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Preparation and Characterization of GFRPC Material

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Abstract

This paper aims to present the mechanical characterization of Glass Fiber Reinforced Polymer Composites (GFRPC) under different load conditions. The dynamic mechanical properties of GFRPC are estimated experimentally. Different dies are designed for preparing and testing the GFRPC by multi directional loading. Samples are prepared at different compositions and reinforced with glass fiber at various mass fractions and plies by conventional hand lay-up process. The specimens are post cured for active bonding between reinforcement and matrix material, so that thermo-mechanical properties are enhanced to exercise for different applications. The glass fibers are added proportional to load bearing and optimized the reinforcement by weight percentage for particular grade applications. The prepared composites are characterized by Differential Scanning Calorimeter (DSC) to identify the glass transition temperature and nature of the interface between the phases, morphology of the prepared composites. Dynamic Mechanical Analysis (DMA) to determine the intrinsic properties as well as dynamic mechanical properties of GFRPC by three point flexural method. Slow Strain Rate Tensile Testing (SSRTT) exposes the stress, elongation and time at ultimate load and at the point of fracture. Here the test specimen is stretched monotonically in axial tension at a slow rate until failure. The SSRTTM testing is that it produces a result in a reasonably short time, within 6-12 hours in most cases, depending on strain rate to evaluation the effect of metallurgical variables such as alloy composition, heat treatment and processing and/or environmental parameters in a relatively short period testing.

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*Keywords:*Glass Fiber; Matrix; DSC; DMA; SSRTT;

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1. Introduction

Composite material is a structural material that consists of two or more combined constituents that are combined at a macroscopic level. Generally, a composite material is composed of reinforcement in any form such as fibers, particulates, flakes, and/or fillers which are embedded in a matrix material (polymers, metals, or ceramics). The matrix holds the reinforcement to form the desired shape while the reinforcement improves the overall mechanical properties of the matrix. When designed and prepared properly, the new combined composite material exhibits better strength than would each individual material. Composites are used in aircraft structures, electronic packaging, medical equipment, and space vehicles to home building. Polybenzoxazines are exhibit numerous exclusive properties, such as near-zero volume changes upon polymerization with high mechanical integrity, low water adsorption at room temperature, surprisingly high glass transition temperatures (Tg), rapid development of physical and mechanical properties during polymerization processes very high char yields, and low surface energies [1-4].The resin properties can be extracted from degree-of-cure, the heat flow during exotherm, the glass transition temperature and the melting and degradation temperatures [5-7]. Cure monitoring of liquid resins with a DMA is achieved using either an inert impregnation made of glass fibers [8, 9]. Since the observation of broad calorimetric glass transitions [10, 11] and the failure of time - temperature superposition in some miscible polymer blends, it has been appreciated that miscible blend dynamics are often complex relative to neat homo polymer [10-13]. Related effects are present in miscible polymer/plasticizer mixtures [14]. This is the arena of lightweight materials, by hybriding the properties of composite materials useful active properties are developed. Slow strain rate is significant mechanical behaviour, which is important property in performing CAE design. The vehicle body materials are tested at quasi static strain rate to 500 S^{-1} [15]. Some researchers are reported the dynamic mechanical properties of material in terms of Hopkinson bar [16]. Research groups and automobile manufacturers worked together to establish test standards to obtain tensile properties of materials covering the strain rate from 10^{-3} s^{-1} to 10^{3} s^{-1} [17, 18].

Nomenclature

GFRPC	Glass Fiber Reinforced Polymer Composite
DSC	Differential Scanning Calorimeter
DMA	Dynamic Mechanical Analysis
SSRT	Slow Strain Rate Tensile Testing
T_g	Glass Transition Temperature
E’	Storage Modulus
E”	Loss Modulus

1.1. Benefits of Composites

Advanced technologies in manufacturing and burning desires of updated design engineers demands the hybrid properties of individual materials. These GFRPCs are one of the promising material for light weight applications with good mechanical stiffness. Upgrading properties of GFRPCs are as follows:

- GFRPCs are economical than the conventional metals with reasonable mechanical properties and least production time.
- Its weight to strength ratio is more than many metallic components.
- Having high dimensional accuracy, good surface properties, and environmental friendliness with least finishing cost.
- The addition of the reinforcement to the polymer matrix increases the creep resistance for properly designed FRP part.
- Possess good thermal and electrical stable properties.
- Exhibits non-reactive nature with organic and inorganic substances.

2. Preparation of Glass Fiber Reinforced Polymer Composites (GFRPC)

2.1. Preparation of die

The Dies are prepared according to the requirement of the samples. Some of the dies with M.S material are fabricated and this can be with stand a load up to 80 kg. the other dies are fabricated with ply wood and teak wood with varying dimensions. The ply wood and teak wood are chosen because of their low cost and ease of fabrication. These dies are used where the load applied is nominal in nature.

2.2. Preparation of composite material

This part of the work summarizes and priorities the key environmental issues facing each of the eight common manufacturing process reviewed and given below. It is recognized that the choice of manufacturing process will primarily be influenced by economic and design factors, this work consider preparation and characterization of "GFRP" composite material.

For preparing GFRPC, major ingredientsare E-Glass Fiber mat, Epoxy resin (Company code: L/556) with hardener, Die.

The Hand Lay-up process is being adopted for preparing GFRPC as shown in Fig.1. It is a production technique suitable for prototypes and low volume production of fiber composite parts. The composite part will have a nice smooth surface on one side and a very rough one on the other. This is the simplest method of producing a fiber-reinforced part. To produce a part with this process by using fiber glass and a polyester. Initially E-glass fiber mat is cut to required dimensions and placed on a clean and flat surfaced table. Now the epoxy resin (the resin and fibers are supplied by is taken and mixed with hardener in the required ratio in order to get good strength (for 100% of resin 2% of hardener is taken). Now the mixture is thoroughly applied to the fiber mat with a soft, clean brush. After applying the resin mixture, it is rolled over with a smooth roller in order to eliminate the air bubbles. Now it is properly folded in to one half and again the resin mixture is applied. The process is continued until required size and thickness is attained and allowed to dry for some time.

Fig.1. Hand Lay-up process of composite preparation

The prepared sample is kept in the die and sufficient load is applied for a period of a minimum of 24 hours for getting proper bond between the fiber and matrix. More layers can be added and, after drying, the composite part can be removed from the mould. The prepared samples are post cured in special oven suited for the epoxy composites. This post curing had strong impact on the thermal and mechanical behavior of thermosetting polymers. The main advantages and disadvantages of Hand Lay-up process are easy to control fibers orientation. Furthermore, the process is very flexible as it can produce from very small, up to very large part of different kinds of geometry. The cycle time per part is very long, and only small series can be produced.

3. Experimental Methods

3.1. DSC Testing

Differential Scanning Calorimeter (DSC) monitors heat effects associated with phase transitions and chemical reactions as a function of temperature. In a DSC the difference in heat flow to the sample and a reference at the same temperature, is recorded as a function of temperature. The reference is and inert material such as alumina, or just an empty aluminum pan. The temperature of both the sample and reference are increased at a constant rate. Since the DSC is at constant pressure, heat flow is equivalent enthalpy changes:

$$\left(\frac{dq}{dt}\right)_p = \left(\frac{dH}{dt}\right) (1)$$

Here dH/dt is the heat flow measured in mcal. sec^{-1}. The heat flow difference between the sample and the reference is given by:

$$\Delta \left(\frac{dH}{dt}\right) = \left(\frac{dH}{dt}\right)_{sample} - \left(\frac{dh}{dt}\right)_{reference} \quad (2)$$

And can be either positive or negative.in an endothermic process, such as most phase transitions, heat is absorbed and, therefore, heat flow to the sample is higher than that to the reference. Hence $\Delta dH/dt$ is positive. Other endothermic processes include helix-coil transitions in DNA, protein denaturation, reduction reactions, and some decomposition reactions. In an exothermic process, such as crystallization, some cross-linking processes, oxidation reactions, and some decomposition reactions, the opposite is true and $\Delta dH/dt$ is negative. The DSC testing was performed in both isothermal and dynamic heating modes. An argon gas sample purge was used for all DSC tests. The prepreg material is very sticky, and a hermetic stainless steel DSC pan with an O-ring seal was used for each sample. The average actual cure temperature during the cure time of each prepreg sample was within 2^oC of the set point temperature. For dynamic testing, three linear heating rates of 2, 5 and 10^oC/min were used over temperature range of 30-300^oC for also determining prepreg cure kinetics. The 10^oC/min heating rete was used following each partial cure for determining the T_g and residual heat of reaction after curing to sometime t.($H_{t,resid}$).

3.2. DMA Testing

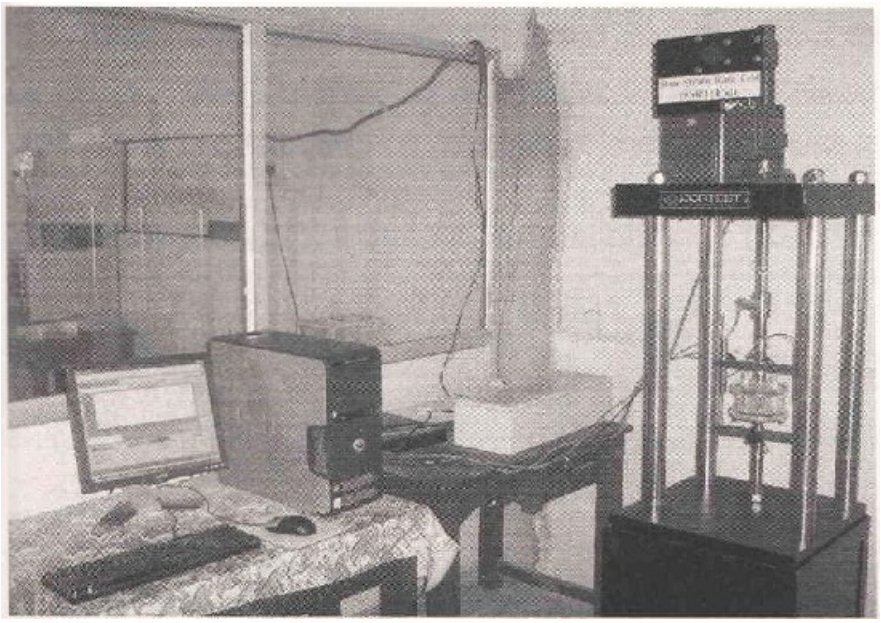
Dynamic Mechanical Analysis (DMA) is one of the most powerful tools to study the behaviour of plastic and polymer composite materials. The pyris Diamond DMA Viscoelasticity Measurement Module (temperature range:150^{o}C~600^{o}C) measures the creep recovery and dynamic viscoelasticity and stress relaxation using 3 point bending, tension, shear and compression methods to measure things such as polymeric materials simply and with high precision, as a function of temperature, frequency, or time. DMA testing was performed in the fixed frequency mode at oscillation amplitude of 0.2 mm, with inert gas sample purge used. Each cured sample was tested in the vertical DMA testing mode at fixed frequency measurements.

3.3. SSRT Testing

Slow Strain Rate Tensile Testing was used to test the prepared Glass Fiber Reinforced Polymer Composite Material to find out the Stress, % Elongation, Time taken to Failure, Slow-Strain-Rate testing is a dynamic load testing. It is an important method which involves relatively slow-strain-rate tensile testing of a specimen. Here the test specimen is stretched monotonically in axial tension at a slow rate until failure. This method is also known as constant extension-rate tensile testing. The benefit of SSRT testing is that it produces a result in a reasonably short time, within 6-12 hours in most cases, depending on strain rate. It also reduces incubation time to the onset of cracking in susceptible materials through the application of the dynamic plastic straining.The main benefit of the SSRT test is it allows the evaluation of the effect of metallurgical variables such as alloy composition, heat treatment and processing and/or environmental parameters in relatively short period of testing. Strain rates utilized

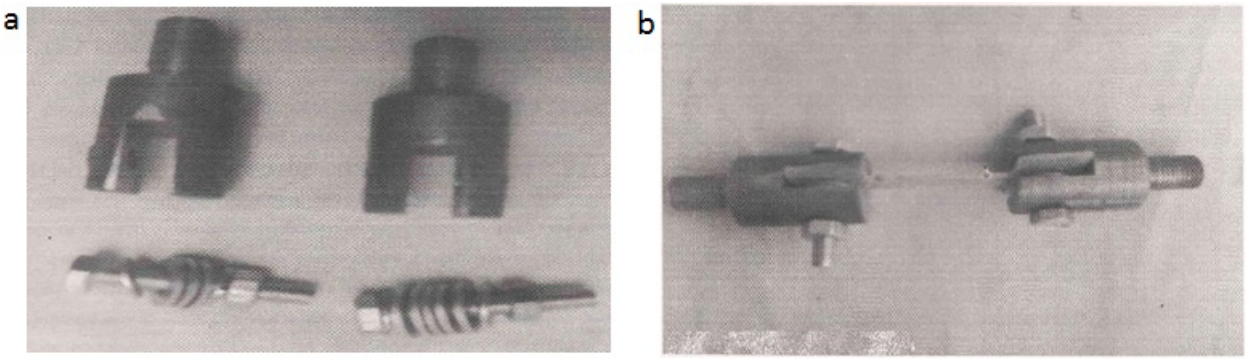
for SSRT testing are typically in the range of 1- $4x10^{-6}S^{-1}$. At a strain rate of 4 $x10^{-6}S^{-1}$, the testing speed is about 1% strain per hour and failure of most engineering materials will occur within a day or two. Constant-strain-rate apparatus requirements include sufficient stiffness to resist significant deformation under the loads necessary to fracture the test specimens, a system to provide reproducible and constant strain rates over the range of 10^{-4} to 10^{-8} S^{-1}, and a cellto contain the test solution. Auxiliary equipment is used to control environmental conditions and to record test data. The testing equipment can also be instrumented to record load-elongation curves.

Fig. 2. A photograph of slow strain rate test unit



A photograph of a SSRT test unit is given in Fig. 2. Curtest incorporated (USA) Company manufactured and supply the SSRT unit with cross head speed range 10^{-7} to 4.5x 10^{-4} mm/sec and maximum load capacity of 4536 Kgf. The arrangement of the sample setup for the Slow Strain Rate Tensile Testing is shown in Fig. 3 (a)and(b).

Fig. 3. (a) Grippers prepared for SSRT Machine; (b) Final setup of samples for SSRTMachine.

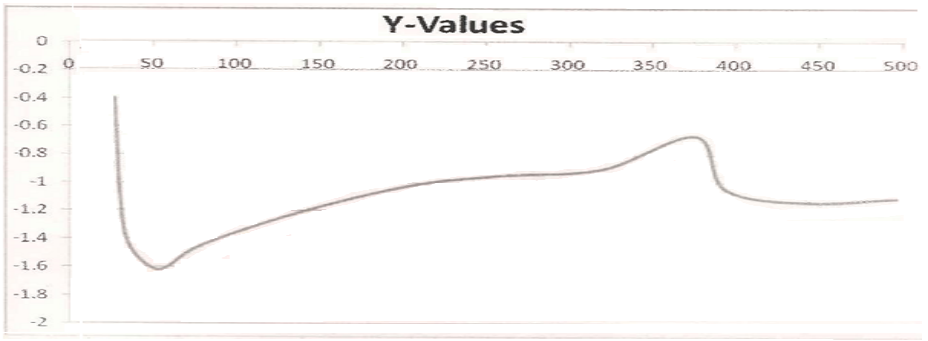


4. Results and Discussion

4.1. DSC Data

The output of the Dynamic Differential Scanning Calorimeter reveals many parameters by plotting the graphbetween heat flows versus temperature as shown in Fig. 4. 'As received' prepreg from graph at a heating rate of 20°c/min revealing one crystallization peak, one melting peak, one exothermic, one endothermic, one glass transition temperature.

Fig. 4. Heat flow (w/g) (Y-axis) V_s Temperature °C (X-axis)



4.2. DMA Data

The graphical figures obtained after conducting experiments on DMA machine and are shown in Fig. 5. It is clear from the graph that the storage modulus (E') of the samples is abruptly decreasing to very small value in the region of glass transition temperature of the sample. This is due to weakening of bonding between the cross linked molecules of the resin matrix of the composite. The loss modulus (E") of the sample is taking high values because there is a sudden decrease of modulus of the sample due to starting of glass transition temperature region in the sample. The tanδ also represents loss of angle of modulus of the sample which is also taking high values in the glass transition temperature region of the sample which has undergone for testing.

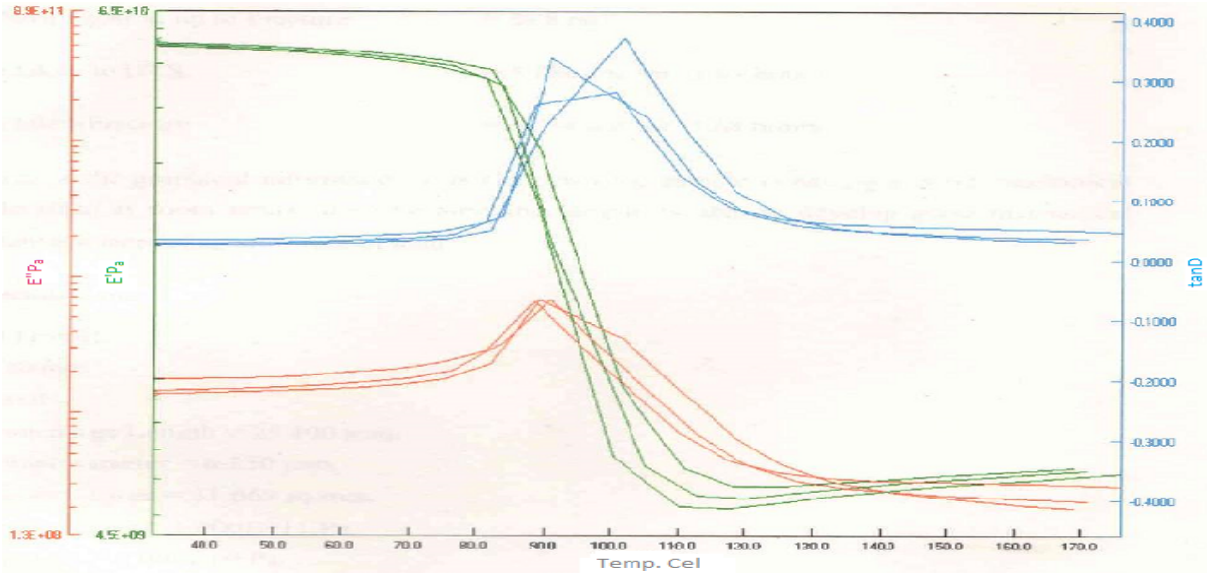


Fig. 5. Experimental Results for DMA

4.3. SSRT Data

Table 1.Slow Strain Rate of GFRPC at different loads.

Time (Sec)	LVDT 1 (mm)	LVDT 2 (mm)	Extension (mm)	Load (Kgf)
3383603310	0	0	0	41.82
3383603340	0.0083	-0.0022	0.003	42.86
3383603370	0.0339	-0.003	0.0154	46.05
3383603400	0.0565	-0.0037	0.0264	48.17
3383603430	0.0745	-0.0007	0.0369	50.45
3383603460	0.0828	0.0097	0.0463	52.04
3383603490	0.0903	0.0239	0.0571	53.62
3383603520	0.0986	0.0373	0.0681	55.37
3383603550	0.1069	0.0493	0.0781	56.73
3383603580	0.1182	0.062	0.0901	58.39
3383603610	0.128	0.0747	0.1013	59.98
3383608890	2.3855	2.4382	2.4119	145.55
3383608930	2.3991	2.4516	2.4254	145.62
3383608960	2.4156	2.4628	2.4392	145.72
3383608980	2.4299	2.4748	2.4524	145.78
3383609020	2.4442	2.486	2.4651	146.08
3383609050	2.4563	2.5002	2.4782	146.31
3383609090	2.4698	2.5144	2.4921	146.39
3383609120	2.4849	2.5263	2.5056	146.69
3383609150	2.506	2.5353	2.5206	146.82
3383609190	2.524	2.545	2.5345	147.28
3383609230	2.5398	2.5562	2.548	147.22
3383609260	2.5549	2.5689	2.5619	147.08
3383609290	2.5692	2.5831	2.5762	146.61
3383609330	2.585	2.5943	2.5897	146.38
3383609350	2.5986	2.6078	2.6033	145.49
3383609394	2.6136	2.6182	2.6159	145.26

The data obtained after conducting Slow Strain Rate Tensile Testing results of GFRPC are given in Table. 1 and resulting graphical figure is shown in Fig. 6. By observing of the graph and data systematically, it is clear that the stress developed in the sample is increasing almost proportionality with % of elongation of the sample.

The maximum stress at UTS = 181.62 N/mm^2
The fracture strength of the sample = 179.10 N/mm^2
The % of elongation up to UTS = %8.55
The % of elongation up to Fracture = % 8.88
Time taken up to UTS = 5,868 sec ‘or’ 1.63 hours
Time taken up to Fracture = 6,084 sec ‘or’ 1.69 hours

By analyzing the graphical information it is clear that the sample is having a good mechanical tensile strength at room temperature because the sample is able to develop good mechanical resistance with increasing slow rate of load.

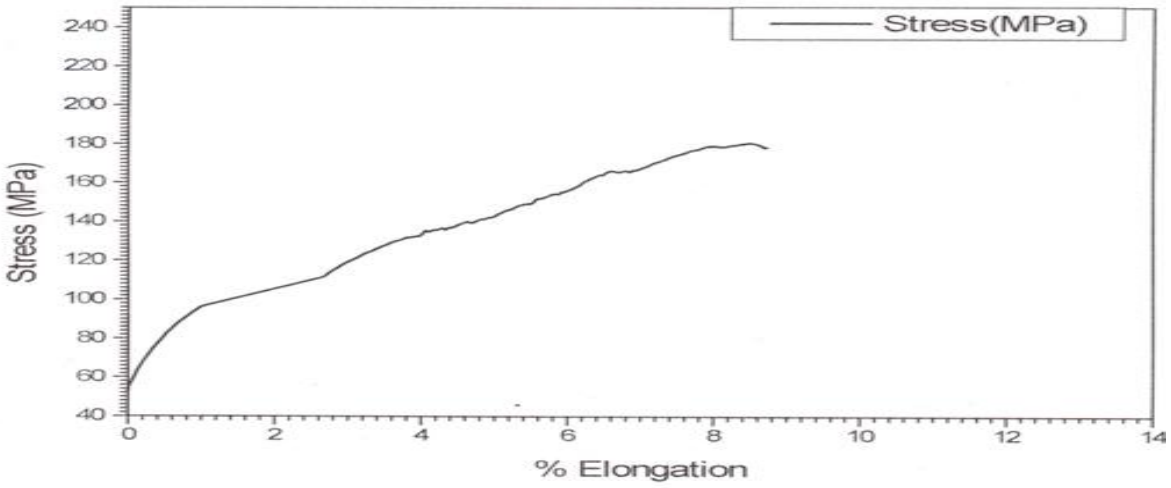


Fig.6. Slow Strain Rate Tensile Testing curve

5. Conclusions

By observing the figure of DMA testing, it is clear that the glass transition temperature is in the region starting from 80°C to 120°C. So the usage of the sample should be below 60°C to 65°C or else the sample will not be working satisfactorily for the intended use. The data from DSC graph figure indicates that the glass transition is near about 100°C which is coincing with DMA output results for storage modulus and loss modulus. So we have conformed DMA results to the data from DSC output results. The Slow Strain Rate Tensile Test data depicted in figure shows that the sample prepared is having good structural integrity because there is direct proportionality between stress and strain.

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