ISIS@MACH ITALIA

Science and Technology Facilities Council
ISIS Neutron and Muon Source

Experiment Proposal

Experiment number GP2023004

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Experiment title	FIB-SEM characterization of CNT-based surface composites for sensors applications
MRF Instrument	**FIB-SEM GAIA 3**
Access Route	Direct Access
Science Areas	Materials, Medicine
Sponsored Grant	None
Grant Title	-
Start Date	-
Similar Submission?	-
Industrial Links	-

Days requested: 2
Previous GP Number: GP2022008
DOI: -
Sponsor: -
Grant Number: -
Finish Date: -

Non-Technical Abstract

Surface composite made of polymer films and carbon nanotubes (CNTs) are a promising class of composite materials used in innovative stretchable sensors. By choosing the polymer substrate, one can obtain different electrical and mechanical behaviours, making them suitable to different applications. To better understand the grafting mechanisms of the CNTs on the polymer substrates, many investigations have been made with conventional SEM and CRM, to clarify the interaction between the CNTs below the polymer surfaces. We propose to complement preliminary characterizations of two CNT surface composites, each one based on a different polymer substrate representing two different classes of polymers (i.e. thermoplastic and thermosetting), with a SEM equipped with a FIB, to investigate the composite layer by layer along its thickness, to unveil the penetration depth of CNT inside the polymer matrix.

Publications

Fazi, L., et al. "Stretchable conductors made of single wall carbon nanotubes self-grafted on polymer films." Journal of Physics: Conference Series. 1548, 1, 2020

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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	

Sample record sheet

Principal contact Mr Valerio Scacco, University of Rome Tor Vergata, ITALY
MRF Instrument **FIB-SEM GAIA 3** **Days Requested:** 2
Special requirements:

SAMPLE

Material	polymer CNT composite	-	-
Formula	polymer CNT composite	-	-
Forms	Solid		
Volume	5 cc		
Weight	mg		
Container or substrate	-	-	-
Storage Requirements	-	-	-

SAMPLE ENVIROMENT

Temperature Range	300 - 300 K	-	-
Pressure Range	0.1 - 0.1 MPa	-	-
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 of by instrument scientist	-	-

Scientific Background

Surface composite made of polymer films and carbon nanotubes (CNTs) are a promising class of composite materials that can be used in innovative stretchable sensors [1]. In fact, they couple the elasticity and plasticity of polymer films with the electrical properties of CNTs. By properly choosing the polymer substrate, one can obtain different electrical and mechanical behaviours, making them suitable to different applications ranging from biomedical devices to actuators [2]. Beyond the choice of the material, the composite materials need to be characterized in terms of the grafting mechanisms of the CNTs on the polymer substrates, because from them follow the electrical and mechanical properties [3,4].

To better understand such mechanisms, many investigations have been made with microscopy techniques such as conventional Scanning Electron Microscopy and Confocal Raman Microscopy to try to clarify the interaction between the CNTs below the polymer surfaces. Unfortunately, only partial results have been obtained: conventional SEM is a surface technique and Confocal Raman Microscopy has a too low resolution to unveil the penetration depth of CNTs with the needed precision.

For these reasons, this proposal has the purpose to complement such preliminary characterizations of two CNT surface composites, each one based on a different polymer substrate representing two different classes of polymers (i.e. thermoplastic and thermosetting ones), with a SEM equipped with a Focused Ion Beam that allows to investigate the composite layer by layer along its thickness, allowing to unveil the penetration depth of CNT inside the polymer matrix using the FIB_SEM instrument of the Medium Range Facility 1 - FOURDIM.

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 (GP2022008), have produced some major estimation of the grafting mechanisms and the penetration depths of CNTs inside the polymer. In particular, SEM images (shown in Figure 1), have provided evidences suggesting the “soaking” of CNTs bundles in the polymeric substrate. We performed a cold cut in liquid nitrogen in order to leave unchanged the structure of the composite interface, but this method does not give us information on the grafting dynamics of carbon nanotubes in the polymer nor on their penetration depth.

With Confocal Raman Microscopy some estimations of the depth of penetration of the CNTs inside the polymers have been obtained but the resolution allowed by this technique (from 5 to 15 micron, depending on the depth), does not allow to obtain unambiguous and trustworthy results, especially with substrates with thickness around 20-30 microns.

Proposed Experiment

We propose to perform a Focused Ion Beam enhanced Scanning Electron Micrscopy experiment using the FIB-SEM Gaia 3 instrument at the MRF1 – FOURDIM of the CSGI – University of Florence Unit of ISIS@MACH ITALIA. The FIB will allow to progressively removing very thin layers (around tens of nanometers) of the composite, allowing a layer by layer characterization of the composite. In addition, such instrument is equipped with a Field-Emission emitter, allowing very high resolution during SEM scan, allowing to properly visualize CNTs, whose contrast with respect to the substrate is very low with conventional, filament-emitter SEM. To facilitate measurement by ensuring a homogeneous surface, the sample is immersed in low vapour pressure hardening resin. To perform this experiment, we request 3 days of the FIB_SEM instrument at the MRF1 – FOURDIM facility.

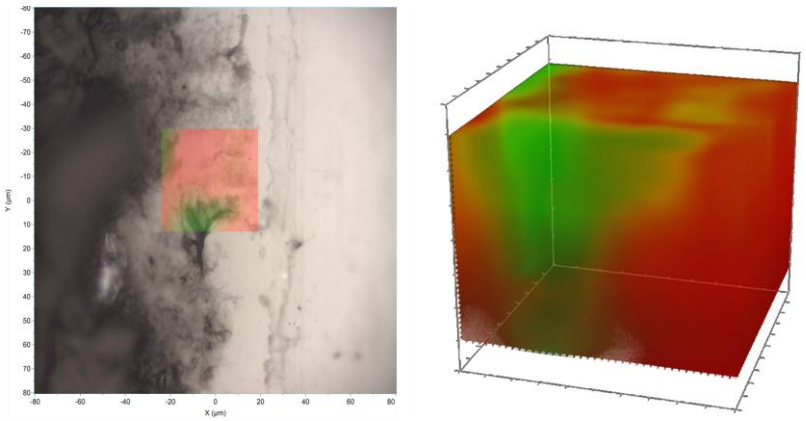

Figure 1 – Raman confocal 3D map that show the carbon nanotube penetration (green signal) into the polymer substrate (red signal).

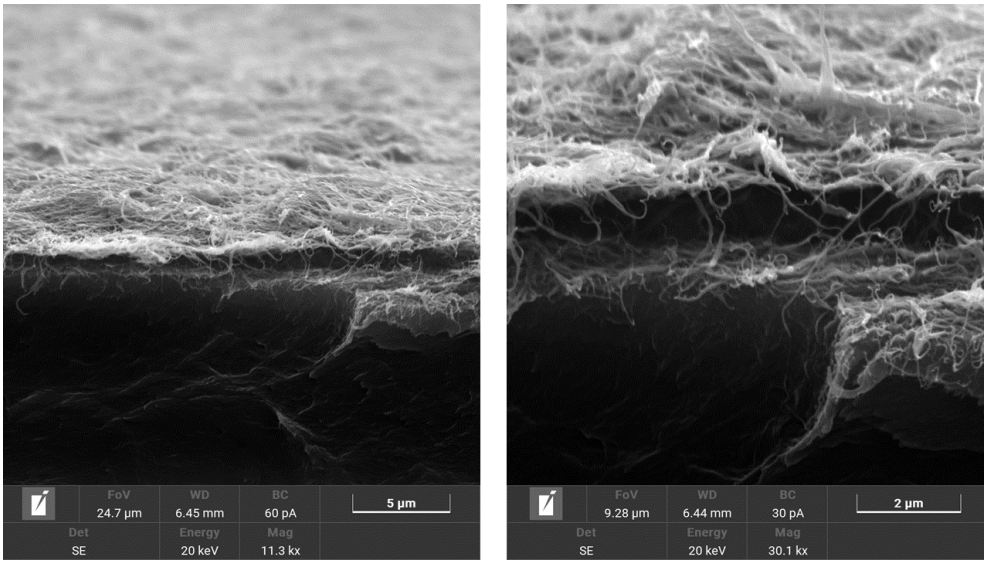

Figure 2 – SEM images of the interface after cold cutting in liquid nitrogen: on the top the carbon nanotube layer and on the bottom the polymer substrate. The left image shows the SWCNTs anchor sites into the substrate.

References

[1] Morales, Piero, et al. "Self-grafting carbon nanotubes on polymers for stretchable electronics." *The European Physical Journal Plus* 133.6 (2018): 1-11.

[2] Pavone, Luigi, et al. "Chronic neural interfacing with cerebral cortex using single-walled carbon nanotube-polymer grids." *Journal of neural engineering* 17.3 (2020): 036032.

[3] Fazi, Laura, et al. "Carbon Nanotube-Based Stretchable Hybrid Material Film for Electronic Devices and Applications." *Journal of Nanoscience and Nanotechnology* 20.7 (2020): 4549-4556.

[4] Fazi, L., et al. "Stretchable conductors made of single wall carbon nanotubes self-grafted on polymer films." *Journal of Physics: Conference Series*. Vol. 1548. No. 1. IOP Publishing, 2020.