

Experiment Proposal

Experiment number GP2023008

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Co-investigator		
Experiment title	TEM characterization of CNT-based surface composites for sensors applications	
MRF Instrument	High Resolution TEM	Days requested: 2
Access Route	Direct Access	Previous GP Number: -
Science Areas	Materials, Medicine	DOI: -
Sponsored Grant	None	Sponsor: -
Grant Title	-	Grant Number: -
Start Date	-	Finish Date: -
Similar Submission?	-	
Industrial Links	-	
Non-Technical Abstract	<p>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. By properly choosing the polymer substrate, one can obtain different electrical and mechanical behaviours, making them suitable to different applications. To understand the grafting mechanisms of the CNTs on the polymer substrates, many investigations have been made with conventional SEM and Confocal Raman Microscopy, in order to clarify the interaction between the CNTs. For this reason, we propose to complement such characterizations of two CNT surface composites, each one based on a different polymer substrate representing two different classes of polymers (i.e. thermoplastic thermosetting ones), with the High Resolution TEM, to obtain a sample estimate of the depth distance of the nanotubes.</p>	

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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ISIS neutron and muon source

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 Mr Valerio Scacco, University of Rome Tor Vergata, ITALY
MRF Instrument **High Resolution TEM** **Days Requested: 2**
Special requirements:

SAMPLE

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

SAMPLE ENVIROMENT

Temperature Range	300 - 300 K	-	-
Pressure Range	0.1 - 0.1 MPa	-	-
Magnetic field range	0 - 0 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 the possibility of slicing the sample with a microtome for TEM analysis, allowing to unveil the penetration depth of CNT inside the polymer matrix using the TEM 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 Transmission Scanning Microscopy experiment using the High Resolution TEM instrument at the MRF1 – FOURDIM of the CSGI – University of Florence Unit of ISIS@MACH ITALIA. By immersing the sample in low vapour pressure hardening resin, it is possible to slice the sample with a microtome and analyze this section frontally using TEM. In this way it is possible to obtain a sample estimate of the depth distance of the nanotubes in the polymer substrate. To perform this experiment, we request 3 days of the TEM instrument



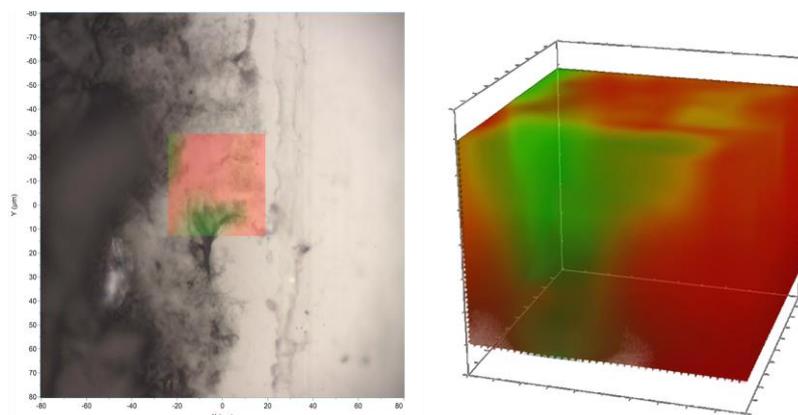


Figure 1 – Raman confocal 3D map that show the carbon nanotube penetration (green signal) into the polymer substrate (red signal).

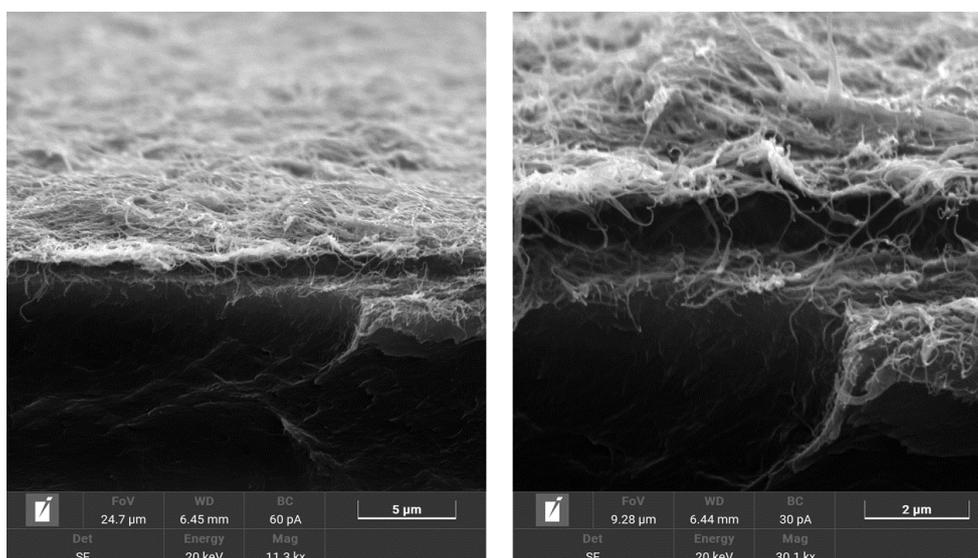


Figure 2 – SEM images of the interface after cold cutting in liquid nitrogen: on the top the carbon nanotube layer and on the bottom the polymer substrate. The left image shows the SWCNTs anchor sites into the substrate.

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.

