Applied Innovative Research
Vol. 1, March 2019, pp. 21-24

# Development of Nano $SiO_2$ Particles Dispersed Shape Memory Epoxy Composites

Krishan Kumar Patel[1]*, Rajesh Purohit[1], S A R Hashmi[2], Ritesh Kumar Gupta[3] & Sandeep Kumar Dwivedi[1]
[1]Maulana Azad National Institute of Technology, Bhopal 462 003, India
[2]CSIR-Advanced Materials and Processes Research Institute, Bhopal 462 026, India
[3]Academy of Scientific & Innovative Research, CSIR- Advanced Materials and Processes Research Institute, Bhopal 462 026, India

*Received 10 September 2018; accepted 16 January 2019*

Thermo responsive shape memory epoxy based composites are being investigated for their excellent mechanical and thermal properties. In present study the thermosetting epoxy containing different composition of $SiO_2$ nano particles ranging from 1 to 4 wt. % have been prepared through solvent casting route. Developed composites have been studied for grain analysis, FTIR, shape recovery, impact strength and hardness. Addition of 3 wt. % $SiO_2$ improves hardness and impact strength significantly. Average grain size of $SiO_2$ particles increase at higher wt. % of $SiO_2$ in polymer matrix due to agglomeration of nanoparticles.

**Keywords:** Polymer, Epoxy, $SiO_2$, Nano particles, FTIR, AFM

## Introduction

Shape memory polymer composites (SMPC) have gained research interest in past few years. SMPCs have excellent properties like light weight, moisture resistant, easy processability and good shape memory behavior makes them suitable candidate for aerospace, robotics, biomedical and structural applications[1-3]. Many researcher improved thermal and mechanical properties of polymer by addition of suitable reinforcement[4-6]. Nano-particles are most promising filler for their exceptional mechanical and thermal properties due to their high surface area to volume ratio[7]. The main objective is to transfer intrinsic properties of nano-filler to the polymer composite, which require proper dispersion and interaction with matrix[8]. The important parameter on which properties of composite depends are volume fraction of filler, filler aspect ratio, particle size, shape of filler, intrinsic properties of constituents, nature of interface degree of dispersion and matrix properties[9,6]. Epoxy is the most common matrix material used for the composite materials development. Silica/epoxy composite material is a widely used material due to their ability to sustain mechanical and thermal loading[10,11]. Singh *et al.*[5] reported an increase in fracture energy and also an improvement in the modulus of epoxy composite by adding higher weight percentage of $SiO_2$ nanoparticles and CTBN liquid rubber. Many researchers[7,11,12] reported that the reinforcement of $SiO_2$ nanoparticles till 4 wt. % in epoxy matrix significantly improved the properties of composites[13,14]. Many of the researchers used 100-300 nm size $SiO_2$ particle which was not much effective in improvement of mechanical properties at less than 4 wt%. In present study $SiO_2$ of very fine size 10-20 nm was used for better dispersion and imparting properties at lower wt. %.

## Materials and Methods

In this study three components polymer composite system was used which consisted of epoxy resin (LY-556), hardener (HY- 951) and nano silica particles with much lesser particle size in the range of 20-30 nm as supplied by Nano Shel Pvt. Ltd. Four composition of $SiO_2$ in epoxy matrix which contains 1, 2, 3, 4 wt % $SiO_2$ in SMPC were prepared. In order to remove the absorbed moisture $SiO_2$ nanoparticles in the powder form were preheated in the oven at 100 °C for 2 h. Pre heated $SiO_2$ nanoparticles was mixed with hardener and slightly heated to reduce the hardener viscosity. To facilitate a homogeneous mixture of $SiO_2$ and hardener, a magnetic stirrer is employed. After the magnetic stirrer mixing the mixture was placed in ultrasonic bath maintained at 80 °C for 30 min to obtain uniform dispersion. Once the mixture reaches to the room temperature, epoxy resin is mixed in the ratio of 1:15. Die is preheated and then a coat of mould relief agent was applied on the inner

---

*Corresponding author (E-mail: krishan.patel4@gmail.com) surfaces of the die to facilitate easy removal of sample after curing. Then die is left for 10 to 12 h curing at room temperature and then the samples were removed from the die.

Prepared composites were characterized for AFM, FTIR, impact and hardness test. AFM was carried on NT-MDT NEXT Solver (make-Russia) at Physics Laboratory of M.A.N.I.T, Bhopal. Impact testing (IZOD test) of the polymer samples were done on Izod and Charpy Impact Tester, Tinius Olsen, USA at CIPET, Bhopal. FT-IR analysis was done on BRUKER – ALPHA FT-IR, at Sagar Institute of Research and Technology (SIRT), Bhopal.

## Results and Discussion

### Mechanical properties

An Izod impact test result shows in Fig. 1 (a) exhibits an increase in impact strength till 3 wt. % of $SiO_2$ nanoparticles and then starts to decrease. It may be attributed to an increase in percentage loading in polymer matrix composite, the ductility of epoxy decreases and possibility of formation of micro cracks increases. So, at higher loadings 4 wt. % of $SiO_2$ impact strength decrease due to increase in the brittle character of composites.

Figure 1 (b) shows that, with increase of $SiO_2$ nanoparticles within the epoxy matrix the hardness were increase significantly up to 3 wt. % and then hardness was decrease at 4 wt. % $SiO_2$. The reason may be up to 3 wt. % $SiO_2$ proper dispersion of nanoparticles after that agglomeration occurs within the epoxy matrix. Due to the agglomeration the grain boundaries reduces which is responsible for decrease the hardness value.

### AFM grain analysis results

In this work from Figs 2, 3 and 4, it can very well be interpreted that on increasing the weight

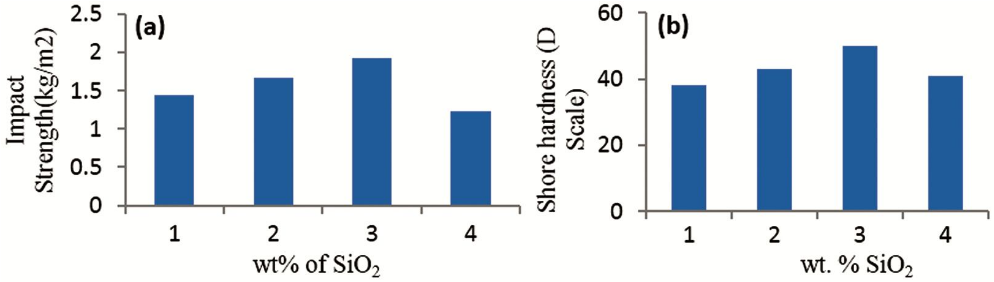

Fig. 1 – Variation of (a) impact strength (kg/m$^2$), (b) hardness for different wt% of nano $SiO_2$ in SMPC.

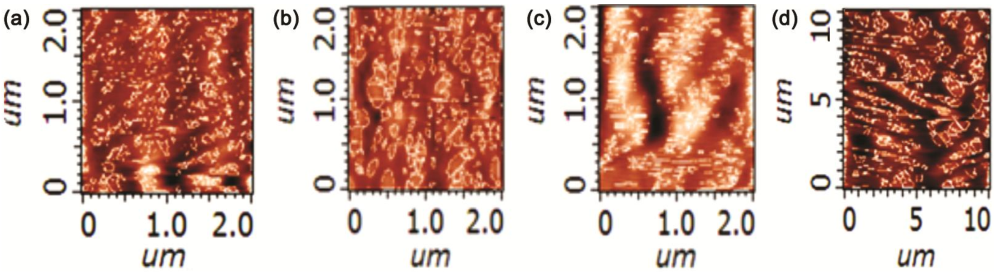

Fig. 2 – SMPC containing (a) 1 wt% $SiO_2$, (b) 2 wt% $SiO_2$, (c) 3 wt% $SiO_2$ and (d) 4 wt% $SiO_2$.

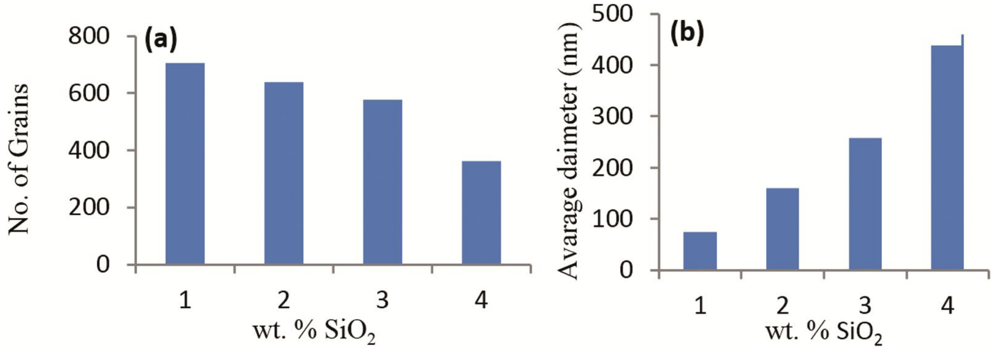

Fig. 3 – Variation in (a) grain numbers and (b) diameters with different wt. % of $SiO_2$.

percentage loading of $SiO_2$ 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 in the resin matrix thereby increasing the average size of the grains. In lower weight percentage loadings there is a proper dispersion of nano particles but on increasing the weight percent of $SiO_2$ agglomeration of nano particles increases.

### Fourier transform infrared spectroscopy

FT-IR spectroscopy is adopted to examine the intermolecular hydrogen bonding of epoxy resin with silica as well as intra molecular bonding within polymer matrix (Fig. 5). The vibration frequency of carboxyl group (-C=0) and hydroxyl group (-OH) shifts from 1694 to 1696 $cm^{-1}$ and 3743 to 3744 $cm^{-1}$, respectively, indicating the occurrence of hydrogen bonding between -C=O and -OH. The vibration frequency of Si-OH was shifted from 3838 to 3895 $cm^{-1}$, this may be attributed to good interaction of silica with polymer matrix. In addition, the frequency shift hydroxyl group (polymer-OH) in composite with silica is more. This indicates that the hydrogen bonding may also form between silica and polymer matrix in the form of –Si-O, H-O- polymer, in which oxygen connected to silicon atom, provides the shared ion pair electrons.

### Shape recovery test

Figure 6 clearly 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. 6 (a) and cool down the specimen in cold water to got 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 s the specimen recover back its permanent shape shows in Fig. 6 (b-d). The shape recovery of specimen because of strain energy stored in specimen during temporary

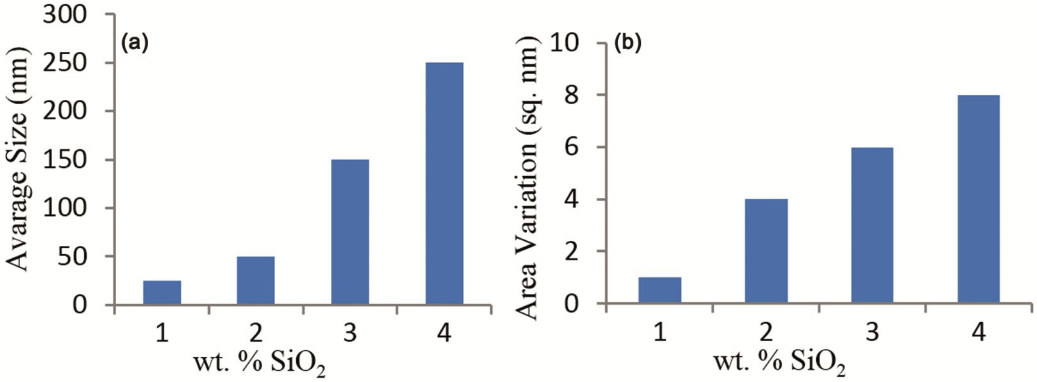

Fig. 4 – (a) average size variation and (b) average area variation with different wt % of $SiO_2$.

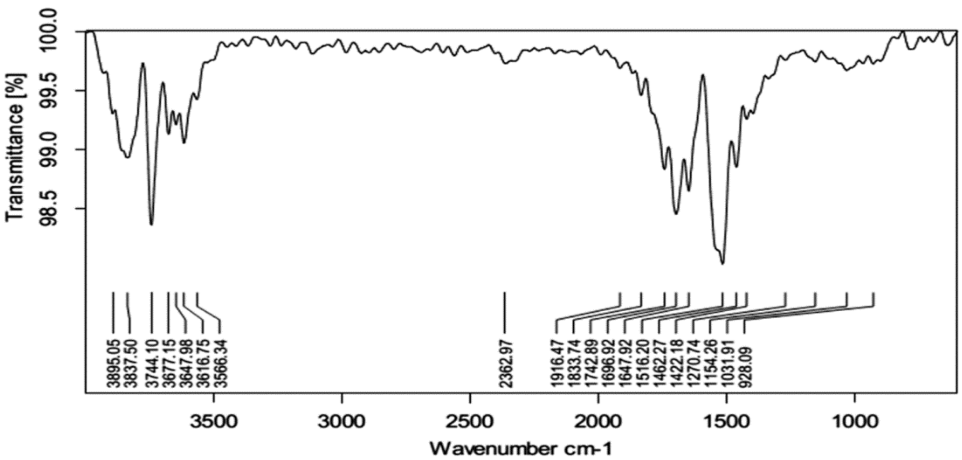

Fig. 5 – FTIR spectroscopy of epoxy + 2 wt. % $SiO_2$ nanoparticles.

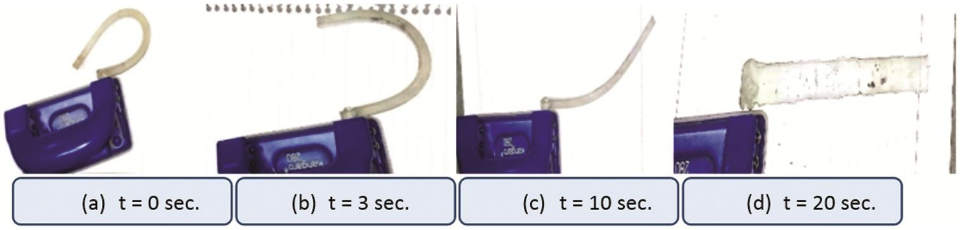

Fig. 6 – Shape recovery demonstration of epoxy specimen at various times to recover original shape.

shape when water cooling at glass transition temperature.

## Conclusions

From this study it has been concluded:

(i) Proper dispersion of nanoparticles in the epoxy resin was obtained by sonication process just after the magnetic stirring at suitable rpm.

(ii) In lower weight percentage loadings there is a proper dispersion of nanoparticles but on increasing the weight percent of reinforcement, i.e., nano silica used here, there are cases of agglomerations seen under AFM.

(iii) Complete shape recovery of this present work epoxy specimen.

(iv) FT-IR graphs very well show the existence of C=O and O–H bond in the PMCs.

(v) There was an increase in the impact and hardness properties till 3 wt. % of $SiO_2$ loading in polymer matrix composites.

## Acknowledgement

The Author Krishan Kumar Patel is highly thankful to MANIT, Bhopal for granting Fellowship under which the present work was carried out.

## References

1 Chen J, Kinloch A J, Sprenger S & Taylor A C, *Polymer*, 54 (2013) 4276.
2 Hsieh T H, Kinloch A J, Masania K, Taylor A C & Sprenger S, *Polymer*, 51 (2010) 6284.
3 Wetzel B, Rosso P, Haupert F & Friedrich K, *Eng Fract Mech*, 73 (2006) 2375.
4 Johnsen B B, Kinloch A J, Mohammed R D, Taylor A C & Sprenger S, *Polymer*, 48 (2007) 530.
5 Singh S K, Singh S, Kohli R, Jain A & Kumar A, *AIP Conf Proc*, 1728 (2016) 020586.
6 Zamanian M, Mortezaei M, Salehnia B & Jam J E, *Eng Fract Mech*, 97 (2013) 193.
7 Goyat M S, Ray S & Ghosh P K, *Composites: Part A*, 42 (2011) 1421.
8 Hunt E M, Plantier K B & Pantoya M L. *Acta Materialia*, 52 (2004) 3183.
9 Purohit R, Patel K K, Gupta G K & Rana R S, *Mater Today Proc*, 4 (2017) 5330.
10 Zaimova D, Bayraktar E, Miskioglu I, Dishovsky N, *Exp Mech Compos Hybrid Multifunctional Mater*, 6 (2014) 65–76.
11 Zaimova D, Bayraktar E, Miskioglu I & Dishovsky N, *Exp Mech Compos Hybrid Multifunctional Mater*, 6 (2014) 157.
12 Gupta G K, Patel K K, Purohit R & Bhagoria P, *Mater Today Proc*, 4 (2017) 5385.
13 Woody D L, Davis J J, Miller P J, *MRS Proceedings, Cambridge University Press*, 418 (1995) 445.
14 Bai Y, Cheng Z Y, Bharti V, Xu H S & Zhang Q M, *Appl Phys Lett*, 76 (2000) 3804.