ISIS@MACH ITALIA

Science and Technology Facilities Council
ISIS Neutron and Muon Source

Experiment Proposal

Experiment number GP2023032

Principal investigator (*) Dr Marcello Marelli, CNR, ITALY
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Experiment title	PVP/CNTs Nanofiber composite: uncovering the inner architecture	
MRF Instrument	**FIB-SEM GAIA 3**	**Days requested:** 2
Access Route	Direct Access	**Previous GP Number:** -
Science Areas	Chemistry, Environment, Materials	**DOI:** -
Sponsored Grant	None	**Sponsor:** -
Grant Title	-	**Grant Number:** -
Start Date	-	**Finish Date:** -
Similar Submission?	-	
Industrial Links	-	

Non-Technical Abstract Polyvinylpyrrolidone (PVP) is an inexpensive and eco-friendly polymer soluble in water, alcohol, and other polar solvents. The novel chemical-physical stability given to the nanofibrous allows new applications as sensors and electrochemical devices. Using carbon nanotubes (CNTs) during the synthesis leads to novel electric properties retaining the original fiber morphology, making indistinguishable a treated fiber from a not-treated one. We want to investigate the structural changes of these systems, disclosing the interior design of the PVP/CNTs. We propose to use FIB-SEM analysis as a unique tool able to disclose those peculiar structures retaining the spatial information. We will collect imaging, from micro- to nano-scale, and form the external to the inner sections. The microfabrication capability of FIB-SEM will be exploited to prepare TEM specimens for further investigation.

Publications

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IM@IT E-platform: No

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Sample record sheet

Principal contact Dr Marcello Marelli, CNR, ITALY
MRF Instrument **FIB-SEM GAIA 3** **Days Requested:** 2
Special requirements:

SAMPLE

Material	PVP nanofiber loaded whit Carbon nanotubes	Basic PVP nanofiber (reference material)	-
Formula	(C6H9NO)n / C (as Carbon Nanotubes)	(C6H9NO)n	-
Forms	Solid	Solid	
Volume	cc	cc	
Weight	mg	mg	
Container or substrate	nanofiber deposited onto electrode, silicon wafer or PTFE foil	nanofiber deposited onto electrode, silicon wafer or PTFE foil	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

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

SAFETY

Prep lab needed	Yes	Yes	-
Sample Prep Hazards	NO	NO	-
Special equip. reqs	As suggested by the person in charge of the FIB-SEM, an ultramicrotome could be used	-	-
Sensitivity to air	No	No	-
Sensitivity to vapour	No	No	-
Experiment Hazards	NO	NO	-
Equipment Hazards	-	-	-
Biological hazards	NO	NO	-
Radioactive Hazards	NO	NO	-
Additional Hazards	-	-	-
Additional Details	-	-	-
Sample will be	Returned to user by instrument scientist (when inactive)	Returned to user by instrument scientist (when inactive)	-

Science Case

1. Background and Context

Polyvinylpyrrolidone (PVP) is an inexpensive and eco-friendly polymer soluble in water, alcohol, and other polar solvents. Commercially it is used as a coating agent but is also used in inks, hair products, paints, pesticides, and toothpaste and as a stabilizer in food. PVP fibers could be manufactured utilizing the electrospinning technique and thanks to a post-treatment under UV light, the polymer chains could crosslink within the fibers to an insoluble new material. The novel chemical-physical stability given to the nanofibrous allows new applications as sensors and electrochemical devices. The possibility to load into the fiber different fillers and/or chemicals could impinge on improved capability as conductivity or selectivity towards specific analyte. From a morphological and structural point of view, these processes are not evident and are hidden in interior architecture. For example, the use of carbon nanotubes (CNTs) as an additive during the synthesis leads to novel electric properties but the original fiber morphology is retained making indistinguishable a treated fiber from a not-treated one. The structural change is visible only inside the fiber and structural variation leads to different electrical properties. These materials will be used as active membranes in chemical sensors, but specific electrical requirements are needed. Nanofibers as sensor material are also promising thanks to the specific response to target molecules even in presence of similar ones.

In this work, we want to exploit the structural changes of these systems, disclosing the interior design of the PVP/CNTs through advanced nanofabrication and imaging analysis. This project is framed in a more wide scientific collaboration between CNR-SCITEC Milano (and in detail by Dr. Marcello Marelli, PI for this proposal) and the CNR-IIA Monterotondo-Rome. The fibers were synthesized at CNR-IIA and part of the characterization is performed in Milano. The characterization part is not supported by a dedicated grant, personal funding at CNR-SCITEC is available for this kind of scientific collaboration used to establish and consolidate an active working network.

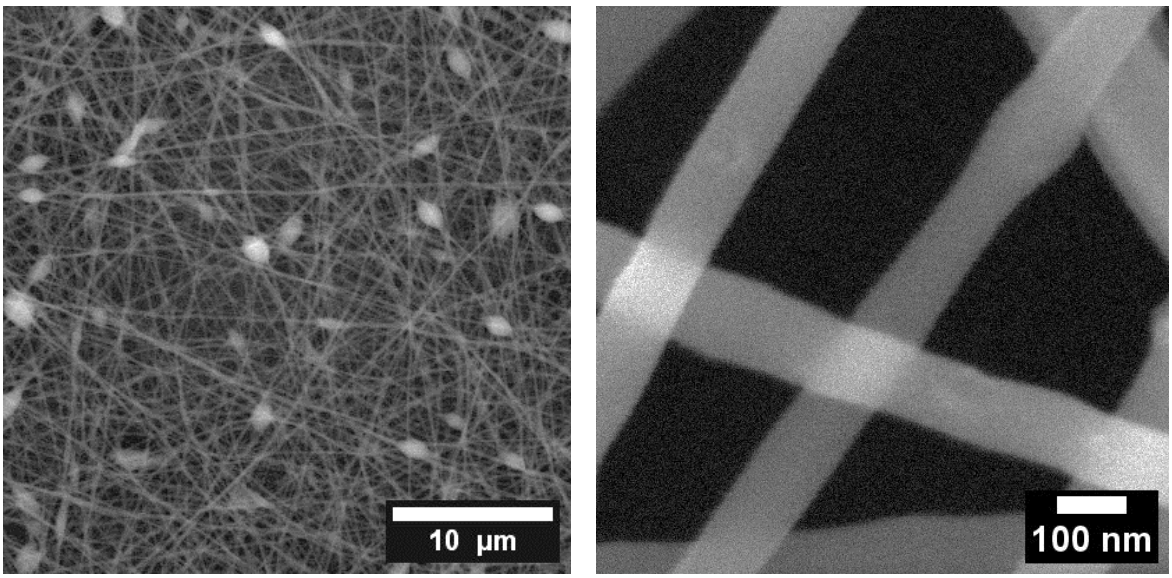
2. Proposed experiment

The proposed experiment aims to disclose the fine structure of PVP nanofiber-based electrochemical sensors at several levels, from the micrometric scale down to the nanometer. CNTs-doped fibers are conductive but morphologically identical to the undoped. If the shape and external appearance are retained, the interior design change with the presence of the CNTs. We want to use the ion gun of FIB-SEM to cut and dig the fibers in order to reveal the inner part and its content. We want to highlight the CNTs location and/or a preferential spatial orientation. We want to use the SEM to image samples in a large range of magnification, from low to high resolution, and prepare some samples suitable for TEM measurement. This preparation part will be made on some fibers deposited onto a TEM grid or producing a thin lamella suitable for TEM analysis. All the proposed analysis could be done by exploiting the unique and high-performance capability of the FIB-SEM TESCAN GAIA 3, the instrument available in the ISIS@MACH ITALIA labs. The undistorted ultra-low magnification imaging next to the extreme high-resolution will enable travel through the sample at all the scale levels. Finally, the FIB gun will be the precise scalpel to cut the material with high accuracy. A good alternative to preparing a TEM lamella could be offered by the use of an ultramicrotome (also available in the labs) as suggested by the person in charge of the instrument. TEM analysis will be performed at different labs after the sample preparation.

The sample batch is composed of two selected samples, obtained at different conditions, with different composition and related to a different activity. A priority list will be established to use the machine time at its best. Specimen will be prepared onto conductive glass/electrode, on TEM grids, and on soft materials (as PTFE foils), in accordance with general procedures shared with the ICCOM labs. Data will be analyzed and managed by Dr. Marcello Marelli, using commercial software, already in use at CNR-SCITEC labs (ImageJ, GMS, Origin and others).

3. Summary of previous experimental proposals or characterisation

Preliminary studies on PVP fibers with a SEM (ESEM Philips XL30) show good stability under the electron beam (20kV) in high vacuum mode, without any metallization or sample pre-treatment. Fibers appear well distributed onto the surface, whit a thickness of 100 nm ca. The Proposal PI, Dr. Marelli, is a senior user at ISTeM Labs and recorded the SEM analysis.



Representative SEM micrographs of PVP/CNTs systems at different magnifications.

4. Justification of experimental proposals request

I request access to the FIB-SEM TESCAN GAIA 3 instrument since offers a state of art analysis both for high-resolution imaging and FIB manufacturing. The peculiar structure of our nanofiber requires an advanced and well-planned analysis strategy that can be accomplished only by high-performance instrumentation. Moreover, the full characterization of these samples could be accomplished by exploiting the FIB capability to prepare thin lamellas/specimens for TEM analysis. Finally, the possibility to prepare a sample with an ultramicrotome available at the same lab is a value added to the proposal.

I request 2 days of machine time. The first day will be focused on a single sample (e.g. PVP fiber loaded whit CNTs) to tune the FIB procedure according to the material properties and collect images at several magnifications. The second day will be focused to prepare TEM lamellas by FIB or by ultramicrotome following the procedure tuned on the first day. The analysis of a second sample (optional, e.g. PVP fibers only) could be performed at the end of the second day (experience on the previous sample could speed up the analysis time).