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Effect of Nanoclay on Dynamic Mechanical and Tensile creep behavior of Glass Fiber Reinforced Polyester

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Abstract: *Fiber reinforced composite material was prepared by reinforcing nanoclay modified isophthalic polyester resin with glass fiber mat. Polyester resin was modified with nanoclay cloisite15A. The Dynamical Mechanical behaviorwas studied for pure polyester modified with 0.5%, 1%, 1.5% and 2% nanoclay and reinforced with 5 layers of glass fiber mat. The tests were done at temperature levels from room temperature to 150 ^{o}C. The storage modulus decreases with temperature and improved performance noticed for the 1% nanoclay filled sample. The creep behavior of the material tested for pure polyester, purepolyester modified with 1% and 2% cloisite15A which reinforced with glass fiber mat. Creeping increases with elapse of time and decreases with the addition of nanoclay. The better performance noticed with 1% cloisite15A filled sample*
Key words: *nanoclay, storage modulus, creep compliance*

I. Introduction

Recently, polymer nanocomposites, have gained wide acceptability as an alternative to conventional polymer composites in many applications. Polymer systems are widely used because of their light weight, design flexibility, and processability.[1,2]Polymer materials exhibit time dependent behavior. The stress and strain induced when a load is applied are a function of time. The stress-strain-time relationship, or constitutive law, can be determined by loading a polymer specimen with constant stress (creep) or constant strain.

When a plastic material is subjected to a constant load, it deforms continuously. The initial strain is roughly predicted by its stress-strain modulus. The material will continue to deform slowly with time indefinitely or until rupture or yielding causes failure. The primary region is the early stage of loading when the creep rate decreases rapidly with time. Then it reaches a steady state which is called the secondary creep stage followed by a rapid increase (tertiary stage) and fracture. This phenomenon of deformation under load with time is called creep.

Creep resistance is an important property for polymeric materials. It is one of the principal properties of natural fibers reinforced polymer composites for many applications such as aerospace, biomedical and civil engineering [3].However, it is often impractical to test long-term creep behavior directly with experiment because of the extremely long time required. Thus, predicting the creep behavior of polymers using short-term testing has gained considerable attention. One of the most useful extrapolation techniques is time-temperature superposition (TTS). It can be used to predict long-term creep behavior of certain polymers by shifting the curves from tests at different temperatures horizontally along a logarithmic time axis to generate a single curve known as the master curve. Thus, long-term experiment can be replaced by shorter tests at higher temperatures. The shifting distance is called shift factor. The materials for which TTS holds are called thermorheologically simple materials and the rest are called thermorheologically complex materials. The influence of high temperature and long time has similar effect on the polymer material. With shifting the single creep curves (measured at different testing temperatures) together (to a selected reference temperature) a master curve can be created. This time-temperature superposition method (TTS) is able to predict the long-term properties of the material from short time creep tests at higher temperature [5-7]. The relation between temperature and the shift factor can generally be described by the Arrhenius Equation [8]. Long term creep estimations based on master curves and heuristic relations are used to provide information on the given small loads and help designers to a limited extent in dimensioning a given product to its whole life span knowing the forces and environment and in estimating the expected failure time of the part even though it does not provide information on failure deformation or life span.

There are two superposition principles, which are important in predicting creep behavior of plastic materials under various test conditions. The first of these is the *Boltzmann Superposition Principle*, which describes the response of a material to different loading histories. The second is the *Time Temperature Superposition Principle* or the WLF equation, which describes the equivalence of time and temperature. The Boltzmann superposition principle states that the response of a material to a given load is independent of the

response of the material to any load, which is already on the material. The deformation of a specimen is directly proportional to the applied stress, when all deformations are compared to equivalent times. It is only valid in linear viscoelastic region. For the case of creep, the total strain may be expressed by

$$\varepsilon(t) = D(t-\tau_1)\sigma_1 + D(t-\tau_2)(\sigma_2 - \sigma_1) + \ldots\ldots + D(t-\tau_i)(\sigma i - \sigma_{i-1})$$

$$\varepsilon(t) = \int_{-\alpha}^{t} D(t-\tau)\, d\sigma(t)$$

Where D*(t)=1/E(t)* is the compliance function, which is a characteristic of the polymer at a given temperature and initial stress.

The WLF equation is: $\log a_T = \dfrac{-C_1(T - T_{\text{ref}})}{C_2 + (T - T_{\text{ref}})}$

a_T horizontal (time) shift factor, C_1 and C_2 are constants, T_{ref} reference temperature(K)

T temperature at which test is performed

Arrhenius equation is $\log a_T = \dfrac{Ea}{R}\left(\dfrac{1}{T} - \dfrac{1}{T_{\text{ref}}}\right)$

Ea is the activation energy, R is the universal gas constant, T_{ref} is the reference temperature(K)

T is the temperature at which test is performed

Creep behavior of fiber reinforced composites depends strongly on stress, temperature, void content, and fiber loading [9-11]. It was concluded, that creep resistance decreases if temperature or stress rises. Several investigations on the influence of adhesion and density of the composites on the creep behavior already done. With increasing consolidation (lower void content) the resistance to creep increases. [12-14]

II. Literature Review

Challa and Progelhof [14] investigated the effect of temperature on the creep characteristics of polycarbonate and developed a relationship based on Arrhenius theory to develop creep master curves. Kishnaswamy [15] conducted creep rupture testing on high density polyethylene pipes at various hoop stress levels and temperatures and reported the dependency of density and crystallinity towards failure. Greco et al. [16] investigated the flexural creep behavior for compression molded glass fiber reinforced polypropylene at various applied stress level. He reported the effect of matrix crystallinity for the improvement in creep properties for glass fiber reinforced polypropylene. Acha et al. [17] studied the influence of interfacial adhesion in discontinuous jute fiber reinforced polypropylene. Relation between interfacial properties and creep deformation were investigated. Higher creep resistance was observed for composites with good interfacial bonding which was confirmed by the observation of the composite fractured surfaces. Findley and Khosla [18] conducted creep tests for unreinforced thermoplastics; polyethylene, polyvinyl chloride and polystyrene. Approximation was carried out for the linear viscoelastic region by power law and compared the creep performance by estimating the power law coefficient and power law exponent. Power law model was modified by Hadid et al. [19] estimated four parameters for describing the deformation occurring in the material and used stress–time superposition principle to predict long-term material creep behavior of injection molded fiber glass reinforced polyamide. Master curves were developed and a perfect superposition of the curves at various stress levels was visualized. Novak [20] used strain energy equivalence theory and developed a creep predictive model to predict the creep behavior of talc filled polypropylene. Banik et al. [21] reported the improvement in creep resistance due to unidirectional reinforcement for polypropylene-polypropylene composites. Liu et al. [22] used multi-Kelvin element theory and power law functions to predict creep compliance in polyethylene material and compared with the tensile creep experiments. Subramanyan et.al.[23] investigated the influence of reinforced fiber length on the creep performance of thermoplastic composite at various stress levels at room temperature condition. Discontinuous fiber reinforced polypropylene composites were injection molded and its short term flexural creep performance is investigated.

According to studies conducted by Nunenz et. al.[24] on kenaf fiber reinforced composite, it is observed that the creep strain and creep compliance decreased as the kenaf fiber content increased. This behavior was expected from the increased rigidity of the composites. Better creep resistance with increase of kenaf loading also reported by Yanjun [25]. There are three roles of the additives on creep resistance of the composites which can be proposed to explain the experimental observations. The first is the volume effect, where the additives reduced the relative volume of viscoelastic polymer matrix, which was prone to creep. The second is the bridging effect, where the additives sustained part of the stress by connecting to each other. The third is the blocking effect, where the additives interacted with the molecular chains of polymer matrix and blocked them from moving under stress.

Feiyi and Chun developed a creep model for stitched and unstitched woven carbon fiber polymer composite. According to them stitched woven fiber composite will take more time to creep than un stitched and found that the shear and the normal stresses have been significantly reduced as a result of stitching. According to them the enhanced creep resistance of stitched composites can be directly attributed to the reductions in the shear and normal stresses. This highlights that the main mechanism for the significant improvement in creep performance, by stitching, is the significant reduction in the interlaminar stresses.[26] It has been found that through-thickness stitching the creep resistance of composites can be improved, provided the stitches are aligned in the direction of loading. At a given time, the stress level required to induce the same amount creep strain in stitched composites was about twice that for unstitched composites. [27]

Investigation of the creep and dynamic mechanical behavior of natural fiber/epoxy composites using functionalized multiwalled carbon nanotube (MWCNT) modified matrix showed that the creep resistance of natural fiber reinforced composites was greatly improved by the addition of MWCNTs.[28] The comparison of the time-displacement data under constant indentation creep load indicates that the addition of MWCNTs results in a noticeable decrease in creep rate, particularly under the conditions of elevated temperature and high nano indentation creep load. This behavior might indicate a failure of the epoxy–MWCNTs interface at high load levels. However, increasing the temperature to a near glass transition did not impact the ability of the MWCNTs to reduce the creep response of the nanocomposites compared to neat epoxy samples.[29]

` Due to the viscoelastic nature of polymeric materials, the analysis of their long term behavior is essential. For a viscoelastic polymer, the modulus is known to be a function of time at a constant temperature. The modulus is also a function of temperature at a constant time. According to this time-temperature correspondence, long term behavior of a polymer may be measured by two different means. First, experiments for extended periods of time can be carried out at a given temperature, and the response measured directly. This technique becomes increasingly time consuming due to the long response times of many polymers. The second method takes advantage of the principles of time temperature correspondence wherein experiments are performed over a short time frame at a given temperature, and then repeated over the same time frame at another temperature. The two methods are equivalent according to the principles of time temperature super positioning. The structural applications of polymer matrix composites (PMC) demand lifetimes of 15, 25 and 50 years. However the mechanical properties of these composites have a time dependent nature, i.e. strength and stiffness are time-dependent due to the viscoelasticity of polymers.

Creep behavior of fiber epoxy composite studied by Lubna Ghalib[30] reported that, the effect the time on the strain of the epoxy, increase with increasing the time at constant temperature. In considering the strain induced in service it is required to take into account not only the stress, but the time for which it is applied. The strain decrease with increasing the volume percent of the fiber as the composite materials is stiffer and stronger than the polymer matrix and the stiffness increase with increasing the percent of the fiber. It has been shown in the literature that the polymers filled with exfoliated layered silicate platelets can give an increased creep resistance than unfilled polymers. The effect of moisture on creep behavior of polymer containing nanoparticles is still unclear and hardly published. Due to moisture absorption glass transition temperature *Tg* can be reduced below the operating temperature and this increases the creep compliance [31]. Studies on the creep behavior of epoxy/clay nano composite described the correlation between the three creep parameters – viscoelastic plus plastic deformation, viscoelastic relaxation and residual (plastic) on creep behavior. All the deformations increase with the increase of filler content till 6 %. Therefore it was confirmed that inclusion of clay nanoparticles to epoxy resin restricts the mobility of polymer chains in dry atmosphere and improves creep resistibility of the polymer, but absorbed moisture drastically plasticized the polymer and changed creep behavior leading to the increase of creep compliance with the increase of filler content. Particularly high deformations were observed for the highest filler content in atmosphere with highest humidity which can be explained by additional "sliding" of silicate nanoparticles within the layered stacks.[32]

The present study aims on the investigation of tensile creep behavior of fiber reinforced plastic from isophthalic polyester resin and the effect of adding nano clay in the resin.

III. Experimental

3.1 Raw Materials

Isophthalic polyester resin used as the matrix material. Cobalt naphtha ate as the accelerator and methyl ethyl ketone peroxide (MEKP) as the catalyst were used. The clay was made into dispersion with styrene and then the dispersion was added to the polyester resin for modification. Cloisite15A, quaternary ammonium modified montmorrillonite was used as the nano filler. 200 gsm glass fiber mat used for the reinforcement.

3.2 Methodology for specimen preparation

Firstly, the filler (Cloisite15A) was dried in an oven at a temperature of 80 °C for 24hrs to remove the moisture. Then pre calculated amount of clay and isophthalic polyester resin were mixed together in a beaker with mechanical stirrer for 30 minutes at ambient temperature conditions. Then the mixture was placed in a high intensity ultra sonicator for 20 minutes. External ice bath was used to prevent the heating of the mix during ultrasonication process. Once the process was completed, weighed quantity of catalyst, cobalt Naphthalene added and stirred well using a glass rod. Then the accelerator Methyl Ethyl Ketone Peroxide (MEKP) is added and stirred. Further the mix is used for the preparation of Glass fiber reinforced polyester nano composite through hand layup technique. A ceramic tile of smooth surface is placed over the table is applied with wax as mold releasing agent to facilitate the easy removal of the sample. Then a coat of the mix polyester and nano clay prepared is applied over the tile surface using a brush and a roller is applied to make the coating of uniform thickness. Then the first layer of glass fiber mat (200 gsm seven mil cloth) is placed over the coat of mix. Then the mix is again applied in thin layer by using the brush. The processes have been done for a five layers of glass fiber mat. The closed mould was kept under pressure for 24 hrs at room temperature. To ensure complete curing the blended nano composite samples were post cured at 70ºC for 1 hr. and the test specimens of required sizes were cut out from the sample sheet by water jet machining. The specimens have been prepared through same procedure for varying the weight of clay i.e. without clay (pure polyester- 0 % clay), 0.5 % clay, 1 % clay, 1.5 % clay and 2 % clay. A similar procedure had been adopted by Chakradar *et.al.* [33] and Mirmohseni et.al. [34].

3.3 Dynamic Mechanical Analysis (DMA)

The thermo mechanical behavior has been studied by using DMA. The experiment conducted in a DMA Q800 apparatus (TA Instruments, New Castle, USA) A very common method to measure the damping of polymers. It measures the modulus (stiffness) and damping (energy dissipation) properties of materials. The polymer sample is subjected to an oscillating stress and the resulting strain is recorded continuously. The ratio of dynamic stress to dynamic strain is the *complex modulus, G**, which can be resolved into the *storage modulus, G'*, and the *loss modulus, G''*. The storage modulus represents the ability of a material to store energy for every oscillation and it is related to the stiffness of the material. The loss modulus represents the heat dissipated by the material due to its molecular motions and this reflects the damping characteristics of the polymer. The specimen has been taken from nano clay modified isophthalic polyester reinforced with 200 gsm glass fiber (seven mil cloth). The polyester resin modified with 0.5%, 1.0%, 1.5% and 2.0 % Cloisite15A for different types of specimen. Pure polyester (0% filler) reinforced with 200 gsm seven mil cloths also used for analysis. The glass transition temperature of the specimen obtained from experiment conducted in the Tg run mode of DMA. Dual cantilever configuration used for the same. The experiment conducted for a frequency 1 Hz for the temperature range from room temperature to 136 ^{o}C.

3.4 Creep study

Tensile creep behavior of the material has been studied by using the DMA Q800 apparatus (TA Instruments, New Castle, USA). The specimen prepared in the size 25 mm lengths, 4 mm width and 2.5 mm thickness from the casted glass fiber mat composite. The experiment conducted for the samples with 0 wt%, 1wt% and 2 wt% Cloisite15A as filler. The samples prepared from the glass mat laminate used for DMA. The test was conducted for constant stress of 1MPa and 2MPa each separately at reference temperature 30 ^{o}C (approximated to room temperature). Creep performance is commonly represented by creep compliance $J(t) = \varepsilon(t)/\sigma$, where $\varepsilon(t)$ is creep strain and σ is applied stress.[35]

The dynamic mechanical performance of the of the material at reference temperature 30^{o}C at low frequency range varying from very low frequency to 3 Hz conducted. The specimen with the same specification that used for constant stress experiment has been used.

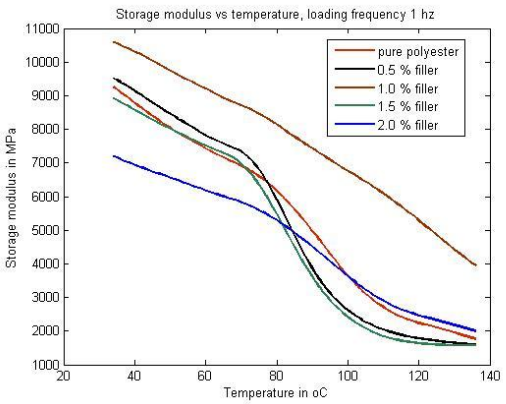
IV. Results And Discussion

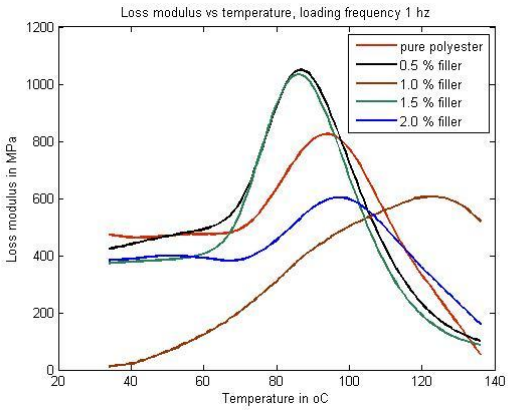
4.1 Dynamic Mechanical Analysis

The variation of storage modulus with temperature is as shown in Fig: 01(a). Percentage volume of filler showed a notable change in the storage modulus. The increase in storage modulus for the sample with 1% filler as compared to 0% and 0.5% filler is evident. Whereas the storage modulus value is low for 2% filled sample as compared to 1% filled sample. At lower percentage level of filler the stiffness of the material increased with increase of filler. Whereas at high percentage of filler the agglomeration of clay particles leads to non uniform mixing, will contribute to the decrease of stiffness. The decrease in stiffness will be the result of non uniform mixing of the filler as well as decrease in intermolecular bonding. The decrease in storage modulus with increase of temperature is due to the decrease in the stiffness from the softening of the matrix.

The effect of clay reinforcement on loss modulus and tan delta (Tan δ) of Polyester/Nanoclay nanocomposites can be seen from fig. 01 (b) and (c). The storage modulus decreases, for nanocomposites for nanoclay content above 1%. The average values of glass transition temperature, Tg for different wt. % of Cloisite15A is in the range 95 – 100 ^{o}C . There is a markedly high increment in the Tg value with 1% filler. Any how the addition of Cloisite15A did not significantly change the glass transition temperature of the FRP nano composite except that for 1% clay filled condition as special case. For the nanocomposite with 1% filler, due to complete exfoliation of the clay in to resin, the molecular mobility may be restricted at high temperature and hence the increase in Tg. Whereas for 2% filled samples the Tg low as compared to 1% filled samples. Here due to increased percentage of clay the absence of entanglement surrounding the nanoclay, the effect due to surface modifiers, un reacted resin plasticization, and a lower cross-link density have been attributed to the decrease in Tg. [36]

Tan δ increase with increase of filler weight percentage. Various mechanisms like matrix viscoelasticity, filler/filler interfacial friction, etc., could increase the damping capacity of the polymer composite materials. However, the molecular motion at room temperature is frozen, and this may not contribute to the damping mechanisms. At Tg, the Tan δ value is higher for nanocomposites indicating the viscous damping because of the segmental motion in the polymer. Tan δ at Tg of the nanocomposites with 0.5 % and 1.5 % filler is higher than that of pure polyester FRP. This increase in the damping factor can be attributed to the restriction to the molecular movements of nano filler, which caused reduction in the matrix viscoelasticity. However, this did not agree with the samples with 1% and 2 % nano filler. [36]





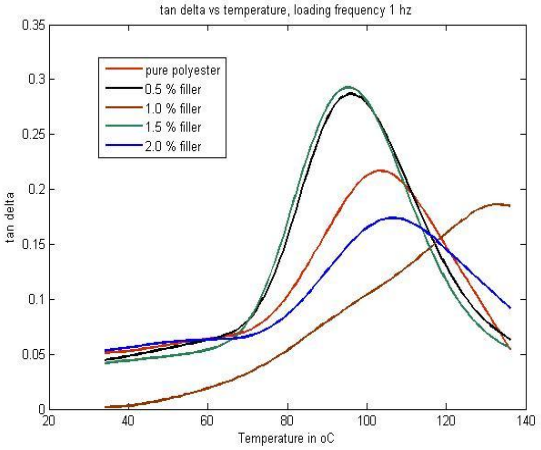


Fig: 1 Variation of (a) Storage modulus with temperature, (b) Loss modulus with temperature, (c) tan(delta) with temperature at frequency 1 Hz

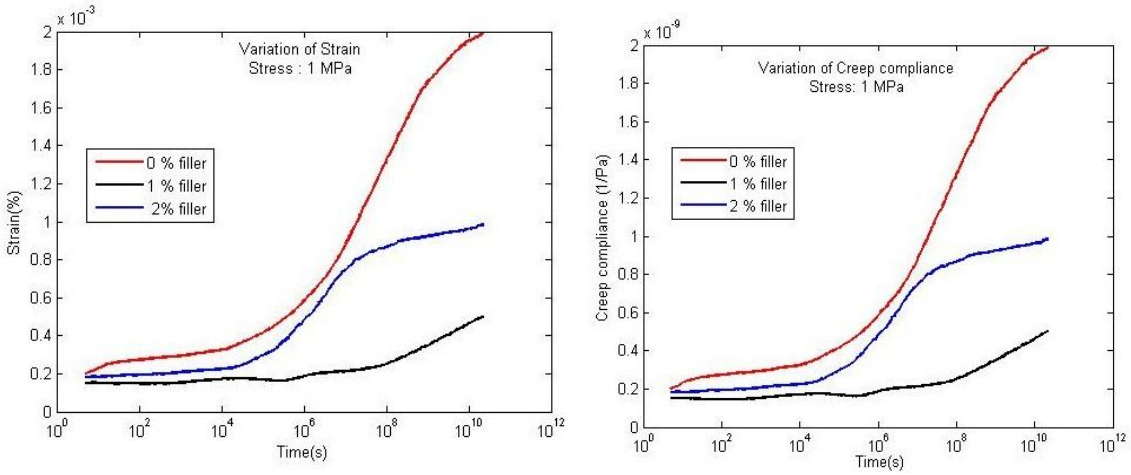


Fig: 02, (a) creep strain and (b) creep compliance with time at stress 1 MPa

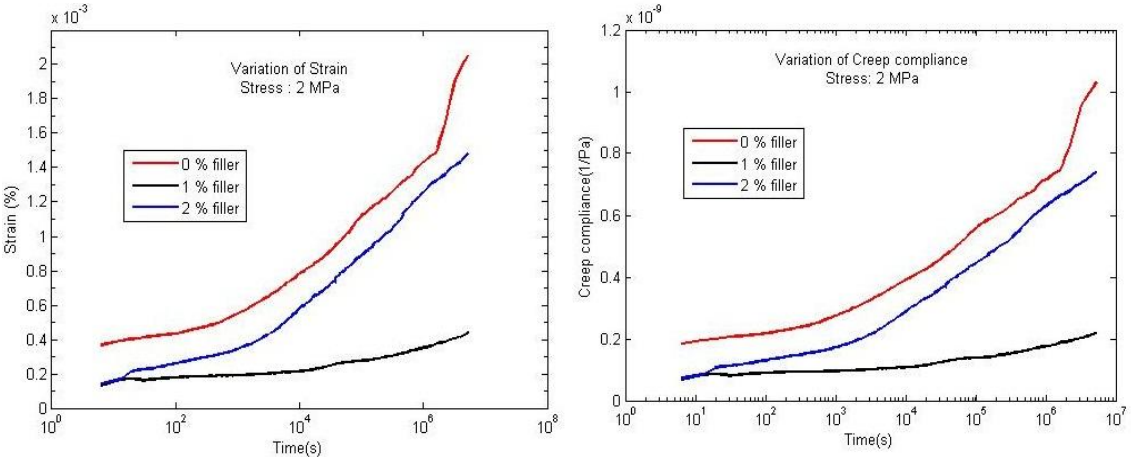


Fig: 03, (a) creep strain and (b) creep compliance with time at stress 2 MPa

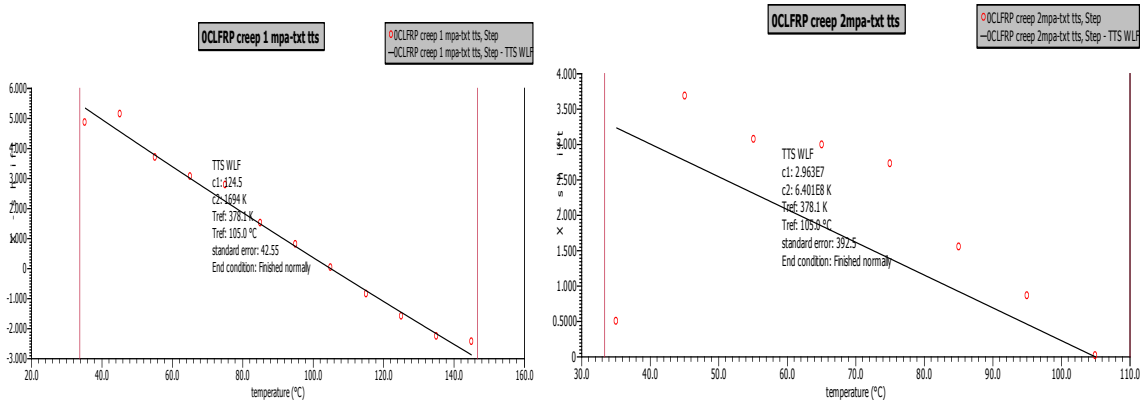


Fig:04 , Variation of shift factor for the specimen with 0% filler at (a) stress 1 MPa and (b) stress 2 MPa

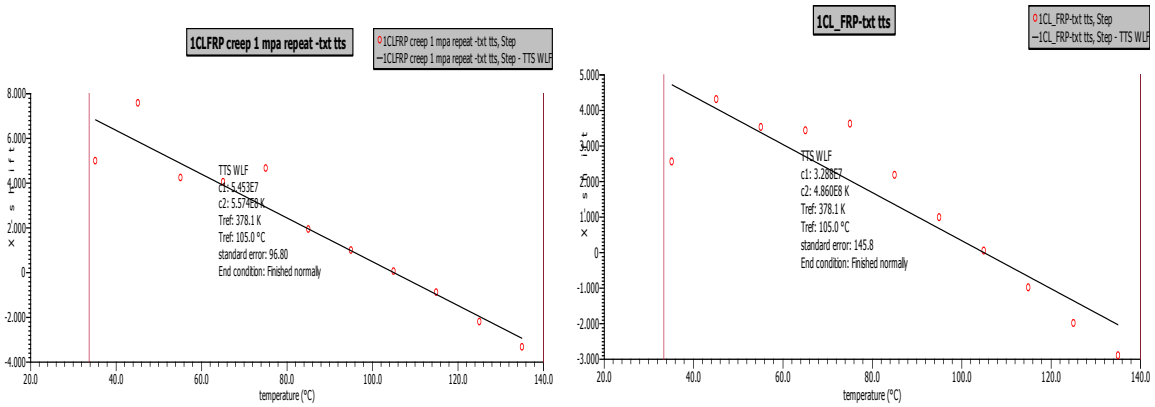


Fig:05, Variation of shift factor for the specimen with 1 % filler at (a) Stress 1 MPa and (b) Stress 2 MPa

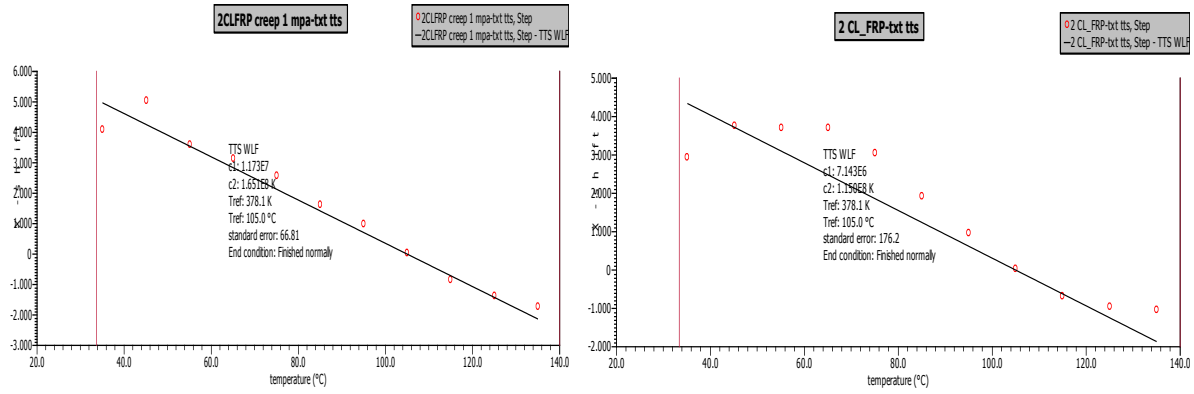


Fig:06, Variation of shift factor for the specimen with 2% filler at (a) Stress 1 MPa and (b) Stress 2 MPa

4.2 Creep data analysis

The experimental results of tensile creep behaviour analysis is given in fig. 2 and 3 respectively for the experiment conducted at constant stress 1 MPa and 2 MPa. Tensile creep properties of fiber reinforced composites modified with 1% and 2% weight nano particles as well as without nano particle (0%) have been determined. The master curve obtained with reference temperature of 30^{o}C for the creep compliance and creep strain with time at constant stress 1 MPa is as in fig 2(a) and (b). The creep strain as well as creep compliance is almost constant up to 10000 seconds of loading. After that the creep compliance and creep strain increases with time. The rate of increase in pure polyester FRP is high. Also the creep compliance and strain is minimum for

1% nano clay filled specimen. And for 2% nano clay filled specimen the creep compliance and creep strain are high but low as compared to pure polyester (0% filler) FRP. 1% filled FRP has creep compliance and strain constant over a long time. Which indicates the improvement in the performance of the material by the addition of nano filler and the sustainable performance of the material at 1% nano clay inclusion. This may be attributed to the restricted motion of the polymer chain by the addition of nano filler which occupies the interface. The nano platelets also help to establish a good bonding between the fiber and polymer matrix, which also restricts the motion of the polymer matrix. For the 2% filled nano composite the possibility for the agglomeration of the clay particles is high which may result in improper mixing.

The master curve plotted with experimental results at reference temperature 30^{o}C and frequency 0.5 Hz, 1.1 Hz, 2.3 Hz, 5 Hz, 10.8 Hz, 23.2 Hz and 50 Hz is as shown in figure 7. The creep properties describe the effect of nano filler on the long term behavior under continues loading. It is observed that the storage modulus increases first and reaches a stable condition with increase of frequency. The change in the nature of curve reaches the limiting value corresponds to the peak of loss modulus curve as well as tan (delta). The peak of damping coefficient curve shifted to higher frequency range slightly with the addition of nano filler, which is a clear indication of the damping nature of nano composite. With the creep deformation of nano composites with 1 wt% Cloisite15A shows the minimum value and which shows improved creep resistance from nano filler. The creep resistance does not show any notable change with the applied stress from the experimental data of 1MPa and 2 MPa applied stress. This also adds to the improved performance of the material. [28]

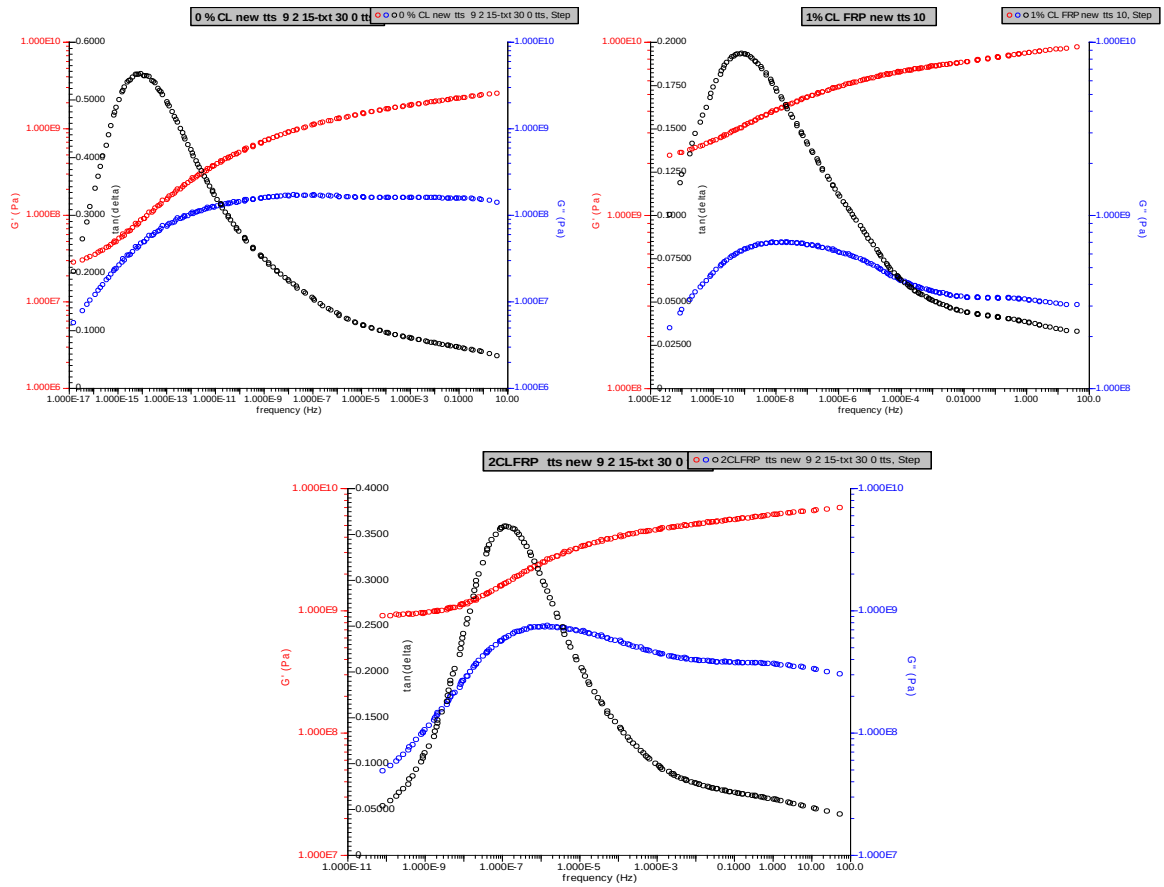


Fig 7, Time temperature super position data obtained for various nano composite: (a) 0 % filled FRP, (b) 1 % filled FRP and (c) 2 % filled FRP.

V. Conclusions

The following conclusions can be drawn from the studies.

1. The storage modulus shows improvement with the addition of nanoclay to polyester matrix
2. Adding 1% clay provides maximum Dynamic mechanical properties. Also the value of storage modulus decreased with further increase of nano filler.
3. Creep compliance of the nanocomposite is least at 1% nanoclay content and remains almost steady for a longer period of time.

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