



Dispersion of SiO₂ Nano particles on Epoxy Based Polymer Nano composites and Its Characterization

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ABSTRACT

In present work we developed and characterized the thermosetting epoxy polymer nano composites containing different weight percentage (wt.%) of SiO₂ nano particles ranging from 1 to 4 wt.% were prepared through solvent casting route. grain analysis, FTIR, shape recovery, impact stress, tensile, flexural strength and hardness were studied. The properties of composites were improved at 3 wt.% SiO₂ nano particles, further loading of nano particles beyond 3 wt. % properties goes to suppressed which may depends on the dispersion of particles within matrix. Shape recovery test was done in this epoxy which may open the new gate for low cost shape memory polymer as we can say novelty of this experiment. The distribution and size of nano particles in epoxy based polymer matrix were examined through AFM analysis finally outlined.

Keywords: Polymer, Epoxy, SiO₂, Nano particles, FTIR, AFM etc.

INTRODUCTION

Epoxy based composites materials are gaining popularities in the world wide in last decades because of its light weight, ease of fabrications, excellent electrical insulator, high water and chemical resistance, low price and excellent heat and moisture resistance¹⁻². Polymer nano composites are widely accepted materials for researchers now days. Polymers are material which exhibits excellent properties from other metallic and ceramics materials³. Polymer materials are the much light in weight, very good corrosion resistance

capacity, electrical insulator, water resistance, ease of fabrication but low in strength. So that for improving the strength of polymers secondary phase high strength nano particles and fibre introducing inside the polymer matrix as desired proportions⁴. Epoxy based polymers nano composites is a high performance of thermosetting which is widely used in aerospace, medicals appliances, automobiles, industries and robotics now a days worldwide⁵⁻⁷. Whenever epoxy polymer has low strength, toughness, hardness due to their highly cross linked structures^{2,8,9}. This cross link structures promotes the brittle behavior resulting the crack initiating



and growth. For improving the properties of epoxy polymer micro and nano particles of SiC, TiC, and SiO₂ were reinforced into the matrix¹⁰⁻¹². Many researches^{8, 9, 11}, also reported that, by reinforcing suitable natural fiber such as sisal, bamboo, banana etc. enhance the mechanical properties of polymer composites. Reinforcing the nano particles in polymer matrix gives better results within the 3 wt.%, further increasing the reinforcement of nano particles the properties suppressed due to agglomeration of particles^{4,11}. An addition of SiC, TiC, SiO₂ nanofillers in epoxy matrix leads to enhance the mechanical properties of base polymer, specially toughness, tensile strength, flexural strength etc. the properties of nano composites strongly depends on the dispersion and loading weight percentage of nano particles¹². The addition of nano fillers tends to increase the interface area as compared with the ordinary polymers¹³. This large interfacial area creates a large volume fraction of interfacial composites with the different properties from the bulk polymers even at the very low percentage of nanofillers^{1,9,13}. In present work, an attempt has been made to dispersion of SiO₂ nano particles in epoxy polymer matrix by homogeneous dispersion of nano particles by ultra-sonication and followed by magnetic stirrer and the effect of particles loading within the matrix and their dispersion on mechanical properties.

EXPERIMENT AND MATERIALS

In present experiment the three components thermosetting hardener (HY951) and resin (LY556), and nano size silicon oxide particles (SiO₂) with size 15-35 nm. These were purchased which is supplied by Nano Shel Pvt. Ltd. India. As suggested by supplier the glass transition temperature of this epoxy was 70°C and mixing ration resin: hardener is 15:1. Both the epoxy resin and hardener were pre sintered at 70°C in muffle furnace to reduce the viscosity for 30 minutes. To make the desired proportional SiO₂ nano particles is added various weight percentage (wt. %) ranging from 1 to 4 wt. % with hardener. And then desired proportion of SiO₂ nano particles were mixed in pre sintered hardener with the help of magnetic stirrer for 30 minutes. To

facilitate a homogeneous mixing of nano particles mixed solution is placed in ultrasonic bath for 30 min. at 70°C after the magnetic stirring. After that the resin is mixed with solution as desired proportion 1:15 with magnetic stirrer for 2 min. and then suddenly cure in die to facilitate desire shape and size at room temperature and left for 24 h then samples removed from die.

Characterization and Testing Instruments

Fourier-transform infrared (FTIR) spectroscopy (type IR Tracer-100/shimadzu co./ USA) analysis was done at Sagar Institute of Research and Technology (SIRT), Bhopal. AFM was carried on NT-MDT NEXT Solver (make-Russia) at Physics Laboratory of M.A.N.I.T, Bhopal. Tensile properties of polymer composites were measured by using (Tinius Olsen, model 50ST, Redhill, UK) with loading rate 4 mm/ min at room temperature. Scanning electron microscopy (SEM) facility used in MSME department MANIT Bhopal. Impact, tensile strength, hardness were tested at CIPET Bhopal MP.

RESULTS AND DISCUSSIONS

FT-IR Analysis

FTIR spectroscopy is used to examine intermolecular hydrogen bonding of polymer matrix with silica. The observations have been evaluated as shown in Figure 1.

- Peak shift from 1693 to 1698 cm⁻¹ and 3744 to 3748 cm⁻¹ respectively which directly insure that occurrence of hydrogen bonding between the -OH and -C=O.
- The vibrational frequency of Si-OH shift from 3837 to 3896 cm⁻¹ which is suggested that involvement of silica in epoxy matrix.

In the Fig. 2 clearly shows that the tensile stress increases with the loading percentage of nano SiO₂ particles within the epoxy polymer matrix. The increase of strength is due to the reinforcing the high strength of nano particles. Further increasing the nano particles beyond 4 wt.% the tensile strength was suppressed due to the agglomeration of nano particles in the matrix. Similar observations were also reported by several researchers^{9,11}. The tensile

strength of epoxy composites was highest at 3 wt. % nano SiO₂ reinforcement. The distribution of nano

particles were homogeneous at 3 wt. % clearly seen in AFM and SEM image.

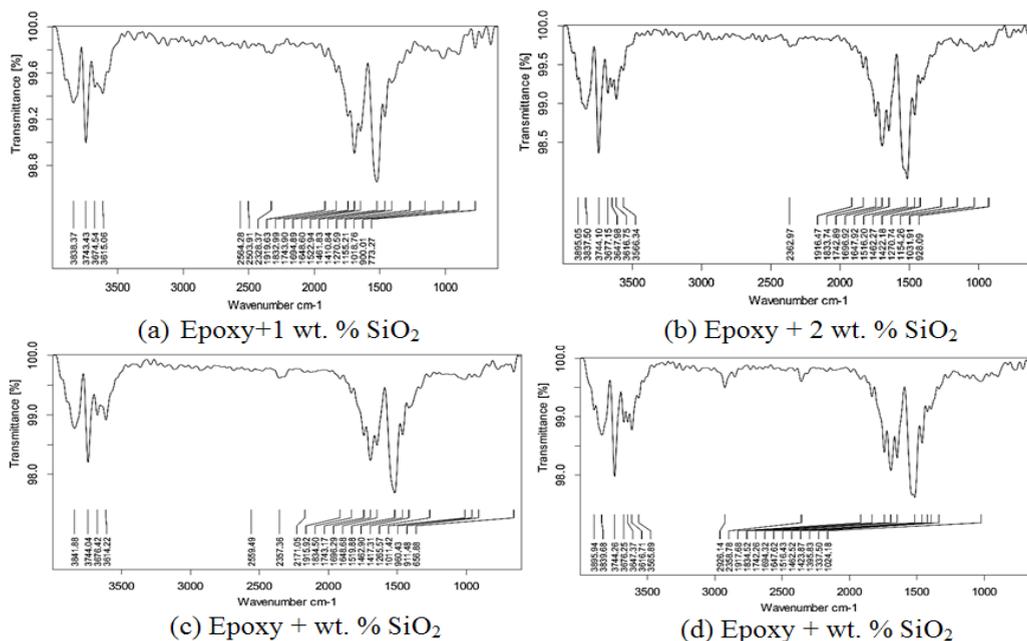


Fig. 1. FT-IR graphs for Polymer Matrix Composites of Epoxy + different wt.% of nano SiO₂ (a to d)

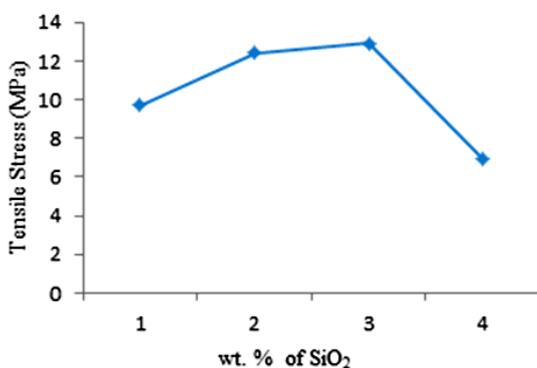


Fig. 2. Tensile stress with respect to loading wt.% of SiO₂

Flexural test results also shows Fig. 3 similar trend just like tensile test and impact test results, flexural load and strength increased upto 3 wt.% loading of SiO₂ in epoxy/nano SiO₂ polymer nano composite. On further loading the polymer epoxy based matrix with 4 wt.% of SiO₂ the flexural properties decreased. Reason that can be interpreted for the decreasing trend at high loading is the lack of homogeneity of distribution of reinforcement in the matrix and due to formation of agglomerations as seen by grain analysis under AFM. This decrease can also be attributed to the trapped bubbles and weak boundaries between matrix and nanoparticles. Flexural test result was lowered by adding 4 wt.% nano SiO₂ in epoxy polymer matrix. This may be

because of weak interaction between epoxy and nano SiO₂ particles which resulted in decrease of property.

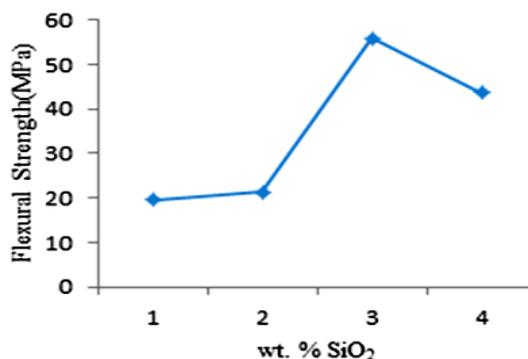


Fig. 3. Flexural strength at various wt.% of SiO₂ nano particles in polymer matrix

Scanning Electron Microscopy (SEM) Analysis

From the SEM characterization shows in Fig. 4 it can be very well interpreted that at lower wt.% of nano particles reinforcement within the epoxy matrix gives good result. At the 3 wt.% of SiO₂ nano particles the dispersion is homogeneous and further increasing the wt.% of SiO₂ the agglomeration of particles start which is clearly seen in SEM image. So that we can say the distribution of nano particles within the matrix at 3 wt.% of SiO₂ are more uniform and homogeneous. Further increasing the

loading wt. % of silica the number of agglomerations increases and their size also is increased. It also shows that at high loadings of nanoparticles the chances of lump formation is very high, owing to decrease in the properties of material due to heterogeneous distribution of silica nanoparticles in the matrix. Similar observations also reported by many researchers^{8,11}.

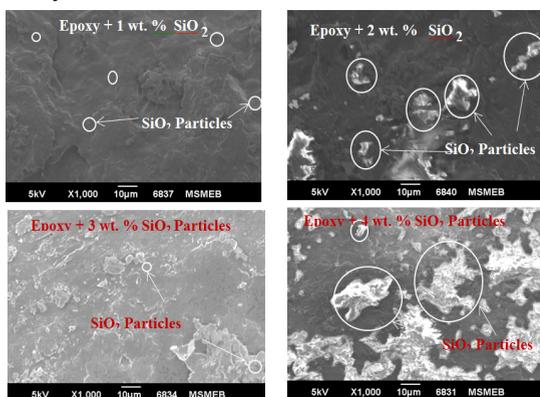


Fig. 4. SEM image of Epoxy Silica PMC

AFM Roughness Analysis

Figure 6 Average Roughness Variation with different wt.% of nano SiO₂ in Epoxy/SiO₂ PMCs. These AFM results show in Fig. (5a, 5b, 5c and 5d) very much correlate with the SEM results where grain analysis showed a decrease in number of grains at higher loadings due to clustering and agglomerations. From the graph Fig. 6, it is seen that on increasing the weight percent of SiO₂ loading the average roughness, tends to increase.

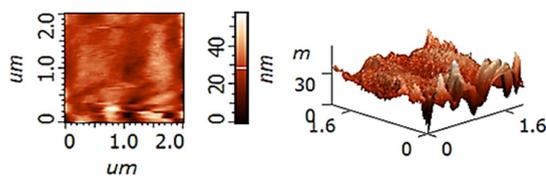


Fig. 5a. Dimensional and 3D image of Epoxy + 1 wt.% of SiO₂

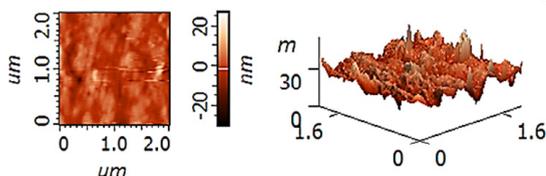


Fig. 5b. Dimensional and 3D image of Epoxy + 2 wt.% of SiO₂

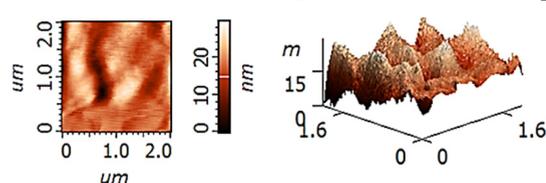


Fig. 5c. Dimensional and 3D image of Epoxy + 3 wt.% of SiO₂

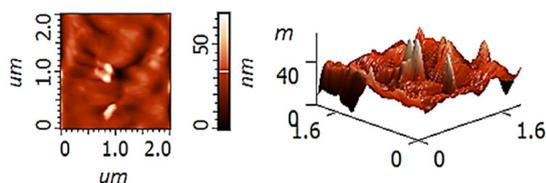


Fig. 5d. Dimensional and 3D image of Epoxy + 4 wt.% of SiO₂

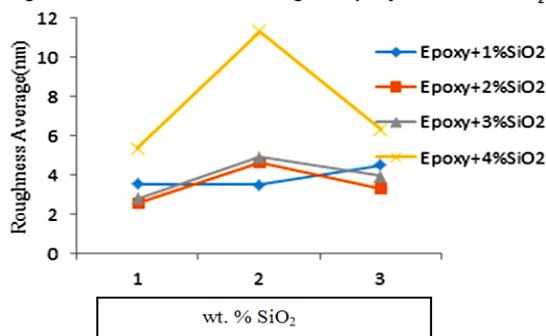


Fig. 6. Average Roughness Variation with different wt.% of nano SiO₂ in Epoxy/SiO₂ PMCs

AFM Grain Analysis Results

In this work AFM was carried on NT-MDT NEXT Solver (make-Russia) at Physics Laboratory of M.A.N.I.T, Bhopal. AFM grain analysis Fig. 7, Fig. 8 and Fig. 9 it can very well be interpreted that on increasing the weight percentage loading of SiO₂ nanoparticles in the epoxy matrix, the number of grains show a decreasing trend whereas diameter, average size and area show increasing trend, this concludes that high weight percentage loading clustering and agglomerations of the nanoparticles take place inside resin matrix thereby increasing the average size of the grains. at lower weight percentage of nano particles loading within the epoxy polymer matrix there are proper dispersion of nano particles but on increasing the weight percent of nano particles of SiO₂ agglomeration increases drastically within the matrix.

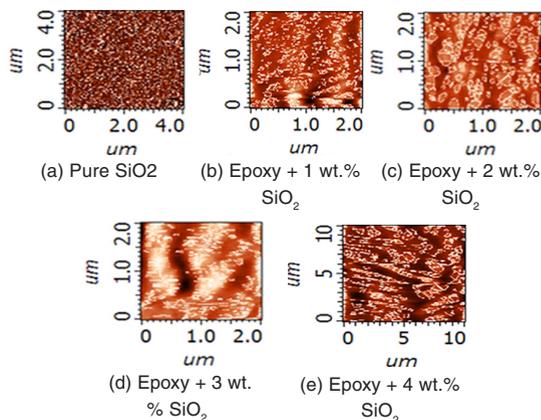


Fig. 7. AFM grain analysis of pure SiO₂ and Epoxy + different wt.% of SiO₂ nano particles (a to e)

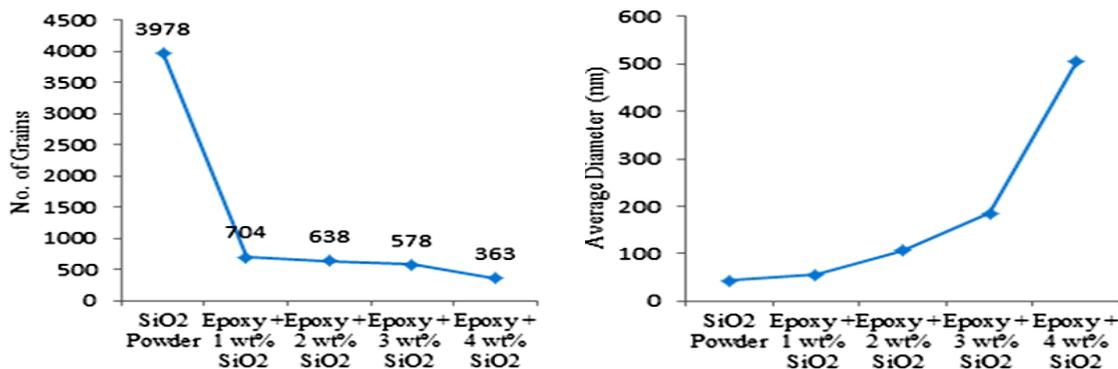


Fig. 8. Variation in grain numbers and diameters with different wt.% of SiO₂

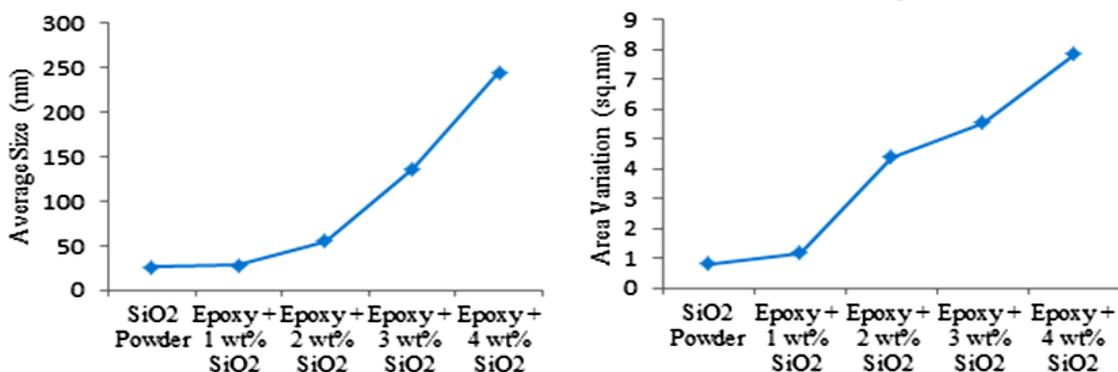


Fig. 9. Average Size variation and average Area variation with different wt % of SiO₂

In Fig. 10 impact test results shows. The impact strength was highest at 3 wt.% loading of nano SiO₂, further loading the ductility of epoxy decreases and possibility of formation of micro cracks increases. So, at higher loadings 4 wt.% of SiO₂ impact strength decrease due to increase in the brittle character of composites.

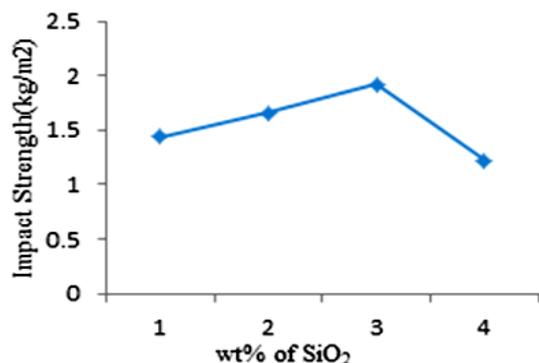


Fig. 10. Variation of Impact Strength (kg/m²) at loading of various wt.% of nano SiO₂ within matrix

In shape recovery demonstration Fig.11 shows that shape recovery of epoxy specimen when heated at 70°C in open environment. In shape recovery test first specimen heated at 70°C which

is glass transition temperature of epoxy, then bend the specimen shows in Fig. 11 (a) and cool down the specimen in cold water to get temporary shape. After that the test initiated at 70°C environment temperature. At t=0 second the specimen got temporary shape after that till 20 sec. the specimen recover back its permanent shape shows in Fig. 11 (a to d). The shape recovery of specimen because of strain energy stored in specimen during temporary shape when water cooling at glass transition temperature. This epoxy has glass transition temperature is 70°C which is very useful for further research in high temperature thermo-responsive shape memory polymer composites. So that, this type of epoxy opens the new gate for researchers to development and characterized the thermo-responsive shape memory properties.

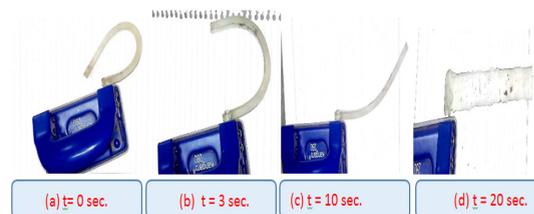


Fig. 11. Shape recovery demonstration at 70°C of epoxy specimen at various time to recover original shape (a to d)

Figure 12 clearly shows that, with increase of SiO₂ nano particles within the epoxy matrix the hardness were increase significantly upto 3 wt.% and then hardness was decrease at 4 wt.% SiO₂. The reason may be, upto 3 wt.% SiO₂ proper dispersion of nano particles after that agglomeration occurs within the epoxy matrix. Due to the agglomeration the grain boundaries reduces which is responsible for decrease the hardness value.

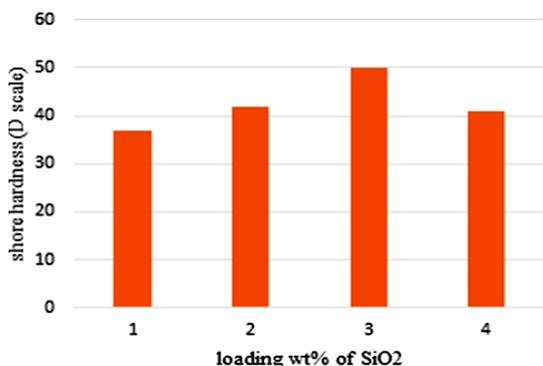


Fig. 12. Hardness curve at various wt.% of SiO₂ nano particles

CONCLUSION

- This present experiment we demonstrate shape recovery phenomena of this low cost epoxy which open the new gate for

high temperature thermo responsive shape memory polymer composites. This existing epoxy may very useful for replacement of widely spared high coastally polyurethane based shape memory polymer, which is further need for research and development.

- Homogeneous dispersion of SiO₂ nano particles with the help of magnetic stirrer followed by the ultrasonic liquid bath. At lower wt.% of nano particles proper mixing seen in AFM and SEM.
- Surface roughness increase with the increasing wt. % of SiO₂ nano particles due to the increase in coefficient of friction.
- FT-IR graphs very well show the existence of C=O and O-H bond in the PMCs.
- Tensile strength, flexural strength, hardness were significantly improved at 3 wt.% of SiO₂ nano particles.

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