Mechanical Properties of Silicon Nitride Particles Filled Epoxy Composites

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Abstract*— Present work concentrate on development of polymer composites *consist of epoxy as a matrix material with a micro-size silicon nitride as a filler material. Five sets of composite specimen with filler content ranging upto 50 wt. % has been fabricated using simple hand lay-up technique. Mechanical properties of the samples were studied. The various mechanical property evaluated are tensile strength, compressive strength and hardness. From the experimental results, it is found that silicon nitride filled epoxy composites possess high compressive strength and hardness. Through, tensile strength of the composite increases with increase in filler content till certain filler content and later the decreasing trend is noticed in the fabricated composites.

Keywords—Composites. Silicon nitride, Epoxy, Mechanical properties.

I. INTRODUCTION

Composite materials are extending the horizon of the designers in all branches of engineering. In composite materials are combines in such a way as to facilitate us to make enhanced use of their virtues while diminishing to some extent the effects of their insufficiencies. This process of optimization can release a designer from the constraints associated with the selection and manufacture of conventional materials. They can make use of tougher and lighter materials, with properties that can be tailored to suit particular design requirements. And because of the ease with which complex shapes can be manufactured, the complete rethinking of an established design in terms of composites can often lead to both cheaper and better solutions.

Polymer matrix composites (PMCs) are the best established form of advanced composite materials. Of the two classes of polymers used as matrices, thermosets and thermoplastics, thermosets dominate the market for structural applications. Current research is being conducted on composite material that can be used for microelectronic applications. For these applications, high thermal conductivity, low coefficient of thermal expansion and high glass transition temperature are the most important requirements. Most metal matrix composites and ceramic matrix composites are thermally conductive but are highly dense and electrically conductive as well, which may not be suitable for electronic packaging application. So the present research has been focused on developing a composite, which are polymer matrix composites, where polymers are embedded with thermally conductive and electrically insulative ceramic particulate fillers. The amount of filler that is incorporated inside the matrix is considered to be the most significant factor which can alter the performance of composite system. It has been shown by many researchers that dramatic improvement in mechanical properties can be achieved by incorporation of either micro or nano-particles, since rigid inorganic particles generally have a much higher stiffness than polymer matrices [1].

The effect of $CaCO_3$ volume fraction on the notched Izod impact toughness of high density polyethylene (HDPE)/CaCO3 composites is shown by Liu et al. [2]. Cho et al. [3] underlined the interest of replacing micro-scale particles by its nano-scale counterparts smaller particle size yields higher fracture toughness. Wetzel et al. [4] study the effect of micro and nano-sized ceramic particulates into an epoxy resin. They study the effect of SiC and TiO2 particles for different particle loading and particle size as well as with surface modification of particles and observed that with increase in filler loading and decrease in particle size, the specific wear rate decreases whereas modification of particles has only a little effect on the wear performance. However, Zang et al. [5] reported that grafting of PAAM onto nano-silica increases the interfacial interaction between the particles and the matrix through chemical bonding. Antunes et al. [6] found that increasing average particle dimension tends to decrease the volume removed by wear in the composite and increase it in the antagonist body. Recently, Anjum et al. [7] studied specific wear rate of SiO2 filled glass-epoxy composite. Likewise, the incorporation of hard particles i.e. SiC, ZrO, Ti3SiC2 has led to enhancement in wear resistance [8-10].

Against this background, in present work, a class of composite is fabricated in which the continuous phase is thermoset epoxy matrix and a discontinuous phase is micro-size silicon nitride particles. Simple hand lay-up method is used for fabrication of composites with wide range of filler content. The various testing involve physical testing in which microstructure study of composites was performed together with evaluation of density and void content. Under mechanical testing, tensile strength, compressive strength and hardness were evaluated.

II. MATERIALS AND METHODS

A. *Material used*

Thermoset resin Lapox L12 is a liquid, unmodified epoxy resin of medium viscosity is used as the matrix material in present investigation. Hardener K6 is commonly employed with Lapox L12. The matrix material system selected is supplied by ATUL India Ltd., Gujarat, India. Silicon nitride of size 50 microns is used in present investigation supplied by Intelligent Materials Private Limited, Mohali. It is of high purity grade with alpha crystal structure. It delivers superior thermal shock resistance. It also has good fracture toughness. It has good oxidation resistance and creep resistance. Hence there are multiple reason for selecting silicon nitride as filler material in present investigation.

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B. Composite Fabrication

Simple hand lay-up technique is used in the present investigation for fabrication of silicon nitride particles in epoxy matrix. This method is considered as the simplest technique for composite fabrication. The fabrication of composite using hand lay-up method involves following steps:

1. The room temperature curing epoxy resin epoxy resin (Lapox-12) and corresponding hardener (HY 951) are mixed in a ratio 10:1 by weight as recommended.
2. Micro-size silicon nitride particles were then added to the mixture of epoxy and hardener which is later mixed thoroughly by hand stirring.
3. A coating of silicon spray is mandatory over the mould before pouring the mixture into it, a silicon spray is done over the mold so that it will easy to remove the composite after curing. The uniformly mixed dough is then slowly poured into the mould.
4. The cast is than cured for 8 hours before it was taken from the mould.

Composites were fabricated with different weight fraction of filler ranging from 0 wt. filler i.e. neat epoxy to 50 wt. % filler. The composite fabricated under the investigation is given in table 1

TABLE I

EPOXY COMPOSITES FILLED WITH SILICON NITRIDE

S.No.	**Composition**
1	Neat Epoxy
2	Epoxy + 10 wt % Silicon Nitride
3	Epoxy + 20 wt % Silicon Nitride
4	Epoxy + 30 wt % Silicon Nitride
5	Epoxy + 40 wt % Silicon Nitride
6	Epoxy + 50 wt % Silicon Nitride

C. Mechanical Characterization

The tensile strength of the composites is measured with a computerized Tinius Olsen universal testing machine in accordance with ASTM D638 procedure by applying uni-axial load through both the ends at a cross head speed of 0.5 mm/min. In the same process, one more property of the developed material can be evaluated i.e. elongation at break. This gives that before the material breaks, what is the extension it undergone. This help to evaluate the ductility of the material. If the extension is more, the material can be said relative ductile in nature and material with low extensile can be considered as brittle in nature. Static uniaxial compression tests and flexural test on specimens are carried out using the same computerized Tinius Olsen universal testing machine. The method by which the compression test is conducted is in accordance with ASTM D695. Affri LD250 hardness measuring instrument is used to determine the micro-hardness of the fabricated composite. The tests are in accordance with ASTM E384.

III. RESULTS AND DISCUSSION

A. Hardness

Figure 1 shows the variation in the value of hardness of the composite material for different content of silicon nitride in epoxy matrix. Silicon nitride is known for its high hardness. Hardness of silicon nitride is 19.25 GPa which is around 22 times that of neat epoxy. Hence it is obvious that incorporation of silicon nitride in epoxy matrix will enhance the value of hardness of the composite and this increasing trend continues as the content of filler is increasing. The same can be seen from figure 4.7. presently fabricated samples. Increased filler content resulted in increase in modulus of the composite, leading to a corresponding increase in the hardness of the composite. Hardness is very important property while developing material for microelectronic applications. It is seen that if the material possesses low hardness value, it will damage the material while implementing it in practical use. Hence improved hardness is required to overcome the wear during the installation of different component over the chip. Also high hardness is required to protect the delicate component from foreign attack and also it helps the material to get damage when it falls accidently. With the addition of 50 wt. % of silicon nitride, hardness of the composite body increases form 0.87 GPa for neat epoxy to 4.18 GPa. This is a remarkable enhancement in the value of hardness which is of around which is come out to be of 380 % over neat epoxy.

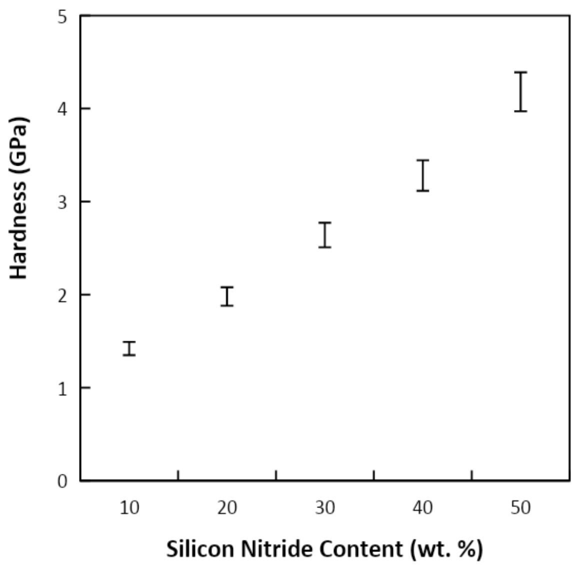

Fig. 1 Hardness of epoxy filled with silicon nitride

B. Tensile Properties

The dependence of ultimate tensile strength of epoxy composites filled micro-size silicon nitride with different content is shown in figure 2. The tensile strength of the epoxy composites increases with increase in silicon nitride content till the filler content is limited upto 20 wt. %. The tensile strength of neat epoxy is measured to be 27 MPa. The value increases to 32.5 MPa with 10 wt. silicon nitride content and is further increases to 39.8 MPa when the filler content increases to 20 wt. %. The tensile strength value obtained at this filler content is maximum where the improvement of 47.4 % over neat epoxy is registered. When the content of silicon nitride increases beyond 20 wt. %, tensile strength starts to show decreasing trend with increase in filler content. It is seen that when 50 wt. % filler is added in epoxy matrix, tensile strength of the composite is 25.1 MPa. This reduction is 36.9 % when compared to maximum tensile strength at 20 wt. % filler and 7 % when compared to neat epoxy. The increasing-decreasing trend is obtained because a limited quantity of filler can successfully remedy the defects form epoxy self-curing. Meanwhile, silicon nitride can transfer stress and avoid the expanding of cracks, thus enhancing tensile strength. Also at low filler content, proper wetting of filler took place and also uniform distribution of filler took place within matrix body. This also supports the improvement in tensile strength with limited filler content. The drastic decline in tensile strength, as a result of high loading of silicon nitride, may be clarified by the fact that excessive fillers served as local stress-concentration points in the resin matrix. Consequently, some voids or interfacial defects, pitiable interfacial wettability between the silicon nitride and the epoxy matrix might appear in the composites, causing in the weakening in tensile strength.

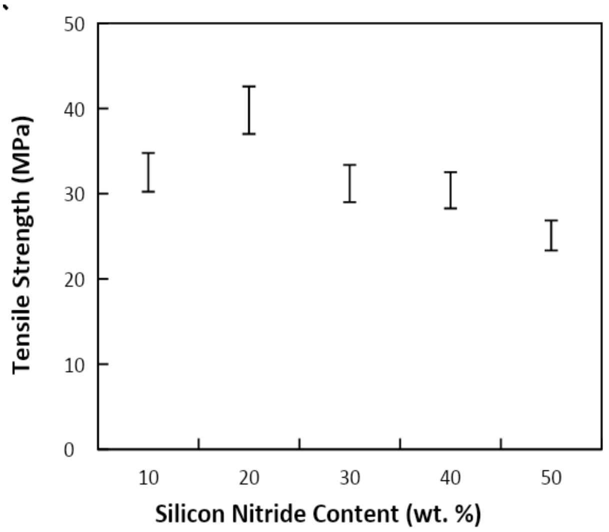

Fig. 2 Ultimate tensile strength of epoxy filled with silicon nitride

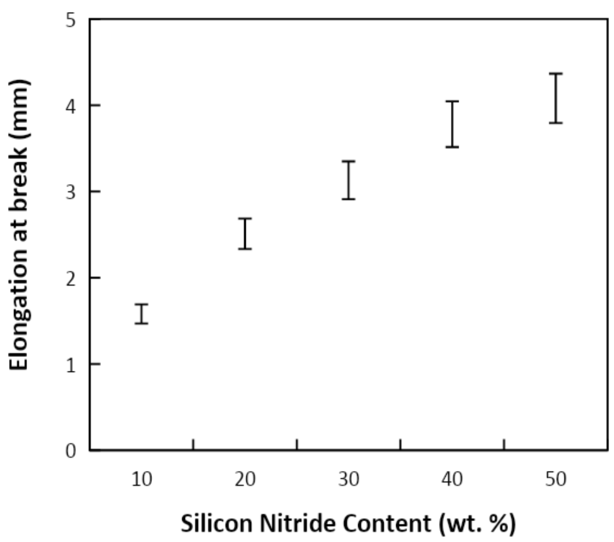

Fig. 3 Elongation at break of epoxy filled with silicon nitride

Figure 3 shows the variation in elongation at break as a function of filler content. Epoxy is highly brittle in nature with extension of only 0.88 mm before it breaks. This limited the application of epoxy as highly brittle nature is not desirable in many applications especially where repeated loading took place. It can be seen that incorporation of silicon nitride slightly changes its behavior from being highly brittle to slightly ductile where extension at break increases with increase in filler content. Elongation at break increases to 4.08 mm with 50 wt. % of silicon nitride. This is an increment of 363.6 %.

C. Compressive Strength

The dependence of compressive strength of epoxy composites filled with silicon nitride with different filler content shown in figure 4. It can be seen from the figure that with increase in content of silicon nitride, compressive strength of the composites increases and this increasing trend continuous till the maximum content of filler is added in epoxy resin. The compressive strength of neat epoxy is 82 MPa which increases to 114.8 MPa at a loading of 50 wt% of micro size silicon nitride. In this case 40 % enhancement in the value of compressive strength is reported for maximum content of filler.

The improvement in compressive strength with filler addition is mainly because of the high compressive strength of filler material. Also, the increase in compressive strength with increased filler content is due to the favorable deformation processes facilitated by the presence of fillers in the matrix. Under a compressive loading situation, the fillers apparently aid the load bearing capability of a composite, rather than acting as stress raiser as is the case in tensile loading. Further, the fact that in a compression test, any crack or flaw introduced by dispersion of the filler will, if at all, get healed (closed) and made ineffective, contrary to the crack opening mechanism occurring in a tensile loading situation.

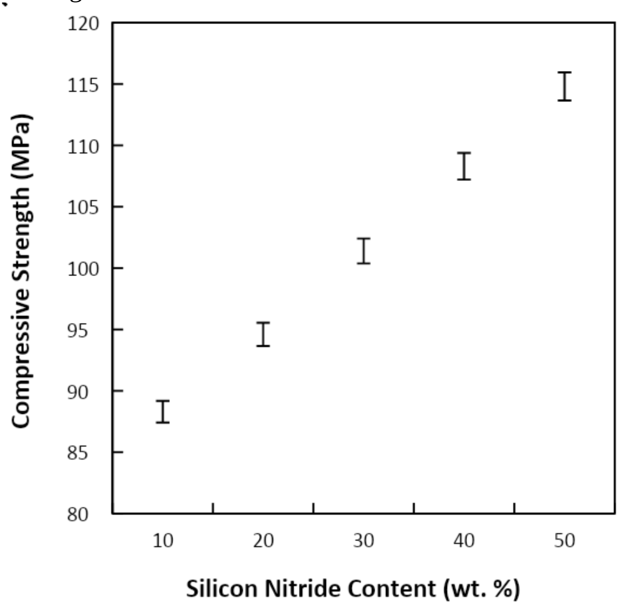

Fig. 4 Compressive strength of epoxy filled with silicon nitride

IV. CONCLUSIONS

This experimental investigation has led to the following specific conclusions:

1. Epoxy matrix composites reinforced with micro-size silicon nitride is possible by simple hand-lay-up technique.
2. The tensile strength of the epoxy composites increases with increase in silicon nitride content till the filler

content is limited upto 20 wt. %. The value increases to 39.8 MPa with 20 wt. % silicon nitride. When the content of silicon nitride increases beyond 20 wt. %, tensile strength starts to show decreasing trend with increase in filler content. At 50 wt. % filler, tensile strength of the composite reduced to 25.1 MPa.

3. Elongation at break increases with filler content changing the behavior of composites from brittle to slight ductile. Maximum elongation noticed is 4.08 mm with 50 wt. % of micro-size silicon nitride. This is an increment of 363.6 %.
4. Compressive strength of the composites increases with increase in silicon nitride content. The compressive strength of neat epoxy is 82 MPa which increases to 114.8 MPa at a loading of 50 wt% of micro size silicon nitride. In this case 40 % enhancement in the value of compressive strength is reported for maximum content of filler.
5. With addition of silicon nitride fillers, micro-hardness of the composites improved and this improvement is mainly a function of the filler content. With the addition of 50 wt. % of silicon nitride, hardness of the composite body increases form 0.87 GPa for neat epoxy to 4.18 GPa. This is a remarkable enhancement in the value of hardness which is of around which is come out to be of 380 % over neat epoxy.

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