International Journal of Advancements in Mechanical and Aeronautical Engineering – IJAMAE
Volume 1 : Issue 1
Publication Date : 15 May 2013

Effect of Unsilanized and Silanized Fly Ash on Damping Properties of Fly Ash Filled Fiber Reinforced Epoxy Composite.

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Abstract— **The damping characteristic of fly ash filled fiber reinforced epoxy resin was examined by varying the fly ash concentration from 0 to 30% by weight. Dynamic Mechanical Analyzer (DMA) was used to determine the temperature dependency of damping factor (*tan δ*). The testing was done under 3-point bending mode with frequency input of 10Hz at ramp rate of 5°C/min. The unsilanized and silanized fly ash concentration effect was compared and fly ash concentration limit for better damping property was ascertained. It was found that the glass transition temperature (*Tg*) and *tan δ* values improved with addition of fly ash up to certain point beyond which any further addition decreased *Tg* and *tan δ*. However the storage modulus and loss modulus increase with an increase in fly ash concentration. It was also found that silanized fly ash had lower *tan δ* value as compared to unsilanized fly ash.**

Key words— **fly ash, coupling agent, DMA.**

I. INTRODUCTION

Polyepoxide, commonly known as epoxy, finds wide range of applications such as composite material, adhesives, coatings etc. because of its excellent heat and moisture resistance, low volatility and good adhesion to many substrates. However, its use is very limited due to lack of dimensional stability and low heat distortion. This draw back can be overcome up to certain extent by using fibers and other inorganic materials such as quartz, kaolin, clay, talc, fly ash etc. as filler.

Fly ash is a byproduct of coal based power plant and is recognized as an environmental hazard, due to which considerable research work has been undertaken worldwide to find its alternate usage. Fly ash is used in Portland pozzolana cement, manufacturing of ash bricks and other building products. Wang et al [1] found it as promising absorbent for removal of various pollutants like So_x, No_x, organic compounds and mercury in air.

Many researchers investigated the effect of fly ash filled composite [2-5]. Gupta et al [6] studied impact resistance and compressive strength of fiber reinforced fly ash composite and concluded that with small amount of fly ash, compressive strength decreases but impact resistance increases by 300%. Chaowasakoo et al [7] analyzed the effect of thermal and microwave curing on epoxy/fly ash composite and concluded that microwave cured samples had lower tensile moduli. Chauhan et al [8] examined the wear resistance and dry sliding behavior of epoxy/fly ash composite and concluded that wear resistance increases with fly ash addition. Baccaro et al [9] studied the effect of radiation induced in epoxy/fly ash composite. The test result showed that irradiation decreases the flexural strength but increases tensile strength, hardness and impact strength. Kulkarni et al [10] studied the effect of surface treatment and size of fly ash particle on compressive strength and found that silane treatment improves the strength and smaller particles size give better strength. Yadong el al [11] examined the fly ash filled recycled PET and found that compressive strength was significantly increased and the linear shrinkage was reduced by 7 times with higher amount of fly ash concentration. Sen et al [12] investigated the cured and uncured state properties of fly ash filled unsaturated polyster composite and concluded that addition of unsilanized fly ash increases the cure time.

Fiber-reinforced thermoplastics have typical advantages over polymer matrix composites such as high strength, high stiffness and corrosion resistance and vibration energy absorption [13]. Damping of vibration energy is very momentous in the design of automotive, marine and aerospace structures. Many efforts have been made to control the vibration and noise. One of most common methods is the application of a viscoelastic material, such as a rubber with a broad and high damping peak in glass transition region (Tg). However, the improvement in the damping efficiency of such a material is limited to some extent as *Tg* of most rubbers are lower than the application temperature and the enhancement of damping usually causes corresponding reduction in stiffness and strength [14].

The objective of this work is to study the effect of silanized and unsilanized fly ash concentration on the damping properties of glass fiber reinforced polymer composite and ascertain the fly ash concentration limit that increases the damping.

II. MATERIAL

A. *Resin*

Araldite GY-257 (Bisphenol-A, mono-functional reactive diluents) epoxy resin was used as structural matrix due to its low viscosity (500-650 mPa-s) and density (1.15g/cm3). Its congenial hardener, HY-951, was mixed in ratio of 100:10 by weight.

B. *Fibers*

E-glass fiber of 10 µm diameter was used as reinforcement. The fiber was cut into 1" size and reinforced into epoxy matrix with random orientation.

C. *Fly ash*

Class F-fly ash was conflated in the fiber reinforced matrix in varying concentration to suss out its effect in composite. Fly ash is the ash particle which escapes through the coal based power plant chimney along with flue gases. It can be extracted by electrostatic precipitators. The average particle size used in experiment was found to be 2.06 µm, as shown in Fig. 1. The assorted shape and size of fly ash as observed in SEM analysis is shown in Fig. 2. The total content of silica and alumina was ascertained to be 74.2%. The overall chemical composition of fly ash is listed in Table 1.

Table 1: Chemical composition of fly ash

Testing of fly ash as per IS:1727-1985 Sample identification: **FLY ASH**		
Sr. no.	*Testing parameters*	*Test values (%)*
1	Loss of ignition	1.2
2	silica	56.5
3	Ferris oxide	11.0
4	Alumina (Al2O3)	17.7
5	Magnesia	5.4
6	Calcium oxide	3.2

D. *Coupling Agent*

γ-aminopropyl triethoxy silane was selected for the substrate treatment due to its compatibility with epoxy and substrate. It has low specific gravity (0.94) and refractive index (1.420) at room temperature.

III. PROCESSING

A. *Mould*

Two mould plates each of size 480 X 260 X 10 mm were braced together to prepare the composite plate of 450 X 330 X 2 mm size. Specimens of size 50 X 6 X 2 mm were cut from the plate for DMA testing.

B. *Substrate treatment*

The fly ash treatment was done with 1% of silane coupling agent and later dried at 150°C for 10-12 minutes. The amount of silane was determined by the following expression:

$$Wt.\,of\ silane(g) = \frac{amt.\,of\ filler\,(g)\,X\ surface\,area\,of\ filler\,(m^2/g)}{min.\,coating\,area\,of\ silane\,agent\,(m^2/g)} \qquad (1)$$

C. *Sample preparation*

The required amount of araldite and hardener were mixed together slowly to forfend the bubbles formation chances. Then glass fiber and there after fly ash were appended and stirred gently for protracted duration for proper mixing of ingredients. The mixture was then degassed at 40-50°C for another 5 minutes and compressed in between the mould plate inside the hydraulic press at 1.5 ton per sq. in. It was allowed to cure for 10-12 hours at normal room temperature and further for 10 hours at 90°C.

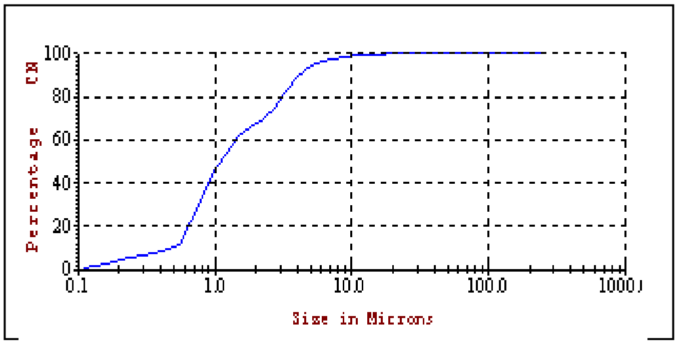

Figure 1: Average fly ash particle size.

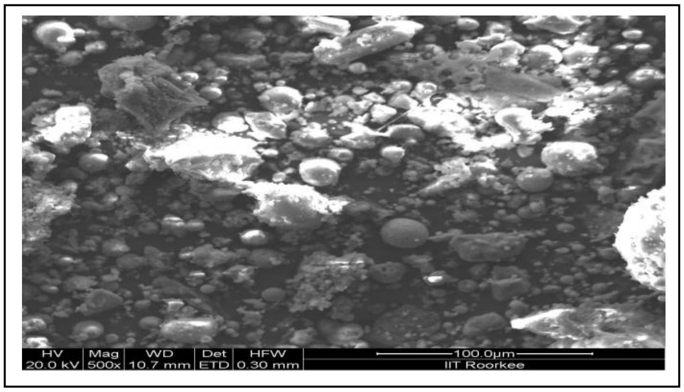

Figure.2: SEM micrograph of fly ash particles

Two different sets of samples were prepared, one set with untreated fly ash, denoted as "EP/FR/FA" which has fiber (4% by weight), fly ash (varying from 0-30% by weight) and remaining part as epoxy. The second was prepared with silane treated fly ash and denoted as "EP/FR/FA/SC". The sample characterization is listed in Table 2.

Table 2 Sample characterization:

Sample	***Fiber***	***Fly ash***

EP/FR/FA-1 or EP/FR/FA/SC-1	4%	0%
EP/FR/FA-2 or EP/FR/FA/SC-2	4%	05%
EP/FR/FA-3 or EP/FR/FA/SC-3	4%	10%
EP/FR/FA-4 or EP/FR/FA/SC-4	4%	15%
EP/FR/FA-5 or EP/FR/FA/SC-5	4%	20%
EP/FR/FA-6 or EP/FR/FA/SC-6	4%	25%
EP/FR/FA-7 or EP/FR/FA/SC-7	4%	30%

IV. EXPERIMENTAL SETUP

The experiment was performed in Triton Dynamic Mechanical Analyzer under 3-point bending mode. DMA is a thermal analysis technique used to characterize the viscoelastic behavior of materials. The sample is mounted inside an environmental chamber and the storage modulus (elastic response) and loss modulus (viscous response) are measured as a function of temperature by applying small sinusoidal stress to the sample at fix rate and measuring the resulting displacement. The magnitude of applied stress and the resultant strain are used to calculate the stiffness of material under stress and the phase lag (δ) between the two is used to determine *tan δ*, the damping factor. The working model of DMA is shown in Fig. 3.

To determine the damping property of composite, which is expressed in terms of tan δ, the sample of 50 x 6 x 2 mm size was mounted between the ends of two parallel arms inside the machine clamp. Temperature was varied from room temperature to 180°C with deformation frequency of 10 Hz and ramp rate of 5°C/min.

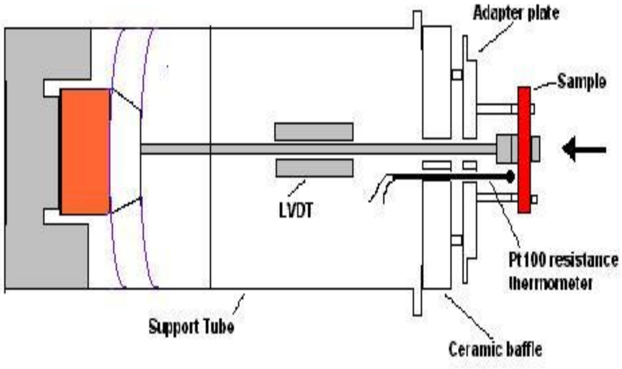

Figure 3: DMA working model.

V. RESULTS AND DISCUSSION

In withering of vibration, termed as damping, kinetic energy is converted into heat energy. These fritter away process inside the material is called material damping. The damping property of polymeric composite is mainly dependent on viscoelasticity of matrix material and therefore *tan δ* value reaches its peak point only at *Tg*. In addition, material damping is also affected by the inclusion of fibers and fillers.

A. Effect of fly ash concentration

Figures 4 and 5 show the temperature dependency of *tan δ* value of unsilanized and silanized fly ash filled composite respectively with varying concentration of fly ash from 0 to 30% by weight. Both figures show that with an increase of fly ash concentration, *tan δ* and *Tg* value initially increase but later they decrease with further inclusion of ash concentration. The composite filled with fly ash have higher value of *Tg* and *tan δ*. The *tan δ* has its peak value of 0.44 at *Tg* of 73.4°C with 15% by weight of unsilanized fly ash concentration and 0.47 at Tg of 74.1°C with 20% by weight silanized fly ash concentration, which are higher than the other 6 samples.

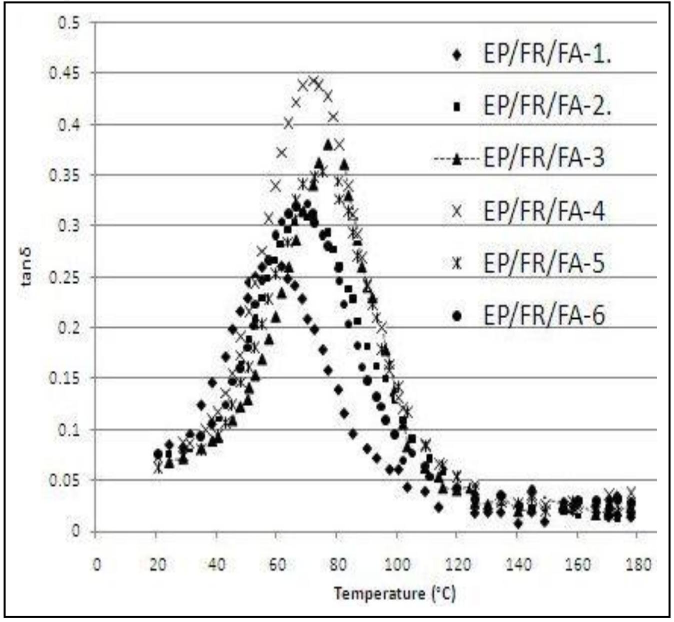

Figure 4: tan δ curve for unsilanized fly ash composite.

Therefore, it is implied that, addition of fly ash improves the damping property and increases the glass transition temperature. The increase in *tan δ* value is probably due to friction between the clumped fly ash particles or between the fly ash particle-matrix interfaces where adhesion is improper.

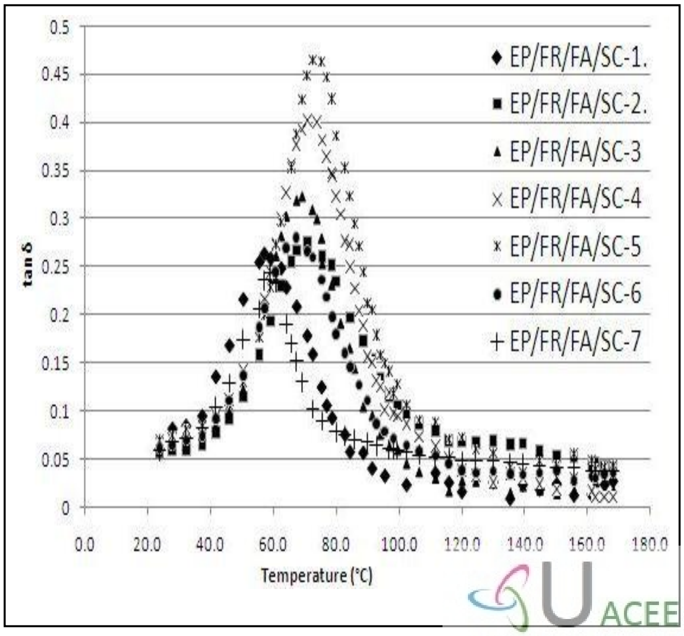

Figure 5: tan δ curve for silanized fly ash composite

The increase in *Tg* value is most likely due to the segmental immobilization of fly ash surface with the increase in its concentration. This result also suggests that the degree of cross linking increases as a result of addition of fly ash [15]. However, addition of fly ash beyond this concentration results in decrease in both *Tg* and *tan δ* value. This decrease can be attributed to the decrease of the matrix material which is the prime reason of material damping due to its viscoelastic nature. Hence more fly ash addition beyond a proper limit results in decrease of damping capacity of composite.

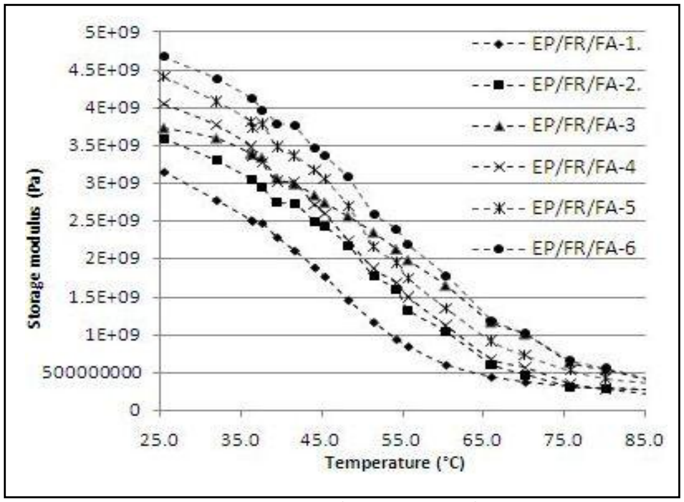

Figure.6: Storage modulus curve for unsilanized fly ash composite.

Fig. 6 shows that the increase in fly ash concentration increases the stiffness. With the addition of fly ash, there is consistent increase in the storage modulus, which represents stiffness. Moreover, it can also be inferred from the figure that beyond the glass transition temperature, the storage modulus is significantly reduced as compared to its value at room temperature resulting in the peak formation of *tan δ*.

B. Effect of silanization of fly ash

The surface treatment acts at the interface between an inorganic substrate and an organic material which results in good interfacial adhesion of fly ash particles with the epoxy matrix.

Table 3 shows that both unsilanized and silanized fly ash filled fiber reinforced epoxy composite have an increasing trend of tan δ value up to certain fly ash concentration and then decreases. However unsilanized fly ash particle filled composite have higher tan δ value when compared with the same concentration of silanized fly ash.

Table 3: Peak Tg and tan δ value of different samples with varying fly ash concentration

Fly ash wt %	***Unsilanized fly ash***		***Silanized fly ash***	
	Tg (°C)	***tan δ***	***Tg (°C)***	***tan δ***
0	59.70	0.24	59.70	0.24
5	70.30	0.30	70.00	0.27
10	74.70	0.37	69.00	0.32
15	73.10	0.44	72.45	0.40
20	73.60	0.36	74.10	0.47
25	67.40	0.32	67.37	0.28
30	--	--	59.20	0.24

This can be attributed to the additional cross linking points created between filler and matrix, formed due to the silanization, resulting in good interfacial adhesion and leads to lower internal friction which ultimately reduces the damping values [16].

Furthermore, it can also be seen with the experimental data that silanized fly ash filled composite can incorporate more amount of fly ash and have higher *tan δ* value at 20% by weight fly ash concentration. Therefore it can be said that, silanization of fly ash improves the damping capacity and glass transition temperature by incorporating higher fly ash concentration which further increase the stiffness of the composite.

VI. CONCLUSION

It is observed that silanization of fly ash results in a higher peak value for *tan δ*. This peak value in case of silanized fly ash corresponds to a higher fly ash concentration as compared to the concentration corresponding to the maximum value of *tan δ* in the unsilanized fly ash. The glass transition temperature initially increase with addition of fly ash and thereafter reduces with further addition of fly ash. The peak value of glass transition temperature is higher in case of silanized fly ash and it also corresponds to a higher concentration of fly ash as compared to the unsilanized case.

ABBREVIATIONS and ACRONYMS

1. amt. – Amount
2. min. – Minium
3. EP – Epoxy
4. FR – Fiber
5. FA – Fly ash
6. SC – Silane coupling
7. SEM – Scanning electron microscope
8. wt. – Weight

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