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Investigation on the Dynamic Mechanical Properties of Self-Healing Glass Fibre Reinforced Plastics

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ABSTRACT:

Self-healing polymeric materials developed in the last decade is one of the marvels in the field of material science and polymer chemistry. Self-healing Glass Fibre Reinforced Plastics (GFRP) was fabricated with the microcapsule based self-healing system which can be triggered by the catalyst, when the capsule breaks open releasing the healing agent, during crack formation. The dynamic mechanical properties of the composite were assessed to find its dependence on temperature, stress and frequency and to report the changes in stiffness and damping. The storage modulus, loss modulus and damping factor were investigated for various frequencies and temperature and discussed.

KEYWORDS:

Dynamic mechanical analysis; Self-healing polymer; Glass fibre reinforced plastics; Frequency sweep; Damping factor

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1. Introduction

Any material carefully developed in the laboratory reaches its functional importance, only when it is able to survive real time loading conditions. Composites, which are commonly used in structural applications, are often subjected to changes in thermal and mechanical stresses in real time applications. Hence a material needs to be tested for its functionality based on the changes in thermal and mechanical stresses simultaneously, rather than testing on any one static change. These kinds of real time simulation of both thermal and mechanical stresses have been made possible using Dynamic Mechanical Analysis (DMA). The mechanical behaviour and properties of polymeric materials under harsh conditions, becomes critical due to its viscoelastic nature and it can be best described with the change in time, temperature, stress and frequency using DMA. In the DMA the following 3 parameters are used:

- Storage modulus: A measure of maximum energy stored in the material;
- Loss modulus: A measure of energy dissipated as heat;
- Damping factor: The ratio of loss modulus to storage modulus.

The dynamic mechanical properties are dependent on the type of fibre, its orientation, volume of fibres, fillers, impact modifiers, coupling agents, mode of testing etc. [1-4]. The glass transition temperature (Tg) where the material moves from hard to rubbery state can be found using the curves of loss modulus (E'') and damping factor (tan δ) [1]. It is also reported that the increase in frequency rate for a constant heating rate shifts (Tg) to the higher end [1, 5].

Kumaresan et al [6] stated that Tg and E'' values increase with increase in silicon carbide filler material in carbon-epoxy composites. Manikandan et al [7] investigated the dynamic mechanical properties of polystyrene composites reinforced with sisal fibres and found that the increase in fibre loading decreases the height and increases the width of (tan δ) curve. Laly et al [8] on their study on the dynamic mechanical properties of banana fibre reinforced polyester composites, highlights that additional peak in the (tan δ) curve is observed which is due to the micromechanical transitions by the immobilised polymer content at high fibre loading. Autonomic self-healing polymeric composites have been a promising field of research, initiated by White et al [9]. Research in this field later gained momentum and was followed by numerous researchers conducting studies by changing the factors involved in the self-healing system like usage of hollow glass fibres for storing the healing material [10], healing system using micro vascular networks [11], vasculature embedded in the composite structure providing healing agent through an external source [12] etc.

The micro encapsulated self-healing composites involve addition of microcapsules filled with healing agent in the matrix material, which is the filler for the composite. The healing agent that is commonly used is Di Cyclo Penta Diene (DCPD) in monomer form which undergoes Ring Opening Metathesis Polymerisation (ROMP) in the presence of Grubb's catalyst. In this study, the dynamic mechanical effect of the microcapsule added GFRP composite is explored at different frequencies at a constant heating rate and the changes in stiffness and damping are reported.

2. DMA

Epoxy LY 556 of bisphenol a diglycidyl ether polymeric resin was used as the matrix material reinforced with E-glass fibre mats of 300gsm by hand layup technique. Microcapsules of diameter 300µm to 900µm made of urea formaldehyde shell filled with dicyclopentadiene monomer was made by the procedure discussed by Brown et al [13]. These capsules along with the I generation Grubb's catalyst were mixed in the epoxy resin before the hand layup process. Grubb's catalyst is the well-defined ruthenium catalyst for olefin metathesis reaction. Grubb's catalyst and microcapsules were mixed with 2.5% by weight and 15% by weight respectively of the total weight percentage of epoxy resin before the layup process. Self-healing GFRP plate of 300mm×300 mm×3mm was fabricated and cut to required specimen dimensions for the study. The microcapsules fabricated for the study and the hand layup of the self-healing GFRP specimen is shown in Fig. 1.

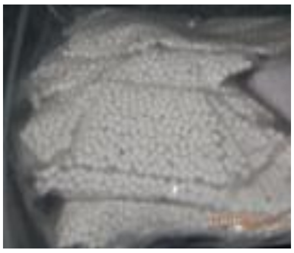

Fig. 1(a): DCPD filler **(b): Microcapsules mixed in epoxy**

DMA was conducted using Dynamic Mechanical Spectrometer DMS6100, as shown in Fig. 2, having a force range of +/-7.8 N. Specimen of size 20mm×10mm was cut and the measurement was done according to ASTM D-4065-01 through dual cantilever bending in synthetic oscillation mode to measure elastic modulus transformation at multiple frequencies. The temperature was varied from 50ºC to 220ºC at the constant rate of 2ºC/min, and tested for frequencies of 0.5Hz, 1Hz, 2 Hz, 5 Hz, 10Hz. The synthetic wave oscillation mode of the spectrometer allows varying between 5 different frequencies.

Fig. 2: Dynamic mechanical spectrometer

3. Results and discussions

The sample was subjected to dynamic frequency scan to get the material flow-elasticity or stiffness as a function of frequency. Sampling frequency with simultaneous temperature scans is the right way to test polymers, due to its viscoelastic nature which turns the material soft at a temperature influencing the dynamic modulus of the material. The storage modulus curves for different frequencies and different temperatures are shown in Fig. 3. A decrease in E' with the decrease in frequency till self-healing GFRP reaches its glass transition phase after which the material turns viscous and the modulus becomes very less. Storage modulus curve can be segmented into 3 different regions-low temperature glassy regions, steep descent in modulus region and the high temperature viscous region [14]. The self-healing GFRP has a steep decrease in E' between 70ºC to 110ºC. Less frequency means the material has more time to respond to the temperature as time and frequency are inversely proportional and so the modulus value decreases [15].

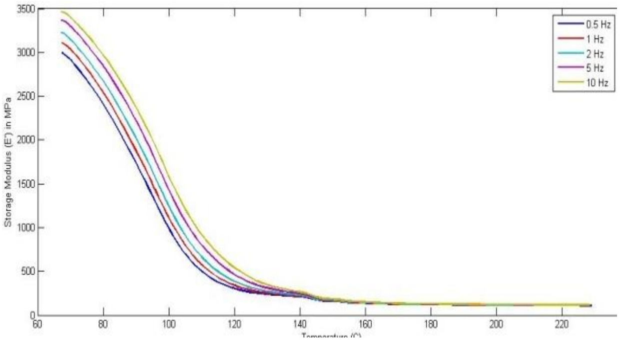

Fig. 3: E' vs. Temperature for various frequencies

Fig. 4 shows the loss modulus (E") curves as a function of temperature at varied frequencies. The peak in loss modulus curve indicates the glass transition region Tg which varies across different frequencies. The Tg represented through loss modulus curve was found to increase with increase in frequency as low frequencies allows the material to flow for long time thus decreasing the loss modulus. The Tg values varied between 85ºC to 110ºC for different frequency values. Till the material reaches the rubbery state the dissipation of heat is higher and after that the material takes in heat in its viscous nature and the curve drops down. The interfacial adhesion between the microcapsules, matrix and glass fibres of Self-healing GFRP can be well understood using the damping curve shown in Fig. 5. The peak of the damping curve denotes Tg of the composite material. Damping peak is affected by the bonding between the individual materials in the composite, curing conditions, nature of the fillers, reinforcements etc. [1]. It was found that the (tan δ) values increased with increased frequencies. The Tg value from the (tan δ) curve ranged across 105ºC to 130ºC for different frequencies. Table 1 summarises the Tg values obtained from loss modulus curve and tan δ curve. The Tg obtained from the loss modulus curve is lesser than the Tg values obtained from (tan δ) curve [1].

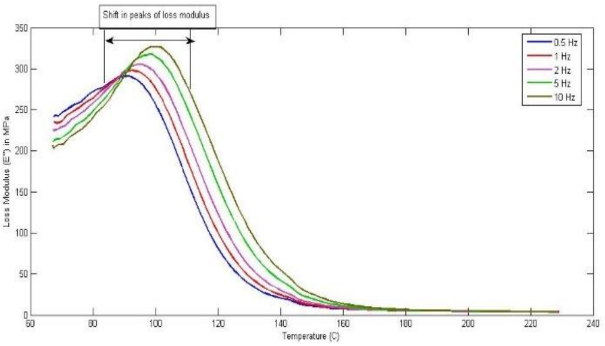

Fig. 4: E'' vs. Temperature for various frequencies

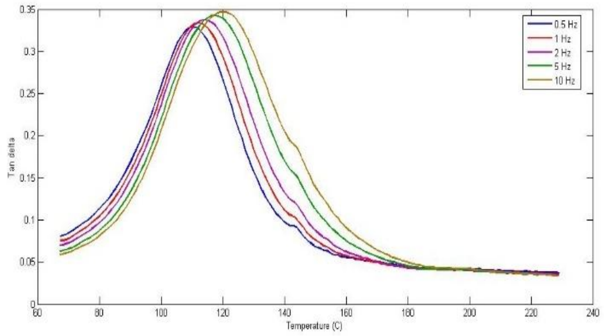

Fig. 5: Damping factor vs. Temperature for various frequencies

Table 1: Tg values obtained from (tan δ) curve and E" curve

Frequency (Hz)	Peak height of tan δ	Tg from tan δ curve (ºC)	Tg from E" curve (ºC)
0.5	0.32	110	98
1	0.33	113	100
2	0.337	115	100
5	0.34	118	101
10	0.35	121	102

Cole-Cole plot is constructed with log (E') vs. log (E") values and its semi-circular nature of the curve indicates that the material is homogenous [16]. To describe the visco-elasticity of a polymer, single relaxation peaks are not enough. Hence, Cole-Cole plot of self- healing GFRP as shown in Fig. 6 is considered as it expresses the dielectric data [2]. The non-semi-circular curve shows that the self-healing GFRP is heterogeneous and indicates good fibre-microcapsules-matrix adhesion. Till the glass transition value, the material behaves elastic. Hence frequency shows little deviation in the Cole-Cole plot. Whereas after the material turns viscous, the frequency doesn't seems to affect.

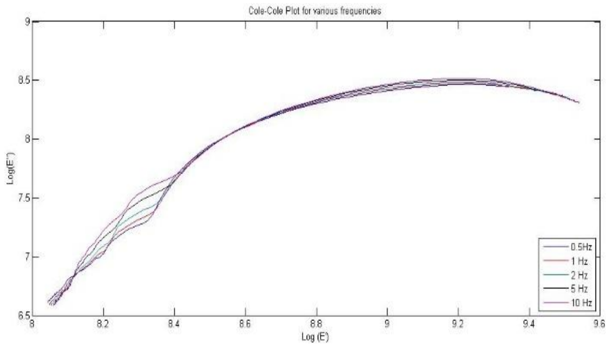

Fig. 6: Cole-Cole plot for self-healing GFRP

4. Conclusion

The dynamic mechanical properties like storage modulus, loss modulus and damping factor were tested and analysed for different frequencies for a constant temperature increase. The viscoelastic behaviour was observed clearly from the data obtained and the Tg values obtained from loss modulus curve were correlated with the Tg obtained from the (tan δ) curve. It was observed that increase in frequency increased the storage modulus, loss modulus and the damping factor as frequency is a function of time. Finally, the irregular, non-semi-circular, Cole-Cole plot confirms the heterogeneity of the self-healing GFRP composite.

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