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Analysis of filler–fibre interaction in fly ash filled short fibre–epoxy composites using ultrasonic NDE

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Abstract. Size and aspect ratio are believed to influence the rheology or the flow in the mixture and in turn the mechanical performance of the composites. Fillers and fibres when used in combination are expected to complement each other's performance resulting in better properties for the composite. They also reduce the extent of matrix polymer required in the system. Composites involving fillers and fibres individually and in combination are studied for the level of defect population using NDE and the mechanical performance under compression correlated with such an analysis. It was found that inclusion filler–fibre combination besides yielding better physical properties like density also yielded improved mechanical properties like strength and modulus. These properties showed an improvement of about 30–40% as compared to the ones where a single reinforcement either ash or fibre was used. Further, they exhibited uniform distribution of defects whose population was least compared to the situation where only one component (either filler or fibre) as reinforcement was tried.

Keywords. Fly ash; filler; fibre; filler–fibre interactions; epoxy; NDE; physical and mechanical properties.

1. Introduction

Non-destructive evaluation (NDE) often helps to eliminate the need for making prototype and test the same for performance, which could be an expensive as well as time involved process. Through NDE, a defect mapping can be performed on the components of the prototype before they can be integrated. The defective regions thus identified and avoided reduce the risk of lower performance by the prototype. With their origin in homogenous materials some decades back, the use of the techniques of NDE to characterize heterogeneous materials such as composites has gained importance in the recent times (Henneke 1990).

Composites on account of their processing schedule and constituent materials are prone to the existence of defects like voids, cracks, debonds and delaminations within them. Many non-destructive testing and evaluation techniques (Register 1998) are used for identifying these defects but ultrasonic testing, owing to its simplicity of usage and ability to test large components, is widely used to address the issue of mapping of defects in composite materials (De Almeida *et al* 1994; Gupta *et al* 1998; Karthikeyan *et al* 1999).

Polymer composites generally are reinforced with fibre or fillers to impart good mechanical properties. Abundantly available fillers, inexpensive and possessing good mechanical properties, when used with fibre helps to reduce the cost of the system, and at the same time either retains or improves the strength properties. Fly ash, a fine particulate waste from thermal power plants has attracted interest (Mohapatra and Rajagopala Rao 2001) lately, because of the abundance in volume of material generated and the environmental linked problems in the subsequent disposal. Fly ash mainly consists of alumina and silica, which are expected to improve the composite properties. Fly ash also consists to some extent, hollow spherical particles (Cenospheres, Pedlow 1978) which aid in maintenance of lower density values for the composite, a feature of considerable significance in weight-specific applications.

Composite properties depend on the size, shape and other physical properties of the reinforcements. The size and the aspect ratio of the reinforcing filler/fibre not only influence the mechanical properties but also the rheology which in turn affects the distribution of filler/fibre and hence the mechanical performance (Ferrigno 1978). Hence filler and fibres are, at times, used in combination to augment each other's properties like in sheet molding compounds (Kabelka *et al* 1996; Kenny and Opalicki 1996) and reinforced syntactic foams (Karthikeyan *et al* 1999). Usage of filler–fibre combination is further expected to reduce the requirement of the amount of matrix material in the composite system. The fly ash is used in unison with fibre either in the form of woven mat (Srivastava *et al* 1988) or in the form of short fibres (Gupta *et al* 2001) with focus on mechanical properties in

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compression and impact. In this study, therefore, an attempt is made to look into the effect of fibre–filler combinations on the defect distribution from the angle of NDE and the attendant changes in the mechanical properties of the composite.

2. Experimental

2.1 *Materials*

The matrix system consists of a medium viscosity epoxy resin (LAPOX L-12) and a room temperature curing hardener with a tetra-amine functional group (K-6) supplied by ATUL India Ltd. The density of cured neat resin is found to be 1120 kg/m^3. Chopped 6 mm length E-glass fibres of density 2500 kg/m^3 treated with silane coupling agent, supplied by Fibre-glass India Ltd are used in the study. The filler used, i.e. fly ash, was obtained from Neyveli Lignite Corporation Ltd., Neyveli (India). This ASTM class 'C' fly ash with bulk density of about 900 kg/m^3 is found to consist of mixture of solid and hollow spheres (figure 1) of assorted sizes. Energy dispersive spectroscopy of the fly ash sample revealed main constituents to be silica and alumina of about 63% and 26%, respectively while traces of other oxides chiefly Fe_2O_3–7% and TiO_2–2·5% were also noticed.

2.2 *Processing*

Measured quantity of epoxy resin was mixed with a pre-weighed amount of fly ash and/or glass fibre and the hardener was added to this with gentle stirring in order to avoid formation of air bubbles. The mixture was then slowly decanted into a mould of size 320 × 170 × 3 mm coated before hand with uniform film of silicone releasing agent. The mixture that was like dough especially for high volume fractions was gently spread to fill the entire mould. The mould was then covered with a heavy lid with its underside having a Teflon sheet smeared with silicone releasing agent. The mixture was left to cure at room temperature for about 24–26 h. Subsequently post-curing was done at a temperature of 75°C for about 90 min. The cured rigid plate sample was withdrawn from the mould and edges were trimmed. In this way, epoxy based systems of constant volume fraction of either filler or fibres were cast (table 1). When both the constituents were introduced into the epoxy the volume fraction of each of these was kept the same to yield a total value that was used to make single reinforcement constituent composites mentioned earlier.

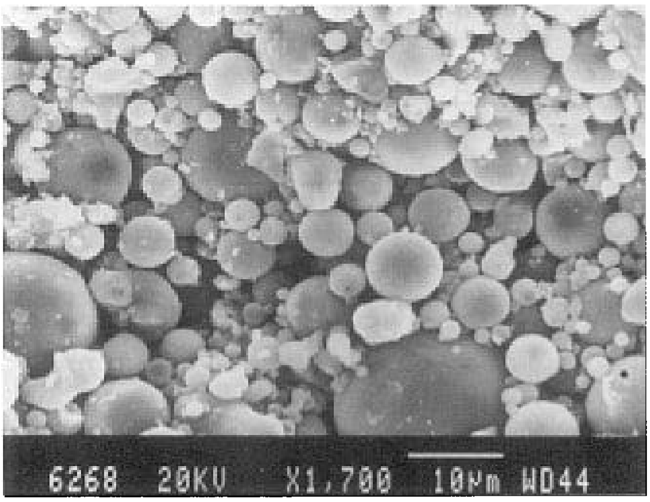

Figure 1. SEM micrograph showing fly ash particles of assorted sizes.

2.3 *Ultrasonic testing*

Testing was done in Ultran NDC-7000 ultrasound testing machine. The machine is computer controlled to scan the sample surface with a probe. Sample movements under the probe are automated so that entire defect mapping of any surface can be performed. Preliminary trial runs were performed with the set up to standardize the data gathering process. From this it was inferred that a 3″ point probe of 5 MHz frequency yielded a good mapping of the attenuation phenomenon. Through transmission technique with water as immersion medium was adopted. A 2-D planar map at half the depth of the specimen was obtained by scanning the sample under the probe. C-scan image, which represents in a colour-code the change in energy of ultrasonic waves in decibels, was captured by a dedicated computer interfaced with the machine. These images are analysed from the mapped attenuation of the ultrasonic energy at different regions to interpret the occurrence of defects.

2.4 *Compression testing*

Compression testing was done using DARTEC 9500, a servo-hydraulic computer controlled testing machine. This machine with a rated load of 100 kN is equipped with an inbuilt software to maintain the constant strain rate by compensating the cross-head travel. The inputs provided to this software are the initial dimensions of the sample

Table 1. Density and void contents for different compositions of ash and fibre.

Sample	Density (kg/m^3)	Voids (%)
Neat epoxy	1120	1·7
30% fly ash	1370	8·1
30% fibre	1788	20·1
15% fly ash + 15% fibre	1452	17·7

(12·5 × 12·5 × 3 mm). With this facility the compression tests were done at a constant strain rate of 0·01 s^{-1}. The machine also has a data logger, which provides the plot of load vs deflection automatically on culmination of the test. Mechanical properties like strength and modulus of the samples were then calculated from the above plot.

3. Results and discussion

When composites are processed through mechanical route, voids are introduced which tend to either distribute or agglomerate to form pockets of clusters in different regions of the sample. The flow during processing generally is influenced by the shape and the volume fraction of the reinforcements. The present study attempts to find out how this void content gets distributed when both filler and fibre are introduced in the composite and the concomitant influences the two introduced systems have on compression strength values.

Figure 2 shows c-scan picture of the neat epoxy sample, where hardly any blue regions, having high-energy attenuation, can be seen. This could be interpreted to mean that very few regions of trapped air (voids) exist within the cast slab. This inference is ably supported from measurement of physical properties, which is also tabulated in table 1. Figure 3 shows c-scan picture of a sample containing 30% fly ash. Here some amount of blue regions can be seen clustered near left and right edges of the sample. A central region also displays the presence of defects. These blue areas are indicative of large attenuation in the ultrasonic energy owing to the presence of clusters or voids. Compared to figure 2 the attenuation being larger in figure 3 can be implied to mean defect content is more in filler bearing composite. This inference too is supported by the void content data presented in table 1 where ash-bearing epoxy has percentage of voids which is more than by a factor of 4. This is understandable as mixing performed to achieve better distribution during processing, yield place to enhancement of air pockets. C-scan picture of sample containing 30% short glass fibres is shown in figure 4, where considerably large amount of these blue areas can be observed indicating a further increase in the amount of voids compared to that seen in either ash bearing composite (figure 3) or neat epoxy (figure 2). This factor is also supported by the data presented in table 1 where an order of change in void increment is seen *vis-à-vis* the neat epoxy case. This increment can be traced to larger churning that each of these higher aspect ratio bearing

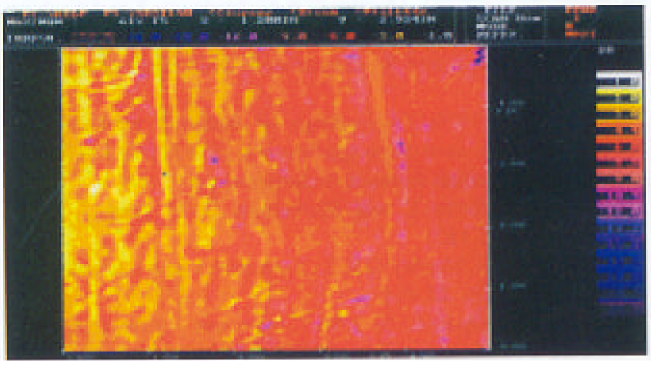

Figure 2. C-scan image of neat epoxy sample.

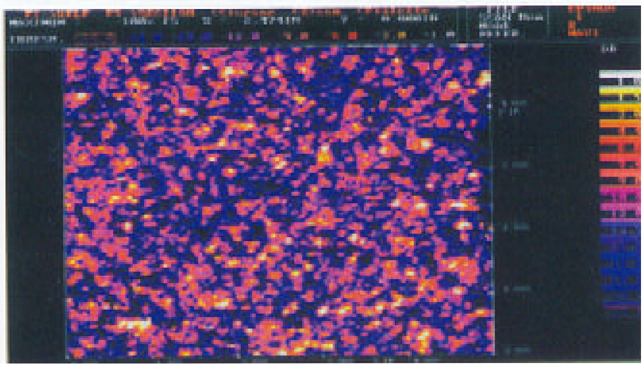

Figure 4. C-scan image of 30 vol.% short glass fibre bearing sample.

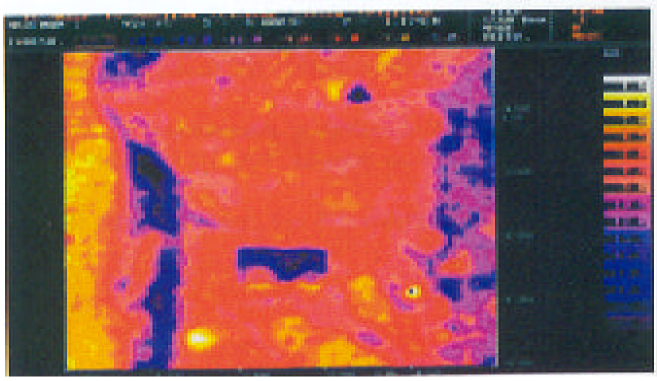

Figure 3. C-scan image of 30 vol.% fly ash bearing sample.

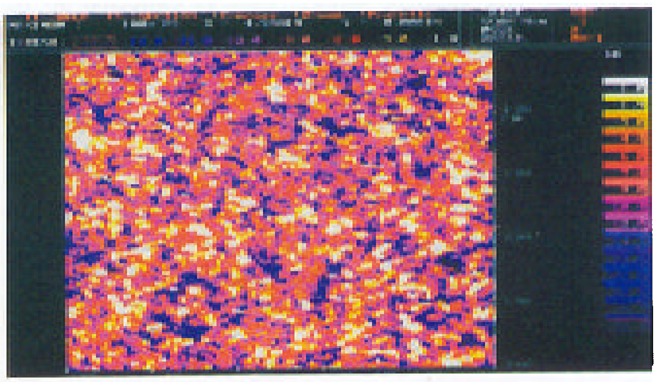

Figure 5. C-scan image of 15 vol.% ash and 15 vol.% fibre bearing sample.

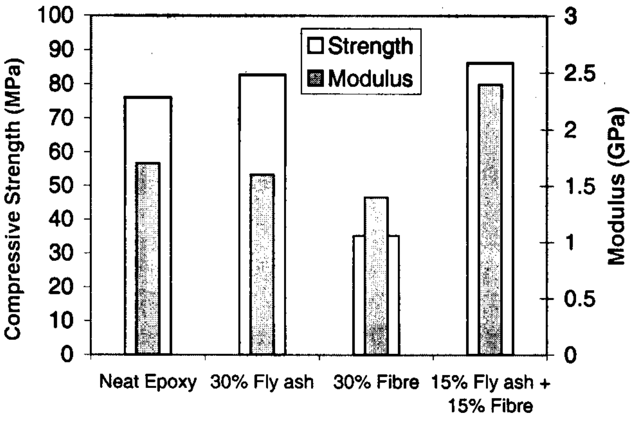

Figure 6. Strength and moduli for ash and fibre bearing composites in compression.

reinforcement bring into the matrix epoxy that surround such fibres. Consequently entrapment of gas pockets leading to formation of voids is a likely process. When fly ash and fibre are added together as reinforcement (15% fly ash and 15% fibre keeping the total reinforcement at 30%), the scenario changes and this is represented in figure 5. It is interesting to note from this picture that an amount of blue area has not only reduced a little as compared to figure 4, but also they are well distributed unlike in figure 3. There is also an increase in the amount of yellow/white regions, which have low attenuation implying sound regions. This improvement could be due to the re-distribution achieved in the presence of filler. Improvement in mechanical properties can also be observed for these filler–fibre hybrid composites (figure 6) coupled with a reduction in density (table 1). It can be noted from the values (figure 6) that the properties in the hybrid system showed an improvement of about 30–40% as compared to the ones where a single reinforcement either ash or fibre was used. The improvement in the properties under compression could be due to reduced population of defects as can be noted from the above NDE analysis or because of the change in the failure mechanism in the presence of particulates (Kulkarni and Kishore 2001). This is expected to be of considerable significance in weight specific applications.

4. Conclusions

When fly ash was used with other reinforcements like fibres having a higher aspect ratio, the amount of voids get reduced and also their distribution is uniform. This way the property changes seen owing to the presence of islands of defect bearing region in a cast laminate is reduced making the reliability of the process better. C-scan study supported by the physical property data measurements, comes in an effective way to identify such systems and processes that yield uniform grade polymerized cast slabs. Further, it was also observed that mechanical properties also showed an improvement of about 30% to 40% owing to introduction of multi-reinforcement into epoxy system coupled with a reduction in density, which is solicited, in weight specific applications.

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