ISIS@MACH ITALIA

Science and Technology Facilities Council
ISIS Neutron and Muon Source

Experiment Proposal

Experiment number GP2023009

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Experiment title	SAXD and WAXD analysis of polyninylalcohol/MXenes nanocomposites
MRF Instrument	**SAXS Xenocs Xeuss**
Access Route	Direct Access
Science Areas	Materials
Sponsored Grant	None
Grant Title	-
Start Date	-
Similar Submission?	-
Industrial Links	-

Days requested: 3
Previous GP Number: -
DOI: -
Sponsor: -
Grant Number: -
Finish Date: -

Non-Technical Abstract

The objective of this proposal is the SAXD and WAXD analysis of nanocomposite samples prepared by the Slovak Academy of Science - Polymer Institute, (SAS-PI) in cooperation with the Institute for Polymers Composites and Biomaterials of the National Resrearch Council of Italy (IPCB CNR).

Samples are constituted by a commercial highly amorphous polyvinylalcohol (HAVOH) and contain variable amounts of single walled carbon nanotubes (SWCNT) and MXenes (MX). By comparison, also nannocomposites containing only SWCNT have been realized. The samples have been already characterized to evaluate their morphology (by TEM analysis), their thermal properties (by DSC) and their elecrical conductivity.

Publications -

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IM@IT E-platform: No

Instruments	
Access Route	
Science Areas	
Sponsored Grant	
Grant Title	
Start Date	
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Sample record sheet

Principal contact Dr Gennaro Gentile, IPCB CNR, ITALY
MRF Instrument **SAXS Xenocs Xeuss** **Days Requested:** 3
Special requirements:

SAMPLE

Material	HAVOH 3% MXenes	HAVOH 3% SWCNT	HAVOH 3% SWCNT 3% MXenes
Formula	polyvinylalcohol, Mxenes (Tin+1Cn)	polyvinylalcohol, SWCNT	polyvinylalcohol, SWCNT, Mxenes (Tin+1Cn)
Forms	Solid	Solid	Solid
Volume	0.025 cc	0.025 cc	0.025 cc
Weight	25 g	25 mg	25 mg
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	300 - 300 K	300 - 300 K	300 - 300 K
Pressure Range	0.1 - 0.1 MPa	0.1 - 0.1 MPa	0.1 - 0.1 MPa
Magnetic field range	- T	- T	- T
Standard equipment	None	None	None
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 of by instrument scientist	Disposed of by instrument scientist	Disposed of by instrument scientist

Science Case

Proposal title:
SAXD and WAXD analysis of polyninylalcohol/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 T_x refer to different functional groups on the surface (e.g. OH, O, F, etc). Moreover, MXenes particles are highly electrically conducting. Shahzad et al. [2] have shown that flexible $Ti_3C_2T_x$ 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, making MXene metallic in nature.
With the objective of preparing new nanocomposites with high electrical conductivity and electromagnetic interference (EMI) shielding properties, in this activity new polyvinylalcohol/MXenes nanocomposites and polyvinylalcohol/single wall carbon nanotubes (SWCNT)/MXenes nanocomposites have been realized at variable composition. As a polymer matrix, an easy water soluble polvinylalcohol, high amorphous polyvinylalcohol (HAVOH) has been used [3], and additivated with commercial SWCNT and MXenes. The most used MXene has been used as a nanofiller in this activity, namely Ti_3C_2, prepared by etching the aluminium from MAX phase Ti_3AlC_2.

2. Proposed experiment

The objective of the experiment is the structural characterization of nanocomposites by SXD/WAXD analysis. 3 samples have been selected for the experiment, namely HAVOH + 3% SWCTN, HAVOH + 3% MXenes, HAVOH + 3% SWCNT + 3% MXENES, to evaluate the effect of SWCNT on the structure of the nanocomposites containing MXenes. If possible, the analysis of neat HAVOH (not listed amongst the samples to be analyzed) would be useful.

3. Summary of previous beamtime or characterisation

The samples have been already characterized to evaluate:

- Their electrical conductivity
- Their EMI shielding properties
- Their morphology (with particular attention to the distribution of MXenes in the polymer matrix) by TEM analysis
- Their thermal properties (by differential scanning calorimetry)

Very interesting results in terms of electrical conductivity and EMI shielding have been obtained. SWCNT and MXenes bot induce an increase of the melting temperature of the HAVOH matrix. As shown in the next figure, the presence of SWCNT hinders the alignment of MXenes along the film direction.

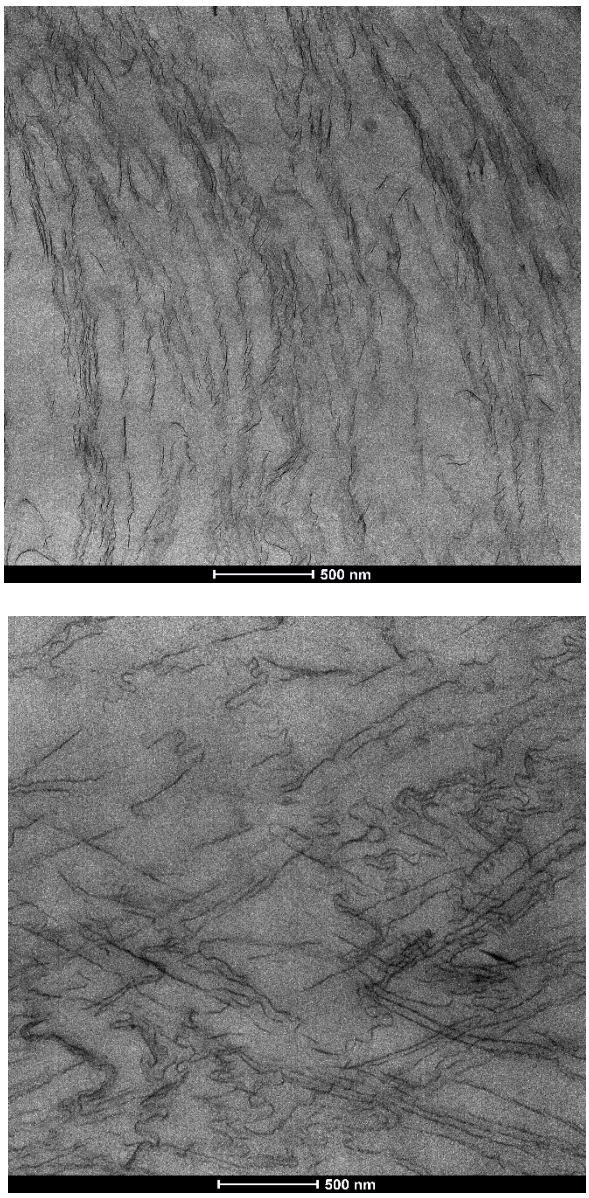

Fig. 1. TEM images of HAVOH + 3% MXenes (up) and HAVOH + 3% SWCNT + 3%MXenes (down)

4. Justification of beamtime request

Structural analysis of the nanocomposites by SAXD/WAXD will allow to better elucidate the effect of SWCNT on the exfoliation distribution of MXenes in the HAVOH matrix.

We have considered 6 hours of experiment time for each sample (total 4 samples).

5. 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