

RESEARCH ARTICLE

Creep compliance of functionalized graphene-epoxy nanocomposites

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Abstract

Creep behavior is observed even at room temperature in polymers and polymer composites under constant stress. In the design of structural elements using these materials, the determination of creep compliance for structural strength and performance analysis is crucial in terms of reliability and usability. In this study, the creep compliance of pure epoxy and functionalized graphene-reinforced epoxy nanocomposites were experimentally investigated and compared at room temperature at constant stress levels of 50, 100, and 200 MPa representing the viscoelastic region, yielding region and viscoplastic region, respectively. To reveal the effect of temperature as well as stress level on this behavior, the creep compliance of epoxy and graphene-epoxy nanocomposites was investigated at 65°C in addition to room temperature after the pure graphene used as a reinforcement element was functionalized with Triton X-100, nanocomposite production was carried out. In 2h creep tests, it was observed that as the constant stress level increased, the creep compliance increased and the creep compliance of the nanocomposite was lower than that of the epoxy at all stress levels. The creep compliance of the epoxy was improved by 65% with the addition of functionalized graphene at room temperature and a stress level of 100 MPa. As the temperature increased, the creep compliance of both epoxy and functionalized graphene-epoxy nanocomposite increased due to molecular mobility and viscous flow. However, at high temperatures, the positive effect of functionalized graphene on the compliance of the epoxy is higher. At 65°C and a stress level of 100 MPa, the improvement rate of creep compliance is 78%. Functionalized graphene exhibited a more effective behavior on creep resistance at high temperatures. The obtained results showed that creep compliance was significantly affected by stress level and temperature.

1. Introduction

Polymeric materials that exhibit viscoelastic-viscoplastic characteristics are greatly influenced by creep behavior, which serves as a significant representation of these characteristics. Epoxy resins, which belong to the group of thermoset polymers, are widely used in various industrial areas due to their excellent adhesion, chemical, and heat resistance, as well as their mechanical and electrical properties [1]. Epoxies are the most commonly preferred matrix material in polymer composites. As such in all polymeric materials, epoxies are subject to creep strain, which is the time-dependent strain under constant stress, even at room temperature. [2]. Due to their inherent brittleness and tendency to creep deformation, the use of epoxies in structures

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subjected to long-term loading is limited [3]. Therefore, research has focused on enhancing the creep resistance of epoxy resins and expanding their application areas. Various reinforcing elements have been employed to improve the creep resistance of epoxy resins.[4,5].

With its discovery in recent years, graphene is a nanomaterial that has attracted attention as a reinforcing material in polymer composites due to its two-dimensional structure and excellent physical, chemical, and electrical properties [6]. The effect of graphene on the mechanical properties of polymer nanocomposites has been recently reported in the literature [7,8]. In addition to mechanical performance, its effect on creep resistance is also an issue that needs to be investigated and developed. A clear understanding of the impact of high-performance graphene on this behavior, which has a negative effect on epoxy, is essential for estimating the fatigue life of structures that are subjected to long-term loading. For this purpose, the fabrication of graphene-epoxy nanocomposites and their characterization to detect creep behavior is the motivation of this study. There are a limited number of studies in the literature investigating the effect of graphene on the creep behavior of epoxy [9,10].

The main challenge in the production of graphene-epoxy nanocomposites is to ensure homogeneous dispersion of graphene in the matrix material. For this purpose, a three-roll Rolling (3RM) method was preferred in this study to overcome the challenge and provide a more effective dispersion [10]. Another problem to be overcome in nanocomposites using graphene as reinforcement material is the interfacial interaction between the two phases of the composite. The agglomeration of graphene sheets is caused by Van der Waals forces between the graphene sheets [11]. Increasing the interfacial interaction between graphene and matrix is critical for a homogeneous distribution and a lower aggregation rate [10]. For this reason, it is important to functionalize graphene with various methods to increase the interfacial interaction with the epoxy matrix [12].

In this study, the creep compliance of pure epoxy and functionalized graphene (f-GNF)-epoxy nanocomposites was experimentally investigated at room temperature at constant stress levels of 50, 100, and 200 MPa. In addition to the effect of stress levels, the creep compliance of epoxy and f-GNF-epoxy nanocomposites containing 0.1wt% f-GNF was also investigated at 65°C in addition to room temperature to reveal the effect of temperature on this behavior. Functionalized graphene improved the creep resistance of the epoxy by preventing it from deforming over time. Accordingly, it has been determined that the material's durability has increased. It has also been reported that as the temperature increases, the creep resistance decreases compared to room temperature. It has been demonstrated that epoxy nanocomposites for structures operating under long-term loadings have better durability with the effect of more functional graphene, supported by experimental data.

2. Materials and methods

2.1. Materials

In this study, the Araldite LY 564 / Aradur 2954 epoxy system was used as the matrix material. The reinforcement material is graphene produced by the electric arc discharge method [13]. To increase the interfacial interaction of the obtained pure graphene with the matrix material and to obtain a more homogeneous distribution, functionalization was carried out by a non-covalent method with Triton X-100 surfactant. The functionalization processes of graphene are given in detail in [10].

2.2. Sample production

In the production of nanocomposite test samples, a three-roll milling method was used to prevent the agglomeration of graphene in the epoxy matrix material. First, graphene and epoxy resin were mixed at 400 rpm for 15 min using a magnetic stirrer. The distance between the rolls of the three-roll milling was set to 5

μm , the roll rotation speed was set to 250 rpm, and the mixture was rolled 5 times. After the graphene was uniformly dispersed in the epoxy matrix, Aradur 2954 hardener was added to the mixture at the manufacturer's recommended ratio of 100:35 by volume. The mixture was stirred at 400 rpm for 15 minutes using a magnetic stirrer. The beaker containing the mixture was placed in the vacuum chamber and degassed at room temperature for 60 minutes and then the mixture was poured into the silicone mold. To remove the gases that may be trapped in the mixture during pouring into the mold, the mold was placed in the vacuum chamber and degassed for 60 minutes. In the next step, the mold was placed in an oven preheated to 80°C and cured for 60 minutes. After the oven temperature was increased to 160°C , post-curing was performed for 240 minutes. The f-GNF-epoxy nanocomposites produced in this study contain 0.1 wt% f-GNF. The f-GNF-epoxy nanocomposite production details are shown in Fig. 1.

2.3. Creep test under uniaxial compression loading

Creep tests of pure epoxy and f-GNF-epoxy nanocomposites containing 0.1 wt% f-GNF under uniaxial compression loading were first carried out at three different constant stress levels at room temperature at a strain rate of $1.E-1 / \text{s}$. These stress levels were determined from the compressive stress-strain curve of pure epoxy. The quasi-static compression test of pure epoxy was carried out at room temperature at a strain rate of $1.E-1 / \text{s}$ using the Instron 5982 model universal static test device. The test setup is shown in Fig. 2a. The dimensions of the compression cylindrical-shaped samples tested with a diameter of 12 mm and a length of 12 mm at a strain rate of $1.E-1 / \text{s}$ at room temperature were determined according to the ASTM D695 standard.

At least three tests of pure epoxy and f-GNF-Epoxy containing 0.1 wt% f-GNF nanocomposite were performed under each condition and the repeatability of the tests was checked and repeated tests were averaged. The repeated test results of pure epoxy and nanocomposite are given in Figs. 3a and 3c, respectively. The result obtained by taking the arithmetic average of the pure epoxy sample tests performed at a strain rate of $1.E-1 / \text{s}$ is given in Fig. 3b. Comparison of the true stress-strain behavior of f-GNF-Epoxy nanocomposite containing 0.1 wt% f-GNF with pure epoxy and the changes in material behavior are shown in Fig. 3d

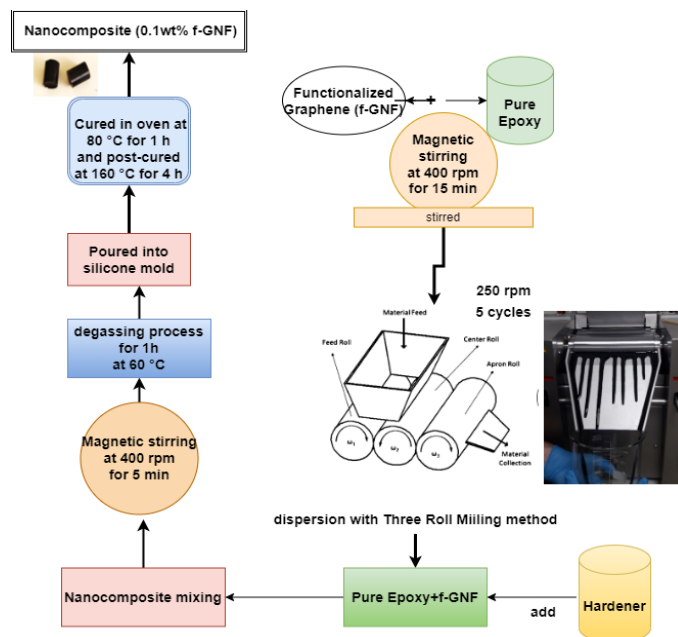


Fig. 1. Production details of F-GNF-Epoxy nanocomposite sample

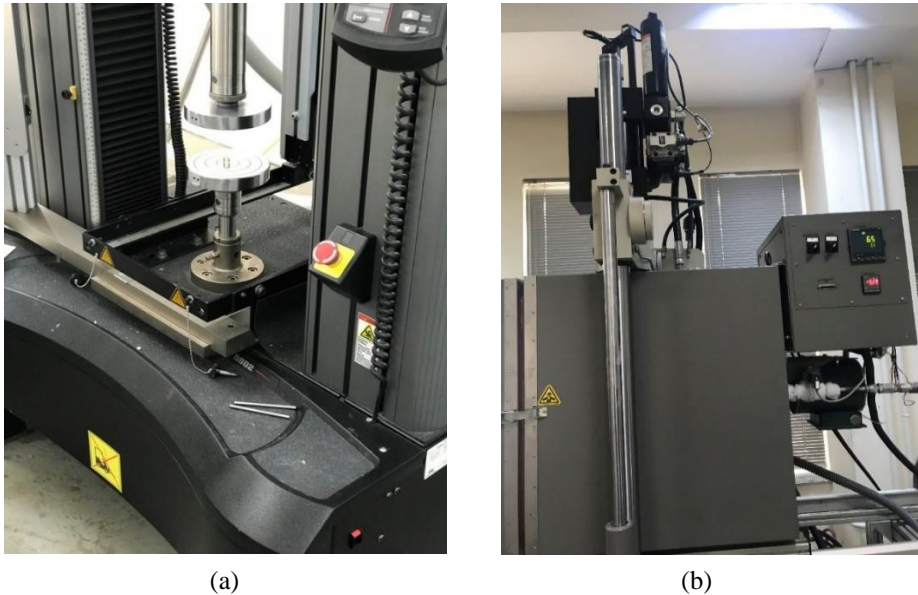


Fig. 2. a) INSTRON 5982, b) 65°C-MTS -322 universal test device

As shown in Figs. 3a and 3c, the repeated test results of both pure epoxy and f-GNF-Epoxy containing 0.1wt% f-GNF nanocomposite are consistent with each other. The results obtained demonstrated the reliability of the tests. The change in stress with increased strain in pure epoxy can be observed in Fig. 3b. In summary, this change consists of a linear increase in the elastic region, a small non-linear behavior in the yield region, a non-obvious strain softening after the yield region, and a very significant non-linear strain hardening that continues until rupture. It can be summarized as non-hardening. These results are consistent with the results in the literature[1,2]. The yield stress of pure epoxy was determined as 137 MPa from the stress-strain behavior at a strain rate of 1.E-1/s.

As seen in Fig. 3d, f-GNF-Epoxy nanocomposites have exhibited significant improvement over pure epoxy when compared in all regions. The functionalization of graphene with Triton X-100 has resulted in the creation of a strong interfacial region between epoxy and graphene. Therefore, the increased interfacial interaction has enabled f-GNF-Epoxy nanocomposites to exhibit more effective material behavior compared to pure epoxy [10]. Yield stress (153 MPa) of f-GNF-Epoxy nanocomposites increased compared to epoxy at a strain rate of 1.E-1 /s. f-GNF improved the material properties by having a barrier effect on the mobility of polymer chains. In addition, the effective distribution of f-GNF in pure epoxy by the three-roll milling method is another factor in increasing the material properties.

Using true stress-strain data for epoxy at a strain rate of 1.E-1 /s, the constant stress level for the creep test was determined. Creep tests were aimed to examine the effect of stress level and temperature on creep behavior and at the same time to reveal the change in creep behavior in viscoelastic and viscoplastic regions. In this context, constant stress levels for creep behavior are 50 MPa, 100 MPa, and 200 MPa to reveal the creep compliance change in the viscoelastic region, the around the yield region, and in the viscoplastic region, respectively. Creep tests of pure epoxy and f-GNF-epoxy nanocomposites were carried out for 2 h. In creep tests, the material was first subjected to uniaxial compression loading at a strain rate of 1.E-1/s up to the stress levels stated above. During the experiment, the measurement of the strain and the speed control was done in a "position-controlled" manner with the 'crosshead' movement of the tester.

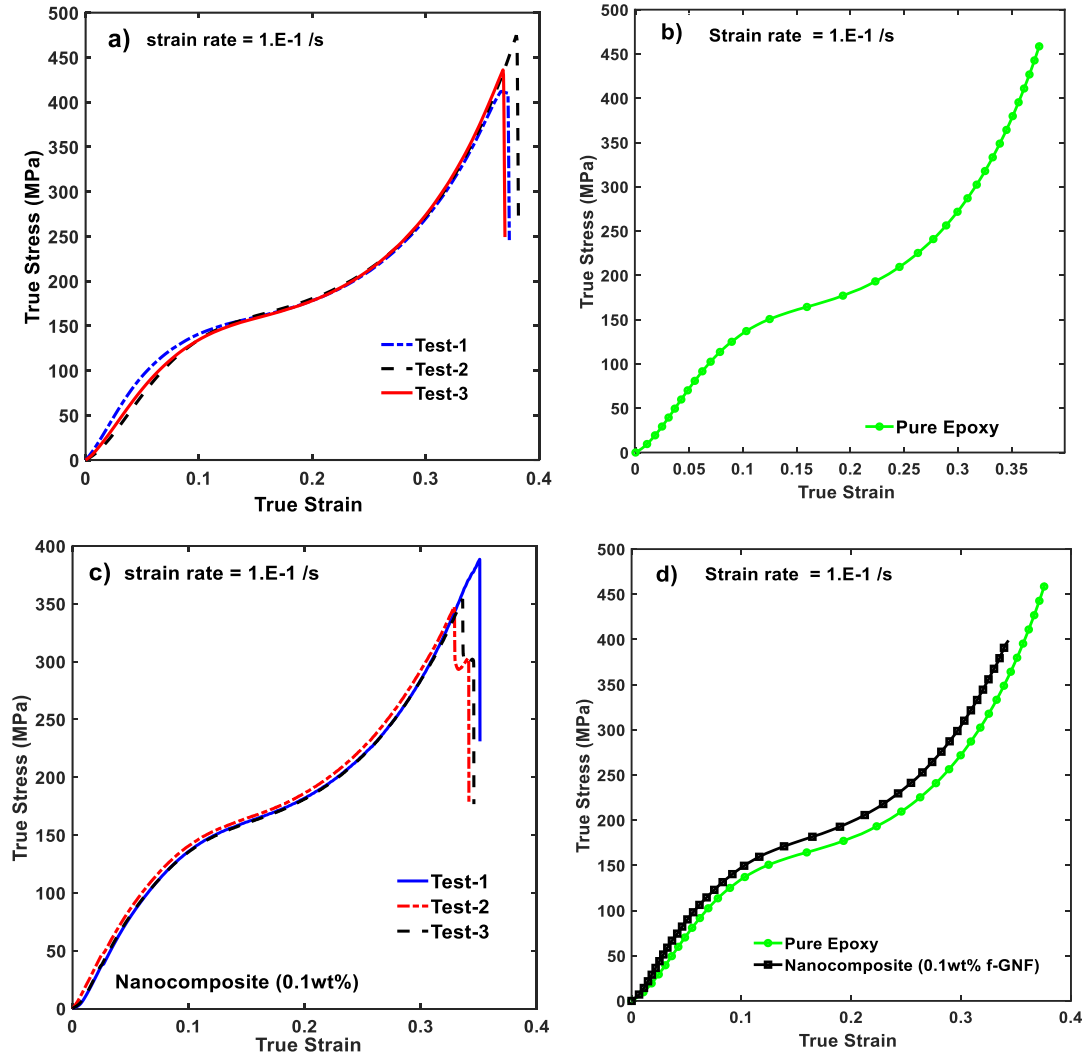


Fig. 3. a) The repeated test results of pure epoxy, b) the average test result of pure epoxy, c) the repeated test results of pure epoxy nanocomposite (0.1wt% f-GNF), d) comparison of the true stress-strain behavior of pure epoxy and nanocomposite

In the next step, the effect of temperature on the creep compliance behavior of both pure epoxy and f-GNF-epoxy nanocomposites under uniaxial compression loading was investigated. The tests were carried out at $65^{\circ}C$ and two different stress levels (50 and 100 MPa) and the effect of temperature was determined by comparing the room temperature results. Temperature-controlled creep tests were also carried out for 2 hours on the MTS 322 Universal test device (Fig. 2b)

3. Experimental results and discussion

3.1. Effect of constant stress levels on creep compliance

In addition to the static-mechanical behavior of f-GNF-epoxy nanocomposites, creep test data used to reveal material behavior in long-term applications are also the results required to predict the time-dependent behavior of materials. Material models implemented in a finite element-based software simulate and predict

viscoelastic-viscoplastic behaviors such as creep, stress relaxation, and recovery. These models need creep compliance data obtained from creep tests to simulate creep-like behavior. Creep compliance ($J(t)$) is defined as the ratio of the time-dependent strain to the constant stress level. (Eq. 1)

$$J(t) = \varepsilon(t)/\sigma_0 \quad (1)$$

where $\varepsilon(t)$ the time-dependent creep strain, σ_0 is the constant stress level.

Compliance results are required to model the effect of many parameters such as stress level, creep time, and temperature on creep behavior, and the model prediction approaches are a data network to be used in the design and analysis stages of structural parts using epoxy and f-GNF-epoxy nanocomposites. Therefore, the stress-dependent change of creep compliance data of epoxy and f-GNF-epoxy nanocomposites is also considered in this study. Creep compliance data of pure epoxy and f-GNF-epoxy nanocomposites containing 0.1 wt% f- GNF at room temperature in the viscoelastic region (50 MPa), the y around yield region (100 MPa) and in the visco-plastic region (200 MPa) are comparatively shown in Figs. 4a, 4b and 4c respectively. After 2 h of creep time, the increase in creep compliance data at three different constant stress levels and the improvement in creep resistance of f-GNF reinforcement compared to epoxy are given in Table 1.

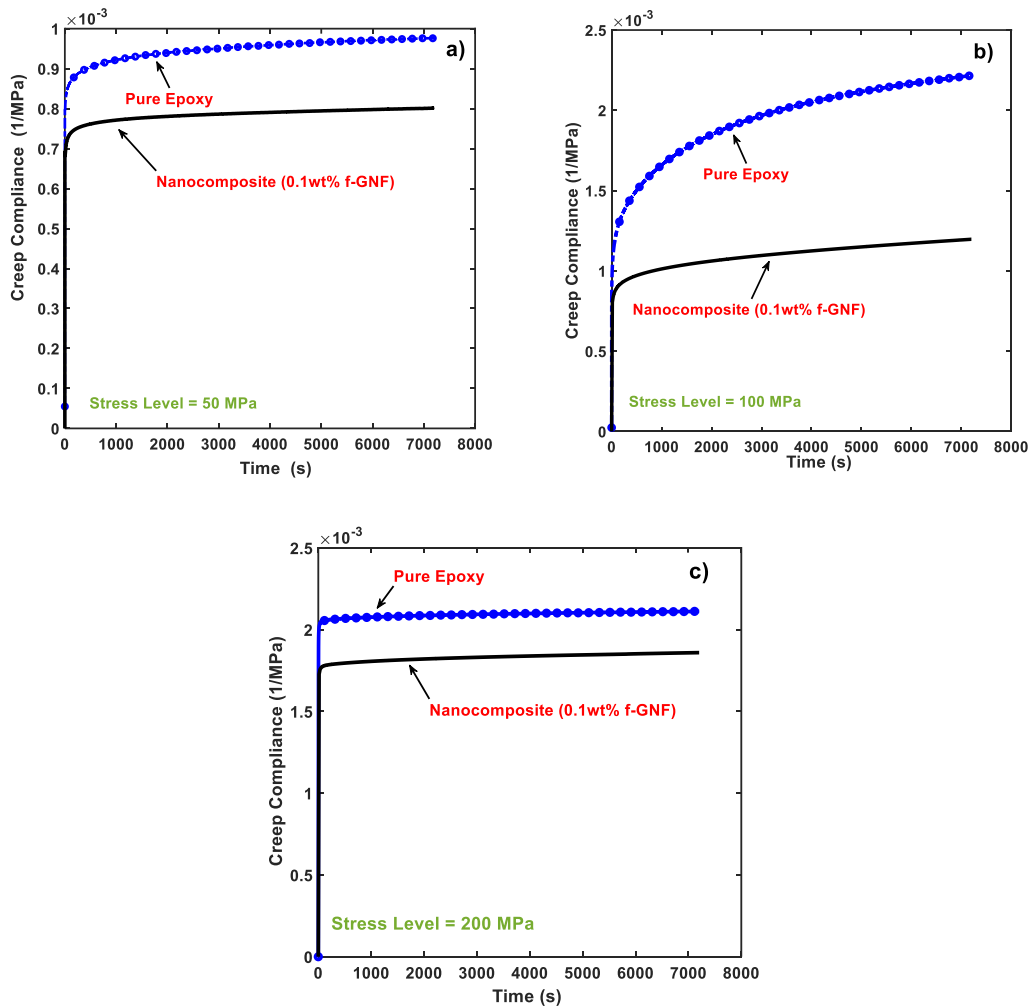


Fig. 4. Time-dependent change of creep compliance for pure epoxy and f-GNF-epoxy nanocomposites containing 0.1 wt% f-GNF at constant stress levels of 50, 100, and 200 MPa

As can be seen in Fig. 4, the creep compliance data of both pure epoxy and f-GNF-epoxy nanocomposite under compression loading are highly dependent on the constant stress level and an increase in creep compliance data is observed with the increase in constant stress level. The obtained result is based on the free volume theory in related studies. Based on the relevant theory, there exists a correlation between viscosity and the free volume fraction, and increasing stress can increase the free volume fraction. This increase results in a decrease in the time-dependent viscosity. These findings may be linked to a rise in molecular thermodynamic motion and an increase in creep data [14]. The creep compliance data of the nanocomposite at all stress levels are lower than epoxy.

When Table 1 is examined, significant improvements in this viscoelastic characteristic were achieved even with only 0.1wt% f-GNF supplementation. As a result of the 2-h test, an improvement of 32% in the viscoelastic deformation regime, 65% in the yield area, and 48% in the viscoplastic deformation regime was achieved. It is thought that the strong interfacial interaction of the C-H-O functional groups in functional graphene with the epoxy groups affects the structure and dynamics of the epoxy and prevents the movements of the epoxide chains, thus reducing the compliance data. When the results are examined, the fact that the viscous flow is low in the viscoelastic linear region is effective in the lower compliance data at these levels. Although the 100 MPa stress level corresponds to just before the yield limit, it is in the region where the flow phenomenon has just begun, in other words, the resistance of the material under mechanical load is broken and the polymer chains are displaced to find a new equilibrium position relative to each other. Therefore, at this load level, the polymer chains are displaced relative to each other without much resistance. In this case, there appears to be a significant increase in compliance on a macro scale [2]. At this stress level, f-GNF increased the creep resistance in the material by providing a stiffness effect on the epoxy matrix. As a result, a high improvement of 65% was achieved. Another important effect of the improvement in compliance data at room temperature is the effect of the effective graphene distribution provided by the three-roll milling method. Breaking the van der Waals bonds, which cause agglomeration in graphene, under the influence of shear stresses in this method is an important factor in minimizing graphene aggregation. As a result, a homogeneous distribution in the epoxy matrix is an important factor in the decrease in creep data [10].

3.2. Effect of temperature increase on creep compliance

The mechanical-material behavior of polymers and polymer-nanocomposites is highly dependent on temperature. In addition to its mechanical behaviors, viscoelastic behaviors such as creep, stress relaxation, and recovery also show temperature dependence. As seen from the previous results, polymeric materials are affected by creep behavior even at room temperature. In this context, it is critical to determine the effect of temperature on time-dependent deformations to reveal the practical applicability of polymeric materials in long-term loading [15]. For this purpose, creep tests were carried out at 65°C to examine the temperature effect on the creep response of both pure epoxy and f-GNF-Epoxy nanocomposite containing 0.1wt% f-GNF. To more clearly reveal the temperature effect on creep deformation, a comparison of the creep compliance data of both pure epoxy and f-GNF-epoxy nanocomposite at two different temperatures is shown in Figs. 5a and 5b. The improvement data in creep resistance obtained at 65°C are given in detail in Table 2.

It can be observed from Fig. 5 and Table 2 that the creep compliance is highly dependent on temperature for both pure epoxy and f-GNF-epoxy due to the highly increased molecular mobility and viscous flow [16]. The increase observed in the creep compliance data of polymers, which are easily deformed due to the weakening of the bonds between molecular chains at high temperatures, was similarly observed in nanocomposite samples. When looked at in terms of stress levels at high-temperature levels, higher compliance data were detected around yield for both epoxy and nanocomposites compared to the viscoelastic deformation regime. This is because epoxy has a chemically cross-linked structure and viscous flow is not expected at 50 MPa, which represents the stress level in the viscoelastic region [17]. The increases in creep

compliance data as the temperature increases can be seen in both Fig. 5 and Table 2. The results reveal that there is an increase in elastic deformation due to temperature increase. The softening of the matrix due to temperature increase is the factor in this case. It can be seen from the results that the creep rate, that is, the slope of the curves in the creep phase, becomes more evident with increasing temperature. The increasing slopes of the curves with temperature indicate that the viscoelastic deformation rate increases, thus increasing deformation at high temperatures [17].

Table 1. Improvement in creep compliance data of f-GNF-epoxy nanocomposites containing 0.1wt% f-GNF compared to pure epoxy at three different stress levels at room temperature

| Stress level (MPa) | Material | Increase in creep compliance end-test | Decrease in creep compliance (1/MPa) | Improvement creep compliance % |
|--------------------|---------------------|---------------------------------------|--------------------------------------|--------------------------------|
| 50 | Pure Epoxy | 0.0001658 | 0.0000536 | 32.32 |
| | f-GNF(0.1wt%)-Epoxy | 0.0001122 | | |
| 100 | Pure Epoxy | 0.0013095 | 0.0008571 | 65.45 |
| | f-GNF(0.1wt%)-Epoxy | 0.0004524 | | |
| 200 | Pure Epoxy | 0.00035075 | 0.00017035 | 48.56 |
| | f-GNF(0.1wt%)-Epoxy | 0.0001804 | | |

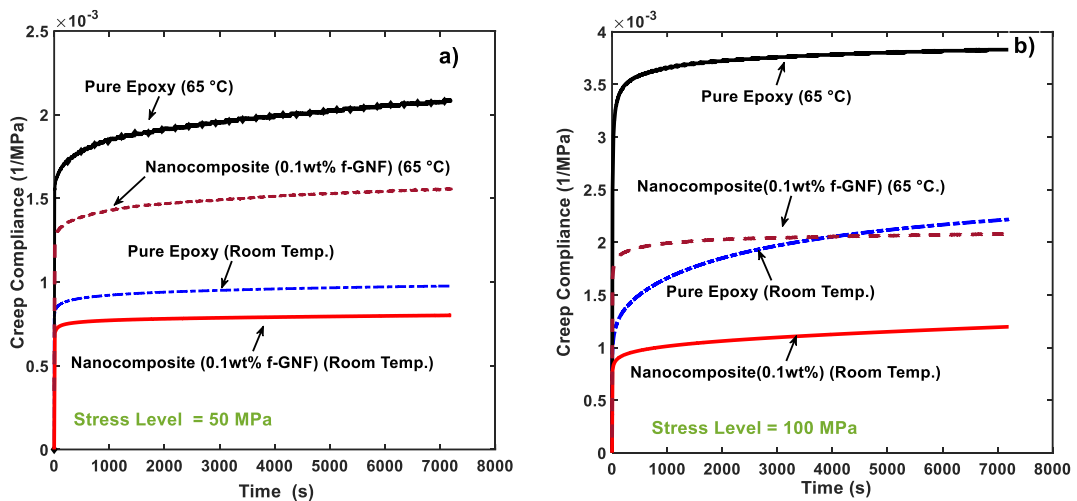


Fig. 5. Comparison of the creep compliance of pure epoxy and f-GNF-epoxy nanocomposite containing 0.1wt% f-GNF at room temperature and 65°C at constant stress levels of a) 50 MPa b) 100 MPa

Table 2. Improvement in creep compliance data of f-GNF-epoxy nanocomposites containing 0.1wt% f-GNF compared to pure epoxy at two different stress levels at 65°C

| Stress level (MPa) | Material | Increase in Creep compliance end-test | Decrease in creep compliance (1/MPa) | Improvement creep compliance % |
|--------------------|---------------------|---------------------------------------|--------------------------------------|--------------------------------|
| 50 | Pure Epoxy | 0,0007 | 0.00662 | 45.7 |
| | f-GNF(0.1wt%)-Epoxy | 0,00038 | | |
| 100 | Pure Epoxy | 0.003348 | 0.00261144 | 78 |
| | f-GNF(0.1wt%)-Epoxy | 0.0073656 | | |

65°C is a temperature close to the glass transition temperature of the material, determined by dynamic mechanical analysis [18]. When the results obtained are examined, as the temperature approaches the glass transition temperature, the orientation of the molecular layers increases during creep loading, and accordingly the creep compliance increases. At the high-temperature level, which is responsible for greater creep compliance, the presence of f-GNF in the epoxy restricts the creep deformation of the epoxy by acting as a barrier to the molecular orientation.

Improvements in creep compliance due to the decreases detected on both elastic and viscoelastic strains were also determined at different stress levels at high temperatures. At the end of the 2h creep test, at 65°C, the improvement rates in the creep compliance data of the nanocomposites were approximately 46% in the viscoelastic deformation regime, while it was 78% in the yield around. As seen in Table 1, these ratios are higher than the room temperature results, that is, f-GNF had a more strengthening effect on the creep resistance at high temperatures. This result is based on the effect of graphene on the activation energy required for the mobility of polymer chains due to their superior thermal conductivity [14]. The heat applied to the nanocomposite with thermal energy is absorbed by the graphene and the activation energy increases accordingly. As a result of this case, the mobility of the polymer chains is further restricted at high temperatures. In this study, the functionalized graphene modified with Triton X-100 and the more effective distribution of f-GNF in the epoxy matrix through the three-roll milling method has significantly influenced the creep behavior under compressive loading and at all temperature levels. The results obtained showed that the creep resistance of epoxy resins improved significantly in all stress regions with the addition of 0.1% f-GNF.

4. Conclusions

In this study, the creep behavior of the fabricated f-GNF-epoxy nanocomposite at room temperature and 65°C was experimentally examined. Since stress level is the primary factor affecting creep, which is defined as the increase in deformation at constant stress, the effects of stress were examined by performing creep tests at 3 different stress levels. Creep compliance, defined as the ratio of creep strain to stress, is a material parameter used in the structural analysis of polymers and polymer matrix composites. This time-dependent parameter is widely used in the design of structural elements and stress-strain analysis. It was observed that the creep compliance of the developed nanocomposite material was lower than that of epoxy. At room temperature and 100 MPa constant stress level, the creep compliance of the f-GNF-epoxy nanocomposite containing 0.1wt% f-GNF decreased by 65% compared to pure epoxy. Since f-GNF reinforcement restricts the molecular mobility of polymer chains, the creep compliance of the nanocomposite material is lower than pure epoxy. The increase in creep resistance at 65°C and 100 MPa constant stress level is 78%. These results show that time-dependent deformation is less in nanocomposites than in epoxy, and the nanocomposite structure will be more stable.

Conflict of interests

The author(s) declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

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Data availability statement

Data generated during the current study are available from the corresponding author upon reasonable request.

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