

## EXPERIMENTAL INVESTIGATION ON THE EFFECT OF SiO<sub>2</sub> NANO PARTICLES IN AXIAL CREEP RUPTURE BEHAVIOUR OF GFRP

### EKSPERIMENTALNO ISTRAŽIVANJE UTICAJA NANOČESTICA SiO<sub>2</sub> NA AKSIJALNO RAZARANJE GFRP PUZANJEM

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#### Keywords

- creep rupture
- GFRP
- ultimate tensile strength
- axial loads
- SiO<sub>2</sub> nano particles

#### Abstract

*Creep strength evaluation is vital for structural materials used in high-temperature applications, like Glass Fibre-Reinforced Polymers (GFRP) in industries such as aerospace, automotive, and marine. Factors like creep behaviour, rupture time, load, and strain are crucial for material design due to extreme working conditions. This study investigates GFRP's creep rupture behaviour reinforced with nanoparticles, examining varied nano SiO<sub>2</sub> content (1 %, 2 %, 3 % wt.) and fibre orientations (0°, 15°, 30°, 45°). Tensile tests are conducted per ASTM D2990 standard to assess material strength, aiding in determining the necessary creep load rate for universal testing machine configuration. Creep tests are performed under axial loads at temperatures ranging from 50 °C to 140 °C. At 110 °C, the highest strain (0.9 %) is observed for 45° and 15° orientations, while 0° and 30° orientations exhibit the lowest strain. All structures show maximal strain at 110 °C, reaching 1 % for 45° and 15° orientations, and least strain for 0° and 30° orientations. Nanoparticle effects vary across orientations, with up to 2 % wt. resulting in increased strain rates at different temperatures. Notably, the 15° and 45° orientations display distinct responses.*

#### INTRODUCTION

Fibre-reinforced polymeric (FRP) composites are widely used in various engineering fields including aerospace, automotive, construction, sports equipment, and marine vehicles. This is mainly because of their excellent weight-to-strength ratio and ease of manufacturing. However, when examining the morphology of these composite materials, it is important to ensure good mechanical performance. In recent years, there has been a focus on improving the mechanical behaviour of FRP composites through the incorporation of nano fillers. This has led to enhanced performance at both the inner laminar and inter planar levels, /1/. To achieve these goals, the concept of hybrid composites can be utilised to overcome lamination issues by combining different types of reinforcements. Proper tailoring plays a crucial role in achiev-

#### Ključne reči

- razaranje puzanjem
- polimeri ojačani staklenim vlaknima (GFRP)
- zatezna čvrstoća
- aksijalno opterećenje
- SiO<sub>2</sub> nanočestice

#### Izvod

*Procena čvrstoće puzanja je važna kod konstrukcionih materijala u primeni na povišenoj temperaturi, na pr. polimeri ojačani staklenim vlaknima (GFRP) u aerokosmotehnici, automobilske i pomorske tehnici. Usled ekstremnih radnih uslova, od velikog značaja u projektovanju materijala je: ponašanje pri puzanju, vreme do loma, opterećenje i deformacije. U radu se istražuje ponašanje loma GFRP, pri puzanju, ojačan nanočesticama, razmatranjem promenljivog sastava nano SiO<sub>2</sub> (1 %, 2 %, 3 % tež.) i orijentacije vlakana (0°, 15°, 30°, 45°). Ispitivanja zatezanjem su izvedena prema ASTM D 2990 radi procene čvrstoće materijala, čime se olakšava i određivanje potrebne brzine opterećenja puzanja za konfigurisanje univerzalne mašine za ispitivanje. Ispitivanja puzanjem su izvedena pod aksijalnim opterećenjem na temperaturama u rasponu 50 °C do 140 °C. Na 110 °C, uočava se najveća deformacija (0,9 %) za orijentacije 45° i 15°, dok se za orijentacije 0° i 30° je ona najmanja. U svim strukturama je najveća deformacija na 110 °C, od 1 % za orijentacije 45° i 15°, a najmanja za 0° i 30°. Uticaji nanočestica se menjaju s obzirom na orijentacije, do 2 % tež. i povećanjem brzine deformacije na različitim temperaturama. Zapravo, orijentacije 15° i 45° pokazuju suprotna ponašanja.*

ing desired results. In the current study, glass fibre/epoxy (GE) composites are chosen due to their wide range of applications and long-lasting durability. The low weight of these composites is also essential for maintaining the structural stability of the polymeric base structure, /2, 3/. Furthermore, the use of reinforcements in composite materials creates interfaces that transform the composite structure into complex multiphase materials with a higher degree of dissimilarity, /4, 5/. Additionally, the mechanical behaviour of these materials is difficult to predict due to their complex details and structure. This is especially true in loading situations such as impact and bending, which are commonly encountered in structural applications /6, 7/. In the last few decades, significant advancements have been made in evaluating the mechanical behaviour of hybrid composites under axial loads

/8/. However, recent reviews have emphasised the importance of studying the tensile behaviour of composite materials /9, 10/. As a result, in recent years, many researchers have focused on incorporating nanoparticles to enhance the mechanical properties of composites /11, 12/. In these composite materials, nanoparticles are added to the matrices as nanofillers and are dispersed throughout the matrix to create a strong matrix and a new composite with enhanced mechanical and chemical properties /13, 14/. The most commonly used nanomaterials in composite materials include nano SiO<sub>2</sub> (silica), clay, carbon nanotubes (CNTs), and SiO<sub>2</sub>. Each type of nanoparticle possesses specific chemical and physical properties that can have different effects on the properties of composites /14, 15/. In an effort to enhance the critical buckling load in hybrid carbon fibre-reinforced laminates, CNTs and clay are applied. The results demonstrate that the addition of 1 wt.% of nano clay and 1.5 wt.% of CNTs increases the buckling force of the laminates by approximately 45 % /16/. MWCNTs are incorporated into polycarbonate composites to study the electrical and rheological percolation thresholds of PC/CNT nanocomposites. The results reveal significant and notable changes in the nanocomposite properties /17, 18/. In the field of material science, creep refers to the time and temperature-dependent deformation that is associated with the yield strength of materials. Creep can occur in materials that are subjected to prolonged periods of heat /19/. The tensile creep behaviour of a glass fibre-reinforced epoxy composite is investigated at three different temperatures: 24.85 °C, 59.85 °C, and 79.85 °C. The composite had fibre orientations of 0° and 90° angles. It is observed that the strain rate at ambient temperature is negligible, but significantly increases at higher temperatures /20, 21/. The tensile creep-rupture behaviour of T800H/2500 unidirectional carbon fibre and epoxy composite is investigated under different fibre orientations, temperatures, and angles /9, 22/. The creep lifetime is predicted by analysing the creep failure mechanism and then compared with experimental results /23-25/. Furthermore, the addition of 1 wt.% nano SiO<sub>2</sub> to polyamide 66 has been found to enhance the creep behaviour of the material /12/. Other studies have reviewed the influence of temperature and load on the creep performance of laminated glass fibre/epoxy composites reinforced with carbon nanotubes (CNTs) /26, 27/. The low-temperature creep resistance of GE composite is enhanced through the reinforcement of CNTs. It is found that oxidised CNTs exhibit a greater reinforcement effect compared to pristine CNTs. However, at elevated temperatures, the CNT-GE composite shows a poor creep response due to interfacial slippage. Additionally, high temperatures and loads promote interfacial slippage and pullout of the CNTs /14, 15, 28/. In another study, the effect of nano graphene/epoxy composites is investigated in a temperature range of 50 °C, 80 °C, and 110 °C. The results show that the addition of nano particles has a positive effect on creep behaviour, and the highest creep rate is observed at 110 °C. In a separate study, it is found that increasing the test temperature from 70 °C to 110 °C results in an increase in strain rate from approximately 24 % to approximately 97 %. This can be attributed to the softening of the polymer at higher temperatures /13,

29/. The aim of the current study is to investigate the impact of varying nano SiO<sub>2</sub> content (1 %, 2 %, 3 % wt.) and different fibre orientations (0-90°, 15°, 30°, 45°) on the material properties. Prior to conducting creep tests, the material needs to be tested to determine the desired load, which will then be used for creep tests. The creep tests will be performed at different temperatures including 50 °C, 80 °C, 110 °C, and 140 °C. It is found that the effect of temperature of 110°C has the most significant impact on material's behaviour /11, 13, 15/.

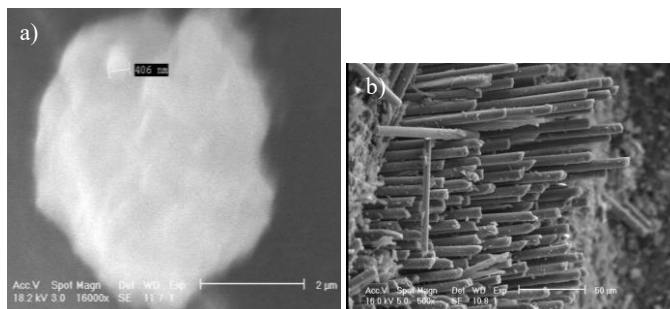
## EXPERIMENTAL DESIGN

### Materials

Epoxy resin utilised in this study is EPR1080, manufactured by Shell Chemicals Co. and supplied by Polymer Co. in Tehran, Iran (Nanophos S.A. Co). It has an epoxide equivalent weight of 185-192 g/eq. The nano SiO<sub>2</sub>, employed as the nano reinforcement in the epoxy matrix, is obtained from the Institute of Petroleum Industry (RIPI) in Iran. The nano SiO<sub>2</sub> has an outer diameter of 10-15 nm, a purity of over 95 %, and maximum length of less than 30 nm, as stated in Table 1 of the study. Tetrahydrofuran (THF), purchased from Merck Co. (Germany), is used as the solvent with a purity (GC) of over 99 %. The glass fibre used in experiments is procured from Toray Company.

### Fabrication of laminated composite

Laminate plates are prepared using 16 layers of glass fibre composite laminates with different orientations (0°, 15°, 30°, 45°) and various nano SiO<sub>2</sub> contents (neat, 1 %, 2 %, 3 %). All specimens are fabricated using the Vacuum Assisted Resin Transfer Moulding (VARTM) or resin infusion method. To prepare the required matrix, different nano silica contents are added to a cured resin and mixed using a magnetic stirrer for two hours at 2000 rpm. This process ensures a well-mixed matrix. The mixed matrix is then subjected to a homogeniser ultrasonic device (ultrasonic SONOPLUS-HD3200) with 80 % amplitude, 20 kHz frequency, and a pulsation cycle of 10 s On and 3 s Off for 10 minutes. This step facilitates the dispersion of silica particles within the epoxy matrix (refer to Fig. 1). Following the ultrasonication process, EPR-1080 (used as a hardener) is added to the nano/epoxy matrix based on a stoichiometric ratio of 15 parts per 100. In the next step, all prepared matrices are moulded into 16 glass fibre layers as shown in Fig. 1. After moulding, all specimens are kept at room temperature for 24 hours to allow initial solidification. Subsequently, the samples undergo final post-curing in a hot oven at 80 °C and 150 °C for 2 hours each.



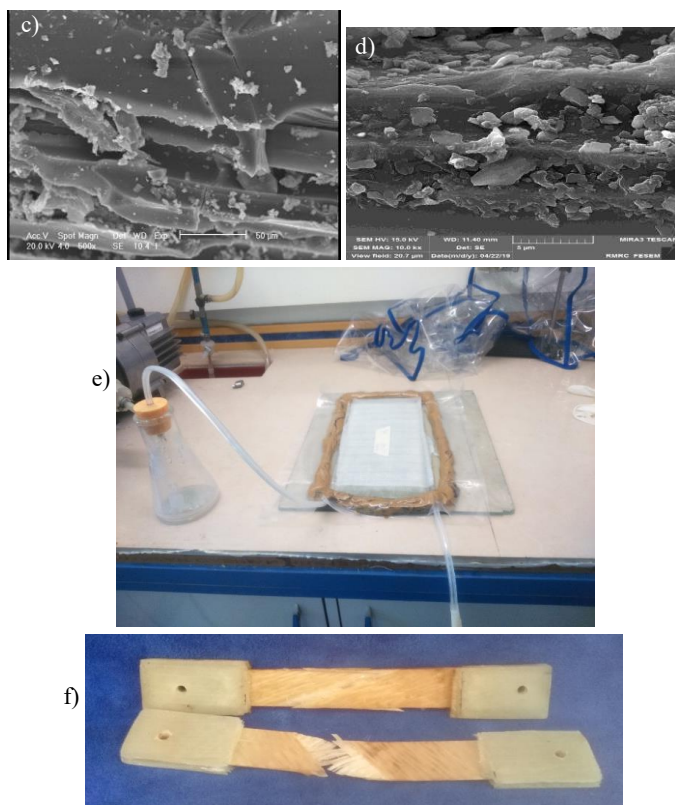


Figure 1. a)-d) Dispersing of nano fillers through epoxy and fibre; and e)-f) moulding process of matrices into 16 layers of GF and cut samples.

### Characterisations

All-composite sheets are cut according to ASTM D2990 with dimensions of 150×40 mm. GFRP base tabs are then attached using a hot method, as shown in Fig. 2. The samples are drilled to prepare them for the creep test holders. The spacing between the holes is set according to the test standard. Samples for the tensile test, following ASTM-D3039 standard, are cut to 130×40 mm. The thickness of all samples is measured using a calliper, and they all had a thickness of 4.3 mm. Both tensile and creep tests are conducted using an STM-150 machine from Santam Company in Iran. The tensile tests are performed at room temperature to determine the yield force, and the tensile curve is obtained from the force-extension results. Creep tests are conducted at the desired temperature, and the strain-time-dependent curves are obtained.

## RESULTS AND DISCUSSION

### Morphology of nano filler

The all-composite sheets are cut into sizes 150×40 mm according to ASTM D2990. Subsequently, GFRP base tabs are attached using a hot press, as depicted in Fig. 2. Following this, the samples are drilled to accommodate the creep test holders, ensuring adherence to the test standard and maintaining the appropriate spacing between the holes. For the tensile tests, additional samples are cut in accordance with ASTM-D3039, with sizes of 130×40 mm. The thickness of all samples is measured using a calliper, and it is determined that they had a uniform thickness of 4.3 mm. The tensile tests are conducted at room temperature to evaluate

the yield force. The force-extension results are utilised to construct the tensile curve. Conversely, the creep tests are carried out at desired temperatures, and the resulting strain-time-dependent curves are obtained using the STM-150 testing equipment from Santam Company (Iran).

### Tensile tests

In Fig. 2, the positioning of the samples between the jaws of the Universal Testing Machine is depicted, as shown in Fig. 3. It is important to note the inclusion of nano SiO<sub>2</sub>, as demonstrated in Fig. 3a. Results indicate that an increase in nano SiO<sub>2</sub> content leads to an improvement in ultimate tensile stress (UTS). However, it is observed that at higher nano SiO<sub>2</sub> contents, the elastic strain decreases while plastic strain increases. This can be attributed to a higher association of nano particles within the composite structure, resulting in a decrease in elastic modulus and total strain.

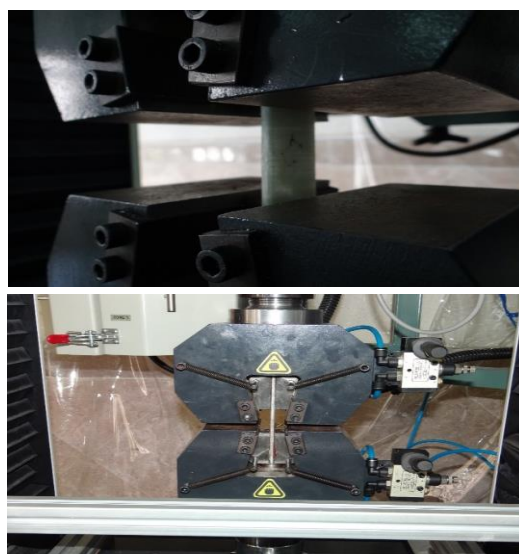
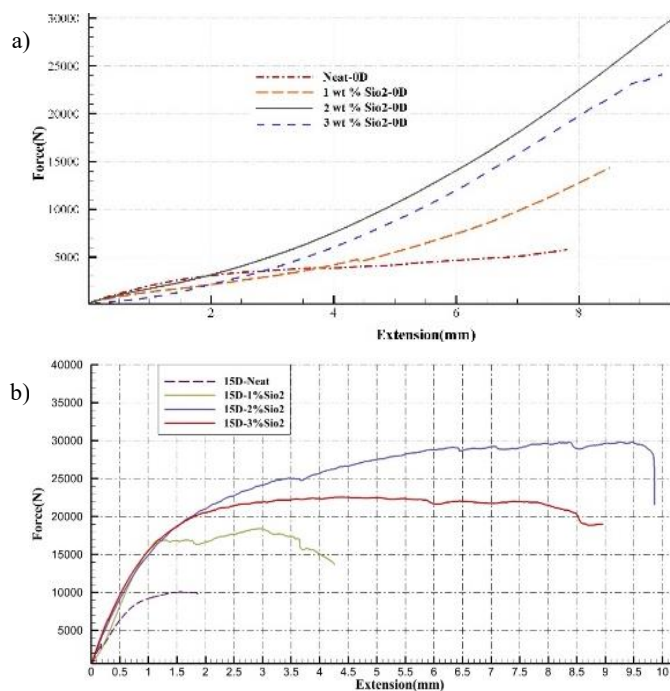


Figure 2. Placement of samples in universal testing machine.





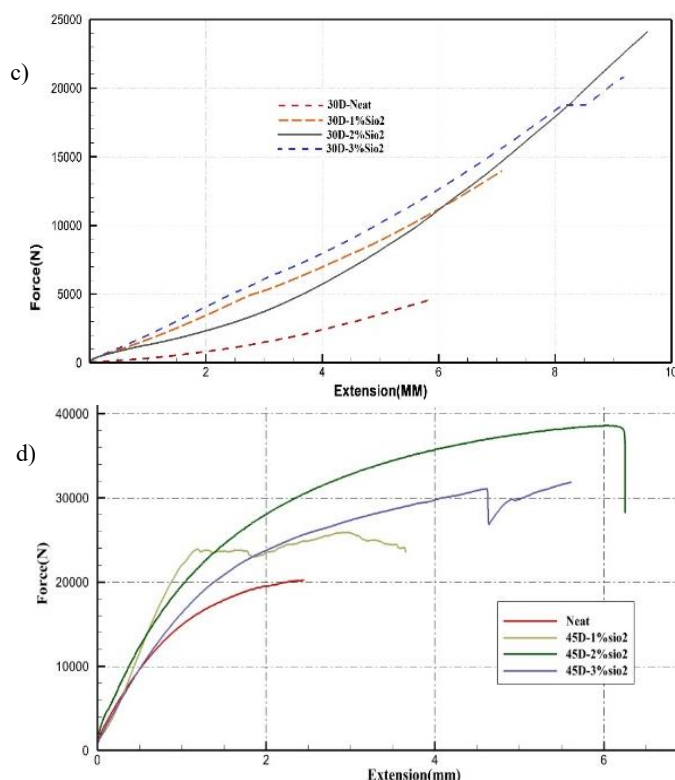


Figure 3. Tensile result of changing both nano SiO<sub>2</sub> content and glass fibre orientation: a) 0°; b) 15°; c) 30°; d) 45°.

When nano SiO<sub>2</sub> is added up to 2 %, both the elastic strain and elastic modulus exhibit improvement compared to the UTS at 3 % nano SiO<sub>2</sub> content, where the UTS relatively decreases. However, the plastic and elastic strain improve, suggesting that nano SiO<sub>2</sub> at this content level can be advantageous in enhancing plastic and elastic strain. At lower nano SiO<sub>2</sub> contents, it helps the fibres resist more and maintain the structure against unexpected failure at lower strains. However, as the nano SiO<sub>2</sub> content increases, the structure becomes more brittle and challenging to handle. The orientation of fibres at 0°, along with the desired nano content, has a significant impact on UTS of the composite structure. The change in material orientation from 0 to 45° can be a critical factor affecting mechanical behaviour of the materials. For example, when the fibre orientation is aligned with the axial load, the fibres and nano particles effectively resist and maintain their positions. However, when considering shear stress, the orientation of laminates can play a significant role in either improving or decreasing various factors. By increasing the nano particle content and adjusting the fibre orientation, the behaviour of composite materials can transition between rigid and flexible structures. The morphology analysis reveals the significant role of nano particles in reducing micro-cracks caused by tension and external stresses. Additionally, the 45° fibre orientation exhibits improved mechanical performance in various aspects, including shear stress and strain, elastic and plastic modulus, and elastic strain. The investigation of inclined sections and the consideration of desired stresses, such as shear and axial stresses in different directions, leads to the conclusion that a 2 wt.% SiO<sub>2</sub> content with 45° fibre orientation demonstrates superior behaviour in terms of axial and shear strain and stress, as

well as maintaining structural integrity. However, the evaluation of UTS reveals variations in different scenarios, highlighting the need for further exploration and analysis. In terms of UTS, it is observed that 2 wt.% SiO<sub>2</sub> with a 45° fibre orientation exhibits favourable behaviour in terms of axial and shear strain and stress. However, it should be noted that achieving the highest UTS values may not necessarily be associated with this specific combination. The mechanical behaviour of composite materials is influenced by a multitude of factors, and finding the right balance between different properties is crucial for optimal performance. Further examination of mechanical properties of composites highlights the crucial significance of the interaction between nano particles and fibres in determining the overall behaviour of the material. The incorporation of nano particles significantly enhances the material's resistance to micro-cracks, resulting in improved strength and durability. It is worth noting that the selected fibre orientation, particularly at 45°, facilitates a favourable distribution of stresses and strains across the composite structure. The chosen fibre orientation, particularly at 45°, facilitates efficient load transfer and the dissipation of stresses, leading to improved mechanical performance in both axial and shear conditions. However, it is important to consider the trade-off between UTS and other mechanical properties. While a specific combination of nano SiO<sub>2</sub> content and fibre orientation may excel in certain aspects, it may not be the optimal choice for all applications. Engineers and designers must carefully assess the specific requirements of their intended applications and select the composite configuration that best optimises the desired mechanical properties. This ensures the composite meets the necessary performance criteria for intended use. In conclusion, the incorporation of nano SiO<sub>2</sub> in GFRP composites has shown promising results in enhancing various mechanical properties. The interaction between nano particles and fibres, along with the chosen fibre orientation, significantly influences the material's behaviour under different loading conditions. Engineers can achieve composite materials with improved resistance to micro-cracks, higher strength, and enhanced mechanical performance by carefully tailoring the nano SiO<sub>2</sub> content and fibre orientation. This leads to the development of more robust and efficient structures for a wide range of applications. Future research in this field should focus on gaining a deeper understanding of the mechanisms underlying the interaction between nano particles and fibres. Exploring novel approaches to optimise the mechanical properties of composite materials will also be valuable. These advancements will contribute to the progress of composite technology, unlocking new possibilities for their utilisation in various engineering applications, including aerospace, automotive, and civil infrastructure.

#### Creep test

##### Effect of temperature

Creep is a time-dependent deformation that occurs in materials when subjected to constant load and temperature. The creep test is specifically designed to measure dimensional changes that occur over time under a constant static load. Conversely, the creep rupture test measures the time it takes for the material to fracture or break under the constant load

condition. In this study, the material is subjected to tension load to apply a constant load. To quantify the deformations, all strains are expressed as a percentage of displacement. This standardised approach allows for consistent comparison of results across different specimens. Measurements of time during the tests are recorded in seconds, reflecting the duration of creep and creep rupture processes.

#### Effect of force

According to the ASTM-D2990 standard, the creep test applied load must be lower than yield stress. The outcome of the test shows that the creep strain increases with time and stress. This is parallel to the effect of temperature on creep behaviour of composite materials. The method of fitting creep results is polynomial; because of the tremendous number of strain and time the result is not clear, and polynomial fitting is used in Tec plot software to change from line segment to polynomial. The effect of creep force and creep rate is significant for the optimum result which is considered as 45 ° and 110 °C. In Fig. 4, it is observed that as the applied force increases, the creep rate also increases. This configuration demonstrates the best creep performance, which is crucial for applications where resistance to time-dependent deformation is of utmost importance.

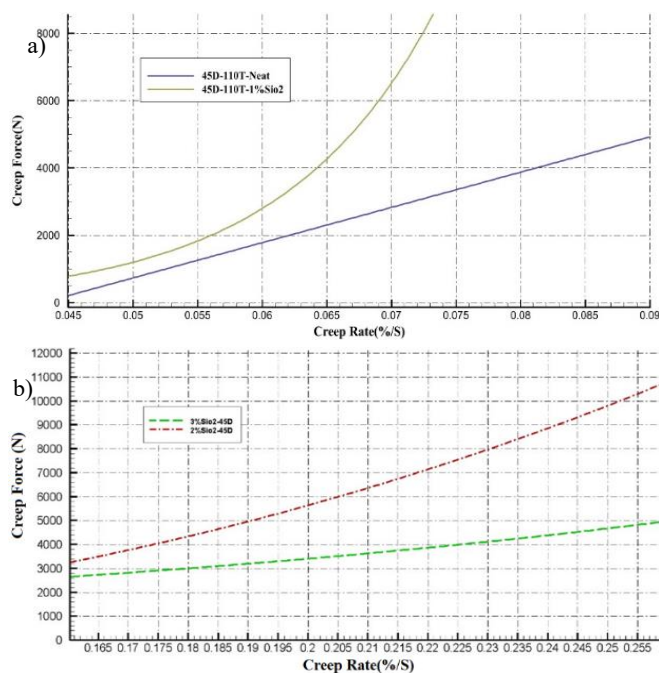


Figure 4. Effect of force on creep rate: a) creep rate of neat and 1 % SiO<sub>2</sub>; b) effect of nano SiO<sub>2</sub> on creep force and creep rate.

#### Effect of 0° and different nanoparticles on creep results

As mentioned previously, the creep test is time-dependent and reveals the essential behaviour of materials under constant load and elevated temperatures, as illustrated in Fig. 5. In initial stages, plastic and elastic strains are negligible. However, as the test progresses, the secondary stage merges with the primary stage, leading to an overlaying effect. This phenomenon can be attributed to the unique nature GFRP and nano SiO<sub>2</sub> as reinforcements in the matrices. As the test approaches its later stages, it eventually culminates in tertiary fracture and failure of the nano composite, as depicted in Fig. 6. By comparing the results, we observe that besides

temperature, nano SiO<sub>2</sub> plays a crucial role in altering the material's mechanical behaviour. The nature of these nano particles is to provide resistance and structural reinforcement against failure. By effectively curing the cracks, they assist glass fibres in withstanding and preventing failure. From Fig. 5a, we deduce that for neat materials (without nano particles), the material's behaviour is characterised by strain rate dependency on time and temperature. In particular, for a given time of 3600 s, the strain rate in materials at 80 °C shows a relative improvement compared to 50 °C. At 50 °C, the material experiences unexpected failure right from the start of the test. This failure can be attributed to combined effects of temperature and constant load tension which results in the presence of micro-cracks in the composite. The lack of sufficient curing agents at the beginning of the creep test contributes to the drastic failure of the composite. However, with the incorporation of nano SiO<sub>2</sub> particles, as shown in Fig. 5- b, c, and d, the material's behaviour significantly improves. The presence of nano SiO<sub>2</sub> particles enhances the material's resistance to micro-cracks and improves its overall strength and durability. This is evident from lower strain rates observed at the same time intervals and temperatures compared to neat material. The nano SiO<sub>2</sub> particles effectively fill and heal the micro-cracks, preventing their propagation and reducing the likelihood of failure. In conclusion, the addition of nano SiO<sub>2</sub> particles in GFRP composites significantly improves their mechanical behaviour, especially in terms of creep resistance. Examining the creep test curve at 80 °C reveals that increasing temperature to 80 °C under a constant load causes the composite structure to shift along the axial vector. This shift indicates the deformation and elongation of the material over time. At lower temperatures, there are porous gaps between the epoxy and fibres, which compromise the durability of the composite structure. These gaps allow for the initiation and propagation of micro-cracks, leading to potential failure. By increasing to 80 °C, the thermal expansion of the material helps to fill these porous gaps, improving the overall integrity of the composite structure. The increased temperature also enhances the mobility of molecules, allowing for better bonding and reducing the likelihood of crack propagation. This results in improved creep resistance and better long-term performance of the material under sustained loads. It is worth noting that the specific behaviour of the nano composite at 80 °C may differ from other temperature conditions. At lower temperatures, results indicate inability to retain its position and exhibit higher elastic strain. As the temperature increases by 30 °C and reaches 110 °C, the composite structure displays a better elastic response. Creep rupture occurs at a higher state of strain. This behaviour is attributed to the nature of thermoset materials. At higher temperatures, the material acts as a healing agent, covering porous gaps and providing more strength to the creep strain. As a result, the structure is able to withstand higher strains and resist failure at lower strain levels. On the contrary, at 140 °C, the temperature brings about a complete transformation in the behaviour of the composite. With another increase of 30 °C, the composite structure weakens, leading to an increase in micro-cracks and porous spaces between the epoxy and glass fibres. This drastic

change results in unexpected failure at lower strain levels and a decrease in elastic strain. Suddenly, the plastic strain changes to tertiary fracture, indicating severe failure. This observation highlights the critical role that temperature plays in the creep behaviour of composite materials. It not only affects the healing and bonding properties of the material but also impacts its overall strength and resistance to failure. Results show that there is an optimum temperature range in which the composite demonstrates improved resistance to creep and better mechanical performance. If the temperature goes beyond this range, the material's properties are negatively affected, resulting in a decrease in structural integrity and increased risk of failure. The addition of nano SiO<sub>2</sub> in concentrations ranging from 1 wt.% to 3 wt.% significantly increases the strain rate. Among these concentrations, the addition of 1 wt.% SiO<sub>2</sub> shows the most substantial increase in strain rate compared to the neat test results. Increasing the nano SiO<sub>2</sub> content to 2 wt.% results in a significant improvement in total strain. Interestingly, there is a reverse relationship observed between strain rate and percentage of strain due to the percentage of total creep strain, as depicted in Fig. 6.

SEM micrograph results reveal a change in material behaviour with increasing nano particle content, Fig. 8. At higher nano SiO<sub>2</sub> content (3 wt.%), agglomeration of nano particles within the epoxy is observed. This agglomeration has an adverse effect on mechanical properties of the composite, leading to weaknesses in its overall performance.

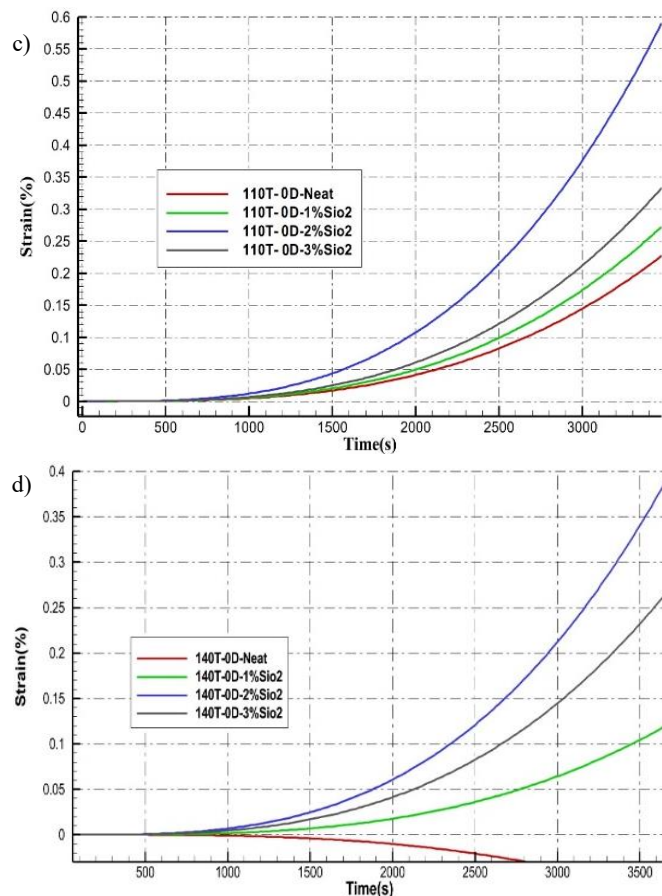
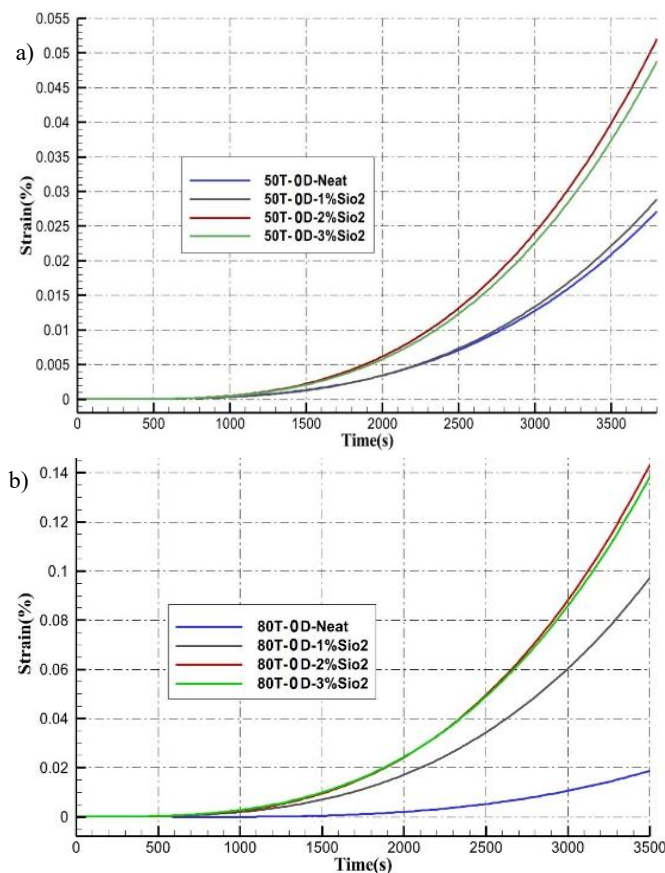


Figure 5. Results of creep strain and time under 50-140 °C and nano SiO<sub>2</sub> from neat-3 wt. % in 0° of fibres: a) neat; b) 1 wt. %; c) 2 wt. %; d) 3 wt. %.







Figure 6. Creep test leading to failure.

The addition of nano SiO<sub>2</sub> to the composite structure has a notable impact on its creep behaviour and mechanical features. While lower concentrations may lead to significant improvements in strain rate, higher concentrations could cause nano particle agglomeration, compromising the composite's mechanical properties. Striking the right balance between nano SiO<sub>2</sub> content and other factors is crucial for developing advanced composite materials with enhanced creep resistance and improved performance in various engineering applications. It is observed that for both 1 wt.% and 3 wt.% nano SiO<sub>2</sub> content at 110 °C and 140 °C, the results are relatively close. In the tertiary stage, the inclusion of nano particles enhances the material's rupture behaviour. Throughout the primary and secondary stages, there have been notable improvements observed over time, indicating increased resistance against fibre pull-out, breakage, and shear stress.

#### Effect of 15° and different nanoparticles on creep results

Analysing the creep results at a 15-degree orientation reveals significant influence of orientation on the material's creep behaviour and strain response. Increasing nano SiO<sub>2</sub> content to 2 % results in a noticeable change compared to the 0° orientation. The addition of nano content increases both creep behaviour and strain, suggesting a higher ratio of displacement to the actual dimension of creep specimens compared to neat specimens. The strain rate for 2 wt.% nano content exceeds that of 1 wt.% and 3 wt.% nano content, indicating enhanced mechanical behaviour, especially tensile behaviour. Caution is necessary when adding higher amounts of nano particles. Excessive aggregation within the matrix can occur, causing nanoparticles to clump together and form large fillers of around 70 µm. This aggregation limit must be carefully considered to avoid detrimental effects on the material's properties. As depicted in Fig. 7, creep behaviour is influenced primarily by two factors: temperature and UTS, with UTS assumed to be 0.4 % of the total UTS in this study. Above 50 °C, a noticeable shift in creep behaviour occurs. Deformation of composite samples becomes increasingly dependent on temperature, and after approximately 2000 s, most samples with higher nano contents transition from the elastic to plastic deformation. This duration of deformation is crucial when considering operational conditions and the lifespan of components. With higher nano content, deformation may be more pronounced, yet the structure remains resilient, showcasing the beneficial role of nanoparticles in maintaining the composite's structural integrity.

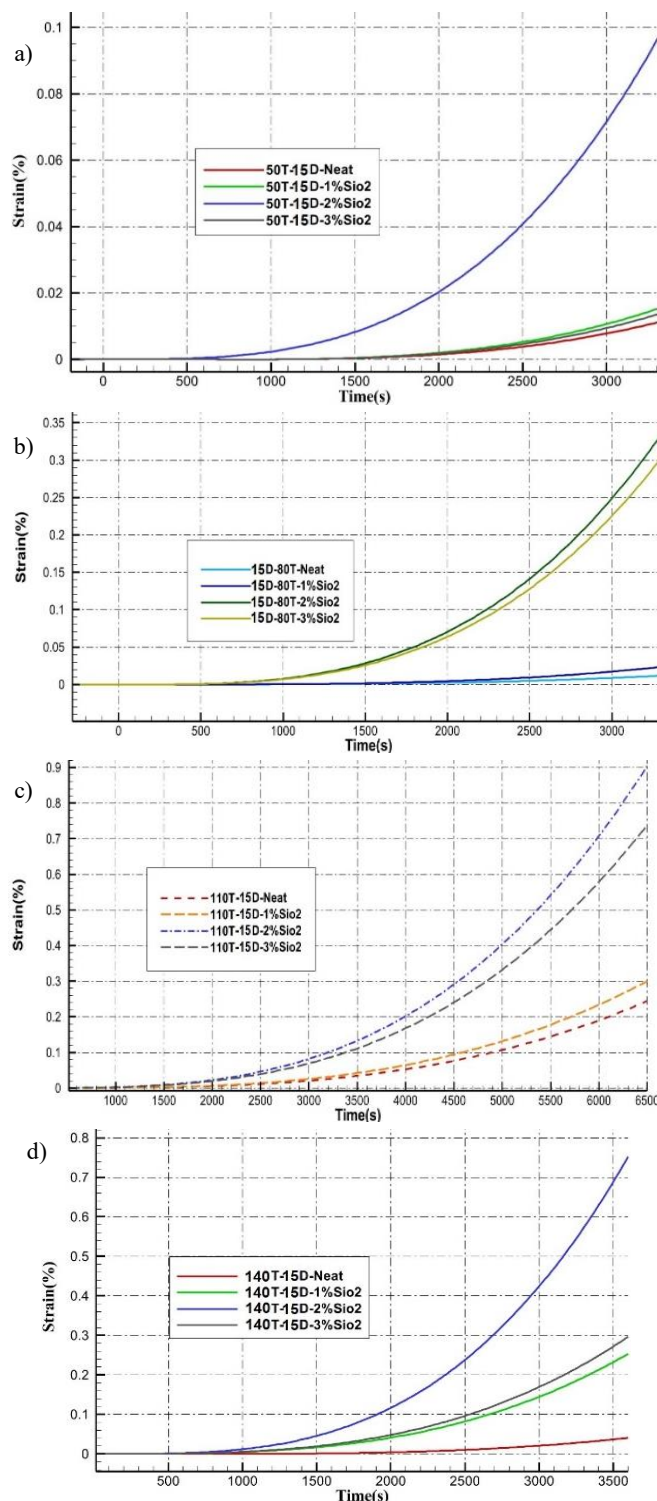


Figure 7. Results of creep strain and time from 50-140 °C and nano SiO<sub>2</sub> from neat-3 wt. % in 15° of fibre: a) 50 °C; b) 80 °C; c) 110 °C; d) 140 °C.

The highest recorded strain rate is observed in samples with 2 wt.% nano content, reaching 0.9 strain rate at 110 °C, demonstrating the positive influence of increased temperature on creep behaviour (Fig. 7). Across all four graphs in Fig. 7, it is apparent that deformation and creep fracture or failure depend on nano content. Lower nano content results in less dispersion of nano particles within the structure, limiting the advantage of crack-filling and leading to early composite

failure. When temperature is raised to 140 °C, creep behaviour decreases compared to 110 °C. This observation suggests that higher temperatures may adversely affect the nanostructure or diminish the activity and effectiveness of nano contents within the composite.

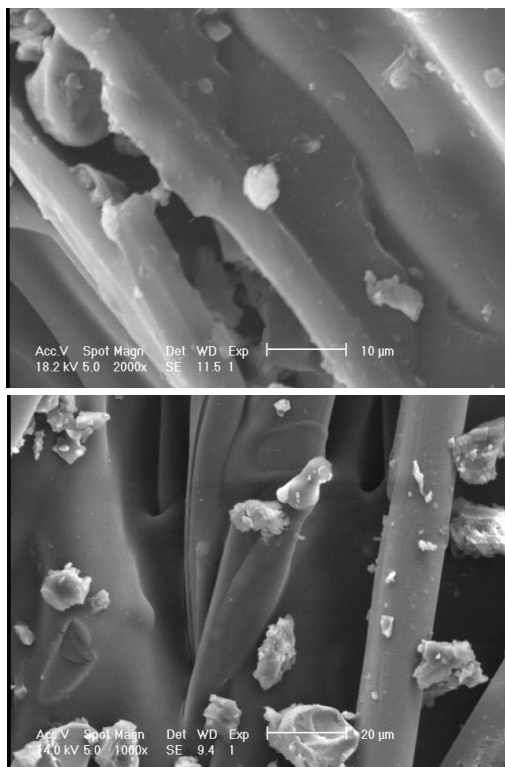


Figure 8. Aggregation and concentration of nanoparticles which influence and create concentrated tension.

#### Effect of 30° and different nanoparticles on creep results

As illustrated in Fig. 9, the increase in fibre orientation at this stage exerts a fascinating effect on creep behaviour, resulting in a decrease in strain rate compared to the 15° orientation. Particularly noteworthy is the significant creep rate observed at 0.25 for 1 wt.% nano SiO<sub>2</sub> content. At this juncture, the influence of temperature becomes highly significant. Additionally, it becomes apparent that incorporating nanoparticles at a 30° orientation negatively impacts creep rate, likely due to weakening of the chain-to-fibre interaction. The orientation vector of the fibre plays a crucial role in determining the creep rate and behaviour, as it can alter the tension tensor and ultimately lead to unexpected structural failures.

The detrimental effect of higher nano SiO<sub>2</sub> content is observed in this scenario. This observation is attributed to the aggregation of particles around the fibre, aiming to reinforce the structure. However, this aggregation leads to localised tension, as depicted in SEM images in Fig. 8. In instances of lower nano content, up to 1 wt.%, the distribution and filling of nanoparticles are superior compared to 3 wt.%. In these composites with lower content, the nanoparticles effectively fill and repair cracks without significantly weakening the structure. Conversely, in the case of 3 wt.% nano content, excessive nanoparticles tend to accumulate around cracks and voids, resulting in structural degradation. In the 1 wt.% nano content scenario, the nanoparticles are more evenly dis-

persed, preventing aggregation and localised tension. Consequently, the structure maintains its integrity at 110 °C, forming robust polymeric bonds, as previously discussed.

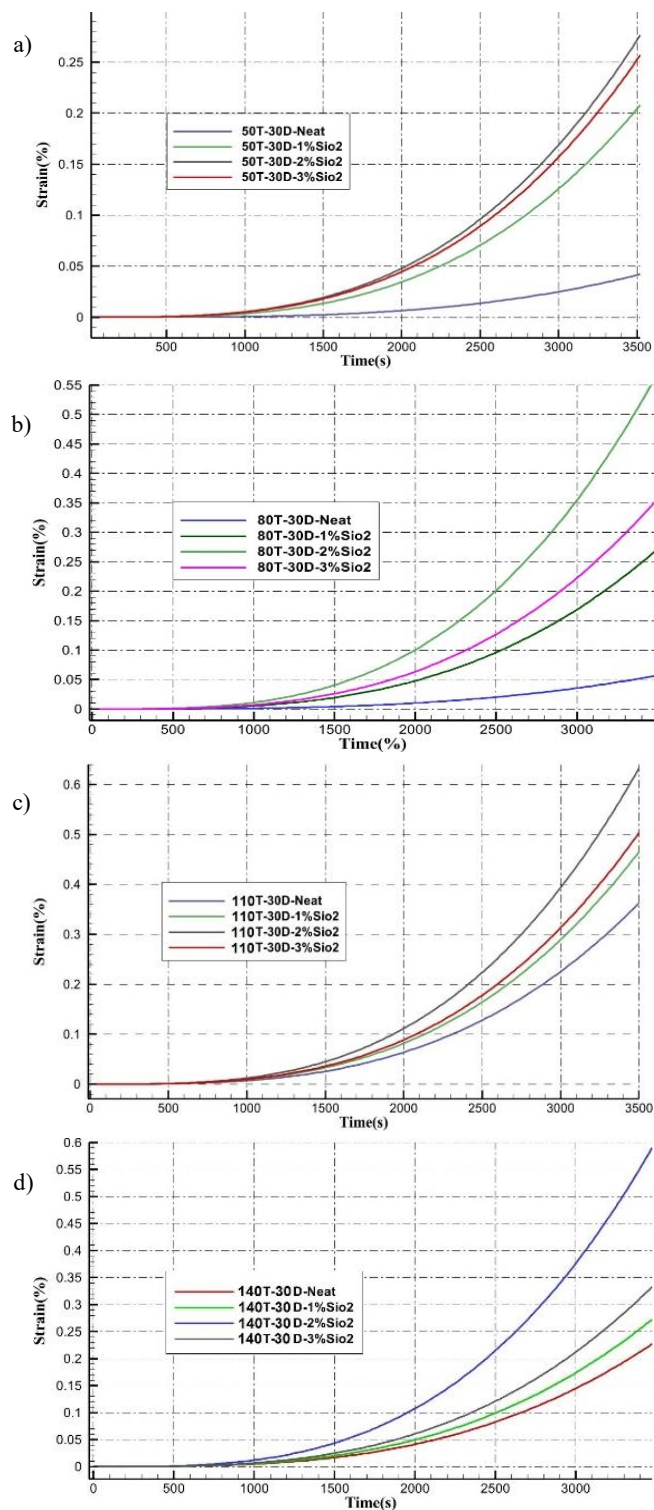


Figure 9. Results of creep strain and time from 50-140 °C and nano SiO<sub>2</sub> from neat-3 wt. % in 30° of fibre: a) 50 °C; b) 80 °C; c) 110 °C; d) 140 °C.

#### Effect of 45° and different nanoparticles on creep results

After a thorough examination of Fig. 10, several valuable and intriguing findings are incorporated into the current study. The observed creep rate varies considerably, ranging



from a lower rate of approximately 0.09 to a higher rate of nearly 1. Notably, most variations in strain rate are observed at 110 °C, highlighting the significant impact of temperature on creep behaviour. As temperature increases, creep rate decreases by up to 50 %. In this context, the critical role of nano particles emerges.

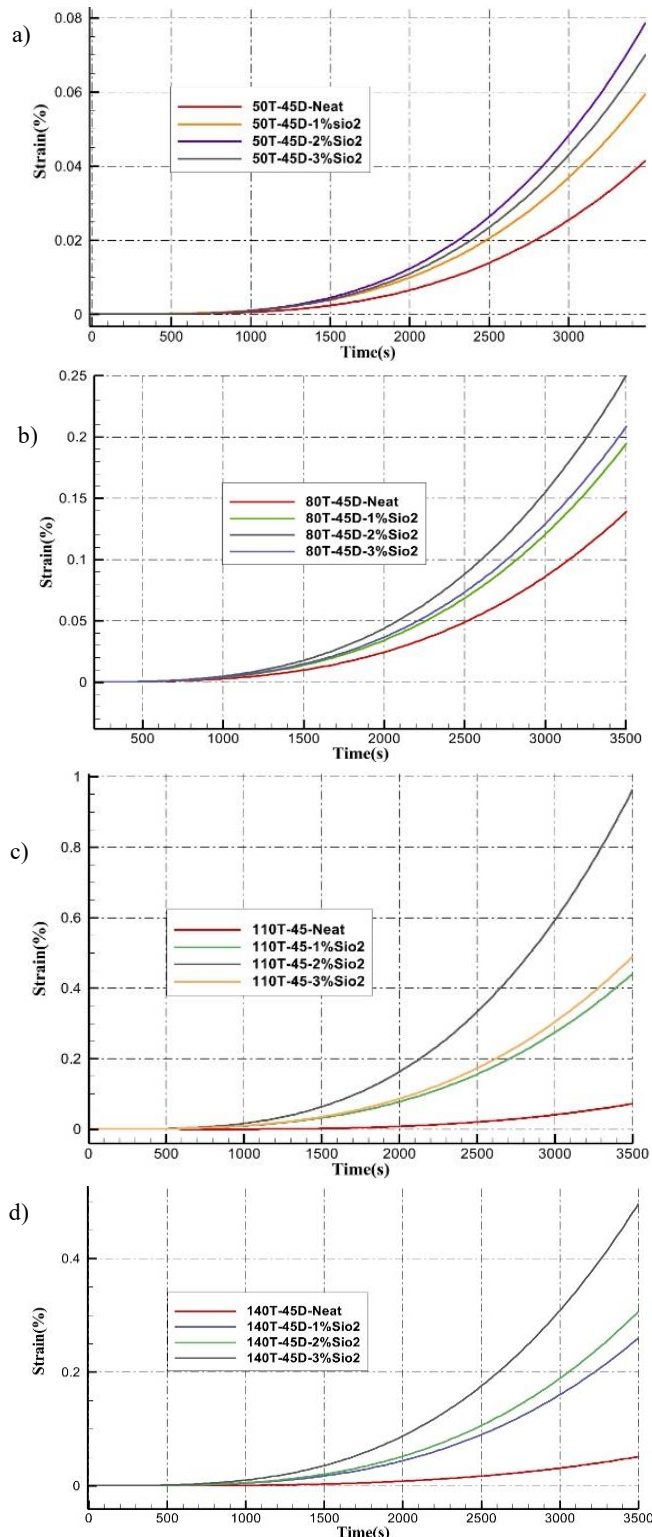
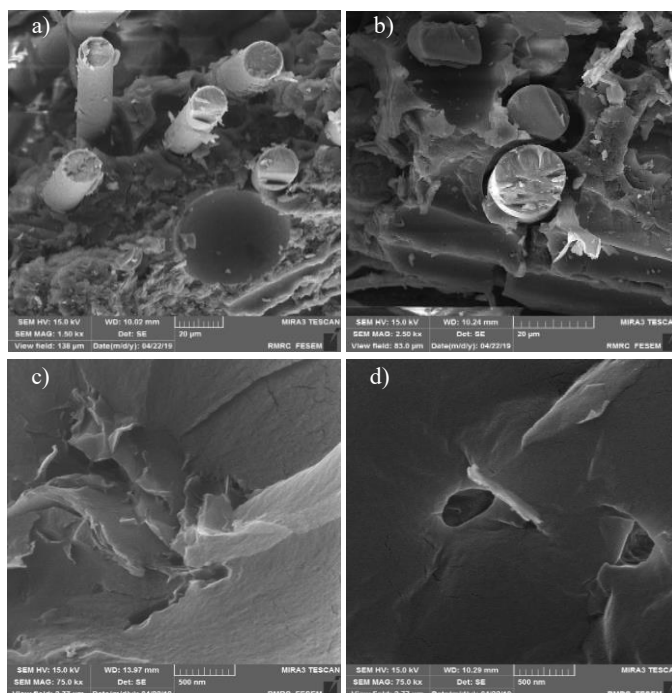


Figure 10. Results of creep strain and time from 50-140 °C and nano SiO<sub>2</sub> from neat-3 wt. % in 45° of fibre: a) 50 °C; b) 80 °C; c) 110 °C; d) 140 °C.

The addition of nano contents up to 2 wt.% results in a notable enhancement of mechanical behaviour and the complete elimination of aggregation, which had previously been a primary cause of unexpected failure. Instead, nano particles play a crucial role in healing and covering, effectively preserving structural integrity. The analogy of nano particles acting as pins in the chain structure of polymerisation underscores their pivotal role in maintaining structural cohesion and facilitating a higher creep rate. Temperature also emerges as an important factor. For instance, at 50 °C, the highest strain rate for neat samples is recorded as 0.09. As the temperature increases, the strain rate varies for composites with 1 to 2 wt.% nano particles, ranging from 0.025 to 0.08 at 80 °C. At 80 °C, the highest strain rates are attributed to 2 and 3 wt.% nano content, reaching 0.2 and 0.255, respectively. With a further temperature increase of 30 to 110 °C, the highest strain rate of 1.0 % is recorded for the 2 wt.% nano content. From these observations, it can be concluded that, in some cases, higher nano particle content can enhance creep behaviour due to their healing role in filling and covering cracks. However, aggregation may still occur at higher nano contents, leading to adverse effects on material properties. This comprehensive analysis underscores the intricate interplay between nano particles, temperature, and strain rate in shaping the creep behaviour of composite materials.

#### Study of SEM figures on creep results

As depicted in Fig. 11 a and b, the fibre orientation has a profound impact on the tension tensor, resulting in variations in creep behaviour. Presence and arrangement of polymerisation bonds within the composite are deeply influenced by both the nano particle contents and fibre orientations. By analysing Figs. 11(a-h), it becomes evident that the proper dispersion of nano particles plays a critical role in preventing tension concentration and promoting structural integrity. In these figures, nano particles are represented by bright dots, and their distribution is notably visible in areas where cracks,



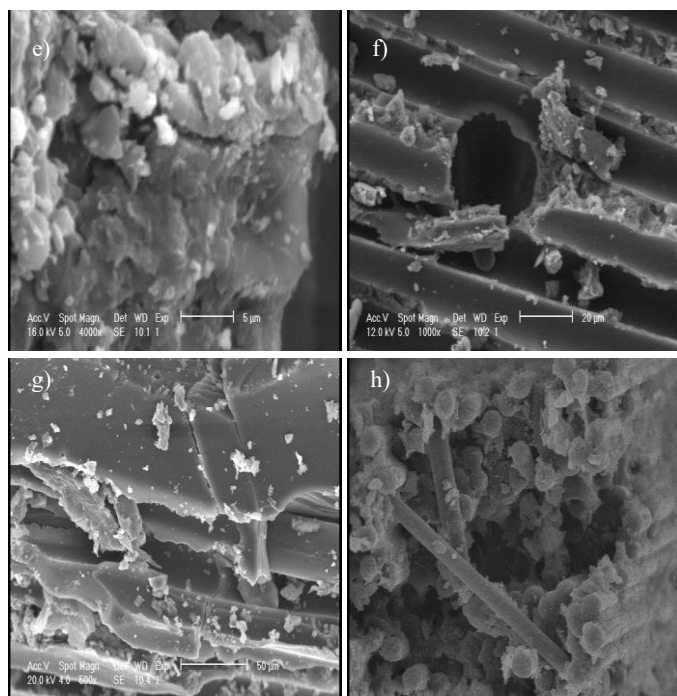


Figure 11. SEM shots of creep samples after creep test.

holes, or inequalities are observed. These nano particles actively work to cover and heal these gaps, effectively strengthening the structure. Well-dispersed nanoparticles act as a reinforcement mechanism, reinforcing the matrix and enhancing the overall mechanical properties of the composite material. The interplay between fibre orientation, nano particle contents, and their effective dispersion plays a vital role in determining creep behaviour and mechanical performance of the composite. Properly dispersed nano particles can significantly improve composite structural integrity and resistance to tension concentration, making them crucial components for designing high-performance composite materials in various engineering applications.

## CONCLUSION

Results of the current study demonstrate that incorporation of nanoparticles plays a crucial role in determining the behaviour of composite structures. This behaviour is influenced by various factors, including fibre orientation, presence of cracks, UTS, and temperature.

The highest strain values are observed at 110 °C, with a maximum recording of 0.09 for both 45° and 15° fibre orientations. On the other hand, the 0° and 30° orientations exhibit the least strain values. The effect of nanoparticles varies with different orientations. For 0° and 30° orientations, adding nanoparticles up to 2 wt.% increases the strain rate, indicating improved mechanical behaviour. The situation is different for 15° and 45° orientations. For the 15° orientation, the addition of nanoparticles up to 1 wt.% is significant in enhancing the strain rate. However, further increasing the nanoparticle content leads to decreased strain rates and weakens the structure due to aggregation and tension concentration. In contrast, for 45° orientation, adding nanoparticles up to 2 wt.% has a significant positive effect. However, adding more nanoparticles results in a decrease in

strain rate and eventually leads to typical fracture behaviour. These findings highlight the importance of carefully optimising the nanoparticle content and considering the specific fibre orientation to achieve desired mechanical properties and creep behaviour in composite materials. By understanding and controlling these factors, engineers can design composite materials with improved performance and reliability for various engineering applications at different temperature and loading conditions.

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