

EVALUATION OF MECHANICAL PROPERTIES OF PRE-LOADED GLASS FIBER/EPOXY COMPOSITE REINFORCED WITH TiO_2 NANOPARTICLES

Hawraa Ali Nasir¹
eng954.hawraa.ali@student.uobabylon.edu.iq

Mustafa Baqir Hunain²
dr_m_hunain@yahoo.com

^{1,2}Mechanical Engineering Department / Faculty of Engineering / University of Babylon/Babylon/Iraq.

ABSTRACT

This research investigates the combined effects of biaxial preloading and the addition of titanium dioxide (TiO_2) nanoparticles (particle size 50nm) on the mechanical characteristic of glass fiber-reinforced epoxy composites. Specimens were prepared using different weight fraction of TiO_2 (0, 1, 3, and 5%), with different preload levels (0, 400, 800, and 1200 N). The mechanical performance was evaluated through flexural, tensile, and impact tests. The results showed that the best enhancement in mechanical properties were attained at 3% weight percentage of TiO_2 with a preload of 800 N, with tensile strength increasing by 75.82%, impact strength by 127.14%, and flexural stress by 425.87% compared to the unloaded and unreinforced sample. In contrast, a performance decline was observed when the weight fraction increased to 5% with a high preload (1200 N), attributed to particle agglomeration and internal damage to the composite structure.

Keywords: Glass fiber epoxy composite, TiO_2 nanoparticles, Preload, Mechanical properties.

NOMENCLATURE

GFRP	glass fiber reinforced polymer.
FRPC	fiber-reinforced polymer composite.
TiO_2	titanium dioxide.
MgO	magnesium oxide.
ANOVA	analysis of variance.

INTRODUCTION

A composite consists of two components: a continuous matrix and discontinuous reinforcement. Glass fiber-reinforced polymer composites are well-known for their lightweight design, corrosion resistance, high strength, excellent mechanical properties, minimal thermal expansion and durability. Nano fillers have the benefit of requiring less fillers to attain certain multifunctional qualities as compared to standard microscale fillers.

Combining glass fiber reinforced polymer (GFRP) composites with Nano fillers improves their behavior dramatically, especially in structural applications (Somaiah, A.et al., 2024). Nanoparticles were used to increase the performance and characteristics of Fiber-reinforced polymer composite (FRPCs) and were identified as a promising approach. A wide variety of nanoparticles, including silica, clay, calcium carbonate, nickel oxide, layered silicate, titanium dioxide, and others, have received special attention for inorganic/organic nanocomposites (Kumar, A.et al.,2021). Pre-stressing of fibers through the curing stage of composite materials is a technique for improving mechanical performance. During this process, the fibers are subjected to tensile load while the resin cures, resulting in internal tension. After curing is complete and the external force is removed, the fibers maintain this tension within the matrix. This causes compressive stresses in the composite structure as the fibers strive to return to their original unstressed state (Nacy, S.M.and Yaser, A.S.,2008).

Many researchers have examined the influence of nanoparticle addition(TiO₂) and the application of prestress on enhancing the mechanical behavior of composite materials. (AL-Ajaj, I.A.et al.,2013) investigated the mechanical characteristics of epoxy resin reinforced with micro particles TiO₂ (50- μ m) and nanoparticles TiO₂ (50-nm) with epoxy volume fractions (1, 2, 3, 4, 5, 7, 10, 15 and 20 %). The results showed that the young's modulus, fracture toughness, and flexural strength of nanocomposite improve when volume fraction is below 7%. However, at elevated volume fractions, both Flexural Strength and Fracture Toughness decrease, while Young's Modulus continues to increase than that of epoxy. An increase in the volume fraction of TiO₂ micro particles leads to a decrease in both flexural strength and fracture toughness, especially at high volume fraction. In contrast, the young modulus improves as volume fraction increases. (Somaiah,A.et al., 2024) studied the influence of TiO₂ and MgO Nanoparticles and fiber stacking sequence on the mechanical properties of (GFRP) composites. The results showed that the addition of nanoparticles improves the ultimate tensile strength and flexural strength by 31.96 and 68.43%. Analysis of variance (ANOVA) revealed that the fiber stacking sequence was the most influential factor on ultimate tensile strength (63.65%) and flexural strength (65.70%), whereas nanoparticles played a major role in enhancing composite hardness by 39.22%. (Kumar, A.et al.,2021) investigated the influence of varying weight fractions (0.5, 1, 1.5, and 2%) of titanium dioxide nanoparticles on glass fiber reinforced epoxy composites. on the mechanical and thermal properties. The results showed that the increase in tensile strength (38.56%), bending strength (30.52%), inter-laminar shear stress (25.22%), impact strength (327.10%), micro-hardness (48.53%), and the energy of fracture (40.19%), with a minimum (GFRP-T1.0) laminates had a lower toughness than (GFRP-T0.0) laminates. In comparison to (GFRP-T0.0) composite laminates, (GFRP-T1.5) enhanced stiffness and rigidity by 52.72% and 34.13%, respectively. (Merad, L.et al.,2011) conducted a study to evaluate the effect of titanium dioxide nanoparticles on the mechanical properties of epoxy resin. Particles with a diameter of 21 nanometers were used at volume fractions of 0.5%, 1.5%, and 2.0%. The results showed a significant improvement in mechanical properties,

including hardness and tensile strength, compared to unreinforced epoxy. These properties gradually increased with increasing nanoparticle TiO_2 content. (Al-Dulaimy, A.K.A. et al., 2020) examined the influence of unidirectional pre-loading at levels of (5, 8 and 10 N) on tensile characteristics of an epoxy reinforced with E-glass fibers at fiber volume fraction of 10%. The results showed that the applying of preload improved the maximum strength, percentage elongation, and rupture strength raised by 38.5, 45.57, and 106.2%, respectively, by comparing them with control samples. The optimum pre-loading level is 10N. (Nishi Y. et al., 2014) investigated the influence of pre-stressing on the Charpy impact strength of epoxy composite reinforced with unidirectional glass fibers at 50% fiber volume fraction. The results demonstrated that applying a pre-stress of 17.6 MPa led to 30% improve in Charpy impact strength. (Mostafa, N.H. et al., 2015) improved the flexural performance of woven E-glass /polyester reinforced composites. They studied the effect of equi-biaxial fabric pre-stressing on flexural characteristic. The findings revealed that the flexural characteristics improved with raised the pre-stressing. The optimum equi-biaxial fabric pre-stress level is 50Mpa. They found that the level of improvement in flexural properties decreased with increasing fabric orientation angle. (Mohmoud, M. et al., 2020) used unidirectional glass fiber reinforced with epoxy resin manufactured under vacuum conditions. Flexural, compression, and tensile tests were performed to identify the influence of pre-stress on mechanical properties, fiber waviness, and fiber volume fraction. The findings revealed an obvious enhancement in mechanical properties when compared to control samples of composites.

Many researchers have investigated the effect of adding particles to polymer matrices, but there is a lack of scientific literature on the effect of adding titanium dioxide (TiO_2) nanoparticles to glass fiber-reinforced epoxy resins, especially when combined with fiber preloading. Therefore, this work aims to investigate the effect of different weight fraction of TiO_2 nanoparticles (0, 1, 3, and 5wt.%), as well as the effect of fiber preloading at different levels (0, 400, 800 and 1200N) on the mechanical characteristics of the composite material, including tensile, impact, and flexural tests.

THE EXPERIMENTAL PROCEDURE

Materials

The materials utilized in the current experimental work include bidirectional woven glass fiber. The glass fiber had a density (2.58g/cm^3), tensile strength (3.45GPa), Young's modulus (72GPa), and Poisson's ratio of 0.22 (Skia Warp, 2020). Epoxy resin (Quick mast 105), produced by DCP company was used as a matrix material with suitable hardener. The mixing ratio of the epoxy and hardener of 4:1 by weight. Its properties include a tensile strength of $\geq 25\text{MPa}$, flexural strength of $\geq 50\text{MPa}$, compressive strength of $\geq 70\text{MPa}$, density 1.1g/cm^3 , and viscosity of 2-5 poise (DCP International). Titanium dioxide (TiO_2) nanoparticles were used as a reinforcement material, with a purity of 99%, an average

particle size of 50 nm, a specific surface area of $210 \pm 10 \text{ m}^2/\text{g}$, and a bulk density of 0.25 g/cm^3 (HWNANO, Product information page).

Mold Design and Fiber Pre-Tensioning

To ensure uniform distribution and controlled pre-tensioning of the glass fibers before composite fabrication, a specially designed steel mold was used with dimensions of $50 \times 50 \text{ cm}$, as shown in Figure 1a. The mold consists of two main sections: a fixed part secured with bolts, and a movable part operated using a screw-nut mechanism. By rotating the screw, tension is gradually applied to the fiber layers. A digital scale connected to the mold is used to monitor the applied load accurately, ensuring consistent fiber tension during preparation. After the tensioning process, the fiber layers were transferred to a secondary steel mold with dimensions of $20 \times 30 \text{ cm}$, shown in Figure 1b, which was used for casting the final composite plates.

Sample preparation

Samples were prepared under controlled laboratory conditions using fiber-reinforced composite materials, with the aim of studying the effect of both preloading and nanoparticle reinforcement on the mechanical properties. The following steps explain the preparing process:

1. Samples were prepared by tensioning bidirectional glass fibers inside a mold using a mechanical tensioning system, with four levels of applied load (0, 400, 800, and 1200 N).
2. The nanocomposite specimens were fabricated by adding titanium dioxide (TiO₂) nanoparticles into the epoxy resin at weight fractions of 1%, 3%, and 5%, respectively.
3. Initially, both titanium dioxide (TiO₂) and epoxy resin were accurately weighted using a digital scale, and then mechanically mixed for 20 minutes at room temperature to prepare the desired composite material.
4. The mixture was placed in an ultrasonic bath as an additional dispersion step to ensure uniform distribution and reduce agglomeration of titanium dioxide nanoparticles within the epoxy matrix.
5. After completing the ultrasonic bath process, the hardener was added to the epoxy-TiO₂ mixture at a weight ratio of 4:1 (epoxy to hardener) and manually stirred for 60 seconds to ensure uniform mixing.
6. For pure specimens, the epoxy resin and hardener were combined immediately at a weight ratio of 4:1 for 60 second at room temperature.
7. The prepared mixture was poured into the mold over the pre-tensioned fibers and allowed to cure at ambient temperature for one day.
8. The laminates were removed from the mold and left in an oven at 70°C for 3 hours in order to attained adequate curing (AL-Sukri, H.M.H.,2007).

9. Finally, the specimens were cut according to the standard of each test using a reciprocating saw; they were left for 3 weeks before testing.
10. Two types of samples were fabricated: one without nanoparticle reinforcement, and the other reinforced with titanium dioxide (TiO₂) nanoparticles.

MECHANICAL TESTS

Tensile test

Tensile testing is one of the common methods used to evaluate the mechanical properties of fiber-reinforced composite materials. The tensile test was conducted in compliance with the ASTM (D3039) standard (ASTM, ASTM D3039, 2014), the tensile specimens were prepared in a rectangular shape with standard dimensions of 250 mm in length and 25 mm in width. The tensile tests were performed using a Universal Testing Machine (WDW-200E) at room temperature, with a constant crosshead speed of 2 mm/min. Each test was repeated three times for every sample, and the average value was reported. The specimens used in this test are shown in Figure 2.

Impact test

The impact test was carried out by using a charpy impact tester (Gunt Hamburg Co./ WP400). The test sample was prepared as per ISO179 (ISO, ISO179-1, 2010) standard, with standard dimensions of 55 mm in length and 10 mm in width. The average of three samples was considered. The samples of this test can be shown in figure 3. The impact strength in kJ/m^2 was calculated as per equation 1 (Karim, A.A. et al., 2018).

$$G_c = \frac{U_c}{A} \dots\dots\dots (1)$$

Where G_c : impact strength (KJ/m^2)

U_c : fracture impact energy (J)

A: cross section area of specimen (m^2)

Flexural test

The flexural testing was performed by using Universal Testing Machine Type (WDW-5E). The test specimens were prepared in accordance with ASTM D790 (ASTM INTERNATIONAL, D0790, 2002), with standard dimensions of 128 mm in length and 12.7 mm in width. A three-point bending test was undertaken at a cross head speed of (1mm/min) at room temperature and flexural modulus, specimens' bending stress before breaking at maximum load were assessed. Each test was repeated three times for every sample, and the mean value was reported. The specimens used in this test are shown in Figure 4.

RESULTS AND DISCUSSION

Tensile test

Identifying the tensile characteristics is essential when designing and developing new composite materials. The overall behavior of the stress-strain curves of the composite material was analyzed by addition titanium dioxide (TiO₂) nanoparticles at different weight percentages (0, 1, 3, and 5%) to the glass fiber-reinforced epoxy resin, under different levels of fiber preloading, as shown in figure 5 (a-d). These curves show that the relationship between stress and strain was nonlinear in all the studied samples. The stress increased progressively with increasing strain. Maximum tensile strength indicates a material's ability to resist failure. Figure 6 shows the relationship between maximum tensile strength and preload. From this figure, it can be noticed that when applying preload of (800N) and adding TiO₂ nanoparticles with (3wt.%) to the composite material led to an increase in the tensile strength, reaching a maximum value of 235.6MPa. This improvement may be attributed to the addition of fine, well-distributed nanoparticles alongside the glass fibers, which contribute to enhancing the interfacial bonding between the matrix and fibers. This improved bonding enables loads and stresses to be transferred more efficiently from the matrix to the reinforcement, which positively affects the mechanical characteristics of the composite. In contrast, preloading generates internal compressive stresses within the composite material, increasing the energy required for crack initiation and propagation. Preloading also reduces fiber waviness, improving the efficiency of load transfer between the fibers and matrix, enhancing the composite's ability to resist failure under load. The combined influence of nanoparticle reinforcement and fiber preloading contributes significantly in improving the overall mechanical characteristics of the composite. Increasing the titanium dioxide (TiO₂) nanoparticle content to 5wt.%, combined with applying a high preload (1200 N), caused a reduction in tensile strength. This decrease is attributed to several factors, including fiber damage or matrix deformation resulting from excessive loading, which reduces the capability of the material to resist mechanical load. Additionally, the agglomeration of TiO₂ particles at this high ratio leads to weak bonding at the interface between the filler and matrix, reducing stress transfer efficiency within the composite and negatively impacting its mechanical performance.

Impact test

Impact test is a measure of the resistance of material when an impact loading is applied. Figure 7 represents the effect of adding different weight fractions of TiO₂ nanoparticles and applying different level of preload on the impact strength of the composite samples. It has been noted that the impact strength rises with increasing both the ratio of nanomaterials and preload, reaching its highest value at 3 wt.%TiO₂ and a preload of 800N. This improvement is attributed to the effect of two main factors. First, the homogeneous distribution of TiO₂ particles within the matrix, which contributed to enhancing the interfacial bonding between the fibers and the matrix. TiO₂ nanoparticles inhibit crack

propagation within the matrix through mechanisms such as crack deflection, stabilization, and energy dissipation. When an external load is applied, the presence of these nanoparticles introduces additional resistance to crack growth, thereby increasing the amount of energy the material can absorb before failure. As a result, the composite exhibits enhanced stiffness and toughness compared to unfilled materials. Second, the fiber preloading reduces fiber waviness within the composite structure, enhancing the efficiency of load transfer from matrix to fiber, thus increasing its shock resistance. It was observed that the tensile strength decreased with the increase of titanium dioxide (TiO_2) particle content to 5 wt.%, along with the application of a high preload of 1200 N. This decline in performance can be attributed to excessive preloading, this causes permanent damage to the bonds between the fibers and the matrix, and agglomeration of TiO_2 nanoparticle, both of which reducing energy absorption efficiency and thus reducing the material's ability to resist impact.

Flexural test

Figure 8 illustrate the flexural stress performance of epoxy composites reinforced with glass fibers and varying weight fractions of TiO_2 nanoparticles under different preload levels. This figure shows that the flexural stress increases with increased weight fraction of TiO_2 nanoparticles and preload level, reaching a maximum value at 3 wt.% of TiO_2 and preload of 800N. This increment may be due to the properties of nanoparticles such as high rigidity, high specific surface area and good interfacial bonding with fiber and matrix. In addition, preloading plays an important role in enhancing flexural stress, as it induces residual stress in the fabric, generating a force component perpendicular to the direction of the applied bending forces. This component increases with increasing deflection of the specimen during bending, enhancing the material's resistance to bending. Preloading also contributes to the straightening of fibers within the composite structure, improving the efficiency of load transfer from the matrix to the fibers, compared to wavy or irregular fibers. The results showed a decrease in flexural stress was observed when TiO_2 nanoparticles content was increased to 5wt.% and a preload of 1200 N was applied. This is attributed to the agglomeration of particles at high concentrations and poor interfacial bonding. In addition, overloading may lead to permanent deformation of the fibers or matrix and increase the residual shear stress at the fiber-matrix interface, which contributes to gradual debonding that weakens the composite material's response to mechanical forces, especially in flexural tests.

CONCLUSION

In this study, the effect of both biaxial preloading and the addition of TiO_2 nanoparticles on the mechanical properties of glass fiber-reinforced epoxy polymer composites was investigated, leading to the following conclusions:

- The mechanical properties of the nanocomposite material are significantly affected by increasing the weight fraction of TiO_2 particles and preload level, a gradual

enhancement in mechanical performance was observed up to a concentration of 3wt.% and preload 800N.

- The tensile strength enhanced by 75.82% when 3% TiO₂ was added and a preload of 800 N was applied compared to the unreinforced and unpreloaded sample.
- The impact strength increased by 127.14% as a result of adding 3 wt.% titanium dioxide to the composite material with a preload of 800 N, compared to the unreinforced and unpreloaded sample.
- The flexural stress increased by 425.87% with increasing fiber preload up to 800N and adding Titanium dioxide by 3 wt.%.
- Using a 5 wt.% TiO₂ concentration with a preload of 1200 N led to decrease in the mechanical properties of the material.

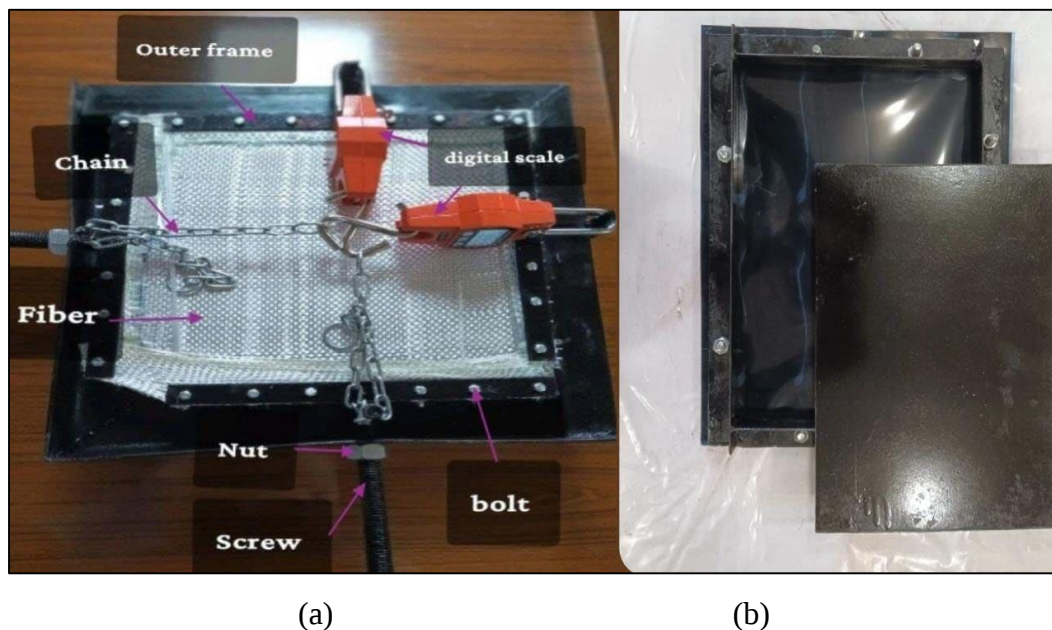


Fig. 1. (a) biaxial loading frame.; (b) manufactured frame.

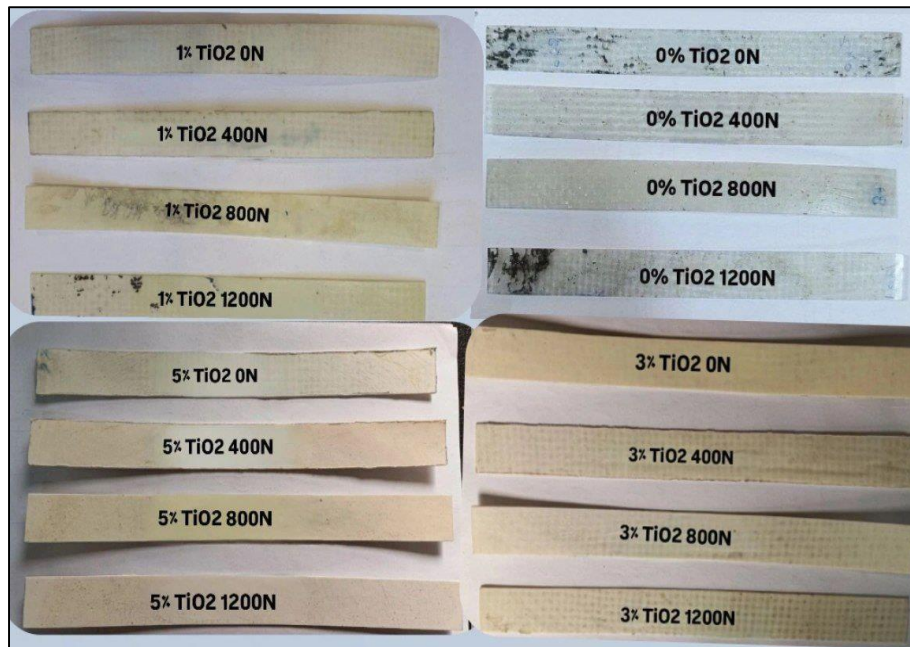


Fig.2.Tensile test samples.

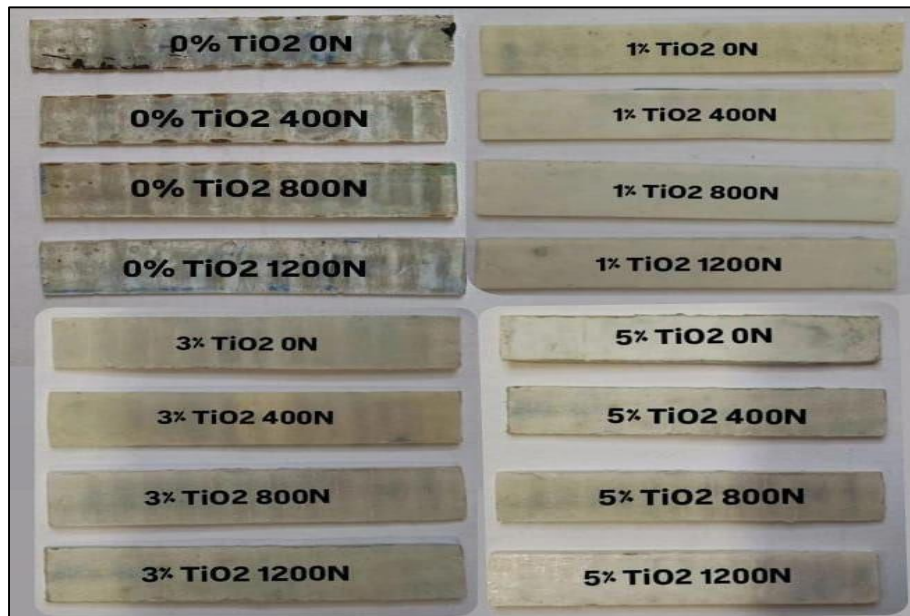


Fig.3. The impact test samples.

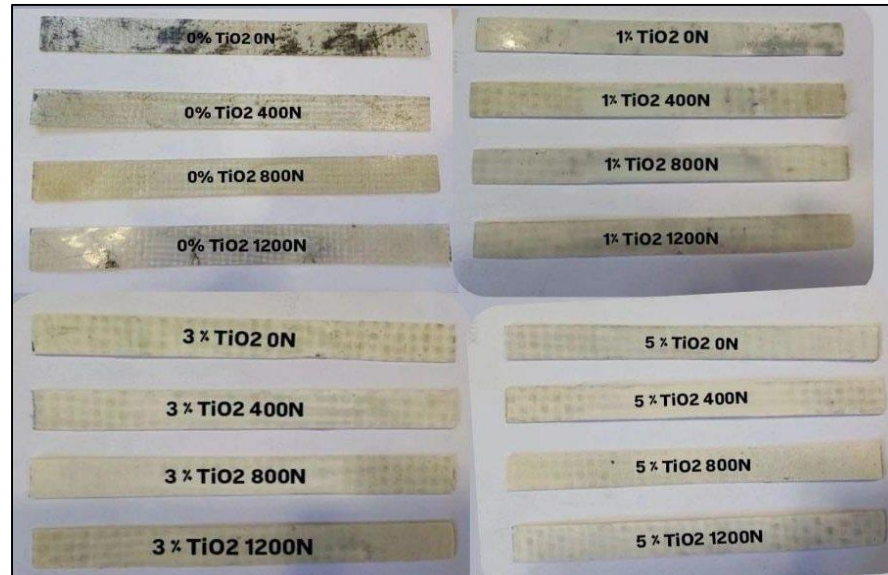
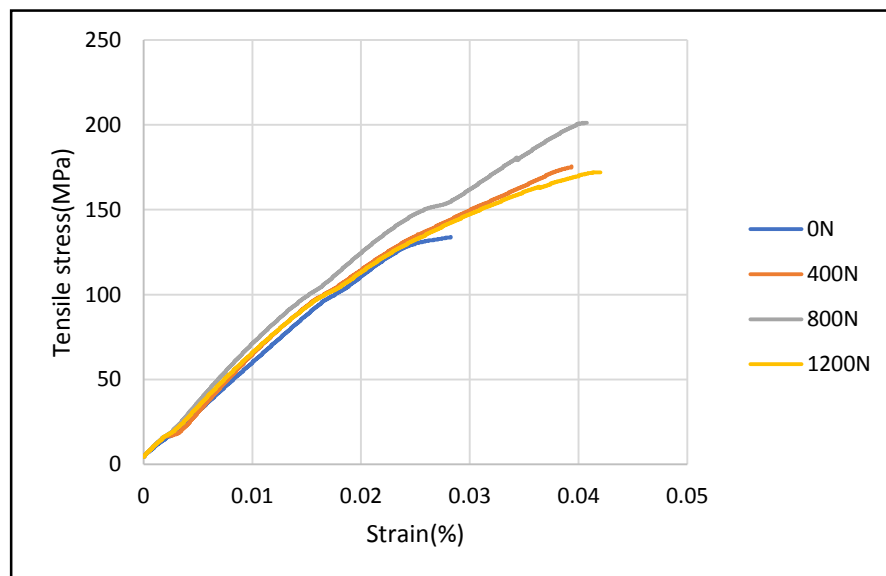
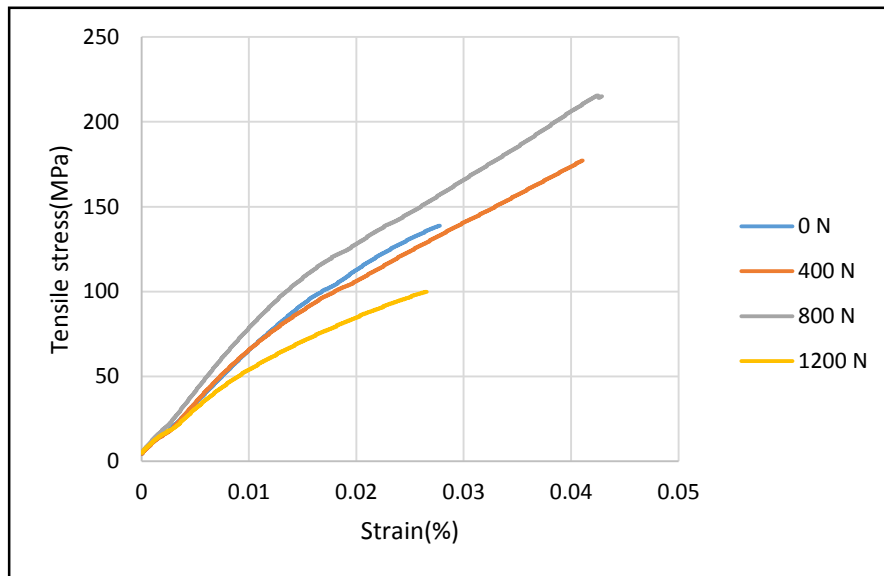


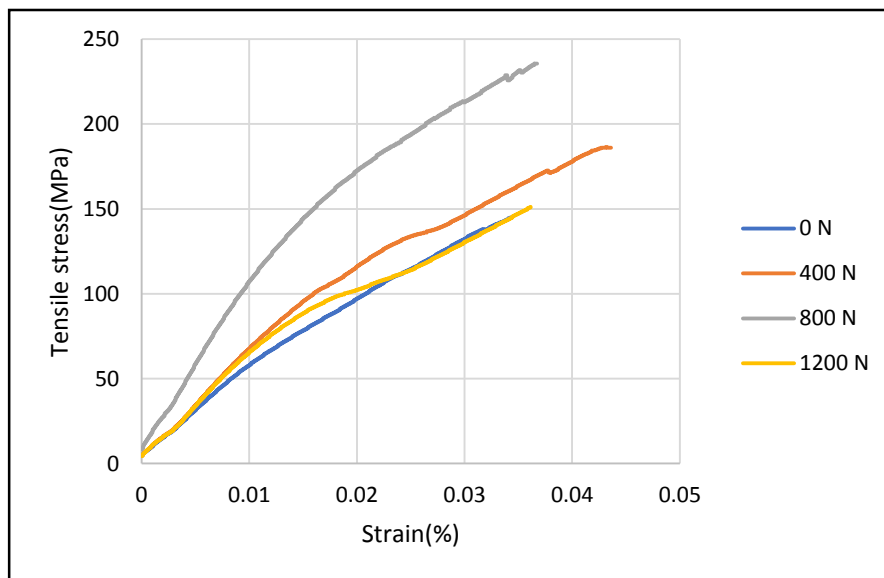
Fig.4. The flexural test specimens.



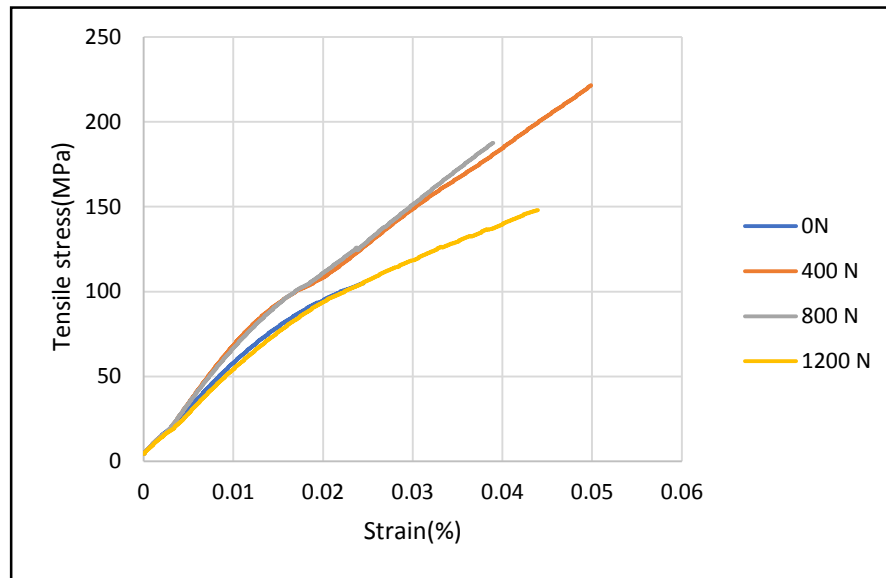
(a)Epoxy + glass fiber +0% TiO_2 nanoparticles.



(b) Epoxy + glass fiber + 1% TiO_2 nanoparticles.



(c) Epoxy + glass fiber + 3% TiO_2 nanoparticles.



(d) Epoxy + glass fiber +5% TiO_2 nanoparticles.

Fig.5. Stress–strain curves of preloaded glass fiber composite.

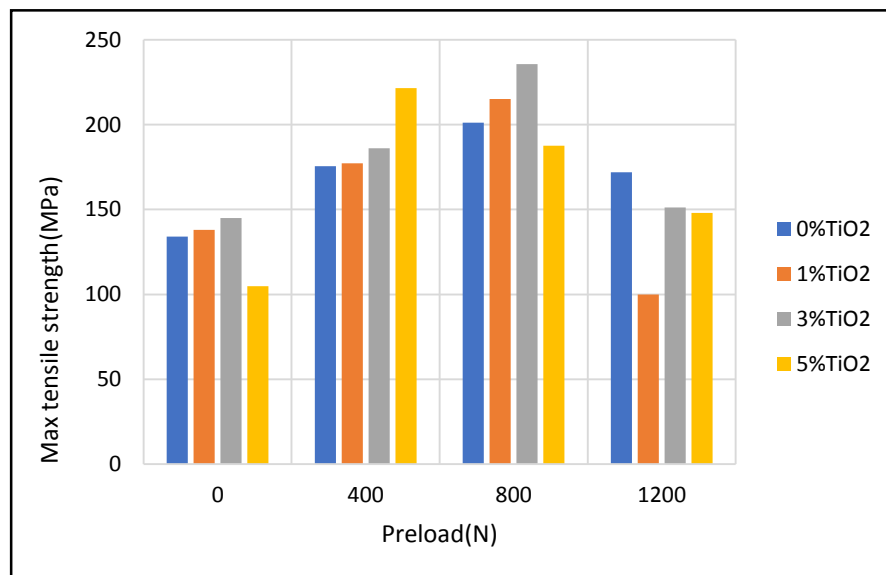


Fig.6.Maximum tensile strength and preload.

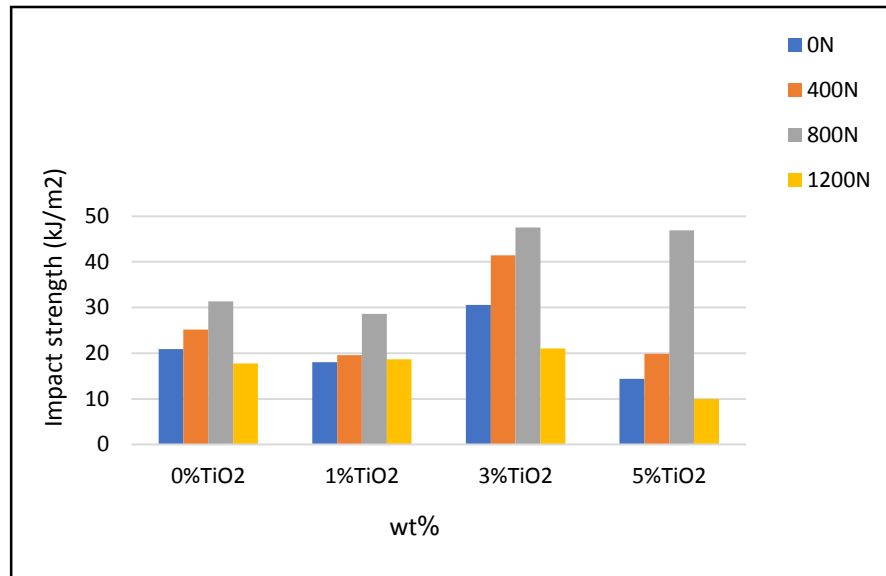


Fig.7. Impact test results.

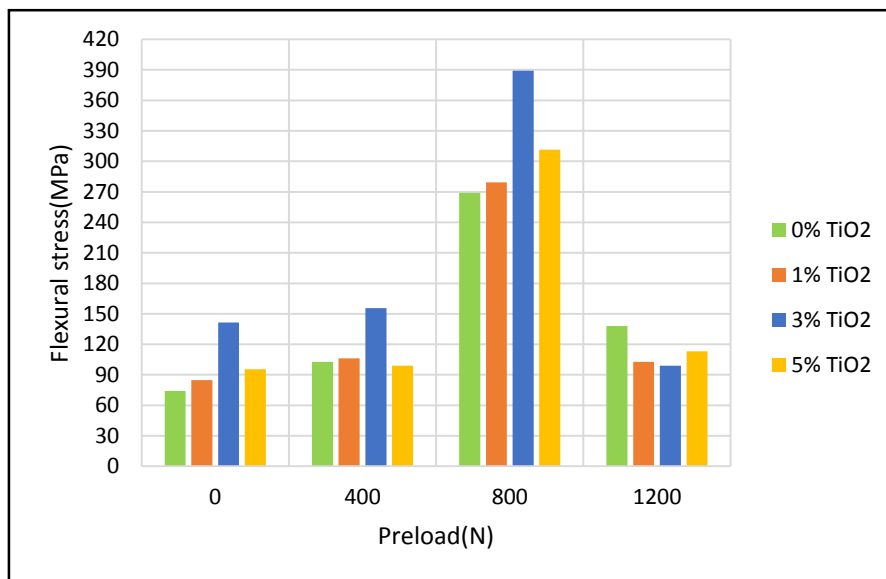


Fig.8. Flexural stress of epoxy composites reinforced with glass fibers.

REFERENCE

Al-Ajaj, I. A., Abd, M. M. and Jaffer, H. I. 'Mechanical Properties of Micro and Nano TiO₂ /Epoxy Composites', International Journal of Mining & Mechanical Engineering, 1(2), 2013. ISSN: 2320-4060.

Al-Dulaimy, A. K. M. A., Al-hassany, M. O. A. and Shakir, S. W. 'The Effect of Unidirectional Pre-load on Tensile Characteristics of E-glass Fiber and Epoxy Composite', Materials Today: Proceedings, doi: <https://doi.org/10.1016/j.matpr.2020.12.571>

ASTM, ASTM D3039/D3039M – Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials, Annual Book of ASTM Standards, pp. 1–13, 2014. doi: 10.1520/D3039

ASTM INTERNATIONAL, Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials. D790, Annual Book of ASTM Standards, i, pp. 1–12, 2002. doi: 10.1520/D0790-17.2

DCP International, Quick Mast 105, available at: www.dcp-int.com

Hayder, M. H. Al-Shukri 'Experimental and Theoretical Investigation into Some Mechanical Properties of Glass Polyester Composite Under Static and Dynamic Loads', M.Sc. Thesis, University of Technology, Baghdad, Iraq, 2007.

HWNANO, Product Information Page. Available at: <https://www.hwnanomaterial.com/>.

ISO, ISO 179-1: Plastics — Determination of Charpy Impact Properties, European Committee for Standardization, 2010. Available at: <https://www.iso.org/obp/ui/#iso:std:iso:179:2:ed-2:v1:en>

Karim, A. A., Kader, E. E., Hamod, A. A. and Abdulrahman, A. J. 'Mechanical Properties of a Hybrid Composite Material (Epoxy Polysulfide Rubber) Reinforced with Fibres', IOP Conference Series: Materials Science and Engineering, 433(1), 2018. doi: 10.1088/1757-899X/433/1/012050

Kumar, A., Saha, A. and Kumar, S. 'Structural Analysis of Sol-Gel Derived TiO₂ Nanoparticles: A Critical Impact of TiO₂ Nanoparticles on Thermo-Mechanical Mechanism of Glass Fiber Polymer Composites', 2021. doi: <https://doi.org/10.21203/rs.3.rs-539836/v1>

Mahmoud, M., Selim, M. M., Ning, H. and Pillay, S. 'Effect of Fiber Pre-stressing on Mechanical Properties of Glass Fiber Epoxy Composites Manufactured by Vacuum-assisted Resin Transfer Molding', Journal of Reinforced Plastics and Composites, 39(1-2), pp. 21-30, 2020.

Merad, L., Benyoucef, B., Abadie, M. J. M. and Charles, J. P. 'Characterization and Mechanical Properties of Epoxy Resin Reinforced with TiO₂ Nanoparticles', Journal of Engineering and Applied Sciences, 2011.

Mostafa, N. H., Ismarrubie, Z. N., Sapuan, S. M. and Sulta, M. T. H. 'The Influence of Equi-biaxially Fabric Pre-stressing on the Flexural Performance of Woven E-glass/Polyester-Reinforced Composites', Journal of Composite Materials, 2015. doi: 10.1177/0021998315620478

Nacy, S. M. and Yaser, A. S. 'The Effect of Fibers Pre-stressing on Tensile Properties of Glass Fiber/Epoxy Composite Materials', 1st Scientific Conference, Technical College-Najaf, Iraq, 2008.

Nishi, Y., Okada, T., Okada, S., Hirano, M., Matsuda, M., Matsuo, A. and Faudree, M. C. 'Effects of Tensile Prestress Level on Impact Value of 50 vol% Continuous Unidirectional 0 Degree Oriented Carbon Fiber Reinforced Epoxy Polymer (CFRP)', Materials Transactions, 55, pp. 318-322, 2014. <https://doi.org/10.2320/matertrans.M2013278>

Sika Wrap ® -300 C, Product Data Sheet, December 2020, pp. 1-5.

Somaiah, A., Prasad, B. A. and Nath, N. K. 'Exploring the Impact of TiO₂ and MgO Nanoparticles on the Mechanical and Topographical Characteristics of Glass Fiber Reinforced Polymer (GFRP) Composites with Varied Lay-up Sequences: A Taguchi Analysis', International Research Journal of Multidisciplinary Technovation, 6(2), pp. 70-83, 2024. doi: <https://doi.org/10.54392/irjmt242>