Experiment Proposal

Experiment number GP2024012

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Experiment title	Characterisation of carbon nanotube network structure in nanocomposites resolved by TEM measurement		
MRF Instrument	**TEM FEI**	**Days requested:**	6
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

Single wall carbon nanotube (SWCNT) reinforced polymers are next-generation, high-performance, multifunctional materials with a wide array of promising applications. The successful introduction of such materials is hampered by the lack of a quantitative understanding of process-structure-property relationships. These relationships can be developed only through the detailed characterization of the nanoscale reinforcement morphology within the embedding medium.

Here, we aim to reveal by high resolution transmission electron microscopy (TEM FEI) operating at the IPCB-CNR Unit of IM@IT the detailed morphology of SWCNT with have been rooted within a polypropylene and polyisoprene substrates.

Publications -

Sample record sheet

Principal contact	Dr Triestino Minniti, University of Rome Tor Vergata, ITALY		
MRF Instrument	**TEM FEI**	**Days Requested:** 6	
Special requirements:			

SAMPLE

Material	Poly-propylene and carbon nanotubes	-	-
Formula	-	-	-
Forms	Solid		
Volume	6e-9 cc		
Weight	5e-9 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	-	-

Characterisation of carbon nanotube network structure in nanocomposites resolved by TEM measurement

1. Background and Context

Polymer-carbon nanotube composites combine the elastic and plastic mechanical properties of polymers with the electrical conduction character of graphitic nanostructures. “As grown” single wall carbon nanotube (SWCNT) bundles and coils, which exhibit their own elasticity, were found to self-graft efficiently on many different polymeric films, rooting into the polymer bulk in a surprisingly stable combination and endowing the composite with a variable electrical conduction [1]. Some of these self-assembled surface composites proved to be useful in the accomplishment of signal capturing microdevices that recorded very efficiently months of cortical level electrical activity from selected areas of brains of laboratory rats that were freely moving in laboratory cages and feeding with a fully normal behavior [2]. Many more application fields can be envisaged in sensor technology and particularly in medicine and prosthetics, depending on the different composite properties that can be achieved using different polymeric matrices. We have observed for example a very different grafting behavior between composites based on thermoplastic polymers and those based on thermosetting elastomers, the former showing diffusion of the SWCNT limited to few micrometers, with a surface adhering thick conductive layer of SWCNT, while the latter allow much deeper diffusion, with consequently higher electrical resistivity [3]. Confocal Micro-Raman spectroscopy allows for identification of spectral features of SWCNT that have drifted into the elastomer more than 100 micrometers. However, 3D maps of the Raman signal suggest that their non uniform bundled structure is often maintained while limited spatial resolution does not allow detection of single ropes or small coils of nanotubes. We therefore lack information on the details of the distribution of the conductive part of the composite, which is important in the development of ultrathin implantable devices. For this reason, by high resolution transmission electron microscopy (TEM), the proponents aim at studying the detailed morphology of SWCNT which have been rooted into polymeric substrates of the two different types mentioned before: thermoplastic polymers (e.g., poly-ethylene (PE), whose microcrystalline structure seems to hinder the diffusion of nanotube bundles into the core, leaving a thick surface shell rich in nanotubes, and therefore displaying a fairly high electrical conductivity; and thermosetting elastomers whose amorphous structure on the contrary allows deep penetration of nanotubes, that appear fully embedded into the polymeric structure and electrically insulated from each other, so that the overall conductivity is about two orders of magnitude lower [3]. Aiming at application to biomedical devices, the best candidates of the two types of composites are poly-ethylene and poly-dimethyl-siloxane (PDMS), both of them being well known for their full biocompatibility, and also representing good examples of highly plastic and highly elastic materials to cover a wide range of application areas. Although less suitable to medical application, poly-propylene (PP) and poly-isoprene (PI) are also good candidates to study the CNT diffusion in thermoplastics and thermosettings. Cross-sections for such samples will be prepared using an ultramicrotome sectioning tool to allow the transmission of electrons through the sample with minimal distortion. Both the TEM microscope and the ultramicrotome equipment are available at the IPCB-CNR Unit of IM@IT. After TEM measurements, the same thin section of both samples will be further studied on the lateral sides by scanning electron microscopy (SEM) to complement TEM data.

Furthermore, the 2D morphology characterization of the samples will be used as input for a full comprehensive proposal for studying such polymer-carbon nanotube composites in 3D by means of advanced electron tomography technique available at the electron Bio-Imaging Centre (eBIC), Diamond Light Source, United Kingdom.

2. Proposed experiment for TEM

In the present proposal we wish to measure the morphology of 2 distinct sample made of SWCNT which are self-grafted onto PE (or PP) and onto PDMS (or PI) substrates. Cross-sections for both samples will be prepared using ultramicrotome sectioning tool to allow the transmission of electrons through the sample with minimal distortion. Both the TEM FEI and the ultramicrotome instruments are available at the IPCB-CNR Unit of IM@IT. After TEM measurements, the same thin section of both samples will be further studied on the lateral sides by scanning electron microscopy (SEM correlative AFM) operating at the UniTOV Unit to complement TEM data.

3. Justification of experimental time requested for TEM

Two distinct polymer-carbon nanotube composite samples will be measured by TEM scans using a field of view and magnification which depends on the dimensions of SWCNT bundles and coils rooting into the polymer bulk of the sample. We predict n. 10 images for each sample. Hence, after discussion with the instrument scientist, we request 6 days of instrument time including the time-consuming sample preparation, set-up and calibration time.

4. References

[1] P. Morales, S. Moyanova, L. Pavone, L. Fazi, D. Mirabile Gattia, B. Rapone, A. Gaglione, R. Senesi *Self-grafting carbon nanotubes on polymers for stretchable electronics* The European Physical Journal Plus, 1 June 2018 (Materials Science, Engineering Physics) DOI:10.1140/EPJP/I2018-12040-0

[2] L. Pavone, S. Moyanova, F. Mastroiacovo, L. Fazi, C. Busceti, A. Gaglione, K. Martinello, S.Fucile, D. Bucci, A. Prioriello, F. Nicoletti, F. Fornai, P. Morales, R. Senesi; *Chronic neural interfacing with cerebral cortex using single-walled carbon nanotube-polymer grids.* Journal of Neural Engineering. 2020 Jul 3;17(3):036032. doi: 10.1088/1741-2552/ab98db.

[3] L. Fazi , C. Andreani , C. D'Ottavi , L. Duranti , P. Morales , E. Preziosi , A. Prioriello , G. Romanelli , V. Scacco, R. Senesi, S. Licoccia *Characterization of Conductive Carbon Nanotubes/Polymer Composites for Stretchable Sensors and Transducers* Molecules 2023 Feb 13; 28(4):1764. doi: 10.3390/molecules28041764