Tensile Tests of Glass Powder Reinforced Epoxy Composites: Pilot Study

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Keywords: Yield strength, tensile strength, Young's modulus, epoxy resin, and glass powder.

Abstract. Epoxy resin was filled with glass powder to optimize the strength of the composite for structural applications by a research centre in the University of Southern Queensland (USQ). In order to reduce costs, the centre wishes to fill as much glass microspheres as possible subject to maintaining sufficient strength and fracture toughness of the composites in structural applications. This project varies the percentage by weight of the glass powder in the composites. After casting the composites to the moulds, they were cured at ambient conditions for 24 hours. They were then post-cured in a conventional oven and subjected to tensile tests. It was found that the best percentage of glass powder by weight that can be added to the epoxy resin to give an optimum yield and tensile strengths as well as Young modulus and cost was five percent. It was also found that the fractured surfaces examined under scanning electron microscope were correlated with the fracture toughness. The contribution of the study was that if tensile properties were the most important factors to be considered in the applications of the composites, glass powder is not a suitable filler. It is also hoped that the discussion and results in this work would not only contribute towards the development of glass powder reinforced epoxy composites with better material properties, but also useful for the investigations of tensile properties in other composites.

Introduction

Organic-inorganic hybrid materials consisting of inorganic materials and organic polymers are a new class of materials, which have received much attention in recent years. The composite materials exhibit characteristics of both inorganic materials and organic polymers. It has been established in recent years that polymer reinforced with a small percentages of strong fillers can significantly improve the mechanical and thermal properties [1].

The most widely used and least expensive polymer resins are the polyesters and vinyl esters; these matrix materials are used primarily for glass fiber-reinforced composites. The epoxies are more expensive and, in addition to commercial applications, are also utilized extensively in polymer matrix composites for aerospace applications; they have better mechanical properties and resistance to moisture than the polyesters and vinyl resins [2]. This research project is to investigate the yield strength, tensile strength and Young's modulus of epoxy composites reinforced with varying percentage by weight of glass powder, the filler, with a view to finding out the optimum percentage by weight of the glass powder that can be added to the composites.

The epoxy resin used in this study is Kinetix R246TX Thixotropic Laminating Resin, an opaque liquid, and the hardener used is kinetic H160 medium hardener which has a pot life of 120 minutes. Other hardeners like H126, H128, H161 and H162 can also be used [3]. The glass powder was first mixed with epoxy resin, after this the hardener, kinetic H160 medium was added. The by weight ratio of resin to hardener used was 4:1 [3]. The composite was then cast to moulds of tensile test pieces and left to cure under ambient conditions for 24 hours. The tensile test specimens were taken out of the moulds and then post-cured in an oven at 40 ^{o}C for 16 hours, and then at 50 ^{o}C for 16 hours and finally at 60 ^{o}C for 8 hours. This is to ensure the heat distortion temperature (HDT) is above 63 ^{o}C. To bring the ultimate HDT to 68 ^{o}C, another 15 hours of post-curing will be required [3]. The specimens were then subjected to tensile tests.

Epoxy resin

The family of polymers gets its name from the epoxy functional group that terminates molecules or that is internal to the structure. Epoxies are really polyethers, because the monomer units have an ether type of structure with oxygen bonds, R—O—R [4]. Whereas the building blocks and chemical reactions involved in producing and crosslinking of unsaturated polyesters are similar for different polyester types, the situation is rather complex with epoxies. A wide range of building blocks are used in polymerization and numerous different compounds in crosslinking. In the following, only one epoxy configuration is considered and will have to serve as a representative for the entire epoxy family. An epoxide, or oxirane, group consists of one oxygen and two carbon atoms arranged in a ring. Often the epoxide group contains yet another carbon atom and is then referred to as a glycidyl group. The most common epoxy is based on condensation polymerization of epichlorohydrin and bisphenol A creating diglycidylether of bisphenol A (DGEBPA or DGEBA) [3]. The number of repeating units in an epoxy molecule is much lower as compared with that of polyesters, typically on the order of 10; at an average n value of 2 DGEBPA is solid at room temperature. Although epoxies are normally referred to as polymers, they are therefore strictly oligomers (few mers).

Glass powder

The glass powder used is SPHERICEL® 60P18 (spherical) hollow glass spheres. They are used to enhance performance and reduce viscosity in paints and coatings and as lightweight additives in plastic parts. They are chemically inert, non-porous, and have very low oil absorption. Typical properties of the spheres are shown in Table 1 [4]. SPHERICEL® 60P18 hollow spheres products offer formulators flexibility in polymer composites. The addition of hollow spheres to fiberglass reinforced plastics (FRP), epoxy, compounds, and urethane castings can provide weight reduction cost, savings and improved impact resistance. Insulating features of hollow spheres also work to the chemists' advantage in thermal shock and heat transfer areas. Two densities available are 0.6 to 1.1 g/cc; it provides choices to best fit mixing and target weight requirements [5]. The density of the hollow glass powder used in this research is 0.6 g/cc because the other filler, ceramic hollow spheres or SLG used in similar study is 0.7 g/cc; this will give a better basis for comparison of results obtained in the future. When used in polymer concrete, hollow spheres provide a cost effective alternative without degrading physical properties.

Table1: Typical properties of hollow glass spheres composites.

Shape	Spherical
Colour	White
Composition	Proprietary Glass
Density	1.1 g/cc and 0.6 g/cc
Particle Size	Mean Diameter 11 and 18 microns
Hardness	6 (Moh's Scale)
Chemical Resistance	Low alkali leach/insoluble in water
Crush Strength	>10,000 psi

Table 2: Tensile strength of alumina trihydrate filled epoxy

Particle size (microns)	**Volume fraction (%)**	**Tensile strength (MPa)**
Unfilled	0	75.9 ± 8.8
1	10	58.0 ± 3.4
8	10	29.9 ± 1.7
12	10	27.2 ± 2.4

The Composite Samples

The reinforcer was glass powder (glass hollow sphere) particulates and they were made 0 % to 35% by weight in the cured epoxy composite, EP/GP (X %), where X is the percentage by weight of the filler. Above 35% by weight of filler, the slurry would be too sticky to be cast into moulds. As the raw materials of the composites are liquid and glass hollow spheres, the tensile test specimens were cast to shape. The resin is an opaque liquid and is first mixed with the catalyst. After that the glass powder is added to the mixture, they are then mixed to give the uncured composite. The mixture of glass powder, resin and accelerator was blended with mechanical blender to ensure a more homogenous mixture. The uncured composite was then cast into the moulds and cured in ambient conditions. After initial 24-hour curing when the test pieces were removed from the mould, they were post-cured for 40 hours. This was achieved by curing the pieces in an oven.The test pieces were then tensile-tested in accordance with an Australian standard [6].

Results and Discussion

Figure 1 illustrates the yield strengths of varying percentage by weight of glass hollow spheres reinforced epoxy matrix composites. The yield strength of the neat resin was 17.95 MPa, which was higher than those of the composites with any percentage by weight of glass powder other than 5 w/t % (18.24 MPa) of glass powder. After dropping to 14.64 MPa at 10% by weight of filler, it remained stable up to 20 % percent by weight of glass powder. After this, it dropped further to 13.62 MPa at 25 % by weight of filler and remained so up to 35 % percent by weight of glass powder. In general, the higher the percentage by weight of glass powder, the lower was the yield strength.

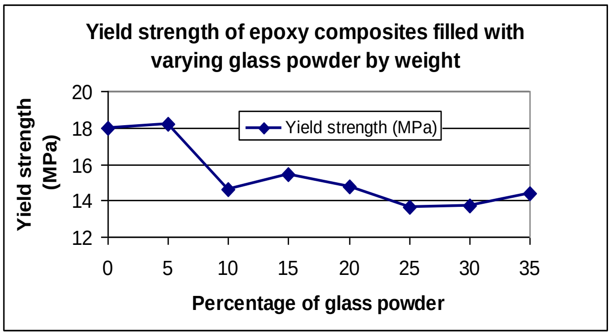

Fig. 1: Yield strength of epoxy composite reinforced with varying glass powder by weight

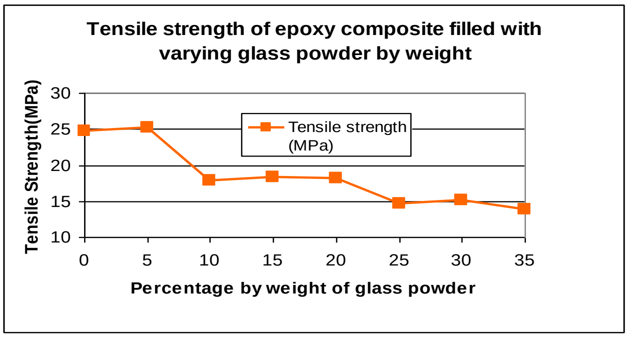

Fig. 2: Tensile strength of epoxy composite reinforced with varying glass powder by weight

Figure 2 shows the tensile strengths of epoxy composites with varying percentage of glass powder by weight. The tensile strength of the neat resin was 24.80 MPa, which was only lower than that (25.14 MPa) of composite with 5 % by weight of filler, but higher than those of the composites with any percentage by weight of glass powder. At 10 percent by weight of filler, the tensile strength dropped to 17.79 MPa; it then remained up to 20 % percent by weight of glass powder; after this glass powder reinforcement dragged the values of tensile strength further down; it dropped to 14.72 MPa when the percentage by weight of filler was 25% remained so up to 35 % percent by weight of glass powder. The variation of tensile strength with respect to percentage by weight of glass powder is the same as that of yield strength. If cost and tensile strength were considered at the same time, composite with 5 % by weight of filler is the best. It can be found that the trend for the graphs of yield and tensile strengths are the same and it can be argued that the results were correct in trend.

The tensile strength of neat resin used in the study (24.8 MPa) is much lower than that used by the studies of Nakamura et al. (77.3 MPa) and Radford (75.9 MPa). The former did not mention the epoxy resin used and the latter used anhydride-cured epoxy resin [7, 8]. In this study, the pot life of the hardener is 120 minutes; therefore, the epoxy resin used must be amine-cured as well. Effects of particle size on the tensile properties of cured epoxy resins, filled with spherical silica particles prepared by hydrolysis of silicon tetrachloride, were studied by Nakamura et al. [7]. Particles were sorted into five kinds of different mean sizes in the range from 6-42 microns. Static tensile tests were carried out. Tensile strengths were found to increase with a decrease in the particle size but with increase particle contents [7]. This trend is supported by the tensile strength results of epoxy/alumina trihydrate particulate composites in Table 2 [7]. In this study, the tensile strengths were found to decrease with increase particulate loading and it can be argued that this happened because the glass powder particles had not been treated by hydrolysis of silicon tetrachloride.

Figure 3 shows the Young's modulus of varying by weight of glass hollow spheres reinforced phenol formaldehyde matrix composite. The Young's modulus of the neat resin was 2.91 GPa and it dropped to 2.63 GPa when the percentage by weight of glass powder was 15%. It remained stable up until 25 % by weight of glass powder. It then bounced back to 3.02 GPa at 35 % by weight of filler. Table 3 shows the values of Young's modulus mentioned above with their standard deviations in brackets. From neat resin to 20 % by weight of glass powder, the yield strength, tensile strength and Young's modulus behaviors of the composites were more or less the same. From 20+ % to 35 % by weight of the filler, values of yield and tensile strength decreased further with increasing particulate loading, while those of Young's modulus moved in the opposite direction.

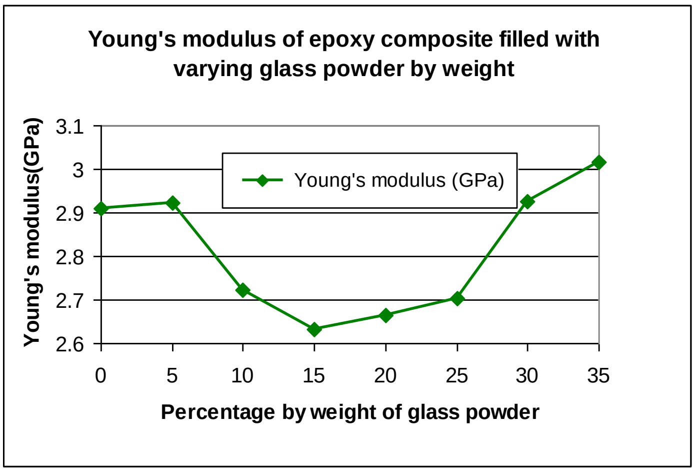

Fig. 3: Young's modulus of epoxy composite reinforced with varying glass powder by weight

It was found that a reduction in cost by one percent is followed by 1.5 % increase in tensile strength. For other percentages by weight of filler, the loss in tensile strength will not be compensated by the reduction in cost. It can be argued that 5 % by weight of filler is the best.

Figure 4 shows the scanning electron microscope image of neat epoxy resin post-cured for a total of 40 hours at 40 ^{o}C, 50 ^{o}C and 60 ^{o}C respectively at a magnification of 200 times. Faint striations followed by a 'turbulent flow' can be found in the fractured surface of the neat resin. This shows that plastic deformation had taken place in the resin. Figure 5 illustrates the scanning electron microscope image of epoxy reinforced by 25 % by weight of glass powder and post-cured for the same number of hours and temperatures at a magnification of 200 times. Holes were spotted and this explained why the tensile strength (24.80 MPa) of neat epoxy resin was stronger than that (14.72 MPa) of epoxy composite with 25 % by weight of glass powder. The holes were formed during the mixing process and the higher the percentage by weight of glass powder, the more holes would be expected.

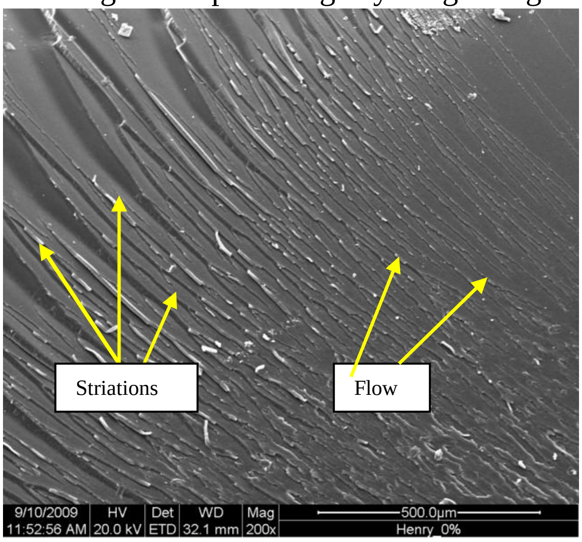

Fig. 4: SEM image of fractured neat epoxy resin, 200X

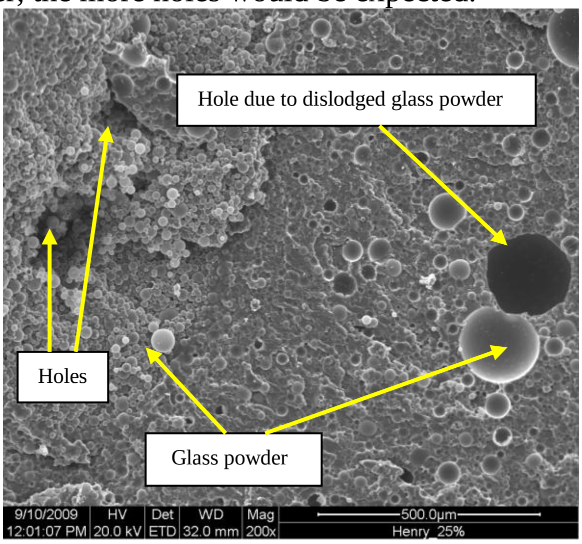

Fig. 5: SEM image of fractured 25 % glass powder filled epoxy composite, 200X

Conclusions

This study has evaluated the yield strength, tensile strength and Young's modulus of varying percentage by weight of glass powder reinforced epoxy resin; in all cases, the fluidity of the slurry composite was high and could be cast easily into moulds. The values with no filler had also been compared with those found by other studies but the tensile properties of some cases did not agree with this study and some did. Since the sizes of porosities of the composites found in this study were very small, it can be argued that the values of tensile properties obtained were very good and reliable as their standard deviations were low. Some air bubbles were found due to imperfect manufacturing of the samples. It can also be argued that the interfacial adhesion between epoxy resin (matrix) and glass powder (reinforcer) would be improved by treating or coating the glass powder and the properties of the composites would be improved.

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