FUNCTIONAL CHARACTERIZATION OF FIBER REINFORCED NANOCOMPOSITES

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Abstract: This work presents an insight into the effect of preparation procedure and the filler content on functional properties of glass fabric reinforced nanocomposite systems. For the preparation of nanocomposites, unidirectional glass fabrics were used. Methyl siloxane was used as resin in the study. As fillers, carbon black (CB) nanoparticles having size less than 50 nm were used.
The characterization of nanocomposites was done using Dynamic Mechanical Analysis (DMA), Thermal Mechanical Analysis(TMA), Impact testing , Tensile testing, Thermal conductivity and electrical conductivity measurements. The morphology of composites was characterized by Scanning Electron Microscopy (SEM). The thermal conductivity of the composite was measured by Differential Scanning Calorimetry (DSC) and Alambeta thermal conductivity tester. The electrical conductivity was measured by 2 electrode probe method.
The dependence of heat flow Q, thermal expansion coefficient, α, the dynamic mechanical parameters, E', E'', tanδ, Tg, thermal and electrical parameters (λ, R and ρ), impact energy are associated with the filler content and is controlled by the employed curing conditions. Experimental results show that some functional properties can be enhanced by the incorporation of nanoparticles.

Keywords: Nanocomposite, glass fiber reinforcement, thermo mechanical analysis, knife penetration, electrical resistivity

1. Introduction

Traditional fiber-reinforced composites have improved over the years with respect to material properties and have gained considerable acceptance in the aerospace industry. The in-plane properties of fiber/polymer composite system are defined by the fiber properties, while the properties along thickness dimension are defined by the characteristics of the matrix resin [1]. The use of an additional phase (e.g. inorganic filler) has been proven to improve the mechanical properties of the resin as well as some nanofillers can be used to get advanced functional properties in the composite material [2,3].

Nano-phased matrix based on organic polymers and inorganic particles such as carbon nanoparticles have attracted great interest because they frequently exhibit unexpected properties including reduced gas permeability, improved solvent resistance, superior mechanical and enhanced flame retardant properties. Different polymer/carbon nanocomposites have been successfully synthesised by incorporating various polymer matrixes such as epoxy [4-6].

The primary focus of this paper is to characterize the effect of carbon nanoparticles on thermal, electrical and mechanical properties of Methyl Siloxane. An ultrasonicator was used to disperse carbon nanoparticles in the resin.

2. Experimental

2.1 Materials and Manufacturing of Nanocomposites

The resin used in this study is commercially available Methyl Siloxane (Lukosil). It is a low viscosity resin. The Carbon nanoparticles used were obtained from Sigma Aldrich, USA having particle size less than 50nm.
A precalculated amount of carbon nanoparticle and resin were carefully weighed and mixed together in a beaker. The mixing was carried out through a high intensity ultrasound

irradiation by a sonicator probe (Bandelin Sonoplus Sonicator, UK) for 10 minutes with maximum energy of 30KJ.
The glass fabric samples were coated with the prepared carbon/siloxane resin and left for room temperature curing for 16 hrs. The test samples were again coated with the same carbon/siloxane resin and post cured at 200^0C for 6 hrs. Finally test samples were cut from the original one for thermal, electrical and mechanical characterization [7].

2.2 Test Procedure

2.2.1 Electrical Conductivity

Hewlett Packard (hp) 4339B high resistance meter was used to measure the surface resistance and volume resistance of the composites. The environmental condition for the measurement was 22^0C temperature and 29.5% relative humidity and voltage used was 100V.

2.2.2 Differential Scanning Calorimetry (DSC)

A Perkin Elmer Differential Scanning Calorimeter (Pyris 6, DSC) was used under Nitrogen atmosphere. The measurements were performed from room temperature to 400^0C at a scanning rate of 10^0C/min.

2.2.3 Thermal Mechanical Analysis (TMA)

A RMI Thermomechanical Analyzer TMA CXO 3RA-T was used for this purpose. The measurements were performed from room temperature to 800^0 C, with static force of 100mN and rate of 5^0C/min.

2.2.4 Thermal Conductivity Measurement

Thermal conductivity of the samples was measured on Alambeta Thermal conductivity tester (hot plate method).

2.2.5 Knife Penetration Resistance

Knife penetration resistance of the composite samples were measured on Impact tester.

3. Results and Discussion

3.1 Electrical Conductivity

Volume resistivity and surface resistivity of the composite samples are shown in Figures 1 and 2 respectively. Volume conductivity and surface conductivity of the nanocomposite samples was found to be higher than the neat resin composite.

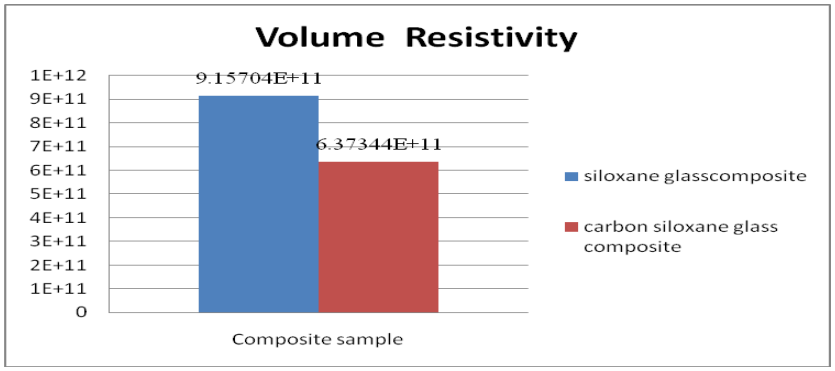


Figure 1. *Volume resistivity (Ωm) of the composite samples*

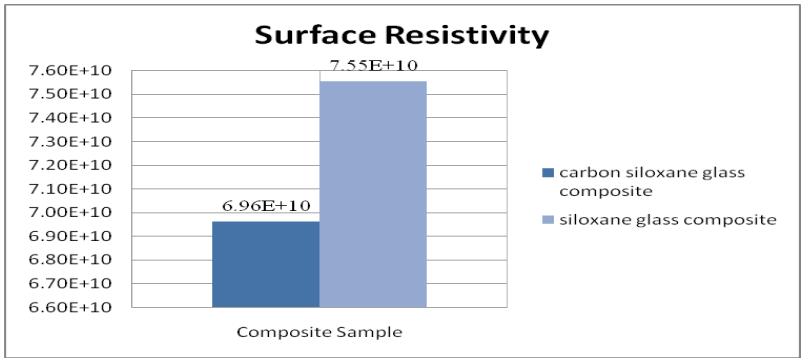


Figure 2. *Surface resistivity (Ωm) of the composite samples*

Higher electrical conductivity of the carbon nanocomposites is due to the formation of conductive path inside the composite according to Maxwell Wagner and Sillars. The simplest model for describing an inhomogeneous structure is a double layer arrangement, where each layer is characterized by its permittivity $\varepsilon'_{1,2}$ and its conductivity $\sigma_{1,2}$. The relaxation time is

then: $$\tau_{MW} = \epsilon_0 \frac{\epsilon_1 + \epsilon_2}{\sigma_1 + \sigma_2}.$$

Importantly this shows that an inhomogeneous material may have frequency dependent response, even though none of the individual inhomogeneities is frequency dependent.

A more sophisticated model for treating interfacial polarization was developed by Maxwell, and later generalized by Wagners and Sillars. Maxwell considered a spherical particle with a dielectric permittivity ε'_2 and radius *R* suspended in an infinite medium characterized by ε_1.

3.2 Heat Flow Rate and Thermal Expansion Coefficient

Heat flow rate through the nanocomposites was higher than the neat resin composite as shown in Figure 3. The nanocomposites have a thermal expansion coefficient lower than the neat resin composites (Figure 4).

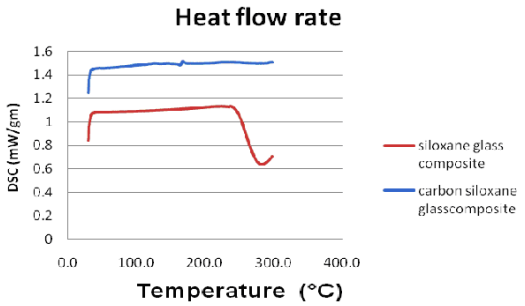


Figure 3*. Heat flow rate through the composite*

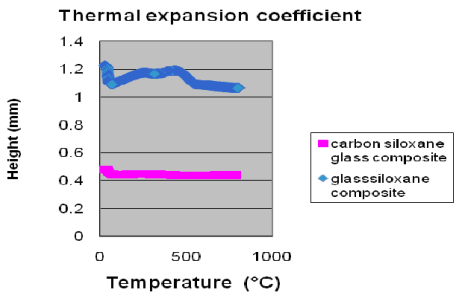


Figure 4*. Thermal expansion coefficient*

A higher heat flow rate in case of carbon nanocomposites is due to better conductivity of carbon which is an integral part of the composite and acts as an interface between the glass reinforcement and the siloxane resin.
However, with the presence of nanoparticles in the matrix system, the mobility of polymer chains is restricted even under high temperature conditions. Thereby the thermal expansion coefficient is lower for this class of materials.

3.3 Thermal Conductivity Parameters

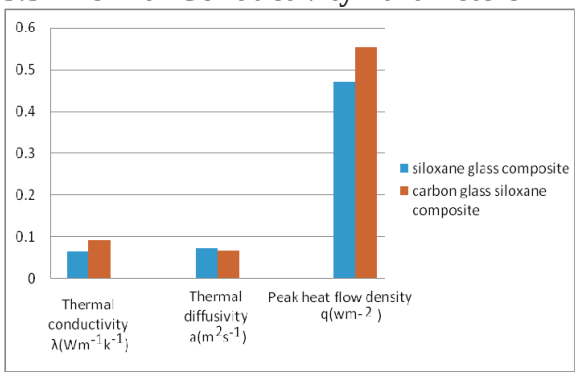


Figure 5. *Thermal conductivity, diffusivity and heat flow density*

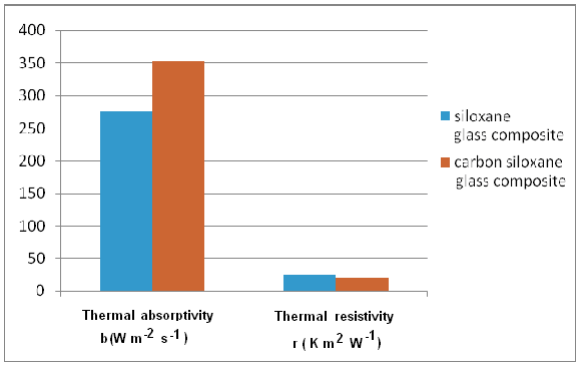
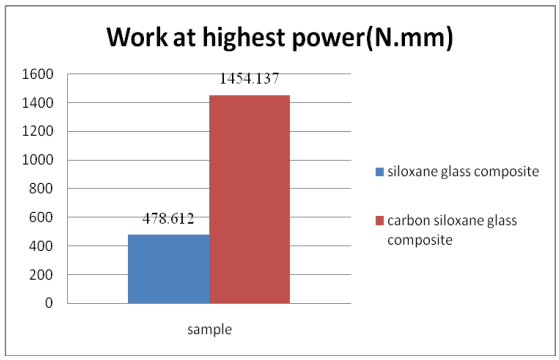


Figure 6*. Thermal absorptivity and thermal resistivity*

the thermal conductivity, heat flow density and absorptivity improve with incorporation of carbon nanoparticles in the matrix. At the same time diffusivity and resistivity fall down. This behaviour is attributed to the excellent thermal conductive nature of carbon, which facilitates better flow of heat through the matrix system by transfer of heat between the adjacent particles.

3.4 Knife Impact Penetration Resistance



F*igure 7**. Work at highest penetration*

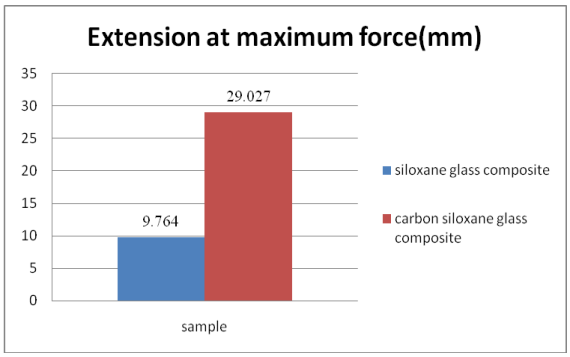
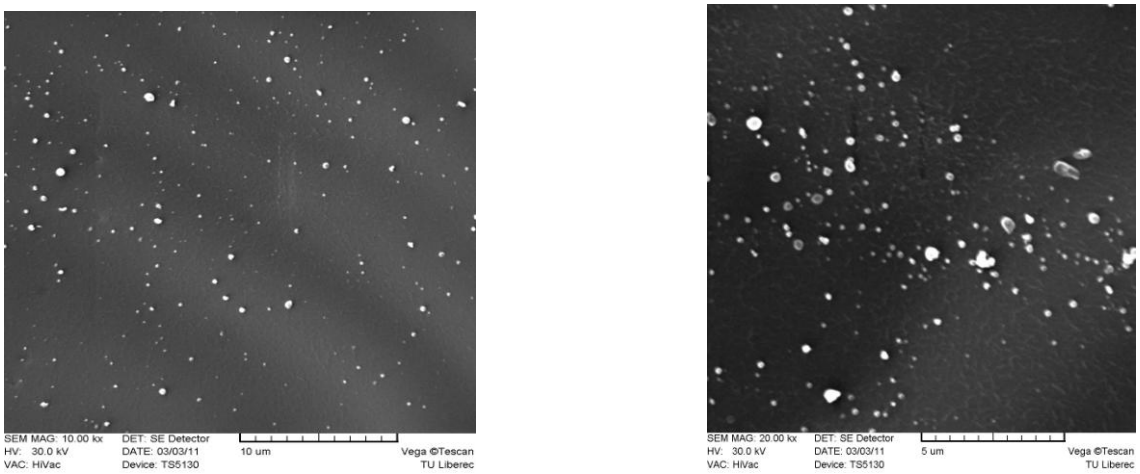


Figure 8*. Extension at maximum force*

It can be observed from Figure 7 above that a higher energy is required for penetration of knife through the carbon particle reinforced nanocomposite compared to pure resin composite. This proves that the carbon nanoparticles create a stronger interface between the matrix and the reinforcement of glass fabric due to their exceptionally high surface interaction. This results in a higher resistance offered to penetration of knife through the fiber -nanoparticle-matrix system. The same is also observed in Figure 8, in terms of higher extension at maximum force for a carbon nanocomposite. The nanoparticles offer higher resistance to deformation upon knife impact.

3.5 SEM Images of Carbon Nanocomposites



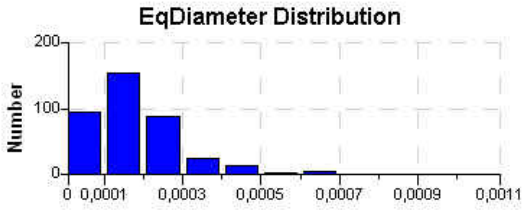
(a) (b)

Figure 9*. SEM image of composite surface (a) 10 µm magnification (b) 5µm magnification*

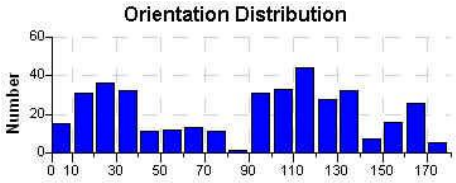
The images obtained from Scanning Electron Microscopy (SEM) show distribution of nanoparticles in the composite system.

3.6 Particle Size Distribution Statistics on the Composite Surface

3.6.1 10µm Magnification



(a)



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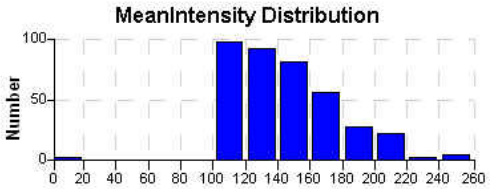

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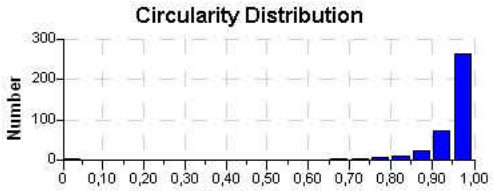
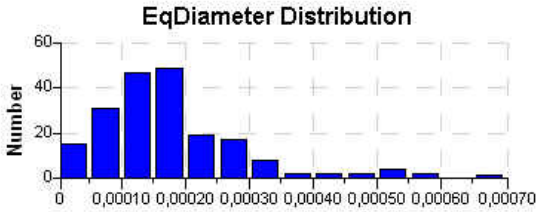
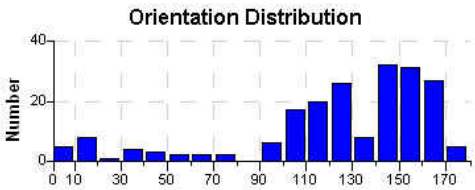


(d)

3.6.2 5µm Magnification



(a)



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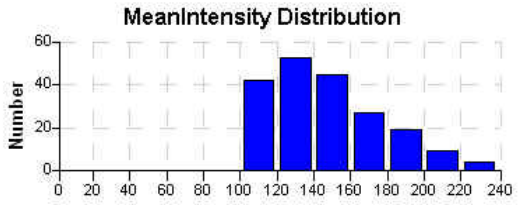

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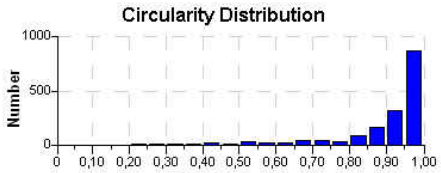

(d)

The particle size distribution as shown in the Figure above indicates that it follows a normal distribution. That means the carbon nanoparticles are uniformly distributed in the matrix.

4. Conclusion

The mechanical strength enhancement can be attributed to the intrinsic characteristics of the nanoparticles based on the rule of mixture. Electrical conductivity, which is observed at about 1% w/w of nanoparticle, indicates the creation of conducting paths and is associated with the Maxwell Wagner Sillars (MWS) relaxation, probably due to the formation of aggregated microstructures in the bulk composite. The results of DSC show that the heat flow rate through nanocomposite is higher than the pure resin composite.

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