DENSE

Development and Electromagnetic Characterization of Nano Structured Carbon Based Polymer CompositEs

S. Bellucci (Resp. INFN), R. Baldini (Tecn.), L. Coderoni (Bors.), G. Giannini (Ass. Ric.), F. Micciulla (Ass. Ric.), I. Sacco (Bors.)

External collaborating Institutions: Univ. Roma La Sapienza, Università degli Studi di SALERNO, Università degli Studi di PALERMO.

We participate as a partner (the INFN unit) to the PROGRAMMA DI RICERCA SCIENTIFICA DI RILEVANTE INTERESSE NAZIONALE (PRIN) of the Italian MINISTERO DELL'ISTRUZIONE DELL'UNIVERSITà E DELLA RICERCA DIREZIONE GENERALE DELLA RICERCA. DENSE has a duration of 36 months and started its activities on 1st January 2010. The consortium binds together three Universities (with four different Departments) and one Research Organization.

Project objectives:

The main objectives of the project DENSE are the realization and characterization of new multifunctional polymeric nanocomposites employing as nanofillers graphene and multiphase systems clay/Carbon Nanotubes (CNTs). Such nanocomposites, based on thermosetting (epoxy) resin are intended for industrial
applications, such as aeronautics, automotive, electronics, where remarkable thermal and mechanical properties and, at the same time, tailored and controlled electromagnetic (EM) performances are required. The design and realization of such nanocomposites will be performed from the tailoring of the electromagnetic performances.
In fact, although several interesting results had been obtained in the realization of new and improved nanocomposites, the researchers are still far from obtaining a precise control of the desired nanostructure and thus optimize the materials performances. To reach such a goal it is required to act on both the choice of the most appropriate nanofillers and the fabrication techniques. The present research proposal pursues both aspects, by deepening the knowledge of the basic chemical and physical mechanisms and enhancing the technological competences required for the production of nanocomposites based on graphene and multiphase clay/CNTs.
These nanocomposites are potentially very interesting for different industrial applications but, up to now, have not been intensively and accurately investigated.
An and extensive structural and morphological analysis will be carried out on nanofillers and nanocomposites in order to provide detailed information on nanostructures. These data will be correlated with the information obtained from an accurate characterization at both micro and macro scale concerning EM
properties (at low and high frequency), thermal and mechanical characteristics in order to improve the physical and numerical models adopted in materials analysis and design.
A further effort is aimed at the global optimization of the base materials properties (nanofillers and polymer) and parameters of the production process able to lead to the desired EM performances of the nanocomposites. Robust optimization techniques will be employed to overcome the drawbacks of the currently adopted approaches consisting in the choice of the best solution for each step of the production process parameters (local optimization) which generally may not lead to the

global optimum. The main objectives are pursued by a multidisciplinary research team composed by five Research Units which develop complementary and integrated research lines.

Relevant results achieved:

The activity carried out by INFN-LNF has concerned the mechanical and electrical characterization of nanocomposite materials, which were made using an epoxy resin (EPIKOTE 828) as matrix, loaded with different types of fillers such as carbon nanotubes, carbon black and graphite. Work has concerned the study of mechanical and electrical properties, carried out on epossidic nanostructured materials. The effects of concentration and dispersion's degree of carbon nanotubes (Cnts in short) on the tensile strength and electrical conductivity of the nanocomposites were studied. In order to realize these materials we used an epoxy resin (EPIKOTE 828) as matrix, which is a liquid of medium-low viscosity at room temperature and it is hardened by the curing reaction with polyamines. The curing agent, called A1 (specific gravity 1.02g/cm3 and viscosity 0.21Pa•s at 25ºC) is obtained from a common TEPA (tetraethylenepentamine) by reaction with formaldehyde (CH2O).This resin is loaded with different percentages of conductive nanofillers such as Cnts, synthesized with the arc discharge method. To check the efficiency of the coating, graphite and carbon black were taken as referencing fillers, on which both mechanical and electrical tests were carried out.
From a mechanical standpoint the analysis showed that:
- The mechanical properties of the matrix are not improved by fillers because they are short fibers (or amorphous).
- At equal concentrations, composites loaded with CNTs are substantially better than the ones loaded with carbon black and graphite.
From the electrical standpoint the analysis showed that:
- The electrical conductivity increases, with the increasing concentration of Cnts. This is probably because they are distributed randomly in such a way as to create a preferential path for current flow inside the matrix.
- At equal concentrations, composites loaded with Cnts have greater electrical conductivity than the ones loaded with carbon black and graphite.
In a second step, since the Cnts have showed values of electrical conductivity higher than the other fillers, we decided to compare them with commercial Swnts and Mwnts, synthesized with the CVD technique (Chemical Vapor Deposition). On the nanocomposites obtained using these fillers, electrical measurements and measurements of epsilon' and epsilon" in microwave range were carried out.
As regards DC electrical measurements, for low percentage Cnts loadings the obtained results seem to indicate that the method of synthesis by arc discharge yields better results than the growth of nanotubes with CVD technique. In this regard the samples were subjected to TEM and SEM micrographies, and the images showed how the Cnts of INFN (arc discharge) have less defects than commercial ones (CVD). Regarding higher concentration of fillers one can see that the values of resistivity of the matrix with INFN-Cnts and the one with Mwnts are comparable with each other, while the value of resistivity of the matrix with Swnts is approximately three orders of magnitude lower. Therefore, increasing the amount of nanotubes, a mechanism of percolation is activated, in which an interconnected network is formed (so we are beyond the percolation threshold).
Finally, as regards the measurements in microwave range, it was found that commercial Mwnts are more effective than Swnts for the realization of DC conductive composites. At the same time, using the same epoxy/Cnts composites in Ka-band (26-37 GHz), the results have shown that in 26-27 GHz both fillers have almost the same efficiency, although Swnts show slightly better results.

Publications by LNF Authors in the Year 2011

S. Bellucci, F. Micciulla, I. Sacco, L. Coderoni and G. Rinaldi, Electrical Properties and Electromagnetic Shielding Effectiveness of Carbon Based Epoxy Nanocomposites, Nanodevices and Nanomaterials for Ecological Security, NATO Science for Peace and Security Series B: Physics and Biophysics, 2012, Part 1, 115-123, DOI: 10.1007/978-94-007-4119-5_10.

A. Pliushch, A. Paddubskaya, P. Kuzhir, S. Maksimenko, L. Coderoni, F. Micciulla, I. Sacco, S. Bellucci, Nanocarbon Modified Epoxy Resin and Microwaves, Fullerenes, Nanotubes and Carbon Nanostructures, Volume 20, Issue 4-7, 2012, pages 496-501

J. Macutkevic, D. Seliuta, G. Valusis, R. Adomavicius, P. Kuzhir, A. Paddubskaya, M. Shuba, S. Maksimenko, L. Coderoni, F. Micciulla, I. Sacco, S. Bellucci, Terahertz time domain spectroscopy of epoxy resin composite with various carbon inclusions, Chemical Physics, Volume 404, 24 August 2012, Pages 129–135

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List of Conference Talks by LNF Authors in the Year 2011

S. Bellucci, “Epoxy-Nano-Carbon Coating for Shielding in Wide Frequency Range”, FM&NT 2012, April 17 – 20, Riga, LATVIA.

F. Micciulla, "New nanocomposites fillers obtained via Self-propagating High-Temperature Synthesis (SHS) method", 7th International Conference on NANOSTRUCTURED POLYMERS AND NANOCOMPOSITES April 24 – 27 Praga, Czech Republic

S. Bellucci, "Microwave probing of graphitic materials – filled composites", GRANADA'12, Graphene Nanoscience:
from Dirac Physics to Applications 9-13 September 2012, Granada, Spain.

S. Bellucci, "Atomistic simulations of defect containing tubular nanostructures", Fundamental and Applied NanoElectroMagnetics (FANEM), Belarusian State University, Minsk, Belarus, May 22-25, 2012

S. Bellucci, “Microwave probing of composites based on exfoliated graphite and nanocarbons”, Nanoscience & Nanotechnology 2012, Laboratori Nazionali di Frascati 1-4 October 2012.