ISIS@MACH ITALIA

Papers for MRF1 Access Panel

Direct Access

9 & 10 October 2023

Important Note from the User Office

Dear Panel Member,

If you have not already done so, please complete your taxi and accommodation requirements online

Accommodation

Accommodation is arranged for you at *The Grand Hotel Villa Torretta* (circle A on the map, page 3) in Via Milanese 3, Sesto San Giovanni, MI. You can reach the IM@IT – University of Milano Bicocca in Piazza della Scienza 1 (circle B on the map, page 4):

- by taxi.
- with a 30-minute walk.
- by the underground line 5 starting from "Bignami "to "Bicocca" stop.
- by tram line 31 from "Parco Nord Clerici" to "Bicocca" stop.

Transport to and from Milan

We are not able to pre-book taxis from Milan airport or train Station to *The Grand Hotel Villa Torretta,* so you will therefore need to take your own taxi. Please ensure you obtain a receipt from the driver if you wish to reclaim this cost.

If you do experience problems in finding a taxi, please do not hesitate to contact us by phone on **0039 3466153289** or **0039 3392759852**.

MEDIUM-RANGE FACILITY 1 ACCESS PANEL
ROUND Direct Access 23-2, 9 & 10 October 2023 *https://isismachitalia.eu/about/*

TIMETABLE

Sunday 8 October

19:00 *Meeting point at The Grand Hotel Villa Torretta (see A in Bicocca MAP, page 3)*

19:30 *Dinner at San Glicerio 1 Restaurant (see C in Bicocca MAP, page 3)*

Monday 9 October

08:30 Meeting point: reception hall of *The Grand Hotel Villa Torretta.* Transport to the Building U1 *(see B in Bicocca MAP, page 3)*.

09:00 – 10:00 MAP Chair Meeting Conference Room T010 Building U1

Refreshments will be available from 08:30.

10:00 – 12.00 MAP Meeting Conference Room T010 Building U1

12:00 – 13:30 *Lunch at "Tutto St'Orto" Restaurant (see D in Bicocca MAP, page 3) and group photo.*

13:30 – 18:30 MAP Meeting Conference Room T010 Building U1

Refreshments will be available at 15:30 and 17:00

18:45 Transport departs outside Building U1 for dropping off at *Primevo Restaurant (see E in Bicocca MAP, page 3).*

19:15 *Dinner at Primevo Restaurant (see E in Bicocca MAP, page 3).*

Tuesday 10 October

08:30 Meeting point: reception of *The Grand Hotel Villa Torretta*. Transport to the building U1.

Refreshments will be available at 08:30 and 10:00.

09:00 – 12:30 MAP Meeting Conference Room T010 Building U1

12:30 – 14:00 *Lunch at "Sottosopra" Restaurant (see F in Bicocca MAP, page 3) and group photo.*

MRF1 Management Group

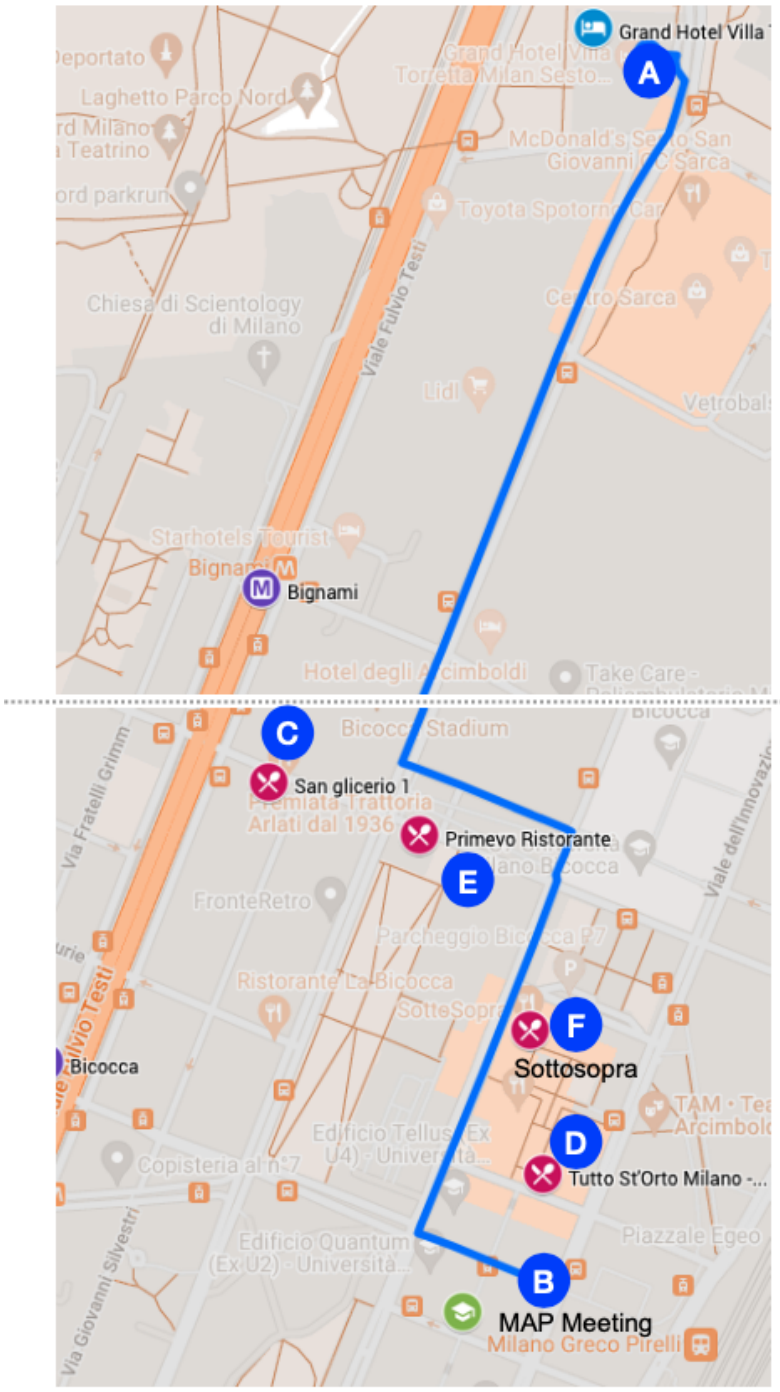
Andreani, Carla	Chair
Parker, Stewart	Vice-Chair
Albani, Giorgia	User Office
Bonini, Massimo	MAP Chair
Romanelli, Giovanni	IM@IT Representative

MAP Members

Bonini, Massimo	Chair	University Florence	ITALY
Parker, Stewart	Secretary	ISIS-STFC	UK
Caciuffo, Roberto	Member	INFN	ITALY
Cazzaniga, Carlo	Member	ISIS-STFC	UK
Faraone, Antonio	Member	NIST	US
Fragneto, Giovanna	Member	ESS	SE
Frost, Christopher	Member	ISIS-STFC	UK
Hyde, Timothy	Member	HRF University of Glasgow	UK
Romanelli, Giovanni	IM@IT Representative	University Tor Vergata	ITALY
Salzmann, Christoph	Member	University College London	UK
Scherillo, Antonella	Member	ISIS-STFC	UK
Telling, Mark	Member	ISIS-STFC	UK
Albani, Giorgia	User Office	University Milano-Bicocca	IT

BICOCCA MAP

A: Gran Hotel Villa Torretta (Via Milanese, 3, 20099 Sesto San Giovanni MI) - Accommodation
B: MAP Meeting (Piazza della Scienza 1, Building U1, Conference Room T010)
C: San Glicerio 1 Restaurant (Viale San Glicerio 6) – Dinner 8th October
D: Tutto St'Orto (Piazza della Trivulziana, 2, 20126 Milano MI) – Lunch 9th October
E: Primevo Restaurant (Viale Sarca, 198, 20126 Milano MI) - Dinner 9th October
F: Sottosopra Restaurant (Viale Piero and Alberto Pirelli 16) – Lunch 10th October



ISIS@MACH ITALIA
Hub for the analysis of composite materials

Guidelines for the Medium Range Facilities Access Panel (MAPs)
isismachitalia.eu

MAP is an external independent peer review panel responsible for the selection and scientific evaluation of the proposals submitted by potential users requesting access to the suite of MRFs. The panel is composed of 9-13 independent members with a collective scientific knowledge of complex materials and interphases (CMI) and atomic-to-micro analysis and technology, covering the science areas supported by the IM@IT Research Infrastructure.

The ***Remit*** of the MAP is:

- To recommend to the Executive Director of IM@IT a balanced science program based upon the criteria of ***scientific excellence and timeliness*** (all within the bounds of technical feasibility and safety implications) and, where appropriate, the potential economic impact and contribution to knowledge exchange and transfer.
- To comment on the appropriateness of the number of instrument days requested for the experiments proposed.
- To identify after each proposal round scientific trends and facility development issues (including software development) which are of relevance to the MRF instrument.

The MRFs suite includes:

AFM	The Nanowizard II – JPK-Bruker
AFM Raman	Raman Spectrometer XploRA Plus
Confocal Microscope 1	Laser Scanning Confocal Microscope Leica TCS SP2
Confocal Microscope 2	Laser Scanning Confocal Microscope Leica TCS SP8
Confocal Microscope 3	Laser lines at 454, 488, 514, 635 nm
Cryogenic Electron Microscopy	CEM in Transmission, model Thermo Scientific™ Glacios™
Dynamic Mechanical Analyzer	DMA Star Systems – Mettler Toledo
FIB-SEM GAIA 3	FIB-SEM with simultaneous milling and EBSD
FT-IR Nexus	Nicolet Nexus 870
FT-IR Nicolet	Endowed with LightDrive Optical Engine components
Fluorescence Microscopy	BX51 microscope
Mass Spectrometer 1	Rapiflex™ MALDI Tissuetyper™
Mass Spectrometer 2	Orbitrap Fusion Tribrid mass spectrometer
NMR 600 MHz	Bruker Avance III 600 MHz NMR
Raman Confocal Microscope	Microscope inViaTM QontorTM model
SAXS GISAXS	Xenocs XEUSS 3.0
SAXS WAXD	Saxspace Anton-Paar
SEM FEI	SEM FEI QUANTA 200
SEM LEO SUPRA	SUPRA 35 Field Emission SEM
SEM ZEISS GEMINI	FEG-SEM with a nominal resolution of 1.2 nm

SEM ZEISS SIGMA	Scanning electron microscope with field-emission source
SEM with correlative AFM	SEM system with EDS-SPM
Spectrofluorimeter	Varian Eclipse Spectrofluorimeter
TEM FEI	LaB6 source (120 kV) and BF detector and FEI Eagle
TEM High Resolution	ThermoFisher Talos F200X
TEM JEOL	JEOL JEM 2100 Plus with a LaB6 emitter
X-Ray diffractometer	Rigaku SmartLab SE
XRD TOMOGRAPHY	RIGAKU Nano3DX

If a panel member is unable to attend, he is requested to notify the panel secretary as soon as possible in advance so that a substitute member can be found if necessary. Written comments are expected from non-attending members. Panel members who are unable to attend in person may attend by Zoom video conference.

Reasonable travel and subsistence costs are reimbursed to members when attending panel meetings.

Panel Working Method and Protocols

MAP members are provided with all the proposals for their panel in advance of the meeting. Each proposal will be assigned to two MAP members who act as primary speakers to give their assessment of the proposal at the MAP meeting. Proposals are then discussed by the MAP, considering any technical issues raised by IM@IT representatives. The MAP should arrive at a grade for each proposal (see ***Proposal Grading and Prioritisation*** in Table 1). The MAP will be notified of the number of days available to them for each instrument being considered, and panels should recommend, based on the days available, which proposals should be awarded instrument time and the number of days to be given.

Comments should be provided by panels to be fed back to proposers, particularly where instrument time is not awarded or significantly reduced.

Panel members should highlight any proposal where they consider there to be ethical issues. This may include unethical practice (*e.g.,* plagiarism), but also where additional protocols may be necessary before an experiment can be allowed to take place (*e.g.,* use of biological material, material from human subjects, genetic modification, etc).

Panel Code of Practice

Panel members should declare all conflicts of interest. Members are expected to leave the room during consideration of these proposals and if proposals from their own departments are being considered. The MAP Chair is responsible for deciding on potential conflicts of interest where these are raised. All

papers relating to the proposal review are to be treated as ***confidential*** and should not be discussed outside the meeting; panel discussions and results of the peer review process should also be kept confidential.

Proposal grading and prioritisation

The MAP will peer review all the submitted proposals and agree on an overall grade for each proposal. The grades and an indication of the associated definitions and expected outcomes are given in the table below. Proposals which are scientifically or technically flawed should be rejected and marked X.

<table>
<tr><th>Grade</th><th>Expected Review Outcome</th><th>Definition – for guidance</th></tr>
<tr><td>10</td><td rowspan="2">Instrument time allocation is essential</td><td rowspan="2">Outstanding, World class</td></tr>
<tr><td>9</td></tr>
<tr><td>8</td><td rowspan="2">Instrument time allocation is recommended</td><td rowspan="2">Excellent</td></tr>
<tr><td>7</td></tr>
<tr><td>6</td><td rowspan="2">Instrument time allocation is possible</td><td rowspan="2">Good</td></tr>
<tr><td>5</td></tr>
<tr><td>4</td><td rowspan="4">Instrument time allocation should not be made</td><td rowspan="2">Fair</td></tr>
<tr><td>3</td></tr>
<tr><td>2</td><td>Uncompetitive</td></tr>
<tr><td>1</td><td>Unsatisfactory</td></tr>
<tr><td colspan="3"></td></tr>
<tr><td>R</td><td>Panel would like to see a resubmission with panel comments addressed</td><td>Resubmit</td></tr>
<tr><td>X</td><td>Panel do not want to see a resubmission</td><td>Reject</td></tr>
</table>

IM@IT Access Mechanisms

1. **Access to Medium Range Facilities**

Direct access is suitable for all service, training and instrument time using MRF1 equipment. Proposals are submitted to two calls for proposals each year with deadlines in April and October each year. All direct access proposals are peer reviewed by the (MAP). Proposals which are allocated beamtime are scheduled by ISIS scientists normally between 2 months and 4 months after the proposal deadline.

2. **Industrial Collaborative Program**

An Industrial Collaborative program (ICP) is also offered. It is a fast-track route for industries based in Italy to use MRF1 equipment for service, training, and instrument time. Requests of time using the ICP route can be submitted at any time. Requests are reviewed by a small panel with appropriate

expertise, including the MAP chair, under strict confidentiality rules. Industrial users may also buy beamtime directly by contacting the IM@IT User Office (useroffice@isismachitalia.eu).

IM@IT User Office
revised: May 17th, 2023

Suite of MRF1 Instrument

MRF1 Instrument - AFM Raman

GPno	*Applicant PI*	*Country*	*Req Days*	*Title*
2023047	Pintacuda Dr F	ITALY	3	*Characterisation of the stress field in SiC MOSFET by means of Raman spectroscopy*
2023054	Turina Mrs V	ITALY	1	*Characterization of collagen and both tanning and colouring materials on leather artefacts from Museo Egizio by AFM-Raman measurements*
2023061	Aglietto Dr I	CZECH_REPUBLIC	4	*Graphene-based thermoplastic composites: AFM Raman characterization*
2023065	Strolin Dr L	SPAIN	2	*Understanding ritual practices in Neolithic Saudi Arabia using Raman spectroscopy on horn sheaths from Mustatils*
2023074	Saliu Dr F	ITALY	2	*Nanofibers from textiles: determining photo-degradation induced physicochemical modification of natural and synthetic fibers surface by AFM-RAMAN*
Proposals		Total Requested Days:	6	

MRF1 Instrument - Confocal Microscope 3

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2023068	Putignano Dr O	ITALY	1	*Measurements of dye uniformity in IFOx sensor oxygen sensing element using Confocal microscope*
2023075	Senesi Professor R	ITALY	2	*Confocal microscopy training for MSci students in Physics*
2023082	Musa Dr Maya	ITALY	1	*Confocal Microscopy on meteorite samples, within a multimodal study*
2023088	Tordi Mr P	ITALY	2	*Study of the internal structure of alginate fibers crosslinked with different cations by confocal laser microscopy*
Proposals	Total Requested Days:		6	

MRF1 Instrument - Dynamic Mechanical Analyzer

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2023064	Foglia Dr F	UNITED_KINGDOM	4	*Characterization of recycled perfluorosulfonic acid membrane for a circular hydrogen economy*
2023086	Foglia Dr F	UNITED_KINGDOM	4	*Characterization of recycled perfluorosulfonic acid membrane for a circular hydrogen economy*
Proposals		Total Requested Days:	8	

MRF1 Instrument - FIB-SEM GAIA 3

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2023081	Capitani Professor G	ITALY	3	*Preparation and Study of TEM lamellae of CaREE-flourcarbonates*
Proposals		Total Requested Days:	3	

MRF1 Instrument - FT-IR Nexus

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2023053	Turina Mrs V	ITALY	1	*Characterization of collagen and both tanning and colouring materials on leather artefacts from Museo Egizio by FT-IR measurements*
Proposals	Total Requested Days:		1	

MRF1 Instrument - Fluorescence Microscopy

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2023073	Senesi Professor R	ITALY	2	*Fluorescence microscopy training for MSci students in Physics*
2023079	Maggioni Professor D	ITALY	3	*Fluorescence microscopy characterization of MULtimodal Anticancer Nanohybrids (MULAN)*
2023089	Torelli Dr M	UNITED_STATES	2	*Characterization of Nitrogen-Vacancy Centers for Improved Quantum Sensing*
Proposals		Total Requested Days:	7	

MRF1 Instrument - NMR 600 MHz

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2023069	Romanelli Dr G	ITALY	2	*Training on the use of NMR spectroscopy to characterize phantom materials for neutron therapy*
Proposals	Total Requested Days:		2	

MRF1 Instrument - Raman Confocal Microscope

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2023085	Resta Dr C	ITALY	2	*Training for Confocal Raman Microscopy on Membrane-electrode assembly components*
Proposals	Total Requested Days:		2	

MRF1 Instrument - SAXS GISAXS

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2023051	Cianchi Prof A	ITALY	2	*GISAXS characterization of cathodes for photoinjectors*
2023052	Turina Mrs V	ITALY	2	*Characterization of collagen and both tanning and colouring materials on leather artefacts from Museo Egizio by WAXS/SAXS/USAXS measurements*
2023066	Strolin Dr L	SPAIN	2	*Understanding ritual practices in Neolithic Saudi Arabia using SAXS on horn sheaths from Mustatils*
2023071	Baumer Professor L	SWITZERLAND	2	*Analysis of nails provided by different antique shipwrecks in the Mediterranean using SAXS*
2023072	Moglianetti Dr M	ITALY	2	*Cerium oxide nanoparticles: SAXS analyses for surface properties engineering*
2023076	Moglianetti Dr M	ITALY	2	*Cerium oxide nanoparticle's growth process: SAXS measurements during synthesis*
2023078	Iberi Mr A	ITALY	2	*Soap formulations: investigation of the relationship between structural properties and their stability and performance*
2023083	Sacco Dr P	ITALY	2	*Investigation of the architecture of agarose-based hydrogels prepared by controlled rate of cooling - AGAROCOOL*
2023084	Resta Dr C	ITALY	2	*Training for SAXS on Membrane-Electrode assembly components*
2023087	Brasili Dr F	ITALY	3	*Effective interactions and phase behavior of PNIPAM-PNIPMAM copolymer microgels*

Proposals Total Requested Days: 21

MRF1 Instrument - SAXS WAXD

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2023058	Grozdanov Prof A	MACEDONIA	3	*SAXS WAXD structural analysis of polyninylalcohol/polyacrylic acid/MXenes nanocomposites*
2023060	Sedlarik Prof V	CZECH_REPUBLIC	3	*Innovative sustainable inks for wearable sensors: structural characterization by SAXS/WAXD*
2023062	Aglietto Dr I	CZECH_REPUBLIC	3	*Graphene-based thermoplastic composites: structural analysis by SAXS/WAXD*
2023091	Marcucci Dr G	UNITED_KINGDOM	1	*Unlocking the structure and composition of a historical silver coin using Wide Angle X-ray Diffraction in combination with Muon and Neutron Techniques*
Proposals		Total Requested Days:	10	

MRF1 Instrument - SEM FEI

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2023057	Grozdanov Prof A	MACEDONIA	2	*SEM FEI morphological analysis of polyninylalcohol/polyacrylic acid/MXenes nanocomposites*
2023059	Sedlarik Prof V	CZECH_REPUBLIC	2	*Innovative sustainable inks for wearable sensors: morphological characterization by SEM FEI*
2023063	Aglietto Dr I	CZECH_REPUBLIC	2	*Graphene-based thermoplastic composites: morphological characterization by SEM FEI*
2023077	Amazio Miss P	ITALY	2	*Morphological characterization of sustainable by design water and oil repellent biobased textile coatings*
Proposals		Total Requested Days:	8	

MAP Instrument SEM ZEISS SIGMA

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2023067	Putignano Dr O	ITALY	1	*Measurements of nanofibers distribution in IFOx sensor oxygen sensing element using SEM techniques*
Proposals	Total Requested Days:		1	

MRF1 Instrument - SEM with correlative AFM

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2023045	Pintacuda Dr F	ITALY	2	*Characterisation of the degree of damage by neutron induced single-event burnout failure in SiC MOSFET by SEM measurements*
2023048	Sbardella Dr D	ITALY	2	*Characterisation of surgically removed vitreous humor samples by SEM measurements*
2023070	Baumer Professor L	SWITZERLAND	3	*Analysis of nails provided by different antique shipwrecks in the Mediterranean using SEM-EDS*
2023092	Tordi Mr P	ITALY	2	*Electrostrictive properties of Alginate-based composites including reduced graphene oxide and metal-based nanostructures*
Proposals		Total Requested Days:	9	

MRF1 Instrument - TEM FEI

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2023049	Sbardella Dr D	ITALY	2	*Characterisation of surgically removed human vitreous samples by TEM measurements*
2023055	Sedlarik Prof V	CZECH_REPUBLIC	1	*Innovative sustainable inks for wearable sensors: analysis of fillers morphology by TEM FEI*
2023056	Grozdanov Prof A	MACEDONIA	2	*Analysis of filler spatial distribution by TEM FEI in polyninylalcohol/polyacrylic acid/MXenes nanocomposites*
Proposals		Total Requested Days:	5	

MRF1 Instrument - X-Ray diffractometer

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2023046	Pintacuda Dr F	ITALY	4	*Characterisation of the stress field in SiC MOSFET by means of X-Ray diffraction*
Proposal	Total Requested Days:		4	

MRF1 Instrument - XRD TOMOGRAPHY

GPno	*Applicant*	*Country*	*Req Days*	*Title*
2023044	Pintacuda Dr F	ITALY	4	*Characterisation of the degree of damage by neutron induced single-event burnout failure in SiC MOSFET by means of X-Ray tomography*
2023050	Sbardella Dr D	ITALY	3	*Characterisation of surgically removed human vitreous samples by X-Ray tomography*
2023080	Paladini Dr G	ITALY	4	*X-ray diffraction tomography to study the effect of the application of phosphate-based coatings on the emission of ionizing radiations of lithotypes used as building materials*
2023090	Marcucci Dr G	UNITED_KINGDOM	1	*Unlocking the structure and composition of a historical silver coin using XRD Tomography in combination with Muon and Neutron Techniques*
Proposals		Total Requested Days:	12	

AFM Raman

AFM Raman

Experiment Proposal

Experiment number GP2023047

Principal investigator	Dr Francesco Pintacuda, STMicrocelectronics, ITALY
Co-investigator (*)	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Roberto Senesi, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Carla Andreani, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Characterisation of the stress field in SiC MOSFET by means of Raman spectroscopy
MRF Instrument	**AFM Raman** — **Days requested:** 3
Access Route	Direct Access — **Previous GP Number:** -
Science Areas	Energy, ICT, Materials, Physics — **DOI:** -
Sponsored Grant	None — **Sponsor:** -
Grant Title	- — **Grant Number:** -
Start Date	- — **Finish Date:** -
Similar Submission?	-
Industrial Links	STMicroelectronics

Non-Technical Abstract

We propose to perform the stress field characterisation of SiC MOSFETs devices, already irradiated with fast neutron on the ChipIR beamline, using the AFM Raman instrument operating at the University of Rome Tor Vergata Unit of IM@IT. In this study we wish to perform residual stress analysis of survived SiC MOSFETs from neutron-induced SEBs and compare results with independent measurements using X-Ray diffraction, which we requested in a separate proposal. The degree of damage by neutron induced SEBs failure on SiC occurred after the ChipIr neutron irradiation will be studied by means of SEM measurements and X-Ray tomography data. All the physical quantities inferred in this study have a direct impact on the understating of the mechanisms triggering SEBs in SiC power MOSFETs.

Publications

Pintacuda et al., Prototyping and characterization of radiation hardened SiC MOS structures, 2019 European Space Power Conference (ESPC).
F. Principato et al., Sensors 20 (2020), 3021; F. Principato et al., Sensors 21 (2021), 5627.
AJ Allen, MT Hutchings, CG Windsor, C Andreani, Neutron diffraction methods for the study of residual stress fields, Advances in Physics, 34, 445-473 (1985).

ISIS neutron and muon source — **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Science and Technology Facilities Council
ISIS Neutron and Muon Source

Sample record sheet

Principal contact	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
MRF Instrument	**AFM Raman** — **Days Requested:** 3
Special requirements:	

SAMPLE

Material	SiC	-	-
Formula	SiC	-	-
Forms	Solid		
Volume	0.004 cc		
Weight	12.84 mg		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	293 - K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Characterisation of the stress field in SiC MOSFET by means of Raman spectroscopy

1. Background and Context

Silicon carbide (SiC) is a IV–IV compound material with unique physical and chemical properties. The strong chemical bonding between Si and C atoms gives this material very high hardness, chemical inertness, and high thermal conductivity [1]. As a semiconductor, SiC exhibits a wide bandgap, high critical electric field strength, and high saturation drift velocity. Wide-bandgap semiconductors promise high tolerance to extreme environments, such as ionizing radiation, energetic particles, high and low temperatures. Both n- and p-type control across a wide doping range is relatively easy in SiC; this makes SiC exceptional among wide bandgap semiconductors. The ability of SiC to form silicon dioxide (SiO_2) as a native oxide is an important advantage for device fabrication. Because of these properties, SiC is a promising semiconductor for high-power and high-temperature electronics [2-4] both in space and at ground level. In comparison to silicon, SiC is a superior material thanks to its higher breakdown field and thermal conductivity. For terrestrial applications, power semiconductor devices like SiC metal–oxide–semiconductor field-effect transistor (SiC MOSFET) or insulated-gate bipolar transistors (IGBTs) [5] suffer of single-event burnout (SEB) failure when irradiated by high-energy neutrons [6] which constitute the 97% of the total [7] flux of high-energy particles impinging on such device at sea level. Experiments of neutron-induced SEBs in SiC MOSFET at high-voltage have shown the formation of cracks at the device surface [8] as shown in Figure 1.

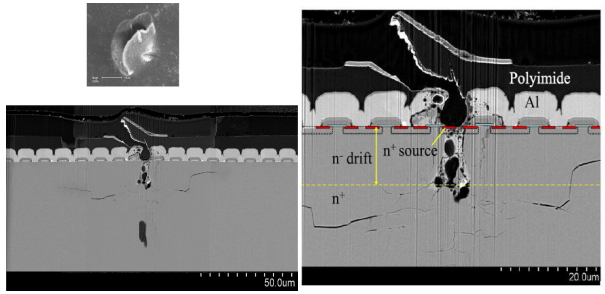

Figure 1: (Left panel) Cross-sectional SEM image of SEB damage for a SiC MOSFET. (Right panel) Magnified view of the damage and device structure [8].

We plan to perform non-destructive characterisations of damaged and survived SiC MOSFETs, following the neutron induced SEBs by fast neutron test at the ChipIr beamline, using scanning electron microscopy (SEM) and X-ray computed topography (XCT); in addition, the stress field [9] will be studied using the X-ray diffraction (XRD) and Raman spectroscopy. To this aim by four distinct proposals, we have requested the use of SEM with correlation AFM, XRD Tomography (for XCT), X-Ray Diffractometer, and the AFM Raman. All the instrumentation is available at IM@IT and operating at the Universities of Rome Tor Vergata and Milano Bicocca, and IPCB-CNR Units.
Aim of this proposal is to perform a residual stress analysis [9] of neutron induced SEBs in SiC MOSFETs using the Raman Spectroscopy, exploiting the relationship between the stress and the relative Raman frequency shift [8]. The stress field will be also independently measured using the high-resolution X-ray diffraction (XRD) instrument and requested in a separate proposal. The SEBs damage in SIC MOSFETs will also be characterised through SEM and XCT. Two separate proposals have been submitted for using SEM-EDS (University of Rome Tor Vergata Unit) and the XRD Tomography (IPCB-CNR Unit).

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Science and Technology Facilities Council
ISIS Neutron and Muon Source

2. Proposed experiment

We aim to measure the stress field in n. 5 as manufactured and n. 5 survived SiC MOSFETs which not undergo to neutron induced SEBs during the test performed at the ChipIr beamline by means AFM Raman, located at the University of Rome Tor Vergata Unit. Such strains will be further measured by independent High Resolution X-ray diffraction following the analysis procedure reported here [8].

3. Justification of experimental time requested

Both as manufactured and survived SiC MOSFETs after neutron induced SEBs on ChipIr have dimensions of about 4mm x 5mm and a thickness of about 200 µm.
We aim to measure n. 5 as manufactured and n. 5 survived SiC MOSFETs by neutron induced SEBs using the AFM Raman instrument with a visible laser source (532 nm in wavelength), a spectral resolution of 1.0 cm^{-1}. The strain will be mapped with a spatial resolution of 0.5 mm. We request 3 days of instrument time which account also for setup time and calibration time.

4. References

[1] Harris, G.L. (1995) Properties of Silicon Carbide, INSPEC.
[2] Davis, R.F., Kelner, G., Shur,M. et al. (1991) Proc. IEEE, 79, 677.
[3] Ivanov, P.A. and Chelnokov, V.E. (1992) Semicond. Sci. Technol., 7, 863.
[4] Morkoç, H., Strite, S., Gao, G.B. et al. (1994), J. Appl. Phys., 76, 1363.
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Experiment Proposal

Experiment number GP2023054

Principal investigator	Mrs Valentina Turina, Fondazione Museo Antichità Egizie, ITALY
Co-investigator (*)	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Carla Andreani, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Lucy Skinner, University of Northampton and the British Museum, UNITED_KINGDOM
Co-investigator	Dr Robert Robinson, University of Wollongong, AUSTRALIA
Co-investigator	Professor Salima Ikram, American University in Cairo, EGYPT
Co-investigator	Miss Giulia Pallottini, Fondazione Museo Antichità Egizie, ITALY
Co-investigator	
Experiment title	Characterization of collagen and both tanning and colouring materials on leather artefacts from Museo Egizio by AFM-Raman measurements
MRF Instrument	**AFM Raman** **Days requested:** 1
Access Route	Direct Access **Previous GP Number:** -
Science Areas	Cultural Heritage, Materials, Physics **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	Fondazione Museo Egizio

Non-Technical Abstract

Within the Museo Egizio collection there are 200 precious and unique leather artefacts belonging to different historical periods including the Old Kingdom, New Kingdom, Roman and Byzantine eras. Hence, it is paramount to understand degradation mechanism of ancient leather probably related to the way the skins were prepared and made durable. The proponents aim to study by WAXS/SAXS/USAXS the assembly and orientation of the collagen fibrils in the samples and extend by means of FT-IR and Raman spectroscopy measurements its characterization and both tanning and colouring materials found in ancient leather. In the present proposal, we wish to measure the intra- and inter- molecular vibrational spectra of collagens as well as both the tanning and colouring materials components, most found in ancient leather using Raman spectroscopy on the AFM Raman instrument.

Publications

G. Romanelli, et al., “Neutron-Enhanced Information on the Laboratory Characterization of Ancient Egyptian Leathers...”, Information, 2022, 13, 467
G. Pallottini, Graduate Thesis, "La coperta Provv.5062 del Museo Egizio di Torino: studio, restauro e valorizzazione" (2021).

ANSTO Reactor

Brief abstract

Two other measurements for performing 2D/3D neutron imaging and neutron USANS have been scheduled to DINGO and KOOKABURRA neutron beamlines at ANSTO (Australia), respectively.

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Sample record sheet

Principal contact	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
MRF Instrument	**AFM Raman** **Days Requested:** 1
Special requirements:	

SAMPLE

Material	Leather	-	-
Formula	Collagen	-	-
Forms	Solid		
Volume	1 cc		
Weight	1 mg		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Characterization of collagen and both tanning and colouring materials on leather artefacts from Museo Egizio by AFM-Raman measurements

1. Background and Context

The collection of the Museo Egizio (Turin) houses over 200 leather artifacts belonging to different historical periods, including the Old Kingdom, New Kingdom, Roman and Byzantine eras. Leather is the main material and the only common element of these objects. Leather was used throughout the entire society, from low to high status and often subject to a variety of uses, from decorative to intense use. Ancient leather presents a heterogeneous composition of both organic and inorganic materials that show an evident reactivity. Its proper preservation remains challenging as some aspects of its chemical composition, degradation and effectiveness of conservation treatments are still not fully understood. Archeologists and conservators were able to identify, through the constant conservative monitoring of the artifacts, different types of degradation and, above all, a correspondence between their dating and the type of documented deterioration. Indeed, the different types of degradation are probably related to the way the skins were prepared and made durable. Of particular concern for the collection of Museo Egizio (Turin) is that the skin processing method (including any coloring treatments) and the substances used to make it more durable are not known. Not so many processes are attested for this period [2], and the substances that were used to treat the skin and the likely connection with the types of deterioration that are documented are closely linked to collagen, the most important fibrous protein. Collagen is the principal protein constituent of a wide variety of connective tissues in animals. Its structure has been investigated extensively by electron microscopy and by diffraction techniques using X-rays and neutrons [3-8]. Recently [9], a characterization of Egyptian leather samples was completed by combining non-destructive techniques, including surface probes (X-ray fluorescence, Raman scattering, and scanning electron microscopy enhanced by X-ray energy spectroscopy) and neutron-based bulk techniques (inelastic and deep-inelastic neutron scattering).

The proponents aim to study by wide/small/ultra small angle X-ray scattering (WAXS/SAXS/USAXS) the assembly and orientation of the collagen fibrils in the samples already investigated in Ref. [9], and by distinct proposals perform a complementary characterization using both Fourier-transform infrared spectroscopy (FT-IR) and Raman spectroscopy (AFM-Raman). These spectroscopic techniques will be used to study the intra- and inter- molecular vibrational spectra of collagens as well as both the tanning and colouring materials components, most found in ancient leather [10]. Hence, we propose to use the SAXS GISAXS, FT-IR Nexus, and AFM Raman instrument operating at the CSGI-Unifi and the Univ. Tor Vergata Units of in the suite of IM@IT. Further characterization on the same samples will be done by neutron imaging (DINGO beamline) and USANS (Kookaburra beamline) at the Australian Centre for Neutron Scattering (ANSTO).

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2. Proposed experiment for AFM-Raman

In the present proposal, we wish to measure the degree of assembly of the collagen fibrils of n. 6 ancient leather artifacts (same samples that will be measure at ANSTO) using Raman measurements on the AFM-Raman instrument. Results from AFM-Raman measurements will be compared and extended by WAXS/SAXS/USAXS and FT-IR widely used for the characterization of collagen and both tanning and colouring materials found in ancient leather as reported in this work [10], which will be submitted in separate proposals.

3. Justification of experimental time requested for AFM-Raman

We aim to measure n. 6 ancient leather artifacts (same samples that will be measure at ANSTO) on the AFM-Raman instrument in the 3100-350 cm^{-1} range, averaging at least ten acquisitions per sample. Hence, we request one day of instrument time including set-up and calibration time.

4. References

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Experiment Proposal

Experiment number GP2023061

Principal investigator	Dr Ivano Aglietto, GrapheneUP SE, CZECH_REPUBLIC
Co-investigator	Dr Gennaro Gentile, IPCB CNR, ITALY
Co-investigator	Dr Marino Lavorgna , CNR, ITALY
Co-investigator (*)	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Graphene-based thermoplastic composites: AFM Raman characterization
MRF Instrument	**AFM Raman** **Days requested:** 4
Access Route	Direct Access **Previous GP Number:** no
Science Areas	Engineering, Materials **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	GrapheneUP SE, Studeněves 13, 273 79 Studeněves, Czech Republic
Non-Technical Abstract	The proposal is addressed to perform the AFM Raman characterization of graphene composites based on polyethylene, polypropylene and polyamide produced by film extrusion, injection moulding and fabric yarn extrusion. The aim is to get insights in the interfacial interactions of the 2D filler with the polymeric matrix and to correlate the preparation approaches to the final properties of the materials. In distinct experiments, the morphological characterization by SEM FEI and the structural characterization by SAXS/WAXD of the samples is requested. All requested characterization will contribute to have a clear understanding of filler distribution at different length-scale, by controlling the chemistry of interfaces through a fine functionalization of the filler realized by GrapheneUp.
Publications	-

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
MRF Instrument **AFM Raman** **Days Requested:** 4
Special requirements:

SAMPLE

Material	few layers graphene (FLG), 3 samples with different functionalization	FLG composites with polyethylene (PE), polypropylene (PP), polyamide (PA) realized by film extrusion, injection moulding and fabric yarn extrusion (9 samples)	-
Formula	C	FLG + PE; FLG + PP; FLG + PA	-
Forms	Solid	Solid	
Volume	0.100 cc	1 cc	
Weight	100 mg	1000 mg	
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	300 - K	300 - K	-
Pressure Range	- mbar	- mbar	-
Magnetic field range	- T	- T	-
Standard equipment	None	None	-
Special equipment	N/A	N/A	-

SAFETY

Prep lab needed	No	No	-
Sample Prep Hazards	-	-	-
Special equip. reqs	no	no	-
Sensitivity to air	No	No	-
Sensitivity to vapour	No	No	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	no	no	-
Radioactive Hazards	no	no	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	-

Graphene-based thermoplastic composites: AFM Raman characterization

1. Background and Context

Thermoplastic materials are of interest in industry due to their low cost and ease of processing and recyclability, in addition to other properties such as rigidity and high impact strength. However, plastics degrade very slowly over hundreds of years, and one of the biggest problems today is the waste produced annually by their use and the long-lasting effects that it has on the environment [1]. The graphene integration in thermoplastic polymers may enhanced significantly the materials performance, by contributing significantly toward sustainability (ie through a reduction of manufacts weight) and enhanced recyclability (ie through the improvement of re-processing as well as the performances of the recycled materials). The improvement in the functional and structural properties of graphene-based polymer nanocomposites is intimately associated with the control of the spatial distribution of graphene in the matrix. This improvement is linked to both the filler synthesis and composite processing techniques, as reported in the literature [2]. A second important problem regards the poor interfacial interactions with the polymer matrix, resulting in the poor dispersion of graphene and low load-transfer from matrix to filler, consequently affecting the final performance of the polymer nanocomposite [3,4]. Modification of graphene is achieved by adding functional groups to the surface or edge of graphene through covalent bonding and non-covalent bonding [5].
Owing that, the aim of the proposal is to study, by using the instrument suite of IM@IT, the correlation between the “chemistry” of Few Layers Graphene (FLGs), the filler spatial distribution and the processing parameters related to the main processing technologies such as film extrusion, injection molding and fabric yarn extrusion. The scope is to investigate how the chemical functionalization of FLGs may affect the spatial distribution as consequence of the different technology adopted for the processing of the composites. In particular, AFM RAMAN will provide chemical info about the pristine FLGs and their interface interaction in the several polymeric matrices. Moreover, SAXS/WAXD will contribute to evaluate the orientation of the filler and its aggregation as well as the effect of filler on the crystallinity of the polymeric phase, which both contribute to enhance properties of the resulting composite, whereas SEM FEI will provide info about assembling of nanoplatelets and spatial filler distribution.

2. Proposed experiment

The graphene-based composites will be prepared by GrapheneUP by using different polymer matrix (i.e. polyethylene, polypropylene and polyamide) and FLGs characterized by different functionalization such as dodecyl amine (DA), p-phenylenediamine (PPD) hexamethylene diamine (HMD), dodecyl amine (DA) or silanes groups and alkylsilanes (AS). Different technologies (i.e. film extrusion, injection molding and fabric yarn extrusion) will be used for the production of composites. The sample size will be compliant with the needs of the different characterization techniques. The following characterization will be performed:

- AFM Raman Spectroscopy (Unit NAST Centre - University of Rome Tor Vergata): will be performed on the custom functionalized graphene, and the composite materials.

In distinct proposals we asked to characterize the same samples by SAXS/WAXD and by SEM FEI.

3. Summary of previous experimental proposals or characterisation

No previous experiments have been carried out on these samples

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4. Justification of experimental time requested

We have requested the AFM Raman Spectroscopy (Unit NAST Centre - University of Rome Tor Vergata) to evaluate to obtain chemical info on FLGs and their interface interaction in several polymeric matrices. It is proposed to measure n. 12 samples (9 composites corresponding to three polymeric matrix realized by using three processing technologies, and 3 pristine FLGs) by using a visible laser source (532, 638 and 785 nm in wavelength), a spectral resolution of 1.0 cm-1, and mapping the samples with a spatial resolution of down to the atomic scale, depending on the final geometry of the sample and interaction with source selected.
After discussion with the instrument scientist, we request 4 days of AFM Raman beam time for the characterization of the above-described materials. The foreseen beam time accounts set up and for the data collection on the samples.

References

[1] Jagadeesh, P., et al., (2022), Sustainable recycling technologies for thermoplastic polymers and their composites: A review of the state of the art, Polymer Composites.2022;43:5831–5862.
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[4] Francisco, D.L., et al., (2018) Advances in polyamide nanocomposites: A review, Polym. Compos., 40, 851–870
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Experiment Proposal

Experiment number GP2023065

Principal investigator (*)	Dr Laura Strolin, Institut Català de Arqueologia Clàssica, SPAIN
Co-investigator	Professor Luisa Cifarelli, University of Bologna and INFN-Bologna, ITALY
Co-investigator	Professor Maria Pia Morigi, University of Bologna, ITALY
Co-investigator	Dr Melissa Kennedy, The University of Sydney, AUSTRALIA
Co-investigator	Dr Thomas Hugh, The University of Sydney, AUSTRALIA
Co-investigator	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Roberto Senesi, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Experiment title	Understanding ritual practices in Neolithic Saudi Arabia using Raman spectroscopy on horn sheaths from Mustatils

MRF Instrument	**AFM Raman**	**Days requested:**	2
Access Route	Direct Access	**Previous GP Number:**	-
Science Areas	Cultural Heritage	**DOI:**	-
Sponsored Grant	None	**Sponsor:**	-
Grant Title	-	**Grant Number:**	-
Start Date	-	**Finish Date:**	-
Similar Submission?	-		
Industrial Links	-		

Non-Technical Abstract

Archaeological research in Saudi Arabia is going through a period of intense development that is constantly leading to important discoveries. Mustatils are massive stone structures serving ritual purpose that were built in hundreds in Northwest Arabia 7500 years ago by nomadic pastoral populations. An exceptional category of finds is represented by horn sheaths, made of the outer keratin shell of the horn. Due to its organic protein composition, the sheath is usually not preserved in archaeology and lacks research. To shed light on the horn treatment, desiccation through deliberate heating, colouring and degradation, we propose a surface characterization using Confocal Raman spectroscopy and, in a separate proposal, small-angle X-ray scattering. A surface characterization via vibrational spectroscopy is expected to provide information on the materials applied on the horn outer layers which prevented degradation of keratin, and possibly explaining the sample colouring.

Publications

Neutron-Enhanced Information on the Laboratory Characterization of Ancient Egyptian Leathers: Hydration and Preservation Status, G. Romanelli et al., Information, 13, 10, 2022

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E-platform: No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Laura Strolin, Institut Català de Arqueologia Clàssica, SPAIN		
MRF Instrument	**AFM Raman**	**Days Requested:**	2
Special requirements:			

SAMPLE

Material	Animal horn	-	-
Formula	Keratin	-	-
Forms	Solid		
Volume	5 cc		
Weight	5 g		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Background and Context

Archaeological research in Saudi Arabia is going through a period of intense development that is constantly leading to important discoveries. Namely, previously unknown monumental structures dating back to the Neolithic are being investigated for the first time: the 'mustatils'. Mustatils are massive stone structures serving ritual purpose that were built in hundreds in Northwest Arabia 7500 years ago by nomadic pastoral populations [1-3]. So far, little is known about their culture, economy, and habits. The main finds in mustatils are skulls of selected horned animals (cattle, goat, gazelle), intentionally deposited in specific offering chambers where hearths are also present [2, 4]. Moreover, these faunal remains are the most ancient attestation of domestic cattle and goat in Arabia.

An exceptional category of find is present, the horn sheath, that is the outer keratin shell of the horn. Due to its organic protein composition, the sheath is usually not preserved in archaeology and lacks research. Therefore, the exceptional preservation of horn sheaths in mustatils opens the unique possibility to investigate this material not only for better understanding the ritual universe and technical knowledge of Neolithic nomadic people of ancient Arabia, but also for clarifying the circumstances of sheath desiccation as related to paleoclimatic conditions. In addition, we target possible conservation methods as archaeologic horn is a highly fragile and perishable material.

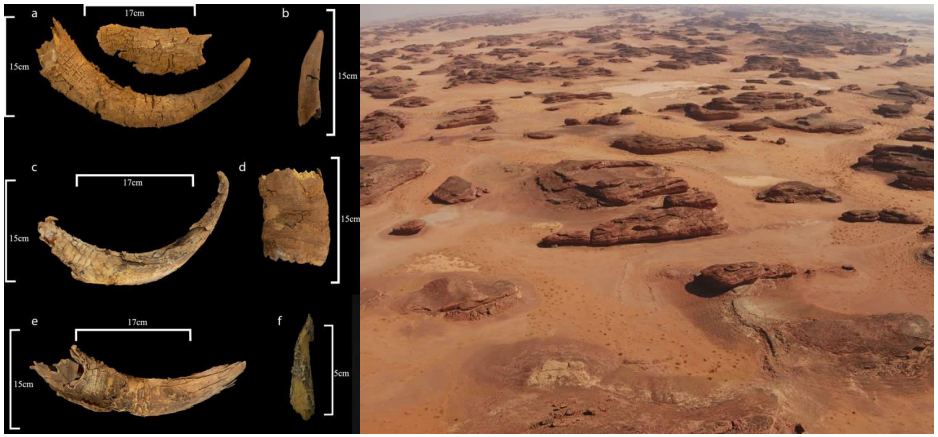

Figure 1: a selection of the horn sheaths found in Mustatil IDIHA-F-0011081 (left); the mustatil in the arid landscape of Northwest Arabia (right).

The research questions motivating this proposal are: were the sheaths treated prior to deposition in the mustatil (as part of the ritual, for preservation purposes) and how? Were some of the sheaths deliberately heated? Why do the sheaths present different colours and levels of degradation? What is their current state of desiccation and degradation?

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Proposed experiment

To answer these questions, we propose a surface characterization of a series of fragments and pieces from a selection of horn sheaths, i.e., smaller portions of the finds in Figure 1 (left), through confocal Raman spectroscopy available at the University of Rome Tor Vergata – IM@IT using the AFM Raman XploRA Plus. Vibrational spectroscopy of the sample surface will provide information on any materials applied on the horn outer layers which prevented degradation of the keratin organic material, as well as shedding light on any desiccation and preservation processes related to such samples and on the reason why different colours are observed. Ancient samples will be compared with modern ones to facilitate the interpretation of the experimental data.

In addition to Raman spectroscopy, through separate proposals, we will request access to the SAXS/WAXD, located at the CSGI-IM@IT Unit, to characterize the aggregation state of individual keratin filaments, providing information on the protein structure and, thus, on the desiccation of the material.

Summary of previous investigations

Mustatils are the object of a wide research programme carried out previously by the University of Western Australia and currently the University of Sydney (Prehistoric AlUla and Khaybar Excavation Project – PAKEP) with the support of the Royal Commission for AlUla. The research focuses on Neolithic mustatils and settlements, as well as on Bronze Age tombs. As such, it aims at enlightening all aspects of ancient societies in the area. It also includes remote sensing, helicopter photography, ground survey, excavation, and material analyses, with a special attention to outreach.

Justification of experimental time requested

We request 2 days of instrument time on the *AFM Raman* MRF located at the Tor Vergata – IM@IT unit, to be used as follows: up to 3 hour of measurements per horn sheath fragment (for a total of about 4 fragments per day) both for the Mustatils finds and for reference horn and keratin samples.

References

[1] Kennedy D. 2017. 'Gates': a new archaeological site type in Saudi Arabia. Arabian Archaeology and Epigraphy 28: 153–74.
[2] Thomas H., Kennedy M., Dalton M., McMahon J., Boyer D. and Repper R. 2021. The Mustatils: Cult and Monumentality in Neolithic north-western Arabia. *Antiquity* 95(381): 605–626.
[3] Abu-Azizeh W., Studer J., Al-Ahmari S., Boyle A., Dausse L., Quartermaine J., Strolin L., Tombret O. and Zazzo A. 2022. The Horn Chamber Mustatil: A Neolithic open-air sanctuary evidencing pastoral nomadic ritual activity in the north-western Arabian Desert (al-ʿUlā [AlUla]). In Foote R., Guagnin M., Périssé I. and Karacic S. (eds.). Revealing Cultural Landscapes in North-West Arabia. Proceedings of the Seminar for Arabian Studies 51,133-156.
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[5] Mattiello S, Guzzini A, Del Giudice A, Santulli C, Antonini M, Lupidi G, Gunnella R. 2022. Physico-Chemical Characterization of Keratin from Wool and Chicken Feathers Extracted Using Refined Chemical Methods. Polymers, 15(1):181

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Experiment Proposal

Experiment number GP2023074

Principal investigator (*)	Dr Francesco Saliu, Universita&039; Milano Bicocca, ITALY
Co-investigator	Dr Massimiliano Clemenza, INFN, ITALY
Co-investigator	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Nanofibers from textiles: determining photo-degradation induced physicochemical modification of natural and synthetic fibers surface by AFM-RAMAN
MRF Instrument	**AFM Raman**
Days requested:	2
Access Route	Direct Access
Previous GP Number:	no
Science Areas	Environment
DOI:	-
Sponsored Grant	None
Sponsor:	-
Grant Title	-
Grant Number:	-
Start Date	-
Finish Date:	-
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

Nowaday Microfibers (MFs) pollution is widespread. Textile washing has been identified as the major source. However, the identification of the factors that influence the MFs release from textiles is underway. Moreover, MFs negative effects on living organisms has been less studied than those associated with spherical particles and very few research has focused on submicrometric fibers (NFs).This may accounted as a significant knowledge gap. The research aim to define the chemo-physical process causing microfiber and nanofiber release, identify the main modification occuring on the fiber surface and suggest technical solution to limit their environmental impacts. Reference material will be submitted to artificial weathering under laboratory controlled condition to mimick different environmental stresses. The MRF instrumentation will be used for the identification of the key chemo-physical modfiication induced by photo-degradation leading to NFs release

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact Dr Francesco Saliu, Universita&039; Milano Bicocca, ITALY
MRF Instrument **AFM Raman** **Days Requested:** 2
Special requirements:

SAMPLE

Material	polymeric microfibers (cellulose, polyethilene, polyesther subjected to artificial weathering) deposided on a glass slide (or we can provide colloidal solution to be despersed on the stub)	cellulose nanofibrillated	Photoaged PET fibers
Formula	CxHxOx (polypropylene with unknow degree of surficial oxidation)	CnHnOn nanofibrillated cellulose with unknow degree of photo-degradation	CnHnOn PET with unknow degree of surface oxidation
Forms	Solid	Solid	Solid
Volume	cc	cc	cc
Weight	50 mg	100 mg	100 mg
Container or substrate	samples can be provided despersed as colloidal solution in a glass vial or deposited onto a glass slide or onto the required stab	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	290 - 310 K	290 - 320 K	- K
Pressure Range	- mbar	- mbar	- mbar
Magnetic field range	- T	- T	- T
Standard equipment	Sample Changer	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	No	Yes	Yes
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	No	No
Sensitivity to vapour	No	No	No
Experiment Hazards	no	-	-
Equipment Hazards	-	-	-
Biological hazards	no	-	-
Radioactive Hazards	no	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Removed By User	Disposed by IS	Disposed by IS

Nanofibers from textiles: determining photo-degradation induced physicochemical modification of natural and synthetic fibers surface by AFM-RAMAN

Background and Context

Recent environmental research highlighted how microfibers (MFs) pollution is widespread. MFs were detected from subsurface oceanic seawater to the deep sea, from the atmosphere to the living organism (Suaria et al 2020). Textile washing has been identified as the major source of MFs. However, the identification of the factors that influence the MFs release from textiles is underway (Saliu et al. 2021). Moreover, the negative effects of MFs on living organisms have been less studied than those associated with spherical particles and very little research has focused on submicrometric fibers (NFs). This may be accounted as a significant knowledge gap in the current literature. The research carried out by the Environmental Chemistry research group at the University of Milano Bicocca aims to define under laboratory conditions the chemo-physical process causing microfiber and nanofiber release and identify the main modification occurring on the fiber surface caused by weathering. This information may help in understanding MFs environmental fate and impacts (including biological interaction and in the development of new technical solutions. Reference materials are submitted to artificial weathering under laboratory-controlled conditions to mimic different environmental stresses and are characterized by employing a wide range of analytical instrumentation available in my department. The research is currently financed by University of Milano-Bicocca funds (FAR and MUSA) and involves several international collaborations (University of Stockholm, University of South Carolina, CNR-ISMAR, University of Foggia)

Proposed Experiment

The AFM-RAMAN MRF instrumentation will be used to add new information regarding the photo-degradation mechanism of fibers in textiles through the identification of the key surface chemo-physical modification, directly on the nanofiber surface Specifically, it is requested the chemical characterization of the surface of polypropylene nanofiber obtained after photodegradation under Xenon lamp with a focus on the recognition of the degree of oxidation and the identification of the main functional groups originated by the chemical photo-oxidation, in relation to the nanofiber morphology (e.g., cracks and pitches). AFM and Raman spectroscopy have been used to characterise plastic particles ith sizes < 100 nm (Fang et al., 2020, Stawikowska and Livingston, 2013). Compared with SEM, AFM can characterize particles in a more comprehensive way (Fu et al., 2020). Coupling Raman spectroscopy affords the nanoscale chemical description of the sample surfaces (Fu and Zhang, 2017). To date, only a few studies applied hybrid AFM techniques (AFM/IR or AFM/Raman) to detect and characterize nano plastics, and none NFs.

Summary of previous instrument time or characterization

Materials to be submitted to the AFM-Raman analysis were already preliminarily characterized by FTIR and Raman, photo-aged, characterized by SEM, DLS, and Mass Spectrometry, and toxicological assayed with different biological models. The nanofiber may be provided as colloidal solutions in water or dispersed onto opportune surfaces (glass slides or stubs). Therefore the sample will result as a collection of several polypropylene nanofibers of different lengths and diameters (DLS showed an average hydrodynamic radius 134 nm) while SEM showed different aggregates (it must be considered the artifacts induced by deposition).

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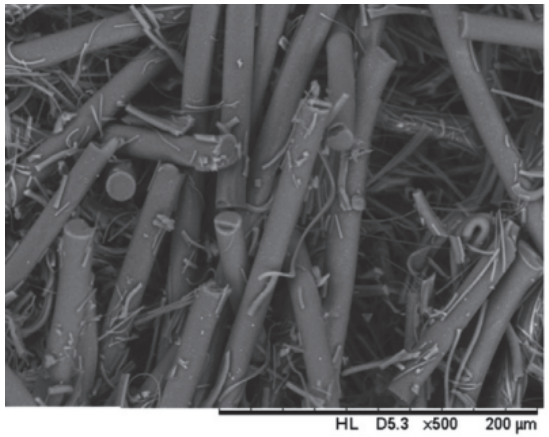
Justification of instrument time request

The request is for 2 days of the AFM-RAMAN instrument time for the collection of a statistically-significant number of Raman signal collection points to characterize the main chemical modification of the material induced by the photo-degradation. We assume about 3 hours for set-up of the experimental parameters on the AFM-RAMAN MRF, up-to 1.5 hours per sample measurement, and an overall number of 10 samples (from 3 different polymeric materials), to be divided between the 2 days requested.



References

G. Suaria et al. Microfibers in oceanic surface waters: A global characterization. Sci Adv. 23, (2020):

F. Saliu et al . The release process of microfibers: from surgical face masks into the marine environment. Environ. Adv.4 (2021)

C. Fang, et al. Identification and visualisation of microplastics/ nanoplastics by Raman imaging : smaller than the diffraction limit of laser? Water Res. (2020), p. 183

W. Fu, W. Zhang Hybrid AFM for nanoscale physicochemical characterization: recent development and emerging applications Small, 13 (2017)

J. Stawikowska, A.G. Livingston Assessment of atomic force microscopy for characterisation of nanofiltration membranes J. Membr. Sci., 425 (2013), pp. 58-70

Confocal Microscope 3

Confocal Microscope 3

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Experiment Proposal

Experiment number GP2023068

Principal investigator (*)	Dr Oscar Putignano, CNR, ITALY
Co-investigator	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Gabriele Croci, University of Milano - Bicocca, ITALY
Co-investigator	Dr Andrea Muraro, CNR, ITALY
Co-investigator	Dr Marco Tardocchi, CNR, ITALY
Co-investigator	Dr Enrico Perelli Cippo, Consiglio Nazionale delle Ricerche, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Measurements of dye uniformity in IFOx sensor oxygen sensing element using Confocal microscope.

MRF Instrument	**Confocal Microscope 3**	**Days requested:**	1
Access Route	Direct Access	**Previous GP Number:**	no
Science Areas	Materials, Medicine, Physics	**DOI:**	-
Sponsored Grant	None	**Sponsor:**	-
Grant Title	-	**Grant Number:**	-
Start Date	-	**Finish Date:**	-
Similar Submission?	-		
Industrial Links	-		

Non-Technical Abstract

The recent COVID-19 pandemics highlighted the need to develop innovative diagnosis tools for lung conditions. A collaboration with clinicians, started during the acute phase of the pandemics, led to the development of a proof-of-concept prototype of a fast, mainstream oxygen sensor called IFOx sensor. One of the key element is represented by its optical sensing element whose geometrical and surface feature impact on the sensor performance. With this experiment we want to evaluate all the geometrical features by performing microscopic measurements in order to improve the optical sensing element design.

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Oscar Putignano, CNR, ITALY		
MRF Instrument	**Confocal Microscope 3**	**Days Requested:**	1
Special requirements:			

SAMPLE

Material	stinless steel, nylon, PtTFPP	-	-
Formula	Fe Ni Cr Nylon PtTFPP	-	-
Forms	Solid		
Volume	1 cc		
Weight	10 mg		
Container or substrate	no	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	270 - 290 K	-	-
Pressure Range	900 - 1100 mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	no	-	-

SAFETY

Prep lab needed	No	-	-
Sample Prep Hazards	no	-	-
Special equip. reqs	no	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	no	-	-
Equipment Hazards	-	-	-
Biological hazards	no	-	-
Radioactive Hazards	no	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	-	-

Structure of the Science Case

Measurements of dye uniformity in IFOx sensor oxygen sensing element using Confocal microscope.

1. Background and Context

The recent COVID-19 pandemics highlighted the need to develop innovative diagnosis tools for lung conditions. A collaboration with clinicians, started during the acute phase of the pandemics, led to the development of a proof-of-concept prototype of a fast, mainstream oxygen sensor called IFOx sensor. Off the shelf oxygen sensors work in side-stream configuration i.e., a sample of gas is spilled from the main airway and analyzed by the sensor. Most medical oxygen sensors rely on a chemical reaction to detect the fraction of oxygen in the sample gas mixture; this leads to aging of the sensing element. Moreover, the side-stream configuration and the fact that the typical reaction time of medical oxygen sensor is of some seconds, make a correlation measurement of the gas flow and oxygen concentration nearly impossible. For these reasons the IFOx sensor is designed to work in main-stream mode i.e., it measures the gas flowing in the totality of the airway. The mainstream configuration allows for seamless correlation of the gas flow and oxygen concentration measurements. The core of the IFOx sensor is an optical sensing element (OSE) based on a metal organic dye called Pt(II)-tetra-pentafluorophenyl-porphyrin (PtTFPP) dye that changes it fluorescence time depending on the oxygen concentration of its surroundings. It is important to notice that the fluorescence quencing of the OSE is not based on a chemical reaction, so ite OSE does not suffer from aging, as it is with electrochemical sensors. To maximize the surface exopsed to the gas and gas permeability the PtTFPP dye is embedded in a mesh of nano fibers obtained with electrospinning technique. Electrosoinning involves an electrostatic field to produce ultrafine fibers from polymer solutions deposited onto a suitable heating element. Electro-spun fibers have an average size of about 100 nm with narrow size distribution. The nanofibers are dyed by dipping into a suitable solution containing the PtTFPP. The uniformity of the dye process is crucial as it ensures light emission uniformity form the OSE.

2. Proposed experiment

The dye is deposited on the OSE at CNR-STIIMA laboratories in Biella. We plan to prepare a set of OSEs to be analyzed to verify the dye uniformity on multiple samples. Moreover, we plan to analyze a sample after extreme usage, using gas flows at least twice the maximum intended value to verify the robustness of the dye.

3. Summary of previous experimental proposals or characterisation

We do not have previous proposals.

4. Justification of experimental time requested

We think that a working day on Confocal Microscope 3 (of University of Milano-Bicocca) is sufficient for the needed characterization. This measurement will be complemented by requesting another working day on SEM ZEISS SIGMA. This request is the subject of another proposal.

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Experiment Proposal

Experiment number GP2023075

Principal investigator	Professor Roberto Senesi, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Laura Fazi, University of Rome Tor Vergata, ITALY
Co-investigator (*)	Dr Francesco Stellato, Università degli Studi di Roma Tor Vergata, ITALY
Co-investigator	Dr Anna Prioriello, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Confocal microscopy training for MSci students in Physics
Training MRF	**Confocal Microscope 3** — **Days requested:** 2
Access Route	Direct Access — **Previous GP Number:** -
Science Areas	Biology and Bio-materials, Materials, Medicine, Physics — **DOI:** -
Sponsored Grant	None — **Sponsor:** -
Grant Title	- — **Grant Number:** -
Start Date	- — **Finish Date:** -
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	We propose a training access to confocal microscopy for MSci students in Physics with curricula in condensed matter and biophysics. This has the aim of providing knowledge and awareness of research infrastructures and instrumentation at the undergraduate level and before MSci thesis, for perspective industrial and public sector users. E-gate for INES at ISIS will be requested for diffraction analyses.
Publications	-

Instruments	**INES**	**Days Requested:** 2
Access Route	Direct Access	**Previous RB Number:**
Science Areas		**DOI:**
Sponsored Grant	None	**Sponsor:**
Grant Title	-	**Grant Number:**
Start Date	-	**Finish Date:**
Similar Submission?		
Industrial Links		

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Sample record sheet

Principal contact	Dr Francesco Stellato, Università degli Studi di Roma Tor Vergata, ITALY		
Training Instrument	**Confocal Microscope 3**	**Days Requested:** 2	
Special requirements:			

SAMPLE

Material	-	-	-
Formula	-	-	-
Forms			
Volume			
Weight			
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	-	-	-
Pressure Range	-	-	-
Magnetic field range	-	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	-	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	-	-	-
Sensitivity to vapour	-	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	-	-	-

Confocal microscope training for MSci students in Physics

Background and Context

Career development of physics students from Tor Vergata leads often to employment in industrial R&D environments (e. g. Leonardo, Thales, Elettronica spa, SONY-Eriksson, Biotech companies etc.). Once part of the industrial environment, alumni are frequently set within industrial programmes whose needs on characterization of materials are an opportunity IM@IT as well as for companies/institutions involved.

In order to align training on access to infrastructures such as IM@IT and the individual career paths there is a need to connect and raise awareness on access during MSci course attendance.

Fulfilling these needs has an added value for all undergraduates, but would add specific importance to those following career paths in the private/industrial sector.

Objectives and summary of previous experimental proposals

The main objective of the proposal is to put in place routine training accesses for MSci students with a transformative use of research instrumentation for teaching purposes.This activity will specifically target to climb the ladder of instrumentation complexity, with a timing tailored before students' theses assignments.

Since 2015 this approach has been proven successful within the Condensed Matter Laboratory course (responsible R. Senesi- see figure below), MSci in Physics, second semester, in bringing more than 25 students as part of the experimental teams in ISIS experiments, 10 of which are now employees in the above mentioned companies.

There is now the opportunity to: 1) temper the complexity of access through IM@IT; 2) extend the access to students attending the Biological Physics Laboratory course (responsible F. Stellato); 3) establish a path followed by a plan of return on investment by engagement with the community of industrial alumni previously involved in the access.

The proposed measurement on the two classes of samples, namely a polymer- carbon nanotube composite and protein microcrystals, convey both training aspects on the identification of interfaces, multi scale morphologies, amorphous/crystalline content, and at the same time provide opportunities to gain an insight into the polymer-nanotube interaction and penetration depths and distribution and to establish methods to discriminate between organic and inorganic crystals, which are currently under investigation in the proponents'research programmes.[Fazi 2023, Stellato 2014]

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Proposed experiment

Request of access to two instruments: confocal and fluorescence microscopies on composite samples, and e-Gate to ISIS. Instrument time of 2 days each for Confocal Microscope 3 and Fluorescence Microscopy is estimated to be sufficient for the purpose of the present proposal.E-gate to INES beamline will be also requested.

References

[Fazi2023] Fazi, L. et al, Molecules 28, 1674 (2023)

[Stellato2014] Stellato, F, *IUCrJ* 1.4, 204-212 (2014)

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Experiment Proposal

Experiment number GP2023082

Principal investigator	Dr Maya Musa, Università di Pavia, ITALY	
Co-investigator (*)	Dr Daniela Di Martino, University of Milano Bicocca, ITALY	
Co-investigator	Dr Margaux Bouzin, Università degli Studi di Milano-Bicocca, ITALY	
Co-investigator	Professor Maddalena Collini, Università degli Studi di Milano Bicocca, ITALY	
Co-investigator	Dr Massimiliano Clemenza, INFN, ITALY	
Co-investigator	Professor Maria Pia Riccardi, Università di Pavia, ITALY	
Co-investigator	Dr Riccardo Rossini, University of Pavia, ITALY	
Co-investigator	Dr Giulia Marcucci, ISIS Neutron and Muon Source, UNITED_KINGDOM	
Co-investigator		
Experiment title	Confocal Microscopy on meteorite samples, within a multimodal study	
MRF Instrument	**Confocal Microscope 3**	**Days requested:** 1
Access Route	Direct Access	**Previous GP Number:** No
Science Areas	Cultural Heritage, Materials	**DOI:** -
Sponsored Grant	None	**Sponsor:** -
Grant Title	-	**Grant Number:** -
Start Date	-	**Finish Date:** -
Similar Submission?	-	
Industrial Links	Planetario e Osservatorio Astronomico G. Giacomotti - Cà del Monte - Cecima (PV), Italy	
Non-Technical Abstract	A confocal microscopy experiment on meteorite samples, has been designed within a multimodal study. The combination of neutron, micro-Raman spectroscopy mapping and SEM/EDS mapping, with images provided by confocal microscopy is expected to play an essential role for the currently ongoing quantitative interpretation of super-resolution thermographic data. Confocal imaging will help developing a multimodal approach for the comprehensive analysis of thin sections, to be subsequently extended and adapted to bulk meteorite specimens.	
Publications	-	

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact Dr Daniela Di Martino, University of Milano Bicocca, ITALY
MRF Instrument **Confocal Microscope 3** **Days Requested:** 1
Special requirements:

SAMPLE

Material	meteorite fragments, mostly silicate	-	-
Formula	-	-	-
Forms	Solid		
Volume	cc		
Weight	mg		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	room temperature - K	-	-
Pressure Range	no applied pressure - mbar	-	-
Magnetic field range	no applied magnetic field - T	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	No	-	-
Sample Prep Hazards	None	-	-
Special equip. reqs	None	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	None	-	-
Equipment Hazards	-	-	-
Biological hazards	No biohazards	-	-
Radioactive Hazards	No radioactive hazards	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Scientific case

Meteorites can be grouped into three main categories: stony, iron and stony-iron meteorites. Stony meteorites consist of mostly silicate minerals and may contain small spheroidal grains (known as chondrules and containing mafic minerals), while iron meteorites consist primarily of an iron-nickel alloy, being a source of metallic iron in ancient times. The most common stony meteorites (ordinary chondrites) usually have an inhomogeneous phase and elemental distribution throughout their volume. These fascinating and rare materials play a fundamental role in the study of planetary and cosmological research [1]. To derive the original provenance or dating of a meteorite the main quest is the determination of its composition and traditionally this goal is achieved by means of destructive measurements (like mass spectrometry or metallographic techniques). Therefore, a non-destructive technique would be desirable, though challenging for home laboratories. To obtain an in-depth information about the meteorite compositions, a multitechnique approach has been designed. Techniques based upon X-rays or neutrons were recently applied, in the perspective of using less invasive methods [2-4]. In this regard, we recently set up a protocol for a non-destructive in-depth characterization of stony meteorites [5-6], and we got time for a new experiment at INES (ISIS, RB2220740) to be performed to derive new details on their composition. In parallel, even though meteorite samples are considered rare and further sampling should be avoided, some thin-sections are already available, since they are commonly used for petrographic-mineralogical observations, and they were imaged by a recently developed super-resolution photo-thermal imaging strategy [7]. The results we have obtained to date suggest that photo-activated far-infrared thermography could represent a complementary technique for the assessment of the composition of meteorite sections, with the advantages of fast imaging times over wide (mm^2- to cm^2- sized) sample areas. In this framework, we propose here a joint confocal microscopy study: with ~ 500 nm spatial resolution, mineralogical phases can be better discriminated and further information on texture and crystal shapes can be obtained. The combination of neutron, micro-Raman spectroscopy mapping and SEM/EDS mapping, with images provided by confocal microscopy is expected to play an essential role for the currently ongoing quantitative interpretation of super-resolution thermographic data. Confocal imaging will help developing a multimodal approach for the comprehensive analysis of thin sections, to be subsequently extended and adapted to bulk meteorite specimens. We emphasize that thin sections will be available for further study, so this experiment can be considered non-invasive.

Technical details

A set of twelve samples of stony and iron meteorites (see fig. 1a) has been provided by the *Planetario e Osservatorio Astronomico G. Giacomotti - Cà del Monte - Cecima* (PV), Italy. Preliminarly, bulk density measurements and XRF analysis have been performed, as for major elements (Si, Fe, Mg, Al, Ca), minor elements (i.e. Na, Ni, Mn) and trace elements (i.e. Ba, Cl, Sr, Ti). Images in super-resolution far-infrared thermography have been acquired (see fig. 1c,d) from one of the thin sections (see fig. 1d). The resulting temperature-based images of the samples, reconstructed at ~10-µm spatial resolution, clearly reveal the heterogeneity of meteorite sections in terms of photo-thermal properties and, indirectly, in terms of the underlying elemental distribution.

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Aiming at quantitatively disclosing the meteorites molecular, elemental and phase features at both the meso- and micro-scale, we plan to correlate far-infrared imaging with micro-Raman mapping, SEM/EDS mapping and neutron spectroscopy. In addition, we submit this proposal for a confocal microscopy experiment on the MRF1 facility Confocal Microscope 3. Each thin section will be imaged in tile-scan (mosaic) mode in confocal reflectance configuration and transmission geometry to provide reference frames of the sample morphology. The spectral dependence of the sample light transmission/reflectance properties will be explored by adjustment of the excitation wavelength in the visible spectral region, as allowed by the available He-Ne, Argon and DPSS laser sources. Along with the absence of required pre-processing or additional preparation of the meteorite section, the employed low (~1-10 µW) excitation cw laser power on the sample plane will comply with the necessary non-invasiveness.

Selected individual chondrules will be mapped at the highest available radial spatial resolution enabled by the 0.5-N.A. air objective (~500 nm), whereas wider sample areas will be imaged at a typical 1 µm pixel size. The corresponding imaging time is estimated to vary from ~15 seconds over 100x100 μm^2 fields of view up to ~60 minutes for 1x1 cm^2 regions sampled at multiple z-depths.

We have estimated a total of one day to carry out the necessary measurements on the twelve available thin sections. Particularly, a high resolution at high magnification collage of the entire section, in order to characterize the morphology dimensions and texture details of the meteorites samples will be performed, combined with the relative spatial distribution of the phases. This analytical step represents a key-test forwarding fundamental information about the sample for the subsequent complementary techniques.

The instrument of choice, and the built-in software implementation of the tile-scan imaging mode, provide full automation in the acquisition and a-posteriori reconstruction of mosaic frames. Subsequent image processing and data analysis will be performed by custom Python codes. The proposed experiment will try to further develop an innovative protocol for the study of meteorites, to be applied to similar samples too, following a multitechnique and multidisciplinary route, to obtain both bulk and surface information.

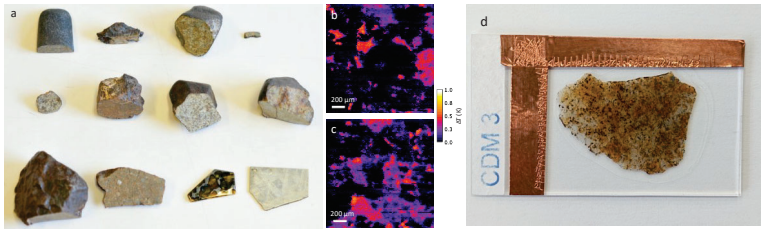

Figure 1: (a) Picture of the samples to be investigated (Credit: Maria Pia Riccardi); (b),(c) Exemplary super-resolution photo-thermal images acquired on two regions of interest, acquired on one thin section (d).

References [1] Bouvier, A. et al. Earth and Planetary Science Letters 273, 48-57 (2008). [2] Tsuchiyama, A. et al. Science 333, 1125-8 (2011). [3] Peetermans, S. et al. Analyst 138, 5303–5308 (2013). [4] Caporali, S. et al. Minerals 6, 14 (2016). [5] Musa, M. et al., Materials 14(24), 7585 (2022) [6] Rossini, R. et al. J. Anal. At. Spectrom. 38, 293 (2023). [7] Bouzin, M. et al., Nat. Commun., 10:5523 (2019).

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Experiment Proposal

Experiment number GP2023088

Principal investigator (*)	Mr Pietro Tordi, University of Florence & CSGI, ITALY
Co-investigator	Dr Rita Gelli, University of Florence & CSGI, ITALY
Co-investigator	Professor Francesca Ridi, University of Florence & CSGI, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Study of the internal structure of alginate fibers crosslinked with different cations by confocal laser microscopy
Training MRF	**Confocal Microscope 3** — **Days requested:** 2
Access Route	Direct Access — **Previous GP Number:** No
Science Areas	Chemistry, Materials — **DOI:** -
Sponsored Grant	None — **Sponsor:** -
Grant Title	- — **Grant Number:** -
Start Date	- — **Finish Date:** -
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

Alginate (Alg) is a biocompatible and biodegradable anionic polysaccharide with high application potential which can be easily shaped into hydrogel fibers using an extrusion and crosslinking strategy, taking advantage of different metal cations. Those fibers are of interest in a number of applications including wound healing, water purification and flame retardancy. Preliminary investigations suggest that fibers from different cations have a characteristic internal morphology, due to the specific diffusion and crosslinking. The goal of this training is to learn how to investigate the internal structure of swollen Alg fibers crosslinked with different cations using confocal laser microscopy. Such technique will allow for the observation of the inner structure of Alg fibers in the swollen state at the micro-scale, upon staining the polymer with a fluorescent probe. The obtained results will relate the features of the internal structure of the fibers with their macroscopic properties.

Publications -

ISIS neutron and muon source — **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Mr Pietro Tordi, University of Florence & CSGI, ITALY
Training Instrument	**Confocal Microscope 3** — **Days Requested:** 2
Special requirements:	

SAMPLE

Material	-	-	-
Formula	-	-	-
Forms			
Volume			
Weight			
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	-	-	-
Pressure Range	-	-	-
Magnetic field range	-	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	-	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	-	-	-
Sensitivity to vapour	-	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	-	-	-

Training Case for ISIS@MACH ITALIA Experimental Proposal
"Study of the internal structure of alginate fibers crosslinked with different cations by confocal laser microscopy"

1. Background and Context

Alginate (Alg) is a biocompatible and biodegradable anionic polysaccharide with high application potential due to its reactivity and selectivity towards metal cations. The hydroxyl and the carboxylate groups on the polymer backbone are involved in the coordination of divalent metal cations, producing crosslinked hydrogels with characteristic porosities and mechanical properties. We developed a facile extrusion and crosslinking approach for the preparation of M^{2+}-crosslinked Alg fibers, which can reach lengths up to some meters thanks to the optimization of the processing parameters. Furthermore, the type of cation and the related complexation geometries allow one to obtain fibers suitable for several applications, including wound healing, water purification and flame retardancy. The diffusion and crosslinking processes responsible for the fiber-formation still need to be unraveled, although their knowledge is crucial for the design of systems with finely tuned properties. This study is part of Pietro Tordi's research activity as a PhD student in co-tutorship between the University of Florence (Italy) and the University of Strasbourg (France), funded by the Italian Ministry of University and Research (MUR) for three years. The aim of the project is the realization of Alg-based composites for wound healing, water/air purification and pressure-based sensors. Part of the studies are currently being carried out at the *Institut de Science et d'Ingeniérie Supramoléculaires* (ISIS, University of Strasbourg), in the Nanochemistry Lab of Prof. Paolo Samorì. The in-depth characterization of the self-assembly properties of Alg in the presence of cations will be beneficial for the preparation of 2D Alg-graphene pressure-sensitive devices.

2. Proposed Training

People to be trained include a PhD student (Pietro Tordi), a researcher (Dr. Rita Gelli), and a professor (Prof. Francesca Ridi), working at the Department of Chemistry of the University of Florence and interested in learning the potentialities of confocal microscopy for the characterization of hydrogels, in particular Alg-based ones. Confocal laser microscopy represents a remarkable tool to unravel the inner structure of Alg fibers at the micrometric scale, as it allows for the observation of the hydrogel in the swollen state and does not require any drying procedure that could lead to artifacts in the structure. MRF staff members contacted in advance prior to the submission will carry out the training.

3. Summary of previous training proposals

No training proposals have been previously submitted through the ISIS@MACH ITALIA infrastructure; nevertheless, we already had the possibility of studying Alg fibers crosslinked with different cations through Small Angle X-ray Scattering experiments, thanks to the allocation of beamtime in the Call for Direct Access round 23-1 (proposal GP2023028). Such characterization led to interesting results (see Section 4, Fig. 1a), and is expected to contribute to the publication of a scientific paper.

4. Justification of experimental proposals request

The aim of the experiment is to observe the internal morphology of swollen Alg fibers crosslinked with various cations (Ca^{2+}, Sr^{2+}, Ba^{2+}, Mn^{2+}, Cu^{2+} and Zn^{2+}) by means of confocal laser microscopy.

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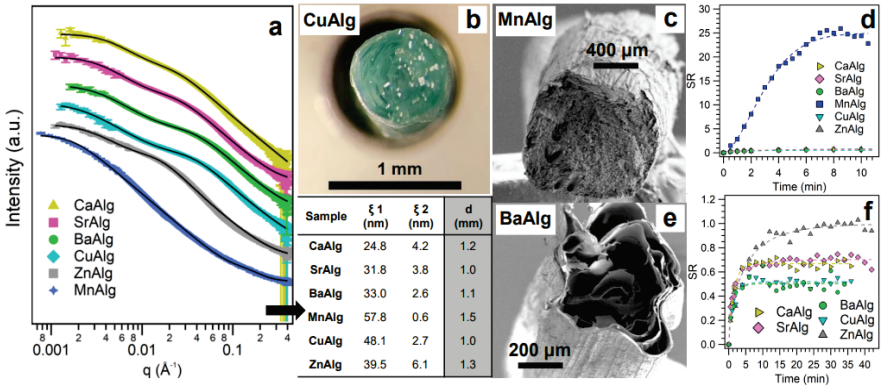
It is believed that different cations impart a different structural organization to Alg chains, due to their different size/charge/coordination geometry. As highlighted by our results, a different assembly at the nanoscale (SAXS, Fig. 1a) results in significant variations in the fibers properties (*e.g.* morphology [c,e] and water absorption [d,f]). The characterization of the internal structure of the swollen fiber at the microscale, possible with confocal laser microscopy, would be fundamental to clarify the diffusion process of the different ions within the hydrogel matrix and the crosslinking process, eventually relating the features of the internal structure of the fibers with their macroscopic properties.



Sample	ξ 1 (nm)	ξ 2 (nm)	d (mm)
CaAlg	24.8	4.2	1.2
SrAlg	31.8	3.8	1.0
BaAlg	33.0	2.6	1.1
MnAlg	57.8	0.6	1.5
CuAlg	48.1	2.7	1.0
ZnAlg	39.5	6.1	1.3

Figure 1. (**a**) SAXS curves of M^{2+}-Alg fibers. (**b**) Swollen Cu^{2+}-Alg fiber's cross-section. SEM micrographs of the lyophilized Mn^{2+}-Alg (**c**) and Ba^{2+}-Alg (**e**) cross-sections. (**d,f**) Swelling profiles of the M^{2+}-Alg samples. * Characteristic correlation lengths (ξ) and swollen diameters (d) of the M^{2+}-Alg samples are reported in the table.

The training would allow us to answer some fundamental questions about the structure of Alg fibers, namely: *i)* Is it possible to observe a different internal structure of Alg fibers depending on the cation used to crosslink the matrix? *ii)* If the fiber is crosslinked with a cation that imparts a specific structure, is it possible to exchange the crosslinking cation and follow in real time the modification of the structure?
Our samples consist of six swollen Alg fibers crosslinked with Ca^{2+}, Sr^{2+}, Ba^{2+}, Mn^{2+}, Cu^{2+} and Zn^{2+}, having diameters ranging from 1 to 1.5 mm. Considering that the field-of-view of Leica TCS SP5 II is 750 µm, the possibility of acquiring in tile scan mode will be taken into account. Alg can be fluorescently-labeled with a probe interacting with the carboxylic units on the polymeric backbone, in order to distinguish between the different porous/layered structures of the Alg fiber. Prior to the experiments, staining tests with different fluorescent probes will be performed at the University of Florence, following the advice of the instrument scientist, in order to determine the most suitable probe in terms of stability and binding conditions. As suggested by the instrument scientist, the attention will be devoted to fluorescent probes with excitation wavelength close to 488 nm (according to the literature, fluorescein isothiocyanate (FITC) might be a promising candidate). To carry out the training and the subsequent analysis of the six samples, a total of 2 days of operation is required (safety considerations: 1 h, instrument set-up: 1 h, measurement run: 2 h for each sample, total 12 h, data analysis: 2 h).

Dynamic Mechanical Analyzer

Dynamic Mechanical Analyzer

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Experiment Proposal

Experiment number GP2023064

Principal investigator	Dr Fabrizia Foglia, University College London, UNITED_KINGDOM
Co-investigator (*)	Mr Keenan Smith, University College London, UNITED_KINGDOM
Co-investigator	Dr Tom Miller, University College London, UNITED_KINGDOM
Co-investigator	Professor Silvia Licoccia, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Peter Fouquet, Institut Laue-Langevin, FRANCE
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Characterization of recycled perfluorosulfonic acid membrane for a circular hydrogen economy
MRF Instrument	**Dynamic Mechanical Analyzer** **Days requested:** 4
Access Route	Direct Access **Previous GP Number:** No
Science Areas	Energy **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	ILL; for Figaro and Spin Echo
Industrial Links	-

Non-Technical Abstract

Perfluorinated sulfonic-acid (PFSA) ionomers, such as Nafion introduced by DuPont >50 years ago, are a superior class of ion-conducting polymers used in fuel cells and electrolysers due to their remarkable ion conductivity and chemical and mechanical stability. Fuel cell lifetime is, however, curtailed by chemical and physical degradation of the PFSA in the electrode and electrolyte membrane (amongst other mechanisms) and lead to unusable end of life cells. Furthermore, with increased adoption of sustainable energy technologies, increased demand for PFSAs is forecast. This translates into the necessity to recycle membrane components. Here we intend to study the water dynamics in degraded and recycled Nafion, as well as compare these to pure recast Nafion using the DMA instrument of the IM@IT Unit University of Rome Tor Vergata.

Publications

Nafion matrix and ionic domain tuning for high performance composite proton exchange membranes. Advanced Functional Materials, 2304061; 2023

Disentangling water, ion and polymer dynamics in an anion exchange membrane. Nature Materials 21 (5) 555; 2022

Foglia F, et al. Aquaporin-like water transport and nanoconfinement in nanoporous crystalline layered carbon nitride. Science Advances 6 (39), eabb6011; 2020

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Mr Keenan Smith, University College London, UNITED_KINGDOM
MRF Instrument	**Dynamic Mechanical Analyzer** **Days Requested:** 4
Special requirements:	

SAMPLE

Material	C7HF13O5S·C2F4	-	-
Formula	C7HF13O5S·C2F4	-	-
Forms	Solid		
Volume	cc		
Weight	100 mg		
Container or substrate	sample is thin-film	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	83 - 473 K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	no	-	-

SAFETY

Prep lab needed	No	-	-
Sample Prep Hazards	no	-	-
Special equip. reqs	no	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	Yes	-	-
Experiment Hazards	no	-	-
Equipment Hazards	-	-	-
Biological hazards	no	-	-
Radioactive Hazards	no	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Removed By User	-	-

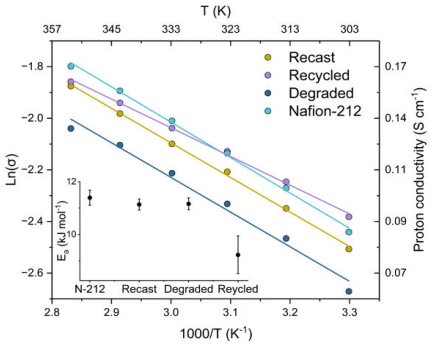
Characterization of recycled perfluorosulfonic acid membrane for a circular hydrogen economy

1. Background and Context

Ion transport is a critical element of energy conversion devices such as fuel cells (FC), water electrolysers and flow batteries. Perfluorinated sulfonic-acid (PFSA) ionomers, such as Nafion introduced by DuPont >50 years ago[1], are a class of ion-conducting polymers known for their remarkable ion conductivity and chemical and mechanical stability. PFSA ionomers are typically formed from a hydrophobic Teflon-like backbone with pendent hydrophilic sulfonic acid bearing side chains which phase separate to form a morphology with superior ion and water transport[2]. PFSAs are essential in both the proton exchange membrane (PEM), transporting H^+ ions between electrodes while blocking the flow of reactant gases and ions (H_2, O_2 and VO^-), and as thin, 2-50 nm films, coating catalyst particles in the electrodes for efficient electrocatalysis[3]. FC lifetime is, however, curtailed by chemical degradation from peroxides/radicals and physical degradation of pinholes and microcracks due to high temperature and low humidity operation[4].

With increased adoption of sustainable energy technologies, an increased demand for PFSAs is forecast as well as generation of increased quantity of used PFSA material. PFSA synthesis relies on precursors from non-renewable fossil fuel industries and requires reaction steps exceeding 500 °C. Current End of life technologies are based on hydrometallurgical and pyro-hydrometallurgical methods for the recovery of noble metal catalysts whilst generating corrosive and hazardous fluorine and HF gas form PFSA waste pructs[5]. Therefore, an approach to separate, regenerate and re-use the various material components of spent devices will establish a sustainable life cycle for hydrogen technologies, with environmental and economic benefits.

At UCL we have developed a low-cost solvent-based approach to extract PFSA from the carbon and platinum in degraded FC membrane electrode assemblies (MEAs) allowing both components to separately be regenerated. Through subsequent processing and assembly, the PFSA component can be incorporated into 2nd life FCs as the PEM or electrode ionomer with no deterioration in FC performance. Producing a recycled ionomer also has the potential to realise composite membranes as well as advanced fabrication methods, such as direct membrane deposition, due to the solution cast nature.



Whilst recast membranes achieved equivalent conductivities to pristine Nafion, reduced mechanical strength, despite similar crystallinity and chemical signatures, raise questions of atomic structure and molecular morphology. PFSA's chemical and mechanical properties are interrelated through their phase-separated morphology, where the transport properties are primarily due to the hydrated ionic domains, while the hydrophobic backbone provides the mechanical support. These features arrange at multiple length scales and thus a complete understanding of chemical changes requires approaches investigating different length scales. Thin film confinement of Nafion on a substrate has been shown to induce anisotropic phase separation in plane, which has been used to understand morphological arrangement due to ionomer side chain length and EW[6]. Degradation induced main chain or side chain scission or sulphonic anhydride crosslinking will affect the domain and crystallite morphology and result in modified transport processes. Structural and dynamical investigation are critical to reveal these changes.

We have already been performed time on FIGARO (ILL reflectometer; experiments were scheduled in April 2023) to study structural changes and got allocated time on WASP (ILL Spin Echo spectrometer; experiments are scheduled in November 2023) to investigate water dynamics within these membranes and, therefore, best understand the structure dynamics interplay. We now intend to extend our study using DMA 1 Star Systems – Mettler Toledo – to measure the mechanical and viscoelastic properties of our sample as a function of temperature, and relative humidity levels. This project is related to our work carried out within the EPSRC fellowship (EP/V057863/1).

2. Proposed experiment

We plan to perform experiments using the DMA 1 Star Systems – Mettler Toledo – available via ISIS@MACH to measure the mechanical and viscoelastic properties on recycled Nafion and compared these with untreated as well as degraded Nafion.

3. Justification of experimental proposals request

Experiments will be performed on: i) pure recast Nafion; ii) FC degraded recycled Nafion (Nafion-FC); and iii) Fenton's reagent degraded Nafion (Nafion-FT) at low-hydration (λ~7; where λ represents the water uptake per sulphonic group). Based on the number of membranes (three) and conditions (6 Temperatures from 83 to 473 K at both λ~7 and ~18) to be investigated we plan for a Total: 36 samples; we therefore request a total of **4 days**.

References:

[1] KA Mauritz & RB Moore, Chem. Rev. **104**, 4535 (2004);

[2] A Kusoglu & AZ Weber, Chem. Rev. **117**, 987 (2017);

[3] TAM Suter, et al, Nanomaterials **11**(10) 2530 (2021);

[4] R Borup, et al, Chem. Rev. **107**, 3904 (2007);

[5] L Duclos, et al, Green Chem. **22**, 1919 (2020);

[6] UN Shrivastava, H Fritzsche & K Karan, Macromolecules **51**, 9839 (2018).

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Experiment Proposal

Experiment number GP2023086

Principal investigator	Dr Fabrizia Foglia, University College London, UNITED_KINGDOM
Co-investigator (*)	Mr Keenan Smith, University College London, UNITED_KINGDOM
Co-investigator	Dr Tom Miller, University College London, UNITED_KINGDOM
Co-investigator	Professor Silvia Licoccia, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Peter Fouquet, Institut Laue-Langevin, FRANCE
Co-investigator	Professor Christoph Salzmann, University College London, UNITED_KINGDOM
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Characterization of recycled perfluorosulfonic acid membrane for a circular hydrogen economy
MRF Instrument	**Dynamic Mechanical Analyzer** **Days requested:** 4
Access Route	Direct Access **Previous GP Number:** No
Science Areas	Energy **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	ILL; for Figaro and Spin Echo
Industrial Links	-

Non-Technical Abstract

Perfluorinated sulfonic-acid (PFSA) ionomers, such as Nafion introduced by DuPont >50 years ago, are a superior class of ion-conducting polymers used in fuel cells and electrolysers due to their remarkable ion conductivity and chemical and mechanical stability. Fuel cell lifetime is, however, curtailed by chemical and physical degradation of the PFSA in the electrode and electrolyte membrane (amongst other mechanisms) and lead to unusable end of life cells. Furthermore, with increased adoption of sustainable energy technologies, increased demand for PFSAs is forecast. This translates into the necessity to recycle membrane components. Here we intend to study the water dynamics in degraded and recycled Nafion, as well as compare these to pure recast Nafion using the DMA instrument of the IM@IT Unit University of Rome Tor Vergata and the particle size analyser (international MRF) at UCL.

Publications

Nafion matrix and ionic domain tuning for high performance composite proton exchange membranes. Advanced Functional Materials, 2304061; 2023

Disentangling water, ion and polymer dynamics in an anion exchange membrane. Nature Materials 21 (5) 555; 2022

Foglia F, et al. Aquaporin-like water transport and nanoconfinement in nanoporous crystalline layered carbon nitride. Science Advances 6 (39), eabb6011; 2020

Internationa MRFs	**Particle Size Analyser**	**Days requested:** 2
ISIS neutron and muon source		**IM@IT E-platform:** No
Instruments		**Days Requested:**
Access Route		**Previous RB Number:**
Science Areas		**DOI:**
Sponsored Grant		**Sponsor:**
Grant Title		**Grant Number:**
Start Date		**Finish Date:**
Similar Submission?		
Industrial Links		

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Sample record sheet

Principal contact	Mr Keenan Smith, University College London, UNITED_KINGDOM		
MRF Instrument	**Dynamic Mechanical Analyzer**	**Days Requested:** 4	
Special requirements:			
	SAMPLE		
Material	C7HF13O5S·C2F4	-	-
Formula	C7HF13O5S·C2F4	-	-
Forms	Solid		
Volume	cc		
Weight	100 mg		
Container or substrate	sample is thin-film	-	-
Storage Requirements	-	-	-
	SAMPLE ENVIROMENT		
Temperature Range	83 - 473 K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	no	-	-
	SAFETY		
Prep lab needed	No	-	-
Sample Prep Hazards	no	-	-
Special equip. reqs	no	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	Yes	-	-
Experiment Hazards	no	-	-
Equipment Hazards	-	-	-
Biological hazards	no	-	-
Radioactive Hazards	no	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Removed By User	-	-

Characterization of recycled perfluorosulfonic acid membrane for a circular hydrogen economy

1. Background and Context

Ion transport is a critical element of energy conversion devices such as fuel cells (FC), water electrolysers and flow batteries. Perfluorinated sulfonic-acid (PFSA) ionomers, such as Nafion introduced by DuPont >50 years ago[1], are a class of ion-conducting polymers known for their remarkable ion conductivity and chemical and mechanical stability. PFSA ionomers are typically formed from a hydrophobic Teflon-like backbone with pendent hydrophilic sulfonic acid bearing side chains which phase separate to form a morphology with superior ion and water transport[2]. PFSAs are essential in both the proton exchange membrane (PEM), transporting H^+ ions between electrodes while blocking the flow of reactant gases and ions (H_2, O_2 and VO^-), and as thin, 2-50 nm films, coating catalyst particles in the electrodes for efficient electrocatalysis[3]. FC lifetime is, however, curtailed by chemical degradation from peroxides/radicals and physical degradation of pinholes and microcracks due to high temperature and low humidity operation[4].

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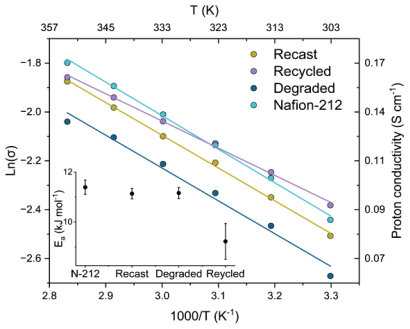
At UCL we have developed a low-cost solvent-based approach to extract PFSA from the carbon and platinum in degraded FC membrane electrode assemblies (MEAs) allowing both components to separately be regenerated. Through subsequent processing and assembly, the PFSA component can be incorporated into 2nd life FCs as the PEM or electrode ionomer with no deterioration in FC performance. Producing a recycled ionomer also has the potential to realise composite membranes as well as advanced fabrication methods, such as direct membrane deposition, due to the solution cast nature.



Whilst recast membranes achieved equivalent conductivities to pristine Nafion, reduced mechanical strength, despite similar crystallinity and chemical signatures, raise questions of atomic structure and molecular morphology. PFSA's chemical and mechanical properties are interrelated through their phase-separated morphology, where the transport properties are primarily due to the hydrated ionic domains, while the hydrophobic backbone provides the mechanical support. These features arrange at multiple length scales and thus a complete understanding of chemical changes requires approaches investigating different length scales. Thin film confinement of Nafion on a substrate has been shown to induce anisotropic phase separation in plane, which has been used to understand morphological arrangement due to ionomer side chain length and EW[6]. Degradation induced main chain or side chain scission or sulphonic anhydride crosslinking will affect the domain and crystallite morphology and result in modified transport processes. Structural and dynamical investigation are critical to reveal these changes.

We have already been performed time on FIGARO (ILL reflectometer; experiments were scheduled in April 2023) to study structural changes and got allocated time on WASP (ILL Spin Echo spectrometer; experiments are scheduled in November 2023) to investigate water dynamics within these membranes and, therefore, best understand the structure dynamics interplay. We now intend to extend our study using DMA 1 Star Systems – Mettler Toledo – to measure the mechanical and viscoelastic properties of our sample as a function of temperature, and relative humidity levels. This project is related to our work carried out within the EPSRC fellowship (EP/V057863/1).

2. Proposed experiment

We plan to perform experiments using the DMA 1 Star Systems – Mettler Toledo – available via ISIS@MACH to measure the mechanical and viscoelastic properties on recycled Nafion and compared these with untreated as well as degraded Nafion. Additionally, we wish to perform light scattering experiments using the Mastersizer Particle Size Analyser at UCL through the international MRF route. This will give insights into potential degradation processes of the Nafion materials.

3. Justification of experimental proposals request

Experiments will be performed on: i) pure recast Nafion; ii) FC degraded recycled Nafion (Nafion-FC); and iii) Fenton's reagent degraded Nafion (Nafion-FT) at low-hydration (λ~7; where λ represents the water uptake per sulphonic group). Based on the number of membranes (three) and conditions (6 Temperatures from 83 to 473 K at both λ~7 and ~18) to be investigated we plan for a Total: 36 samples; we therefore request a total of **4 days**. All samples will also be analysed using the Mastersizer at UCL. We request **2 days**, one day to establish this new technique and to determine the optimal experimental conditions for measuring our samples, and a second day for measuring the actual samples.

References:

[1] KA Mauritz & RB Moore, Chem. Rev. **104**, 4535 (2004);
[2] A Kusoglu & AZ Weber, Chem. Rev. **117**, 987 (2017);
[3] TAM Suter, et al, Nanomaterials **11**(10) 2530 (2021);
[4] R Borup, et al, Chem. Rev. **107**, 3904 (2007);
[5] L Duclos, et al, Green Chem. **22**, 1919 (2020);
[6] UN Shrivastava, H Fritzsche & K Karan, Macromolecules **51**, 9839 (2018).

FIB SEM GAIA 3

FIB SEM GAIA 3

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Experiment Proposal

Experiment number GP2023081

Principal investigator	Professor Giancarlo Capitani, University of Milano-Bicocca, ITALY		
Co-investigator (*)			
Co-investigator (*)			
Co-investigator (*)			
Co-investigator (*)			
Co-investigator (*)			
Co-investigator (*)			
Co-investigator (*)			
Co-investigator (*)			
Experiment title	Preparation and Study of TEM lamellae of CaREE-flourcarbonates.		
MRF Instrument	**FIB-SEM GAIA 3**	**Days requested:** 3	
Access Route	Direct Access	**Previous GP Number:** no	
Science Areas	Environment, Materials	**DOI:** -	
Sponsored Grant	None	**Sponsor:** -	
Grant Title	-	**Grant Number:** -	
Start Date	-	**Finish Date:** -	
Similar Submission?	-		
Industrial Links	-		
Non-Technical Abstract	CaREE-fluorcarbonates (CRFC) are main ore for rare earth elements (REE). REE are critical raw materials for electronics and green technologies and there is a global concern for their supply and recycling, in particular from EC countries, which are completely dependent from China. Consequently, many efforts are being spent in geological exploitation, improvement of metallurgical processes and recycling REE from mine waste and RAEE (electronic waste). It is universally recognized, as well as often forgotten, that fundamental research is the nourishment of applied research. Mineralogical and crystallographic studies on CRFC may shed light on their nature and genesis processes, eventually suggesting more successful prospection routes, enhanced metallurgical processes and recycling strategies. In this context, since a few years, at the DISAT, we have undertaken a study of CRFC of the bastnäsite-synchysite series from several localities. The CRFC we are currently studying are systematically		
Publications	-		

ISIS neutron and muon source	**E-platform:** No
Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact
MRF Instrument **FIB-SEM GAIA 3** **Days Requested:** 3
Special requirements:

SAMPLE

Material	Ca and REE fluorcarbonate synchysite-(Ce) embedded in epoxy and polished	-	-
Formula	Ca(Ce,La, Nd)(CO3)2F with inclusions of hematite (Fe2O3) and thorite (ThSiO4)	-	-
Forms	Solid		
Volume	7 cc		
Weight	9 g		
Container or substrate	epoxy cylinder 1/2 inch in diameter	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	TEM grids for FIB applications to fix the lamellae	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	The amount of Th dispersed in the sample is so small that there is not detactable effect	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Removed By User	-	-

:al Proposal – Preparation and Study of TEM lamellae of CaREE-fluorcarbonates.

1. Background and context

CaREE-fluorcarbonates (CRFC) are main ore for rare earth elements (REE). REE are critical raw materials for electronics and green technologies and there is a global concern for their supply and recycling, in particular from EU countries, which are completely dependent from China. Consequently, many efforts are being spent in geological exploitation, improvement of metallurgical processes and recycling REE from mine waste and RAEE (electronic waste). It is universally recognized, as well as often forgotten, that fundamental research is the nourishment of applied research. Mineralogical and crystallographic studies on CRFC may shed light on their nature and genesis processes, eventually suggesting more successful prospection routes, enhanced metallurgical processes and recycling strategies. In this context, since a few years, at the DISAT, we have undertaken a study of CRFC of the bastnäsite-synchysite series from several localities, in Italy (Cuasso al Monte (VA), Cinquevalli (TR)) and outside Italy (Malawi, Turkey), with the aim to contribute to the above issues. The study has been supported by university funding, both competitive (FAQC2022, 25'000 €) and non (FAQD) from 2017 to 2021 (total 7283 €). Over the years, the study has involved several bachelor and master degree students and it is currently the subject of a PhD thesis; it has produced three peer review papers on international journals [1-3] and two more are in preparation. The current proposal aims at finalizing the work in progress.

2. Proposed experiments

The CRFC we are currently studying are systematically found in miarolitic cavities in a granophyre located in the Western Southern Alps (Cuasso al Monte (VA)). These rocks have been never regarded as ore for REE, but rather as dimension stones. Nevertheless, hydro-thermalized outcrops (not good for the market) and quarry wastes are rich of REE. The CRFC are normally less than 100 µm in size, have hexagonal prismatic habit, reddish color and evident zonation (Fig. 1). The latter microstructural aspect is very intriguing, since unveiling inhomogeneous REE distribution, with a Ce-rich core and a Y-rich rim. The rim is also enriched in Th and plenty of Fe-oxides (hematite, determined by Raman). This microstructure may convey information about the minero-genesis, which may potentially guide any ore prospection. Moreover, since the difficulty affecting the separation of REE in metallurgical processes (e.g. [4], it would be of paramount importance to understand the Ce/Y fractionation process operated by nature. The last but not the least, given the huge environmental problem caused by the association of Th to every REE deposit, the understanding of the processes underlying the Th mobility may help in the design of cleaner metallurgical processes. Any genetic and fractionation model may be achieved pushing the investigation down to the nanometer scale, a dimension accessible exclusively by transmission electron microcopy (TEM). Before that, thin TEM lamellae must be extracted from such microscopic samples, a task sometimes more difficult than the TEM observation itself and possible only via the FIB lift-out technique [5]. Basically, a wedge shaped lamella ~20 µm in length must be cut out across the core-rim interface, extracted, welded on a TEM-FIB grid and further thinned down to electron transparency (Fig. 2). The lamella so prepared is then ready to be study be high resolution (HR) and analytical TEM.

3. Summary of previous experimental proposals or characterisation

The proposal has never been submitted before but samples from the same locality have been characterized by a variety of techniques, including scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), single crystal X-ray diffraction and Raman spectroscopy. Overall, these techniques revealed that the CRFC are synchysite-(Ce), with inclusions of hematite (Fe_2O_3) and thorite ($ThSiO_4$) and possess the microstructure and chemical zoning described above. A TEM investigation focused on the core/rim interface would complement these information, possibly leading to a genetic and fractionation model. Previous TEM studies on similar samples from other localities have shown syntactic (crystallographically oriented) intergrowths of different CRFC, compositional and polytypic faults and new polymorphs. All together, these observations have contributed to better define the bastnäsite-synchysite series and to speculate on the formation of the ore [1-3].

4. Justification of experimental proposals request

In order to build up a genetic model, information at the SEM and TEM scale are required. As outlined above, the size and shape of the sample require a dual beam instrument to prepare TEM lamellae. In order to properly locate the core/rim interface, a SEM/EDS map is required. Considering the dimension of the sample (150-200 in diameter) and the volume affected by the extraction (some tens of µm), the preparation of 3 lamellae appears feasible. Three lamellae represent also a reasonable number to be investigated,

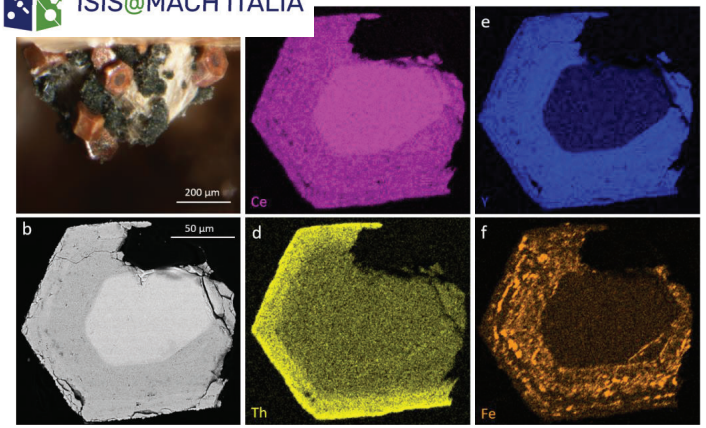

Figure 1. Optical micrograph showing the Ca-REE fluorcarbonates samples within a miarolitic cavity. (b-f) SEM-EDS images showing the chemical zonation. The Ce content is greater in the core with respect to rim (c), whereas Y and Th show an opposite trend (e,d). Fe-oxides inclusions are present only in the rim (f).

considering the poor stability of CRFC under the TEM beam, the complex microstructure and the diversity of TEM techniques planned. Indeed, from the experiment we expect to: i) collect a high-quality SEM-EDS map to describe the microstructure and correctly locate the extraction areas; ii) obtain electron transparent lamellae with the FIB lift-out technique; iii) determine the nature of the boundary (coherent/incoherent) through combined selected area diffraction (SAD) and HRTEM observations; iv) determine the order/disorder nature of the domains on the opposite sides of the boundary, i.e. any presence of stacking faults (conventional TEM); v) determine any presence of compositional faults, by directly measuring the d-spacing on HR images; vi) determine the composition of the ordered domains on both sides of the boundary (EDS); v) the nature of inclusions (hematite, thorite and any other phase detected) through a combination of SAD and EDS. Overall, this information would contribute to build up a genetic model, possibly leading to improved prospection strategies, more efficient and clean ore processing operations and recycling routes. For the goal, HRTEM capabilities (quasi-atomic resolution) and EDS analysis at the nanoscale are required, i.e. a field emission gun (FEG) TEM is required. After a confrontation with the instrument scientist, one day and one half would be sufficient for the preparation of the three lamellae, considering the lack of a priori knowledge of the ion milling rate of CFRC. A similar time extent (half a day/lamella) should be sufficient for their characterization at the TEM.

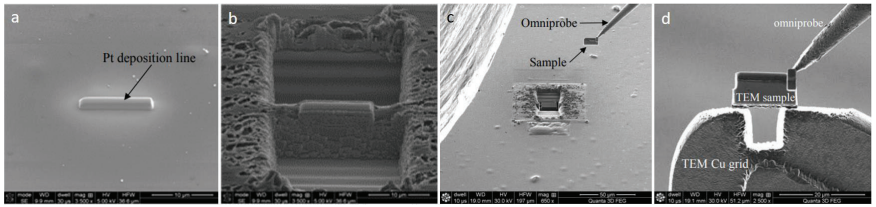

Figure 2. SEM images of lamella preparation steps: (a) Deposition of a Pt strip to protect the extraction area; (b) Cut out the lamella with the ion beam; (c) Lamella extraction with a micromanipulator; (c) Welding the lamella on a TEM grid for further milling.

[1] Capitani, G. HRTEM Investigation of Bastnäsite–Parisite Intergrowths from Mount Malosa (Malawi): Ordered Sequences, Polysomatic Faults, Polytypic Disorder, and a New Parisite-(Ce) Polymorph. *EJM* **2019**, *31* (3), 429–442. https://doi.org/10.1127/ejm/2019/0031-2824. [2] Capitani, G. Synchysite-(Ce) from Cinquevalli (Trento, Italy): Stacking Disorder and the Polytypism of (Ca,REE)-Fluorcarbonates. *Minerals* **2020**, *10* (1), 77. https://doi.org/10.3390/min10010077. [3] Conconi, R.; Fumagalli, P.; Capitani, G. A Multi-Methodological Study of the Bastnäsite-Synchysite Polysomatic Series: Tips and Tricks of Polysome Identification and the Origin of Syntactic Intergrowths. *AmMin* **2023**. https://doi.org/10.2138/am-2022-8678. [4] Cen, P., Bian, X., Liu, Z., Gu, M., Wu, W., Li, B., 2021. Extraction of rare earths from bastnaesite concentrates: A critical review and perspective for the future. *MinEng* 171, 107081. https://doi.org/10.1016/j.mineng.2021.107081. [5] Hyun Jung, K.; Sang, H., C.; Hung-Bin, B.; Tae Woo, L. Transmission Electron Microscopy (TEM) Sample Preparation of Si1-XGex in c-Plane Sapphire Substrate. *NASA/TM–2012-217597* **2012**, 39 pp. https://ntrs.nasa.gov/citations/20120013304.

FT-IR Nexus *FT-IR Nexus*

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Experiment Proposal

Experiment number GP2023053

Principal investigator	Mrs Valentina Turina, Fondazione Museo Antichità Egizie, ITALY
Co-investigator (*)	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Carla Andreani, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Lucy Skinner, University of Northampton and the British Museum, UNITED_KINGDOM
Co-investigator	Dr Robert Robinson, University of Wollongong, AUSTRALIA
Co-investigator	Professor Salima Ikram, American University in Cairo, EGYPT
Co-investigator	Miss Giulia Pallottini, Fondazione Museo Antichità Egizie, ITALY
Co-investigator	
Experiment title	Characterization of collagen and both tanning and colouring materials on leather artefacts from Museo Egizio by FT-IR measurements
MRF Instrument	**FT-IR Nexus** **Days requested:** 1
Access Route	Direct Access **Previous GP Number:** -
Science Areas	Cultural Heritage, Materials, Physics **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	Fondazione Museo Egizio

Non-Technical Abstract

Within the Museo Egizio collection there are 200 precious and unique leather artefacts belonging to different historical periods including the Old Kingdom, New Kingdom, Roman and Byzantine eras. Hence, it is paramount to understand degradation mechanism of ancient leather probably related to the way the skins were prepared and made durable. The proponents aim to study by WAXS/SAXS/USAXS the assembly and orientation of the collagen fibrils in the samples and extend by means of FT-IR and Raman spectroscopy measurements its characterization and both tanning and colouring materials found in ancient leather. In the present proposal, we wish to measure the intra- and inter- molecular vibrational spectra of collagens as well as both the tanning and colouring materials components, most found in ancient leather using FT-IR measurements on the FT-IR Nexus instrument.

Publications

G. Romanelli, et al., “Neutron-Enhanced Information on the Laboratory Characterization of Ancient Egyptian Leathers...”, Information, 2022, 13, 467
G. Pallottini, Graduate Thesis, "La coperta Provv.5062 del Museo Egizio di Torino: studio, restauro e valorizzazione" (2021).

ANSTO Reactor

Brief abstract

Two other measurements for performing 2D/3D neutron imaging and neutron USANS have been scheduled to DINGO and KOOKABURRA neutron beamlines at ANSTO (Australia), respectively.

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Sample record sheet

Principal contact	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
MRF Instrument	**FT-IR Nexus** **Days Requested:** 1
Special requirements:	

SAMPLE

Material	Leather	-	-
Formula	Collagen	-	-
Forms	Solid		
Volume	1 cc		
Weight	1 mg		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Characterization of collagen and both tanning and colouring materials on leather artefacts from Museo Egizio by FT-IR measurements

1. Background and Context

The collection of the Museo Egizio (Turin) houses over 200 leather artifacts belonging to different historical periods, including the Old Kingdom, New Kingdom, Roman and Byzantine eras. Leather is the main material and the only common element of these objects. Leather was used throughout the entire society, from low to high status and often subject to a variety of uses, from decorative to intense use. Ancient leather presents a heterogeneous composition of both organic and inorganic materials that show an evident reactivity. Its proper preservation remains challenging as some aspects of its chemical composition, degradation and effectiveness of conservation treatments are still not fully understood. Archeologists and conservators were able to identify, through the constant conservative monitoring of the artifacts, different types of degradation and, above all, a correspondence between their dating and the type of documented deterioration. Indeed, the different types of degradation are probably related to the way the skins were prepared and made durable. Of particular concern for the collection of Museo Egizio (Turin) is that the skin processing method (including any coloring treatments) and the substances used to make it more durable are not known. Not so many processes are attested for this period [2], and the substances that were used to treat the skin and the likely connection with the types of deterioration that are documented are closely linked to collagen, the most important fibrous protein. Collagen is the principal protein constituent of a wide variety of connective tissues in animals. Its structure has been investigated extensively by electron microscopy and by diffraction techniques using X-rays and neutrons [3-8]. Recently [9], a characterization of Egyptian leather samples was completed by combining non-destructive techniques, including surface probes (X-ray fluorescence, Raman scattering, and scanning electron microscopy enhanced by X-ray energy spectroscopy) and neutron-based bulk techniques (inelastic and deep-inelastic neutron scattering).

The proponents aim to study by wide/small/ultra small angle X-ray scattering (WAXS/SAXS/USAXS) the assembly and orientation of the collagen fibrils in the samples already investigated in Ref. [9], and by distinct proposals perform a complementary characterization using both Fourier-transform infrared spectroscopy (FT-IR) and Raman spectroscopy (AFM-Raman). These spectroscopic techniques will be used to study the intra- and inter- molecular vibrational spectra of collagens as well as both the tanning and colouring materials components, most found in ancient leather [10]. Hence, we propose to use the SAXS GISAXS, FT-IR Nexus, and AFM Raman instrument operating at the CSGI-Unifi and the Univ. Tor Vergata Units of in the suite of IM@IT. Further characterization on the same samples will be done by neutron imaging (DINGO beamline) and USANS (Kookaburra beamline) at the Australian Centre for Neutron Scattering (ANSTO).

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2. Proposed experiment for FT-IR

In the present proposal, we wish to measure the degree of assembly of the collagen fibrils of n. 6 ancient leather artifacts (same samples that will be measure at ANSTO) using FT-IR measurements on the FT-IR Nexus instrument. Results from FT-IR measurements will be compared and extended by WAXS/SAXS/USAXS and Raman spectroscopy widely used for the characterization of collagen and both tanning and colouring materials found in ancient leather as reported in this work [10], which will be submitted in separate proposals.

3. Justification of experimental time requested for FT-IR

We aim to measure n. 6 ancient leather artifacts (same samples that will be measure at ANSTO) on the FT-IR Nexus instrument in the 4000-650 cm^{-1} range at a resolution of 4 cm^{-1} [10], averaging at least ten acquisitions per sample each of 300 s. Hence, we request one day of instrument time including set-up and calibration time.

4. References

[1] E. Schiaparelli, Relazione sui lavori Della Missione Archeologica Italiana in Egitto (anni 1903–1920), second volume: The intact tomb of the architect Kha in the necropolis of Thebes (AdArte, 2008).

[2] Driel-Murray, van, C. 2000. Leatherwork and Skin Products. In: Nicholson, P.T. & I. Shaw. Eds. 2000. Ancient Egyptian Materials and Technology. – Cambridge, Cambridge University Press: 299-319.

[3] Miller, A. Philos. Trans. R. Soc. Lond. B. 304 (1984), pp. 455-477.

[4] R.D.B. Praser et al., J. Mol. Biol. 193 (1987), pp. 115-125.

[5] T. J. Wess et al., J. Mol. Biol. 2131-5, (1990).

[6] M. Karplus et al., Biophysical Journal 69 (1195) pp. 660-673.

[7] H. D. Middendorf et al., Biophysical Journal 69 (1995), pp. 660-673.

[8] J. Li, J. Chem. Phys. 105 6733-6755 (1996)

[9] G. Romanelli et al., Information 13 (2022), 467.

[10] A. Elmaggar et al., Archaeometry 59 (2017), pp. 133-147.

Fluorescence Microscopy

Fluorescence Microscopy

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Experiment Proposal

Experiment number GP2023073

Principal investigator	Professor Roberto Senesi, University of Rome Tor Vergata, ITALY	
Co-investigator	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY	
Co-investigator	Dr Laura Fazi, University of Rome Tor Vergata, ITALY	
Co-investigator (*)	Dr Francesco Stellato, Università degli Studi di Roma Tor Vergata, ITALY	
Co-investigator	Dr Anna Prioriello, University of Rome Tor Vergata, ITALY	
Co-investigator		
Co-investigator		
Co-investigator		
Co-investigator		
Experiment title	Fluorescence microscopy training for MSci students in Physics	
Training MRF	**Fluorescence Microscopy**	**Days requested:** 2
Access Route	Direct Access	**Previous GP Number:** -
Science Areas	Biology and Bio-materials, Materials, Medicine, Physics	**DOI:** -
Sponsored Grant	None	**Sponsor:** -
Grant Title	-	**Grant Number:** -
Start Date	-	**Finish Date:** -
Similar Submission?	-	
Industrial Links	-	
Non-Technical Abstract	We propose a training access to fluorescence microscopy for MSci students in Physics with curricula in condensed matter and biophysics. This has the aim of providing knowledge and awareness of research infrastructures and instrumentation at the undergraduate level and before MSci thesis, for perspective industrial and public sector users. E-gate for INES at ISIS will be requested for diffraction analyses.	
Publications	-	

Instruments	**INES**	**Days Requested:** 2
Access Route	Direct Access	**Previous RB Number:**
Science Areas		**DOI:**
Sponsored Grant	None	**Sponsor:**
Grant Title	-	**Grant Number:**
Start Date	-	**Finish Date:**
Similar Submission?		
Industrial Links		

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Sample record sheet

Principal contact	Dr Francesco Stellato, Università degli Studi di Roma Tor Vergata, ITALY	
Training Instrument	**Fluorescence Microscopy**	**Days Requested:** 2
Special requirements:		

SAMPLE

Material	-	-	-
Formula	-	-	-
Forms			
Volume			
Weight			
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	-	-	-
Pressure Range	-	-	-
Magnetic field range	-	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	-	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	-	-	-
Sensitivity to vapour	-	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	-	-	-

Fluorescence microscopy training for MSci students in Physics

Background and Context

Career development of physics students from Tor Vergata leads often to employment in industrial R&D environments (e. g. Leonardo, Thales, Elettronica spa, SONY-Eriksson, Biotech companies etc.). Once part of the industrial environment, alumni are frequently set within industrial programmes whose needs on characterization of materials are an opportunity IM@IT as well as for companies/institutions involved.

In order to align training on access to infrastructures such as IM@IT and the individual career paths there is a need to connect and raise awareness on access during MSci course attendance.

Fulfilling these needs has an added value for all undergraduates, but would add specific importance to those following career paths in the private/industrial sector.

Objectives and summary of previous experimental proposals

The main objective of the proposal is to put in place routine training accesses for MSci students with a transformative use of research instrumentation for teaching purposes.This activity will specifically target to climb the ladder of instrumentation complexity, with a timing tailored before students' theses assignments.

Since 2015 this approach has been proven successful within the Condensed Matter Laboratory course (responsible R. Senesi- see figure below), MSci in Physics, second semester, in bringing more than 25 students as part of the experimental teams in ISIS experiments, 10 of which are now employees in the above mentioned companies.

There is now the opportunity to: 1) temper the complexity of access through IM@IT; 2) extend the access to students attending the Biological Physics Laboratory course (responsible F. Stellato); 3) establish a path followed by a plan of return on investment by engagement with the community of industrial alumni previously involved in the access.

The proposed measurement on the two classes of samples, namely a polymer- carbon nanotube composite and protein microcrystals, convey both training aspects on the identification of interfaces, multi scale morphologies, amorphous/crystalline content, and at the same time provide opportunities to gain an insight into the polymer-nanotube interaction and penetration depths and distribution and to establish methods to discriminate between organic and inorganic crystals, which are currently under investigation in the proponents'research programmes.[Fazi 2023, Stellato 2014]

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Proposed experiment

Request of access to two instruments: confocal and fluorescence microscopies on composite samples, and e-Gate to ISIS. Instrument time of 2 days each for Confocal Microscope 3 and Fluorescence Microscopy is estimated to be sufficient for the purpose of the present proposal. E-gate to INES beamline will be also requested.

References

[Fazi2023] Fazi, L. et al, Molecules 28, 1674 (2023)

[Stellato2014] Stellato, F, *IUCrJ* 1.4, 204-212 (2014)

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Experiment Proposal

Experiment number GP2023079

Principal investigator	Professor Daniela Maggioni, Università degli Studi di Milano, ITALY
Co-investigator	Professor Laura D&039;Alfonso, Università degli Studi di Milano-Bicocca, ITALY
Co-investigator (*)	Professor Giuseppe Chirico, Università degli Studi di Milano-Bicocca, ITALY
Co-investigator	Miss Veronica Schifano, Università degli Studi di Milano, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Fluorescence microscopy characterization of MULtimodal Anticancer Nanohybrids (MULAN)
MRF Instrument	**Fluorescence Microscopy** **Days requested:** 3
Access Route	Direct Access **Previous GP Number:** no, I have not
Science Areas	Biology and Bio-materials, Chemistry, Physics **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	The project aims at developing multimodal organic-inorganic hybrid nanomaterials (multifunctional nanohybrids, NH), which combine different tools in a single carrier for cancer treatment. The combination of photodynamic (PDT) and plasmon photothermal (PPTT) therapies can result in synergic cancer treatment improving the targeting by localized photosensitizer (PS) light-switching. The NH will be constituted by linear PAA as a versatile backbone, decorated with ruthenium (Ru) complexes as PS and gold nanostars (GNS), to achieve light-responsive behaviour for diagnosis and targeted treatment. Fluorescence microscopy will be used both to characterize the cellular uptake and distribution of the NH and to assess the extent of the damage induced by PDT and/or PPTT under varying experimental conditions.
Publications	-

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact Professor Giuseppe Chirico, Università degli Studi di Milano-Bicocca, ITALY
MRF Instrument **Fluorescence Microscopy** **Days Requested:** 3
Special requirements:

SAMPLE

Material	Gold nanoparticles, polyamidoamines, Ruthenium complexes	-	-
Formula	-	-	-
Forms	Solid		
Volume	cc		
Weight	mg		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

MULtimodal Anticancer Nanohybrids (MULAN)

1. Background and Context

Emerging approaches in cancer treatments point to the tumour selective destruction by light excitation of suitable sensitizers. The combination of photodynamic (PDT) and plasmon photothermal (PPTT) therapies can result in synergic cancer treatment improving the targeting by localized photosensitizer (PS) light-switching. For both applications, nanoparticles (NPs) have shown unique potential, though it is important to characterise their interactions with cells. This multimodal approach can be extended combining the efficacy of plasmon NPs with long-lived emitting molecules producing cytotoxic $^1O_2$.

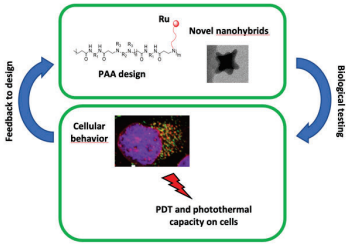
The project aspires at combining on a unique nanosized carrier many therapeutic and diagnostic functionalities, to obtain theranostic products with improved properties as new tools for cancer treatments.

We have already extensively investigated polyamidoamines (PAA), synthetic biodegradable and biocompatible polymers, that can complex small oligos delivering them in the nucleus [1, 2]. Thus might help NPs to escape endosomes and reach cytosol or the nucleus, where PDT effect is stronger. We have also shown that transition metal complexes bound to PAAs does not loose PS properties, showing even a beneficial effect on the metal-induced cytotoxicity [3]. A collaboration with Groningen University is ongoing and part of a PhD student research project is on this topic.

[1] D. Maggioni et al. *Appl. Mater. & Interfaces* 2020*, 12,* 34576.
[2] D. Maggioni et al. *Inorg. Chem.* 2019, *58*, 14586-14599.
[3] D. Maggioni et al. *Inorg. Chem.* 2015, 54, 544.

2. Proposed experiment

The project aims at developing multimodal organic-inorganic hybrid nanomaterials (**multifunctional nanohybrids, NH**), which combine different tools in a single carrier for cancer treatment. A key feature of the proposed NH is the ability to pass the nuclear membrane barrier, to efficiently transfect cells for gene delivery and treat cells through PDT and PPTT while also allowing imaging. The NH will be constituted by **linear PAA** as a versatile backbone, decorated with **ruthenium (Ru) complexes** as PS and **gold nanostars** (**GNS**), to achieve light-responsive behaviour for diagnosis and targeted treatment.



Scheme of the proposed experiment

We plan to test cell uptake and PAA intracellular trafficking on model cancer cells. By imaging correlation methods, exploiting Ru NIR luminescence, we will monitor PAA and NHs ability to pass cell membranes. Uptake kinetics, light-triggered endosomal escape and their final intracellular location will be studied as well.

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The PPTT efficacy of the selected NH colloids, relying on the GNS photothermal features, will be studied in detail vs. irradiation wavelength (700-1000 nm NIR region) at increasing laser intensities. The thermal response of the samples will be recorded by a thermocamera and the heating profiles will allow to determine both the process characteristic times and the associated temperature enhancements.

PDT and PPTT efficacy of NHs will be measured after NIR irradiation for variable time intervals and irradiation intensities on cell cultures. We will determine what NH intracellular locations give higher PDT efficacy.

3. Justification of experimental time requested

The **fluorescence microscopy** setup at the University of Milano-Bicocca, led by Prof. G.Chirico, comprising a tunable pulsed NIR laser and a scanning microscope, will allow us to both detect the distribution of the developed NHs inside the cells (low power excitation of the NPs luminescence) and to investigate the combined PDT and PPTT effects induced by high power irradiation obtained from the same laser source.

We are going to need a total of 3 days on the setup:
1 day to acquire images of the NHs in cells vs time at different incubation times (0h, 24h, 48h);
1 day to study the NH thermal response at different wavelengths (5) and powers (5);
1 day to acquire after irradiation images of the cells to determine PDT and PPTT efficacy.

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Experiment Proposal

Experiment number GP2023089

Principal investigator (*)	Dr Marco Torelli, Adamas Nanotechnolgoies, Inc., UNITED_STATES
Co-investigator	Dr Andrea Morales, QZabre Ltd, SWITZERLAND
Co-investigator	Professor Massimo Bonini, CSGI - University of Florence, ITALY
Co-investigator	Dr Olga Shenderova, Adamas Nanotechnologies, UNITED_STATES
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Characterization of Nitrogen-Vacancy Centers for Improved Quantum Sensing
Training MRF	**Fluorescence Microscopy** **Days requested:** 2
Access Route	Direct Access **Previous GP Number:** No
Science Areas	Biology and Bio-materials, Chemistry, Energy, Materials, Medicine, Physics **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

Adamas Nanotechnologies, a global producer of nanodiamonds with nitrogen-vacancy (NV) centers used for quantum sensing, is proposing to partner with Qzabre, a leader in advanced optics, to characterize advancements made to diamond materials to create the next generation of quantum sensors. Adamas is currently researching means to improve the quality of NV centers within diamond, including by modification of the lattice and surface functionality. For quantum sensing applications, correlating these modifications with improvements in optical NV properties such as optical spin-lattice (T1) and spin-spin (T2) coherence times is critical to development. This proposal will provide means for Q-Zabre to analyze samples to provide iterative information on treatments such that the best possible material is developed. An outcome for quantum researchers is that this information can be made available such that they can choose the most appropriate material for their given application.

Publications -

Internationa MRFs	**QSM - Quantum Scanning Microscope**	**Days requested:** 2
ISIS neutron and muon source		**IM@IT E-platform:** No
Instruments		**Days Requested:**
Access Route		**Previous RB Number:**
Science Areas		**DOI:**
Sponsored Grant		**Sponsor:**
Grant Title		**Grant Number:**
Start Date		**Finish Date:**
Similar Submission?		
Industrial Links		

ISIS@MACH ITALIA

Science and Technology Facilities Council
ISIS Neutron and Muon Source

Sample record sheet

Principal contact	Dr Marco Torelli, Adamas Nanotechnolgoies, Inc., UNITED_STATES	
Training Instrument	**Fluorescence Microscopy**	**Days Requested:** 2
Special requirements:		

SAMPLE

Material	-	-	-
Formula	-	-	-
Forms			
Volume			
Weight			
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	-	-	-
Pressure Range	-	-	-
Magnetic field range	-	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	-	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	-	-	-
Sensitivity to vapour	-	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	-	-	-

Training Case: Characterization of Nitrogen-Vacancy Centers for Improved Quantum Sensing

1. Background and Context

Fluorescent live-cell imaging is one of the leading technologies in fundamental and translational biology correlating individual protein function with biological outcomes, however, bleaching of organic reporters used in protein conformational studies and scarcity of local physiological parameters limit its exploratory and translational capability. Developments in quantum sensing are aimed to surpass the limits of classical measurements in sensitivity, resolution, and speed to improve the understanding of complex events in molecular disease biology and enable new modalities for drug and biomarker discovery. Fluorescent nanodiamond containing nitrogen-vacancy (NV) centers (NDNV) has potential as a novel sensor for single-molecule studies. NDNV are capable of sensing local electromagnetic fields, free radicals, temperature and pH with nanoscale resolution. The ability to measure particle orientation allows NDNV to report protein conformational dynamics in physiological conditions with high spatiotemporal resolution. At molecular sizes (<20 nm) however, a large fraction of these NV^- emitters become inactive due to charge transfer and electromagnetic noise from a particle's surface. Currently, Adamas is developing methods to improve the quality of NV^- emitters, both by improvement of the lattice quality through specialized thermal treatments methods in addition to surface treatments which stabilize the particle surface. These treatments have successfully shown that the proportion of NV^- to NV^0 can be altered as validated by fluorescence emission spectroscopy (Fig 1). However, these changes have not yet been correlated to quantum characteristics relevant to sensing, specifically optical T1 and T2 coherence times. Correlating the changes in NV^- content with these optical properties will be critical in optimizing selection of the functionalization methods. Moreover, this data is important for users of these materials in selecting candidates for use. Currently however, this optical data is difficult to obtain.

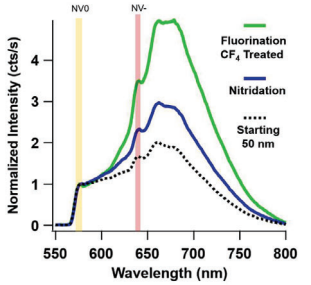

Figure 1: Comparison of fluorescence emission after plasma fluorination and nitridation treatment of standard 50 nm carboxylated particles. Spectra were normalized to NV0 for comparison of NV^- character. Both nitridation and fluorination produce a significant enhancement of NV^- content, with CF_4 producing a more dramatic change.

2. Proposed Training

The proposed work leverages the unique capabilities of the ISIS@MACH Italia coalition to bring these measurements to fruition. Involvement will consist of MRF1 staff (both at Milano Bicocca Unit and at Q-Zabre International MRF) interfacing with Adamas staff, where novel samples are provided for characterization. This will provide a basis to learn how to best prepare samples on the Adamas side, while allowing Q-Zabre to determine the best way to analyse samples. It is anticipated there will be some optimization required. The quantum microscope to be used is unique in providing optical T1 and T2 characterization which is specific to NV centers in diamond.

3. Summary of previous training proposals

This materials development has been funded by DOE SBIR phase I & phase II grants DE-SC0022441 and DOE SBIR phase I grant DE-SC0022858, however these did not include advanced characterization proposed here.

4. Justification of experimental proposals request

Q-Zabre's quantum microscope is capable of providing the necessary characterization to advance Adamas' material. Currently this capability is difficult to find. It is expected that this initial collaboration will be a strong start in working together in the future towards other projects. Two days total are proposed. This time is expected to be sufficient for two rounds of iterative particle characterization, where adjustments to particle functionalization are made based on the characterization, with some initial time for optimization of data collection. The MRF1 fluorescent microscope likewise will provide complementary characterization. Diamond has the capability for two-photon excitation, however this ability is often underutilized. Data relating to these processes will more thoroughly describe any optical changes occurring through modification of the diamond particles.

Sample	
Material:	Diamond nanoparticles dried onto substrate
Form:	Solid
Weight:	<100 mg
Substrate:	Glass
Storage Requirements: RT	

Sample Environment	
Temperature Range:	Room Temp
Pressure Range:	Atmospheric

Safety	
Prep Lab Needed:	Minimal
Sample Prep Hazards:	No
Sensitive to Air:	No
Additional Details:	Diamond is well-demonstrated to be biologically non-toxic. Films will prevent any dust/particle hazard
Samples will be:	Discarded or returned

NMR 600 MHz

NMR 600 MHz

Experiment Proposal

Experiment number GP2023069

Principal investigator (*)	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
Co-investigator	Ms Margherita Simoni, University of Rome Tor Vergata, ITALY
Co-investigator	Mr Matteo Castellani, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Cristina Airoldi, University of Milano-Bicocca, ITALY
Co-investigator	Dr Alessandro Palmioli, University of Milano Bicocca, ITALY
Co-investigator	Professor Emiliano Fratini, CSGI - Università Degli Studi DI Firenze, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Training on the use of NMR spectroscopy to characterize phantom materials for neutron therapy
Training MRF	**NMR 600 MHz** **Days requested:** 2
Access Route	Direct Access **Previous GP Number:** -
Science Areas	Medicine, Technique Development **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

We propose a training activity to establish and validate a quantitative procedure whereby information from nuclear magnetic resonance (NMR) spectroscopy and imaging is used to provide patient-specific neutron scattering libraries to be used to optimize transport codes in neutron capture therapy. In particular, using chemical shift spectra from 1H-NMR, the idea is to reconstruct the relative concentrations of organic functional groups in standard samples, and use these data to build the sample-specific macroscopic cross section at thermal neutron energies using the Average Functional Group Approximation. Therefore, we propose to collect NMR spectroscopy data on samples prepared in a controlled procedure and previously characterized using neutron transmission measurements at the ISIS Neutron and Muon Source. Samples are hydrogel systems composed of semi-interpenetrated polymer networks(SIPN) to be suggested and used as radiation protection standards.

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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ISIS Neutron and Muon Source

Sample record sheet

Principal contact	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
Training Instrument	**NMR 600 MHz** **Days Requested:** 2
Special requirements:	

SAMPLE

Material	-	-	-
Formula	-	-	-
Forms			
Volume			
Weight			
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	-	-	-
Pressure Range	-	-	-
Magnetic field range	-	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	-	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	-	-	-
Sensitivity to vapour	-	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	-	-	-

Background and Context

Neutron capture therapy [1] is a cancer-treatment technique based on the irradiation of the human body with epithermal neutrons, their moderation within organic matter, and their eventual absorption by suitable drugs, often rich in boron, taken by the patient and delivered to the cancer region. Following neutron absorption, the heavy ions produced in the nuclear reaction deliver a large amount of energy over small regions of few micrometres, destroying cancer cells without affecting heathy ones. At present, the transport and moderation of neutrons in the human body is modelled using well-characterized materials, such as polymethyl methacrylate (pMMA) phantoms, in a crude simplification of the complexity of the human body. A possible way forward would be to reconstruct the chemical and physical composition of the region of interest in a patient-specific manner, using phenomenological libraries specific of the actual molecular composition. Proton magnetic resonance spectroscopy [2] methods can provide a quantitative information on the molecular composition of human body (e.g., see the case of fat and water in Ref. [3]), by relaxometry and chemical-shift-based approaches. In both cases, properties related to the presence of hydrogen atoms in specific functional groups are related to their abundance in any voxel of the volume under investigation. While the thermal neutron cross section of specific organic systems is known only for a handful of materials, a method was recently presented, referred to as the Average Functional Group Approximation (AFGA) [4], that allows the accurate prediction of the mass attenuation factor at thermal neutron energies of hydrogen-rich materials by a simple rationalisation of its constituent functional groups. Within this framework, one can hypothesize a procedure whereby the information on the abundances of hydrogen-containing functional groups, provided, e.g., by NMR spectroscopy or chemical-shift imaging, is given as an input to a transport code where the position-specific neutron attenuation functions are calculated using AFGA. If reliable, such procedure would allow safer, patient-specific, and more efficient treatments in the case of neutron capture therapy.

Proposed Training

We propose a training activity to perform a series of measurements to show the accuracy and/or limitations of ^{1}H-NMR spectroscopy in the solid phase and in solution, either using or not High-Resolution Magic Angle Spinning (HR-MAS), and to establish the effect of the relaxation times on the evaluation of the functional-group populations in the final applications, likely to be in the solid state. We propose to carry out the measurements on two samples of polyhydroxyethylmethacrylate (pHEMA) at different hydration levels (pHEMA+10% H_2O and pHEMA+40% H_2O), previously characterized with neutrons (see Figure 1). pHEMA represents a material with a similar stoichiometry to pMMA yet with the possibility of including some amounts of water, as a first step to better reproduce human body.

We propose a training activity to be carried out by the instrument scientists of the NMR Spectrometer (academic staff from the University of Milano-Bicocca) towards the other members of the experimental team (academic staff and 2 Ph.D/M.Sc students) from the University of Rome Tor Vergata and CSGI). The need of an NMR spectrometer is dictated by the final applications related to NMR imaging at medical facilities and its availability at the IM@IT unit of Milano-Bicocca represents an opportunity to consolidate cooperation across different units of the ISIS@MACH ITALIA Research Infrastructure, as well as the ISIS Neutron and Muon Source.

The need for testing the samples in the several experimental preparation possibilities is dictated by the fact that the pHEMA+H2O samples would more suitably be measured via liquid NMR, thus losing part of the information on the amount adsorbed water, while the final application would rather require solid NMR. Understanding the limitations of such approaches is vital for the final medical application.

Summary of previous proposals and characterizations The samples for the NMR training were synthesized at the IM@IT – CSGI unit in Florence and characterized at the ISIS Neutron and Muon Source using neutron transmission experiments (ISIS Proposal RB 2310166 carried out in July 2023) to measure their mass attenuation factors as functions of the incident neutron energy. Preliminary results, to be benchmarked by NMR characterizations, are reported in Figure 1 as percentage difference of pHEMA samples compared with standard pMMA, both in the case of the experimental data (solid line) and model predictions using AFGA (dashed line).

Justification of experimental proposals request

We request two days of time to use the **NMR spectrometer (Bruker Avance III 600 MHz NMR spectrometer**) available at the IM@IT – Milano-Bicocca unit. Having discussed the training plan with the instrument scientist, we envisage 0.5 days as a general introduction to the instrument set-up and how to run a measurement, and 0.25 days to test two samples in three different configurations (solid, solution and HR-MAS). Therefore, we request 0.5 + 3x2x0.25 = 2.0 days of instrument time.

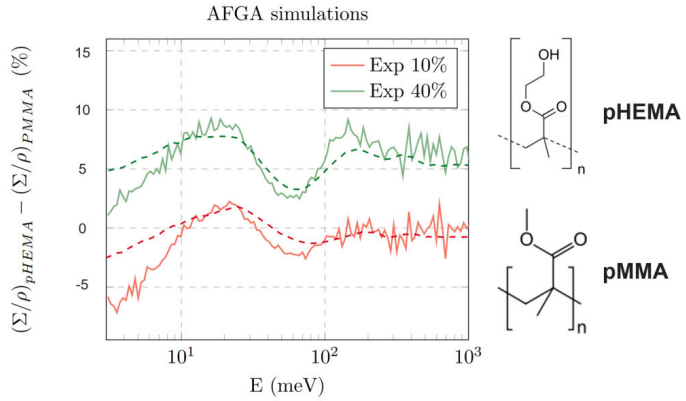

Figure 1. Relative difference between the mass attenuation factor of pHEMA (at two hydration levels) and pMMA, both experimental and modelled. The difference, in some regions of the order of 5-10%, can be used as a simple way to assess the error in the dose calculation in a patient depending on the phantom used during the simulation stage of treatment planning.

References

[1] Z.P. Zagorski, Radiation Physics and Chemistry 56, 559–565 (1999).
[2] Nuclear Magnetic Resonance Spectroscopy, J.B. Lambert and E.P. Mazzola (2004).
[3] H. H. Hu and H. E. Kan, NMR Biomed., 26(12), 1609–1629 (2013).
[4] G. Romanelli et al., J. Phys.: Condens. Matter 33, 285901 (2021).

Raman Confocal Microscope

Raman Confocal Microscope

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Science and Technology Facilities Council
ISIS Neutron and Muon Source

Experiment Proposal

Experiment number GP2023085

Principal investigator (*)	Dr Claudio Resta, Enapter SRL, ITALY
Co-investigator	Dr Gabriele Agonigi, enapter s.r.l., ITALY
Co-investigator	Dr Massimo Rosa, Enapter s.r.l., ITALY
Co-investigator	Dr Antonio Filpi, Enapter srl, ITALY
Co-investigator	Mr Stefano Catanorchi, Enapter S.r.L., ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Training for Confocal Raman Microscopy on Membrane-electrode assembly components
Training MRF	**Raman Confocal Microscope** — **Days requested:** 2
Access Route	Direct Access — **Previous GP Number:** 2023031
Science Areas	Chemistry, Energy, Environment — **DOI:** -
Sponsored Grant	None — **Sponsor:** -
Grant Title	- — **Grant Number:** -
Start Date	- — **Finish Date:** -
Similar Submission?	-
Industrial Links	Enapter s.r.l.

Non-Technical Abstract

Enapter produces scalable and modular AEM electrolysers, a relatively new technology, to produce hydrogen and oxygen from water splitting electrochemical reaction. Key components are MEA (Membrane Electrode Assemblies) and PTL (Porous Transport Layer). AEM technology combines advantages of both classical alkaline and PEM water electrolysis, to produce high purity hydrogen at relatively high pressure and high current density without using expensive or scarce materials (e.g. Tit, Ir, Pl). Our research programmes would relevantly benefit by using powerful characterization techniques . Potentialities of those techniques have only been barely explored in companies' framework and may constitute a breakthrough on the analysis of the MEA components in AEM systems. Specifically, the possibility to receive a training would, in our opinion, constitute a remarkable asset and grant a more proficient and effective investigation and technology development.

Publications -

ISIS neutron and muon source — **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Science and Technology Facilities Council
ISIS Neutron and Muon Source

Sample record sheet

Principal contact	Dr Claudio Resta, Enapter SRL, ITALY		
Training Instrument	**Raman Confocal Microscope**	**Days Requested:** 2	
Special requirements:			

SAMPLE

Material	-	-	-
Formula	-	-	-
Forms			
Volume			
Weight			
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	-	-	-
Pressure Range	-	-	-
Magnetic field range	-	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	-	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	-	-	-
Sensitivity to vapour	-	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	-	-	-

Case for ISIS@MACH ITALIA Training Proposal

Training on Confocal Raman Microscopy for Membrane-electrode assembly components analysis

1. Background and Context

Enapter produces scalable and modular AEM electrolysers to produce hydrogen and oxygen from water splitting electrochemical reaction. Key components to allow efficient and durable performances are MEA (Membrane Electrode Assemblies) and PTL (Porous Transport Layer). AEM technology combines advantages of both classical alkaline and PEM water electrolysis, allowing to produce high purity hydrogen at relatively high pressure and high current density without using expensive or scarce materials (e.g. Titanium, Iridium, Platinum). Being the AEM technology relatively new, every single constituent of the final product needs to be extensively characterized to provide a deeper knowledge and speed up technological improvements. (e.g. connection between morphology and physical-chemical properties). Due to the novelty of the technology, very few advanced characterization techniques are routinely used in the field. Preliminary data showed that our research programmes could relevantly benefit by having access and gaining expertise on some powerful characterization techniques (like confocal Raman microscopy); potentialities of those techniques have only been barely explored in companies' framework and may constitute a breakthrough on the analysis of the MEA components in AEM systems. Additionally, the possibility to receive a specific training would, in our opinion, constitute a remarkable asset and grant a more proficient and effective investigation and technology development. Our main financial support comes from the holder Enapter AG, additionally Enapter earned a grant from PNRR programme from italian government and it is involved in an Horizon 2020 project ("CHANNEL").

2. Proposed training

This training proposal would allow 5 members of the R&D chemistry department of Enapter srl to gain expertise in Confocal Raman technique. All the selected members are chemists with relevant expertise in the field of AEM electrolysis and material characterization, Confocal Raman Microscopy can, in our opinion, be important to understand the morphological and chemical organisation and composition of the different materials they are composed of. In particular, Raman spectroscopy combined with confocal microscopy can highlight the distribution of organic and inorganic portions on the surface and through the cross section. Being sensible to different organic moieties and allowing for the analysis of selected regions of the sample, this technique could spot if chemical modification occurred on the different materials during its manufacturing or if the different organic components composing the MEA have preferential distributions. Some of the components of our devices already proved to be suitable to be investigated through confocal Raman microscopy. Being the simultaneous characterisation of their morphology and composition the final aim of our investigation, we need to acquire a deeper and more comprehensive knowledge of this technique. In our opinion this would guide us in the data analysis and modelling and help us to understand its actual potential. The training would be carried out at CSGI – "Consorzio interuniversitario per lo Sviluppo dei sistemi a Grande Interfase" – Università degli Studi di Firenze, with whom we are already in contact and agreed on.

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3. Summary of previous training proposals

No previous training proposal has been presented. However, in a first round of measures, Raman spectroscopy has been proved to be suitable to investigate the morphological organization of MEA components as well as the distribution of organic and inorganic compounds throughout it. Since MEA components have been extensively characterised by Enapter srl in terms of their performances, Raman spectroscopy would allow to cross-correlate performances with morphological/compositional results and extract general guidelines for the development of efficient MEA components.

4. Justification of training proposals request

Based on the previous experiments (GP2023031), Raman confocal microscopy would be very helpful in correlating MEA performances with their component's distribution and morphology, thus guiding MEA optimization. Since is a unique instrument when aiming at the characterisation of morphological and compositional properties of polymeric and composite surfaces we are interested in gaining more practice on the instrument. At the same time, with the training we aim to better learn instrument features as well as be independent in the interpretation of the results. In our experiment, we target the acquisition of compositional maps of the surface of MEA components. Given the number of samples and the time typically required to acquire maps with accurate resolution, following the suggestions by the ISIS@MACH Italia team we request two days of training.

SAXS GISAXS *SAXS GISAXS*

Experiment Proposal

Experiment number GP2023051

Principal investigator	Professor Alessandro Cianchi, University of Rome Tor Vergata , ITALY
Co-investigator (*)	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Enrica Chiadroni, Sapienza University of Rome, ITALY
Co-investigator	Dr Luigi Faillace, INFN-LNF - Frascati, Rome, ITALY
Co-investigator	Dr Mario Galletti, Universitá di Roma Tor Vergata, ITALY
Co-investigator	Dr Andrea Liedl, INFN, ITALY
Co-investigator	Dr Riccardo Pompili, INFN, ITALY
Co-investigator	Professor Marco Laurati, CSGI, ITALY
Co-investigator	
Experiment title	GISAXS characterization of cathodes for photoinjectors
MRF Instrument	**SAXS GISAXS**
Access Route	Direct Access
Science Areas	Materials, Physics
Sponsored Grant	None
Grant Title	-
Start Date	-
Similar Submission?	-
Industrial Links	-

Days requested: 2
Previous GP Number: 2023003
DOI: -
Sponsor: -
Grant Number: -
Finish Date: -

Non-Technical Abstract

High brightness machines, like Free Electron lasers, are driven by photoinjectors. The quality of the electron beam extracted from the source is paramount for the whole machine's performance. Copper cathodes are widely used, as electron sources, for their reasonable quantum efficiency, robustness, simple treatment, and implementation. However, copper cathodes, exposed to strong electric fields and continuously bombarded with high-intensity lasers, can degrade their quantum efficiency. To this end, we have performed preliminary experiments at IM@IT investigating the morphology of our copper cathodes, using SEM - EDX and Profilometry, which evidenced structures up to the micrometer scale. To complement these characterizations, we propose to study the same cathodes, either after having been used or without exposure to the laser, with GISAXS. This is a resubmission related to proposal GP2023003, for which the Panel was unable to allocate instrument time because of oversubscription.

Publications

J. Scifo et al, Nucl. Inst. Meth. A., 909, 2018, 233-238 https://doi.org/10.1016/j.nima.2018.01.041

ISIS neutron and muon source

E-platform: No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

ISIS@MACH ITALIA

Science and Technology Facilities Council
ISIS Neutron and Muon Source

Sample record sheet

Principal contact Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
MRF Instrument **SAXS GISAXS** **Days Requested:** 2
Special requirements:

SAMPLE

Material	Cu Oxygen free 99.95% exposed to RF	-	-
Formula	Cu	-	-
Forms	Solid		
Volume	50 cc		
Weight	500 g		
Container or substrate	-	-	-
Storage Requirements	limited exposure to air required	-	-

SAMPLE ENVIROMENT

Temperature Range	300 - 300 K	-	-
Pressure Range	0 - 1000 mbar	-	-
Magnetic field range	0 - 0 T	-	-
Standard equipment	None	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	No	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Removed By User	-	-

Scientific Background

High brightness machines, like free electron lasers, are driven by photoinjectors. The quality of the electron beam extracted from the source is paramount for the whole machine's performance. Copper cathodes are widely used, at electron sources such as SPARC_LAB (INFN, Italy) [1], for their reasonable quantum efficiency, robustness, simple treatment, and implementation. However, copper cathodes, exposed to strong electric fields and continuously bombarded with high-intensity lasers, can degrade their quantum efficiency for several reasons. The most common problem is surface contamination, mainly due to carbon ions, but also thermal stress can induce dislocations inside the material, leading to extrusion, creating a tip where the electric field can grow up, thus causing a dangerous discharge. The change in quantum efficiency or, even worse, the difference in this parameter point by point can dramatically affect the beam properties, degrading the emittance and producing poor radiation source performances.

To better understand the degrade in quantum efficiency of copper cathodes, surface analyses can be used to estimate both contamination and thermal-induced roughness before and after exposure to the RF laser, as well as after nanomachining [2]. The nanomachining process consists in diamond milling and blowing with dry nitrogen. This procedure reduces the roughness of the cathode surface to about 5 nm and prevents surface contamination introduced by other techniques, such as polishing with diamond paste or the machining with oil. While during use and exposure to high-energy lasers the roughness can increase to several micrometres, resolution of experimental techniques to characterise the surface of copper cathodes needs to span over several spatial scales.

Here, we propose to complement preliminary characterizations of exposed copper cathodes at the micrometre spatial scale, with a characterization of the roughness at the nanometre scale using Grazing-Incidence Small Angle X-ray Scattering (GISAXS) using the SAXS Xenocs Xeuss instrument of the Medium Range Facility 1 - FOURDIM. This is a **resubmission related to proposal GP2023003**, for which the Panel was unable to allocate instrument time because of oversubscription of the instrument.

Previous Characterizations

Previous characterizations, using the Small Research Facilities available at the ISIS@MACH ITALIA laboratories of the unit at University of Rome Tor Vergata (GP2022013), have shown the presence of a series of craters at the micrometre scale, whose frequency of appearance decreases going from the centre of the copper cathode, where the high-intensity laser hits the surface more often, towards the border. Such trend can be appreciated looking at the series of SEM images in Figure 1. A detail of a region with several craters, each with average dimensions of the order of 10 micrometres, is provided in Figure 2 using both secondary and back-scattered electrons. The roughness level in a region including a crater was measured using a profilometer to about 2 µm, while in a region just outside the border of the crater to about 60-80 nm, a value approaching the one for a recently nano-machined surface. However, such value of roughness is approximately at the resolution limit of the profilometer, and the estimate of the roughness in regions further away from the beam centre becomes limited by the instrument resolution.

Proposed Experiment

We propose to perform a GISAXS experiment using the SAXS Xenocs Xeuss instrument at the MRF1 – FOURDIM of the CSGI – University of Florence Unit of ISIS@MACH ITALIA. By analysing the SAXS patterns in the grazing incidence geometry as a function of the distance from the cathode centre, we aim at measuring the roughness value at the nanometre scale, checking whether it approaches the nominal value of few nanometres, obtained after nanomachining, in the outer regions where the high-intensity laser is expected

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to have created a negligible number of craters. Experiments will be performed on both an exposed copper cathode and on a nanomachined one. We expect the SAXS patterns to be dominated by the roughness contribution, as the cathode is about 2 cm thick and only composed of copper. A distorted wave Born approximation (DWBA) will be used to analyse the roughness distribution [3] in the 1 – 100 nm range.

To perform this experiment, we request 2 days of the GISAXS instrument at the MRF1 – FOURDIM facility.

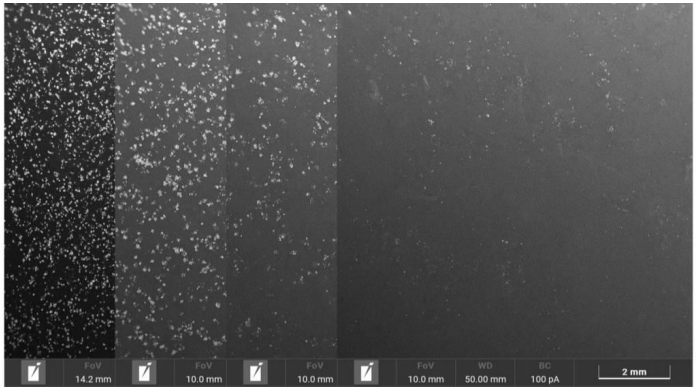

Figure 1 – Series of SEM images of the copper cathode surface, moving from the centre (left image) to the outer region (right image) showing the different frequency of craters.

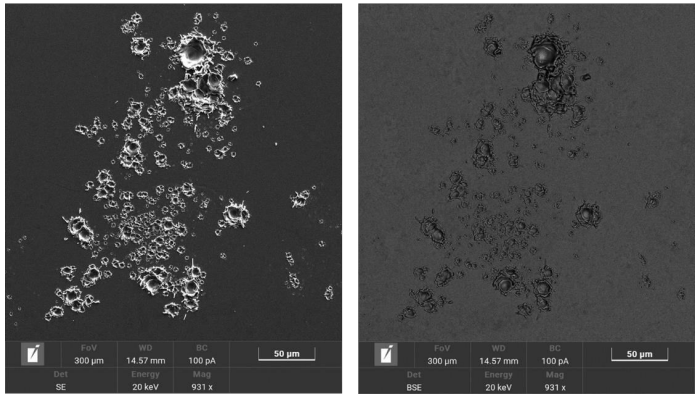

Figure 2 – SEM images of the copper cathode surface showing craters at the micrometre scale using secondary (left) and back-scattered electrons (right).

References

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Experiment Proposal

Experiment number GP2023052

Principal investigator	Mrs Valentina Turina, Fondazione Museo Antichità Egizie, ITALY
Co-investigator (*)	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Carla Andreani, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Lucy Skinner, University of Northampton and the British Museum, UNITED_KINGDOM
Co-investigator	Dr Robert Robinson, University of Wollongong, AUSTRALIA
Co-investigator	Professor Salima Ikram, American University in Cairo, EGYPT
Co-investigator	Miss Giulia Pallottini, Fondazione Museo Antichità Egizie, ITALY
Co-investigator	
Experiment title	Characterization of collagen and both tanning and colouring materials on leather artefacts from Museo Egizio by WAXS/SAXS/USAXS measurements
MRF Instrument	**SAXS GISAXS** **Days requested:** 2
Access Route	Direct Access **Previous GP Number:** GP2023002
Science Areas	Cultural Heritage, Materials, Physics **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	Fondazione Museo Egizio
Non-Technical Abstract	Within the Museo Egizio collection there are 200 precious and unique leather artefacts belonging to different historical periods including the Old Kingdom, New Kingdom, Roman and Byzantine eras. Hence, it is paramount to understand degradation mechanism of ancient leather probably related to the way the skins were prepared and made durable. The proponents aim to study by WAXS/SAXS/USAXS the assembly and orientation of the collagen fibrils in the samples and extend by means of FT-IR and Raman spectroscopy measurements its characterization and both tanning and colouring materials found in ancient leather. In the present proposal, a resubmission of (GP2023002), we wish to measure the degree of assembly of the collagen fibrils of ancient leather artifacts using WAXS/SAXS/USAXS measurements on the SAXS GISAXS instrument.
Publications	G. Romanelli, et al., “Neutron-Enhanced Information on the Laboratory Characterization of Ancient Egyptian Leathers...”, Information, 2022, 13, 467 G. Pallottini, Graduate Thesis, "La coperta Provv.5062 del Museo Egizio di Torino: studio, restauro e valorizzazione" (2021).

ANSTO Reactor

Brief abstract	Two other measurements for performing 2D/3D neutron imaging and neutron USANS have been scheduled to DINGO and KOOKABURRA neutron beamlines at ANSTO (Australia), respectively.

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Sample record sheet

Principal contact	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY		
MRF Instrument	**SAXS GISAXS**	**Days Requested:** 2	
Special requirements:			
	SAMPLE		
Material	Leather	-	-
Formula	Collagen	-	-
Forms	Solid		
Volume	1 cc		
Weight	1 mg		
Container or substrate	-	-	-
Storage Requirements	-	-	-
	SAMPLE ENVIROMENT		
Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	-	-	-
Special equipment	-	-	-
	SAFETY		
Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Characterization of collagen and both tanning and colouring materials on leather artefacts from Museo Egizio by WAXS/SAXS/USAXS measurements

1. Background and Context

The collection of the Museo Egizio (Turin) houses over 200 leather artifacts belonging to different historical periods, including the Old Kingdom, New Kingdom, Roman and Byzantine eras. Leather is the main material and the only common element of these objects. Leather was used throughout the entire society, from low to high status and often subject to a variety of uses, from decorative to intense use. Ancient leather presents a heterogeneous composition of both organic and inorganic materials that show an evident reactivity. Its proper preservation remains challenging as some aspects of its chemical composition, degradation and effectiveness of conservation treatments are still not fully understood. Archeologists and conservators were able to identify, through the constant conservative monitoring of the artifacts, different types of degradation and, above all, a correspondence between their dating and the type of documented deterioration. Indeed, the different types of degradation are probably related to the way the skins were prepared and made durable. Of particular concern for the collection of Museo Egizio (Turin) is that the skin processing method (including any coloring treatments) and the substances used to make it more durable are not known. Not so many processes are attested for this period [2], and the substances that were used to treat the skin and the likely connection with the types of deterioration that are documented are closely linked to collagen, the most important fibrous protein. Collagen is the principal protein constituent of a wide variety of connective tissues in animals. Its structure has been investigated extensively by electron microscopy and by diffraction techniques using X-rays and neutrons [3-8]. Recently [9], a characterization of Egyptian leather samples was completed by combining non-destructive techniques, including surface probes (X-ray fluorescence, Raman scattering, and scanning electron microscopy enhanced by X-ray energy spectroscopy) and neutron-based bulk techniques (inelastic and deep-inelastic neutron scattering).

The proponents aim to study by wide/small/ultra small angle X-ray scattering (WAXS/SAXS/USAXS) the assembly and orientation of the collagen fibrils in the samples already investigated in Ref. [9], and by distinct proposals perform a complementary characterization using both Fourier-transform infrared spectroscopy (FT-IR) and Raman spectroscopy (AFM-Raman). These spectroscopic techniques will be used to study the intra- and inter- molecular vibrational spectra of collagens as well as both the tanning and colouring materials components, most found in ancient leather [10]. Hence, we propose to use the SAXS GISAXS, FT-IR Nexus, and AFM Raman instrument operating at the CSGI-Unifi and the Univ. Tor Vergata Units of in the suite of IM@IT. Further characterization on the same samples will be done by neutron imaging (DINGO beamline) and USANS (Kookaburra beamline) at the Australian Centre for Neutron Scattering (ANSTO).

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2. Proposed experiment for WAXS/SAXS/USAXS

In the present proposal, a resubmission of (GP2023002), we wish to measure the degree of assembly of the collagen fibrils of n. 6 ancient leather artifacts (same samples that will be measure at ANSTO) using WAXS/SAXS/USAXS measurements on the Xenocs Xeuss instrument. Results from WAXS/SAXS/USAXS will be compared and extended by Fourier-transform infrared spectroscopy (FT-IR) and Raman spectroscopy measurements widely used for the characterization of collagen and both tanning and colouring materials found in ancient leather as reported in this work [10], which will be submitted in separate proposals.

3. Justification of experimental time requested for WAXS/SAXS/USAXS

We aim to measure n. 6 ancient leather artifacts (same samples that will be measure at ANSTO) on the Xenocs Xeuss instrument equipped with a copper anode microsource (λ = 0.15405 nm) in the range of scattering vector from 1.5 to around 31 nm^{-1}. We propose to measure small angle X-ray scattering data for each leather sample directly mounted on the sample holder with an acquisition time of 1800 s. Hence, we request two days instrument time including set-up and calibration time.

4. References

[1] E. Schiaparelli, Relazione sui lavori Della Missione Archeologica Italiana in Egitto (anni 1903–1920), second volume: The intact tomb of the architect Kha in the necropolis of Thebes (AdArte, 2008).

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Experiment Proposal

Experiment number GP2023066

Principal investigator (*)	Dr Laura Strolin, Institut Català de Arqueologia Clàssica, SPAIN
Co-investigator	Professor Luisa Cifarelli, University of Bologna and INFN-Bologna, ITALY
Co-investigator	Professor Maria Pia Morigi, University of Bologna, ITALY
Co-investigator	Dr Melissa Kennedy, The University of Sydney, AUSTRALIA
Co-investigator	Dr Thomas Hugh, The University of Sydney, AUSTRALIA
Co-investigator	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Roberto Senesi, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Experiment title	Understanding ritual practices in Neolithic Saudi Arabia using SAXS on horn sheaths from Mustatils

MRF Instrument	**SAXS GISAXS**	**Days requested:**	2
Access Route	Direct Access	**Previous GP Number:**	-
Science Areas	Cultural Heritage	**DOI:**	-
Sponsored Grant	None	**Sponsor:**	-
Grant Title	-	**Grant Number:**	-
Start Date	-	**Finish Date:**	-
Similar Submission?	-		
Industrial Links	-		

Non-Technical Abstract

Archaeological research in Saudi Arabia is going through a period of intense development that is constantly leading to important discoveries. Mustatils are massive stone structures serving ritual purpose that were built in hundreds in Northwest Arabia 7500 years ago by nomadic pastoral populations. An exceptional category of finds is represented by horn sheaths, made of the outer keratin shell of the horn. Due to its organic protein composition, the sheath is usually not preserved in archaeology and lacks research. To shed light on the horn treatment, desiccation through deliberate heating, colouring and degradation, we propose a structural characterization using small-angle Xray scattering and, in a separate proposal, Confocal Raman spectroscopy. A structural characterization at the nm scale is expected to provide information on the the protein structure and, thus, on the desiccation of the material.

Publications

Neutron-Enhanced Information on the Laboratory Characterization of Ancient Egyptian Leathers: Hydration and Preservation Status, G. Romanelli et al., Information, 13, 10, 2022

ISIS neutron and muon source		**E-platform:**	No
Instruments		**Days Requested:**	
Access Route		**Previous RB Number:**	
Science Areas		**DOI:**	
Sponsored Grant		**Sponsor:**	
Grant Title		**Grant Number:**	
Start Date		**Finish Date:**	
Similar Submission?			
Industrial Links			

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Sample record sheet

Principal contact	Dr Laura Strolin, Institut Català de Arqueologia Clàssica, SPAIN		
MRF Instrument	**SAXS GISAXS**	**Days Requested:**	2
Special requirements:			

SAMPLE

Material	Animal horn	-	-
Formula	Keratin	-	-
Forms	Solid		
Volume	5 cc		
Weight	5 g		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Background and Context

Archaeological research in Saudi Arabia is going through a period of intense development that is constantly leading to important discoveries. Namely, previously unknown monumental structures dating back to the Neolithic are being investigated for the first time: the 'mustatils'. Mustatils are massive stone structures serving ritual purpose that were built in hundreds in Northwest Arabia 7500 years ago by nomadic pastoral populations [1-3]. So far, little is known about their culture, economy, and habits. The main finds in mustatils are skulls of selected horned animals (cattle, goat, gazelle), intentionally deposited in specific offering chambers where hearths are also present [2, 4]. Moreover, these faunal remains are the most ancient attestation of domestic cattle and goat in Arabia.

An exceptional category of find is present, the horn sheath, that is the outer keratin shell of the horn. Due to its organic protein composition, the sheath is usually not preserved in archaeology and lacks research. Therefore, the exceptional preservation of horn sheaths in mustatils opens the unique possibility to investigate this material not only for better understanding the ritual universe and technical knowledge of Neolithic nomadic people of ancient Arabia, but also for clarifying the circumstances of sheath desiccation as related to paleoclimatic conditions. In addition, we target possible conservation methods as archaeologic horn is a highly fragile and perishable material.

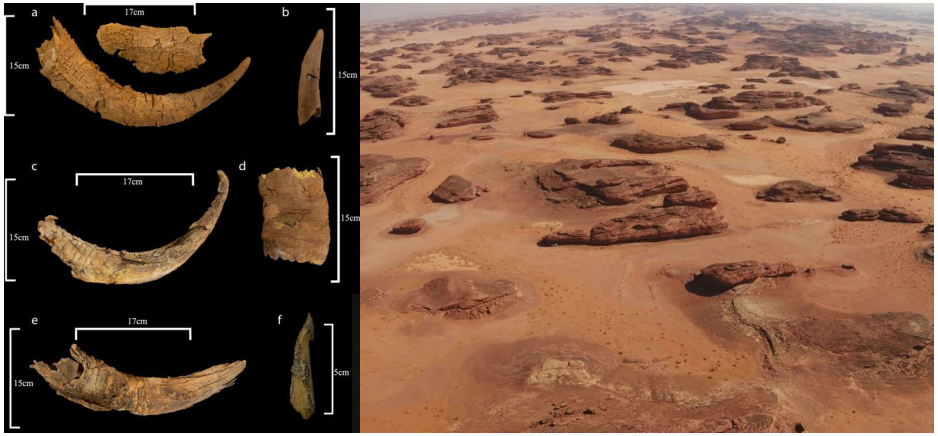

Figure 1: a selection of the horn sheaths found in Mustatil IDIHA-F-0011081 (left); the mustatil in the arid landscape of Northwest Arabia (right).

The research questions motivating this proposal are: were the sheaths treated prior to deposition in the mustatil (as part of the ritual, for preservation purposes) and how? Were some of the sheaths deliberately heated? Why do the sheaths present different colours and levels of degradation? What is their current state of desiccation and degradation?

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Proposed experiment

To answer these questions, we propose a structure characterization of a series of fragments and pieces from a selection of horn sheaths, i.e., smaller portions of the finds in Figure 1 (left), through small-angle x-ray scattering (SAXS) using the MRF located at the CSGI-IM@IT Unit. Structural information will provide details on the aggregation state of individual keratin filaments [5], shedding light on the protein structure and, thus, on the desiccation of the material.

In addition to SAXS investigations, through separate proposals, we will request access to the confocal Raman spectroscopy available at the University of Rome Tor Vergata – IM@IT using the AFM Raman XploRA Plus. Vibrational spectroscopy of the sample surface will provide information on any materials applied on the horn outer layers which prevented degradation of the keratin organic material, as well as shedding light on any desiccation and preservation processes related to such samples and on the reason why different colours are observed. Ancient samples will be compared with modern ones to facilitate the interpretation of the experimental data.

Summary of previous investigations

Mustatils are the object of a wide research programme carried out previously by the University of Western Australia and currently the University of Sydney (Prehistoric AlUla and Khaybar Excavation Project – PAKEP) with the support of the Royal Commission for AlUla. The research focuses on Neolithic mustatils and settlements, as well as on Bronze Age tombs. As such, it aims at enlightening all aspects of ancient societies in the area. It also includes remote sensing, helicopter photography, ground survey, excavation, and material analyses, with a special attention to outreach.

Justification of experimental time requested

We request 2 days of instrument time on the *SAXS GISAXS* MRF located at the CSGI – IM@IT unit, to be used as follows: up to 2 hour of measurements per horn sheath fragment (for a total of about 5 fragments per day) both for the Mustatils finds and for reference horn and keratin samples.

References

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[5] Mattiello S, Guzzini A, Del Giudice A, Santulli C, Antonini M, Lupidi G, Gunnella R. 2022. Physico-Chemical Characterization of Keratin from Wool and Chicken Feathers Extracted Using Refined Chemical Methods. Polymers, 15(1):181

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Experiment Proposal

Experiment number GP2023071

Principal investigator	Professor Lorenz Baumer, Université de Genève, SWITZERLAND
Co-investigator	Professor Luisa Cifarelli, University of Bologna and INFN-Bologna, ITALY
Co-investigator	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Roberto Senesi, University of Rome Tor Vergata, ITALY
Co-investigator (*)	Dr Laura Strolin, Institut Català de Arqueologia Clàssica, SPAIN
Co-investigator	Professor Maria Pia Morigi, University of Bologna, ITALY
Co-investigator	Dr Maria Grazia Griffo, Museo Archeologico Regionale Lilibeo–Marsala, ITALY
Co-investigator	
Co-investigator	
Experiment title	Analysis of nails provided by different antique shipwrecks in the Mediterranean using SAXS
MRF Instrument	**SAXS GISAXS** **Days requested:** 2
Access Route	Direct Access **Previous GP Number:** -
Science Areas	Cultural Heritage **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	Ship nails can provide important information about the construction techniques of ancient ships and, depending on their typology, alloys, and internal structure, deliver information on the ship provenance and travel routes. The study of their production and mechanical treatment allows to approach questions like if there was, all over the Mediterranean a general standardization or not, based on a cultural exchange, or if there are culturally different and chronologically evolving technologies used to produce the nails. Here we propose a structure characterization of several ship nails, from different findspots, and belonging to different cultures and periods, based on X-ray diffraction techniques to provide information on the crystal structure in the surface. In separate proposals, we will request access to neutron techniques and SEM-EDS to characterize the elemental analysis of the materials used, both on the surface and in the bulk.
Publications	-

Instruments	**INES**	**Days Requested:** 2
Access Route	Direct Access	**Previous RB Number:**
Science Areas		**DOI:**
Sponsored Grant	None	**Sponsor:**
Grant Title	-	**Grant Number:**
Start Date	-	**Finish Date:**
Similar Submission?		
Industrial Links		

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Sample record sheet

Principal contact	Dr Laura Strolin, Institut Català de Arqueologia Clàssica, SPAIN		
MRF Instrument	**SAXS GISAXS**	**Days Requested:** 2	
Special requirements:			
	SAMPLE		
Material	Bronze nail	-	-
Formula	Cu, Sn	-	-
Forms	Solid		
Volume	10 cc		
Weight	90 g		
Container or substrate	-	-	-
Storage Requirements	-	-	-
	SAMPLE ENVIROMENT		
Temperature Range	300 - 300 K	-	-
Pressure Range	0 - 1000 mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	-	-	-
Special equipment	-	-	-
	SAFETY		
Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Background and Context

Underwater excavations regularly provide an important number of nails, usually in bronze or copper alloys, used for different purposes. The so-called treenails consisting of a nail driven through a wooden peg are used fixing the planks and the frames of the ship (fig. 1a-1b), whereas wooden pegs have been used to stabilize the tenons keeping the planks in place. Shorter nails have been used to protect the outside of the hull with thin lead sheets. As the analysis of one single plank from the Antikythera shipwreck is showing (fig. 2), there is an enormous number of nails used in antique ship construction (yellow and red dots). The nails are therefore not only a fundamental part of a ship but can also deliver a rich amount of information from scientific analytical methods.

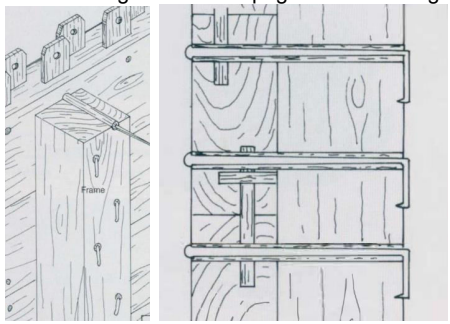

Fig. 1a and 1b: schematic representation of treenails used in antique ship construction.

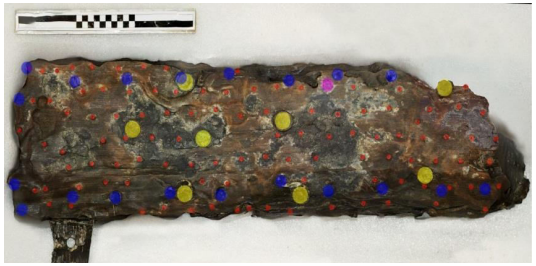

Fig. 2: Analysis of the number of different types of nails used in a single plank from the Antikythera shipwreck, 1st century BC. (yellow: treenails; red: bronze nails; blue: wooden pegs)

While ship construction was during the antiquity a highly specialized industry, e.g., for the Phoenicians, the Greeks, and the Romans, and producing a large series of special ship types for all kind of purposes (war ships, long distance cargo ships, etc.), it is astonishing that the nails, as fundamental as they are, have so far only found little interest. Aside of giving important information about the construction techniques, they can, by their typology, by their alloys, and by their internal structure deliver information e.g., about the provenance and, by analyzing reparations, about the routes of the ships. Moreover, investigating production techniques and mechanical treatment of the nails, will help understanding if there were standard technologies shared in the whole Mediterranean - thanks to cultural exchange - and how they evolved through time.

Proposed experiment

We propose a characterization of several ship nails, coming from different findspots, and belonging to different cultures and periods. In that interest, nails coming from at least three different shipwrecks will be analyzed, allowing the comparison of their metal composition, their provenance, and their mechanical treatment. For the time being, three shipwrecks have been selected as a starting point of the project. From each shipwreck, between 3 and 12 nails will be selected for analysis. The three ships are: the Marsala Punic (Phoenician) military ship (3rd century BC), the Antikythera Greek (?) cargo ship [3, 4, 5, 6] (1st century BC), and the Marausa Roman merchant ship found near Trapani. While surface characterizations were already performed on samples from the Punic ship [2], here we

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plan a **systematic comparison of nails from the three different ships using Small and Wide Angle X-ray Scattering** using the instrument Gi-*SWAXS* XEUSS 3.0HR, located at the CSGI - IM@IT Unit. This instrument is particularly suitable for analysing these samples given it has a suitably large sample volume where ship nails, with linear dimensions of the order of 10 cm, can be easily accommodated. Specifically, the crystal structure over a wide spatial scale will be assessed, mainly on the edges and external surfaces of the artifact, due to the high Z numbers of the main elements in bronze, providing information on the manufacturing techniques of the nails, thus on their provenance.

Information on the crystal structures of the outer layers gathered with the SAXS/WAXD will be complemented requesting, in a distinct proposal, a Scanning Electron Microscopy and Energy Dispersive X-Ray Spectroscopy (SEM-EDS) analysis using the instrument located at the Tor Vergata Unit, and neutron diffraction and neutron resonance capture analysis at the INES beamline of the ISIS Facility. The combination of this set of analyses will give us comprehensive information on the elemental composition and structure (manufacturing), both on the surface and in the bulk, as well as provide information on the manufacturing procedures of these artifacts and on their origin.

Summary of previous characterizations.

As for today, only the nails of the Marsala Punic ship have found a partial analysis, whereas the nails of the two other ships remain unstudied. The members of the research group have already been working on some of the shipwrecks that will deliver the samples: 3D tomography of a few planking elements has already been done for the Marsala Punic ship, providing important information about the ship's construction [1]. The University of Geneva is leading since 2021 an international underwater excavation mission on the Antikythera shipwreck, delivering new and important information, and materials [4, 5, 6].

Justification of experimental time requested

We request 2 days of instrument time on the *SAXS-GISAXS* MRF, to be used as follows: up to 1 hour of measurements per ship nail (for a total of about 10 ship nails per day) for each of the three ships selected. The nails to be measured will be selected during the experiment, amongst the available ones, depending on the data being collected and to maximize the statistical significance of the systematic characterization.

References

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[3] Kaltsas N. et al, ed., *The Antikythera Shipwreck. The ship, the treasures, the mechanism,* Athens, National Archaeological Museum 2012.
[4] Simosi A., Baumer L., E., *L'épave d'Anticythère livre peu à peu ses secrets*, Archéologia, 614, novembre 2022, 56-63.
[5] Simosi A., Baumer L., *Anticythère 2021*, Antike Kunst 65, 2022, 155-157. 163. https://www.jstor.org/stable/27164586
[6] Simosi A., Baumer L., *Anticythère 2022*, Antike Kunst 66, 2023, 119-124 (in print).

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Experiment Proposal

Experiment number GP2023072

Principal investigator (*)	Dr Mauro Moglianetti, Italian Institute of Technology
Co-investigator	Dr Arianna Traviglia, Istituto Italiano di Tecnologia, ITALY
Co-investigator	Professor Federica Menegazzo, Ca&039; Foscari University Venice, ITALY
Co-investigator	Dr Maria Stratigaki, IIT, ITALY
Co-investigator	Mr Paolo Guzzonato, Università Ca&039; Foscari Venezia, ITALY
Co-investigator	Dr Erica Galvagno, Università Ca&039; Foscari di Venezia, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Cerium oxide nanoparticles: SAXS analyses for surface properties engineering
MRF Instrument	**SAXS GISAXS** **Days requested:** 2
Access Route	Direct Access **Previous GP Number:** no
Science Areas	Chemistry, Cultural Heritage **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	The catalytic properties of cerium oxide nanoparticles make them an ideal material to target some of the degradation issues affecting Cultural Heritage assets. Still, the characteristics of these nanoparticles need to be fully uncovered to achieve the necessary performances. Size and aggregation are key parameters to achieve this goal. SAXS measurements will guide us to develop synthetic protocols to achieve ultra-small and highly stable nanomaterials suitable for application.
Publications	-

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Mauro Moglianetti, Italian Institute of Technology
MRF Instrument	**SAXS GISAXS** **Days Requested:** 2
Special requirements:	

SAMPLE

Material	cerium oxide nanoparticles	-	-
Formula	CeO2	-	-
Forms	Liquid		
Volume	20 ml		
Weight	mg		
Container or substrate	standard cuvette	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	Sample Changer	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	no	-	-
Special equip. reqs	no	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	no	-	-
Equipment Hazards	-	-	-
Biological hazards	no	-	-
Radioactive Hazards	no	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Cerium oxide nanoparticles: SAXS analyses for surface properties engineering
Proposers: Erica Galvagno, Federica Menegazzo, Maria Stratigaki, Paolo Guzzonato, Mauro Moglianetti (co-main proposer), Arianna Traviglia (co-main proposer) - Beamlines requested: SAXS

Background and Context: The catalytic properties of cerium oxide nanoparticles (CeO_2 NPs) make them an ideal material to target some of the degradation issues affecting Cultural Heritage. However, the characteristics of these nanoparticles need to be fully uncovered in order to achieve the desired performances. Interestingly, CeO_2 NPs present optimal antioxidant activity thanks to the favorable reversible conversion between Ce(III) and Ce(IV). This property is linked to the stability of both these oxidation states and is clearly dependent on their relative ratio on the surface of the nanoparticles [1]. Therefore, the performances are intimately linked to the surface physico-chemical properties. To control these crucial characteristics, we need to fine-tune the size and morphology of the nanoparticles. Consequently, we need to decrease the size to obtain the highest surface area, aiming at achieving the best activity. For these reasons, we developed innovative synthetic protocols through which we are able to produce CeO_2 NPs with dimensions below 5.0 nm. Transmission Electron Microscopy (TEM) has been employed to characterize the synthetized nanoparticles, allowing to estimate their size and dimensional distribution. However, this technique becomes extremely complicated at ultra-small dimensions and does not provide information about the aggregation state and stability in solution, crucial parameters for future application. Moreover, commonly available Dynamic light scattering (DLS) instruments fail at dimensions below 10 nm. Therefore, Small Angle X-ray Scattering (SAXS) [2] is extremely necessary to provide information on size, morphology, and aggregation state at ultra-small scale, allowing to analyze the nanoparticles while they are in solution. SAXS needs to be associated with techniques such as X-ray photoelectron spectroscopy (XPS) to get a complete view of the catalytic properties of the NPs [3].

Proposed experiment: Considering that advanced techniques are fundamental to analyze the key properties of ultra-small CeO_2 NPs, due to the intrinsic limits of conventional techniques, we require to use SAXS instrumentation. Since the information obtained from the techniques used so far (TEM and DLS) has not been satisfactory we are convinced that the SAXS beamline is the only choice to provide information about the size distribution and aggregation state directly in the solution. We plan to analyze samples in solution, in different environmental conditions and synthetized with different reaction parameters, overcoming the step of sample preparation (required in TEM) that alters the aggregation state of NPs.

Summary of previous experimental proposals or characterization: We have recently developed a highly innovative method to achieve ultra-small CeO_2 NPs based on hydrothermal processes. The interplay between the catalyst and the concentration of cerium ions, together with the temperature allows to stop the growth of the nanomaterials, thus achieving sizes of few nanometers. We have already performed Transmission Electron Microscopy (TEM), the elective technique to characterize CeO_2 NPs. The results in Figure 1 show that we have achieved dimensions below 5.0 nm. However, TEM analysis does not provide information on the aggregation state in solution. On the other hand, we also used DLS. But it definitely fails at ultra-small dimensions. Furthermore, we have performed Flow field-flow fractionation, a sophisticated approach in which both separation and

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characterization of the sample can be carried out. Once again, the small size of the samples constituted a problem during the analysis, since the multi angle light scattering detector works best for dimensions above 10 nm.

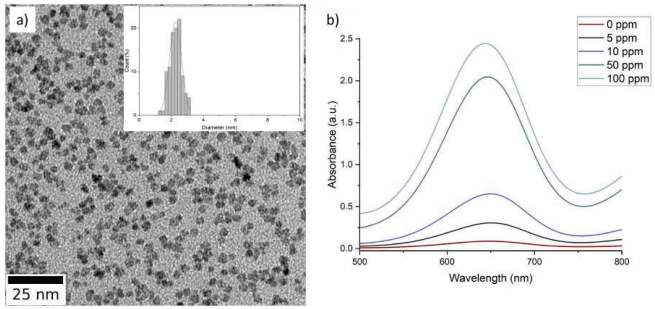

Figure 1. TEM image (left) and size distribution (inset) of CeO_2 NPs. Catalytic activity of the NPs in the oxidation of tetramethylbenzidine (right) at different concentrations (Uv-Vis spectrum).

We are in the progress of performing SPECS EnviroESCA (10^{th} -16^{th} Sept. at Prague Charles University) to study in detail the ratio between the two oxidation states [4]. Through UV-visible spectroscopy, we have already observed important differences in the activity between samples synthesized according to different protocols and samples aged in different environmental conditions (Figure 1, right).

Justification of experimental time requested: By using SAXS lab instrument at the unit CSGI/ University of Florence we will measure in total 200 samples divided as follows: 10 samples (synthesized as illustrated in Table 1) obtained from different reaction protocols, measured in 3 different concentrations at different pHs (5 values) to evaluate the behavior in aqueous environment: this sums up to 150 samples. Two of these samples will also be measured at different ageing times to study stability in different storing conditions (4 samples x 3 concentrations x 5 pHs= 60). We therefore ask for 2 days of beamtime, taking into account the time needed for the sample preparation and setup. We request the use of the robotic sample handlers and the use of the instrument during nighttime.

Table 1. Reaction conditions for the synthesis of CeO_2 NPs.

Protocol	Microwave								Conventional	
Temperature (°C)	Low				Medium		High		Low	
pH influence	1		2		1	2	1	2	1	2
Reaction time (min)	15	30	15	30	15	15	15	15	240	240

References: [1] Seal S., *Nanoscale*, 2020, **12**, 6879, DOI: 10.1039/d0nr01203c; [2] Moglianetti M., *Chem. Sci.*, 2014, **5**, 1232-1240, DOI: 10.1039/C3SC52595C; [3] Moglianetti M., *ACS Nano*, 2018, **12**, 8, 7731–7740, DOI: 10.1021/acsnano.8b01612; [4] Mehmood R., *Inorg. Chem.*, 2019, **58**, DOI: 10.1021/acs.inorgchem.9b00330.

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Experiment Proposal

Experiment number GP2023076

Principal investigator (*)	Dr Mauro Moglianetti, Italian Institute of Technology
Co-investigator	Dr Arianna Traviglia, Istituto Italiano di Tecnologia, ITALY
Co-investigator	Professor Federica Menegazzo, Ca&039; Foscari University Venice, ITALY
Co-investigator	Dr Maria Stratigaki, IIT, ITALY
Co-investigator	Mr Paolo Guzzonato, Università Ca&039; Foscari Venezia, ITALY
Co-investigator	Dr Erica Galvagno, Università Ca&039; Foscari di Venezia, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Cerium oxide nanoparticle's growth process: SAXS measurements during synthesis
MRF Instrument	**SAXS GISAXS** **Days requested:** 2
Access Route	Direct Access **Previous GP Number:** no
Science Areas	Chemistry, Cultural Heritage **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	The catalytic properties of cerium oxide nanoparticles make them an ideal material to target some of the degradation issues affecting Cultural Heritage assets. Still, the characteristics of these nanoparticles need to be fully uncovered to achieve the necessary performances. Size and aggregation are key parameters to achieve this goal. SAXS measurements performed during the nanoparticle's growth process will guide us to develop synthetic protocols to achieve ultra-small and highly stable nanomaterials suitable for application.
Publications	-

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Mauro Moglianetti, Italian Institute of Technology		
MRF Instrument	**SAXS GISAXS**	**Days Requested:** 2	
Special requirements:			
	SAMPLE		
Material	cerium oxide nanoparticles	-	-
Formula	CeO2	-	-
Forms	Liquid		
Volume	20 ml		
Weight	mg		
Container or substrate	standard cuvette	-	-
Storage Requirements	-	-	-
	SAMPLE ENVIROMENT		
Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	Sample Changer	-	-
Special equipment	-	-	-
	SAFETY		
Prep lab needed	Yes	-	-
Sample Prep Hazards	no	-	-
Special equip. reqs	no	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	no	-	-
Equipment Hazards	-	-	-
Biological hazards	no	-	-
Radioactive Hazards	no	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Cerium oxide nanoparticle's growth mechanism from SAXS analysis
Proposers: Erica Galvagno, Federica Menegazzo, Maria Stratigaki, Paolo Guzzonato, Mauro Moglianetti (co-main proposer), Arianna Traviglia (co-main proposer)
Beamlines requested: SAXS

Background and Context: The catalytic properties of cerium oxide nanoparticles (CeO_2 NPs) make them an ideal material to target some of the degradation issues affecting Cultural Heritage. However, the characteristics of these nanoparticles need to be fully uncovered in order to achieve the desired performances. Interestingly, CeO_2 NPs present optimal antioxidant activity thanks to the favorable reversible conversion between Ce(III) and Ce(IV). This property is linked to the stability of both these oxidation states and is clearly dependent on their relative ratio on the surface of the NPs [1]. Therefore, the performances are intimately linked to the surface physico-chemical properties and to control them, we need to fine-tune the size and morphology of the NPs. Consequently, we need to decrease the size to obtain the highest surface area, aiming at achieving the best activity. For these reasons, we are developing innovative synthetic protocols through which we are able to produce CeO_2 NPs with dimensions below 5.0 nm. Transmission Electron Microscopy (TEM) has been employed to characterize the synthetized nanoparticles, but this technique becomes extremely complicated at ultra-small dimensions and does not provide information about the growth mechanisms and the aggregation behavior in solution, crucial parameters for future applications. Moreover, commonly available Dynamic light scattering (DLS) instruments fail at dimensions below 10 nm. Therefore, Small Angle X-ray Scattering (SAXS) [2] is extremely necessary to provide information on the growth mechanism, through real time data on size, morphology, and aggregation state at ultra-small scale. The deep study on the growth mechanisms of NPs based on SAXS measurements, will clearly be associated to the characterization of the final products with the adequate techniques (e.g., X-ray photoelectron spectroscopy, X-ray diffraction).

Proposed experiment: Due to the intrinsic limits of conventional techniques, we require to use SAXS instrumentation to study the growth mechanism of ultra-small CeO_2 NPs. Since the data obtained from the techniques used so far (TEM and DLS) has solely allow us to get partial information on the final products, we are convinced that the clarification of the synthetic steps of nucleation and growth through the SAXS beamline is essential in the development of optimized protocols. Thanks to real-time measurements of size distribution (seeds stage, growth process, and final results) directly in the solution, we will be able to compare synthetic protocols with different reaction conditions and get a deeper insight in a mechanism that has yet to be fully explored. SAXS is fundamental to overcome the step of sample deposition on grids and the limit of nanoparticles poor statistics as in the case of TEM techniques.

Summary of previous experimental proposals or characterization: We have recently developed a highly innovative method to achieve ultra-small CeO_2 NPs based on hydrothermal processes. The interplay between the catalyst and the concentration of cerium ions, together with the temperature allows to stop the growth of the nanomaterials, thus achieving sizes of few nanometers. We have already performed TEM, the elective technique to characterize CeO_2 NPs. The results in Figure 1a show that we have achieved dimensions below 5.0 nm. However, TEM analysis does not provide information on the growth mechanism with statistical significance (very limited number of nanoparticles (200-300) that can be

analyzed). On the other hand, we also used DLS. But it fails at ultra-small dimensions. Furthermore, we have performed Flow field-flow fractionation, a sophisticated approach in which both separation and characterization of the sample can be carried out. Once again, the small size of the samples constituted a problem during the analysis, since the multi angle light scattering detector works best for dimensions above 10 nm.

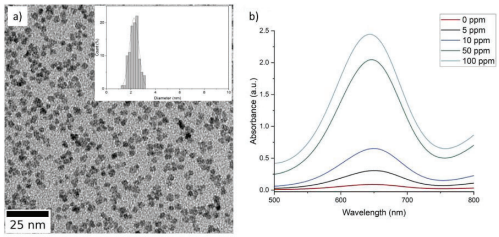

Figure 1. TEM image (left) and size distribution (inset) of CeO_2 NPs. Catalytic activity of the NPs in the oxidation of tetramethylbenzidine (right) at different concentrations (Uv-Vis spectrum).

We are in the progress of performing SPECS EnviroESCA (10^{th} -16^{th} Sept. at Prague Charles University) to study in detail the ratio between the two oxidation states [4]. Through UV-visible spectroscopy, we have already observed important differences in the activity between samples synthesized according to different protocols and samples aged in different environmental conditions (Figure 1b).

Justification of experimental time requested: By using SAXS lab instrument at the unit CSGI/ University of Florence we will monitor 11 different synthetic protocols as described in Table 1. The reactions require from 30 mins up to 4 hours (plus 1/2 hour for the setting up) for a total of 37 hours. Taking in account the possibility to monitor two reactions simultaneously, we estimate 20 hours of beamtime. We therefore ask for 2 days. We request the use of the robotic sample handlers, sample heater and the use of the instrument during nighttime for the longer reactions.

Table 1. Reaction conditions for the synthesis of CeO_2 NPs.

Temperature	Low		High								
Time (hours)	4.0		4.0					1.0		0.5	
pH influence	Base 1	Base 2	Base 1				Base 2	Base 1		Base 1	
Stabilizer amount	1	1	1	1	½	¼	1	1	1	1	1
Reaction stopping	RT	RT	RT	Ice bath	RT	RT	RT	RT	Ice bath	RT	Ice bath

References: [1] Seal S., *Nanoscale*, 2020, **12**, 6879, DOI: 10.1039/d0nr01203c; [2] Moglianetti M., *Chem. Sci.*, 2014, **5**, 1232-1240, DOI: 10.1039/C3SC52595C; [3] Moglianetti M., *ACS Nano*, 2018, **12**, 8, 7731–7740, DOI: 10.1021/acsnano.8b01612; [4] Mehmood R., *Inorg. Chem.*, 2019, **58**, DOI: 10.1021/acs.inorgchem.9b00330.

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Experiment Proposal

Experiment number GP2023078

Principal investigator (*)	Mr Alessandro Iberi, Ludovico Martelli S.p.A., ITALY
Co-investigator	Dr Marco Lombardi, Ludovico Martelli S.p.A, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Soap formulations: investigation of the relationship between structural properties and their stability and performance.
MRF Instrument	**SAXS GISAXS** — **Days requested:** 2
Access Route	Direct Access — **Previous GP Number:** -
Science Areas	Chemistry — **DOI:** -
Sponsored Grant	None — **Sponsor:** -
Grant Title	- — **Grant Number:** -
Start Date	- — **Finish Date:** -
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	The aim of the study is to characterize the lyotropic liquid crystal phases of soap- and surfactant- based cosmetic formulations, in order to 1) correlate the structure with chemical-physical properties, such as the rheological behaviour; 2) investigate their stability during manufacturing and shelf life; 3) find a relationship between structure and in-use performance. SAXS and WAXS analyses (Xenocs XEUSS 3.0 system) have been chosen to investigate the structure, while rheological analyses will be performed by Ludovico Martelli S.p.A. SAXS/WAXS and rheological analyses can also be performed in a wide temperature range (20-90°C) in order to determine structure variations during the manufacturing stages of the product (for example during and after the saponification process). We therefore request a total of 2 days for the instrument Xenocs XEUSS 3.0.
Publications	-

ISIS neutron and muon source — **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact Mr Alessandro Iberi, Ludovico Martelli S.p.A., ITALY
MRF Instrument **SAXS GISAXS** **Days Requested:** 2
Special requirements:

SAMPLE

Material	Soap-based formulation	Soap-based formulation	Surfactant-based formulation
Formula	Triethanolamine salts of fatty acids	Sodium and/or potassium salts of fatty acids	Aminoacids-derived surfactants
Forms	Liquid	Solid	Liquid
Volume	cc	cc	cc
Weight	mg	mg	mg
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	- K	- K
Pressure Range	- mbar	- mbar	- mbar
Magnetic field range	- T	- T	- T
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	Yes	Yes
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	No	No
Sensitivity to vapour	No	No	No
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	Disposed by IS

Soap formulations: investigation of the relationship between structural properties and their stability and performance.

1. Background and Context

Soaps and synthetic surfactants have historically been used in a wide variety of cosmetic formulations, such as cleansing products, shaving preparations, and emulsions for skin care.
According to the stage of manufacturing and the composition of the product, different liquid crystal phases (e.g. lamellar or hexagonal) can be identified in the structure of many soap- and surfactant-based formulations[1-2].
Some chemical-physical properties of these formulations, for example the rheological behaviour during manufacturing and shelf life, are deeply influenced by phase structure[3]. An accurate phase characterisation of soap- and surfactant-based formulations can therefore help improve the production processes and sensory characteristics of cosmetic products.

2. Proposed experiment

The aim of the study is to characterize the lyotropic liquid crystal phases of soap- and surfactant-based cosmetic formulations, to:

- correlate the structure with chemical-physical properties, such as the rheological behaviour,
- investigate their stability during manufacturing and shelf life,
- find a relationship between structure and in-use performance.

Small and wide-angle X-ray scattering (SAXS and WAXS) analyses are usually chosen to investigate the structure of soap- and surfactant-based formulations, as they can provide unique information about phase characterisation and interaction between the liquid crystal layers.
Results of SAXS and WAXS characterisation will be merged with the rheological analyses of the same samples, in particular:

- flow curve and/or
- amplitude sweep and/or
- frequency sweep

to put into relationship the structure with rheology and with stability prediction inferred from rheological analyses.
Rheological analyses will be performed by Ludovico Martelli S.p.A. using an Anton Paar MCR102e Rheometer.
SAXS/WAXS and rheological analyses can also be performed in a wide temperature range (20-90°C) to determine structure variations during the manufacturing stages of the product (for example during and after the saponification process).

3. Summary of previous experimental proposals or characterisation

According to preliminary experiments, SAXS profiles can provide accurate phase characterisation of our cosmetic formulations. As shown by the following figure, sample is characterised by many diffraction orders, which can be traced back to at least 2 lamellar phases.

1 H. Sagitani, Stability Conditions and Mechanism of Cream Soaps: Role of Glycerol, *J. Oleo Sci.* 63, (4) 365-372 (2014).
2 H. Ren et al., Preparation and Characterization of Alpha Gel Formed by Fatty Alcohol and Amino Acid Surfactants, *J Surfact Deterg* (2021).
3 K. Ichihara et al., Rheology of α-Gel Formed by Amino Acid-Based Surfactant with Long-Chain Alcohol: Effects of Inorganic Salt Concentration, *Langmuir 2021*, 37, 7032−7038.

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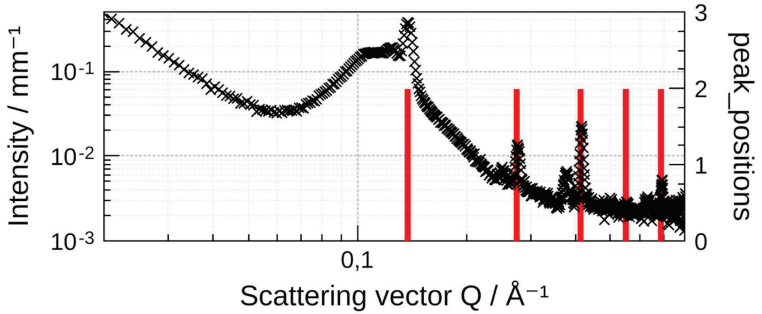

4. Justification of experimental time requested

We propose the following analyses:

- SAXS: 0.7 days for 14 samples, to be analysed at room temperature. The acquisition will be done in high resolution mode with atmocap to minimize sample evaporation, using a Genix generator with microfocus copper tube operated at a maximum power of 30 W. The detector sample distance will be set to 0.3 m, so to have a scattering vector range of 0.01-0.9 1/A. The acquisition time will be ~20 minutes.
- SAXS performed in the temperature range 20-90°C: 1 days for 7 samples.
- WAXS: 0.3 days for 7 samples.

We therefore request a total of 2 days for the instrument Xenocs XEUSS 3.0.

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Experiment Proposal

Experiment number GP2023083

Principal investigator (*) Dr Pasquale Sacco, Università degli Studi di Trieste, ITALY
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator
Co-investigator

Experiment title	Investigation of the architecture of agarose-based hydrogels prepared by controlled rate of cooling - AGAROCOOL		
MRF Instrument	**SAXS GISAXS**	**Days requested:**	2
Access Route	Direct Access	**Previous GP Number:**	No
Science Areas	Biology and Bio-materials, Chemistry, Materials	**DOI:**	-
Sponsored Grant	None	**Sponsor:**	-
Grant Title	-	**Grant Number:**	-
Start Date	-	**Finish Date:**	-
Similar Submission?	-		
Industrial Links	-		

Non-Technical Abstract

Extracellular matrices (ECMs) and, more broadly, living tissues can be described as complex biopolymer-based networks endowed with particular physical-mechanical properties. The cells that make up the tissues can sense this biophysical milieu and convert the resulting external physical information into intracellular biochemical signals through a process called 'mechanotransduction'. Our research group is interested in recapitulating this physical information in ECM mimetics in the form of hydrogels and using them as a platform to study important mechanotransduction processes in cells. Previous results have shown a direct correlation between the chemical composition of the assembled biopolymers and the mechanical response of the hydrogels. The aim of AGAROCOOL is to investigate the role that the methylation pattern of agarose samples plays in the formation of three-dimensional hydrogels. The architecture of these biomaterials will be investigated by Ultra-/SAXS and possibly by cryo-EM.

Publications

None
None
None

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Pasquale Sacco, Università degli Studi di Trieste, ITALY		
MRF Instrument	**SAXS GISAXS**	**Days Requested:**	2
Special requirements:			

SAMPLE

Material	The hydrogels will be assembled using three agaroses with different chemical composition (methylation degree)	-	-
Formula	Biopolymers formed by alternating D-galactose and 3,6-anhydro-L-galactopyranose linked by α-(1→3) and β-(1→4) glycosidic bonds	-	-
Forms	Friable powder		
Volume	cc		
Weight	500 mg		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	Water Bath	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	None	-	-
Special equip. reqs	To dissolve agaroses in deionized water we need autoclave or microwave	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	None	-	-
Equipment Hazards	-	-	-
Biological hazards	None	-	-
Radioactive Hazards	None	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Removed By User	-	-

1. Background and Context

Extracellular matrices (ECMs) and, more broadly, living tissues can be described as complex biopolymer-based networks endowed with particular physical/mechanical properties. The cells that make up the tissues can sense this biophysical milieu and convert the resulting external physical information into intracellular biochemical signals through a process called mechanotransduction.[1] ECM mimics in the form of hydrogels are urgently needed to recapitulate the correct ECM composition and mechanics and use them to understand various biochemical aspects in both cell biology and pathology. Therefore, understanding the structural and mechanical properties of these systems is crucial for the intended need. Recently, we have focused our attention on the biopolymer agarose, a linear polysaccharide derived from red algae. It is a thermoresponsive biopolymer that dissolves completely in water when heated and forms a wall-to-wall hydrogel when cooled to room temperature. While it is generally assumed that gelation occurs by liquid–liquid phase separation of agarose on cooling,[2–4] the two main proposed mechanisms for hydrogel formation are spinodal decomposition or nucleation and growth. We have recently shown that controlled cooling of agarose is critical for the surface-to-core distribution of the biopolymer in the hydrogel network and this affects surface nanomechanical properties, bulk mechanical behaviour and response of cells.[5]

2. Proposed experiment

Considering our previous findings, the possibility of gaining access to the instrumentation of MRFs would be a great opportunity to expand our knowledge of the relationship between hydrogel architecture and related behaviour. Agaroses with different chemical compositions in terms of methylation content[6] are used to prepare hydrogels using a well-established experimental protocol.[5] Ultra-/SAXS may be the best technique for this type of analysis as it is a powerful tool for revealing the structure of biomaterials and allows samples to be studied in their wet state without the need for special sample preparation procedures. Another advanced and useful technique is cryogenic electron microscopy (cryo-EM), which would allow resolution of the hydrated hydrogels at the nano-scale.

3. Summary of previous experimental proposals or characterisation

We have already thoroughly investigated the effects of the rate of cooling upon heating (quenching) of a agarose sample on the mechanical properties of the resulting hydrogels.[5] The experimental protocol consists of dissolving 1% w/V agarose by autoclaving (*i.e.* 121°C) and then controlling cooling-steps (85, 60 and 42 °C) before gelling at room temperature (Figure 1). The bulk properties of the hydrogels were characterised by uniaxial compression and rheological tests, which showed that these networks relax the stress rapidly and exhibit similar stiffness, but have a linear elasticity that increases with decreasing cooling rate. The greatest effect of quenching is observed when the hydrogel surface, *i.e.* the hydrogel-air interface, is considered. Atomic force microscopy and nanoindenter analyses were undertaken to investigate the surface stiffness and topology. The results show that the lower the curing temperature of the agarose solutions, the lower the hydrogel surface stiffness. A combination of environmental SEM and confocal microscopy imaging has evidenced a clear agarose film on the surface of the hydrogel that gradually disappears on the cooling rate.

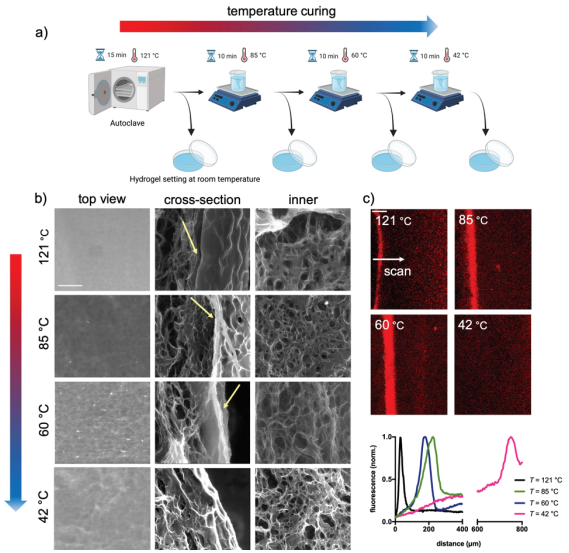

Figure 1. Temperature curing of agarose solution defines hydrogels that have different networks. (a) Cartoon recapitulating the experimental setup used in this study, which involves controlled cooling steps before the hydrogel forms at room temperature. (b) Scanning electron micrographs in environmental state of agarose hydrogels obtained at different curing temperatures: top view (left), cross-section (middle) and inner part of the hydrogel (right); the yellow arrows indicate the agarose abundance; the scale bar is 100 µm. (c) Confocal scanning electron microscopy of hydrogels prepared at different curing temperatures. The hydrogels were prepared with Atto Rho101 NHS ester-labelled agarose and sectioned to visualize the internal structure. The scan starts from the surface boundary and proceeds toward the inner part of the hydrogel as indicated in the images; the scale bar is 100 µm. The agarose profile (normalized fluorescence intensity) is shown in the plot.

4. Justification of experimental time requested

Ultra-/SAXS, and possibly cryogenic electron microscopy, are advanced tools that would allow us to better understand the relationship between hydrogel architecture and its response to mechanical stimulation, which is a key aspect in the field of mechanotransduction. In particular, Ultra-/SAXS allows for the determination of the characteristic mesh and inhomogenity sizes proper of a hydrogel architecture. We have three agarose samples with well-identified chemical compositions, which we have previously studied by NMR analyses.[6] These agaroses are used for the preparation of hydrogels, using the temperature-assisted gelation protocol described above. We assume that a total of 9 samples need to be analysed. The estimated time for the entire set-up and analysis considering the Ultra-/SAXS measurements on a Xeuss 3HR so to cover the dimensional range between few nm and few microns would be 2 days in total.

References

[1] *Nature,* **2020**, *584*, 535.
[2] *Macromolecules,* **1974**, *7*, 527.
[3] *Phys. Rev. E,* **1999**, *59*, 2222.
[4] *Polymer (Guildf),* **2002**, *43*, 5299.
[5] *Advanced Healthcare Materials,* **2023**, 2300973.
[6] *Advanced Functional Materials,* **2023**, *Accepted.*

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Experiment Proposal

Experiment number GP2023084

Principal investigator (*)	Dr Claudio Resta, Enapter SRL, ITALY
Co-investigator	Dr Gabriele Agonigi, enapter s.r.l., ITALY
Co-investigator	Dr Massimo Rosa, Enapter s.r.l., ITALY
Co-investigator	Dr Antonio Filpi, Enapter srl, ITALY
Co-investigator	Mr Stefano Catanorchi, Enapter S.r.L., ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Training for SAXS on Membrane-Electrode assembly components
Training MRF	**SAXS GISAXS** **Days requested:** 2
Access Route	Direct Access **Previous GP Number:** 2023033
Science Areas	Chemistry, Energy, Environment **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	Enapter s.r.l.
Non-Technical Abstract	Enapter produces scalable and modular AEM electrolysers, a relatively new technology, to produce hydrogen and oxygen from water splitting electrochemical reaction. Key components are MEA (Membrane Electrode Assemblies) and PTL (Porous Transport Layer). AEM technology combines advantages of both classical alkaline and PEM water electrolysis, to produce high purity hydrogen at relatively high pressure and high current density without using expensive or scarce materials (e.g. Tit, Ir, Pl). Our research programmes would relevantly benefit by using powerful characterization techniques . Potentialities of those techniques have only been barely explored in companies' framework and may constitute a breakthrough on the analysis of the MEA components in AEM systems. Specifically, the possibility to receive a training would, in our opinion, constitute a remarkable asset and grant a more proficient and effective investigation and technology development.
Publications	-

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Sample record sheet

Principal contact	Dr Claudio Resta, Enapter SRL, ITALY		
Training Instrument	**SAXS GISAXS**	**Days Requested:** 2	
Special requirements:			

SAMPLE

Material	-	-	-
Formula	-	-	-
Forms			
Volume			
Weight			
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	-	-	-
Pressure Range	-	-	-
Magnetic field range	-	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	-	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	-	-	-
Sensitivity to vapour	-	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	-	-	-

Case for ISIS@MACH ITALIA Training Proposal

Training on SAXS for Membrane-electrode assembly components analysis

1. Background and Context

Enapter produces scalable and modular AEM electrolysers to produce hydrogen and oxygen from water splitting electrochemical reaction. Key components to allow efficient and durable performances are MEA (Membrane Electrode Assemblies) and PTL (Porous Transport Layer). AEM technology combines advantages of both classical alkaline and PEM water electrolysis, allowing to produce high purity hydrogen at relatively high pressure and high current density without using expensive or scarce materials (e.g. Titanium, Iridium, Platinum). Being the AEM technology relatively new, every single constituent of the final product needs to be extensively characterized to provide a deeper knowledge and speed up technological improvements. (e.g. connection between morphology and physical-chemical properties). Due to the novelty of the technology, very few advanced characterization techniques are routinely used in the field. Preliminary data showed that our research programmes could relevantly benefit by having access and gaining expertise on some powerful characterization techniques (like SAXS); potentialities of those techniques have only been barely explored in companies' framework and may constitute a breakthrough on the analysis of the MEA components in AEM systems. Additionally, the possibility to receive a specific training would, in our opinion, constitute a remarkable asset and grant a more proficient and effective investigation and technology development. Our main financial support comes from the holder Enapter AG, additionally Enapter earned a grant from PNRR programme from italian government and it is involved in an Horizon 2020 project ("CHANNEL").

2. Proposed training

This training proposal would allow 5 members of the R&D chemistry department of Enapter srl to gain expertise in SAXS technique. All the selected members are chemists with relevant expertise in the field of AEM electrolysis and material characterization.
In order to work properly and efficiently, a very delicate equilibrium between the physical and chemical properties of MEA components and their relationship is required. In this regard, a deeper understanding of morphological and structural features of each MEA component is crucial for their optimization and improvement. Specifically, SAXS and reflectometry/GISAXS experiments could provide us with information on the nanoscale morphology of each MEA components (e.g. the presence and organisation at the nanoscale of different inorganic phases in the electrodes surface, as well as the structure and organisation of hydrophilic/hydrophobic moieties within the membrane) that can be extremely helpful for their engineering and optimization. Some of the components of our devices already proved to be suitable to be investigated through SAXS techniques. However, in order to obtain reliable data and, even more importantly, a reasonable interpretation of them, a knowledge of these technique more profound than the one we have in our company is required. This training would help us in the development and design of the proper experiments as well as it would guide us in the data analysis and modelling.
The training would be carried out at CSGI – "Consorzio interuniversitario per lo Sviluppo dei sistemi a Grande Interfase" – Università degli Studi di Firenze, with whom we are already in contact and agreed on

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3. Summary of previous training proposals

No previous training proposal has been presented. However, membranes and electrodes have been extensively analysed by Enapter in terms of their functional performances, and in a first round of measures, SAXS has been proved to be suitable for the investigation of the morphological feature of MEA components at the nanoscale.

4. Justification of training proposals request

The Xenocs SAXS available at ISIS@MACH Italia is a unique instrument in Italy, in terms of its flexibility to perform SAXS/USAXS/GISAXS analysis on solid samples/surfaces, without the need to apply vacuum at the sample stage, when this is undesirable in terms of sample stability or evaporation. Based on the previous experiments (GP2023033) conducted on the same instrument, we think that a further investigation through this facility would provide us with very precious information on all the components of the MEA package. Moreover, being trained to it would allow us for a more detailed understanding of its features and dramatically improve our capability of data analysis and modelling. After discussing with ISIS@MACH Italia staff, we are requesting 2 days of training.

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Experiment Proposal

Experiment number GP2023087

Principal investigator (*)	Dr Francesco Brasili, National Research Council, ITALY
Co-investigator	Dr Emanuela Zaccarelli, CNR, ITALY
Co-investigator	Professor Marco Laurati, CSGI, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Effective interactions and phase behavior of PNIPAM-PNIPMAM copolymer microgels
MRF Instrument	**SAXS GISAXS** **Days requested:** 3
Access Route	Direct Access **Previous GP Number:** no
Science Areas	Materials, Physics **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

Thermoresponsive microgels are of great interest both as model systems in soft matter physics and as key components in diverse applications. Their main feature, the volume phase transition (VPT), consists in the collapse of the polymer network at high temperature, due to the lower solvent affinity. We are focused on tuning the VPT temperature (VPTT) and in setting it close to physiological temperature, to enable promising biomedical applications. To this aim, we study composite microgels obtained by the copolymerization of N-isopropylacrylamide (NIPAM) and NIPMAM (N-isopropylmethacrylamide), combining experiments and simulations. Moving from previous SANS results, that revealed the formation of segregated poly-NIPAM domains when close to the VPTT, in this proposal we study the effect of this heterogeneous deswelling on the interparticle interactions. Hence, we will perform experiments at varying temperature and microgel concentration to determine the state diagram of the dispersion.

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Francesco Brasili, National Research Council, ITALY		
MRF Instrument	**SAXS GISAXS**	**Days Requested:** 3	
Special requirements:			

SAMPLE

Material	Poly-N-isopropylacrylamide-co-n-isopropylmetacrylamide microgels in water	-	-
Formula	H2C=CHCONHCH(CH3)2 ; H2C=C(CH3)CONHCH(CH3)2	-	-
Forms	Liquid		
Volume	10 cc		
Weight	10 g		
Container or substrate	plastic tubes, capillars	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	293 - 323 K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	no	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	no	-	-
Equipment Hazards	-	-	-
Biological hazards	no	-	-
Radioactive Hazards	no	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Removed By User	-	-

1. Background and Context

Microgels are stimuli-responsive particles with many uses, including filtration processes, drug delivery, sensors and 3D printing. They are also a model system for the investigation of fundamental questions in condensed matter physics, such as glass transitions and jamming. Microgels made of poly-N-isopropylacrylamide (PNIPAM) present a volume phase transition (VPT) that originates from the decrease of solvent affinity of the polymer upon increasing temperature T and gives rise to a responsive collapse of the microgels at high T, which is particularly suitable for applications. Scattering and microscopy experiments have shown that this transition is intimately linked to the evolution of the internal structure of the particles. Recently some of us introduced a new realistic coarse-grained simulation model of microgels [1], that reproduces the internal structure and the swelling behavior of experiments. The use of composite microgels expands the possibilities to tuning their properties, including the volume phase transition temperature (VPTT), thereby also enlarging the range of possible applications. However, the effect of copolymerization on microgel responsiveness is highly non-trivial. We are combining experiments and the new simulation method mentioned before to investigate microgels with double thermoresponsivity (we recently applied a similar approach on PNIPAM-co-PEG (poly-ethylene-glycol) microgels [2]). As a first step towards systems with a VPTT close to physiological temperature, we started studying PNIPAM-co-PNIPMAM (poly-N-isopropylmethacrylamide) microgels. While PNIPAM has a VPTT at ~32 ºC, PNIPMAM collapses above ~42 ºC. We recently investigated by SANS the internal structure of single composite microgels, founding evidence of heterogeneous deswelling with the formation of segregated domains of PNIPAM when approaching the VPTT. The domain size is affected by the relative PNIPAM:PNIPMAM composition. Due to the hydrophobicity of PNIPAM in the collapsed state, we expect that these domains significantly modify the interparticle interactions across the VPTT and therefore the state diagram of microgel dispersions as a function of T and concentration. Establishing a link between internal microstructure and phase behavior is fundamental for tuning the system for applications, such as injectable scaffolds for tissue engineering and auxetic materials.

2. Summary of previous results

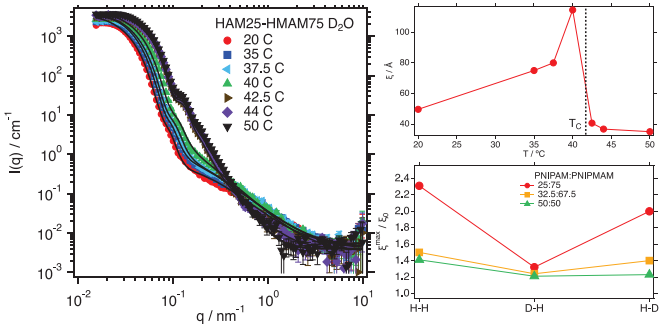

Figure 1: Left: Experimental scattering intensities for sample with composition 25:75 and different temperatures. Lines are fits to the model described in the text. Right: polymer domain correlation length as a function of T (top), and its maximum variation for different compositions and partial deuterations (bottom).

Fig.1 (left) shows exemplary form factors (FF) measured for a fully protonated PNIPAM-PNIPMAM (HAM-HMAM) microgel with composition 25:75 in D_2O at different T. The FF evidences the transition from a swollen to compact shape when crossing the VPTT (T_C = 41.7 °C). Fits using a fuzzy sphere + domain scattering model were used to extract a domain correlation length ξ that shows a non-monotonic trend as a function of T (Fig.1, right), with a maximum that indicates presence of larger heterogeneities at T_C before collapse. Note that this is very different from pure PNIPAM microgels, in which ξ decreases monotonically with increasing T. A plot of the relative variation between the maximum value of ξ, ξ^{max}, and its value in the swollen state, ξ_0, shows that the domain size variation decreases with increasing PNIPAM content. Moreover, values for the microgel containing partially deuterated PNIPAM are significantly smaller than those of the other two microgels, indicating that the observed variation of ξ is mainly due to PNIPAM domains.

3. Proposed experiment

We plan to investigate how the formation of domains across the VPTT for the microgel with PNIPAM:PNIPMAM composition 25:75 affects interactions and thus structural arrangements as a function of packing fraction. In detail, we plan to measure fully protonated microgel dispersions with 8 different effective packing fractions ϕ_{eff} = 0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.8, 1.0, for 7 different T = 20, 35, 37.5, 40, 42.5, 44, 50 °C across the VPT. The experimental scattering curves will be analyzed using analytical form factor models [3], as well as simulated form factors [1,2], while structure factors will be obtained in all cases from the effective potentials extracted from simulations [2,4].

4. Justification of experimental time requested

Experiments will be performed on the Xeuss 3.0 SAXS of CSGI Florence using a High Resolution collimation setup. Using two Sample-Detector distances equal to 30 and 180 cm we will get access to a q-range 0.04nm^{-1} < q < 7nm^{-1}. Since the particle hydrodynamic radius is R_H = 90nm, this configuration will allow us to reach sufficiently low q-values to measure the structure factor peak even at the smallest effective packing fraction and lowest T. Samples will be loaded in capillaries and measured in air using the Peltier capillary stage of the instrument to control T. Even at the highest ϕ_{eff} investigated, the sample viscosity is sufficiently low to allow loading in capillaries. Provided the relatively low contrast between particles and water background, in particularly at low T, we estimate an average measurement time of 1hr for each ϕ_{eff} and T, corresponding to a total time of 1hr × 8 samples × 7 T = 56 hrs. In addition, water will be measured at the same temperatures, corresponding to additional 8 hrs. According to these calculations and considering equilibration times for the different temperatures, we ask for 3 days of beamtime.

References

[1] Gnan et al., Macromolecules 50, 8777 (2017)
[2] Rivas-Barbosa et al., Macromolecules 55, 1834 (2022)
[3] M. Keerl, J. S. Pedersen, W. Richtering. JACS: 131, 8, 3093-3097 (2009)
[4] Ruiz-Franco et al. Soft Matter, 19: 3614 (2023).

SAXS WAXD *SAXS WAXD*

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Experiment Proposal

Experiment number GP2023058

Principal investigator	Professor Anita Grozdanov, Skopje University, MACEDONIA
Co-investigator (*)	Dr Marino Lavorgna , CNR, ITALY
Co-investigator	Dr Gennaro Gentile, IPCB CNR, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	SAXS WAXD structural analysis of polyninylalcohol/polyacrylic acid/MXenes nanocomposites
MRF Instrument	**SAXS WAXD** **Days requested:** 3
Access Route	Direct Access **Previous GP Number:** No
Science Areas	Engineering, Materials **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	The proposal aims to perform a structural characterization of polyninylalcohol/polyacrylic acid/MXenes nanocomposites by small and wide angle X-ray diffraction, using the SAXS WAXD. In distinct proposals, we aim to perform both a morphological analysis using the scanning electron microscope SEM FEI and the analysis of the filler spatial distribution in the polymer matrix by the transmission electron microscope TEM FEI, all operating at the IPCB-CNR Unit. This proposal is specifically addressed to get new insights in the exfoliation degree and the spatial distribution of 2D Mxenes nanofillers in highly amorphous polyninylalcohol (HAVOH)/polyacrylic acid (PAA) blends and to correlate the preparation approaches to the structure and morphology and to the final properties of the materials, with particular attention on their electrical conductivity, their EMI shielding and their gas barrier properties.
Publications	-

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact Dr Marino Lavorgna , CNR, ITALY
MRF Instrument **SAXS WAXD** **Days Requested:** 3
Special requirements:

SAMPLE

Material	HAVOH neat (1 sample)	HAVOH + PAA (3 samples)	HAVOH + PAA + MXENES (2 samples)
Formula	polyvinylalcohol	polyvinylalcohol + polyacrylic acid	polyvinylalcohol + polyacrylic acid + mxenes
Forms	Solid	Solid	Solid
Volume	0.1 cc	0.1 cc	0.1 cc
Weight	100 mg	100 mg	100 mg
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	300 - 300 K	300 - 300 K	300 - 300 K
Pressure Range	- MPa	- mbar	- mbar
Magnetic field range	- T	- T	- T
Standard equipment	None	None	None
Special equipment	N/A	N/A	N/A

SAFETY

Prep lab needed	No	No	No
Sample Prep Hazards	no	no	no
Special equip. reqs	N/A	no	N/A
Sensitivity to air	Yes	Yes	Yes
Sensitivity to vapour	Yes	Yes	No
Experiment Hazards	no	no	no
Equipment Hazards	-	-	-
Biological hazards	no	no	no
Radioactive Hazards	no	no	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	Disposed by IS

SAXS WAXD structural analysis of polyninylalcohol/polyacrylic acid/MXenes nanocomposites

1. Background and Context

Polymer composites with nanoparticles as fillers are a growing group of materials with interesting properties for variety of application. Although numerous composites with nanofillers have been prepared and studied in last decade, mainly with carbon based fillers as carbon nanotubes or graphene, there are still challenges when new type of nanoparticles are discovered or synthetized.
MXenes are new types of 2D materials described first in the paper of Barsoum et al. in 2011 [1]. General formula for MXenes is Mn+1XnTx (n = 1–3), where M represents transition metals (Sc, Ti, Zr, Hf, V, Nb, Ta, Cr, Mo, etc.), X is carbon and/or nitrogen and Tx refer to different functional groups on the surface (e.g. OH, O, F, etc). Moreover, MXenes particles are highly electrically conductive. Shahzad et al. [2] have shown that flexible Ti3C2Tx films exhibit excellent electrical conductivity and electromagnetic interference (EMI) shielding capacity. Electrical conductivity reached 4600 S/cm, what originates from the high electron density of states near the Fermi level. In addition, due to their 2D morphology, MXenes are very promising to impart high gas barrier properties to polymer nanocomposites.
With the objective of preparing new nanocomposites with high gas barrier properties, high electrical conductivity and electromagnetic interference (EMI) shielding properties, in this activity new polymer blends filled with MXenes have been realized at variable composition. As a polymer matrix, an easy water soluble polvinylalcohol, high amorphous polyvinylalcohol (HAVOH) has been used [3], blended with polyacrylic acid (PAA) at variable molecular weight. Indeed, after thermal treatments, HAVOH/PAA blends are prone to give light crosslinking, with improvement of their stability to high relative humidity environments. HAVOH/PAA blends have been additivated with MXenes, in particular Ti3C2, prepared by etching the aluminium from the MAX phase Ti3AlC2.

2. Proposed experiment

The HAVOH/PAA nanocomposites at variable PAA molecular weight and HAVOH/PAA weight ratio, and containing 5 phr of MXenes have been realized by Skopje University - Faculty of Technology and Metallurgy, in cooperation with IPCB-CNR. In particular, HAVOH/PAA blends in water solutions have been prepared and additivated with the MXenes. Then films (about 50 micrometer thick) have been prepared by water casting. On the obtained films thermal treatments have been performed in oven to promote crosslinking between the HAVOH and the PAA phase. The following samples have been prepared for their characterization by SAXD/WAXD and, in distinct proposals with SEM FEI and TEM FEI: 1) HAVOH neat; 2) HAVOH/PAA_4k 70/30; 3) HAVOH/PAA_4k 50/50; 4) HAVOH/PAA_240k 50/50; 5) HAVOH/PAA_4k 50/50 + 5phr Mxenes; 6) HAVOH/PAA_240k 50/50 + 5phr Mxenes.
The following characterization will be performed on these samples to evaluate the effect of the composition (HAVOH/PAA ratio, MW of PAA, MXenes additivation) on the structure of the composites:

- Small and Wide-Angle X-ray Diffractometer (SAXS/WAXD) to obtain info about orientation of 2D fillers and crystallinity degree of polymeric phase. It is proposed to measure n. 6 samples (1 HAVOH neat, 3 on HAVOH/PAA blends, 2 nanocomposites filled with 5 phr Mxenes) by modulating the acquisition time to optimize the spectra and highlight the presence of the fillers, by scanning the accessible q range from 0.06nm-1 to 40.7 nm-1. Hence, we request 3 days of beamtime which account also for setup time, and eventual beam loss time.

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In distinct proposals the same samples will be analyzed by Scanning and Transmission Electron Microscopy (SEM FEI and TEM FEI, respectively), available at the IPCB CNR Unit.

3. Summary of previous experimental proposals or characterisation

No previous experiments have been carried out on these samples

4. Justification of experimental time requested

We have requested the SAXS WAXD equipment to evaluate the effect of the composition (HAVOH/PAA ratio, PAA molecular weight, MXenes additivation) on the structure of the composites.

We request 3 days of SAXS WAXD beam time, necessary for the structural characterization of the 6 above-described materials, after discussion with the instrument scientist. The foreseen beam time accounts set up and for the data collection on the samples.

References

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[3] C. Santillo, A.P. God, R.K.Donato, R.J.Espanhol Andrade, G.G. Buonocore, H. Xia, M. Lavorgna, A. Sorrentino. Tuning the structural and functional properties of HAVOH-based composites via ionic liquid tailoring of MWCNTs distribution. Composites Science and Technology, 207, 2021, 108742.

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Experiment Proposal

Experiment number GP2023060

Principal investigator	Professor Vladimir Sedlarik, Tomas Bata University in Zlin, CZECH_REPUBLIC
Co-investigator (*)	Dr Marino Lavorgna , CNR, ITALY
Co-investigator	Dr Gennaro Gentile, IPCB CNR, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Innovative sustainable inks for wearable sensors: structural characterization by SAXS/WAXD
MRF Instrument	**SAXS WAXD** **Days requested:** 3
Access Route	Direct Access **Previous GP Number:** no
Science Areas	Engineering, Materials **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	The proposal is aimed at characterizing new sustainable inks based on polyurethane, modified with several fillers (1D and 2D and hybrid systems) and applied by conventional deposition techniques on selected textiles for the realization wearable sensors. The challenge is to have control of the deposition procedure to increase the filler-filler contacts and enhance the electron conductivity, at lower filler content, by maximizing the coating durability in washing cycles. Tomas Bata University needs to further improve its understanding of the developed systems by investigating the spatial filler distribution and correlating results to the coating processing conditions. This proposal is addressed to perform the structural characterization by SAXS/WAXD of carbonaceous filler-based sustainable inks. In distinct proposals the morphologycal characterization by SEM FEI of the samples and the morphology of the fillers by TEM FEI is requested. All equipments are available at the IPCB CNR Unit.
Publications	-

ISIS neutron and muon source	**E-platform:** No
Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Marino Lavorgna , CNR, ITALY
MRF Instrument	**SAXS WAXD** **Days Requested:** 3
Special requirements:	

SAMPLE

Material	cotton fabrics treated with PU + 1D fillers (MWCNTs) by different deposition technologies (i.e. rod coaters, dip coating, spray coating) (3 samples)	cotton fabrics treated with PU + 2D fillers (graphene) by different deposition technologies (i.e. rod coaters, dip coating, spray coating) (3 samples)	cotton fabrics treated with PU + 1D fillers (MWCNTs) + 2D fillers (graphene) by different deposition technologies (i.e. rod coaters, dip coating, spray coating) (3 samples)
Formula	cotton, polyurethane (PU), MWCNTs	cotton, polyurethane (PU), graphene	cotton, polyurethane (PU), MWCNTs, graphene
Forms	Solid	Solid	Solid
Volume	0.500 cc	0.500 cc	0.5 cc
Weight	500 mg	500 mg	500 mg
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	300 - K	300 - K	300 - K
Pressure Range	- mbar	- mbar	- mbar
Magnetic field range	- T	- T	- T
Standard equipment	None	None	None
Special equipment	N/A	N/A	N/A

SAFETY

Prep lab needed	Yes	Yes	No
Sample Prep Hazards	no	-	no
Special equip. reqs	-	no	no
Sensitivity to air	No	No	No
Sensitivity to vapour	No	No	No
Experiment Hazards	no	no	no
Equipment Hazards	-	-	-
Biological hazards	no	no	no
Radioactive Hazards	no	no	no
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	Disposed by IS

Innovative sustainable inks for wearable sensors: structural characterization by SAXS/WAXD

1. Background and Context

The rapid development of IoT and smart wearable devices has contributed to the enormous demand for smart flexible strain sensors. Unfortunately, the realization of smart textiles isn't always sustainable. [1] Thus, technological interests are growing in developing green composite materials as inks for conductive connections and piezo resistors by embedding nano carbons, such as 1D nanotubes, or 2D platelets. [2, 3] The advantages of polymeric nanocomposites with carbonaceous fillers are the low cost, lightweightness, and ease of dispersibility in environmentally friendly solvents. Another benefit of 1D and 2D nano carbons is their high aspect ratio, which ensures an efficient electrical percolation network at low loadings. These non-metal inks do not require a post-coating sintering step, which can reach damaging temperatures for common flexible polymer substrates such as cotton and cellulose. They create a stable conductive ink with time and, in some instances, are biocompatible, enabling easier processing and a more comprehensive range of applications. [4] Functionalizing standard fabrics with conductive materials is a popular approach. Methods like screen printing, dip-, spray-, blade-coating, and solution deposition of inks or pastes are efficient for large-area functionalization of textiles at ambient temperature and pressure. [5] Since signal transmission, electronic conduction, and thermal property depend on the integrity of the conductive paths, wearable interconnects require the stability of the electrical performance of the conductive textile upon deformation and washing. A green wearable conductor tunable and adaptable in terms of change in resistance with deformation would be ideal since it could satisfy divergent needs with a single solution, which would bring us closer to the demand of electronics. The challenge is coating the textile with sustainable conductive ink, realized using 1D, 2D carbonaceous filler and hybrid systems and controlling the formulation as well as the three-dimensional distribution to exploit the divergent needs of high and stable conductivity and piezo-resistivity using sustainable polymers and solvents made by mixing water and biodegradable surfactants such as polymers based on PVA.
Owing that, the aim of the proposal is to study, by using the instrument suite of IM@IT, the correlation between the filler spatial distribution and the coating parameters adopted by Bata University to deposit the inks on the textile substrate. The scope is to investigate how the aspect ratio and shape of the filler (1D and 2D filler) may affect the spatial distribution as consequence of the different technology adopted for the processing of the composites.

2. Proposed experiment

The sustainable Inks prepared by Bata University containing carbonaceous fillers (1D: multiwalled carbon nanotubes, MWCNTs; 2D: graphene derivatives; mixtures of 1D/2D nanofillers) applied by using different deposition technologies (i.e. rod coaters, dip coating, spray coating), for a total of 9 samples will be characterized with the following technique.

- Small and Wide Angle X-ray Diffractometer (SAXS/WAXD) (Unit CNR-IPCB): to obtain info about filler orientation and hierarchical structure and crystallinity degree of polymeric phase adopted in the Inks formulation. The sample size will be compliant with the characterization technique:

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In distinct proposals the same samples will be analyzed by Scanning Electron Microscopy (SEM FEI) to evaluate the coating morphology, whereas the inks from polyurethane water dispersions will be analyzed by transmission electron microscopy (TEM FEI) to evaluate their morphology in the polyurethane matrix.

3. Summary of previous experimental proposals or characterisation

No previous experiments have been carried out on these samples

4. Justification of experimental time requested

We request the SAXS/WAXD equipment to evaluate the effect of the filler nature and the application process on the structure of the coatings after their application on a cotton substrate. After discussion with the instrument scientist we request 3 days of SAXS/WAXD beam time, necessary for the analysis of n. 9 inks corresponding to 3 different fillers (1D, 2D and hybrid systems) deposited on the cotton substrates through rod coating, dip-coating and spray-coating technology, by modulating the acquisition time to optimize the spectra and highlight the presence of the fillers, by scanning the accessible q range from 0.06 nm-1 to 40.7 nm-1.
The foreseen beam time accounts for set up and for the data collection on the samples.

References

[1] S. R. Joshi, S. Kumar, and S. Kim, “Ecofriendly Polymer–Graphene-Based Conductive Ink for Multifunctional Printed Electronics,” Adv.Mater. Technol., vol. 2201917, pp. 1–9, 2023, doi:10.1002/admt.2022019172.
[2] P. Cataldi et al., “A Green Electrically Conductive Textile with Tunable Piezoresistivity and Transiency,” Adv. Funct. Mater., 2023, doi:10.1002/adfm.2023015423.
[3] L. Jiang, H. Hong, and J. Hu, “Facile thermoplastic polyurethane-based multi-walled carbon nanotube ink for fabrication of screen-printed fabric electrodes of wearable e-textiles with high adhesion and resistance stability under large deformation,” Text. Res. J., vol. 91, no.21–22, pp. 2487–2499, 2021, doi: 10.1177/004051752110086134.
[4] S. Mondal, “Phase change materials for smart textiles – An overview,” Appl. Therm. Eng., vol. 28, no. 11–12, pp. 1536–1550, 2008, doi: 10.1016/j.applthermaleng.2007.08.0095.
[5] A. Tiwari and L. Uzun, “Advanced Functional Materials,” Adv. Funct. Mater., pp. 1–577, 2015, doi: 10.1002/9781118998977.

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Experiment Proposal

Experiment number GP2023062

Principal investigator	Dr Ivano Aglietto, GrapheneUP SE, CZECH_REPUBLIC
Co-investigator	Dr Gennaro Gentile, IPCB CNR, ITALY
Co-investigator (*)	Dr Marino Lavorgna , CNR, ITALY
Co-investigator	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Graphene-based thermoplastic composites: structural analysis by SAXS/WAXD
MRF Instrument	**SAXS WAXD** **Days requested:** 3
Access Route	Direct Access **Previous GP Number:** no
Science Areas	Engineering, Materials **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	GrapheneUP SE, Studeněves 13, 273 79 Studeněves, Czech Republic
Non-Technical Abstract	The proposal is addressed to perform the structural characterization by SAXS/WAXD of graphene composites based on polyethylene, polypropylene and polyamide produced by film extrusion, injection moulding and fabric yarn extrusion. The aim is to get insights in the spatial orientation of the 2D filler with the polymeric matrix and to correlate the preparation approaches to the final properties of the materials. In distinct experiments, the AFM/Raman and the morphological characterization by SEM FEI of the samples is requested. All requested characterization will contribute to have a clear understanding of filler distribution at different length-scale, by controlling the chemistry of interfaces through a fine functionalization of the filler realized by GrapheneUp.
Publications	-

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact Dr Marino Lavorgna , CNR, ITALY
MRF Instrument **SAXS WAXD** **Days Requested:** 3
Special requirements:

SAMPLE

Material	few layers graphene (FLG), 3 samples with different functionalization	FLG composites with polyethylene (PE), polypropylene (PP), polyamide (PA) realized by film extrusion, injection moulding and fabric yarn extrusion (9 samples)	-
Formula	C	FLG + PE; FLG + PP; FLG + PA	-
Forms	Solid	Solid	
Volume	0.100 cc	1 cc	
Weight	100 mg	1000 mg	
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	300 - K	300 - K	-
Pressure Range	- mbar	- mbar	-
Magnetic field range	- T	- T	-
Standard equipment	None	None	-
Special equipment	N/A	N/A	-

SAFETY

Prep lab needed	No	No	-
Sample Prep Hazards	-	-	-
Special equip. reqs	no	no	-
Sensitivity to air	No	No	-
Sensitivity to vapour	No	No	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	no	no	-
Radioactive Hazards	no	no	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	-

Graphene-based thermoplastic composites: structural analysis by SAXS/WAXD

1. Background and Context

Thermoplastic materials are of interest in industry due to their low cost and ease of processing and recyclability, in addition to other properties such as rigidity and high impact strength. However, plastics degrade very slowly over hundreds of years, and one of the biggest problems today is the waste produced annually by their use and the long-lasting effects that it has on the environment [1]. The graphene integration in thermoplastic polymers may enhanced significantly the materials performance, by contributing significantly toward sustainability (ie through a reduction of manufacts weight) and enhanced recyclability (ie through the improvement of re-processing as well as the performances of the recycled materials). The improvement in the functional and structural properties of graphene-based polymer nanocomposites is intimately associated with the control of the spatial distribution of graphene in the matrix. This improvement is linked to both the filler synthesis and composite processing techniques, as reported in the literature [2]. A second important problem regards the poor interfacial interactions with the polymer matrix, resulting in the poor dispersion of graphene and low load-transfer from matrix to filler, consequently affecting the final performance of the polymer nanocomposite [3,4]. Modification of graphene is achieved by adding functional groups to the surface or edge of graphene through covalent bonding and non-covalent bonding [5].
Owing that, the aim of the proposal is to study, by using the instrument suite of IM@IT, the correlation between the “chemistry” of Few Layers Graphene (FLGs), the filler spatial distribution and the processing parameters related to the main processing technologies such as film extrusion, injection molding and fabric yarn extrusion. The scope is to investigate how the chemical functionalization of FLGs may affect the spatial distribution as consequence of the different technology adopted for the processing of the composites. In particular, SAXS/WAXD will contribute to evaluate the orientation of the filler and its aggregation as well as the effect of filler on the crystallinity of the polymeric phase, which both contribute to enhance properties of the resulting composite. Moreover, AFM RAMAN will provide chemical info about the pristine FLGs and their interface interaction in the several polymeric matrices, whereas SEM FEI will provide info about assembling of nanoplatelets and spatial filler distribution.

2. Proposed experiment

The graphene-based composites will be prepared by GrapheneUP by using different polymer matrix (i.e. polyethylene, polypropylene and polyamide) and FLGs characterized by different functionalization with dodecyl amine (DA), p-phenylenediamine (PPD) hexamethylene diamine (HMD), dodecyl amine (DA) or silanes groups and alkylsilanes (AS). Different technologies (i.e. film extrusion, injection molding and fabric yarn extrusion) will be used for the production of composites. The sample size will be compliant with the needs of the different characterization techniques. The following characterization will be performed:

- Small and Wide Angle X-ray Diffractometer (SAXS/WAXD) (Unit CNR-IPCB): to obtain info on the structure of FLGs and on the orientation of 2D fillers and their effect on the crystallinity degree of polymeric phase.

In distinct proposals we asked to characterize the same samples by AFM Raman and by SEM FEI.

3. Summary of previous experimental proposals or characterisation

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No previous experiments have been carried out on these samples

4. Justification of experimental time requested

We have requested the SAXS WAXD equipment available at IPCB CNR to evaluate to obtain info on the structure of FLGs and on the orientation of 2D fillers and their effect on the crystallinity degree of polymeric phase.
We propose to measure n. 12 samples (9 composites corresponding to three polymeric matrices realized by using three processing technologies, and 3 pristine FLGs) by modulating the acquisition time to optimize the spectra and highlight the presence of the fillers, by scanning the accessible q range from 0.06 nm-1 to 40.7 nm-1.
After discussion with the instrument scientist request 3 days of SAXS WAXD beam time for the structural characterization of the materials. The foreseen beam time accounts set up and for the data collection on the samples.

References

[1] Jagadeesh, P., et al., (2022), Sustainable recycling technologies for thermoplastic polymers and their composites: A review of the state of the art, Polymer Composites.2022;43:5831–5862.
[2] Salzano De Luna, et al., (2019) Nanocomposite polymeric materials with 3D graphene-based architectures: from design strategies to tailored properties and potential applications, Progress in Polymer Science, 89, 213-249.
[3] Ma, J., et al., (2018) Solubility study on the surfactants functionalized reduced graphene oxide, Colloids Surf. A Physicochem. Eng. Asp., 538, 79–85
[4] Francisco, D.L., et al., (2018) Advances in polyamide nanocomposites: A review, Polym. Compos., 40, 851–870
[5] Li, A., et al., (2017) Thermal conductivity of graphene-polymer composites: mechanisms, properties, and applications, Polymers, 9: 437.

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Experiment Proposal

Experiment number GP2023091

Principal investigator	Dr Giulia Marcucci, ISIS Neutron and Muon Source, UNITED_KINGDOM
Co-investigator (*)	Dr Daniela Di Martino, University of Milano Bicocca, ITALY
Co-investigator	Dr Massimiliano Clemenza, INFN, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Unlocking the structure and composition of a historical silver coin using Wide Angle X-ray Diffraction in combination with Muon and Neutron Techniques
MRF Instrument	**SAXS WAXD** **Days requested:** 1
Access Route	Direct Access **Previous GP Number:** NO
Science Areas	Cultural Heritage, Materials **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	The INFN has funded the CHNET_TANDEM collaboration aimed at the development of a non-destructive analytical technique using negative muon beams. As part of this effort, an 18th-century Portuguese coin was used to compare the muon technique with other methods, in collaboration with the IAEA. The muon beam technique revealed the coin elemental composition and depth profile, showing a possible silver enrichment. The main objectives of this proposal are to further investigate the coin composition and structure with a non-destructive approach exploiting the complementarity of information of a multi-technique protocol. We plan to use Wide Angle X-ray Diffraction to investigate the structure and potential alterations in the coin phases structure and help understand its production technology, in combination with XRD tomography and cutting-edge scientific techniques with heritage science to assess the results obtained with previous muons and neutrons analyses.
Publications	-

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Daniela Di Martino, University of Milano Bicocca, ITALY		
MRF Instrument	**SAXS WAXD**	**Days Requested:** 1	
Special requirements:			

SAMPLE

Material	Copper and Silver coin	-	-
Formula	Cu, Ag	-	-
Forms	Solid		
Volume	0.22 cc		
Weight	2 g		
Container or substrate	no	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	room temperature - K	-	-
Pressure Range	no applied pressure - mbar	-	-
Magnetic field range	no applied magnetic field - T	-	-
Standard equipment	None	-	-
Special equipment	none	-	-

SAFETY

Prep lab needed	No	-	-
Sample Prep Hazards	no	-	-
Special equip. reqs	none	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	no	-	-
Equipment Hazards	-	-	-
Biological hazards	no	-	-
Radioactive Hazards	no	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Background and Context

The INFN has funded the CHNET_TANDEM collaboration aimed at the development of a non-destructive analytical technique for Cultural Heritage using negative muon beams. Proof-of-principle experiments using negative muons for elemental analysis were conducted on the Port 4 beamline of the ISIS Neutron and Muon Source from April 2015, including calibration on standard materials [1] and feasibility tests on at many archaeological artefacts, such as “bronze age” artefacts (CHNET_TANDEM INFN experiment), Roman Empire coins and ancient swords to name but a few [2-4].

As part of this project, an 18th-century Portuguese coin has been used for a round-robin comparison in participation to the IAEA (International Atomic Energy Agency) Coordinated Research Project (CRP) F11021 [5] “Enhancing Nuclear Analytical Techniques to Meet the Needs of Forensic Science” with the Muonic Atom X-ray Spectroscopy performed at PORT4 of the ISIS Neutron and Muon Source. This CRP allowed introducing, in the IAEA framework, the use of negative muons as a reference technique for non-destructive elementary characterization measurements for unique samples, such as those of cultural heritage or those measured for forensic reasons.

The application of the Muonic Atom X-ray spectroscopy allowed to perform an elemental depth profile of the coin, determining the Ag/Cu ratio from the surface to the inner core of the sample and therefore disclosing a slight silver enrichment, as shown in Fig. 1. Preliminary XRF measurements were carried out and main results for composition are listed as follow: Ag: 91.2%, Cu: 3.7%, Cl: 1.2% Au: =0.7%, Fe: 0.5%, Pb: 0.2% plus other minor components. We can also confirm that, on the surface, the coin is silver-based, with copper as a minor alloy constituent and other elements between 0.2 -1%. The main interest of this proposal is to cross-check this relatively new nuclear investigation with consolidated non-invasive techniques to reveal the exact composition (surface and bulk) and homogeneity along the depth profile and to expand the punctual elemental analysis to the phase composition representative of the entire sample, to also determine the production process, whether by minting or casting.

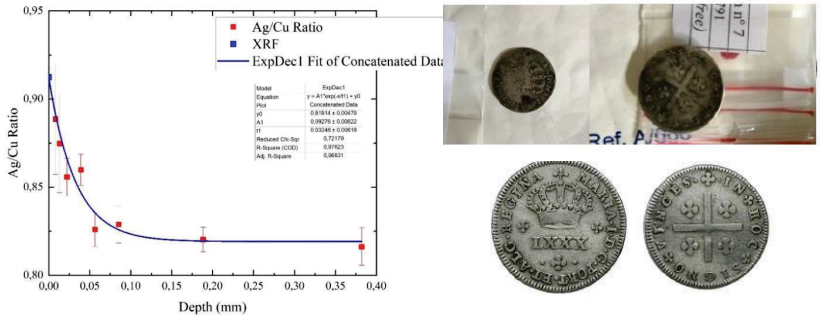

Fig. 1 On the left: Depth profile of the Ag/Cu ratio obtained through Muonic Atom X-ray spectroscopy measurements at the ISIS Neutron and Muon Source. On the right: (top) Front and rear of the Portuguese coins, 80 reis, coinage under Maria I (2 cm in diameter and 0.7 mm in thickness). (bottom) A Portuguese coin, 80 reis (coinage under Maria I) from a recent auction [6].

Sample description

A Portuguese coin, dating to the late 18th century will be investigated and is part of the round-robin comparison in CRP F11021. This coin is shown in Fig. 1. During the 18th century, the Portuguese monetary unit was the reis. The etymology comes from “*rei*” (literally meaning king), the plural being “*reis*”. Different types of coinage can be found and are either copper-, silver- or gold-based. The Portuguese coinage consisted of 5, 10, 20 and 40 reis pieces in either copper or bronze; a silver coinage of 60, 80, 120, 200 and 400 reis and gold coinage of 480, 800, 1,200, 1,600, 3,200 and 6,400 reis. Our coin has inscribed on it “*LXXX*” and is therefore 80 reis. In addition, the name of the queen (Queen Maria I who ruled from 1777 to 1799). A picture of the sample (front and rear) is shown below.

Proposed experiment

The primary objectives of this study are as follows:

i. Phase composition and distribution analysis: Perform XRD tomography to determine the precise composition of the coin, including the ratio of silver to copper and the presence of any alloying elements to cross-check the Muonic Atom X-ray Spectroscopy results;
ii. Structural Composition: Investigate the structure and potential alterations in the coin's phases structure by WAXD analysis caused by historical factors such as copper depletion and minting techniques.
iii. Historical Context: Correlate the findings with historical records and numismatic data to provide insights into the coin's origin, purpose, and significance.

We propose to use WAXD analysis to accomplish our research objectives, considering also this three-fold motivation: 1) the sample is an ancient artefact, and non-destructive analyses should be used to preserve its uniqueness; 2) no cleaning will be performed on the sample– we will be able to perform the measurement also in the presence of corrosion layers or deposits, suggested by XRF measurements; 3) the sample is bulky, and we want to infer not only the mean bulk composition but the depth profile. In this regard, another proposal will be submitted for the same sample for XRD tomography measurements to accomplish the phase composition investigation. These two experiments will be useful in complementing the information collected through neutron diffraction and neutron resonance capture analysis carried out at the INES beamline at ISIS (RB2010534, “Combination of neutron based techniques to derive the composition of an 18th-century coin”)."

We would like to underline that this round-robin is on a real sample. Other measurements have been done on standards; however, the study of a real case is mandatory when these techniques are to be used on real specimens and historical artefacts are always not homogeneous and present different issues in comparison to a standard sample.
Therefore, we aim to measure n. 1 sample in three different positions with a Cu Kα radiation source, in the diffraction range up to 60° 2theta. Hence, we request, I day which accounts also for setup time.

References

[1] A.D. Hillier et al, Microchemical Journal. Vol. 125, March 2016, Pages 203–207.
[2] M. Clemenza et al. Nucl. Instrum. Meth. Phys. Res. A . 936, (2019), Pages 27-28
[3] A.D. Hillier, A. M. Pollard, A. Wilson, D. MckPaul, et al in prep, see expt report RB 1520462.
[4] A. I. Wilson 'The metal supply of the Roman Empire', in E. Papi and B. Scardigli (eds), Supplying Rome.
[5]A. Fajgelj et al The IAEA’s Analytical Quality Control Services (AQCS) Programme on Intercomparison Runs and Reference Materials. IAEASM-344/3. 1997
[6] See for example “Lot 337 Auction 23” where 4 of these coins were estimated at 40 euros https://numismaticaleiloes.bidinside.com/en/lot/335/portugal-d-pedro-ii-to-d-maria-i-4-/

SEM FEI

SEM FEI

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Experiment Proposal

Experiment number GP2023057

Principal investigator	Professor Anita Grozdanov, Skopje University, MACEDONIA
Co-investigator	Dr Marino Lavorgna , CNR, ITALY
Co-investigator (*)	Dr Gennaro Gentile, IPCB CNR, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	SEM FEI morphological analysis of polyninylalcohol/polyacrylic acid/MXenes nanocomposites
MRF Instrument	**SEM FEI** **Days requested:** 2
Access Route	Direct Access **Previous GP Number:** No
Science Areas	Engineering, Materials **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	The proposal aims to perform a morphological characterization of polyninylalcohol/polyacrylic acid/MXenes nanocomposites by scanning electro microscope SEM FEI. In distinct proposals, we aim to perform both a structural analysis of the samples using the small and wide angle X-ray diffraction, using the SAXS WAXD and the analysis of the filler spatial distribution in the polymer matrix by the transmission electron microscope TEM FEI, all operating at the IPCB-CNR Unit. This proposal is specifically addressed to get new insights in the exfoliation degree and the spatial distribution of 2D Mxenes nanofillers in highly amorphous polyninylalcohol (HAVOH)/polyacrylic acid (PAA) blends and to correlate the preparation approaches to the structure and morphology and to the final properties of the materials, with particular attention on their electrical conductivity, their EMI shielding and their gas barrier properties.
Publications	-

ISIS neutron and muon source	**E-platform:** No
Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Gennaro Gentile, IPCB CNR, ITALY
MRF Instrument	**SEM FEI** **Days Requested:** 2
Special requirements:	

SAMPLE

Material	HAVOH neat (1 sample)	HAVOH + PAA (3 samples)	HAVOH + PAA + MXENES (2 samples)
Formula	polyvinylalcohol	polyvinylalcohol + polyacrylic acid	polyvinylalcohol + polyacrylic acid + mxenes
Forms	Solid	Solid	Solid
Volume	0.1 cc	0.1 cc	0.1 cc
Weight	100 mg	100 mg	100 mg
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	300 - 300 K	300 - 300 K	300 - 300 K
Pressure Range	- MPa	- mbar	- mbar
Magnetic field range	- T	- T	- T
Standard equipment	None	None	None
Special equipment	N/A	N/A	N/A

SAFETY

Prep lab needed	No	No	No
Sample Prep Hazards	no	no	no
Special equip. reqs	N/A	no	N/A
Sensitivity to air	Yes	Yes	Yes
Sensitivity to vapour	Yes	Yes	No
Experiment Hazards	no	no	no
Equipment Hazards	-	-	-
Biological hazards	no	no	no
Radioactive Hazards	no	no	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	Disposed by IS

SEM FEI morphological analysis of polyninylalcohol/polyacrylic acid/MXenes nanocomposites

1. Background and Context

Polymer composites with nanoparticles as fillers are a growing group of materials with interesting properties for variety of application. Although numerous composites with nanofillers have been prepared and studied in last decade, mainly with carbon based fillers as carbon nanotubes or graphene, there are still challenges when new type of nanoparticles are discovered or synthetized.
MXenes are new types of 2D materials described first in the paper of Barsoum et al. in 2011 [1]. General formula for MXenes is Mn+1XnTx (n = 1–3), where M represents transition metals (Sc, Ti, Zr, Hf, V, Nb, Ta, Cr, Mo, etc.), X is carbon and/or nitrogen and Tx refer to different functional groups on the surface (e.g. OH, O, F, etc). Moreover, MXenes particles are highly electrically conductive. Shahzad et al. [2] have shown that flexible Ti3C2Tx films exhibit excellent electrical conductivity and electromagnetic interference (EMI) shielding capacity. Electrical conductivity reached 4600 S/cm, what originates from the high electron density of states near the Fermi level. In addition, due to their 2D morphology, MXenes are very promising to impart high gas barrier properties to polymer nanocomposites.
With the objective of preparing new nanocomposites with high gas barrier properties, high electrical conductivity and electromagnetic interference (EMI) shielding properties, in this activity new polymer blends filled with MXenes have been realized at variable composition. As a polymer matrix, an easy water soluble polvinylalcohol, high amorphous polyvinylalcohol (HAVOH) has been used [3], blended with polyacrylic acid (PAA) at variable molecular weight. Indeed, after thermal treatments, HAVOH/PAA blends are prone to give light crosslinking, with improvement of their stability to high relative humidity environments. HAVOH/PAA blends have been additivated with MXenes, in particular Ti3C2, prepared by etching the aluminium from the MAX phase Ti3AlC2.

2. Proposed experiment

The HAVOH/PAA nanocomposites at variable PAA molecular weight and HAVOH/PAA weight ratio, and containing 5 phr of MXenes have been realized by Skopje University - Faculty of Technology and Metallurgy, in cooperation with IPCB-CNR. In particular, HAVOH/PAA blends in water solutions have been prepared and additivated with the MXenes. Then films (about 50 micrometer thick) have been prepared by water casting. On the obtained films thermal treatments have been performed in oven to promote crosslinking between the HAVOH and the PAA phase. The following samples have been prepared for their characterization by SEM FEI and, in distinct proposals, with SAXS WAXD and TEM FEI: 1) HAVOH neat; 2) HAVOH/PAA_4k 70/30; 3) HAVOH/PAA_4k 50/50; 4) HAVOH/PAA_240k 50/50; 5) HAVOH/PAA_4k 50/50 + 5phr Mxenes; 6) HAVOH/PAA_240k 50/50 + 5phr Mxenes.
The following characterization will be performed on these samples to evaluate the effect of the composition (HAVOH/PAA ratio, MW of PAA, MXenes additivation) on the morphology of the composites:

- Morphological analysis by scanning electron microscope (SEM FEI) (Unit IPCB CNR): to obtain info about the effect of the blend composition and the additivation of 2D fillers on the morphology of the samples. It is proposed to measure n. 6 samples (1 HAVOH neat, 3 on HAVOH/PAA blends, 2 nanocomposites filled with 5 phr Mxenes). For SEM of blends and nanocomposites, cryo-fractured surfaces of the samples will be analyzed. SEM analysis should be performed at suitable acceleration voltage using secondary electron detectors.

In distinct proposals the same samples will be analyzed by small and wide angle X ray diffraction (SAXS WAXD) and Transmission Electron Microscopy TEM FEI), available at the IPCB CNR Unit.

3. Summary of previous experimental proposals or characterisation

No previous experiments have been carried out on these samples

4. Justification of experimental time requested

We request the SEM FEI equipment available at the IPCB CNR Unit to evaluate the effect of the composition (HAVOH/PAA ratio, PAA molecular weight, MXenes additivation) on the morphology of the composites.
We request 2 days of SEM FEI beam time, necessary for the morphological characterization of the 6 above described materials, after discussion with the instrument scientist. The foreseen beam time accounts for set up and for the data collection on the samples.

References

[1] M. Naguib, M. Kurtoglu, V. Presser, J. Lu, J. Niu, M. Heon, L. Hultman, Y. Gogotsi, and M. W. Barsoum. Two-dimensional nanocrystals produced by exfoliation of Ti 3AlC 2. Adv. Mater. 23 (2011), p. 4248–4253.
[2] F. Shahzad, M. Alhabeb, C. B. Hatter, B. Anasori, S. M. Hong, C. M. Koo, and Y. Gogotsi. Electromagnetic interference shielding with 2D transition metal carbides (MXenes). Science 353 (2016), p. 1137–1140.
[3] C. Santillo, A.P. God, R.K.Donato, R.J.Espanhol Andrade, G.G. Buonocore, H. Xia, M. Lavorgna, A. Sorrentino. Tuning the structural and functional properties of HAVOH-based composites via ionic liquid tailoring of MWCNTs distribution. Composites Science and Technology, 207, 2021, 108742.

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Experiment Proposal

Experiment number GP2023059

Principal investigator	Professor Vladimir Sedlarik, Tomas Bata University in Zlin, CZECH_REPUBLIC
Co-investigator	Dr Marino Lavorgna , CNR, ITALY
Co-investigator (*)	Dr Gennaro Gentile, IPCB CNR, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Innovative sustainable inks for wearable sensors: morphological characterization by SEM FEI
MRF Instrument	**SEM FEI** — **Days requested:** 2
Access Route	Direct Access — **Previous GP Number:** no
Science Areas	Engineering, Materials — **DOI:** -
Sponsored Grant	None — **Sponsor:** -
Grant Title	- — **Grant Number:** -
Start Date	- — **Finish Date:** -
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	The proposal is aimed at characterizing new sustainable inks based on polyurethane, modified with several fillers (1D and 2D and hybrid systems) and applied by conventional deposition techniques on selected textiles for the realization wearable sensors. The challenge is to have control of the deposition procedure to increase the filler-filler contacts and enhance the electron conductivity, at lower filler content, by maximizing the coating durability in washing cycles. Tomas Bata University needs to further improve its understanding of the developed systems by investigating the spatial filler distribution and correlating results to the coating processing conditions. This proposal is addressed to perform the morphological characteriaztion by SEM FEI of carbonaceous filler-based sustainable inks. In distinct proposals the structural characterization by SAXS/WAXD of the samples and the morphology of the fillers by TEM FEI is requested. All equipments are available at the IPCB CNR Unit.
Publications	-

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Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Gennaro Gentile, IPCB CNR, ITALY
MRF Instrument	**SEM FEI** — **Days Requested:** 2
Special requirements:	

SAMPLE

Material	cotton fabrics treated with PU + 1D fillers (MWCNTs) by different deposition technologies (i.e. rod coaters, dip coating, spray coating) (3 samples)	cotton fabrics treated with PU + 2D fillers (graphene) by different deposition technologies (i.e. rod coaters, dip coating, spray coating) (3 samples)	cotton fabrics treated with PU + 1D fillers (MWCNTs) + 2D fillers (graphene) by different deposition technologies (i.e. rod coaters, dip coating, spray coating) (3 samples)
Formula	cotton, polyurethane (PU), MWCNTs	cotton, polyurethane (PU), graphene	cotton, polyurethane (PU), MWCNTs, graphene
Forms	Solid	Solid	Solid
Volume	0.500 cc	0.500 cc	0.5 cc
Weight	500 mg	500 mg	500 mg
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	300 - K	300 - K	300 - K
Pressure Range	- mbar	- mbar	- mbar
Magnetic field range	- T	- T	- T
Standard equipment	None	None	None
Special equipment	N/A	N/A	N/A\

SAFETY

Prep lab needed	Yes	Yes	No
Sample Prep Hazards	no	-	no
Special equip. reqs	-	no	no
Sensitivity to air	No	No	No
Sensitivity to vapour	No	No	No
Experiment Hazards	no	no	no
Equipment Hazards	-	-	-
Biological hazards	no	no	no
Radioactive Hazards	no	no	no
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	Disposed by IS

Innovative sustainable inks for wearable sensors: morphological characterization by SEM FEI

1. Background and Context

The rapid development of IoT and smart wearable devices has contributed to the enormous demand for smart flexible strain sensors. Unfortunately, the realization of smart textiles isn't always sustainable. [1] Thus, technological interests are growing in developing green composite materials as inks for conductive connections and piezo resistors by embedding nano carbons, such as 1D nanotubes, or 2D platelets. [2, 3] The advantages of polymeric nanocomposites with carbonaceous fillers are the low cost, lightweightness, and ease of dispersibility in environmentally friendly solvents. Another benefit of 1D and 2D nano carbons is their high aspect ratio, which ensures an efficient electrical percolation network at low loadings. These non-metal inks do not require a post-coating sintering step, which can reach damaging temperatures for common flexible polymer substrates such as cotton and cellulose. They create a stable conductive ink with time and, in some instances, are biocompatible, enabling easier processing and a more comprehensive range of applications. [4] Functionalizing standard fabrics with conductive materials is a popular approach. Methods like screen printing, dip-, spray-, blade-coating, and solution deposition of inks or pastes are efficient for large-area functionalization of textiles at ambient temperature and pressure. [5] Since signal transmission, electronic conduction, and thermal property depend on the integrity of the conductive paths, wearable interconnects require the stability of the electrical performance of the conductive textile upon deformation and washing. A green wearable conductor tunable and adaptable in terms of change in resistance with deformation would be ideal since it could satisfy divergent needs with a single solution, which would bring us closer to the demand of electronics. The challenge is coating the textile with sustainable conductive ink, realized using 1D, 2D carbonaceous filler and hybrid systems and controlling the formulation as well as the three-dimensional distribution to exploit the divergent needs of high and stable conductivity and piezo-resistivity using sustainable polymers and solvents made by mixing water and biodegradable surfactants such as polymers based on PVA.
Owing that, the aim of the proposal is to study, by using the instrument suite of IM@IT, the correlation between the filler spatial distribution and the coating parameters adopted by Bata University to deposit the inks on the textile substrate. The scope is to investigate how the aspect ratio and shape of the filler (1D and 2D filler) may affect the spatial distribution as consequence of the different technology adopted for the processing of the composites.

2. Proposed experiment

The sustainable Inks prepared by Bata University containing carbonaceous fillers (1D: multiwalled carbon nanotubes, MWCNTs; 2D: graphene derivatives; mixtures of 1D/2D nanofillers) applied by using different deposition technologies (i.e. rod coaters, dip coating, spray coating), for a total of 9 samples will be characterized by scanning electron microscopy (SEM FEI) (Unit CNR-IPCB): to obtain info about the morphology of the coatings applied onto cotton substrate the following technique. The sample size will be compliant with the characterization technique:

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In distinct proposals the same samples will be analyzed by small and X-ray diffraction (SAXS/WAXD) to evaluate the coating structure, whereas the inks from polyurethane water dispersions will be analyzed by transmission electron microscopy (TEM FEI) to evaluate their morphology in the polyurethane matrix.

3. Summary of previous experimental proposals or characterisation

No previous experiments have been carried out on these samples

4. Justification of experimental time requested

We request the SEM FEI equipment to evaluate the effect of the filler nature and the application process on the morphology of the coatings after their application on a cotton substrate.
After discussion with the instrument scientist we request 2 days of SEM FEI beam time, to measure n. 9 inks corresponding to 3 different fillers (1D, 2D and hybrid systems) deposited on the cotton substrates through rod coating, dip-coating and spray-coating technology. SEM experiments will be conducted in high vacuum mode at acceleration voltages established by the Instrument scientist to better evidence the sample morphology.
The foreseen beam time accounts for set up and for the data collection on the samples.

References

[1] S. R. Joshi, S. Kumar, and S. Kim, “Ecofriendly Polymer–Graphene-Based Conductive Ink for Multifunctional Printed Electronics,” Adv.Mater. Technol., vol. 2201917, pp. 1–9, 2023, doi:10.1002/admt.2022019172.
[2] P. Cataldi et al., “A Green Electrically Conductive Textile with Tunable Piezoresistivity and Transiency,” Adv. Funct. Mater., 2023, doi:10.1002/adfm.2023015423.
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Experiment Proposal

Experiment number GP2023063

Principal investigator	Dr Ivano Aglietto, GrapheneUP SE, CZECH_REPUBLIC
Co-investigator (*)	Dr Gennaro Gentile, IPCB CNR, ITALY
Co-investigator	Dr Marino Lavorgna , CNR, ITALY
Co-investigator	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Graphene-based thermoplastic composites: morphological characterization by SEM FEI
MRF Instrument	**SEM FEI** **Days requested:** 2
Access Route	Direct Access **Previous GP Number:** no
Science Areas	Engineering, Materials **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	GrapheneUP SE, Studeněves 13, 273 79 Studeněves, Czech Republic
Non-Technical Abstract	The proposal is addressed to perform the morphological characterization by SEM FEI of graphene composites based on polyethylene, polypropylene and polyamide produced by film extrusion, injection moulding and fabric yarn extrusion. The aim is to get insights in the spatial distribution of the 2D filler with the polymeric matrix and to correlate the preparation approaches to the final properties of the materials. In distinct experiments, the AFM/Raman and the structural characterization by SAXS/WAXD of the samples is requested. All requested characterization will contribute to have a clear understanding of filler distribution at different length-scale, by controlling the chemistry of interfaces through a fine functionalization of the filler realized by GrapheneUp.
Publications	-

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact Dr Gennaro Gentile, IPCB CNR, ITALY
MRF Instrument **SEM FEI** **Days Requested:** 2
Special requirements:

SAMPLE

Material	few layers graphene (FLG), 3 samples with different functionalization	FLG composites with polyethylene (PE), polypropylene (PP), polyamide (PA) realized by film extrusion, injection moulding and fabric yarn extrusion (9 samples)	-
Formula	C	FLG + PE; FLG + PP; FLG + PA	-
Forms	Solid	Solid	
Volume	0.100 cc	1 cc	
Weight	100 mg	1000 mg	
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	300 - K	300 - K	-
Pressure Range	- mbar	- mbar	-
Magnetic field range	- T	- T	-
Standard equipment	None	None	-
Special equipment	N/A	N/A	-

SAFETY

Prep lab needed	No	No	-
Sample Prep Hazards	-	-	-
Special equip. reqs	no	no	-
Sensitivity to air	No	No	-
Sensitivity to vapour	No	No	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	no	no	-
Radioactive Hazards	no	no	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	-

Graphene-based thermoplastic composites: morphological characterization by SEM FEI

1. Background and Context

Thermoplastic materials are of interest in industry due to their low cost and ease of processing and recyclability, in addition to other properties such as rigidity and high impact strength. However, plastics degrade very slowly over hundreds of years, and one of the biggest problems today is the waste produced annually by their use and the long-lasting effects that it has on the environment [1]. The graphene integration in thermoplastic polymers may enhanced significantly the materials performance, by contributing significantly toward sustainability (ie through a reduction of manufacts weight) and enhanced recyclability (ie through the improvement of re-processing as well as the performances of the recycled materials). The improvement in the functional and structural properties of graphene-based polymer nanocomposites is intimately associated with the control of the spatial distribution of graphene in the matrix. This improvement is linked to both the filler synthesis and composite processing techniques, as reported in the literature [2]. A second important problem regards the poor interfacial interactions with the polymer matrix, resulting in the poor dispersion of graphene and low load-transfer from matrix to filler, consequently affecting the final performance of the polymer nanocomposite [3,4]. Modification of graphene is achieved by adding functional groups to the surface or edge of graphene through covalent bonding and non-covalent bonding [5].
Owing that, the aim of the proposal is to study, by using the instrument suite of IM@IT, the correlation between the “chemistry” of Few Layers Graphene (FLGs), the filler spatial distribution and the processing parameters related to the main processing technologies such as film extrusion, injection molding and fabric yarn extrusion. The scope is to investigate how the chemical functionalization of FLGs may affect the spatial distribution as consequence of the different technology adopted for the processing of the composites. In particular, SEM FEI will provide info about assembling of nanoplatelets and spatial filler distribution. Moreover, SAXS/WAXD will contribute to evaluate the orientation of the filler and its aggregation as well as the effect of filler on the crystallinity of the polymeric phase, which both contribute to enhance properties of the resulting composite, whereas AFM RAMAN will provide chemical info about the pristine FLGs and their interface interaction in the several polymeric matrices.

2. Proposed experiment

The graphene-based composites will be prepared by GrapheneUP by using different polymer matrix (i.e. polyethylene, polypropylene and polyamide) and FLGs characterized by different functionalization with dodecyl amine (DA), p-phenylenediamine (PPD) hexamethylene diamine (HMD), dodecyl amine (DA) or silanes groups and alkylsilanes (AS). Different technologies (i.e. film extrusion, injection molding and fabric yarn extrusion) will be used for the production of composites. The sample size will be compliant with the needs of the different characterization techniques. The following characterization will be performed:

- Scanning Electron Microscopy (SEM/TEM) (Unit CNR-IPCB): to obtain more insights into the morphology of FLGs and their spatial distribution within the polymer composites.

In distinct proposals we asked to characterize the same samples by AFM Raman and by SAXS/WAXD.

3. Summary of previous experimental proposals or characterisation

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No previous experiments have been carried out on these samples

4. Justification of experimental time requested

We have requested the SEM FEI equipment available at IPCB CNR to evaluate to obtain info on the morphology of FLGs and their spatial distribution in the polymeric phase.
It is proposed to measure n. 12 samples (3 pristine FLGs and 9 composites corresponding to three polymeric matrix realized by using three processing technologies). For SEM of FLGs, FLGs dispersions will be deposited on aluminium stubs. For SEM of composites, cryo-fractured surfaces of the samples will be analyzed. SEM analysis should be performed at suitable acceleration voltage using secondary electron detectors.
After discussion with the instrument scientist, we request 2 days of SEM FEI beam time, for a fully and thorough morphological characterization of the materials. The foreseen beam time accounts set up and for the data collection on the samples.

References

[1] Jagadeesh, P., et al., (2022), Sustainable recycling technologies for thermoplastic polymers and their composites: A review of the state of the art, Polymer Composites.2022;43:5831–5862.
[2] Salzano De Luna, et al., (2019) Nanocomposite polymeric materials with 3D graphene-based architectures: from design strategies to tailored properties and potential applications, Progress in Polymer Science, 89, 213-249.
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Experiment Proposal

Experiment number GP2023077

Principal investigator	Miss Paola Amazio, Next Technology Tecnotessile, ITALY
Co-investigator (*)	Dr Gennaro Gentile, IPCB CNR, ITALY
Co-investigator	Dr Marino Lavorgna , CNR, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Morphological characterization of sustainable by design water and oil repellent biobased textile coatings
MRF Instrument	**SEM FEI** — **Days requested:** 2
Access Route	Direct Access — **Previous GP Number:** no
Science Areas	Engineering, Materials — **DOI:** -
Sponsored Grant	None — **Sponsor:** -
Grant Title	- — **Grant Number:** -
Start Date	- — **Finish Date:** -
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	The project aims to the morphological characterization of 2 new biobased coatings based on functionalized biomonomers (waterborne organic and hybrid coatings and hybrid sol-gel coatings) applied on 2 textile substrates (polyester and polyamide). The morphology of the fabrics and the homogeneity of the coatings will be evaluated by SEM FEI available at IPCB CNR also after domestic washing cycles, to evaluate their durability. Results will allow to select the most performant coatings and best application conditions
Publications	-

ISIS neutron and muon source — **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Gennaro Gentile, IPCB CNR, ITALY
MRF Instrument	**SEM FEI** — **Days Requested:** 2
Special requirements:	

SAMPLE

Material	neat textile samples (2 samples)	polyester textiles coated with biobased coatings (4 samples)	polyamide samples coated with biopolymers (4 samples)
Formula	polyester, polyamide 6,6	polyester, biopolymer coating	-
Forms	Solid	Solid	Solid
Volume	1 cc	1 cc	1 cc
Weight	1 g	1 g	1 g
Container or substrate	-	-	no
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	- K	- K
Pressure Range	- mbar	- mbar	- mbar
Magnetic field range	- T	- T	- T
Standard equipment	None	None	None
Special equipment	-	no	no

SAFETY

Prep lab needed	No	No	No
Sample Prep Hazards	no	no	no
Special equip. reqs	no	no	no
Sensitivity to air	No	No	No
Sensitivity to vapour	No	No	No
Experiment Hazards	no	no	no
Equipment Hazards	-	-	-
Biological hazards	no	no	no
Radioactive Hazards	no	no	no
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	Disposed by IS

Morphological characterization of sustainable by design water and oil repellent biobased textile coatings

Background and Context

In textile applications, polyester and polyamide 6.6 (nylon) are the main fibers and substrates used, because of their strength and general resistance to moisture, oils, micro-organisms and many common chemicals (1). Generally, polyester is more resistant to light and ultraviolet (UV) degradation than nylon while nylon is more resistant to hydrolysis.
For these relevant textile substrates, new water and oil repellent biobased textile coatings will be applied, based on functional biomonomers based on soybean vegetable oil. In particular, acrylated vegetable oils will be synthesized with controlled acrylation degree in two step process (Route 1:epoxidized intermidiates-partially acrylated oils) and in one step process (Route 2: acrylation). Formulations developed will be applied onto the texile substrate by direct coating, and subsequently subjected to validation. Processing equipment at will be used and new prototypes will be evaluated to perform the coating process with bio-formulations (2,3). The morphology of the fabrics and the homogeneity of the coatings will be evaluated by optical and scanning electron microscopy. The durability of the functionalisation by SEM will be analysed by measuring the performance before and after UV ageing and domestic washing. Results obtained with the lab scale approach will be validated comparison with real scale washing tests performed with commercial washing machines. Consecutive washing cycles will be performed on the fabrics to evaluate the trend of the release during subsequent washings (4). The activity will start with the experimental tests to characterize textile structure before treatment, the internal textile structure (warp, weft). SEM analysis to acquire the internal structure of textile. The images acquired will be used to model the textile structure (warp, weft). The different type of coating deposition will be studied to evaluate the quality and the effectiveness of deposition. The surface analysis methods for characterizing textile materials will be be an essential process in the understanding and optimization of surface modification.

2. Proposed experiment
SEM is commonly used for examining the surface morphology and structures of textile surfaces. The structures of textiles are affected not only by fibers but also by the processing techniques involved. An understanding of the effects of fibers and processes on the properties of the finished materials is of importance in manufacturing textiles with the desired properties. For this reason, samples treated by Next Technology Tecnotessile will be analyzed using the SEM FEI equipment available at IPCB. 10 samples of coatings applied with different processing conditions and on different textile substrates will be analyzed to evaluate best coating formulations and coating application conditions.

3. Summary of previous experimental proposals or characterisation
No previous experiments have been carried out on these samples

4. Justification of experimental time requested
We have requested the SEM FEI equipment available at IPCB CNR to evaluate to obtain info on the morphology of the two different textiles, namely polyamide and polyester, before and after treatment. It is proposed to measure n. 10 samples, (2 sample before treatment for polyamide and polyester textiles), 4 coated polyester samples (2 before, 2 after washing cycles to evaluate

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the coating durability), and 4 coated polyamide samples (2 before, 2 after washing cycles to evaluate the coating durability. For SEM analysis, textile samples will be mounted on aluminium stubs. SEM analysis will be performed at suitable conditions useful to evaluate the coating morphology at different scale lengths. After discussion with the instrument scientist, we request 2 days of SEM FEI access, for a fully and thorough morphological characterization of the materials. The foreseen beam time accounts set up and for the data collection on the samples.

References
(1) Ajmeri Jr and Ajmeri C j (2002), 'Special textiles for industry applications', Textile Magazine, 43(12), 70–72.
(2) Gandini A, Lacerda TM. From monomers to polymers from renewable resources: recent advances. Prog Polym Sci 2015;48:1–39. doi: 10.1016/j. progpolymsci.2014.11.002

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SEM ZEISS SIGMA

SEM ZEISS SIGMA

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Experiment Proposal

Experiment number GP2023067

Principal investigator (*)	Dr Oscar Putignano, CNR, ITALY
Co-investigator	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Gabriele Croci, University of Milano - Bicocca, ITALY
Co-investigator	Dr Andrea Muraro, CNR, ITALY
Co-investigator	Dr Marco Tardocchi, CNR, ITALY
Co-investigator	Dr Enrico Perelli Cippo, Consiglio Nazionale delle Ricerche, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Measurements of nanofibers distribution in IFOx sensor oxygen sensing element using SEM techniques.
MRF Instrument	**SEM ZEISS SIGMA**
Days requested:	1
Access Route	Direct Access
Previous GP Number:	no
Science Areas	Materials, Medicine, Physics
DOI:	-
Sponsored Grant	None
Sponsor:	-
Grant Title	-
Grant Number:	-
Start Date	-
Finish Date:	-
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

The recent COVID-19 pandemics highlighted the need to develop innovative diagnosis tools for lung conditions. A collaboration with clinicians, started during the acute phase of the pandemics, led to the development of a proof-of-concept prototype of a fast, mainstream oxygen sensor called IFOx sensor. One of the key element is represented by its optical sensing element whose geometrical and surface feature impact on the sensor performance. With this experiment we want to evaluate all the geometrical features by performing microscopic measurements in order to improve the optical sensing element design.

Publications -

ISIS neutron and muon source

E-platform: No

Instruments	
Days Requested:	
Access Route	
Previous RB Number:	
Science Areas	
DOI:	
Sponsored Grant	
Sponsor:	
Grant Title	
Grant Number:	
Start Date	
Finish Date:	
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Oscar Putignano, CNR, ITALY		
MRF Instrument	**SEM ZEISS SIGMA**	**Days Requested:**	1
Special requirements:			

SAMPLE

Material	stinless steel, nylon, PtTFPP	-	-
Formula	Fe Ni Cr Nylon PtTFPP	-	-
Forms	Solid		
Volume	1 cc		
Weight	10 mg		
Container or substrate	no	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	270 - 290 K	-	-
Pressure Range	900 - 1100 mbar	-	-
Magnetic field range	0 - 0 T	-	-
Standard equipment	None	-	-
Special equipment	no	-	-

SAFETY

Prep lab needed	No	-	-
Sample Prep Hazards	no	-	-
Special equip. reqs	no	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	no	-	-
Equipment Hazards	-	-	-
Biological hazards	no	-	-
Radioactive Hazards	no	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	-	-

Structure of the Science Case

Measurements of nanofibers distribution in IFOx sensor oxygen sensing element using SEM techniques.

1. Background and Context

The recent COVID-19 pandemics highlighted the need to develop innovative diagnosis tools for lung conditions. A collaboration with clinicians, started during the acute phase of the pandemics, led to the development of a proof-of-concept prototype of a fast, mainstream oxygen sensor called IFOx sensor. Off the shelf oxygen sensors work in side-stream configuration i.e., a sample of gas is spilled from the main airway and analyzed by the sensor. Most medical oxygen sensors rely on a chemical reaction to detect the fraction of oxygen in the sample gas mixture; this leads to aging of the sensing element. Moreover, the side-stream configuration and the fact that the typical reaction time of medical oxygen sensor is of some seconds, make a correlation measurement of the gas flow and oxygen concentration nearly impossible. For these reasons the IFOx sensor is designed to work in main-stream mode i.e., it measures the gas flowing in the totality of the airway. The mainstream configuration allows for seamless correlation of the gas flow and oxygen concentration measurements. The core of the IFOx sensor is an optical sensing element (OSE) based on a metal organic dye called Pt(II)-tetra-pentafluorophenyl-porphyrin (PtTFPP) dye that changes it fluorescence time depending on the oxygen concentration of its surroundings. It is important to notice that the fluorescence quencing of the OSE is not based on a chemical reaction, so ite OSE does not suffer from aging, as it is with electrochemical sensors. To maximize the surface exopsed to the gas and gas permeability the PtTFPP dye is embedded in a mesh of nano fibers obtained with electrospinning technique. Electrosoinning involves an electrostatic field to produce ultrafine fibers from polymer solutions deposited onto a suitable heating element. Electro-spun fibers have an average size of about 100 nm with narrow size distribution. The nanofibers are dyed by dipping into a suitable solution containing the PtTFPP. The uniformity of the dye process is crucial as it ensures light emission uniformity form the OSE.

2. Proposed experiment

The dye is deposited on the OSE at CNR-STIIMA laboratories in Biella. We plan to prepare a set of OSEs to be analyzed to verify the dye uniformity on multiple samples. Moreover, we plan to analyze a sample after extreme usage, using gas flows at least twice the maximum intended value to verify the robustness of the dye.

3. Summary of previous experimental proposals or characterisation

We do not have previous proposals.

4. Justification of experimental time requested

We think that a working day on SEM ZEISS SIGMA is sufficient for the needed characterization. This measurement will be complemented by requesting another working day on Confocal Microscope 3 (of University of Milano-Bicocca). This request is the subject of another proposal.

SEM with correlative AFM

SEM with correlative AFM

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Experiment Proposal

Experiment number GP2023045

Principal investigator	Dr Francesco Pintacuda, STMicrocelectronics, ITALY
Co-investigator (*)	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Roberto Senesi, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Carla Andreani, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Characterisation of the degree of damage by neutron induced single-event burnout failure in SiC MOSFET by SEM measurements
MRF Instrument	**SEM with correlative AFM** — **Days requested:** 2
Access Route	Direct Access — **Previous GP Number:** -
Science Areas	Energy, Engineering, ICT, Materials, Physics — **DOI:** -
Sponsored Grant	None — **Sponsor:** -
Grant Title	- — **Grant Number:** -
Start Date	- — **Finish Date:** -
Similar Submission?	-
Industrial Links	STMicroelectronics

Non-Technical Abstract

We propose to perform materials-to-circuits characterisation of SiC MOSFETs devices, already irradiated with fast neutron on the ChipIR beamline, using the SEM with correlative AFM, operating at the University of Rome Tor Vergata Unit of IM@IT. In this measurement we wish to access the degree of damage by neutron induced SEBs failure on SiC occurred after neutron irradiation. In addition, we propose in separate proposals a 3D reconstruction analysis of the damage, using the XRD Tomography instrument located at the IPCB-CNR Unit, as well as residual stress analysis of survived SiC MOSFETs from neutron-induced SEBs, using the high-resolution X-Ray diffractometer and Raman spectrtoscopy instruments, located at the University of Milano Bicocca and at the University of Rome Tor Vergata Units, respectively. All the physical quantities inferred in this study have a direct impact on the understating of the mechanisms triggering SEBs in SiC power MOSFETs.

Publications

Pintacuda et al., Prototyping and characterization of radiation hardened SiC MOS structures, 2019 European Space Power Conference (ESPC).
F. Principato et al., Sensors 20 (2020), 3021; F. Principato et al., Sensors 21 (2021), 5627
AJ Allen, MT Hutchings, CG Windsor, C Andreani, Neutron diffraction methods for the study of residual stress fields, Advances in Physics, 34, 445-473 (1985)

ISIS neutron and muon source — **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
MRF Instrument	**SEM with correlative AFM** — **Days Requested:** 2
Special requirements:	

SAMPLE

Material	SiC	-	-
Formula	SiC	-	-
Forms	Solid		
Volume	0.004 cc		
Weight	12.84 mg		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	293 - K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Characterisation of the degree of damage by neutron induced single-event burnout failure in SiC MOSFET by SEM-EDS measurements

1. Background and Context

Silicon carbide (SiC) is a IV–IV compound material with unique physical and chemical properties. The strong chemical bonding between Si and C atoms gives this material very high hardness, chemical inertness, and high thermal conductivity [1]. As a semiconductor, SiC exhibits a wide bandgap, high critical electric field strength, and high saturation drift velocity. Wide-bandgap semiconductors promise high tolerance to extreme environments, such as ionizing radiation, energetic particles, high and low temperatures. Both n- and p-type control across a wide doping range is relatively easy in SiC; this makes SiC exceptional among wide bandgap semiconductors. The ability of SiC to form silicon dioxide (SiO_2) as a native oxide is an important advantage for device fabrication. Because of these properties, SiC is a promising semiconductor for high-power and high-temperature electronics [2-4] both in space and at ground level. In comparison to silicon, SiC is a superior material thanks to its higher breakdown field and thermal conductivity. For terrestrial applications, power semiconductor devices like SiC metal–oxide–semiconductor field-effect transistor (SiC MOSFET) or insulated-gate bipolar transistors (IGBTs) [5] suffer of single-event burnout (SEB) failure when irradiated by high-energy neutrons [6] which constitute the 97% of the total [7] flux of high-energy particles impinging on such device at sea level. Experiments of neutron-induced SEBs in SiC MOSFET at high-voltage have shown the formation of cracks at the device surface [8] as shown in Figure 1.

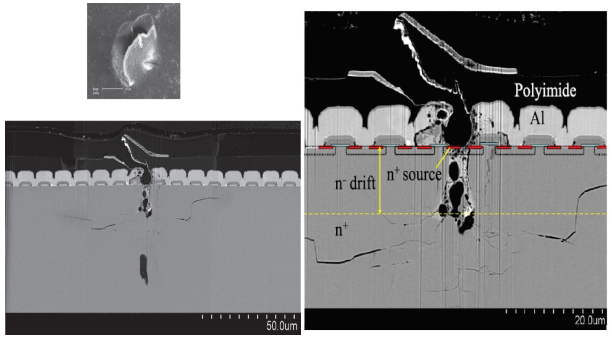

Figure 1: (Left panel) Cross-sectional SEM image of SEB damage for a SiC MOSFET. (Right panel) Magnified view of the damage and device structure [8].

We plan to perform non-destructive characterisations of damaged and survived SiC MOSFETs, following neutron induced SEBs by fast neutron test at the ChipIr beamline, using scanning electron microscopy (SEM) and X-ray computed topography (XCT); in addition, the stress field [9] will be studied using X-ray diffraction (XRD) and Raman spectroscopy measurements. To this aim by four distinct proposals, we have requested the use of SEM with correlation AFM, XRD Tomography (for XCT), X-Ray Diffractometer, and the AFM Raman. All the instrumentation is available at IM@IT and operating at the Universities of Rome Tor Vergata and Milano Bicocca, and IPCB-CNR Units.

In the present proposal we wish to measure the SEBs damaged SIC MOSFETs using the SEM instrument. The SEM measurements are the gold standard procedure for assessing the possible damage present in semiconductors. By a comparison of the SEM images, like the one shown in Figure 1, with the 3D reconstruction of the SiC MOSFETs obtained from XCT independent measurements we will then perform a non-destructively benchmark. The XCT, 2D and 3D reconstruction, will help us to identify the presence of micro-burning or multiple burning, which are not easily observed with microscope after decapsulation of the package. Furthermore, the residual stress analysis will be assessed by high resolution X-ray diffraction (XRD) and Raman spectroscopy using the same procedure as reported in [8].

2. Proposed experiment on SEM with correlative AFM

In this experiment we aim to perform SEM measurements of n. 5 damaged and n. 5 survived SiC MOSFETs samples already undergone to neutron induced SEBs during a test performed at the ChipIr beamline, ISIS neutron and muon source. Cross-sectional SEM image of SEBs damage SiC MOSFET, like the one shown in Figure 1, will be compared with XCT, 2D and 3D reconstruction of the damage.

3. Justification of experimental time requested on SEM-EDS

The damage and survived SiC MOSFETs after neutron induced SEBs on ChipIr have dimensions of about 4mm x 5mm and a thickness of about 200 µm.

We aim to measure n. 5 damaged and 5 survived SiC MOSFETs using a field of view and magnification which depends on the size of the damage. We predict n. 6 image per sample. Hence, after discussion with the instrument scientist, we request 2 days of instrument time including set-up and calibration time.

4. References

[1] Harris, G.L. (1995) Properties of Silicon Carbide, INSPEC.
[2] Davis, R.F., Kelner, G., Shur,M. et al. (1991) Thin film deposition and microelectronic and optoelectronic device fabrication and characterization in monocrystalline alpha and beta silicon carbide. Proc. IEEE, 79, 677.
[3] Ivanov, P.A. and Chelnokov, V.E. (1992) Recent developments in SiC single-crystal electronics. Semicond. Sci. Technol., 7, 863.
[4] Morkoç, H., Strite, S., Gao, G.B. et al. (1994) Large-band-gap SiC, III-V nitride, and II-VI ZnSe-based semiconductor device technologies. J. Appl. Phys., 76, 1363.
[5] Pintacuda et al., Prototyping and characterization of radiation hardened SiC MOS structures, 2019 European Space Power Conference (ESPC).
[6] Principato et al., Accelerated Tests on Si and SiC Power Transistors with Thermal, Fast and Ultra-Fast Neutrons, Sensors 20 (2020), 3021;
[7] J. F. Ziegler, IBM J. Res. Dev. 40, 19 (1996).
[8] Yeong-Jae Yu et al., Residual stress analysis of 4H-SiC crystals obtained by a top-seeded solution growth method, Cryst. Eng. Comm. 19 (2017), 6731.
[9] AJ Allen, MT Hutchings, CG Windsor, C Andreani, Advances in Physics, 34, 445-473 (1985).

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Experiment Proposal

Experiment number GP2023048

Principal investigator	Dr Diego Sbardella, IRCCS Fondazione G.B. Bietti, ITALY	
Co-investigator (*)	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY	
Co-investigator	Professor Alessio Bocedi, University of Rome, Tor Vergata, ITALY	
Co-investigator	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY	
Co-investigator	Dr Laura Fazi, University of Rome Tor Vergata, ITALY	
Co-investigator	Professor Roberto Senesi, University of Rome Tor Vergata, ITALY	
Co-investigator	Dr Luigi Ambrosio, National Research Council, ITALY	
Co-investigator	Dr Tommaso Rossi, IRCCS Fondazione Bietti ONLUS, ITALY	
Co-investigator		
Experiment title	Characterisation of surgically removed vitreous humor samples by SEM measurements	
MRF Instrument	**SEM with correlative AFM**	**Days requested:** 2
Access Route	Direct Access	**Previous GP Number:** No
Science Areas	Biology and Bio-materials, Medicine, Physics	**DOI:** -
Sponsored Grant	Yes	**Sponsor:** Other
Grant Title	Profiling of physical and proteomics parameters of vitreous body in retinal detachment	**Grant Number:** 5*1000 to IRCCS Fondazione Bietti
Start Date	01/03/2023	**Finish Date:** 01/03/2025
Similar Submission?	-	
Industrial Links	BVI Medical	

Non-Technical Abstract

Rhegmatogenous Retinal Detachment (RD) is a severe eye disease that occurs when the retina becomes detached from the Retinal Pigment Epithelium due to the presence of retinal tears or holes. The gold standard treatment of RD is vitrectomy, that is the removal of part of the vitreous humor (VH) using vitreous cutters. A major question still unanswered, is whether there is a relation between the morphology (dimensions) of VH fragments generated by cutters when set with different frequency parameters. The proponents aim to study by high resolution microscopy measurements (scanning electron microscopy (SEM-AFM), Transmission electron microscopy (TEM), X-ray computed tomography (XCT)) the morphology of VH fragments surgically isolated from RD patients. In the present proposal we wish to measure the morphology and topography feature of 6 distinct VH fragments (3 samples for each vitreous cutter frequency, i.e. 5000 CPM and 20000 CPM) using the SEM with correlative AFM instrument.

Publications

T. Rossi et al., Retina 34 (2014), 1896-904.
T. Rossi et al., Invest Ophthalmol Vis Sci. 12 (2014), 8289-94.
T. Rossi et al., Translational Vision Science & Technology 11 (2022), 29.

ISIS neutron and muon source — **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY		
MRF Instrument	**SEM with correlative AFM**	**Days Requested:** 2	
Special requirements:			

SAMPLE

Material	Humor vitreous	-	-
Formula	-	-	-
Forms	Liquid		
Volume	0.002 ml		
Weight	2 mg		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Characterisation of surgically removed human vitreous samples by SEM measurements

1. Background and Context

Rhegmatogenous Retinal Detachment (RD) is a severe eye disease [1] that occurs when the retina becomes detached from the Retinal Pigment Epithelium (RPE) due to the presence of retinal tears or holes. The gold standard treatment of RD is vitrectomy, that is the removal of part of the vitreous humor (VH), a gel-like fluid that shapes the eye globe, using vitreous-cutters. About 15-20% of all RDs relapse within the first 6 months through a process called Proliferative Vitreo-Retinopathy [2] (PVR), which is characterized by inflammation, collagen deposition and retinal contraction. PVR is highly invalidating and often accompanied by sight loss, thus carrying a huge burden for the quality of life and for social and economic costs. All vitreous cutters base on the mechanism of a reciprocating blade moving within a hollow cylinder in a proximal-to-distal fashion, with cut-rates comprised between 1,000 and 20,000 cuts per minute. Given the miniaturization of retinal surgery instrumentation, cutters have evolved from 20G (0.9 mm out diameter in section) to 25G (0.5mm) and even 27G (0.4mm), making the internal fluidics even more challenging and requiring high aspiration vacuum up to 650 mmHg to win the hydraulic resistance of the highly viscous human vitreous material. High suction and blade motion applied to the collagen mesh of vitreous exert traction on the retina especially when the peripheral “vitreous base” is removed and more so when the retina is mobile during retinal detachment surgery. For this reason, the intraoperative creation of iatrogenic retinal tears and the amount of traction exerted on the retina causing further damage and possibly giving rise to Proliferative Vitreoretinopathy remains and important issue, largely unresolved. A major question still unanswered, is whether there is a relation between intraoperative retinal traction, PVR onset and the morphology (dimensions) of VH fragments (mostly collagen and proteoglycan) generated by cutters when set with different frequency (cuts per minute, CPM) parameters or whether these parameters have no effects on VH fragmentation [3]. The contribution of turbulent vitreous fluidics at the cutter port to the consistency of vitreous fragment dimensions is also a matter of speculation. If different fragments are produced, then the tensile force generated over the retina layer (which adheres to VH) as well as the mechanical stress, which is definitely responsible for intra-operative retinal traction and iatrogenic break formation and likely relate with PVR onset, may be influenced by cutter parameters. This proposal fits into a wider multidisciplinary research program of IRCCS Fondazione Bietti (IFB), a main Italian clinical center for the study and research in Ophthalmology, granted by the Ministry of Health (Profiling of physical and proteomics parameters of vitreous body in retinal detachment) and supported by industries. The proponents aim to study by high resolution microscopy measurements (scanning electron microscopy (SEM-AFM), Transmission electron microscopy (TEM), X-ray computed tomography (XCT)) the morphology of VH fragments surgically isolated from RD patients and its protein composition that can help predicting the proportion of those patients who most likely will develop PVR. To this end, we wish to use the SEM with correlative AFM, TEM FEI, XRD Tomography instruments. All the instrumentation is available at IM@IT and operating at the Universities of Rome Tor Vergata and IPCB-CNR Units. It is worth mentioning that VH fragments, generated by vitreous cutters used at two frequencies, i.e., 5000 and 20000 CPM, will be isolated from the same patient eye during two surgical phases, using

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an established surgical procedure and Good Medical Practices [3]. Documentation on the ethical issues associated to the use of VH fragments will be provided upon request.

2. Proposed experiment for SEM

In the present proposal we wish to measure the morphology and topography feature of 6 distinct VH fragments (3 samples for each vitreous cutter frequency, i.e., 5000 CPM and 20000 CPM) using the SEM with correlative AFM instrument. Results from SEM images (mean size and standard deviation of each macromolecular fragments in VH) and AFM topography (EDX used to identify collagen fibrils) measured in this experiment will be compared with TEM data and reconstruction of X-Ray computed tomography (XCT) data proposed by the proponents in other two separated proposals.

3. Summary of previous experimental proposals or characterisation

The performance of vitreous cutters, by means of hydraulic resistance posed by cut VH during aspiration, has been investigated for frequencies < 12000 CPM. Furthermore, cutter blade action determines instantaneous flow rate fluctuation that interferes significantly with VH aspiration posing a possible risk of inadvertent retinal entrapment [3,4].
Nevertheless, since VH fragments generate are supposed to be > 10 µm, current biochemical and molecular biology techniques cannot easily be applied to address the aim this proposal deals with.

4. Justification of experimental time requested for SEM

VH fragments (6 in total, 3 samples for each vitreous cutter frequency, i.e., 5000 CPM and 20000 CPM) will be measured by SEM, EDX and AFM scans using a field of view and magnification which depends on the macromolecular fragments in VH. We predict n. 5 images per sample and few AFM topography images. Hence, after discussion with the instrument scientist, we request 2 days of instrument time including set-up and calibration time.

5. References

[1] T. Schick et al., Klin Monbl Augenheilkd. 12 (2020), pp. 1479-1491.

[2] S. Yang et al., Discov Med. 110 (2015), 207.

[3] T. Rossi et al., Retina 34 (2014), 1896-904.

[4] T. Rossi et al., Invest Ophthalmol Vis Sci. 12 (2014), 8289-94.

[5] S. Pastor-Idoate et al., PLoS ONE 12 (2017), e0173883.

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Experiment Proposal

Experiment number GP2023070

Principal investigator	Professor Lorenz Baumer, Université de Genève, SWITZERLAND		
Co-investigator	Professor Luisa Cifarelli, University of Bologna and INFN-Bologna, ITALY		
Co-investigator	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY		
Co-investigator	Professor Roberto Senesi, University of Rome Tor Vergata, ITALY		
Co-investigator (*)	Dr Laura Strolin, Institut Català de Arqueologia Clàssica, SPAIN		
Co-investigator	Professor Maria Pia Morigi, University of Bologna, ITALY		
Co-investigator	Dr Maria Grazia Griffo, Museo Archeologico Regionale Lilibeo–Marsala, ITALY		
Co-investigator			
Co-investigator			
Experiment title	Analysis of nails provided by different antique shipwrecks in the Mediterranean using SEM-EDS		
MRF Instrument	**SEM with correlative AFM**	**Days requested:** 3	
Access Route	Direct Access	**Previous GP Number:** -	
Science Areas	Cultural Heritage	**DOI:** -	
Sponsored Grant	None	**Sponsor:** -	
Grant Title	-	**Grant Number:** -	
Start Date	-	**Finish Date:** -	
Similar Submission?	-		
Industrial Links	-		

Non-Technical Abstract

Ship nails can provide important information about the construction techniques of ancient ships and, depending on their typology, alloys, and internal structure, deliver information on the ship provenance and travel routes. The study of their production and mechanical treatment allows to approach questions like if there was, all over the Mediterranean a general standardization or not, based on a cultural exchange, or if there are culturally different and chronologically evolving technologies used to produce the nails.

Here we propose a surface characterization of several ship nails, from different findspots, and belonging to different cultures and periods, based on Scanning Electron Microscopy, for morphological analysis, and concurrent Energy-Dispersive X-ray Spectroscopy, to provide elemental analysis of the materials used. X-ray diffraction and neutron measurements, requested in separate proposals, will provide information on the crystal structure in the surface and in the bulk.

Publications -

Instruments	**INES**	**Days Requested:** 2	
Access Route	Direct Access	**Previous RB Number:**	
Science Areas		**DOI:**	
Sponsored Grant	None	**Sponsor:**	
Grant Title	-	**Grant Number:**	
Start Date	-	**Finish Date:**	
Similar Submission?			
Industrial Links			

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Sample record sheet

Principal contact	Dr Laura Strolin, Institut Català de Arqueologia Clàssica, SPAIN		
MRF Instrument	**SEM with correlative AFM**	**Days Requested:** 3	
Special requirements:			

SAMPLE

Material	Bronze nail	-	-
Formula	Cu, Sn	-	-
Forms	Solid		
Volume	10 cc		
Weight	90 g		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	300 - 300 K	-	-
Pressure Range	0 - 1000 mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Background and Context

Underwater excavations regularly provide an important number of nails, usually in bronze or copper alloys, used for different purposes. The so-called treenails consisting of a nail driven through a wooden peg are used fixing the planks and the frames of the ship (fig. 1a-1b), whereas wooden pegs have been used to stabilize the tenons keeping the planks in place. Shorter nails have been used to protect the outside of the hull with thin lead sheets. As the analysis of one single plank from the Antikythera shipwreck is showing (fig. 2), there is an enormous number of nails used in antique ship construction (yellow and red dots). The nails are therefore not only a fundamental part of a ship but can also deliver a rich amount of information from scientific analytical methods.

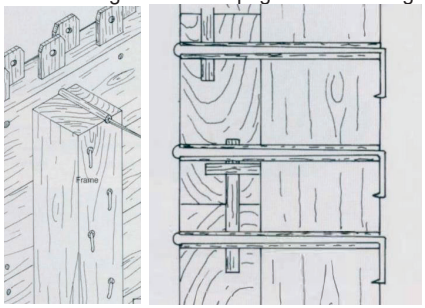

Fig. 1a and 1b: schematic representation of treenails used in antique ship construction.

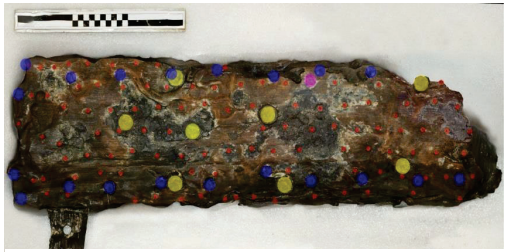

Fig. 2: Analysis of the number of different types of nails used in a single plank from the Antikythera shipwreck, 1st century BC. (yellow: treenails; red: bronze nails; blue: wooden pegs)

While ship construction was during the antiquity a highly specialized industry, e.g., for the Phoenicians, the Greeks, and the Romans, and producing a large series of special ship types for all kind of purposes (war ships, long distance cargo ships, etc.), it is astonishing that the nails, as fundamental as they are, have so far only found little interest. Aside of giving important information about the construction techniques, they can, by their typology, by their alloys, and by their internal structure deliver information e.g., about the provenance and, by analyzing reparations, about the routes of the ships. Moreover, investigating production techniques and mechanical treatment of the nails, will help understanding if there were standard technologies shared in the whole Mediterranean - thanks to cultural exchange - and how they evolved through time.

Proposed experiment

We propose a surface characterization of several ship nails, coming from different findspots, and belonging to different cultures and periods. In that interest, nails coming from at least three different shipwrecks will be analyzed, allowing the comparison of their metal composition, their provenance, and their mechanical treatment. For the time being, three shipwrecks have been selected as a starting point of the project. From each shipwreck, between 3 and 12 nails will be selected for analysis. The three ships are: the Marsala Punic (Phoenician) military ship (3rd century BC), the Antikythera Greek (?) cargo ship [3, 4, 5, 6] (1st century BC), and the Marausa Roman merchant ship found near

Trapani. While surface characterizations were already performed on samples from the Punic ship [2], here we plan a **systematic comparison of nails from the three different ships using Scanning Electron Microscopy and Energy Dispersive X-ray Spectroscopy** using the instrument SEM with correlative AFM, a TESCAN VEGA SEM, located at the University of Rome Tor Vergata, IM@IT Unit. This instrument is particularly suitable for analysing this samples given it has a suitably large sample volume where ship nails, with linear dimensions of the order of 10 cm, can be easlity accommodated and analysed. Specifically, the mapping of the elemental composition on the surface of each nail will provide information of the type of bronze alloy used, thus on its origin.

Information on the surface morphology and elemental composition gathered with the SEM-EDS will be complemented requesting, in a distinct proposal, a X-Ray analysis using X-ray diffraction/ SAXS GISAXS located at the CSGI Unit, and neutron diffraction and neutron resonance capture analysis at the INES beamline of the ISIS Facility. The combination of this set of analysis will give us comprehensive information on the elemental composition and structure (manufacturing), both on the surface and in the bulk, as well as provide information on the manufacturing procedures of these artifacts and on their origin.

Summary of previous characterizations.

As for today, only the nails of the Marsala Punic ship have found a partial analysis, whereas the nails of the two other ships remain unstudied. The members of the research group have already been working on some of the shipwrecks that will deliver the samples: 3D tomography of a few planking elements has already been done for the Marsala Punic ship, providing important information about the ship's construction [1]. The University of Geneva is leading since 2021 an international underwater excavation mission on the Antikythera shipwreck, delivering new and important information, and materials [4, 5, 6]

Justification of experimental time requested

We request 3 days of instrument time on the *SEM with correlative AFM* MRF, to be used as follows: up to 2 hours of measurements per ship nail (for a total of 4-5 ship nails per day) for each of the three ships selected. The nails to be measured will be selected during the experiment, amongst the available ones, depending on the data being collected and in order to maximize the statistical significance of the systematic characterization.

References

[1] Albertin F., Baumer L. E., Bettuzzi M., et al., *X-ray computed tomography to study archaeological clay and wood artefacts at Lilybaeum*, The European Physical Journal Plus 136, 513 (2021). https://doi.org/10.1140/epjp/s13360-021-01465-1
[2] Armetta F., Celeste Ponterio R., et al., *New Insight on Archaeological Metal Finds, Nails and Lead, Sheathings of the Punic Ship from Battle of the Egadi Islands,* Molecules 28(4), February 2023:1968. https://doi.org/10.3390/molecules28041968
[3] Kaltsas N. et al, ed., *The Antikythera Shipwreck. The ship, the treasures, the mechanism,* Athens, National Archaeological Museum 2012.
[4] Simosi A., Baumer L., E., *L'épave d'Anticythère livre peu à peu ses secrets*, Archéologia, 614, novembre 2022, 56-63.
[5] Simosi A., Baumer L., *Anticythère 2021*, Antike Kunst 65, 2022, 155-157. 163. https://www.jstor.org/stable/27164586
[6] Simosi A., Baumer L., *Anticythère 2022*, Antike Kunst 66, 2023, 119-124 (in print).

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Experiment Proposal

Experiment number GP2023092

Principal investigator	Mr Pietro Tordi, University of Florence & CSGI, ITALY		
Co-investigator	Professor Paolo Samorì, University of Strasbourg and CNRS, FRANCE		
Co-investigator	Professor Massimo Bonini, CSGI - University of Florence, ITALY		
Co-investigator	Professor Pietro Morales, University of Rome Tor Vergata, ITALY		
Co-investigator	Dr Laura Fazi, University of Rome Tor Vergata, ITALY		
Co-investigator	Dr Anna Prioriello, University of Rome Tor Vergata, ITALY		
Co-investigator	Professor Roberto Senesi, University of Rome Tor Vergata, ITALY		
Co-investigator (*)			
Co-investigator (*)			
Experiment title	Electrostrictive properties of Alginate-based composites including reduced graphene oxide and metal-based nanostructures		
MRF Instrument	**SEM with correlative AFM**	**Days requested:**	2
Access Route	Direct Access	**Previous GP Number:**	NO
Science Areas	Chemistry, Materials, Physics	**DOI:**	-
Sponsored Grant	None	**Sponsor:**	-
Grant Title	-	**Grant Number:**	-
Start Date	-	**Finish Date:**	-
Similar Submission?	-		
Industrial Links	-		

Non-Technical Abstract

Hydrogels are the subject of an increasing number of scientific studies where biomimetic approaches towards the preparation of mechanical/pressure sensors and actuators are investigated. In particular, thanks to their high deformability, self-healing and biocompatibility, hydrogels are especially interesting in biomedical applications, such as in the development of blood pressure sensors and artificial muscles. Alginate is a biocompatible and biodegradable anionic polysaccharide with high application potential due to its ability to form 2D (films) and 1D (fibers) structures thanks to its reactivity and selectivity towards metal cations and to its ability to act as a dispersant for carbon based materials. Here we propose the morphological and functional characterization of composite fibres prepared by wet spinning of alginate solutions in Cu2+ or Ag+ crosslinking baths, as well as films obtained through spray coating of alginate-reduced graphene oxide (rGO) dispersions.

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact

MRF Instrument **SEM with correlative AFM** **Days Requested:** 2

Special requirements:

SAMPLE

Material	Allginate, Copper, Silver, Graphite, Graphene, Graphene oxide	-	-
Formula	C, Cu, Ag, O, H	-	-
Forms	Solid		
Volume	0,1 cc		
Weight	100 mg		
Container or substrate	not needed	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	Room T - K	-	-
Pressure Range	up to 5 - mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	No	-	-
Sample Prep Hazards	No	-	-
Special equip. reqs	Non	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	No	-	-
Equipment Hazards	-	-	-
Biological hazards	No	-	-
Radioactive Hazards	No	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Experiment description of proposal "Electrostrictive properties of Alginate-based composites including reduced graphene oxide and metal-based nanostructures"

1. Background and Context

Hydrogels are the subject of an increasing number of scientific studies where biomimetic approaches towards the preparation of mechanical/pressure sensors and actuators are investigated. In particular, thanks to their high deformability, self-healing and biocompatibility, hydrogels are especially interesting in biomedical applications, such as in the development of blood pressure sensors and artificial muscles.(Jia et al., 2022) Some of the applicants have recently demonstrated the possibility to develop molecule-graphene hybrid materials with tunable mechano-response to be used as highly sensitive pressure sensors for health monitoring.(Huang et al., 2019) In this context, alginate is a biocompatible and biodegradable anionic polysaccharide with high application potential due to its ability to form 2D (films) and 1D (fibers) structures thanks to its reactivity and selectivity towards metal cations and to its ability to act as a dispersant for carbon based materials.(Srivastava and Choudhury, 2023; Tordi et al., 2023) Composites including graphite, graphene and graphene oxide are now being studied by the applicants: in fact, this study is part of Pietro Tordi's research activity as a PhD student in co-tutorship between the University of Florence (Italy) and the University of Strasbourg (France), funded by the Italian Ministry of University and Research (MUR) for three years. The aim of the project is the realization of Alg-based composites for pressure-based sensors and actuators. Currently part of the studies are carried out at the Institut de Science et d'Ingeniérie Supramoléculaires (ISIS, University of Strasbourg), in the Nanochemistry Lab of Prof. Paolo Samorì. The characterisation of the electro-striction properties of alginate-based composites would pave the way towards novel perspectives for the application of these materials.

2. Proposed experiment

Some of the applicants have recently reported very interesting results when characterizing the electromechanical behaviour of conductive carbon nanotubes/polymer composites intended to be used as stretchable sensors and transducers.(Fazi et al., 2023) In this proposal we aim at establishing a scientific collaboration between Italian and French research groups, from the Universities of Strasbourg, Florence and Rome, where the combination of the respective chemical and physical backgrounds would allow to characterize the electrostrictive properties of alginate-based composites. In particular, composite films and fibres will be characterized, focussing on resistivity, I/V curves, and the strain dependence of stress and current. Fibers are prepared by wet spinning of alginate solutions in Cu^{2+} or Ag^{+} crosslinking baths, while films are obtained through spray coating of alginate-reduced graphene oxide (rGO) dispersions. A green reducing agent such as ascorbic acid is used for the reduction of Cu^{2+}, Ag^{+} and GO (to obtain CuNPs, AgNPs and rGO respectively), to impart electrical conductivity to the composite.

3. Summary of previous experimental proposals or characterisation

The samples have been already characterized in terms of the alginate interaction with copper(Tordi et al., 2023), silver and graphene oxide (articles in preparation).TGA, DSC, SEM-EDX, XPS, Raman, FT-IR, tensile tests and I/V analysis were used to investigate the obtained composites, proving that their chemical composition, thermal stability, morphology, mechanical and electrical properties can be tuned as a function of the preparation. In this proposal we aim at the extension of the investigation towards the electrostrictive properties. The expertise and instrumentation available at the UTOV unit in the characterization of such properties would allow

4. Justification of experimental time requested

We are requesting the instrument "SEM with correlative AFM" as the microscope operation is available either in high or low vacuum (with a partial pressure from 7 up to 500 Pa in nitrogen and water vapour). The possibility to use low vacuum is especially important for our research as our samples consist of hydrogel films, where a small amount of water vapour is needed to keep their deformability. We request 2 days for the experiments: in fact, we expect that the first day would be needed to optimize measurement conditions and perform a screening of the most interesting samples (in terms of their electrostrictive properties), while the second day will be used to perform actual measurements on the selected samples. These are the expected figures: 12 samples will be pre-screened during the first day (30 minutes per sample, i.e. a total time of 6 hours plus the time needed to change the samples and evacuate the chamber); 6 samples in the second day, where more refined analysis will be performed, such as the cross-section analysis and the EDX compositional investigation.

References

Fazi, L., Andreani, C., D'Ottavi, C., Duranti, L., Morales, P., Preziosi, E., Prioriello, A., Romanelli, G., Scacco, V., Senesi, R., Licoccia, S., 2023. Characterization of Conductive Carbon Nanotubes/Polymer Composites for Stretchable Sensors and Transducers. Molecules 28, 1764. https://doi.org/10.3390/molecules28041764

Huang, C.-B., Witomska, S., Aliprandi, A., Stoeckel, M.-A., Bonini, M., Ciesielski, A., Samorì, P., 2019. Molecule-Graphene Hybrid Materials with Tunable Mechanoresponse: Highly Sensitive Pressure Sensors for Health Monitoring. Advanced Materials 31, 1804600. https://doi.org/10.1002/adma.201804600

Jia, W., Wang, L., Fang, F., Xu, Y., Zhao, G., Ma, X., 2022. Investigation into a conductive artificial muscle based on sodium alginate/cellulose with good response characteristics. Smart Mater Struct 31, 105018. https://doi.org/10.1088/1361-665X/ac8efd

Srivastava, N., Choudhury, A.R., 2023. Stimuli-Responsive Polysaccharide-Based Smart Hydrogels and Their Emerging Applications. Ind Eng Chem Res 62, 841–866. https://doi.org/10.1021/acs.iecr.2c02779

Tordi, P., Ridi, F., Bonini, M., 2023. A green and sustainable approach for the preparation of Cu-containing alginate fibers. Colloids Surf A Physicochem Eng Asp 132396. https://doi.org/10.1016/j.colsurfa.2023.132396

TEM FEI

TEM FEI

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Experiment Proposal

Experiment number GP2023049

Principal investigator	Dr Diego Sbardella, IRCCS Fondazione G.B. Bietti, ITALY		
Co-investigator (*)	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY		
Co-investigator	Professor Alessio Bocedi, University of Rome, Tor Vergata, ITALY		
Co-investigator	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY		
Co-investigator	Dr Laura Fazi, University of Rome Tor Vergata, ITALY		
Co-investigator	Professor Roberto Senesi, University of Rome Tor Vergata, ITALY		
Co-investigator	Dr Luigi Ambrosio, National Research Council, ITALY		
Co-investigator	Dr Tommaso Rossi, IRCCS Fondazione Bietti ONLUS, ITALY		
Co-investigator			
Experiment title	Characterisation of surgically removed human vitreous samples by TEM measurements		
MRF Instrument	**TEM FEI**	**Days requested:** 2	
Access Route	Direct Access	**Previous GP Number:** No	
Science Areas	Biology and Bio-materials, Medicine, Physics	**DOI:** -	
Sponsored Grant	Yes	**Sponsor:** Other	
Grant Title	Profiling of physical and proteomics parameters of vitreous body in retinal detachment	**Grant Number:** 5*1000 to IRCCS Fondazione Bietti	
Start Date	01/03/2023	**Finish Date:** 01/03/2025	
Similar Submission?	-		
Industrial Links	BVI Medical		

Non-Technical Abstract

Rhegmatogenous Retinal Detachment (RD) is a severe eye disease that occurs when the retina becomes detached from the Retinal Pigment Epithelium due to the presence of retinal tears or holes. The gold standard treatment of RD is vitrectomy, that is the removal of part of the vitreous humor (VH) using vitreous cutters. A major question still unanswered, is whether there is a relation between the morphology (dimensions) of VH fragments generated by cutters when set with different frequency parameters. The proponents aim to study by high resolution microscopy measurements (scanning electron microscopy (SEM-AFM), Transmission electron microscopy (TEM), X-ray computed tomography (XCT)) the morphology of VH fragments surgically isolated from RD patients. In the present proposal we wish to measure the morphology of 6 distinct VH fragments (3 samples for each vitreous cutter frequency, i.e. 5000 CPM and 20000 CPM) by transmission electron microscopy using the TEM FEI instrument.

Publications

T. Rossi et al., Retina 34 (2014), 1896-904.
T. Rossi et al., Invest Ophthalmol Vis Sci. 12 (2014), 8289-94.
T. Rossi et al., Translational Vision Science & Technology 11 (2022), 29.

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E-platform: No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY		
MRF Instrument	**TEM FEI**	**Days Requested:** 2	
Special requirements:			

SAMPLE

Material	Humor vitreous	-	-
Formula	-	-	-
Forms	Liquid		
Volume	0.002 ml		
Weight	2 mg		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Characterisation of surgically removed human vitreous samples by TEM measurements

1. Background and Context

Rhegmatogenous Retinal Detachment (RD) is a severe eye disease [1] that occurs when the retina becomes detached from the Retinal Pigment Epithelium (RPE) due to the presence of retinal tears or holes. The gold standard treatment of RD is vitrectomy, that is the removal of part of the vitreous humor (VH), a gel-like fluid that shapes the eye globe, using vitreous-cutters. About 15-20% of all RDs relapse within the first 6 months through a process called Proliferative Vitreo-Retinopathy [2] (PVR), which is characterized by inflammation, collagen deposition and retinal contraction. PVR is highly invalidating and often accompanied by sight loss, thus carrying a huge burden for the quality of life and for social and economic costs. All vitreous cutters base on the mechanism of a reciprocating blade moving within a hollow cylinder in a proximal-to-distal fashion, with cut-rates comprised between 1,000 and 20,000 cuts per minute. Given the miniaturization of retinal surgery instrumentation, cutters have evolved from 20G (0.9 mm out diameter in section) to 25G (0.5mm) and even 27G (0.4mm), making the internal fluidics even more challenging and requiring high aspiration vacuum up to 650 mmHg to win the hydraulic resistance of the highly viscous human vitreous material. High suction and blade motion applied to the collagen mesh of vitreous exert traction on the retina especially when the peripheral “vitreous base” is removed and more so when the retina is mobile during retinal detachment surgery. For this reason, the intraoperative creation of iatrogenic retinal tears and the amount of traction exerted on the retina causing further damage and possibly giving rise to Proliferative Vitreoretinopathy remains and important issue, largely unresolved. A major question still unanswered, is whether there is a relation between intraoperative retinal traction, PVR onset and the morphology (dimensions) of VH fragments (mostly collagen and proteoglycan) generated by cutters when set with different frequency (cuts per minute, CPM) parameters or whether these parameters have no effects on VH fragmentation [3]. The contribution of turbulent vitreous fluidics at the cutter port to the consistency of vitreous fragment dimensions is also a matter of speculation. If different fragments are produced, then the tensile force generated over the retina layer (which adheres to VH) as well as the mechanical stress, which is definitely responsible for intra-operative retinal traction and iatrogenic break formation and likely relate with PVR onset, may be influenced by cutter parameters. This proposal fits into a wider multidisciplinary research program of IRCCS Fondazione Bietti (IFB), a main Italian clinical center for the study and research in Ophthalmology, granted by the Ministry of Health (Profiling of physical and proteomics parameters of vitreous body in retinal detachment) and supported by industries. The proponents aim to study by high resolution microscopy measurements (scanning electron microscopy (SEM-AFM), Transmission electron microscopy (TEM), X-ray computed tomography (XCT)) the morphology of VH fragments surgically isolated from RD patients and its protein composition that can help predicting the proportion of those patients who most likely will develop PVR. To this end, we wish to use the SEM with correlative AFM, TEM FEI, XRD Tomography instruments. All the instrumentation is available at IM@IT and operating at the Universities of Rome Tor Vergata and IPCB-CNR Units. It is worth mentioning that VH fragments, generated by vitreous cutters used at two frequencies, i.e., 5000 and 20000 CPM, will be isolated from the same patient eye during two surgical phases, using an established surgical procedure and Good Medical Practices [3]. Documentation on the ethical issues associated to the use of VH fragments will be provided upon request.

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2. Proposed experiment for TEM

In the present proposal we wish to measure the morphology of 6 distinct VH fragments (3 samples for each vitreous cutter frequency, i.e. 5000 CPM and 20000 CPM) by transmission electron microscopy using the TEM FEI instrument available at the IPCB-CNR Unit of IM@IT. Results from TEM images (mean size and standard deviation of each macromolecular fragments in VH) measured in this experiment will be compared with SEM/AFM data and reconstruction of X-Ray computed tomography (XCT) data proposed by the proponents in other two separated proposals.

3. Summary of previous experimental proposals or characterisation

The performance of vitreous cutters, by means of hydraulic resistance posed by cut VH during aspiration, has been investigated for frequencies < 12000 CPM. Furthermore, cutter blade action determines instantaneous flow rate fluctuation that interferes significantly with VH aspiration posing a possible risk of inadvertent retinal entrapment [3,4].
Nevertheless, since VH fragments generate are supposed to be > 10 µm, current biochemical and molecular biology techniques cannot easily be applied to address the aim this proposal deals with.

4. Justification of experimental time requested for TEM

VH fragments (6 in total, 3 samples for each vitreous cutter frequency, i.e., 5000 CPM and 20000 CPM) will be measured by TEM scans using a field of view and magnification which depends on the macromolecular fragments in VH. We predict n. 6 images for each sample. Hence, after discussion with the instrument scientist, we request 2 days of instrument time including set-up and calibration time.

5. References

[1] T. Schick et al., Klin Monbl Augenheilkd. 12 (2020), pp. 1479-1491.

[2] S. Yang et al., Discov Med. 110 (2015), 207.

[3] T. Rossi et al., Retina 34 (2014), 1896-904.

[4] T. Rossi et al., Invest Ophthalmol Vis Sci. 12 (2014), 8289-94.

[5] S. Pastor-Idoate et al., PLoS ONE 12 (2017), e0173883.

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Experiment Proposal

Experiment number GP2023055

Principal investigator	Professor Vladimir Sedlarik, Tomas Bata University in Zlin, CZECH_REPUBLIC
Co-investigator	Dr Marino Lavorgna , CNR, ITALY
Co-investigator (*)	Dr Gennaro Gentile, IPCB CNR, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Innovative sustainable inks for wearable sensors: analysis of fillers morphology by TEM FEI
MRF Instrument	**TEM FEI** **Days requested:** 1
Access Route	Direct Access **Previous GP Number:** no
Science Areas	Engineering, Materials **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	The proposal is aimed at characterizing new sustainable inks based on polyurethane, modified with several fillers (1D and 2D and hybrid systems) and applied by conventional deposition techniques on selected textiles for the realization wearable sensors. The challenge is to have control of the deposition procedure to increase the filler-filler contacts and enhance the electron conductivity, at lower filler content, by maximizing the coating durability in washing cycles. Tomas Bata University needs to further improve its understanding of the developed systems by investigating the spatial filler distribution and correlating results to the coating processing conditions. This proposal is addressed to perform the morphological characteriaztion by TEM FEI of carbonaceous fillers . In distinct proposals the structural characterization by SAXS/WAXD and the morphological characterization by SEM FEI of the inks applied on cotton is requested. All equipments are available at the IPCB CNR Unit.
Publications	-

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact Dr Gennaro Gentile, IPCB CNR, ITALY
MRF Instrument **TEM FEI** **Days Requested:** 1
Special requirements:

SAMPLE

Material	1D fillers (MWCNTs) in a polyurethane water dispersion	2D fillers (graphene) in polyurethane water dispersion	1D fillers (MWCNTs) + 2D fillers (graphene) in polyurethane water dispersion
Formula	polyurethane (PU), MWCNTs	polyurethane (PU), graphene	polyurethane (PU), MWCNTs, graphene
Forms	Liquid	Liquid	Liquid
Volume	1 cc	1 cc	1 cc
Weight	1000 mg	1000 mg	1000 mg
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	300 - K	300 - K	300 - K
Pressure Range	- mbar	- mbar	- mbar
Magnetic field range	- T	- T	- T
Standard equipment	None	None	None
Special equipment	N/A	N/A	N/A

SAFETY

Prep lab needed	Yes	Yes	No
Sample Prep Hazards	no	-	no
Special equip. reqs	-	no	no
Sensitivity to air	No	No	No
Sensitivity to vapour	No	No	No
Experiment Hazards	no	no	no
Equipment Hazards	-	-	-
Biological hazards	no	no	no
Radioactive Hazards	no	no	no
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	Disposed by IS

Innovative sustainable inks for wearable sensors: analysis of filler morphology by TEM FEI

1. Background and Context

The rapid development of IoT and smart wearable devices has contributed to the enormous demand for smart flexible strain sensors. Unfortunately, the realization of smart textiles isn't always sustainable. [1] Thus, technological interests are growing in developing green composite materials as inks for conductive connections and piezo resistors by embedding nano carbons, such as 1D nanotubes, or 2D platelets. [2, 3] The advantages of polymeric nanocomposites with carbonaceous fillers are the low cost, lightweightness, and ease of dispersibility in environmentally friendly solvents. Another benefit of 1D and 2D nano carbons is their high aspect ratio, which ensures an efficient electrical percolation network at low loadings. These non-metal inks do not require a post-coating sintering step, which can reach damaging temperatures for common flexible polymer substrates such as cotton and cellulose. They create a stable conductive ink with time and, in some instances, are biocompatible, enabling easier processing and a more comprehensive range of applications. [4] Functionalizing standard fabrics with conductive materials is a popular approach. Methods like screen printing, dip-, spray-, blade-coating, and solution deposition of inks or pastes are efficient for large-area functionalization of textiles at ambient temperature and pressure. [5] Since signal transmission, electronic conduction, and thermal property depend on the integrity of the conductive paths, wearable interconnects require the stability of the electrical performance of the conductive textile upon deformation and washing. A green wearable conductor tunable and adaptable in terms of change in resistance with deformation would be ideal since it could satisfy divergent needs with a single solution, which would bring us closer to the demand of electronics. The challenge is coating the textile with sustainable conductive ink, realized using 1D, 2D carbonaceous filler and hybrid systems and controlling the formulation as well as the three-dimensional distribution to exploit the divergent needs of high and stable conductivity and piezo-resistivity using sustainable polymers and solvents made by mixing water and biodegradable surfactants such as polymers based on PVA.
Owing that, the aim of the proposal is to study, by using the instrument suite of IM@IT, the correlation between the filler spatial distribution and the coating parameters adopted by Bata University to deposit the inks on the textile substrate. The scope is to investigate how the aspect ratio and shape of the filler (1D and 2D filler) may affect the spatial distribution as consequence of the different technology adopted for the processing of the composites.

2. Proposed experiment

The carbonaceous fillers (1D: multiwalled carbon nanotubes, MWCNTs; 2D: graphene derivatives; mixtures of 1D/2D nanofillers) after drying from polyurethane water dispersion will be characterized in terms of morphology by the following technique:

- Transmission electron microscopy (TEM FEI) (Unit CNR-IPCB): to obtain info about filler morphology and interactions with the polyurethane phase. It is proposed to measure n. 3 different fillers (1D, 2D and hybrid systems). Hence, we request 1 day of beamtime which accounts also for setup time.

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In distinct proposals the sustainable Inks prepared by Bata University containing carbonaceous fillers (1D: multiwalled carbon nanotubes, MWCNTs; 2D: graphene derivatives; mixtures of 1D/2D nanofillers) applied on cotton substrates by using different deposition technologies (i.e. rod coaters, dip coating, spray coating), will be analyzed by Scanning Electron Microscopy (SEM FEI) to evaluate the coating morphology and by small and by wide angle X ray diffraction (SAXS/WAXD) to evaluate the coating structure and the filler orientation.

3. Summary of previous experimental proposals or characterisation

No previous experiments have been carried out on these samples

4. Justification of experimental time requested

We request the TEM FEI equipment to evaluate the morphology of the fillers and their interactions with the polyurethane phase.
We have requested 1 day of TEM FEI beam time, necessary for the analysis of the 3 above listed samples after discussion with the instrument scientist. The foreseen beam time accounts for set up and for the data collection on the samples.

References

[1] S. R. Joshi, S. Kumar, and S. Kim, “Ecofriendly Polymer–Graphene-Based Conductive Ink for Multifunctional Printed Electronics,” Adv.Mater. Technol., vol. 2201917, pp. 1–9, 2023, doi:10.1002/admt.2022019172.
[2] P. Cataldi et al., “A Green Electrically Conductive Textile with Tunable Piezoresistivity and Transiency,” Adv. Funct. Mater., 2023, doi:10.1002/adfm.2023015423.
[3] L. Jiang, H. Hong, and J. Hu, “Facile thermoplastic polyurethane-based multi-walled carbon nanotube ink for fabrication of screen-printed fabric electrodes of wearable e-textiles with high adhesion and resistance stability under large deformation,” Text. Res. J., vol. 91, no.21–22, pp. 2487–2499, 2021, doi: 10.1177/004051752110086134.
[4] S. Mondal, “Phase change materials for smart textiles – An overview,” Appl. Therm. Eng., vol. 28, no. 11–12, pp. 1536–1550, 2008, doi: 10.1016/j.applthermaleng.2007.08.0095.
[5] A. Tiwari and L. Uzun, “Advanced Functional Materials,” Adv. Funct. Mater., pp. 1–577, 2015, doi: 10.1002/9781118998977.

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Experiment Proposal

Experiment number GP2023056

Principal investigator	Professor Anita Grozdanov, Skopje University, MACEDONIA
Co-investigator	Dr Marino Lavorgna , CNR, ITALY
Co-investigator (*)	Dr Gennaro Gentile, IPCB CNR, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Analysis of filler spatial distribution by TEM FEI in polyninylalcohol/polyacrylic acid/MXenes nanocomposites
MRF Instrument	**TEM FEI** **Days requested:** 2
Access Route	Direct Access **Previous GP Number:** No
Science Areas	Engineering, Materials **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

The proposal aims to analyze the filler spatial distribution in polyninylalcohol/polyacrylic acid/MXenes nanocomposites by transmission electron microscopy using TEM FEI. In distinct proposals, we aim to perform both a structural analysis of the samples using the small and wide angle X-ray diffraction, using the SAXS WAXD and their morphological analysis by the scanning electron microscopy, using the SEM FEI, all operating at the IPCB-CNR Unit. This proposal is specifically addressed to get new insights in the exfoliation degree and the spatial distribution of 2D Mxenes nanofillers in highly amorphous polyninylalcohol (HAVOH)/polyacrylic acid (PAA) blends and to correlate the preparation approaches to the structure and morphology and to the final properties of the materials, with particular attention on their electrical conductivity, their EMI shielding and their gas barrier properties.

Publications -

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Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact Dr Gennaro Gentile, IPCB CNR, ITALY
MRF Instrument **TEM FEI** **Days Requested:** 2
Special requirements:

SAMPLE

Material	HAVOH + PAA (2 samples)	HAVOH + PAA + MXenes (2 samples)	-
Formula	polyvinylalcohol + polyacrylic acid	polyvinylalcohol + polyacrylic acid + mxenes	-
Forms	Solid	Solid	
Volume	0.1 cc	0.1 cc	
Weight	100 mg	100 mg	
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	300 - 300 K	300 - 300 K	-
Pressure Range	- MPa	- mbar	-
Magnetic field range	- T	- T	-
Standard equipment	None	None	-
Special equipment	N/A	N/A	-

SAFETY

Prep lab needed	No	No	-
Sample Prep Hazards	no	no	-
Special equip. reqs	N/A	no	-
Sensitivity to air	Yes	Yes	-
Sensitivity to vapour	Yes	Yes	-
Experiment Hazards	no	no	-
Equipment Hazards	-	-	-
Biological hazards	no	no	-
Radioactive Hazards	no	no	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	Disposed by IS	-

Analysis of filler spatial distribution by TEM FEI in polyninylalcohol/polyacrylic acid/MXenes nanocomposites

1. Background and Context

Polymer composites with nanoparticles as fillers are a growing group of materials with interesting properties for variety of application. Although numerous composites with nanofillers have been prepared and studied in last decade, mainly with carbon based fillers as carbon nanotubes or graphene, there are still challenges when new type of nanoparticles are discovered or synthetized.
MXenes are new types of 2D materials described first in the paper of Barsoum et al. in 2011 [1]. General formula for MXenes is Mn+1XnTx (n = 1–3), where M represents transition metals (Sc, Ti, Zr, Hf, V, Nb, Ta, Cr, Mo, etc.), X is carbon and/or nitrogen and Tx refer to different functional groups on the surface (e.g. OH, O, F, etc). Moreover, MXenes particles are highly electrically conductive. Shahzad et al. [2] have shown that flexible Ti3C2Tx films exhibit excellent electrical conductivity and electromagnetic interference (EMI) shielding capacity. Electrical conductivity reached 4600 S/cm, what originates from the high electron density of states near the Fermi level. In addition, due to their 2D morphology, MXenes are very promising to impart high gas barrier properties to polymer nanocomposites.
With the objective of preparing new nanocomposites with high gas barrier properties, high electrical conductivity and electromagnetic interference (EMI) shielding properties, in this activity new polymer blends filled with MXenes have been realized at variable composition. As a polymer matrix, an easy water soluble polvinylalcohol, high amorphous polyvinylalcohol (HAVOH) has been used [3], blended with polyacrylic acid (PAA) at variable molecular weight. Indeed, after thermal treatments, HAVOH/PAA blends are prone to give light crosslinking, with improvement of their stability to high relative humidity environments. HAVOH/PAA blends have been additivated with MXenes, in particular Ti3C2, prepared by etching the aluminium from the MAX phase Ti3AlC2.

2. Proposed experiment

The HAVOH/PAA nanocomposites at variable PAA molecular weight and HAVOH/PAA weight ratio, and containing 5 phr of MXenes have been realized by Skopje University - Faculty of Technology and Metallurgy, in cooperation with IPCB-CNR. In particular, HAVOH/PAA blends in water solutions have been prepared and additivated with the MXenes. Then films (about 50 micrometer thick) have been prepared by water casting. On the obtained films thermal treatments have been performed in oven to promote crosslinking between the HAVOH and the PAA phase. The following samples have been prepared for their characterization by TEM FEI and, in distinct proposals, with SEM FEI and SAXS/WAXD: 1) HAVOH neat; 2) HAVOH/PAA_4k 70/30; 3) HAVOH/PAA_4k 50/50; 4) HAVOH/PAA_240k 50/50; 5) HAVOH/PAA_4k 50/50 + 5phr Mxenes; 6) HAVOH/PAA_240k 50/50 + 5phr Mxenes.
The following characterization will be performed on these samples to evaluate the effect of the composition (HAVOH/PAA ratio, MW of PAA, MXenes additivation) on the filler spatial distribution in the composites:

- Transmission electron microcopy analysis (TEM FEI) (Unit IPCB CNR): to obtain info about the spatial distribution of MXenes in the composites. It is proposed to analyze n. 4 samples (2 above described nanocomposites filled with 5 phr Mxenes, by comparison the 2 HAVOH/PAAA blends not additivated with MXenes). TEM analysis will be performed in bright field mode. Samples will be prepared by ultramicrotomy.

In distinct proposals the same samples will be analyzed by Scanning Electron Microscopy (SEM FEI) and by small and wide angle X-ray diffraction (SAXS/WAXD), available at the IPCB CNR Unit.

3. Summary of previous experimental proposals or characterisation

No previous experiments have been carried out on these samples

4. Justification of experimental time requested

We request the TEM FEI equipment available at the IPCB CNR Unit to evaluate the effect of the MXenes additivation on the filler spatial distribution in the composites.
We have requested 2 days of TEM FEI beam time, necessary for the analysis of the 4 above listed samples after discussion with the instrument scientist. The foreseen beam time accounts set up and for the data collection on the samples.

References

[1] M. Naguib, M. Kurtoglu, V. Presser, J. Lu, J. Niu, M. Heon, L. Hultman, Y. Gogotsi, and M. W. Barsoum. Two-dimensional nanocrystals produced by exfoliation of Ti 3AlC 2. Adv. Mater. 23 (2011), p. 4248–4253.
[2] F. Shahzad, M. Alhabeb, C. B. Hatter, B. Anasori, S. M. Hong, C. M. Koo, and Y. Gogotsi. Electromagnetic interference shielding with 2D transition metal carbides (MXenes). Science 353 (2016), p. 1137–1140.
[3] C. Santillo, A.P. God, R.K.Donato, R.J.Espanhol Andrade, G.G. Buonocore, H. Xia, M. Lavorgna, A. Sorrentino. Tuning the structural and functional properties of HAVOH-based composites via ionic liquid tailoring of MWCNTs distribution. Composites Science and Technology, 207, 2021, 108742.

X-Ray diffractometer

X-Ray diffractometer

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Experiment Proposal

Experiment number GP2023046

Principal investigator	Dr Francesco Pintacuda, STMicrocelectronics, ITALY
Co-investigator (*)	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Roberto Senesi, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Carla Andreani, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Characterisation of the stress field in SiC MOSFET by means of X-Ray diffraction
MRF Instrument	**X-Ray diffractometer** **Days requested:** 4
Access Route	Direct Access **Previous GP Number:** -
Science Areas	Energy, Engineering, ICT, Materials, Physics **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	STMicroelectronics

Non-Technical Abstract

We propose to perform the stress field characterisation of SiC MOSFETs devices, already irradiated with fast neutron on the ChipIR beamline, using the X-Ray diffractometer instrument operating at the Medium Range Facility 1 (MRF1) of the University of Milano Bicocca Unit of ISIS@MACH ITALIA. Aim is to perform residual stress analysis of survived SiC MOSFETs from neutron-induced SEBs and compare results with independent measurements based on Raman spectroscopy and submitted as a separate proposal. Furthermore, the degree of damage by neutron induced SEBs failure on SiC occurred after the ChipIr neutron irradiation will be studied by means of X-Ray tomography data and results compared with scanning electron microscopy measurements. Both experiments have been puth forward by two separate proposals.
All the physical quantities inferred in this study have a direct impact on the understating of the mechanisms triggering SEBs in SiC power MOSFETs.

Publications

Pintacuda et al., Prototyping and characterization of radiation hardened SiC MOS structures, 2019 European Space Power Conference (ESPC).
F. Principato et al., Sensors 20 (2020), 3021; F. Principato et al., Sensors 21 (2021), 5627.
AJ Allen, MT Hutchings, CG Windsor, C Andreani, Neutron diffraction methods for the study of residual stress fields, Advances in Physics, 34, 445-473 (1985).

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY	
MRF Instrument	**X-Ray diffractometer**	**Days Requested:** 4
Special requirements:		

SAMPLE

Material	SiC	-	-
Formula	SiC	-	-
Forms	Solid		
Volume	0.004 cc		
Weight	12.84 mg		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Characterisation of the stress field in SiC MOSFET by means of X-Ray diffraction

1. Background and Context

Silicon carbide (SiC) is a IV–IV compound material with unique physical and chemical properties. The strong chemical bonding between Si and C atoms gives this material very high hardness, chemical inertness, and high thermal conductivity [1]. As a semiconductor, SiC exhibits a wide bandgap, high critical electric field strength, and high saturation drift velocity. Wide-bandgap semiconductors promise high tolerance to extreme environments, such as ionizing radiation, energetic particles, high and low temperatures. Both n- and p-type control across a wide doping range is relatively easy in SiC; this makes SiC exceptional among wide bandgap semiconductors. The ability of SiC to form silicon dioxide (SiO_2) as a native oxide is an important advantage for device fabrication. Because of these properties, SiC is a promising semiconductor for high-power and high-temperature electronics [2-4] both in space and at ground level. In comparison to silicon, SiC is a superior material thanks to its higher breakdown field and thermal conductivity. For terrestrial applications, power semiconductor devices like SiC metal–oxide–semiconductor field-effect transistor (SiC MOSFET) or insulated-gate bipolar transistors (IGBTs) [5] suffer of single-event burnout (SEB) failure when irradiated by high-energy neutrons [6] which constitute the 97% of the total [7] flux of high-energy particles impinging on such device at sea level. Experiments of neutron-induced SEBs in SiC MOSFET at high-voltage have shown the formation of cracks at the device surface [8] as shown in Figure 1.

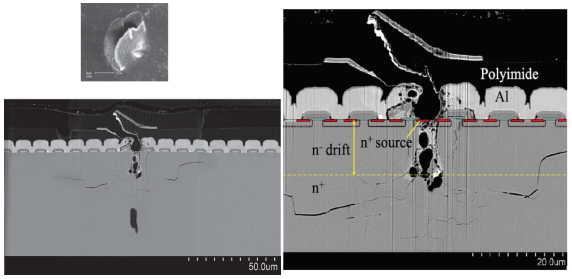

Figure 1: (Left panel) Cross-sectional SEM image of SEB damage for a SiC MOSFET. (Right panel) Magnified view of the damage and device structure [8].

We plan to perform non-destructive characterisations of damaged and survived SiC MOSFETs, following the neutron induced SEBs by fast neutron test at the ChipIr beamline, using scanning electron microscopy (SEM) and X-ray computed topography (XCT); in addition, the stress field [9] will be studied using the X-ray diffraction (XRD) and Raman spectroscopy. To this aim by four distinct proposals, we have requested the use of SEM with correlation AFM, XRD Tomography (for XCT), X-Ray Diffractometer, and the AFM Raman. All the instrumentation is available at IM@IT and operating at the Universities of Rome Tor Vergata and Milano Bicocca, and IPCB-CNR Units.

Aim of this proposal is to perform a residual stress analysis [9] of neutron induced SEBs in SiC MOSFETs using the high-resolution X-ray diffraction (XRD). The stress field will be also independently measured using the AFM Raman instrument, requested in a separate proposal, by using the relationship between the stress and the relative Raman frequency shift [8]. The SEBs damage in SIC MOSFETs will also be characterised through SEM and XCT. Two separate proposals have been submitted for using SEM-EDS (University of Rome Tor Vergata Unit) and the XRD Tomography (IPCB-CNR Unit).

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2. Proposed experiment

We aim to measure the stress field in n. 5 as manufactured and n. 5 survived SiC MOSFETs which not undergo to neutron induced SEBs during the test performed at the ChipIr beamline by means of X-ray diffraction (XRD) instrument located at the University of Milano Bicocca Unit. Such strains will be further measured by independent Raman spectroscopy measurements as reported here [8].

3. Justification of experimental time requested

Both as manufactured and survived SiC MOSFETs after neutron induced SEBs on ChipIr have dimensions of about 4mm x 5mm and a thickness of about 200 µm.

We aim to measure n. 5 as manufactured and n. 5 survived SiC MOSFETs by neutron induced SEBs using the X-Ray diffractometer with a Cu Kα radiation source. We request, after discussions with the instrument scientist, 4 days of instrument time including set-up and calibration time.

4. References

[1] Harris, G.L. (1995) Properties of Silicon Carbide, INSPEC.
[2] Davis, R.F., Kelner, G., Shur,M. et al. (1991) Proc. IEEE, 79, 677.
[3] Ivanov, P.A. and Chelnokov, V.E. (1992) Semicond. Sci. Technol., 7, 863.
[4] Morkoç, H., Strite, S., Gao, G.B. et al. (1994), J. Appl. Phys., 76, 1363.
[5] Pintacuda et al., 2019 European Space Power Conference (ESPC).
[6] Principato et al., Sensors 20 (2020), 3021;
[7] J. F. Ziegler, IBM J. Res. Dev. 40, 19 (1996).
[8] Yeong-Jae Yu et al., Cryst. Eng. Comm. 19 (2017), 6731.
[9] AJ Allen, MT Hutchings, CG Windsor, C Andreani, Advances in Physics, 34, 445-473 (1985).

XRD TOMOGRAPHY

XRD TOMOGRAPHY

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Experiment Proposal

Experiment number GP2023044

Principal investigator	Dr Francesco Pintacuda, STMicrocelectronics, ITALY
Co-investigator (*)	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Roberto Senesi, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Carla Andreani, University of Rome Tor Vergata, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Characterisation of the degree of damage by neutron induced single-event burnout failure in SiC MOSFET by means of X-Ray tomography
MRF Instrument	**XRD TOMOGRAPHY**
Days requested:	4
Access Route	Direct Access
Previous GP Number:	-
Science Areas	Energy, Engineering, ICT, Materials, Physics
DOI:	-
Sponsored Grant	None
Sponsor:	-
Grant Title	-
Grant Number:	-
Start Date	-
Finish Date:	-
Similar Submission?	-
Industrial Links	StMicroelectronics

Non-Technical Abstract

We propose to perform materials-to-circuits characterisation of SiC MOSFETs devices, already irradiated with fast neutron on the ChipIR beamline, by means of X-Ray tomography, operating at the IPCB-CNR Unit of IM@IT. Our aim is to access the degree of damage by neutron induced SEBs failure on SiC occurred after the ChipIr neutron irradiation by means of XCT data and compare these results with SEM images of the damage using the SEM with correlative AFM operating at the University of Rome Tor Vergata Unit, which we requested in a separate proposal. In two other proposals, we will investigate the residual stress field of survived SiC MOSFETs from neutron induced SEBs, by means of high resolution X-ray diffraction and AFM Raman, located at the University of Milano Bicocca and at the University of Rome Tor Vergata Units, respectively. All the physical quantities inferred in this study have a direct impact on the understating of the mechanisms trigger SEBs in SiC power MOSFETs.

Publications

Pintacuda et al., Prototyping and characterization of radiation hardened SiC MOS structures, 2019 European Space Power Conference (ESPC).
F. Principato et al., Sensors 20 (2020), 3021; F. Principato et al., Sensors 21 (2021), 5627
AJ Allen, MT Hutchings, CG Windsor, C Andreani, Neutron diffraction methods for the study of residual stress fields, Advances in Physics, 34, 445-473 (1985)

ISIS neutron and muon source	**E-platform:** No
Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY		
MRF Instrument	**XRD TOMOGRAPHY**	**Days Requested:**	4
Special requirements:			

SAMPLE

Material	SiC	-	-
Formula	SiC	-	-
Forms	Solid		
Volume	0.004 cc		
Weight	12.84 mg		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	293 - K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	None	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Characterisation of the degree of damage by neutron induced single-event burnout failure in SiC MOSFET by means of X-Ray tomography

1. Background and Context

Silicon carbide (SiC) is a IV–IV compound material with unique physical and chemical properties. The strong chemical bonding between Si and C atoms gives this material very high hardness, chemical inertness, and high thermal conductivity [1]. As a semiconductor, SiC exhibits a wide bandgap, high critical electric field strength, and high saturation drift velocity. Wide-bandgap semiconductors promise high tolerance to extreme environments, such as ionizing radiation, energetic particles, high and low temperatures. Both n- and p-type control across a wide doping range is relatively easy in SiC; this makes SiC exceptional among wide bandgap semiconductors. The ability of SiC to form silicon dioxide (SiO_2) as a native oxide is an important advantage for device fabrication. Because of these properties, SiC is a promising semiconductor for high-power and high-temperature electronics [2-4] both in space and at ground level. In comparison to silicon, SiC is a superior material thanks to its higher breakdown field and thermal conductivity. For terrestrial applications, power semiconductor devices like SiC metal–oxide–semiconductor field-effect transistor (SiC MOSFET) or insulated-gate bipolar transistors (IGBTs) [5] suffer of single-event burnout (SEB) failure when irradiated by high-energy neutrons [6] which constitute the 97% of the total [7] flux of high-energy particles impinging on such device at sea level. Experiments of neutron-induced SEBs in SiC MOSFET at high-voltage have shown the formation of cracks at the device surface [8] as shown in Figure 1.

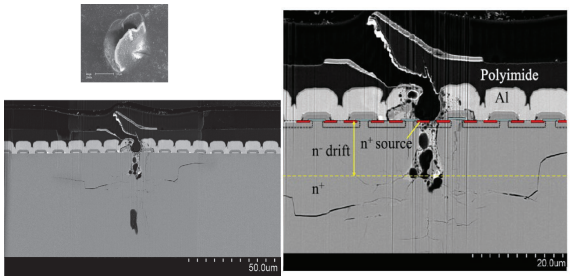

Figure 1: (Left panel) Cross-sectional SEM image of SEB damage for a SiC MOSFET. (Right panel) Magnified view of the damage and device structure [8].

We plan to perform non-destructive characterisations of damaged and survived SiC MOSFETs, following the neutron induced SEBs by fast neutron test at the ChipIr beamline, using scanning electron microscopy (SEM) and X-ray computed topography (XCT); in addition, the stress field [9] will be studied using the X-ray diffraction (XRD) and Raman spectroscopy. To this aim by four distinct proposals, we have requested the use of SEM with correlation AFM, XRD Tomography (for XCT), X-Ray Diffractometer, and the AFM Raman. All the instrumentation available at IM@IT and operating at the Universities of Rome Tor Vergata and Milano Bicocca, and IPCB-CNR Units.

In the present proposal we wish to study the SEBs damage in SIC MOSFETs through a series of XCT scans. The 2D and 3D reconstructions from XCT data will provide non-destructive evaluations

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of the damage for the semiconductors under investigation. The XCT scans will also provide information on the presence of micro-burning or multiple burning, which are not easily observed with microscope after decapsulation of the package. On the other hand, a comparison of XCT results with independent SEM analysis, considered the gold standard procedure for evaluation of damage in semiconductors, we enable us to perform an experimental benchmark. Furthermore, the residual stress analysis will be assessed by high-resolution X-ray diffraction (XRD) and Raman spectroscopy using the same procedure as reported in [8].

2. Proposed experiment

We aim to measure a total of n. 5 damaged and n. 5 survived SiC MOSFETs by means of X-ray computed topography for samples which undergo to neutron induced SEBs during a previous test performed at the ChipIr beamline, ISIS neutron and muon source. The 3D reconstruction of SEB damaged SiC MOSFET extract from XCT data will be compared with cross-sectional SEM image of the same sample, like the one shown in Figure 1, and used here to benchmark XCT results.

3. Justification of experimental time requested

The damaged and survived SiC MOSFETs after neutron induced SEBs on ChipIr have dimensions of about 4mm x 5mm and a thickness of about 200 µm. We aim to measure n. 5 damaged and 5 survived SiC MOSFETs using a field of view of 10.64 mm x 10.64 mm, pixel size of 5.2 µm, and about 1570 projections to fulfil the Niquist-Shannon sampling theorem. With an exposure time per projection of 5 s, each tomography will last about 2 hours. Hence, after discussion with the instrument scientist, we request 4 days of instrument time including set-up and calibration time.

4. References

[1] Harris, G.L. (1995) Properties of Silicon Carbide, INSPEC.
[2] Davis, R.F., Kelner, G., Shur,M. et al. (1991) Proc. IEEE, 79, 677.
[3] Ivanov, P.A. and Chelnokov, V.E. (1992) Semicond. Sci. Technol., 7, 863.
[4] Morkoç, H., Strite, S., Gao, G.B. et al. (1994), J. Appl. Phys., 76, 1363.
[5] Pintacuda et al., 2019 European Space Power Conference (ESPC).
[6] Principato et al., Sensors 20 (2020), 3021;
[7] J. F. Ziegler, IBM J. Res. Dev. 40, 19 (1996).
[8] Yeong-Jae Yu et al., Cryst. Eng. Comm. 19 (2017), 6731.
[9] AJ Allen, MT Hutchings, CG Windsor, C Andreani, Advances in Physics, 34, 445-473 (1985).

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Experiment Proposal

Experiment number GP2023050

Principal investigator	Dr Diego Sbardella, IRCCS Fondazione G.B. Bietti, ITALY
Co-investigator (*)	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Alessio Bocedi, University of Rome, Tor Vergata, ITALY
Co-investigator	Dr Giovanni Romanelli, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Laura Fazi, University of Rome Tor Vergata, ITALY
Co-investigator	Professor Roberto Senesi, University of Rome Tor Vergata, ITALY
Co-investigator	Dr Luigi Ambrosio, National Research Council, ITALY
Co-investigator	Dr Tommaso Rossi, IRCCS Fondazione Bietti ONLUS, ITALY
Co-investigator	
Experiment title	Characterisation of surgically removed human vitreous samples by X-Ray tomography
MRF Instrument	**XRD TOMOGRAPHY** **Days requested:** 3
Access Route	Direct Access **Previous GP Number:** No
Science Areas	Biology and Bio-materials, Medicine, Physics **DOI:** -
Sponsored Grant	Yes **Sponsor:** Other
Grant Title	Profiling of physical and proteomics parameters of vitreous body in retinal detachment **Grant Number:** 5*1000 to IRCCS Fondazione Bietti
Start Date	01/03/2023 **Finish Date:** 01/03/2025
Similar Submission?	-
Industrial Links	BVI Medical
Non-Technical Abstract	Rhegmatogenous Retinal Detachment (RD) is a severe eye disease that occurs when the retina becomes detached from the Retinal Pigment Epithelium due to the presence of retinal tears or holes. The gold standard treatment of RD is vitrectomy, that is the removal of part of the vitreous humor (VH) using vitreous cutters. A major question still unanswered, is whether there is a relation between the morphology (dimensions) of VH fragments generated by cutters when set with different frequency parameters. The proponents aim to study by high resolution microscopy measurements (scanning electron microscopy (SEM-AFM), Transmission electron microscopy (TEM), X-ray computed tomography (XCT)) the morphology of VH fragments surgically isolated from RD patients. In the present proposal we wish to measure the morphology of 6 distinct VH fragments (3 samples for each vitreous cutter frequency, i.e., 5000 CPM and 20000 CPM) using XCT on the XRD Tomography instrument.
Publications	T. Rossi et al., Retina 34 (2014), 1896-904. T. Rossi et al., Invest Ophthalmol Vis Sci. 12 (2014), 8289-94. T. Rossi et al., Translational Vision Science & Technology 11 (2022), 29.

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact Dr Triestino Minniti, University of Rome Tor Vergata, ITALY
MRF Instrument **XRD TOMOGRAPHY** **Days Requested:** 3
Special requirements:

SAMPLE

Material	Humor vitreous	-	-
Formula	-	-	-
Forms	Liquid		
Volume	0.002 ml		
Weight	2 mg		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	- K	-	-
Pressure Range	- mbar	-	-
Magnetic field range	- T	-	-
Standard equipment	-	-	-
Special equipment	-	-	-

SAFETY

Prep lab needed	Yes	-	-
Sample Prep Hazards	-	-	-
Special equip. reqs	-	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	-	-	-
Equipment Hazards	-	-	-
Biological hazards	-	-	-
Radioactive Hazards	-	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Characterisation of surgically removed human vitreous samples by X-Ray tomography

1. Background and Context

Rhegmatogenous Retinal Detachment (RD) is a severe eye disease [1] that occurs when the retina becomes detached from the Retinal Pigment Epithelium (RPE) due to the presence of retinal tears or holes. The gold standard treatment of RD is vitrectomy, that is the removal of part of the vitreous humor (VH), a gel-like fluid that shapes the eye globe, using vitreous-cutters. About 15-20% of all RDs relapse within the first 6 months through a process called Proliferative Vitreo-Retinopathy [2] (PVR), which is characterized by inflammation, collagen deposition and retinal contraction. PVR is highly invalidating and often accompanied by sight loss, thus carrying a huge burden for the quality of life and for social and economic costs. All vitreous cutters base on the mechanism of a reciprocating blade moving within a hollow cylinder in a proximal-to-distal fashion, with cut-rates comprised between 1,000 and 20,000 cuts per minute. Given the miniaturization of retinal surgery instrumentation, cutters have evolved from 20G (0.9 mm out diameter in section) to 25G (0.5mm) and even 27G (0.4mm), making the internal fluidics even more challenging and requiring high aspiration vacuum up to 650 mmHg to win the hydraulic resistance of the highly viscous human vitreous material. High suction and blade motion applied to the collagen mesh of vitreous exert traction on the retina especially when the peripheral “vitreous base” is removed and more so when the retina is mobile during retinal detachment surgery. For this reason, the intraoperative creation of iatrogenic retinal tears and the amount of traction exerted on the retina causing further damage and possibly giving rise to Proliferative Vitreoretinopathy remains and important issue, largely unresolved. A major question still unanswered, is whether there is a relation between intraoperative retinal traction, PVR onset and the morphology (dimensions) of VH fragments (mostly collagen and proteoglycan) generated by cutters when set with different frequency (cuts per minute, CPM) parameters or whether these parameters have no effects on VH fragmentation [3]. The contribution of turbulent vitreous fluidics at the cutter port to the consistency of vitreous fragment dimensions is also a matter of speculation. If different fragments are produced, then the tensile force generated over the retina layer (which adheres to VH) as well as the mechanical stress, which is definitely responsible for intra-operative retinal traction and iatrogenic break formation and likely relate with PVR onset, may be influenced by cutter parameters. This proposal fits into a wider multidisciplinary research program of IRCCS Fondazione Bietti (IFB), a main Italian clinical center for the study and research in Ophthalmology, granted by the Ministry of Health (Profiling of physical and proteomics parameters of vitreous body in retinal detachment) and supported by industries. The proponents aim to study by high resolution microscopy measurements (scanning electron microscopy (SEM-AFM), Transmission electron microscopy (TEM), X-ray computed tomography (XCT)) the morphology of VH fragments surgically isolated from RD patients and its protein composition that can help predicting the proportion of those patients who most likely will develop PVR. To this end, we wish to use the SEM with correlative AFM, TEM FEI, XRD Tomography instruments. All the instrumentation is available at IM@IT and operating at the Universities of Rome Tor Vergata and IPCB-CNR Units. It is worth mentioning that VH fragments, generated by vitreous cutters used at two frequencies, i.e., 5000 and 20000 CPM, will be isolated from the same patient eye during two surgical phases, using an established surgical procedure and Good Medical Practices [3]. Documentation on the ethical issues associated to the use of VH fragments will be provided upon request.

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2. Proposed experiment for XCT

In the present proposal we wish to measure the morphology of 6 distinct VH fragments (3 samples for each vitreous cutter frequency, i.e., 5000 CPM and 20000 CPM) using XCT on the XRD Tomography instrument available at the IPCB-CNR Unit of IM@IT. Results from reconstruction 2D and 3D XCT data will be compared with results obtained from SEM/AFM and TEM measurements which have been proposed by the proponents in other two separated proposals.

3. Summary of previous experimental proposals or characterisation

The performance of vitreous cutters, by means of hydraulic resistance posed by cut VH during aspiration, has been investigated for frequencies < 12000 CPM. Furthermore, cutter blade action determines instantaneous flow rate fluctuation that interferes significantly with VH aspiration posing a possible risk of inadvertent retinal entrapment [3,4].
Nevertheless, since VH fragments generate are supposed to be > 10 µm, current biochemical and molecular biology techniques cannot easily be applied to address the aim this proposal deals with.

4. Justification of experimental time requested for XCT

VH fragments (6 in total, 3 samples for each vitreous cutter frequency, i.e., 5000 CPM and 20000 CPM) will be XCT scan using a field of view of 2.66 mm x 2.66 mm, pixel size of 1.3 µm, and about 3100 projections to fulfil the Niquist-Shannon sampling theorem. With an exposure time per projection of 5 s, each tomography will last about 4 hours. Hence, after discussion with the instrument scientist, we request 3 days of instrument time including set-up and calibration time.

5. References

[1] T. Schick et al., Klin Monbl Augenheilkd. 12 (2020), pp. 1479-1491.

[2] S. Yang et al., Discov Med. 110 (2015), 207.

[3] T. Rossi et al., Retina 34 (2014), 1896-904.

[4] T. Rossi et al., Invest Ophthalmol Vis Sci. 12 (2014), 8289-94.

[5] S. Pastor-Idoate et al., PLoS ONE 12 (2017), e0173883.

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Experiment Proposal

Experiment number GP2023080

Principal investigator (*)	Dr Giuseppe Paladini, University of Catania, ITALY
Co-investigator	Professor Valentina Venuti, Università di Messina, ITALY
Co-investigator	Dr Francesco Caridi, Università degli Studi di Messina, ITALY
Co-investigator	Professor Vincenza Crupi, University of Messina, ITALY
Co-investigator	Professor Paola Cardiano, Università degli Studi di Messina, ITALY
Co-investigator	Professor Gabriele Lando, University of Messina, ITALY
Co-investigator	Professor Domenico Majolino, Università degli Studi di Messina, ITALY
Co-investigator	
Co-investigator	
Experiment title	X-ray diffraction tomography to study the effect of the application of phosphate-based coatings on the emission of ionizing radiations of lithotypes used as building materials
MRF Instrument	**XRD TOMOGRAPHY** **Days requested:** 4
Access Route	Direct Access **Previous GP Number:** No
Science Areas	Cultural Heritage, Environment, Materials, Physics **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	-
Non-Technical Abstract	The assessment of the radiological risk to individuals from both external and internal exposure to ionizing radiation in stone materials, along with the development of restoration intervention protocols that incorporate radiation protection measures, is emerging as a critically important and widely discussed issue in the field of material and conservation science. Here we propose to investigate the correlations between the microstructural changes, based on the evaluation of the 3D spatial distribution of the mineralogical phases prior and after treatment with a specific phosphate-based consolidant, and the emission of ionizing radiations for two different porous lithotypes (i.e., granodiorite and limestone), taking advantage of the XRD Tomography at ISIS@MACH ITALIA.
Publications	G. Romanelli et al. Neutron-Enhanced Information on the Laboratory Characterization of Ancient Egyptian Leathers: Hydration and Preservation Status. Information 2022, 13, 467

ISIS neutron and muon source	**E-platform:** No
Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Sample record sheet

Principal contact	Dr Giuseppe Paladini, University of Catania, ITALY
MRF Instrument	**XRD TOMOGRAPHY** **Days Requested:** 4
Special requirements:	

SAMPLE

Material	granodiorite	limestone	-
Formula	(Ca,Na)(Al,Si)4O8+SiO2+K(Fe,Mg)3(AlSi3O10)(OH)2; (Ca,Na)(Al,Si)4O8+SiO2+K(Fe,Mg)3(AlSi3O10)(OH)2+phosphate-based consolidant	CaCO3; CaCO3+phosphate-based consolidant	-
Forms	Solid	Solid	
Volume	12.57 cc	12.57 cc	
Weight	33.31 g	25.14 g	
Container or substrate	No sample holder, container or substrate	No sample holder, container or substrate	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	room temperature - K	room temperature - K	-
Pressure Range	room pressure - mbar	room pressure - mbar	-
Magnetic field range	No magnetic field - T	No magnetic field - T	-
Standard equipment	None	None	-
Special equipment	No need for special equipment	No need for special equipment	-

SAFETY

Prep lab needed	No	No	-
Sample Prep Hazards	No other hazards associated with the sample preparation	No other hazards associated with the sample preparation	-
Special equip. reqs	No special equipment requirements	No special equipment requirements	-
Sensitivity to air	No	No	-
Sensitivity to vapour	No	No	-
Experiment Hazards	No other hazards associated with experiment	No other hazards associated with experiment	-
Equipment Hazards	-	-	-
Biological hazards	No biological hazards associated with the sample	No biological hazards associated with the sample	-
Radioactive Hazards	Radioactive hazards associated with the sample will be evaluated prior the XRD Tomography analysis	Radioactive hazards associated with the sample will be evaluated prior the XRD Tomography analysis	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Removed By User	Removed By User	-

1. Background and Context

The application of organic/inorganic products in the consolidation of lithic materials of historical-artistic interest is currently a major topic in the field conservation and material science. Such approach is aimed at reducing the impact of aging and decay phenomena [1-3], in the view of retrieving the original stone mechanical strength and grain cohesion after exposition to natural weathering. In the last 30 years, the evaluation of the radiological risk for human beings due to external and internal exposure to ionizing radiations in stone materials, as well as to the design of restoration intervention protocols based on radiation protection measures, is becoming a hot-topic of paramount relevance. Building materials, including stones used for historical sites and monuments, contain natural radionuclides (such as ^{226}Ra, ^{232}Th and ^{40}K) which can determine significant exposure to gamma rays and contribute to indoor radon concentrations, thus representing a serious radiological concern regarding the public health.

As it is well known, the rate of ageing in such materials is strongly affected by several physico-chemical properties including the porosity, permeability, texture, and the mineralogical composition/distribution within the 3D crystal structure. Accordingly, also the emission of ionizing radiations, being originating from specific isotope-bearing minerals, and how such minerals are distributed within the structure, are likely to experience variations as one of the aforementioned parameters varies. In this sense, the application of different inorganic products, already largely employed to boost the stone performance against natural weathering phenomena, is also expected to change the radioactive-related outcomes of the stone, following the establishment of physico-mechanical interactions occurring between a specific consolidant product and the chosen substrate, which yield to newly-formed phases accompanied by an overall 3D phase re-organization.

It is worth of note that the knowledge of such aspects furnishes novel insights for the development of optimized procedures for radiological and conservation purposes.

2. Proposed experiment

Here we propose to use XRD Tomography at ISIS@MACH ITALIA to map in 3D the crystallization products formed upon the establishment of physico-mechanical interactions occurring between a specific inorganic phosphate-based consolidant, and two different porous lithotypes of the Calabrian territory, southern Italy, *i.e.*, granodiorite and limestone, widely employed as building materials.

The experiment aims at addressing the following points:

- Evaluation of the microstructural changes in terms of porous distribution and connectivity prior and after treatment.
- Assessment of the variations in the 3D spatial distribution of the observed phases of interest within the sample volume, with particular regard to the evaluation of the depth-resolved spatial profiles of each crystallization minerals (from the treated surface) prior and after treatment. Notably, this information is not available from conventional XRD technique.
- Both the aforementioned aspects will be then correlated to the expected changes in the emission of ionizing radiations, *i.e.* radon exhalation, which will be measured prior and after treatment through Closed Chamber Method (CCM) [4,5], respectively.

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Starting from the XRD Tomography dataset, the reconstruction images associated to selected crystalline phases of interest will be achieved by plotting the integrated area of a “marker” peak of the XRD pattern as a function of all pixels of the array. Ad-hoc software will be used to proper analyse and visualize the obtained data.

3. Summary of previous experimental proposals or characterisation

No previous experimental proposals or characterization are available.

4. Justification of experimental time requested

We intend to make use of the RIGAKU Nano3DX instrument at ISIS@MACH ITALIA to get simultaneous knowledge of the mineralogical phases present within the investigated lithotypes and their 3D distribution prior and after treatment, not accessible through conventional XRD and/or μ–CT techniques.

4 days of experimental time are requested.

Measurements will be performed on **4 cylinder fragments** sampled from the investigated lithotypes, untreated and treated with a phosphate-based consolidant. In particular, considering a 180° rotation and the vertical beam size, each dataset would require **~ 9 h,** for a total measuring time of **~ 36 h**. We expect at most **~ 1 h** for the setup time for each sample.

5. References

1. L. Randazzo et al, J. Cult. Herit. 2020, 46, 31–41.
2. V. Crupi et al, Constr. Build. Mater. 2018, 166, 464-471.
3. M. Baglioni et al, Molecules 2021, 26, 3967.
4. L. Zhang et al, J. Rad. Prot. 2012, 32, 315–323.
5. A.I. Amasi et al, J. Environ. Earth Sci. 2015, 5, 57–63.

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Experiment Proposal

Experiment number GP2023090

Principal investigator	Dr Giulia Marcucci, ISIS Neutron and Muon Source, UNITED_KINGDOM
Co-investigator (*)	Dr Daniela Di Martino, University of Milano Bicocca, ITALY
Co-investigator	Dr Massimiliano Clemenza, INFN, ITALY
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Co-investigator	
Experiment title	Unlocking the structure and composition of a historical silver coin using XRD Tomography in combination with Muon and Neutron Techniques
MRF Instrument	**XRD TOMOGRAPHY** **Days requested:** 1
Access Route	Direct Access **Previous GP Number:** No
Science Areas	Cultural Heritage, Materials **DOI:** -
Sponsored Grant	None **Sponsor:** -
Grant Title	- **Grant Number:** -
Start Date	- **Finish Date:** -
Similar Submission?	-
Industrial Links	-

Non-Technical Abstract

The INFN has funded the CHNET_TANDEM collaboration aimed at the development of a non-destructive analytical technique using negative muon beams. As part of this effort, an 18th-century Portuguese coin was used to compare the muon technique with other methods, in collaboration with the IAEA. The muon beam technique revealed the coin elemental composition and depth profile, showing a possible silver enrichment. The main objectives of this proposal are to further investigate the coin composition and structure with a non-destructive approach exploiting the complementarity of information of a multi-technique protocol. We plan to use XRD tomography to achieve a 3D map of the coin metallic phase composition and distribution and help understand its production technology. Overall, this project combines cutting-edge scientific techniques with heritage science to unravel the secrets of a historical coin and to assess the results obtained with previous muons and neutrons analyses.

Publications -

ISIS neutron and muon source **E-platform:** No

Instruments	**Days Requested:**
Access Route	**Previous RB Number:**
Science Areas	**DOI:**
Sponsored Grant	**Sponsor:**
Grant Title	**Grant Number:**
Start Date	**Finish Date:**
Similar Submission?	
Industrial Links	

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Science and Technology Facilities Council
ISIS Neutron and Muon Source

Sample record sheet

Principal contact Dr Daniela Di Martino, University of Milano Bicocca, ITALY
MRF Instrument **XRD TOMOGRAPHY** **Days Requested:** 1
Special requirements:

SAMPLE

Material	Copper-Silver coin	-	-
Formula	Cu, Ag	-	-
Forms	Solid		
Volume	0.22 cc		
Weight	2 g		
Container or substrate	none	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	Room Temperature - Room temperature K	-	-
Pressure Range	no applied pressure - no applied pressure mbar	-	-
Magnetic field range	no applied magnetic field - no applied magnetic field T	-	-
Standard equipment	None	-	-
Special equipment	none	-	-

SAFETY

Prep lab needed	No	-	-
Sample Prep Hazards	no	-	-
Special equip. reqs	none	-	-
Sensitivity to air	No	-	-
Sensitivity to vapour	No	-	-
Experiment Hazards	no	-	-
Equipment Hazards	-	-	-
Biological hazards	no	-	-
Radioactive Hazards	no	-	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Disposed by IS	-	-

Background and Context

The INFN has funded the CHNET_TANDEM collaboration aimed at the development of a non-destructive analytical technique for Cultural Heritage using negative muon beams. Proof-of-principle experiments using negative muons for elemental analysis were conducted on the Port 4 beamline of the ISIS Neutron and Muon Source from April 2015, including calibration on standard materials [1] and feasibility tests on at many archaeological artefacts, such as “bronze age” artefacts (CHNET_TANDEM INFN experiment), Roman Empire coins and ancient swords to name but a few [2-4].

As part of this project, an 18th-century Portuguese coin has been used for a round-robin comparison in participation to the IAEA (International Atomic Energy Agency) Coordinated Research Project (CRP) F11021 [5] “Enhancing Nuclear Analytical Techniques to Meet the Needs of Forensic Science” with the Muonic Atom X-ray Spectroscopy performed at PORT4 of the ISIS Neutron and Muon Source. This CRP allowed introducing, in the IAEA framework, the use of negative muons as a reference technique for non-destructive elementary characterization measurements for unique samples, such as those of cultural heritage or those measured for forensic reasons.

The application of the Muonic Atom X-ray spectroscopy allowed to perform an elemental depth profile of the coin, determining the Ag/Cu ratio from the surface to the inner core of the sample and therefore disclosing a slight silver enrichment, as shown in Fig. 1. Preliminary XRF measurements were carried out and main results for composition are listed as follow: Ag: 91.2%, Cu: 3.7%, Cl: 1.2% Au: =0.7%, Fe: 0.5%, Pb: 0.2% plus other minor components. We can also confirm that, on the surface, the coin is silver-based, with copper as a minor alloy constituent and other elements between 0.2 -1%. The main interest of this proposal is to cross-check this relatively new nuclear investigation with consolidated non-invasive techniques to reveal the exact composition (surface and bulk) and homogeneity along the depth profile and to expand the punctual elemental analysis to the phase composition representative of the entire sample, to also determine the production process, whether by minting or casting.

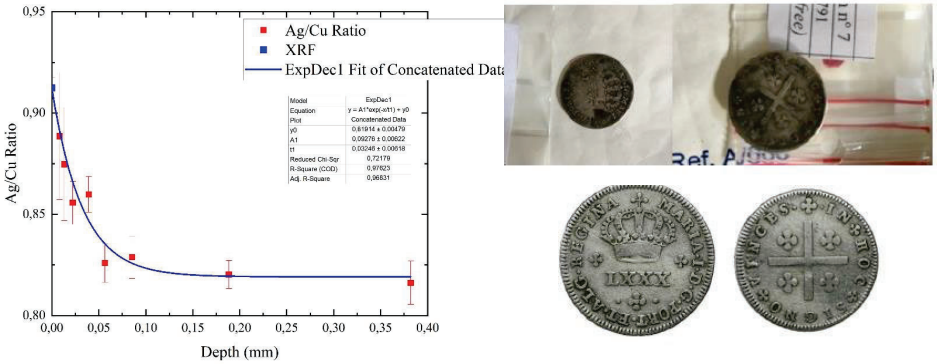

Fig. 1 On the left: Depth profile of the Ag/Cu ratio obtained through Muonic Atom X-ray spectroscopy measurements at the ISIS Neutron and Muon Source. On the right: (top) Front and rear of the Portuguese coins, 80 reis, coinage under Maria I (2 cm in diameter and 0.7 mm in thickness). (bottom) A Portuguese coin, 80 reis (coinage under Maria I) from a recent auction [6].

Sample description

A Portuguese coin, dating to the late 18th century will be investigated and is part of the round-robin comparison in CRP F11021. This coin is shown in Fig. 1. During the 18th century, the Portuguese monetary unit was the reis. The etymology comes from “*rei*” (literally meaning king), the plural being “*reis*”. Different types of coinage can be found and are either copper-, silver- or gold-based. The Portuguese coinage consisted of 5, 10, 20 and 40 reis pieces in either copper or bronze; a silver coinage of 60, 80, 120, 200 and 400 reis and gold coinage of 480, 800, 1,200, 1,600, 3,200 and 6,400 reis. Our coin has inscribed on it “*LXXX*” and is therefore 80 reis. In addition, the name of the queen (Queen Maria I who ruled from 1777 to 1799). A picture of the sample (front and rear) is shown below.

Proposed experiment

The primary objectives of this study are as follows:

i. Phase composition and distribution analysis: Perform XRD tomography to determine the precise composition of the coin, including the ratio of silver to copper and the presence of any alloying elements to cross-check the Muonic Atom X-ray Spectroscopy results;
ii. Structural Composition: Investigate the structure and potential alterations in the coin's phases structure by WAXD analysis caused by historical factors such as copper depletion and minting techniques.
iii. Historical Context: Correlate the findings with historical records and numismatic data to provide insights into the coin's origin, purpose, and significance.

We propose to use XRD tomography to accomplish our research objectives, considering also this three-fold motivation: 1) the sample is an ancient artefact, and non-destructive analyses should be used to preserve its uniqueness; 2) no cleaning will be performed on the sample– we will be able to perform the measurement also in the presence of corrosion layers or deposits, suggested by XRF measurements; 3) the sample is bulky, and we want to infer not only the mean bulk composition but the depth profile. In this regard, another proposal will be submitted for the same sample for Small and Wide Angle X-ray Diffraction measurements to accomplish the structural composition investigation. These two experiments will be useful in complementing the information collected through neutron diffraction and neutron resonance capture analysis carried out at the INES beamline at ISIS (RB2010534, “Combination of neutron based techniques to derive the composition of an 18th-century coin”)."

We would like to underline that this round-robin is on a real sample. Other measurements have been done on standards; however, the study of a real case is mandatory when these techniques are to be used on real specimens and historical artefacts are always not homogeneous and present different issues in comparison to a standard sample.

The use of the XRD tomography technique will involve obtaining a three-dimensional map of the coin's metallic phase distribution, which can provide detailed information about its composition and manufacturing technology: we aim to measure n. 1 sample using a field of view of 5 mm x 5 mm, pixel size of 2.5 µm, with an exposure time per projection of 5 s, each tomography will last about 4 hours. Hence, we request 1 day of beamtime which accounts also for setup time.

References

[1] A.D. Hillier et al, Microchemical Journal. Vol. 125, March 2016, Pages 203–207.
[2] M. Clemenza et al. Nucl. Instrum. Meth. Phys. Res. A . 936, (2019), Pages 27-28
[3] A.D. Hillier, A. M. Pollard, A. Wilson, D. MckPaul, et al in prep, see expt report RB 1520462.
[4] A. I. Wilson 'The metal supply of the Roman Empire', in E. Papi and B. Scardigli (eds), Supplying Rome.
[5]A. Fajgelj et al The IAEA’s Analytical Quality Control Services (AQCS) Programme on Intercomparison Runs and Reference Materials. IAEASM-344/3. 1997
[6] See for example “Lot 337 Auction 23” where 4 of these coins were estimated at 40 euros https://numismaticaleiloes.bidinside.com/en/lot/335/portugal-d-pedro-ii-to-d-maria-i-4-/