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Polymer Nanocomposite Materials for Structural Applications

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1. Introduction

The improvements of physicochemical and thermomechanical properties of polymer composite materials has been a major research interest in the last few decades. Polymer nanocomposites (Mohammad, 2006; Xiangling, 2003) represent a new class of materials alternative to conventional filled polymers. In this new class of material, nanosized inorganic filler (at least one dimension) are dispersed in polymer matrix offering tremendous improvement in performance properties of the polymer. Nanoscale materials have been the subject of research interest in recent years because of their unique properties as compared to the bulk counterparts and their potential applications in a wide variety of areas such as information storage, electronics, sensors, structural components, and catalysis etc. The main challenge in fabrication of these polymer nanocomposites for structural applications is uniform dispersion of nanoparticles in the polymer matrix. However, good dispersion for nanoparticles in polymer composite materials is extremely difficult to achieve, since nanoparticles tend to aggregate together during fabrication. The degree with which the nanoparticles can be homogeneously dispersed in the polymer matrix would significantly influence the thermal, mechanical and optoelectronic properties of the material. Researchers have used several techniques for dispersing nanoparticles may include: 1) mechanical agitation, such as ball milling or magnetic stirring, 2) ultrasonic vibration, 3) shear mixing 4) non-contact mixing 5) using the dispersing agent. The process of curing of these polymer is an another important factor in improving the mechanical, thermal, electrical and optical properties. There is an ever-increasing demand for development of processing of polymer nanocomposites for advanced structural applications. In polymer composite fabrication, curing (polymerization) step is crucial and time-consuming process. The processing thermoplastic nanocomposites were carried out using melt extrusion, or solution casting. The epoxy based thermoset polymers are generally cured using conventional heating. The manufacturing industries of these nanocomposites are also in need of quick and efficient curing method for high temperature or room temperature curing of epoxy polymers. The conventional method of curing is time-consuming, the longer it takes to complete a project, the more expensive it becomes. Several alternate curing methods have been tested and they are: UV rays, Gamma rays, Electron Beam and Microwave (Bogdal & Karen, 2003; Clark & Sutton, 1996), microwave heating curing of room or high temperature epoxy resins. The current chapter is focused on fabrication techniques, synthesis, and characterization of four main polymeric

nanomaterials for various applications: a) Dispersion, synthesis, and curing of epoxy-based nanocomposites, b) Polymeric foam core nanocomposites for lightweight and high strength applications c) Alignment of acicular nanoparticles in thermoplastic polymers to improve the mechanical and antimicrobial properties of textile fibers. d) Development of shear thickening fluid for liquid body armor applications.

2. The dispersion, synthesis, and curing of epoxy-based nanocomposites

The epoxy based nanocomposites are generally prepared by dispersion of known amount of nanoparticles in one of the non-reactive parts of the resin and mix with reactive part of the resin and finally cure. Depending on the physical and chemical properties of nanoparticles, and epoxy resin the processing technique is selected. Most of the commercial metal, metal oxide, and carbon based nanoparticles are used as received. Where as some of the high density and highly agglomerated/entangled nanoparticles such as WO_3 and CNTs require a special modification for better dispersion to obtain optimal improved properties. In one of our recent studies a high density WO_3 nanoparticles were subjected to sized reduction process for better dispersion and improve mechanical properties. The porous WO_3 (pore size 2-5nm) nanoparticles were synthesized using a high intensity ultrasound irradiation of commercially available WO_3 nanoparticles (80nm) in ethanol. The nanocomposites of WO_3 /SC-15 epoxy were prepared by infusion of 1wt%, 2wt% and 3wt% of porous WO_3 nanoparticles into SC-15 epoxy resin by using a non contact (Thinky) mixing technique. In this technique the material container is set at 45 degrees angle inside the mixer and revolves and rotates at high acceleration with the speed of ~ 2000 rpm, dual centrifugal forces were given to the container that keep pressing materials to outward and down along with the slope of inner wall of the container and accomplish powerful mixing and removing air pockets formed in the reaction mixture simultaneously. This technique is non contact and non reactive unlike ultrasound and other mixing techniques (Guo et al., 2006; Isobe et al., 2006; Allaoui et al., 2008; Rao et al., 2003). The mixture was degassed using a vacuum oven for 30 minutes and the finally the neat epoxy and nanocomposites were cured at room temperature for about 24 hours in a plastic rectangular mold. The known percentages of the WO_3 nanoparticles were dispersed in epoxy part-A (SC-15, applied polyamics) using a noncontact defoaming mixer (Thinky, Japan) for 15 minutes. The part-B of the resin was then added to the mixture of part-A containing WO_3 and mixed again using a noncontact defoaming mixer for another 10 minutes. Finally the resin mixture was poured into a polypropylene container and cured at room temperature for 24 hours. This procedure was repeated for three different weight percentages of the WO_3 nanoparticles (1wt%, 2wt% and 3wt %) to make the epoxy nanocomposite and neat epoxy. The samples were cut precisely and used for the microscopic and mechanical testing.

Figure 1(a) shows the TEM picture of the as-received WO_3 nanoparticles. The particles are nearly spherical in shape and the size distribution is wide (~ 30 -100nm). The high resolution micrograph (shown in Figure 1(a)) clearly shows that there are no pores in the as received WO_3 nanoparticles where as the Figure 1(b) clearly shows that the sonochemically modified WO_3 nanoparticles are porous and typical pore size is ~ 2 -5 nm. These TEM results indicate that without significant change in the particles sizes the pores are created uniformly all over the particles using high intensity ultrasound irradiation. SEM micrographs of dispersion of as-received WO_3 nanoparticles and sonochemically modified WO_3 nanoparticles are shown in Figure 1(c) and (d) respectively. It is noteworthy to mention that the infusion of as-

received WO_3 in epoxy resulted in clear separation of particles and epoxy as seen in Figure 1(c). This indicates that the particles are high in density and they are not compatible with the epoxy resin. Whereas the infusion of sonochemically modified WO_3 nanoparticles resulted in uniform dispersion of nanoparticles over the entire volume of epoxy resin presented in Figure 1(d). To study the real effect of dispersion on mechanical properties we have carried out the compressive behaviour of $\text{WO}_3/\text{SC-15}$ epoxy nanocomposites.

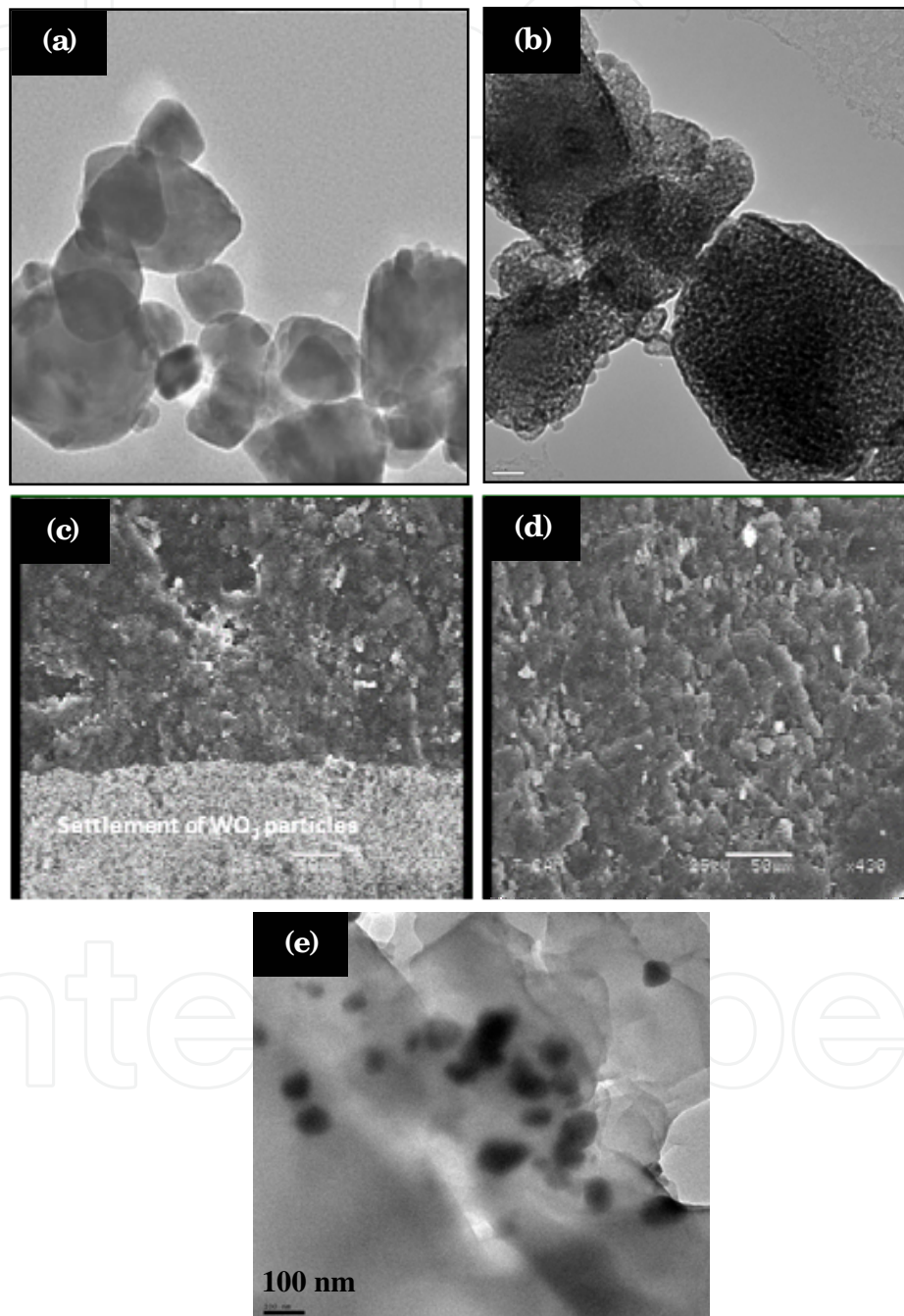


Fig. 1. TEM micrograph of a) as-received WO_3 nanoparticles, b) sonochemically modified WO_3 nanoparticles, c) SEM images of as-received 1wt% WO_3 nanophased SC-15 epoxy nanocomposite and (d) and (e) SEM and TEM micrographs of sonochemically modified 1wt% WO_3 nanophased SC-15 epoxy nanocomposite respectively.

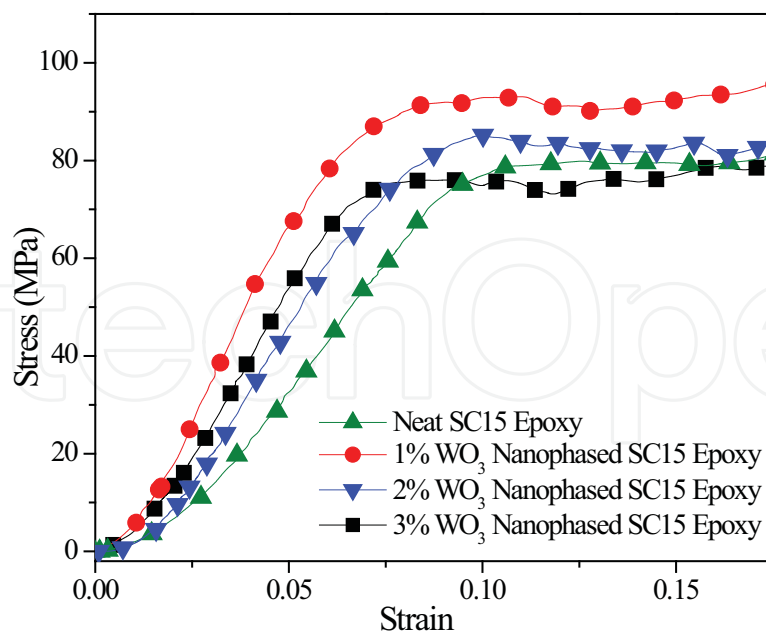


Fig. 2. Compressive stress-strain curves of neat and nanophased SC-15 epoxy nanocomposites

The quasistatic compression tests were carried for neat and nanocomposite samples. Stress-strain curves of 1wt% 2wt% and 3wt% of WO₃/SC-15 epoxy along with neat SC-15 epoxy are shown in Figure 2. It is observed from Figure 2 that the compressive strength and modulus of the 1wt% WO₃/SC-15 epoxy system is 92 MPa and 1773 MPa respectively. Whereas the neat SC-15 epoxy compressive strength and modulus are 79MPa and 894MPa respectively. These results shows that the 16% increase in strength and 98% increase in modulus as compared to the neat SC-15 epoxy system. The 1wt% loading of WO₃ nanoparticles shows the highest mechanical properties among the 2wt% and 3wt% loading of WO₃ nanoparticles. The higher (2wt% and 3wt%) percentage loading of WO₃ nanoparticles shows the inferior properties compared to the 1wt% loading of WO₃ nanoparticles but still they are higher than the neat SC-15 epoxy polymer. The reason for this may be explained as particle loading increases the resulting composites will begin to see more and more particle-to-particle interaction rather than the intended particle-to-polymer interaction. Particle to- particle interaction will lead to agglomerated particles and poor mechanical properties. These mechanical properties are consistence with the thermal and microscopic properties. Figure 2 also contain a stress plateau region that indicates that these composites have an ability to absorb compressive damage. These curves are similar to those observed by other researchers (Gupta & Ricci, 2006).

Because of the wide spread applications of polymer nanocomposites they have created a new opportunity for the development of high performance, smart and multifunctional nanocomposites materials. Recently nanocomposites have shown significant improvements in their mechanical properties. However the advancement of synthetic methods for developing new types of nanofillers in the last two decades has sparked an opportunity for the development of multifunctional nanocomposite materials. A single material with more than two functionalities such as improved mechanical properteis along with magnetic, electrical optical, and sensing capabilities. It is also know in the literature that the decoration of CNTs with metal or metal oxides can improve dispersion of CNTs in solvents (Oh-Kil et al., 2003; Chin et al., 2004; Oymak et al., 2004; Watts et al., 2002; Wu et al.,2002) or reveal new

optical, electric, magnetic properties of CNTs (Lidström et al, 2001; Alexandre et al., 2000; Wiesbrock et al., 2004; Kerep et al., 2006). To address some of the multifunctionalities of a materials we have prepared the iron oxide nanoparticled coated on CNTs. These nanoparticles were further used to fabricate Fe_3O_4 /CNTs epoxy nanocomposite and tested for their mechanical and microscopic properties. Prior to curing, the epoxy resin was mixed with known weight percentages of CNTs (0.5 wt% and 1 wt %) using a non-contact (Thinky hybrid defoaming mixer ARE-250) mixing for 10 minutes. A stoichiometric amount (26 wt %) of the curing agent (epicure W) was added to the reaction mixture containing epoxy and CNTs or Fe_3O_4 /CNTs and mixed for another 10 minutes. The mixture was degassed using a vacuum oven for 30 minutes and the final reaction mixture was transferred into a stainless steel mold for curing. The epoxy resin with and without CNTs or Fe_3O_4 nanoparticles were cured at 120°C for 2 hours and post-cured for 2 hours at 150°C in an oven. The as-fabricated nanocomposite is tested for dispersion and mechanical properties.

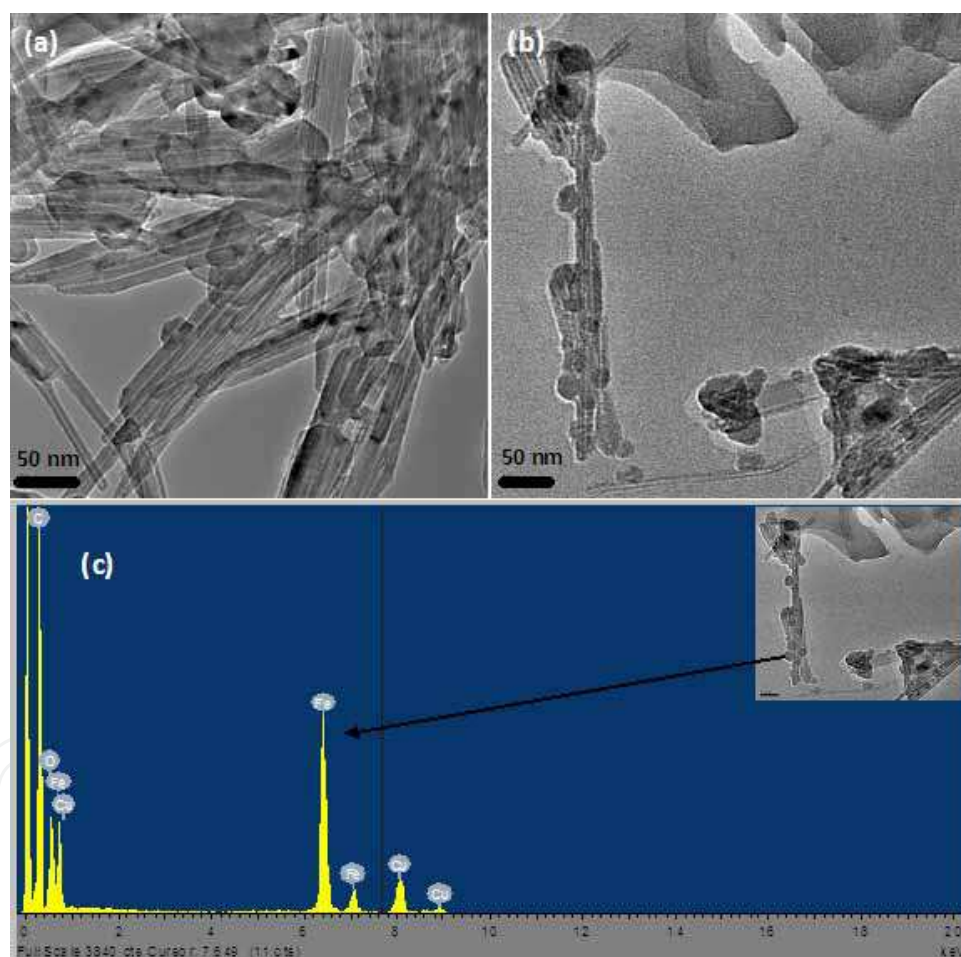


Fig. 3. TEM micrographs of (a) pristine CNTs dispersion in Epon-828, (b) Fe_3O_4 nanoparticles coated on CNTs dispersion in Epon-828, and (c) EDS micrograph showing Fe_3O_4 nanoparticle on CNTs in Epon-828 epoxy

TEM studies were carried out to study the dispersion of CNTs and Fe_3O_4 /CNTs in the Epon-828 resin. The TEM micrographs of nanocomposite samples of a) 0.5wt% CNTs/Epon-828 and b) 0.5wt% Fe_3O_4 /MWCNTs/Epon-828 are presented in Figure 3(a) Figure 3(b) respectively. Figure 3(a) shows that the CNTs are well dispersed in the Epon-828 resin. As

seen in micrograph (Figure 3(a)) the CNTs are completely covered with resin and particles. They are well dispersed in the Epon-828 resin as compared to the pristine CNTs (Figure 3(a)). The dispersion of Fe_3O_4 coated CNTs in Epon-828 resin is shown in Figure 1(b). This Figure clearly shows that the Fe_3O_4 coated CNTs are well dispersed over the entire volume of the Epon-828 resin with a no agglomeration. In this micrograph we can also see that the Fe_3O_4 nanoparticles adhere on the CNTs after dispersion in the Epon-828 resin. This clearly shows that the noncontact mixing method is efficient in mixing the coated nanoparticles in Epon-828 resin. To confirm the coating of Fe_3O_4 nanoparticles on CNTs the EDS analysis was carried out and the results are presented in Figure 3(c). These results show the presence of Fe, O and C elements at nanoscale. The Cu peaks are assigned to the copper grid.

To study the effect of coating of CNTs on mechanical properties, flexural properties of the Fe_3O_4 /CNTs- Epon-828 epoxy were measured. Flexural specimens were prepared according to ASTM standard D790-01. The specimens were tested using Zwick/Roell Material Testing Machine. The load cell used on the Zwick/Roell machine is approximately 2.5 kN. The test was carried out in displacement control mode and the cross-head speed was 2 mm/min. TestXpert software was used to analyze the load-deflection data recorded by the data acquisition system. A Flexure test produces a tensile stress in the convex side of the specimen and compression stress in the concave side. This creates an area of shear stress along the midline. Flexure test measures the force required to bend a specimen under 3 point loading condition. In a 3-point test the area of uniform stress is quite small and point concentrated under the center loading point. The Flexural test measures were carried out under a 3 point bend loading conditions at ambient temperature.

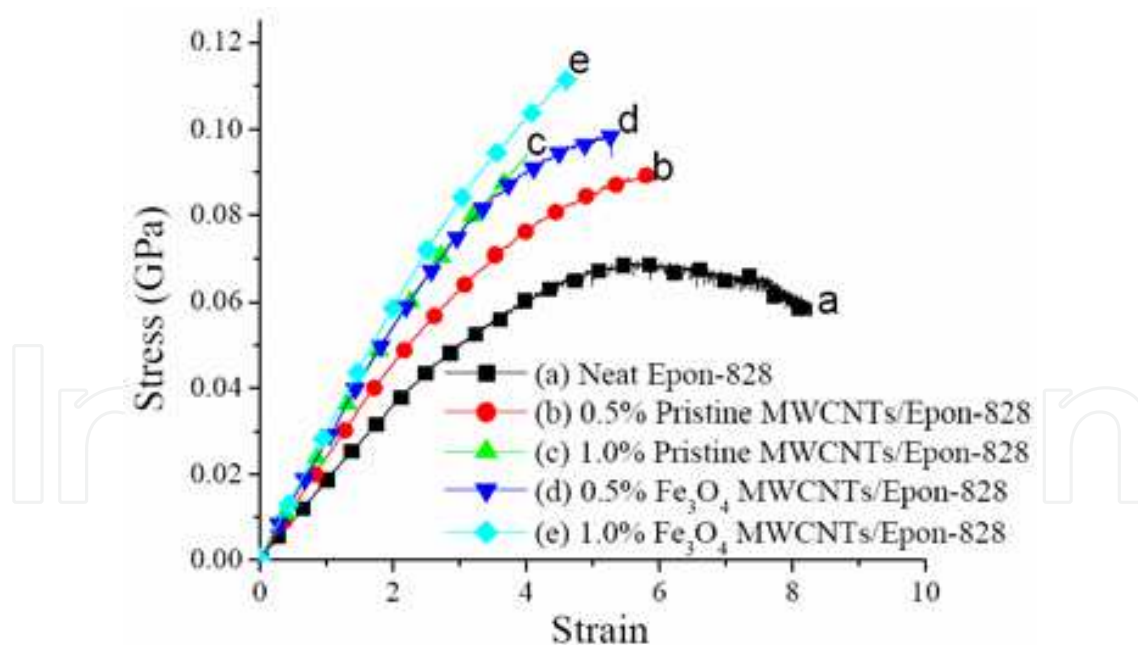


Fig. 4. Flexural stress-strain curves of a) neat Epon-828, b) 0.5wt % of Pristine MWCNTs c) 1wt% of Pristine MWCNTs d) 0.5wt% of Fe_3O_4 coated MWCNTs/Epon-828, and e) 1wt% Fe_3O_4 coated MWCNTs/Epon-828

The stress-strain curves of the neat Epon-828 and coated CNTs and uncoated CNTs infused Epon-828 resin are shown in Figure 4. The Figure 4 shows flexural stress-strain curves of a) neat Epon-828, b) 0.5wt % of uncoated CNTs c) 1wt% of uncoated CNTs d) 0.5wt% of

magnetite coated CNTs and d) 1wt% magnetite coated CNTs. The rest results were presented in Table 1. These results show that there is a gradual increase in the flexural modulus and strength. With the addition of the pristine CNTs (1wt%) and coated CNTs(1wt%) the strength increases to 20% and 50% respectively as compared to the neat Epon-828 epoxy.

Sample	Ultimate Flexural Strength GPa	Flexural Modulus GPa
Neat Epon-828	0.078	2.19
0.5% Pristine/ CNTs/Epon-828	0.093	2.34
1% Pristine /CNTs/Epon-828	0.094	2.67
0.5% Fe ₃ O ₄ /CNTs/Epon-828	0.099	2.70
1% Fe ₃ O ₄ /CNTs/Epon-828	0.117	2.84

Table 1. Flexural properties of neat Epon-828 and nanocomposite systems

There is an ever-increasing demand for development of processing of polymer nanocomposites for advanced structural applications. The manufacturing industries of these nanocomposites are in need of quick and efficient curing method for high temperature epoxy polymer. Over the last five decades the applications of microwave radiation has been increased tremendously in various scientific research fields. The application of microwave heating in curing processes of composites are very limited because of the complexity involved in heat absorption of multiple components at different rates simultaneously. In polymer composite fabrication, curing (polymerization) step is crucial and time-consuming process. Curing can be done by either at room temperature or at high temperature but both types of curing take prolong time (one or two days) (Lidström et al, 2001). This is ultimately adds up to the final cost of the product.

Recent studies have shown that the nanocomposites exhibit remarkable improvements in stiffness, strength, physio-chemical and thermal properties without compromising on density, toughness, or processibility compared to their micro and macro composite counterparts (Alexandre, 2000). Field of polymer nanotechnology is still unrefined in certain aspects, curing is one of them. Novel techniques of curing bring out new breakthrough and there are avenues in nanotechnology, which have not been discovered yet. The bulk manufacturing and energy saving curing methods of nanocomposites is one such major unexplored research areas. The conventional method of curing is time-consuming, the longer it takes to complete a project, the more expensive it becomes. Alternate curing methods have been tried and tested for quite some time now. They are: UV rays, Gamma rays, Electron Beam and Microwave (Bogdal et al., 2003; Clark & Sutton, 1996). Curing using UV light has limited application because of its poor penetrability and limited dose rate; it cannot cure thick materials (Wiesbrock et al., 2004). Gamma rays induced radiation hazard and environmental issues. The most prominent alternatives suggested are Electron Beam (EB) and Microwave heating (Kerep & Ritter, 2006; Sinnwell & Ritter, 2006). Electron Beam curing though efficient and fast unfortunately requires high capital cost for initial setup (Ghosh & Palmese, 2005). It has some other drawbacks like low glass transition temperature

(or low service temperature), low fracture toughness, and high shrinkage when cured (Ghosh & Palmese, 2005). Microwave curing, therefore, is the more economically feasible alternative and is remarkably energy efficient (Soane & Martynenko, 1989; Jacob et al., 1995; Zhou & Hawley, 2003). Microwave heating is unique in the fact that heat is generated in the specimen rather than externally transferred. The potential for microwave curing stems from the fact that there are no major drawbacks for this method. The curing of nanoparticles infused epoxy using microwaves is a relatively unexplored area of research. A novel method has to be formulated from testing different conditions of cure selecting from factors such as power level of microwave, time to cure, percentage of nanoparticles infusion, method of infusion and finally relative cost than other technique. Recently (Papargyris et al., 2008) reported a 50% cure cycle time reduction through the use of microwave curing of carbon fibre epoxy composite prepared by RTM technique without losing its mechanical and thermal properties. (Sandhya et al., 2006) also reported that the microwave curing of the glass-reinforced epoxy resin system is thirty times faster and twenty times more energy-efficient as compared to the oven cured. They also reported that the mechanical performance of the microwave-cured glass-epoxy composite under tension, compression, and flexure, is in close comparison to the thermally cured glass-epoxy composite.

Recently, several studies have been conducted to investigate the interaction between microwave radiation and CNTs. (Mendez, 2003; Watts, 2003; Wu, 2004; Watts, 2003; Grimes et al., 2000; Petit et al., 1997; Imholt et al., 2003). Very recently Chin and his group (Wang et al., 2007) reported the strong bonding between CNT and polymer by microwave heating. Still more studies are required to develop optimal processing techniques to speed up microwave processing for industrial applications. A novel method has to be formulated from testing various polymer cure conditions, microwave power levels, time of exposure to complete cure, percentage of nanoparticles infusion, method of infusion and finally relative cost than other technique.

In this current study, CNTs infused in EPON 862 nanocomposites were prepared with different weight percentage of CNTs by thermal curing (TC & TPC) followed by post curing and microwave curing (MWC). Compression properties were studied. The CNTs were dispersed through acoustic cavitations process. Part-A of EPON 862 was selected for infusion of CNTs because of its less reactivity to ultrasonic irradiation. Pre-calculated amount of CNTs and part A were carefully weighed and mixed together in a beaker. The reaction mixture was irradiated with ultrasonic horn (Ti-horn, 20 kHz, 100W/cm²) at 50% of amplitude for 15 minutes. In order to avoid a temperature increase during the sonication process, external cooling was employed by submerging the mixing beaker in a thermostatic bath at 10°C for the entire period of the ultrasonic irradiation. The Part B was mixed with modified Part A using a THINKY hybrid de-foaming mixture ARE-250 at 2000 rpm for 15 minutes. The trapped air and initial reaction volatiles were removed from the mixture by using a vacuum desiccator. Finally the mixture is cured using a 2.45GHz (Microwave Processing Oven BP210) microwave oven for only 10 minutes instead of 8 hours of conventional oven heating (curing cycle is 4 hrs @ 120°C and post curing @ 170 °C for 4hrs). Same manufacturing procedure was followed for fabrication of neat EPON 862 epoxy sample without CNTs. Thermal and mechanical tests were performed for all samples including thermally and microwave cured samples.

Compression test is carried out to measure the load carrying capacity of a material before fracture. It can be seen in Figure 5 that stress-strain curves have two major distinct phases – an initial elastic response and a protracted plateau. The elastic region is controlled by the

stretching of the EPON-862 polymer. The stress plateau is associated with the time taken to form matrix cracking and crack propagation followed by barreling/bulging and increase in the lateral dimension of the rectangular samples.

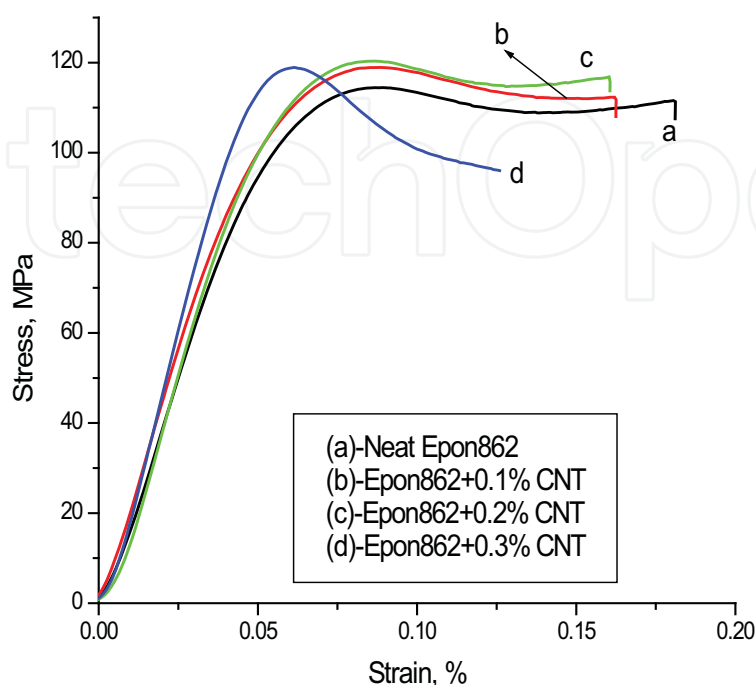


Fig. 5. Compression stress-strain graphs of microwave cured (a) neat EPON-862 (b) EPON-862+0.1wt% CNTs (c) EPON-862+0.2wt% CNTs (d) EPON-862+0.3wt% CNTs.

Figure 5 is the compression graphs of microwave cured neat EPON 862 and EPON 862 with 0.1wt%, 0.2wt%, and 0.3wt% of CNTs loading. The compression strength/modulus calculated from the graph are: 114.44 MPa/2.24 GPa, 118.97 MPa/2.40 GPa, 120.29 MPa/2.52GPa and 118.92 MPa/2.32 GPa for neat EPON 862, EPON 862/0.1wt% CNTs, EPON 862/0.2wt% CNTs, and EPON 862/0.3wt% CNTs respectively. These results show that the 0.2wt% of CNTs infusion in EPON-862 compression modulus (28.57%) and compression strength (5.57%) increased with respect to neat EPON-862 TC and TPC. These results also summarized in table 2 for comparison. The reason for the increase in strength and modulus is may be due to the welding mechanism (Wang et al., 2007). Which is thought to involve the wetting of the CNTs by the surrounding polymer melt; a nanometer-sized melted polymer region is likely formed around the nanotubes due to the dissipation of heat from the CNTs to polymer. This local melting leads to physical intercalation of the CNTs and the polymers and yielding a bonding strength two order of magnitude higher than that obtained for polymer bonded together by epoxy adhesive.

3. Polymeric foam core nanocomposites for lightweight and high strength applications

Polymeric foam materials are widely used in many industrial applications for their properties of light weight, excellent strength to weight ratio, superior insulating abilities, energy-absorbing properties, low thermal conductivity, high sound absorption, and large compressive strains. The main applications include sandwich structures, airframes,

transportation vehicles, boat hulls, radar systems, and space structures (DIAB Inc, 2002; Rohm, 2002; Baltek Corp, 2002; Marsh, 2002). High-performance structural foam materials are fabricated using a blowing agent (surfactants, hydrocarbons) in liquid polymers to expand and form rigid, low-density foams. Some of the leading thermoplastic foams made in this way are polymethacrylimide (PMI) and partly cross-linked polyvinyl chloride (PVC), with trade names Rohacell Rohm, 2002, Divinycell DIAB Inc, 2002; and Expancel Expancel®, 1980). Hollow thermoplastic microspheres are produced under the trade name of Expancel®. These microspheres are small, spherical plastic particles consisting of a polymer shell encapsulating a hydrocarbon gas. When the gas inside the shell is heated, its pressure increases and the thermoplastic shell softens, resulting in a dramatic increase in the volume of the microspheres (Elfvig & Soderberg, 1994). Researchers are using these microspheres for various applications such as car protection, corrosion resistance, acoustic insulation, body fillers and marine hobby putties, underbody coatings and synthetic wood Expancel (Expancel®, 1980; Tomalino & Bianchini, 1997). Young-Wook and his coworkers (Kim et al., 2004) developed a closed-cell silicon oxycarbide foams with cell densities greater than 10^9 cells/cm³ and cells smaller than 30 μ m were obtained from a preceramic polymer using expandable microspheres. (Vaikhanski et al., 2004) were also studied the reinforcement of microspheres in PVC with the aramid fibers and reported the improved mechanical properties.

Expancel-092-DU-120 is unexpanded thermoplastic polymeric powder (particles sizes 28-38 μ m) was received from Expancel Inc. Multi walled carbon nanotubes (CNTs-10-20nm in diameter and 0.5-20 μ m in length) were purchased from Nanostructured & Amorphous materials. The experimental procedure for the coating of CNTs on thermoplastic microspheres and the fabrication of CNTs/thermoplastic microspheres foam panels are as follows: Expancel polymeric powder and a known weight percentage of CNT were dispersed in n-hexane using a high intensity ultrasonic horn (Ti-horn, 20 kHz, and 100 W/cm²) at room temperature for 1-hour. The mixture was then dried in a vacuum for 12-hours and remaining n-hexane was removed by heating the sample at 60°C for 1-hour. The dry CNTs/thermoplastic microspheres mixture was transferred to a rectangular aluminum mold (4"X 4"X1/2") and uniformly spread over the entire volume of the mold. The mold was then heated to ~190°C at a heating rate of 10°C/min for 30 min under a pressure of 15 atm using a MTP-14 programmable compression molding equipment. The as-prepared samples were cut precisely and used for morphological thermal and mechanical testing. The densities of as-prepared foam samples of CNTs / microspheres (nanocomposite) and thermoplastic microspheres only (neat) were measured and presented in Table 2. The as prepared foam samples without CNT are ~ 154 Kg/m³ and with CNT are ~ 153Kg/m³. The smaller variations in densities of nanocomposite and neat foam samples are due to the density differences in as-received microspheres and CNTs.

To examine this we have carried out the SEM analysis at higher magnification as shown in Figure. 6. These results show that the CNT are uniformly coated on the expanded microspheres. This uniform coating with adhering of the MWCNT on microsphere translated in to the enhanced compression strength and modulus. Compression tests were carried for neat and nanocomposite foams. Nanocomposite (1% and 2 by wt %) and neat were tested. Stress-strain curves for neat and nanocomposites are shown in Figure. 7. It is observed that the compressive stress of the 2 wt% nano system is about 51 wt% higher than the neat sample. The 2 wt% nano system also shows 40 wt% improvements in compressive

modulus. This improvement may be the result of increasing the interfacial bond between the nanoparticles and polymeric matrix.

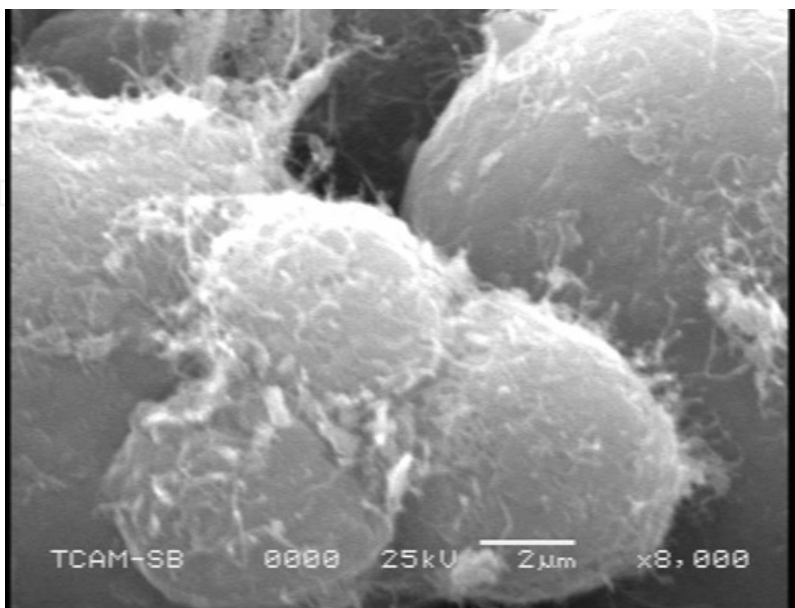


Fig. 6. Coating of CNTs on Expancel microspheres

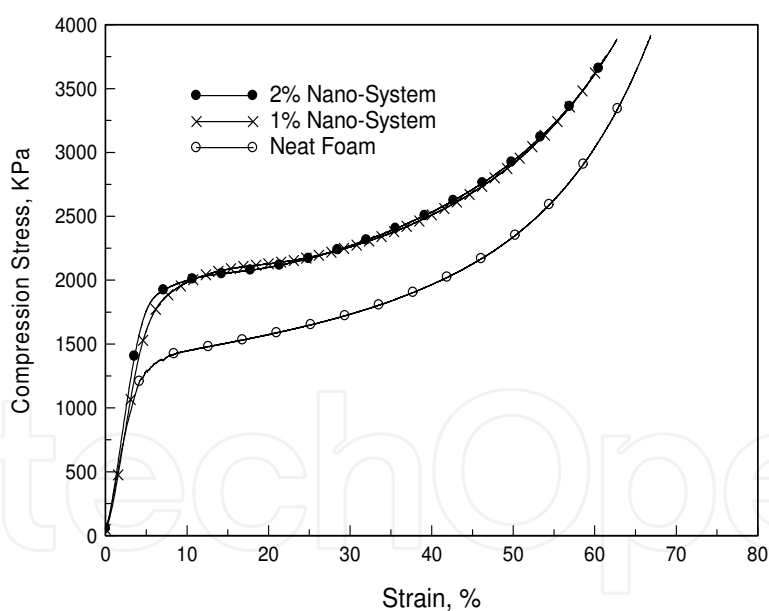


Fig. 7. Compressive stress-strain curves are neat, 1% CNT and 2% CNT nanocomposite foam. Expancel polymeric powder and the 1wt% of SiC or SiO₂ (1wt% of SiC or SiO₂ and 99wt% expancel power) was dispersed in n-hexane using a high intensity ultrasonic horn (Ti-horn, 20 kHz, 100 W/cm²) at room temperature for 1hour. The mixture was then dried in a vacuum for 12hrs and excess n-hexane was completely removed by heating the sample to 60°C for 1-hour. Nanocomposite foam panels were fabricated by heating the dry powder of SiC or SiO₂ coated polymeric powder to ~190°C at a rate of 10°C/min for 30min in a rectangular aluminum mold (4"X 4"X1"). The mold was kept under the pressure of ~3000lbs

using a programmable MTP-14 compression molding machine. The test samples were cut precisely form the as-prepared panel and used for the morphological and mechanical testing.

Material	Compressive Strength [KPa]	Gain/Loss [%]	Compression Modulus [MPa]	Gain/Loss [%]
Neat Foam	1200±50	-	30±3	-
1wt %SiC nanocomposite foam	2230±40	+86	34.1±4.5	+13
1wt %SiO ₂ nanocomposite foam	1418±45	+18	31.5±5.5	+4

Table 2. Compression test results of various samples.

To understand the effect of SiC and SiO₂ coating on polymeric particles the compression tests were carried for neat and nanocomposite foams. Two types of nanocomposite (1wt % SiC and 1wt % SiO₂) samples and the neat samples were tested. Stress-strain curves for neat and nanophased samples shown in Figure. 8. Compression test data is presented in Table 2. It is observed in Figure. 8 that the compressive stress of the 1% SiC and 1% SiO₂ nano systems are about 86% and 18% respectively increased as compared with the neat expandcel foam samples. The 1% SiC nanocomposite also shows 13% improvement in compressive modulus. This improvement are assigned to the increasing the interfacial bond between the nanoparticles and polymeric matrix.

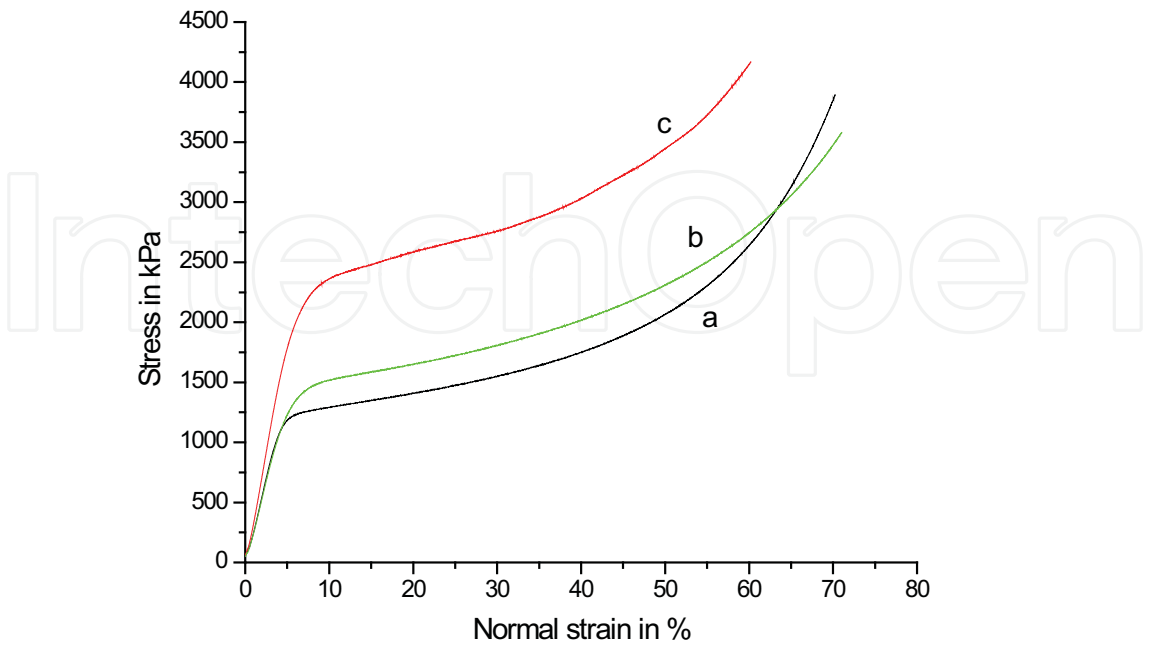


Fig. 8. Compressive stress-strain curves are a) neat, b)1% SiO₂ and c) 1% SiC nanophased expandcel foam

4. Alignment of acicular nanoparticles in thermoplastic polymers to improve the mechanical and antimicrobial properties of textile fibers

The use of nanotechnology in textile industry is rapidly growing because of their wide spread applications in healthcare, body armor, home furnishing etc. Nanoparticles such as silver, silver bromide, zinc oxide, titanium dioxide and copper oxide infused in synthetic polymers such as nylon and polypropylene can impart antimicrobial character to the textile fabrics for various applications (Sang et al., 2003; Zhang et al., 2001). Nylon-6, the semicrystalline polyamide known for its high toughness, tensile strength, low coefficient of friction and good abrasion resistance make it an ideal replacement for a wide variety of materials from metal to rubber. In the last few decades, there has been increased interest in antibacterial finishing on textile materials because of varieties of environmental pollutions. Ag is known to have a wide antibacterial spectrum and with high safety. It is a relatively non-toxic and natural inorganic metal and its nano size level makes its total surface area larger in an identity volume. Recently, Mecking and co-workers showed that hybrids of Ag nanoparticles with amphiphilic hyperbranched macromolecules were effective antimicrobial surface coating agents (Aymonier et al., 2002; Kim et al., 2007). Several salts of Ag and their derivatives are commercially employed as antimicrobial agents, as it is well known that silver ions and Ag-based compounds are highly toxic to microorganisms (Slawson et al., 1992; Zhao & Stevens, 1998) showing strong inhibitory effects on as many as 16 species of bacteria including *E. coli* (Spadaro et al., 1974). Thus, nanoparticles of Ag have aptly been investigated for their antibacterial property (Sondi & Salopek-Sondi, 2004; Panacek et al., 2006; Morones et al., 2005; Baker et al., 2005). Nanoparticles of Ag also have been studied as a medium for antibiotic delivery (Li et al., 2005), and to synthesize composites for use as disinfecting filters and coating materials. However, the bactericidal property of these nanoparticles depends on their stability in the growth medium, since this imparts greater retention time for bacterium-nanoparticle interaction. Therein lays a strong challenge in preparing nanoparticles of Ag stable enough to significantly restrict bacterial growth.

Also, the use of carbon nanotubes as fillers has increased ever since their discovery as they are considered to be the ideal reinforcing agent for high-strength polymer composites because of their tremendous mechanical strength (higher Young's modulus, ~1 TPa which is comparable to that of diamond ~1.2 TPa and good elasticity), nanometer scale diameter, and high aspect ratio (Calvert, 1999; Salvétat et al., 1999; Yu et al., 2000). Carbon nanotubes (CNTs) have been used, in the last two decades, in applications ranging from DNA detection (Kouklin et al., 2005; Saugandhika et al., 2009) to nanorobots (Baxendale, 2003). The nano composite reinforcement is dominated by the collective behavior of the bundles rather than by the strength of the individual tubules (Ajayan et al., 2000). CNTs, which were of ~10-15 nm in diameter and about 5µm long were effective in load transferring mechanism than SWCNTs, as during loading the SWCNTs tend to pull out from the ropes (bundles), thus making load transfer difficult (Guang et al., 2006). Composites containing both noble metal nanoparticles and CNTs, in which nanoparticles are attached on the surface of the CNTs, have potential application in sensors, biomedical applications and heterogeneous catalysis (Yu et al., 2009). Ag decorated CNTs (Ag/CNTs) gained extensive attention due to their potential applications as catalyst (Guo & Li, 2005) [64], advanced materials (Wu, 2006), etc. Peng Cheng et al. proposed an approach for coating CNTs with Ag- NPs based on the wet chemistry reaction (Peng et al., 2006). Generally, a stable and uniform suspension of nanotubes in the polymer is required to obtain a fine dispersion and proper alignment of Ag

coated CNTs within the fibers, which are instrumental for enhancement in structural, thermal and electrical properties of Ag/CNTs/Nylon-6 PNC fibers (Jeong et al., 2006). Ag coated CNTs (Ag/CNTs) nanoparticles were prepared by ultrasonic irradiation of Dimethylformamide (DMF) and silver (I) acetate (Aldrich) in the presence of CNTs. The as-prepared Ag/CNTs were infused into the Nylon-6 polymer fibers through an extrusion process (*Wayne Yellow Label Table Top Extruder*) and these fibers were tested for their thermal, tensile and antimicrobial properties.

Tensile tests of single fiber specimens of the Nylon-6 and composites infused with Ag, CNTs and Ag/CNTs were carried out to estimate the increase in mechanical properties, such as strength and modulus.

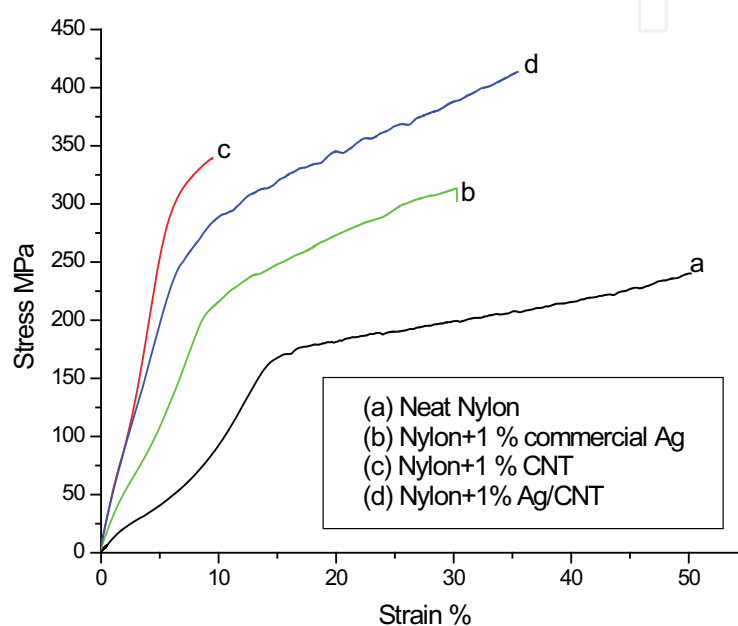


Fig. 9. Tensile response of (a) Neat Nylon-6 (b) commercial Ag-Nylon-6 (c) CNTs-Nylon-6 (d) Ag/CNTs-Nylon-6

The results of tensile properties of the neat Nylon-6, 1.0 wt% commercial Ag/Nylon-6, 1.0 wt% CNTs/Nylon-6, and 1.0 wt% Ag/CNTs/Nylon-6, are and shown in Figure 9. The ultimate tensile strength values for neat Nylon-6, 1wt% commercial Ag-Nylon-6, 1wt% CNTs-Nylon-6, and 1wt% Ag/CNTs-Nylon-6 fibers were found to be about 240 MPa, 313 MPa, 339 MPa and 414 MPa, respectively. The tensile modulus values for neat Nylon-6, 1wt% commercial Ag-Nylon-6, 1wt% CNTs-Nylon-6, and 1wt% Ag/CNTs-Nylon-6 fibers were found to be about 0.84GPa, 2.73GPa, 5.05GPa and 3.76 GPa, respectively. The tensile strength and modulus of the Nylon-6 infused with Ag/CNTs is 172% and 447% higher than the neat Nylon-6 polymer fibers. As expected we observe the highest percentage increase of modulus (650%) and relatively low (141%) increase for 1wt% CNT-Nylon-6. The significant improvement (172 and 447%) of the mechanical properties of the thermoplastic polymer by such 1wt% percentage of Ag/CNT filler is remarkable when compared with the other thermoplastic polymer composite fibers. For instance, in the recent work on polypropylene/F-SWNT composite fibers, the much higher loading of F-SWNTs (10 wt.%) was shown to yield a significantly lower tensile strength enhancement value (125%) (Daneesh et al., 2006).

We have also observed the high tensile strength of 413 MPa for 1.0 wt% loading of Ag/CNTs into Nylon-6. This measured strength value for nanocomposite fiber is more than 172% higher than that of the neat Nylon-6 which indicates the exceptional load bearing capability along with high elongation (35%) of Ag/CNTs and their potential applications in structural composite materials. In comparison with the 1.0 wt% Ag/CNTs/Nylon-6 composite fibers showing the higher tensile strength as 1.0 wt% CNTs/Nylon-6 samples, the former demonstrate better % elongation at break (Figure 7) which overall gives the best combination of mechanical properties among the Nylon-6 composite fibers we have studied. This conclusion is supported by comparison of tensile modulus values observed for neat Nylon-6, and 1wt% Ag//Nylon-6, which are 0.84 GPa and 2.73 GPa respectively. Such significant improvement of tensile modulus can be attributed to the infusion of high strength Ag/CNTs, their alignment in extrusion direction of Nylon-6 polymer fibers (Vijaya, et al., 2009 and 2008; Mahfuz et al., 2005), and the fact that nanotubes/particles do act as nucleation sites for crystallite formation. There are two different mechanisms acting in parallel to account for these results. We have also observed the decrease of elongation (9.6%) by infusion of only CNTs [(Khabashesku et al, 2006; Ignac, 2009; Feng et al., 2005). We know that the strength can be increased by infusion of CNTs in Nylon-6 fiber through melt extrusion process by alignment of CNTs in fiber direction (Vijaya et al., 2009 and 2008; Mahfuz et al., 2005). And also as explained in earlier section the strain to failure can be increases by infusion of smaller size nanoparticles in the polymer (Jeffrey et al., 2005; Mahfuz et al., 2007). In the present case the combination of smaller Ag nanoparticles coating on CNTs leads to increase in strength and stain to failure

5. Development of shear thickening fluid for liquid body armor applications.

Shear thickening is a non-newtonian flow behavior observed as an increase in viscosity with increasing shear rate or applied stress (Barnes, 1989). This phenomenon can occur in colloidal dispersions. In more concentrated colloidal suspensions have been shown to exhibit reversible shear thickening resulting in large, sometimes discontinuous increases in viscosity above a critical shear rate. Two main causes of reversible shear thickening have been proposed: The order-disorder transition [(Hoffmann,1972, 1974 & 1983; Boersma, 1992; Laun et al., 1992) and the “hydrocluster” mechanism (Bender & Wagner, 1995 & 1996; Phung et al., 1976; Melrose et al., 1996; Farr et al., 1997; Brady et al., 1988). This transition from a flowing liquid to a solid-like material is due to the formation and percolation of shear induced transient aggregates, or “hydroclusters,” that dramatically increase the viscosity of the fluid. Recently Wegner’s group and U.S .Army research lab developed a body armor using shear thickening fluid and Kevlar fabric (Young et al., 2003). These research results demonstrate that ballistic penetration resistance of Kevlar fabric is enhanced by impregnation of the fabric with a colloidal shear thickening fluid. Impregnated STF-fabric composites are shown to provide superior ballistic protection as compared with simple stacks of neat fabric and STF. Comparisons with fabrics impregnated with non-shear thickening fluids show that the shear thickening effect is critical to achieving enhanced performance. In the present investigation we report on the synthesis of STF using sonochemical method, which can be potentially used for the body armor applications. Many researchers have used various techniques to prepare the shear thickening fluid. Acoustic cavitations technique is one of the efficient ways to disperse nanoparticles into the liquid polymers. In this study we have synthesized a shear thickening fluid in a single step reaction through high power ultrasound

technique, fabricate STF/fabric composite and characterize it for stab resistance applications. The rheology test results show that as-prepared STF sample exhibits shear thickening behavior. This behavior is very much evident as seen from the graph in Figure 10. The sample viscosity changes from ~ 20 Pa.S at shear rate of 5/S to 410 Pa.S at 12/S shear rate before reversal trend is seen. Continuous increase in sample's viscosity at low and very narrow shear rates range clearly confirm that the sonochemical mixing before evaporation drastically improves the shear thickening effect.

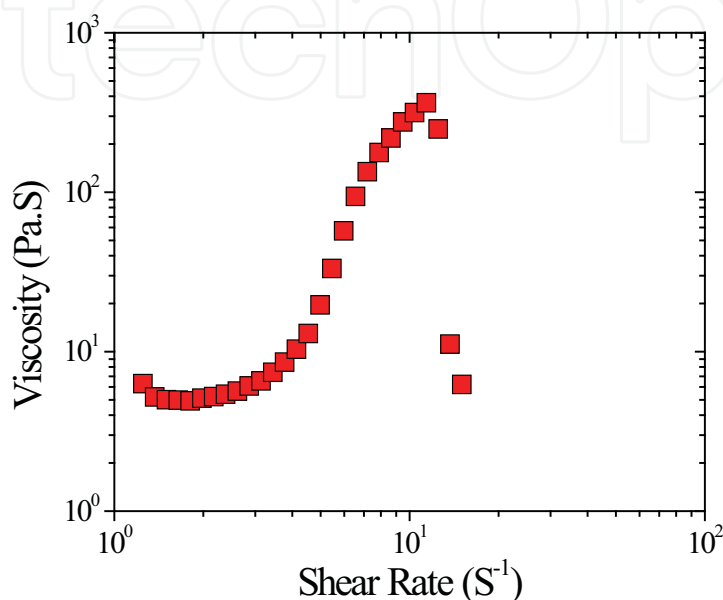


Fig. 10. Rheology graph for as prepared STF sample

Figure 11a shows the drop tower stab performance of neat Kevlar and STF/Kevlar composite targets for the knife and spike threats. As seen in the graph for the knife threat the penetration depth increases as the impact energy increase. STF/Kevlar composite target exhibit slightly less penetration depth and better stab resistance compared to the neat Kevlar target for low values of impact energy. At impact energy around 8 joules both targets reach maximum penetration depth of 5 witness papers. For the spike threat, as impact energy increases, depth of penetration into the backing material also increases. The STF/kevlar composite target exhibits significantly better stab resistance as compared with the neat kevlar target. The neat kevlar target exhibits maximum penetration of 5 witness papers at all impact energy levels. In contrast, STF/kevlar composite only penetrated through 1 witness paper at impact energy of 2.7 Joules and continued to resist the penetration, then showed maximum penetration of 5 witness papers at impact energy of ~ 8 Joules.

The results for neat nylon and STF/nylon composites stab testing for the knife and spike threats are presented in figure 11b. The results show the same trend as impact energy increases, depth of penetration into the backing material also increases. The STF/nylon composite target demonstrates slightly less penetration depth than the neat nylon target for the knife stabbing test.

As seen in the graph STF/Nylon composite target shows only 2 penetrated witness papers at impact energy of 2.7 Joules and when the impact energy reached a value of 5.4 Joules 3 witness papers were penetrated, the fourth witness papers penetrated at impact energy of 8.1 Joules and after that all 5 witness paper were penetrated for higher impact energy levels.

On the other hand the neat nylon target showed maximum penetration of 5 witness papers at impact energy of ~ 8 Joules. The results for the spike threat showed a significant improvement of the stab resistance of the STF/nylon composite target over the neat nylon target with just one penetrated witness paper in the first impact energy level of 2.7 Joules. The neat nylon target did not show any resistance for the spike stab testing with maximum of 5 witness paper penetrated for all impact energy levels. Dynamic stab test results show a significant improvement of target stab resistant and protection in STF/Fabric composite targets compared to neat fabric targets for both spike and knife threats.

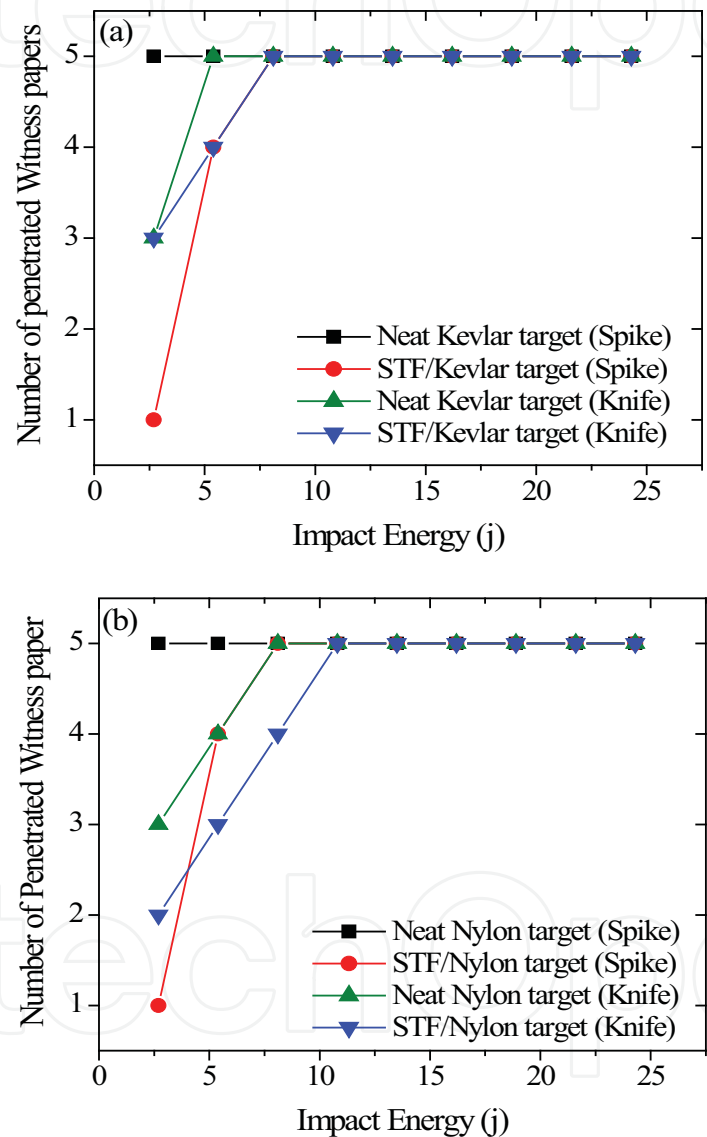


Fig. 11. Dynamic stab test results for (a) neat Kevlar and STF/ Kevlar composite (b) neat nylon and STF/ nylon composite

6. Conclusions

Application of high intensity ultrasound irradiation is proven to be a versatile technique for synthesis, dispersion, and modification of nanoparticles. We have shown here that high intensity ultrasund can be used in synthesis of nanoparticles, modification of surfaces or

uniform dispersion various nanoparticles depending on the solvent, precursor and resin system. In case of WO_3 nanoparticles, the high density commercial WO_3 nanoparticles are modified to porous nanoparticles and reduced the density and increased the dispersion in epoxy. The high resolution TEM micrographs show that porous particles well dispersed in the epoxy resin when compared to nonporous high density nanoparticles. These effects were observed in increasing the mechanical strength of the polymer. We have also shown that the dispersion and synthesis of nanoparticles can increase the mechanical properties which will ultimately lead to the high structural applications. The other aspect of nanocomposite fabrications is curing. The room temperature and microwave curing techniques were also used and shown the applications of nanoparticles in curing with microwave irradiation. The focus is to evaluate the mechanical properties of the neat EPON-862 and CNTs infused EPON-862 nanocomposites to study the fast curing microwave method as good as or better than the prolonged curing regular oven heating method. Improvements are observed for all samples with CNTs infusion in EPON-862 resin irrespective of curing methods. The compressive modulus/strength is increased about 28.57%/ 5.5% for MWC EPON-862/ 0.2wt% CNTs respectively as compared to the neat EPON-862 TC and TPC. These results are significant when consider the timesavings.

The coating of nanoparticles on various surfaces including CNTs and polymeric spheres were also shown. Mechanical tests also indicate that there is a significant increase in strength and modulus about 16% and 98% respectively. This technique can be applied to the other commercially available high density nanoparticle to reduce their sizes for better dispersion in epoxy resin systems for structural applications. High intensity ultrasound is also used in situ synthesis of Fe_3O_4 and Ag nanoparticles on CNTs. Flexural stress-strain results also indicate 50% increase in strength and 30% increase in modulus. TEM shows improvement in dispersion of CNTs in Epoxy system with magnetite coating. Ag/CNTs hybrid nanoparticles were synthesized using sonochemical technique. Neat Nylon-6, commercial Ag, pristine CNTs, and Ag/CNTs infused Nylon-6 polymer composites were fabricated using single screw melt extruder. TEM studies reveal that Ag nanoparticles were uniformly coated on CNTs surfaces and were non-covalently attached through van der Waals forces. The improvement in ultimate tensile strength and elastic modulus is attributed to the alignment of the Ag/CNTs nanoparticles along the direction of extrusion. The increase in thermal stability and crystallinity of Ag/CNTs infused Nylon-6 PNC is correlated with the better cross linking between the nanoparticles and the polymer matrix. The in-situ 1% Ag/CNTs Nylon-6 nanocomposite fibers were demonstrated to have excellent and promising antimicrobial activity as compared to the commercially available Ag nanoparticles, pristine CNTs and neat Nylon-6. It is also noteworthy to mention that this sonochemical synthesis technique can be used to synthesize Ag/CNTs composites in bulk quantities. This technique is also tested to coat uniformly CNTs SiO_2 or SiC on expandable microspheres. These foam materials can be used in many light weight high strength applications. Quasi Static compression test results indicate that there is a significant increase in compressive strength (45- 51%) and modulus 20-40% as compared to neat system. This method can be efficiently used to synthesize shear thickening fluid as well with improved rheological properties and it is developed to prepare STF/ethanol solution in a single step. STF/fabric composites targets exhibit better stab resistance as compared neat fabric targets, this demonstrate that the impregnation of the fabric with a shear thickening fluid have enhanced the fabric performance for body armor applications. The flexibility and thickness of STF/fabric composites are comparable to the neat fabrics.

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8. References

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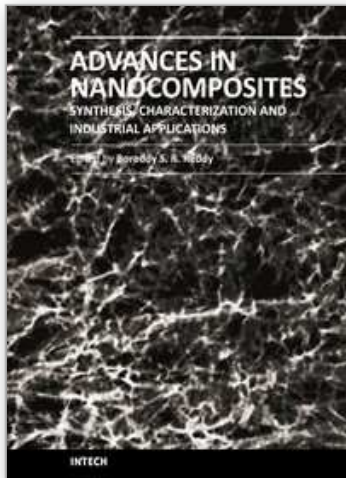
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Advances in Nanocomposites - Synthesis, Characterization and Industrial Applications was conceived as a comprehensive reference volume on various aspects of functional nanocomposites for engineering technologies. The term functional nanocomposites signifies a wide area of polymer/material science and engineering, involving the design, synthesis and study of nanocomposites of increasing structural sophistication and complexity useful for a wide range of chemical, physicochemical and biological/biomedical processes. "Emerging technologies" are also broadly understood to include new technological developments, beginning at the forefront of conventional industrial practices and extending into anticipated and speculative industries of the future. The scope of the present book on nanocomposites and applications extends far beyond emerging technologies. This book presents 40 chapters organized in four parts systematically providing a wealth of new ideas in design, synthesis and study of sophisticated nanocomposite structures.

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