

A COMPREHENSIVE STUDY ON IMPACT AND DYNAMIC MECHANICAL PROPERTIES OF SILICON CARBIDE (SiC) FILLED GLASS FABRIC REINFORCED POLYESTER (G-P) NANOCOMPOSITES

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ABSTRACT

Glass fiber reinforced polyester (G-P) composites are synthesised with an addition of Silicon carbide (SiC) particles by varying weight percentage (0, 2.5, and 5 %) using hand layup technique followed by compression moulding. The prepared specimens were cut according to ASTM standard to determine hardness, and impact strength. The fabricated specimens were characterized by dynamic mechanical analysis (DMA) technique. Density, hardness, and impact strength was found increases with increase in the filler loading. 5 wt% of SiC in to G-P [5 G-P] composites improved the impact strength by 16.27 % compared to that of unfilled G-P composites. More over the dynamic mechanical thermal analysis was carried out to study the Visco elastic behavior of unfilled and SiC filled G-P nanocomposites with varying temperature and frequency.

Keywords: Silicon carbide (SiC), G-P, nanocomposites, DMA, Transition glass temperature (T_g), $\tan\delta$, Storage modulus (E').

INTRODUCTION

Polymer matrix composites reinforced with fibers in different form to increase its mechanical, thermal and tribological properties are referred to as fiber reinforced polymer [FRP] composites. Polyester is most widely used family of thermoset. The success of polyester is mainly based on the low cost, versatile in curing process and mechanical property can be cured by modifying the monomer of the polyester chain. This encourages the use of polyester for various applications. Generally carbon, glass, and Kevlar fibers are reinforced to the polymer matrix due to which it has found in many application like, aerospace, automobile, bridges, transport line, space vehicles etc. All though strength of the glass fiber lower than that of the carbon fiber but it is stiff and less brittle and raw material is less expensive. Also weight to strength ratio of the glass fiber is better than metals. Glass fiber reinforced polymer [GFRPs] are used widely due to their light weight, strong, excellent anti-corrosion property, less density, higher impact and greater tensile strength]. FRPs property can be further improved by incorporating the organic and inorganic fillers. Many researchers proved the significant improvement in mechanical, tribological and thermal property by the addition of nano and micro fillers. Silicon Carbide (SiC) of the ceramic family is a promising candidate because of its many excellent properties, including strength retention, oxidation resistance at higher temperature and thermal shock resistance. In most of the application FRP may be subjected to various types of dynamic stresses hence the studies on their viscoelastic property are of being a greater importance. Dynamic properties of polymeric materials are of considerably important whenever they are determined over a wide range of frequency and temperature due to which influencing parameter like phase transformation on the mechanical properties can be discussed based on this study they are directly relevance to the application related to the vibration or dissipation of vibration energy in engineering applications.

Indra Reddy et al. studied the DMA property of hemp fiber reinforced polymer matrix composites. They concluded that dynamic modulus decrease with increase of glass fiber below the T_g and has positive effect above Glass Transition Temperature [T_g]. Also fiber addition lowers the $\tan\delta$ peak height which improves the matrix fiber adhesion. T_g is shifted to the positive on the addition of fiber. Suresha B et al. showed that addition of nano OMMT into C-E composites decreases the storage modulus than that of unfilled epoxy nano composites. They also concluded that T_g decreases with the addition of nanoclay into C-E composites. Aleksandar Grujie et al experienced that value of storage modulus amplifies in glass as well as in the rubbery state a concentration of filler in composites rises. Martinez-hernandez et al. have studied dynamic, mechanical and thermal analysis of polymer composites reinforced with keratin bio-fiber from chicken feather composites. Incorporation of 1% & 2% weight of bio-fiber showed higher modulus than non reinforced polymer at 35 degree & (E') elastic behaviour at higher Temperature is better for all composite addition of bio keratin fiber. T_g increased with addition of keratin bio-fiber. Duraibabu et al. reported on the thermal, dynamic properties of functionalized nanoalumina reinforced sulphone ether linked tetra glycidyl epoxy nanocomposites. They concluded optimized 3% of (F-nAl) reinforced produced significant improvement on thermal & mechanical stability compound to that of conventional & di-functional epoxy material. Suresh kumar et al. investigated on the mechanical thermal & dynamic properties of untreated (raw) & treated coconut sheath fiber reinforced epoxy compound (TCSE). Tensile, Flexural and impact strength of TCSE compound improved by 21.19%, 18.5%, 24.9% reinforced compound to find that of UTCSE composites.

DMA result shows that storage modulus (E') increased & damping property ($\tan\delta$) was decreased for (TCSE). Improvement in storage modulus & $\tan\delta$ of composite was observed as an effect of alumina platelet reinforcement epoxy as studied by Dharmendra kumar shukla et al. Heitor luiz Orngli et al. reported that increase in the fiber loading improves the storage modulus of sisal /glass fiber reinforced polyester composites. Glass transition temperature [T_g] and loss modulus was found shifting towards the higher temperature following the incorporation of more glass fiber into the composites. Hemanth et al studied the dynamic mechanical analysis and three body abrasive wear behavior of thermoplastic copolyester elastomer composites. They showed that storage and loss moduli of TCE+PTFE composites increased with addition of fiber reinforcement and inclusion of ceramic filler in to the short fiber reinforced TCE+PTFE composites. However lower glass transition T_g registered for TCE+PTFE composites.

From the literature review, mere number of articles is published on the effect of the SiC (nano form) on the polyester composites. Hence main objective of the current investigation is to investigate the influence of SiC particles on the physico-mechanical and dynamic mechanical properties of the glass fiber reinforced polyester (G-P) nanocomposites.

EXPERIMENTAL DETAILS

Materials: Unsaturated Isophthalic polyester (VBR-4503) Resin matrix material, Methyl ethyl ketone peroxides (MEKP) catalyst and Cobalt Naphthenate accelerator were supplied by M/s. Vasavibala Resins Pvt Ltd., Chennai, India. Bi directional E-Glass fabric consist ($\pm 45^\circ$) fiber orientation was used as a primary reinforcing having an aerial density of 380 gm^{-2} supplied by Devold-Norway. Silicon Carbide powder of an average particle size of 52 nm and density 2.65 g/cc used as secondary filler received from M/s. carborundum universal limited Trissur, Kerala, India.

Method of Fabrication: Pre-calculated amount of polyester resin is mixed with 1.5 % MEKP catalyst as prescribed by vendors. SiC powder was dried in a muffle furnace at 150°C for about 1 hr before mixing it with the resin. Precisely weighed SiC powder thoroughly mixed in a resin using a Magnetic stirrer maintained at 1000 rpm for 1 hr to avoid agglomeration followed by 1.5 % Cobalt Naphthenate accelerator added to initiate the curing prior to reinforcement. Polyester/Glass fabric composites containing, 2.5, 5 wt% SiC were prepared. Resin mixture impregnated E-glass fabric layer $220 \times 220 \text{ mm}$ were laid down on the surface of the mold one above the other until desired thickness is achieved. Silicon hard roller and brush facilitates degassing and ensures the uniform distribution of the resin. Each fabricated laminates were cured under pressure of 35 bar using hydraulic press for 24 hrs. Further laminates are removed from the mold and cured for 48 hr at room temperature before use of the composites. All laminates for the mechanical tests were prepared from the eight layer of the E-glass fabric except the hardness test for which 16 layers of fabric were used. All laminates of the hybrid composites were processed at a weight fraction of 60% (± 1.5). Thickness of Laminates maintained uniform thickness of 3.2 mm using a spacer. Laminates for hardness test were made to 6 mm thickness. Table.I. Shows detail Composition of the fabricated Composites.

Table.1.Composition of Unfilled and SiC Filled G-P nanocomposites.

Composite Designation	Quantity of Constituents (wt %)		
	E-Glass Fabric	Polyester matrix	SiC filler
G-P	60	40	0
2.5 G-P	60	37.5	2.5
5 G-P	60	35	5

Experimental Methods: Mechanical Properties Unfilled and SiC Filled G-P Hybrid nanocomposites which were obtained during testing were recorded in Table. II.

Table.2.Physico-MEchanical and DMA properties of Unfilled and SiC Filled G-P nanocomposites.

Composite Designation	Density (g/cc)	Rockwell Hardness (m-scale) (HRm)	Impact strength(J/m)	Tan δ	Glass Transition Temperature (T_g) [$^\circ \text{C}$]
G-P	1.82	89	720	0.24	112
2.5 G-P	1.88	94	810	0.22	108
5 G-P	1.94	102	860	0.21	100

Particle Analyzer using DLS technique: The particle size (diameter) of SiC has been determined using Microtek (Model-nanotrack wave) using Differential light scattering technique (D.L.S.).

In typical DLS experiment particles suspended in the methanol solution are irradiated with monochromatic laser light and fluctuations in the intensity of diffracted light are measured as a function of time. Autocorrelator was

used to receive the intensified data to determine the size of the particles. It is a common tendency to of the immersed particle to collide with an adjacent particle while they are in motion. This type of motion is called Brownian motion and it is vital for DLS analysis because it allow the Stoke-Einstein equation (Eqn. No. 1) to relate the velocity of a particle in solution to its hydrodynamic radius.

$$D = \frac{kT6\pi\eta}{a} \quad (1)$$

In the Stokes-Einstein equation shown (Eqn. 1), D is the diffusion velocity of the particle, k is the Boltzmann constant, T is the temperature, η is the viscosity of the solution and a is the hydrodynamic radius of the particle. The diffusion velocity (D) in the Stokes-Einstein relation is inversely proportional to the radius of the particle (a) and this equation is valid only for the system undergoing Brownian motion. From the earlier experience by researchers it is learnt that small particles should diffuse faster than large ones. This is a key concept in DLS analysis.

Scanning electron microscope and Energy dispersion X-ray analysis (EDX): Scanning electron microscope (SEM) was carried out on a Hitachi model S3400N to analyze the morphology and shape of SiC particles. Semi conductor SiC surfaces were sputtered with gold in a sputtering unit so as to make fully conductive. The images were taken at different magnification and suitable accelerating voltage for the best possible resolution, using secondary electron imaging. Energy dispersion X-ray analysis (EDX) measurement was carried out on oxford Instrument to determine the elements percentage in the SiC particles.

Density: Density of composites was measured using NETTLER-M tester by displacement method, according to ASTM D792, using electronic balance with an accuracy of $\pm 1 \times 10^{-4}$ g/cc.

Hardness: Hardness of samples with specific dimension $25.5 \times 25 \times 6$ mm was determined using SAROJ hardness tester following ASTM D785. During the test Carbide ball indenter with a spherical base of 1 mm diameter was penetrated in to the material under the applied load of 150 kg. Rockwell hardness was determined at different places for each composition. The average value will yield Rockwell hardness m-scale [HRm] for the specific composites.

Impact Strength: Impact Strength was determined using a CEAST-7J pendulum Izod impact testing machine. It is performed in accordance with ASTM D256. The dimension of the samples must be $64 \times 12.7 \times 6$ mm, having a 45° V-notch for a depth of 2.5 mm as shown in the Fig. 1.

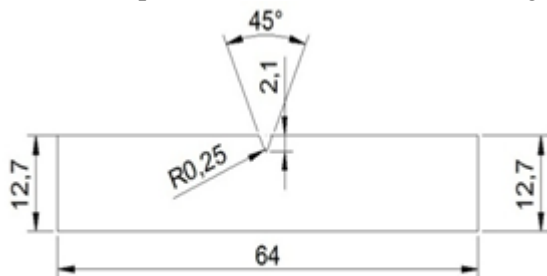


Fig. 1. ASTM D 256 dimensions of the Izod impact test specimen.

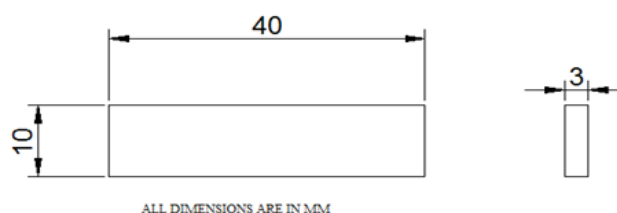


Fig. 2. DMA geometry of unfilled and SiC filled G-P composites.

Dynamic Mechanical analysis (DMA): Dynamic Mechanical analysis (DMA) some time also referred as Dynamic Mechanical Thermal Analysis (DMTA) is a technique used to study the viscoelastic behaviour as function of temperature of the polymer and its based composites. It is performed in accordance with ASTM D 5418[11]. In this present investigation Dynamic Mechanical Analyser (SII-DMS 6100) was used at a varying frequency of 0.5, 1.0, 2.0, 5.0, & 10.0 Hz with $2^\circ\text{C}/\text{min}$ heating rate, with amplitude of $153\mu\text{m}$ using dual cantilever beam technique. The samples were subjected to wide range of temperature from 32°C to 190°C . The sample size used for DMA is $(40 \times 10 \times 3)$ mm as shown in the Fig. 2.

RESULTS AND DISCUSSION

Characterization of SiC Particle: The particle size distribution of SiC is as shown in the Fig. 3. From figure the mean particle size was found to be 53 nm. The scanning electron micrographs of SiC at different magnification are shown in the fig. 4a and b.

Fig. 5a shows the EDX spectrum of the SiC particles. It is confirmed the presence of the Si and C elements. Computer generated quantitative graph of SiC particle is depicted in the Fig. 5b. It is evident from this plot that, contents Si and C are 65 % and 35 % respectively in the SiC powder.

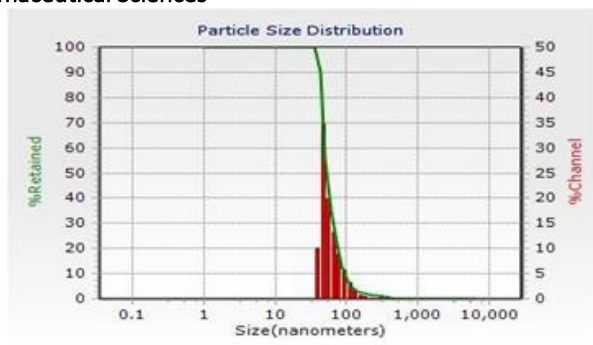


Fig. 3. Particle size distribution of silicon carbide (SiC).

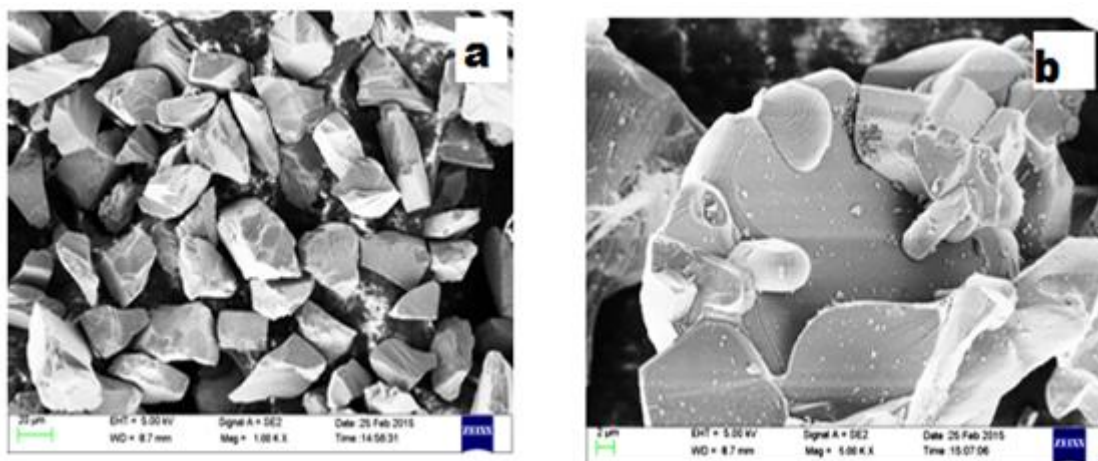


Fig. 4. SEM pictures of SiC (a) at 1000× magnification (b) at 5000× magnification.

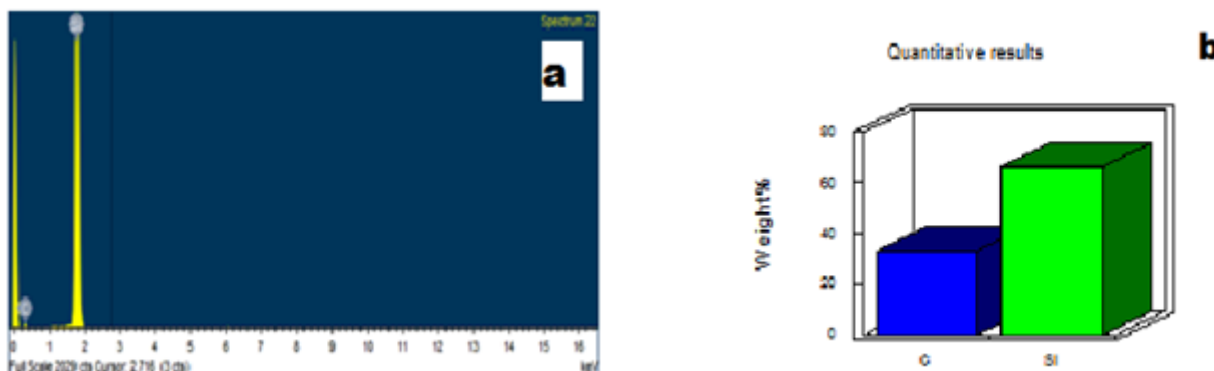


Fig. 5. (a) EDAX spectrum for SiC nano particle (b) weight percentage of the elements present in the SiC.

3.2. Density

Density of the fabricated composites increases with increase in the filler loading as fig. 6. This is mainly because of the inclusion of nano SiC particulates in to polyester matrix.

Hardness

Rockwell Hardness number (HRm) for different composition of G-P composites is shown in the Fig. 7, that hardness of the composite materials increases with increase in SiC filler content. For sample 5 G-P composite hardness found to be 104 HRm compared to 94 HRm of 2.5 G-P, which is increased up to 10%. The resistance to deformation or penetration or increase in the hardness can be ascribed to the fact that when the compression stress is applied on the composites, matrix, fiber and filler are subjected to pressure. This pressure facilitates the primary and secondary reinforcements to contact significantly. Therefore interface will transfer load more effectively though interfacial bond may be weak. Other factors also influence the hardness factor. The increase in adhesion between matrix polyester and reinforcement and reduced in the porosity due to addition of SiC content. Increase in filler content in the fiber reinforced polymer composites decreases inter particle distance, which results in increased hardness. This similar kind of observation is reported by Devendra et al. and Antunes et al..

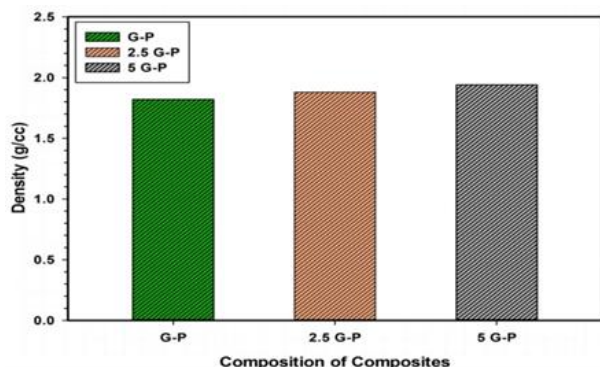


Fig. 6. Density comparison for different composition of the composites.

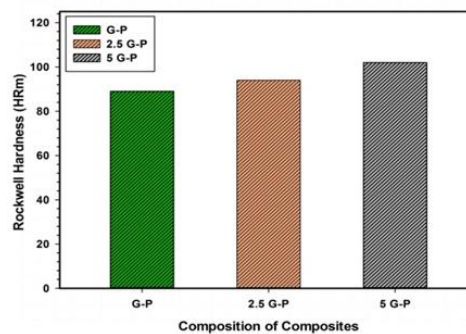


Fig. 7. Rockwell hardness comparison for different composition of the composites.

Impact Strength: Impact strength behaviour is very much influenced by the types of weight percentage of filler and fiber reinforcement. Toughness behaviour of the composite materials can be easily studied under the impact test. Major factors which affect the impact strength of the composites are interfacial adhesion between matrix and filler, matrix fracture and fiber pull out.

Impact strength of the unfilled and SiC filled G-P nanocomposites is presented in the Fig. 8. Impact strength increases with increase in the filler loading. Amongst all, 5 G-P nanocomposites exhibited higher impact strength of 860 J/m which results in 16.27% improvement in impact strength. This indicates that property of polyester matrix changes from brittle to tough. Moreover toughness increases the stiffness too. More amount of energy is absorbed by the material to overcome the resistance to crack propagation. Thus SiC based G-P nano composites have an ability to absorb more energy and has greater fracture strength as compared to unfilled G-P nanocomposites and hence can prevent propagation.

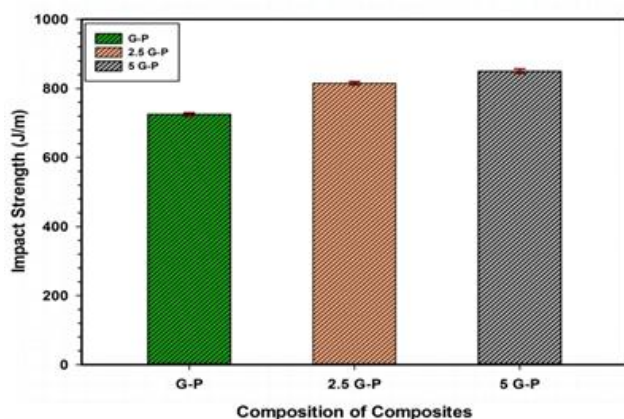


Fig. 8. Impact strength comparison for different composition of the composites.

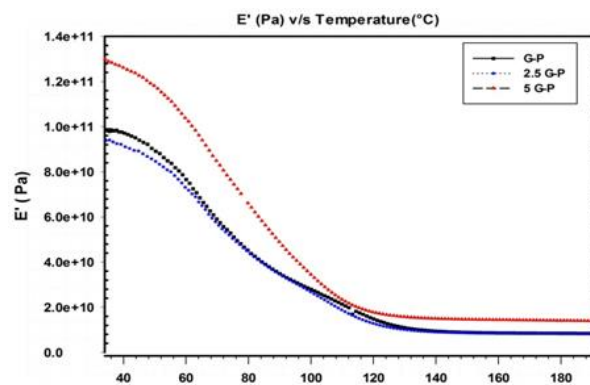


Fig. 9. Storage modulus (E') curve of unfilled and SiC filled G-P nanocomposites as a function of temperature.

Dynamic Mechanical Analysis (DMA)

Storage Modulus (E'): It is defined as the stress in phase with strain in a sinusoidal shearing deformation divided by the stress. Load bearing capacity of the composites can be easily analysed by storage modulus (E') parameter. The variation of storage modulus with the temperature for the unfilled and SiC filled G-P nanocomposites operated at the 1 Hz frequency is shown in the Fig.9.

The 5 G-P hybrid nanocomposites has exhibited higher storage modulus compared to that of the unfilled and 2 G-P nanocomposites. This may be due to the uniform distribution of the nano SiC particles which offer good adhesion between the matrix and primary reinforcement, results in the greater degree of stress transfer at the interface [30]. Storage modulus decreases with increases in the temperature. Decline of the storage modulus curve can be seen in the glass transition temperature region. This is attributed to the increase in the molecular ability of the polymer chain. Also any water molecule adhering to the fiber will get evaporated makes the fiber stiffer which ultimately decreases the storage modulus of the composites at higher temperature.

Damping Parameter ($\tan\delta$): The ratio of loss modulus to the storage modulus is measured as the mechanical loss factor ($\tan\delta$) or simply damping factor. Glass transition (T_g) can be determined from the peak position of the $\tan\delta$

curve due to the transformation of phase between elastic phase and viscous phase in polymeric structure. It is mostly influenced by the addition of fiber and fillers

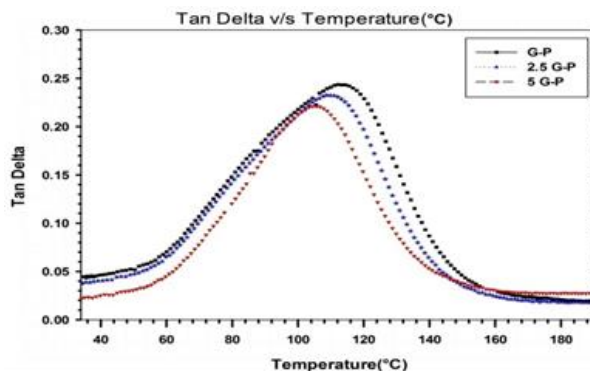


Fig. 10. Damping factor ($Tan\delta$) curve of unfilled and SiC filled G-P nanocomposites as a function of temperature.

The variation of $Tan\delta$ with the temperature for the unfilled and SiC filled G-P nanocomposites at 1 Hz frequency is shown in the Fig. 10. It can be noted from the plot that T_g of SiC/G-P nanocomposites was considerably lower than that of unfilled G-P composites. This can be attributed to the presence of the SiC particles which acts as plasticizers. Further from the Figure it is evident that $Tan\delta$ curve is fairly stable lower temperature. As the temperature increases pronounce peak can be seen which correspond to the high damping property due to the initiation in the segment of the polymer chain. Incorporation of fiber and filler offers resistance to the mobility of the polymer chain, thus raised the storage modulus, and increases the Visco elastic lag between the stress and strain. Hence $Tan\delta$ value increases in the composites.

From the Fig. 10, that $Tan\delta$ value decreases with increase of nano SiC particles in to G-P composites. The 5 G-P hybrid nano composite showed lower value of 0.24 at the glass transition temperature (T_g) 112°C. The lower the value of $Tan\delta$ indicates higher interfacial adhesion between fiber, filler and matrix, good load bearing capacity, and improves stress transfer.

SUMMARY

The synthesis and characterization of unfilled and SiC filled glass fiber reinforced polyester nanocomposites have finished successfully. DLS technique confirms that the size of the SiC particles used in the present investigation is 53 nm. Hardness, density and impact strength was found increases with increases in the SiC filler loading. 5 G-P nanocomposites showed 15% and 16% in the hardness and impact strength respectively. DMA results indicated that storage modulus increases with increase in filler loading in to G-P composites. As temperature increases storage modulus decreases, this trend was found in all polyester composites. 5 G-P nanocomposites have registered the higher value of storage modulus amongst all. Glass transition temperature (T_g) and Damping factor ($Tan\delta$) decreases with increases in the filler loading.

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