

EPOXY - FLY ASH COMPOSITES - A STUDY ON C-SCAN NON DESTRUCTIVE IMAGE PATTERNS, MICROSCOPY AND STRENGTH AS A FUNCTION OF ASH FILLER CONTENT

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Abstract: Fly ash, a by-product of thermal power plants and available in abundance, is considered not an eco-friendly material from an environmental point of view. If ways are found to use this, the resulting efforts can serve the twin purposes of facilitating applications for the ash bearing materials and at the same time reducing pollution. One way to achieve this task is to make ash-bearing composites involving polymer matrices. The ash particles, generally being hollow in nature, display lower densities while oxides present as constituents make them possess high modulus and strength thereby enhancing specific strength and stiffness of polymer systems which by their very nature have lower densities compared to many metal based systems. An effective way to composite with fly ash would be to adopt the mechanical mixing route. This process inherently induces entrapment of air bubbles, which are seen as voids in cast polymer slabs. Non-destructive evaluation comes in handy to assess this defect content in such cases.

This work presents, first, the non destructive pattern obtained on the plain epoxy system; where after the images obtained due to the introduction of 20 and 30% ash fillers into it are included. These ultrasonic C-scan pictures differing in the attenuation features find a good correlation with void content data which varies from 1.7 for the neat epoxy to 9.9 % for the 30% ash bearing case. The work is extended to non destructive evaluation of glass fiber bearing systems and then to a measurement of modulus and correlating this mechanical data with C-scan pictures and features seen under scanning electron microscope.

Introduction: The ability to tailor the composites for specific end applications has been one of the key factors in their usage being expanded in a comparatively shorter period. However, many challenges need be overcome before a successful deployment of the composite component is made. The wide choice available first for choosing materials and their combinations and then in the type of processing methods to be employed to yield the product make the comprehensive selection a truly challenging one. The scientific approach in such cases comes in handy [1]. The configuration of a composite is unique in that it includes matrix, reinforcement and the interface, which could all be varied to attain a specific attribute. The development of composites is a complex one requiring consideration of various parameters such as interface chemistry, component geometry, production volume, shape of reinforcement and the type of matrix molecules, the tooling requirements and finally the costs involved. All these may result in an end product which looks acceptable from a macroscopic outwardly appearance but whose defect population could well be outside the prescribed limits. Hence, in this context, the need for a defect mapping on the components of the prototype, before they can be integrated, arises. Here is where the non-destructive evaluation (NDE) helps by testing the prototypes for their precise performance. The defective regions/defect bearing systems, thus identified, are excluded which in turn aid in reducing the risk of lower performance by the prototype. With a good base built as far as homogenous materials are concerned, stretching its use to characterize heterogeneous materials such as composites has gained importance in the recent times [1] as is evident from a perusal of the literature. Composites on account of their processing schedule and constituent materials are prone to the existence of defects like voids, cracks, debond and delaminations within them. Many non-destructive testing and evaluation techniques [2] are used for identifying these defects. Among these, the ultrasonic testing, owing to its simplicity of usage and ability to test large

components, is widely preferred to tackle the issue of mapping of the defects in composite materials [3-5]. Polymer composites generally are reinforced with fiber or fillers to impart good mechanical properties. Composite properties depend on the size, shape and other physical properties of the reinforcements. The size and the aspect ratio of the reinforcing filler/fiber not only influence the mechanical properties directly but the also through another factor involving the rheology of the system which affects the distribution of filler/fiber and hence the mechanical performance [6]. Hence, filler and fibers are, at times, used in combination to augment each other's properties like in sheet moulding compounds and reinforced syntactic foams [2]. Usage of filler/fiber is further expected to reduce the requirement of the amount of matrix material. An attempt is made in this study to look into the effect of fiber/filler on the defect distribution and the attendant changes in the mechanical (compressive modulus) property of the composite from the view point of NDE. Abundantly available fillers, inexpensive and possessing good mechanical properties, when used with well established matrix systems help to reduce the cost of the system, and at the same time either retain or improve a specific and desirable mechanical property. Fly ash, a fine particulate waste from thermal power plants has attracted interest [7] lately, because of the abundance in terms of the volume of the 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 (termed cenospheres, [8, 9]), which aid in maintenance lower density values for the composite, a feature of considerable significance in weight-specific applications.

Experimental:

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 . E-glass fibers of density 2500 kg/m^3 treated with silane coupling agent chopped to 6 mm length, used in the study, are supplied by Fiber-glass India Ltd. 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 of assorted sizes (Fig. 1). 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.

Processing: Measured quantity of epoxy resin was mixed with a pre-weighed amount of fly ash or glass fiber 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 mm X 170 mm X 3 mm coated before hand with uniform film of silicone releasing agent. The mixture that was like dough, especially for higher volume fractions of reinforcements, 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 hrs. Subsequently, post-curing was done at a temperature of 75°C for about $1\frac{1}{2}$ h. The cured rigid plate sample was withdrawn from the mould and the edges trimmed. In this way, epoxy based systems with varying amounts of filler/fibers (Table 1) were cast. The samples were designated as FA for fly ash filler bearing and GF for glass fiber bearing systems respectively. The number that follows these abbreviations denotes the volume percentage of reinforcement present in the system.

Ultrasonic and Mechanical testing: Ultrasonic 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 the defect mapping of entire surface can be performed. Preliminary trial runs were performed with the set up to standardize the data gathering process. From this it was observed that a 3" point probe with a frequency of 5 MHz yields a good mapping of 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 picture, in color code indicating the change in energy of ultrasonic waves, in decibels, was captured by a dedicated computer interfaced with the machine [10]. These images are analyzed to seek information on occurrences of defects.

Compression testing was done using DARTEC 9500, a servo-hydraulic computer controlled testing machine as per the guidelines in ASTM standard. 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 (12.5 mm X 12.5 mm X 3 mm). With this facility the compression tests were done at a constant strain rate of 0.01s^{-1} . The machine also has a data logger, which provides the plot of load vs. deflection automatically on culmination of the test. Moduli of the samples were then calculated from these plots.

Results and Discussion: The data will be first seen in the light of void content (Table 1) for three compositions of fly ash namely FA10, FA20 and FA30. Then, the three glass bearing compositions of GF10, GF20 and GF30 will be considered. It is noticed that the void content increases from FA10 to FA30 thro' FA20. As far as modulus is concerned, it increases from FA10 to FA20 but decreases for FA30. Thus, a physical data like void content and the modulus values do not show similarity in trends and in this work, hence, recourse to NDE was taken to see whether it can be used to correlate the mechanical property with defect content. The C-scan pictures (Figures 2-5) show the features in neat and FA's of 10, 20 and 30 volume percent ash bearing composites, respectively. From these illustrations, it is seen that where as Fig. 3 shows some defect bearing regions in the sample, this area in Figs. 4 & 5 are lower (FA20) and higher (FA30), respectively. Correspondingly, the modulus for FA 10 to FA 20 shows an increase, while for FA 30 (which showed an increase in defect bearing region) there is a lowering of the value. Thus, the work shows for fly ash bearing system, a direct correlation between defect content (qualitatively assessed based on the attenuation features recorded in these pictures), and the relative values for modulus. Should the modulus be only dependent on ash level, the values should have showed a continuous rise from FA10 to FA30 through FA20, as fly ash is made of inorganic oxides and are know to have a higher modulus compared to the (cured) epoxy matrix material.

It will now be examined for only glass fiber bearing samples whether this variation in values for modulus can be related to ND pictures. Figures 6-8 represent the ND images of glass fiber samples. Before starting on an analysis of the C-Scan pictures, the earlier done comparison between void content and modulus will now be examined. The void content rises rather uniformly from 16.1 to 20.1 for GF10 to GF30 through GF20, which incidentally has void level of 17.3%. But the modulus for GF10 has a high value while the values for GF20 and 30 are much lower and between themselves they differ marginally. Thus, even in this case of glass fiber, the modulus and void content do not show a one to one correlation. Hence, for the glass reinforced (GF) systems also, when NDE was adopted, it showed least the defect-bearing region for GF10, which has the highest modulus. The pictures obtained for GF20 and GF 30 compare well in that both show a fairly wider distribution for high attenuation areas. Earlier, it was seen (Table 1) that these two have a nearly similar value for modulus. Thus, the correlation of modulus values and the C-scan pictures compare well when the reinforcement is changed from near about spherical-looking fly ash to high aspect bearing glass fibers.

Coming to microscopy, two cases in FA will be considered. Fig. 9 shows the features in matrix around ash particles in FA20. The deformation marks in the matrix are noticeable and hence the resistance offered by fly ash and therefore the higher modulus (2.16 GPa) recorded by this system. FA30 (Fig.10) shows the clear interface debonds and cracks [11]. The sample records 1.6 GPa for the modulus (Table 1). Thus there is a correlation with microscopy for these composite materials. Fig. 11 shows the SEM view of GF20 sample. Note the matrix debond from the fibers. The sample recorded value of 1.4 GPa for the modulus (Table 1). This could mean that good

adhesion at the matrix-filler/fiber interface leads to recording of higher mechanical values and vice versa.

Conclusion: Both fly ash and glass fiber in epoxy modify its modulus values. The ash bearing composites display irregular properties with increase in the content. Correlation with void content was not feasible. However, with C-scan pictures a fair degree of correlation could be found. This better correlation was seen with glass fiber-bearing samples too. The qualitative assessment of the sound area by C-scan could be used to judge the trends in modulus value. The SEM pictures also corroborate the nondestructive evaluation results.

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Sample	Density, kg/m ³	Void content, vol. %	Modulus, GPa
Neat epoxy	1120	1.7	1.7
FA 10	1235	1.3	1.9
FA 20	1282	6.1	2.16
FA 30	1370	9.9	1.6
GF 10	1363	16.1	2.27
GF 20	1641	17.3	1.4
GF 30	1788	20.1	1.39

Legend : FA followed by Number indicates the filler as Fly ash by Vol %

GF followed by Number indicated the filler as Short Glass fibre by Vol %

Table 1 Table showing the Densities and the void contents of the samples

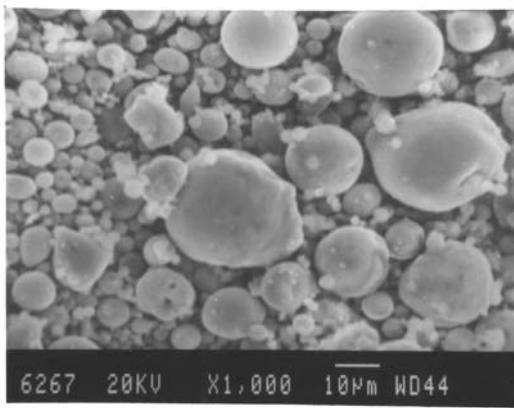


Figure 1 SEM Picture showing the nearly spherical fly ash particles of assorted sizes

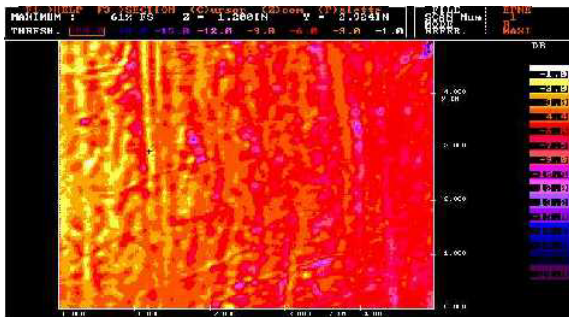


Figure 2 C-scan picture of Neat epoxy (No fly ash/no glass) sample

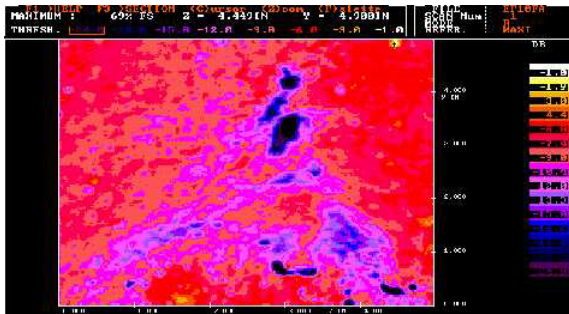


Figure 3 C-scan picture of FA10 sample

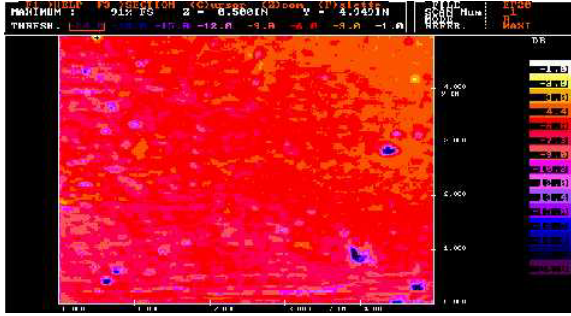


Fig. 4 C-scan picture of FA20 sample

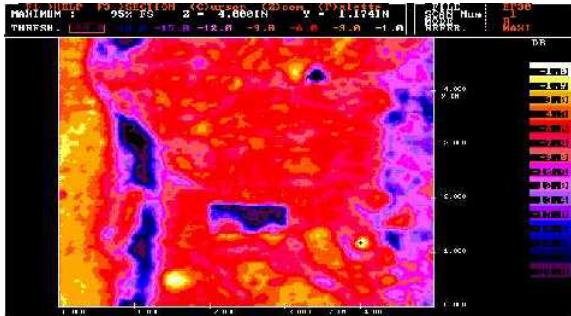


Fig. 5 C-scan picture of FA30 sample

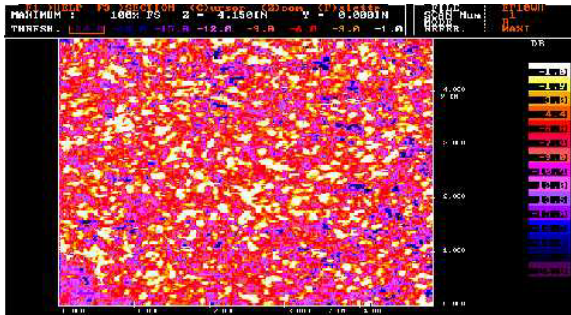


Fig. 6 C-scan picture of GF10 sample

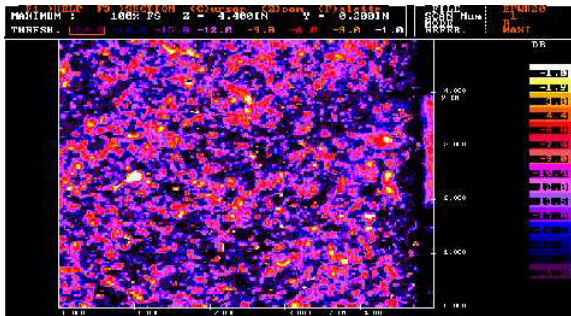


Fig. 7 C-scan picture of GF20 sample

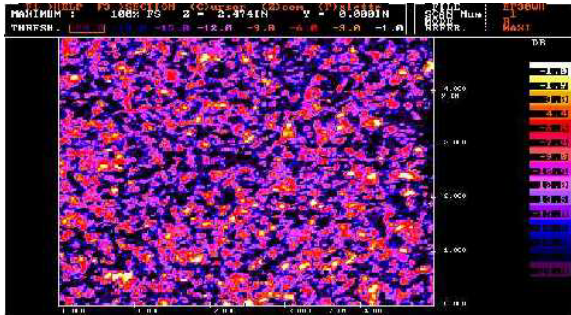


Fig. 8 C-scan image of GF30 sample

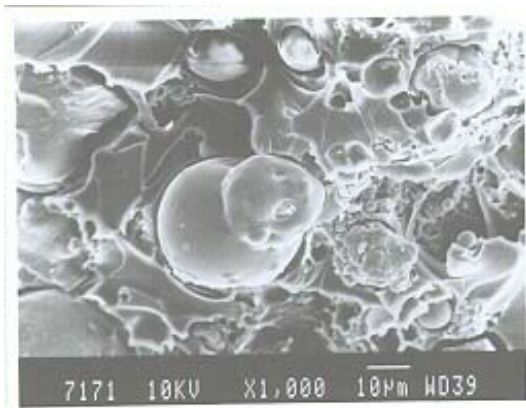


Fig. 9 SEM micrograph of FA20 sample

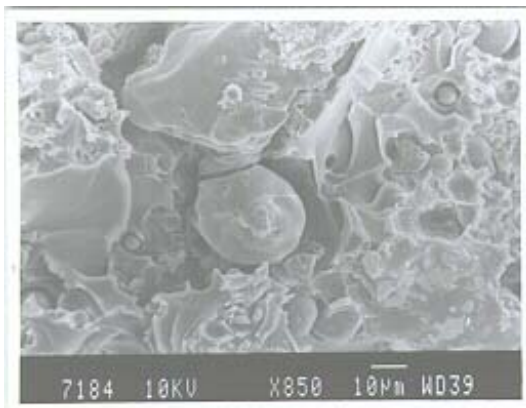


Fig. 10 SEM micrograph of FA30 sample

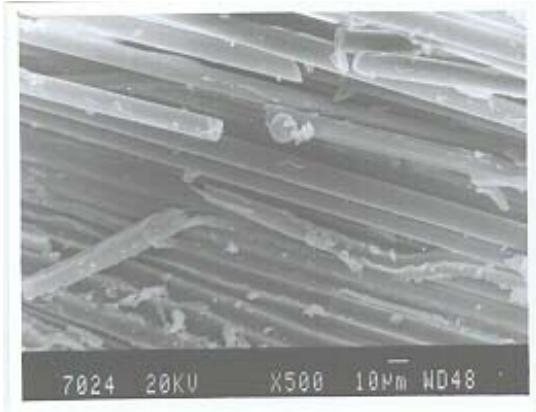


Fig. 11 SEM picture of GF20 sample