

Effect of graphene, silicon, and silver doping on the mechanical and structural properties of advanced epoxy composites

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Abstract: In this study, the effect of incorporating graphene, silicon, and silver nanoparticles into epoxy composites was investigated to enhance their mechanical and structural properties. The research examined various combinations of these materials, including pure epoxy, epoxy/graphene (0.1 wt.%), epoxy/graphene/Si/Ag (0.1-1 wt.%), and epoxy/Si/Ag (0.1-1 wt.%) composites. The structural characteristics were analyzed using XRD and FE-SEM techniques, while mechanical properties were evaluated through tensile testing. Results showed that the addition of graphene significantly improved the crystallinity and structural organization of the composites. The optimal results were achieved in the ternary system (epoxy/graphene/Si/Ag) with 1 wt.% Si +Ag content, showing an enhanced elastic modulus of 0.30 kN/mm² compared to pure epoxy (0.23 kN/mm²). FE-SEM analysis revealed that graphene helped create a uniform network structure that effectively distributed the Si and Ag particles throughout the matrix, leading to improved mechanical performance.

Keywords: Tensile properties, XRD analysis, Hybrid composites, Epoxy/graphene/Silver nanocomposites, Nanostructure.

1. Introduction

The term "nano" is derived from the Greek word for "dwarf" or "abnormally short person." It is used as a prefix for any unit, such as a second or a meter, and it means a billionth (10^{-9}) of that unit. Thus, a nanometer (nm) is a billionth of a meter [1]. Nanocomposites containing nanoparticles can be prepared using mechanical or chemical methods, such as vapor phase deposition, depending on the type of matrix [2]. The term polymer refers to a type of macromolecule that is made up, at least to a first approximation, of a set of regularly repeated chemical units of the same type, or possibly of a very limited number of different types (usually only two), and joined end to end, or sometimes in more complicated ways, to form a chain. If there is only one sort of chemical unit, the resulting polymer is a homopolymer; if there are a few, it is a copolymer [3]. Adding nanofillers to the epoxy matrix is an efficient technique to create innovative materials that combine the benefits of epoxy with nanomaterials [4]. Fillers that are added to epoxy resin to minimize cost, shrinkage, and coefficient of thermal expansion are predicted to contribute to both thermal and electrical conductivity. The size, shape, and orientation of the filler particles should be considered [5]. Epoxies are appropriate for these applications due to their high specific stiffness, specific strength, electrical insulating characteristics, corrosion resistance, chemical compatibility with reinforcing fibers, and relative simplicity of production [6]. The performance of epoxy resins is influenced by factors like the curing temperature, the environment in which they are used, and the amount of hardener mixed with the resin [7]. In order to gain improvements of the performance of epoxy polymers, several authors suggested that low or high aspect ratio fillers should be incorporated into epoxy. These fillers should possess some adequate properties, namely [8]:

- a higher rigidity than the polymer;

- a high specific surface;
- a high filler-matrix bonding;
- small dimensions

Graphene is a single layer of carbon atoms that are tightly bound, giving a honeycomblike appearance to graphene sheets. The unique properties of graphene, including high specific surface area, high young's modulus, a strong filler-matrix interface, and better bonding of the polymer matrix and the filler particles have increased the use of this material [9]. With a Young's modulus (stiffness) of 1 TPa, it is the strongest material ever tested. Graphene possesses other remarkable characteristics: electron mobility is 100× faster than silicon; It has also been utilized as a reinforcing agent to enhance the mechanical properties of biodegradable polymeric nanocomposites [10]. Nanocomposites (e.g. Si NPs/magnetite, Si NPs/silver) have attracted considerable attention, for their multi-functionality is superior to either component [11]. The word 'Silicon' originated from silicium (Latin), meaning what was more generally termed as "the flints" or "Hard Rocks". It is an abundant nonmetallic element found throughout the Universe. On earth, silicon is one of the most common elements after oxygen and carbon [12]. Mechanical strength and flexibility are two important features present in silicon-based polymeric NCs. It is well understood by the scientific community that the mechanical performance of a polymeric system increases dramatically upon the incorporation of silicon-based nanomaterials [13]. Silver nanoparticles (Ag NPs) are increasingly used in various fields, including medical, food, health care, consumer, and industrial purposes, due to their unique physical and chemical properties. These include optical, electrical, and thermal, high electrical conductivity, and biological properties [14].

The term mechanical properties are commonly used to denote stress-strain relationships for polymer systems. Unlike many more familiar materials where these relationships depend essentially only on temperature, in polymeric systems time dependence is also of importance. This time dependence necessitates very careful definitions of parameters such as moduli and compliances which result from experiments involving discontinuous stress or strain levels. In addition, the time dependence may be explored using oscillatory perturbations as is done when investigating dynamic mechanical properties or dielectric relaxation in polymers [15]. X-rays can also reveal various information on the materials, including crystal structure, phase transition, crystalline quality, orientation, and internal stress [16]. The Field Emission Scanning Electron Microscope (FESEM) is an instrument which, just like the SEM, provides a wide variety of information from the sample surface, but with higher resolution and a much greater energy range. It works just like a conventional SEM; the sample surface is scanned with an electron beam while a monitor displays the information that interests us on the basis of the detectors available [17].

2. Materials and Methods

2.1. Materials

2.1.1. Epoxy Risen

The nano polymer composite material was prepared using (epoxy risen-sikadur®52) as a basis material. It is a 2-component, 100% solids, moisture-tolerant, epoxy adhesive. It is a low-viscosity, high-strength adhesive formulated specifically for grouting both dry and damp cracks. It conforms to the current ASTM C-881, types I and II, grade-1, class c and AASHTO M-235 specifications. produced by the Swiss chemical firm Sika, which specializes in building and automotive products. This resin is in the form of a transparent, colorless, viscous liquid with low viscosity at room temperature. This resin is transformed from the liquid state to the solid state by adding its own hardener (Hardener-sikdur®52), manufactured by the same company. It is in the form of a transparent liquid added to the resin. Epoxy at a ratio of (1:2), that means, (2g) for every (1g) of resin at room temperature, and table (1) shows the most important properties of the material. Epoxy used in the research and according to the specifications of the producing company.

Table 1.

Shows the properties of epoxy material.

Density (g / cm³)	1.2
Viscosity (Mixed) at 35°C	200
Tensile strength (MPa)	54
Flexural Strength (MPa)	37.2
Shear Strength (MPa)	29.6
Modulus of Elasticity (MPa)	2,620
Color	Clear, pale yellow

2.1.2. Silicon

In this research the use of silicon produced by the American Company Sigma-Aldrich. The properties of silicon are given in Table (2) according to the properties of Product Company.

Table 2.

Shows the properties of silicon.

Material	Si
Density at 25 °C	2,33 (g/mL)
Molecular weight	28,09 g/mol
Water solubility at 20 °C	0,1 g/l

2.1.2. Silver

In this research the use of silver produced by the American Company (Sky spring nanomaterials, Inc). The properties of silver are given in Table (3) according to the properties of Product Company.

Table 3.

Shows the properties of silver.

Material	Ag
Purity	99.9%
Morphology	spherical
Bulk density	0.35 g/cm ³
True density	10.5 g/cm ³
APS	50-60 nm
Appearance	Dark Grey Powder

2.2 Preparation Method

2.2.1 Preparation Pure Epoxy

Pure epoxy samples were made by mixing epoxy resin with hardener. The epoxy resin and hardener were weighed using an electronic balance at a mixing ratio of 2:1 by weight. The hardener was added to the epoxy resin and mixed until a homogeneous mixture was obtained. The mixture was placed in a dryer to remove air bubbles, then poured into prepared stainless steel molds. The samples were left for 48 hours until they hardened. Then they were removed from the molds and smoothed according to ASTM standard specifications for samples and subjected to a mechanical properties test (tensile test).



Figure 1.
Shows the shape of the molds.

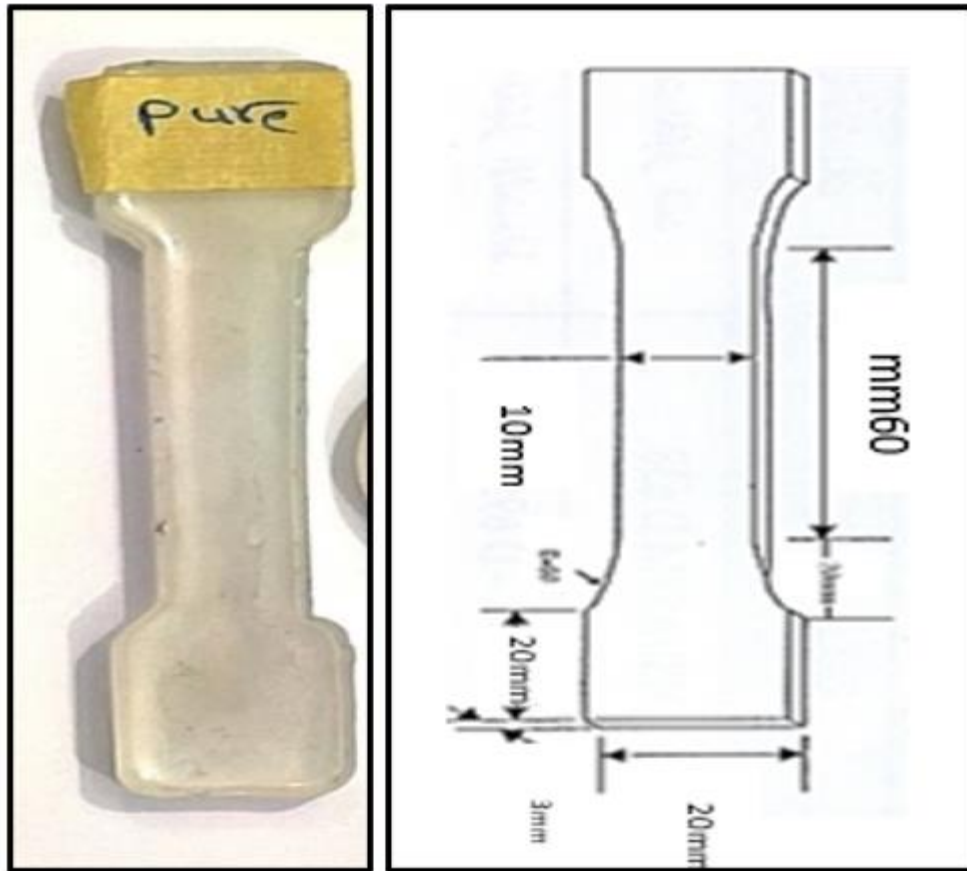


Figure 2.
Shows the sample of the pure epoxy mode

2.2.2. Preparation of Graphene Composite

Graphene/epoxy composite samples were prepared by incorporating 0.1 wt.% graphene into epoxy resin. Components were individually weighed using a sensitive balance. The graphene powder was mixed with epoxy for 15 minutes at 50°C using slow, continuous mixing to ensure homogeneity while preventing bubble formation. The hardener was then added and mixed slowly for 10 minutes. The resulting mixture was carefully poured from one side into pre-prepared metal molds to prevent bubble entrapment, filling them to the required level. Samples were left to cure at room temperature for two days until completely hardened. The cured samples were then extracted, smoothed, and subjected to mechanical and electrical property testing as shown in Figures 3.



Figure 3.
Shows the graphene and epoxy samples.

2.2.3. Preparation of (Graphene/Silicon/Silver) Nanocomposite

Five samples were prepared using fixed 0.1% nano-graphene with different ratios of nano-silicon and nano-silver powders (0.1%, 0.3%, 0.5%, 0.7%, 1%) with epoxy resin. The graphene, silicon, silver, epoxy, and hardener were weighed separately using a sensitive balance. First, graphene powder was mixed with epoxy for 15 minutes at 50°C slowly to get uniform mixing without bubbles. Then, silicon powder was added gradually and mixed for 15 minutes at 50°C using half of the specified weight ratios. After that, silver powder was added and mixed for 15 minutes at 50°C using the remaining half of weight ratios, ensuring the total weight ratio of silicon and silver was 0.1% for the first sample, 0.3% for the second, and so on. Finally, the hardener was added and mixed slowly. The mixture was poured carefully from one side into prepared metal molds to avoid bubbles. The samples were left for two days at room temperature to harden. as shown in Figure 4.



Figure 4.
Shows the (Graphene/silicon /silver) nanocomposite samples

Table 4.
The samples were prepared by fixing epoxy and graphene content while varying Si/Ag concentrations.

Sample Code	Amount of Epoxy (ml)	wt.% graphene(g)	wt.% (Si+Ag) g
C1			0.1
C2			0.3
C3	2:1	0.1	0.5
C4			0.7
C5			1

2.2.4. Preparation of (Silicon/Silver) Nanocomposite

Five samples were prepared using different weight ratios of nano-silicon and nano-silver powders (0.1%, 0.3%, 0.5%, 0.7%, 1%) with epoxy resin. The silicon, silver, epoxy, and hardener were weighed separately using a sensitive balance. First, silicon powder was mixed with epoxy for 15 minutes at 50°C using half of the specified weight ratios to get uniform mixing without bubbles. Then, silver powder was added and mixed for 15 minutes at 50°C using the remaining half of weight ratios, ensuring the total weight ratio of silicon and silver was 0.1% for the first sample, 0.3% for the second, and so on. After that, the hardener was added and mixed slowly. The mixture was poured carefully from one side into prepared metal molds to avoid bubbles. The samples were left for two days at room temperature to harden. as shown in Figure (5).



Figure 5.
Shows the (silicon /silver) nanocomposite samples.

Table 5.

The samples were prepared by fixing epoxy content while varying Si/Ag concentrations.

Sample Code	Amount of Epoxy (ml)	wt.% (Si +Ag) g
D1		0.1
D2		0.3
D3	2:1	0.5
D4		0.7
D5		1

2.3. Characterization

2.3.1. Tensile Test

Mechanical tests were performed according to ASTM: D638-10 (2010) standards using a Zwick-Roell universal testing machine (UTM) equipped with a 5 kN load cell. Five specimens of each composite were tested in each of the loading configurations. Specifically, tensile tests were performed on graphene/silicon/silver nanocomposite specimens with the crosshead speed set at 5 mm/min. Tensile strength and elastic modulus were calculated according to ASTM: D638-10 (2010), as shown in Figure (6).

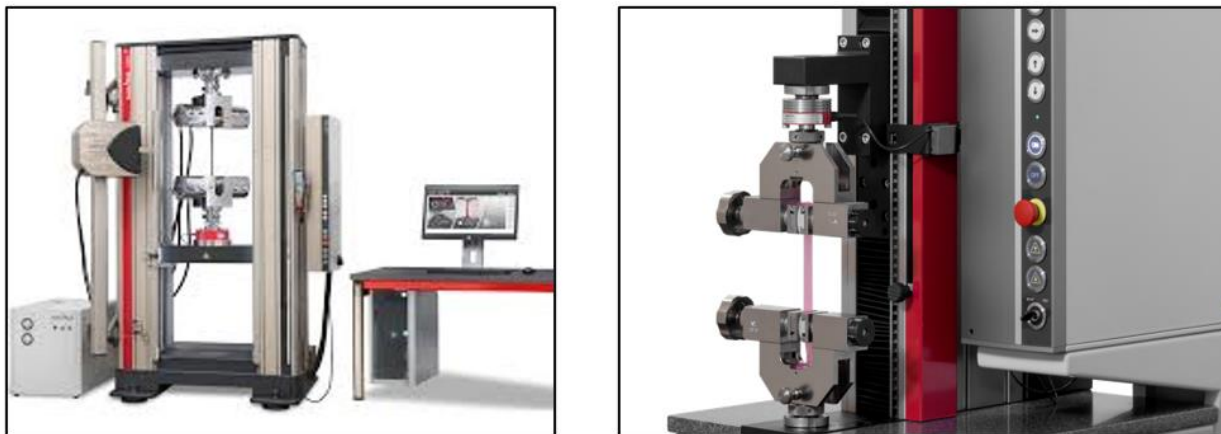


Figure 6.
Shows a tensile testing device.

3. Result and Discussion

3.1. Structural Characterization

3.1.1. XRD Result

The XRD patterns from epoxy-based composites display different crystallographic patterns in samples from groups C and D.

The XRD patterns shown in Figure 1 belong to D-group samples (Epoxy +Si +Ag) and **Figure (8)** features the XRD patterns of C-group samples (Epoxy+Graphene+Si+Ag). The XRD patterns show distinctive peaks for both groups although they display distinct structural patterns.

The epoxy matrix displays a broad peak at $2\theta = 20^\circ$ in both groups because it exists in an amorphous state. The XRD patterns from C-group samples show more pronounced peaks with higher intensity than those of D-group samples which indicates that graphene enhances the crystalline nature of the material. The data indicates that graphene functions as a promoting agent which leads to improved crystalline structure in the composite formation.

The C-group samples produce well-defined peaks at 2θ values of 20° , 40° , and 70° because their organized structure exhibits higher crystallinity. The addition of graphene enables Si+Ag particles to organize more orderly within the epoxy matrix. The XRD technique reveals that crystal structure within composites varies systematically as filler concentration changes from 0.1% to 1%.

The D-group samples present wide peaks with lowered intensity which signifies their amorphous nature. A lack of graphene in the structure leads to reduced ordering among Si+Ag particles because both peak intensities and peak widths become broader. The position of peaks matches the C-group results but their reduced intensity points to inferior crystalline alignment.

Graphene addition to the C-group creates stronger connections between epoxy and Si+Ag particles that result in more orderly structural design. The enhanced crystallinity in the composites matches recent mechanical property assessments therefore indicating a solid structure-property link throughout this system.

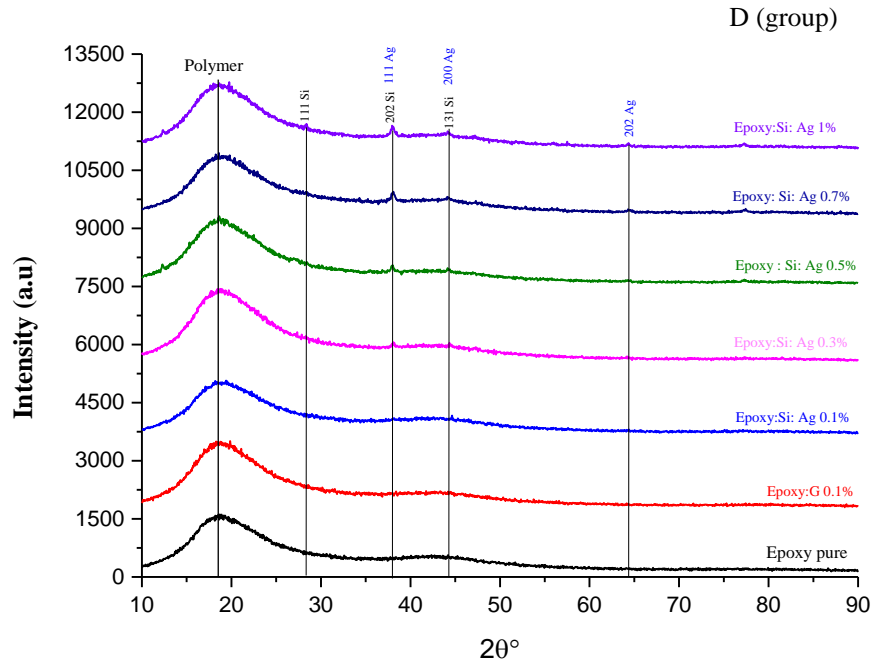


Figure 7.
XRD patterns of D-group samples (Epoxy+Si+Ag).

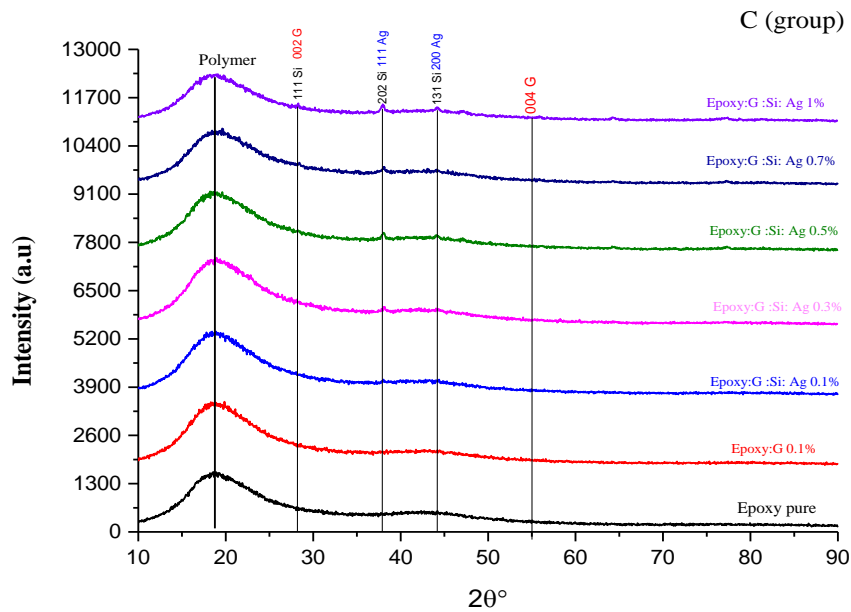
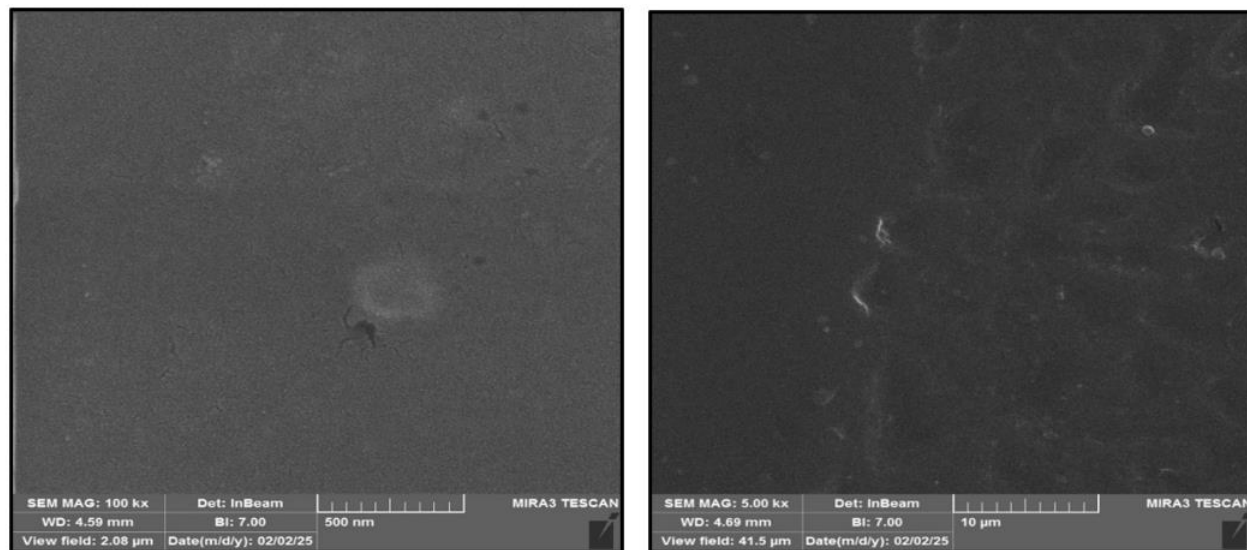


Figure 8.
XRD patterns of C-group samples (Epoxy+Graphene+Si+Ag).

3.1.2. Morphological Characterization

The FE-SEM micrographs display remarkable morphological changes throughout different epoxy composite samples which shed light on their relationship between structure and properties.

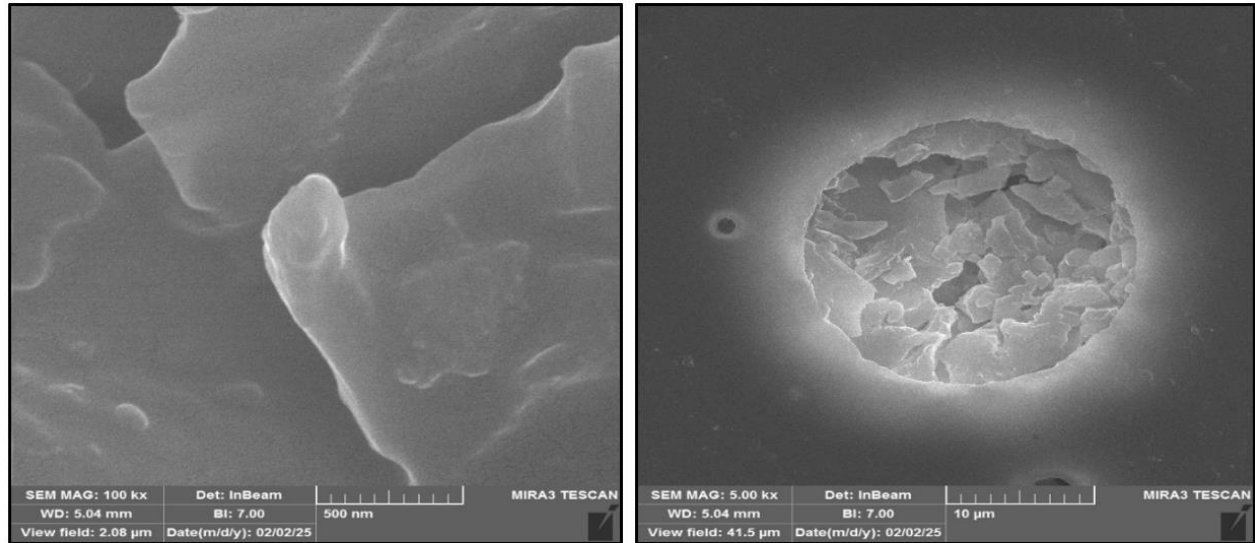
Figure 9 shows the pure epoxy surface morphology at two magnifications demonstrating a mainly smooth and homogeneous surface structure. The moldable structure depicted by pure epoxy samples corresponds to their lower mechanical strength of 0.23 kN/mm^2 and demonstrates amorphous characteristics based on the XRD patterns.



Pure Epoxy

Figure 9.
FE-SEM images of pure epoxy at different magnifications.

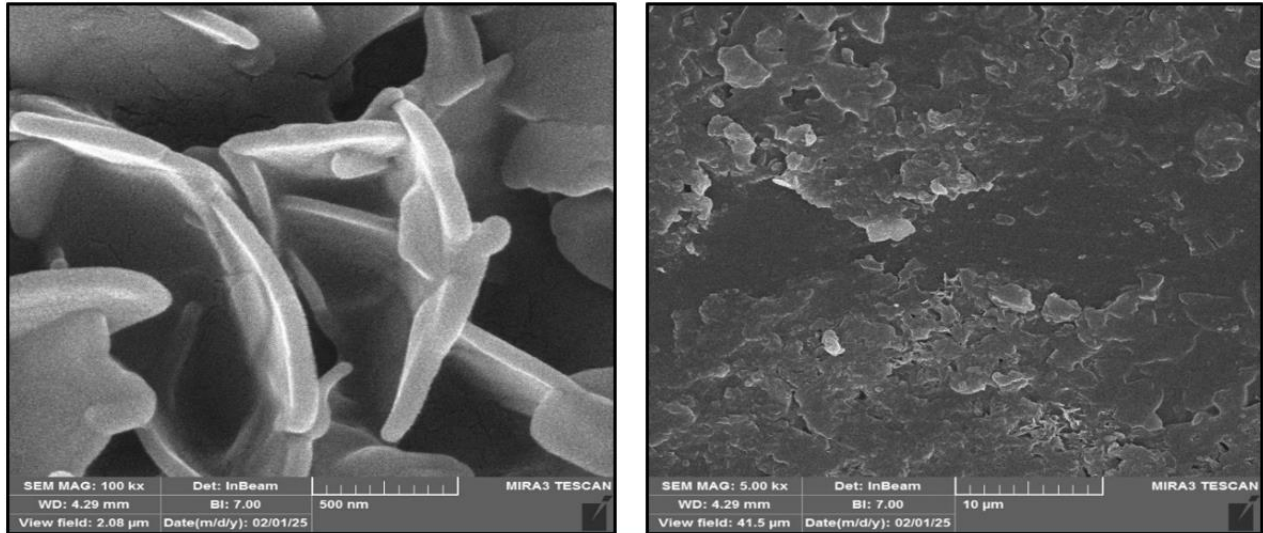
Figure 10 shows the major morphological transformations that occur when incorporating 0.1% graphene into Epoxy. The 100kx magnification reveals distinct layers whereas 5.00kx magnification shows characteristic graphene agglomeration patterns that appear flower-shaped. The newly formed structure leads to a decreased elastic modulus of 0.17 kN/mm^2 during mechanical testing since the agglomerations create localized stress points. The XRD patterns show broader peaks that indicate the sample has an unorganized structural arrangement.



Epoxy + Graphene 0.1%

Figure 10.
Morphology of epoxy/graphene (0.1%) composite showing filler distribution.

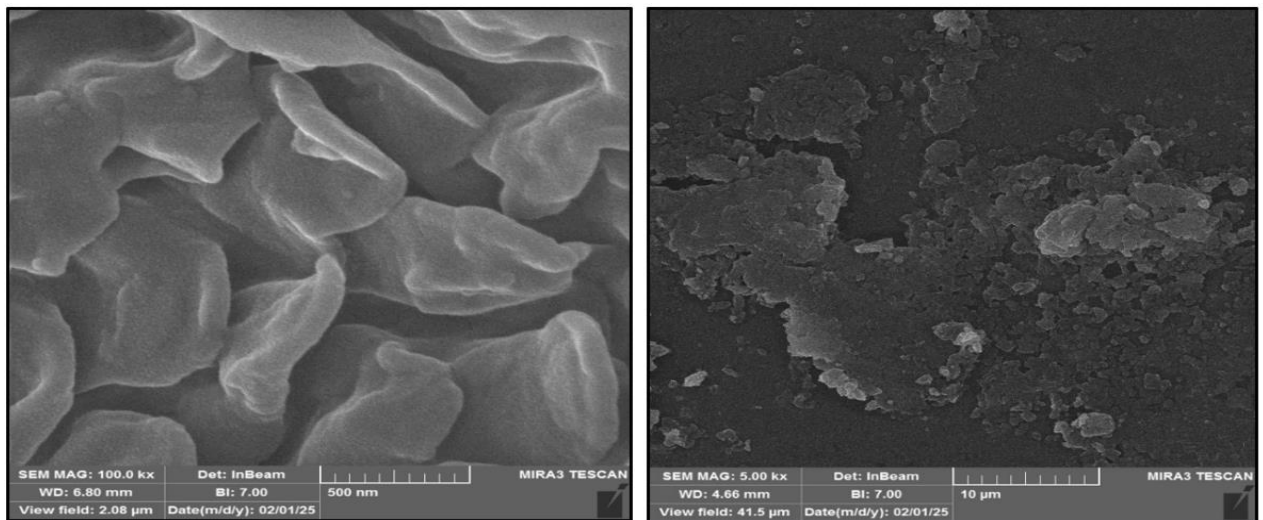
The Epoxy + Graphene + Si +Ag 1% composite contains the most fascinating structure evident in Figure (11). Analysis at high magnification shows a highly complex network structure which spreads with excellent filler dispersion throughout the material. An examination at lower magnification reveals uniform distribution of these networks over the surface which matches the improved mechanical properties ($E = 0.30 \text{ kN/mm}^2$) and XRD peaks with better crystallinity.



Epoxy + Graphen + Si + Ag 1

Figure 11.
Network structure in epoxy/graphene/Si + Ag hybrid composite.

The composite in **figure (12)**; (Epoxy + Si + Ag 1%) shows massive clusters of particles that remain isolated from each other. The inferior filler arrangement leads to reduced mechanical properties ($E = 0.20 \text{ k N/mm}^2$) when compared to the hybrid configuration. The XRD peaks of D-group samples matched the surface features which suggests disorganized structural arrangements.



Epoxy + Si + Ag 1%

Figure 12.
Particle distribution in epoxy/Si+Ag composite.

The morphological investigation results from the samples demonstrate robust evidence showing the combined benefits that graphene and Si+Ag provide to hybrid composites. **Figure (9)** presents an optimized network structure that enables improved load distribution across the whole matrix to achieve superior mechanical outcomes. The macro-scale properties receive direct influence through nanoscale

structural organization which displays a correlation with surface morphology and crystallinity. An extensive analysis proves that hybrid composite performance reaches its pinnacle because of the singular morphology formed by graphene and Si + Ag particle interactions.

3.2. Mechanical properties

3.2.1. Tensile Test

Table 6 show the Epoxy-based composites show complex structural relationships between properties that result from multi-component filler systems. The elastic modulus measurement of pure epoxy shows 0.23 k N/mm² as its baseline value which defines the load-bearing capability of the crosslinked polymer network. sample (epoxy+graphene) 0.1 wt.% graphene nanofiller produced an opposite effect on elastic modulus which declined to 0.17 kN/mm² while displaying indicators of agglomeration and subpar alignment between graphene particles and epoxy boundaries.

The C-series specimens exhibit the most important findings when using Epoxy/Graphene/Si+Ag as the ternary system. The synergistic effects in composition C5 with 1 wt.% Si + Ag led to an optimal elastic modulus value of 0.30 kN/mm². The linear elastic portions of stress-strain curves show elevated slopes which confirm better load capacity and adhering bonds. Stress transfer occurs effectively through the continuous nanofiller network while enhanced interfacial interactions exist between hybrid fillers and epoxy matrix and the Si+Ag particles distribute ideally because graphene layers present two-dimensional morphology [18].

The mechanical performance of D-series samples (Epoxy/Si+Ag binary system) exhibits subpar results indicating elastic moduli rates between 0.20-0.27 kN/mm². The significant difference between D- and C-series illustrates graphene's vital function in the material structure by reinforcing it while dispersing Si+Ag particles. The alteration of filler concentration produces non-monotonic results because dispersion quality and network formation mechanisms compete with each other in complex ways.

Table 6.

Elastic modulus of epoxy nanocomposites (k N/mm²).

Sample Code		E (k N/mm ²)
E	Epoxy	0.23
F	Epoxy + Graphen 0.1%	0.17
C1	Epoxy + Graphen +Si+Ag 0.1%	0.26
C2	Epoxy + Graphen +Si+Ag 0.3%	0.28
C3	Epoxy + Graphen +Si+Ag 0.5%	0.27
C4	Epoxy + Graphen +Si+Ag 0.7%	0.23
C5	Epoxy + Graphen +Si +Ag 1%	0.30
D1	Epoxy +Si+Ag 0.1%	0.23
D2	Epoxy + Si+Ag 0.3%	0.21
D3	Epoxy + Si+ Ag 0.5%	0.27
D4	Epoxy + Si+Ag 0.7%	0.27
D5	Epoxy + Si +Ag 1%	0.20

Figure 13 show the stress-strain curves reveal the complete mechanical behavior by showing various deformation patterns. Initial slopes of C-series specimens remained high while showing an extended linear elastic range because their hybrid filler network both improved structural rigidity and distribute loads efficiently. Proper dispersion of hybrid fillers in nanocomposites leads to synergistic reinforcement effects that adhere to predicted multiple strengthening mechanisms.

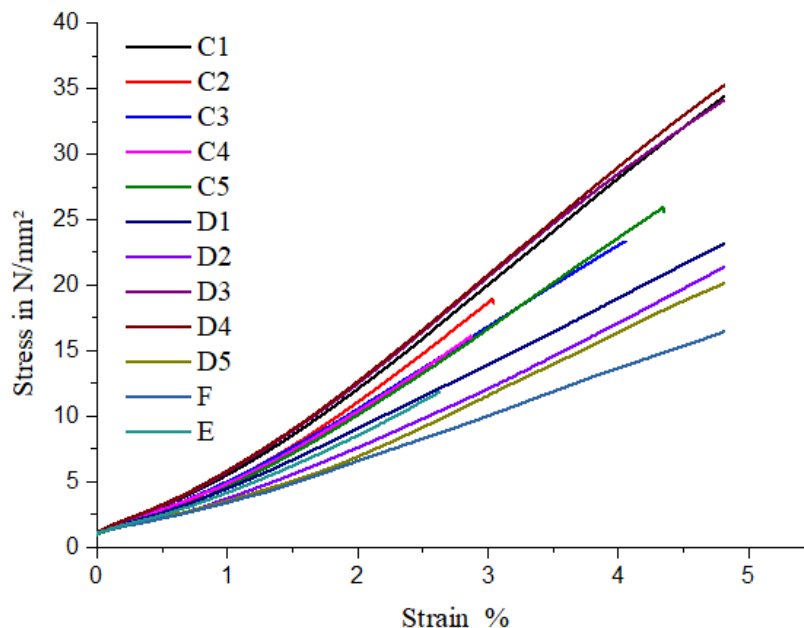


Figure 13.
Stress-strain curves.

Transparency:

The authors confirm that the manuscript is an honest, accurate, and transparent account of the study; that no vital features of the study have been omitted; and that any discrepancies from the study as planned have been explained. This study followed all ethical practices during writing.

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