Multiscale composites within health-monitoring capabilities.

R.Volponi[a]*, P.Spena[b], F.De Nicola[a], Andrea Grilli[b]

[a] *CIRA Italian Aerospace Research Centre, Advanced Materials and Technologies Lab Via Maiorise Capua (CE) Italy*

[b] *IMAST S.c.a.r.l., Technological District on Engineering of polymeric and composite Materials and Structrures, P.zza Bovio – 80133, Napoli, Italy*

** corresponding author: r.volponi@cira.it*

Keywords: Nanocomposites, healt-morning, nanotubes, composites multiscale.

Abstract

In this work piezo-resisitive proprieties epoxy resins charged with carbon nanotubes are been verified.
An efficient solution to integrate nanocharged matrix into a carbon fiber composites are been developed, achieving a multiscale carbon fiber composite.
Finally mechanical and health-monitoring capabilities of multiscale composites are been evaluated.

1. Introduction

In recent years the use of fiber reinforced resin composites has continuously expanded expecially in aerospace.
Inspection and Maintenance are important aspects when considering the availability of aircraft for revenue flights.
Modern airframe design is exploiting new exciting developments in materials and structures to construct ever more efficient air vehicle able to enable ‘smart’ maintenance .So there are a wide interest in developing new composites materials in which is the integrations new functionalities.
One of these new integrated functionality studied in these work is the electrical piezo-resitivity behaviour of polymer charged with carbon nanotube [1]…[5].
Using such nanocharged polymer as matrix into a carbon fiber fabric is possible to obtain a composite in which the intrinsic piezo-resistivity can be used as integrated health-monitoring capability.[7]…[13]

2. Materials

2.1 Piezoresistivity of epoxy resin charged with carbon nanotube

In order to develop a multiscale composite we have studied the electrical beaviour of an epoxy resin charged with carbon nanotube.
Infact charging an epoxy resin with carbon nanotube it became electrical conductive. Furthermore an epoxy resin charged with carbon nanotube show a heavy piezo-resistivity effect: his electrical resistance change when is applied a strain.
So the idea is to use that nano-composite as matrix in a carbon fiber rinforced composite to add an intrinsec health-monitorin funcinality.
The epoxy matrix system choosed is commercialized by Elantas named EC327.
That system has a very low viscosity at room temperature, around 300mPas, a long pot-life of several hours at room temperature, a storage modulus of 3200Mpa and a glass transition temperature of about 190°C.
The resin has been charged with carbon nanotube 3100 Nanocyl through a mechanical system: a three roll mill Exakt 80E, with three different amount of CNT: 0,3%, 0,6%, 0,9%.
Nano-charged resins were cured following the proper cure cycle as given by data sheet.
From the three plates were cutted different samples. Electrical contacts were done using a conductive silver paste at the edge of the sample. Then the sample was incorporated in the pure resin to insulate and to have a mechanical clamping. **(Fig.1)**
Electro-mechanical tests were performed over the sample using an Instron tensile machine and a Keithley 2182A picoammeter with a nanovoltmeter Keithley 6221
Strains were measured by a extensometer staked over the sample and variation of electrical resistance were recorded simultaneously **(Fig.2)**.

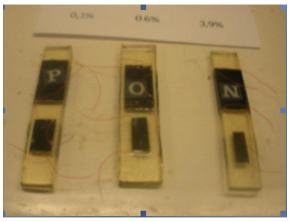


Fig.1 Sample of nanocharded resins

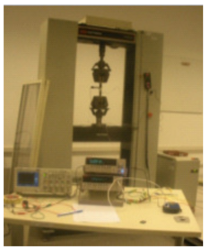


Fig.2 Tensile machine Instron and Keitley ammeter-voltmeter system

Two different kind of test were performed: first a load-unload cycles and a creep test (load, maintaining, unloading).
A very important parameter estimated in the tests was the Gauge factor G_f defined as:

$$GF = \frac{\Delta R/R_G}{\epsilon} \quad (1)$$

It account the variation of the electrical resistance due to the strain. If the Gauge factor is greater than 1 means that there are an amplifier effect.

So for resin charged using 0,3% of CNT the response were:

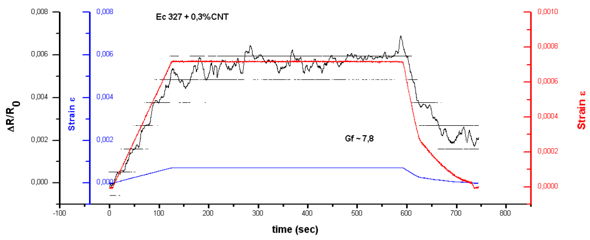


Fig.3 Creep test for 0,3% CNT

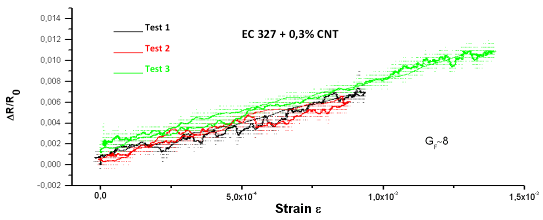


Fig.4 Load-unload test for 0,3% CNT

For 0,6%:

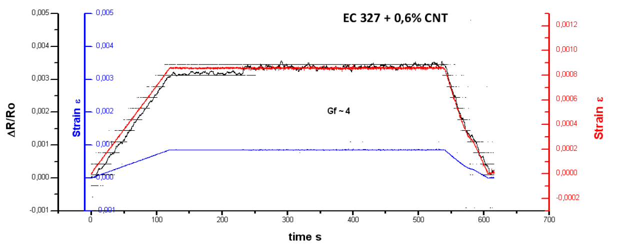


Fig.5 Creep test for 0,6% CNT

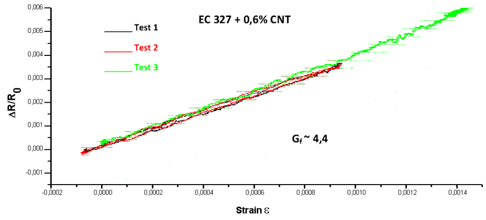


Fig.6 Load-unload test for 0,6% CNT

Finally with 0,9% were found:

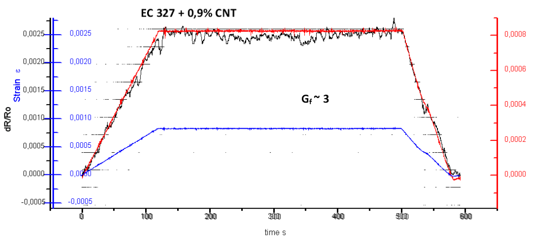


Fig.7 Creep test for 0,9% CNT

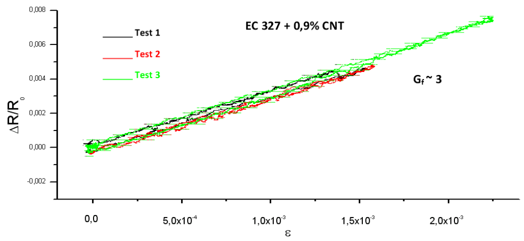


Fig.8 Load-unload test for 0,9% CNT

In the follow table are summarized the Gauge factors, the electrical conductivity and the viscosity founded varying the charge:

Sample	**Gauge Factor**	**Conductivity (S/m)**	**Viscosity @1Hz@ 80°C (Pa*s)**
EC327+0,3%	8	3e-4	1,3
EC327+0,6%	~ 4	1,3e-2	2
EC327+0,9%	3	1,4e-1	30

Table 1 Gauge factors, Conductivity and Viscosity of nanocharged resins

It's possible note that the Gauge factor decrease as the charge increase. This comportment is due to the realization of the conductive network by nanotubes. These conductive network follows a percolative so at low ratio of carbon nanotubes is more sensitive to the variations of shape of the sample due to the mechanical stresses.

2.2 Manufacturing of a Multiscale Composite.

The amount of carbon nanotube chosed as charge for nanocomposite matrix in the multiscale composite was the 0,6%.
That percentage show a good conductivity and an evident gauge factor.
A first attempt to infuse the charged matrix into a 7 plies dry preform was made by liquid infusion thecnique.
The result was a dry pannel with evident voids and an heavy filtering effect **(Fig.10D).**
Infact as a nanofilled resin exceeds the limit of 0.8 Pa*s [6]; the usual liquid infusion becomes unfeasible to infiltrate a carbon fiber laminate.
To overcome this critical point, a new unusual thecnique has been developed.
A thin layer of liquid epoxy mixture containing MWCNTs (0.6%wt) was spread on a release films (Release Ease 234 TFP-HP Airtech) **(see fig.9A).;** then a dry preform (300mm x 300mm) made laminating 20 plies of carbon fiber cloths (SIGMATEX (UK) LDT 193GSM /PW/HTA40 E13 3K) was placed on mixture **(see fig.9B)** forcing it to flow throw the thickness of the preform using an external supplementary pressure inside an autoclave. In this way the length of the impregnation path is considerably reduced and the process can be forced by means of the pressure application. A further advantage of this technology is due to the smaller length of the infiltration path which reduces the effects of infiltration through the preform. In fact, the edges of the preform were sealed to force the resin to only flow through the thickness The laminate was covered by a porous release film and a distribution media to allow to the resin to escape from the upper side and a breather media to receive the excesses of the resin **(see fig.9C).** Finally it was made a vacuum bag and the laminate was putted into the autoclave **(see fig. 9D).**

Fig. 9A Thin layer of nanocharged resin

Fig. 9B Dry carbon fiber preform placed on the layer of resin

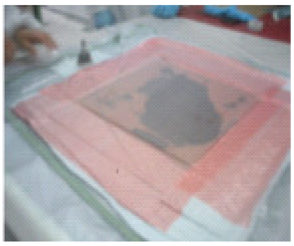


Fig. 9C Vacuum bag

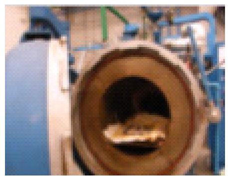


Fig. 9D Autoclave

The panel obtained was well infused, without voids, although it presents an excess of resin on the bottom side**(see fig.10B),** and the ratio resin/fiber varies through the thickness **(see Fig. 10C).**

Fig. 10A Upper side

Fig. 10B Bottom side

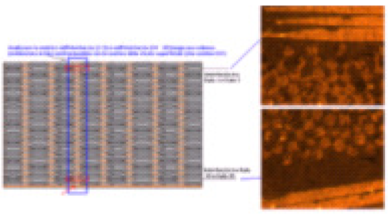


Fig. 10C Microscope analysis: absence of voids

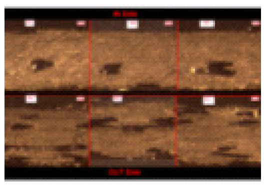


Fig 10D Liquid infusion process: precence of voids**.**

A second panel of 7 plies was infused with pristine resin and the same carbon fiber clothes and was used as reference.

2.3 Piezoresistivity of Multiscale Composite.

First were measured the electrical conductivity through the thickess.
Three square shaped of (40mmX40mm) sample were cutted from multiscale composite and from the reference.
Each side of samples were painted with a silver based paint (**Fig 11A**) and were measured the electrical conductivity through the thickess with Keitley ammeter-voltmeter system.
Finally those values were compared with those found in a commercial 18 plies prepreg composite (**Fig 11B**).

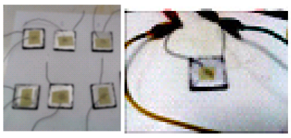


Fig 11A Samples for measuremten of electrical conductivity through the thickess.

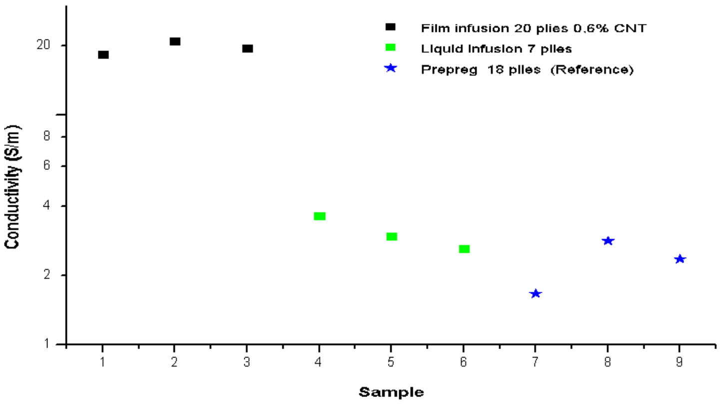
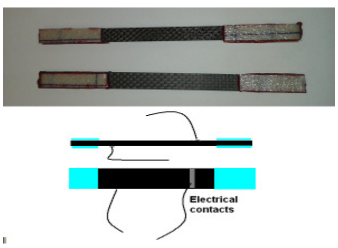


Fig 11B Electrical **c**onductivity through the thickess of different kinds of composites

Is possible to notice that electrical conductivity to the thickness increse about 7 times with nanotube, and the initial values are comparable with the values found in a commercial carbon fiber composite (**Fig.11B**).
These result is very interesting as there are a a great commercial interest to increse the electrical conductivity into the thickness in carbon fiber composites to overcame problems due to ligthning in aircraft.

Subsequently to evaluate the health-monitoring intrinsic capability of multiscale composite panel, a sample lenght 200mm, width 10mm and thick 2,8mm has been cutted.
An other sample lenght 200mm, width 10mm and thick 1,3mm has been cutted from panel infused with pristine resin and has been used as reference.



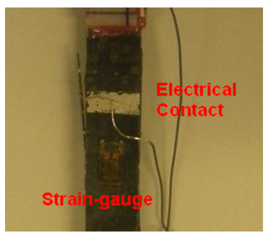


Fig. 12 Samples for tensile test of reference composite and multiscale composite

To achieve an electrical insulation between the samples and the clamps of the tensile test machine, at each ends of the samples were sticked 2 tabs made of glass fiber composite.

Electrical contacts were made on opposite side of sample (**Fig.12**).
Finally on each sample were stiked a strain-gauge sensor.
The electrical behaviour of the reference sample has been tested.
The sample was campled into the Istron machanical testing machine and a costant current of 33mV has been applied between the electrodes.
By means a Vishay datalogger, were recodered in the time, the variations of strain, load, and voltage applied to the sample. Voltage is direct proportional to the electrical resistance measured between the electrodes.
A first charactherization has been made appling 50 cicles of a costant variation of load and unload at rate of a cicle each minute for a maximum strain of 1400 με.

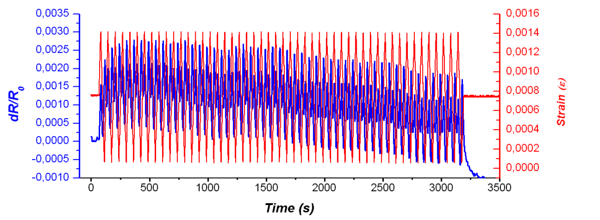


Fig.13A 50 Cycles load-unload test for reference composite

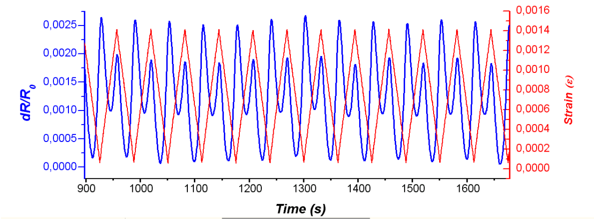


Fig.13B Particolar of 50 Cycles load-unload test for reference composite

From data is possible to see the presence of a delay, and a non linear variation. Infact there a double frequence of maximum in the variation of resistence respect the maximum loads applied. These beaviour means that recovery effects are presents.
The maximum variation of electrical resistance respect the maximum strain applied is 1,7.
So a second kind of test has been performed: tensile test till to break.

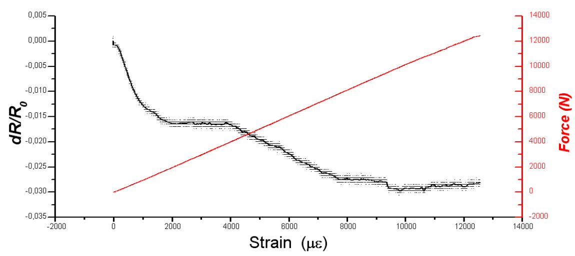


Fig.14 Tensile test till break of reference composite

In these test is evident first of all a negative variation of the electrical resistance. These beavior is completly different to that founded in charged resins, but can be easily explained as applaied strain reduce the distance beetween the carbon fiber and beetween the plies.
Is evident too a change of the slope on the curve of the variation of resistance expecially around 1000με. These beaviour can explain the recovery effects seens in cyclic tests.
Finally the break is fragile and the gauge factor is around -0,25.

For the multiscale carbon fiber composite the sample was underwent to the same previous cycling test:

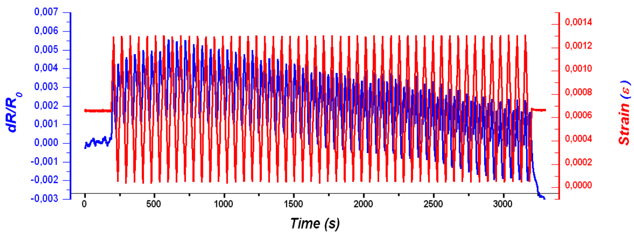


Fig.15A 50 Cycles load-unload test for multiscale composite

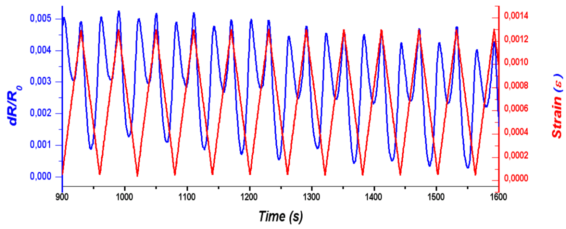


Fig.15B Particolar of 50 Cycles load-unload test for multiscale composite

Also in these case are evident recovery effects and a delay, but the variation of the resistence are 3 times greater that the applayed strain.
More analysis is possible from results tensile test till to break:

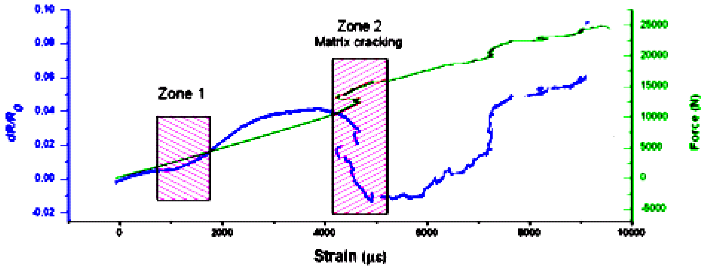


Fig.16 Tensile test till break of multiscale composite

In these case the electrical resistence increase as the strain increase.
Is possible to notice that around 1000 με (Zone 1) the slope of the variation of electrical resistance varies, and as told for the reference composite this explain the delay in the response.
Around 5000 με (Zone 2) is possible to see a sudden variation of response of resistence; it occur in corrispondence of breaking in the linearity between the force applied and the strain recorded (green line). Means that a crack in the matrix has occured.
Is interesting to note how the response of electrical resistance in the multiscale composite is very sensitive to the formation of cracks in matrix.
After that event the signal became very noisy but it follows a positive trend concordantly with stress-strain curve.
At 4000 με the gauge factor is 8.

3 Conclusions

A feseable method for fabrication of carbon fiber composites with a matrix charged with CNT has been assesed.
The strain sensitive capability of a multiscale composite has been tested.
That capability have demostrate an high sensitivity in case of formation of cracks in the matrix..

4. Acknowledgment

The research leading to these results has received funding from the European Union Seventh Framework Programme FP7/2007–13 under grant agreement no. 313978.

References

[1] Wichmann, M. H. G.; Buschhorn, S. T., Gehrmann, J., & Schulte, K. (2009). Piezoresistive response of epoxy composites with carbon nanoparticles under tensile load. *Phys. Rev. B*, Vol. 80, (December 2009), 245437, ISSN 1098-0121

[2] Iosif D.; Rosca, Suong V., Hoa. (2009). Highly conductive multiwall carbon nanotube and epoxy composites produced by three-roll milling. *Carbon*, Vol.47, Issue.8, (July2009), pp. 1958–1968, ISSN 0008-6223

[3] Kang I, Schulz MJ, Kim JH, Shanov V, Shi D. A carbon nanotube strain sensor for structural health monitoring. *Smart Mater Struct* 2006; 15: 737–48.

[4] Hu, N.; Masuda, Z.; Yan, C.; Yamamoto, G.; Fukunaga, H.; Hashida, T. Electrical properties of polymer nanocomposites with carbon nanotube fillers. *Nanotechnology* 2008, 19, 215701

[5] Allaoui A.; Bai S., Cheng H., Bai J. (2002). Mechanical and electrical properties of a MWNT/epoxy composite. *Compos. Sci. Technol*., Vol.62, Issue.15, (November 2002), PP. 1993–1998, ISSN 0266-3538

[6] DW. Becker, Tooling for Resin Transfer Moulding, Wichita State University, Wichita Kansas, no date

[7] Alexopoulos, N.D., Bartholome, C., Poulin, P., Marioli-Riga, Z., 2010. Structural health monitoring of glass fiber reinforced composites using embedded carbon nanotube (CNT) fibers. *Composites Science and Technology* 70, 260–271.

[8] Kang I, Schulz MJ, Lee JW, Choi GR, Jung JY, Choi JB, et al. A carbon nanotube smart material for structural health monitoring. Solid State Phenom 2007; 120: 289–96.

[9] Gao, S.-L.; Zhuang, R.-C., Zhang, J., Liu, J.-W., & Mader, E. (2010). Glass fibers with carbon nanotube networks as multifunctional sensors. *Adv. Funct. Mater*., Vol. 20, (May 2010), pp. 1885–1893, ISSN 1616-301X

[10] Bekyarova, E.; Thostenson, E. T., Yu, A., Kim, H., Gao, J., Tang, J., Hahn, H. T., Chou, T.-W., Itkis, M. E., & Haddon, R. C. (2007). Multiscale carbon nanotube-carbon fiber reinforcement for advanced epoxy composites. *Langmuir*, Vol. 23, pp. 3970-3974, ISSN 0743-7463

[11] Cho, J., Daniel, I.M., Dikin, D.A., 2008. Effects of carbon nanotube reinforcement on mechanical response and damage tolerance of carbon/epoxy composites. Proc. *Am. Soc. Composites, Technical Conference*, 23rd, pp. 89/1–89/14

[12] Thostenson, E.T., Gangloff Jr., J.J., Li, C., Byun, J., 2009. Electrical anisotropy in multiscale nanotube/fiber hybrid composites. *Applied Physics Letters* 95, 073111

[13] Kim, M., Park, Y., Okoli, O.I., Zhang, C., 2009. Processing, characterization, and modeling of carbon nanotube-reinforced multiscale composites. *Composites Science and Technology* 69, 335–342.