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Hydrothermal Modification of Carbon Fiber Fabrics by ZnO Nanorods for Mechanical Strengthening of CFRP Laminates

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Abstract

Hexagonal ZnO nanorods were produced on plain woven carbon fiber using a two-step seed-assisted hydrothermal procedure. By varying the process parameters such as molar concentrations, number of seeding cycles, and growth duration at a controlled growth temperature of 90 °C, it is feasible to produce ZnO nanostructures with a range of morphologies, including nanowires, hexagonal nanorods, and nanoflowers. The developed morphologies were examined using field emission scanning electron microscopy and the elemental compositions by energy-dispersive X-ray spectroscopy. The length of the seeding and growth treatments significantly impacts how nanostructures grow. Using the vacuum bagging technique, a laminated composite comprised of ZnO-orchestrated woven carbon fiber (WCF) with epoxy resin as the matrix. The most intriguing outcome of this work is how generated nanorods affect laminated composite impact strength due to better interfacial contact. The impact energy absorption capacity will alter because of fluctuations in ZnO's convergence over time. ZnO grown on WCF, however, has led to the emergence of unique failure modes that characterize the fracture mechanism of hybrid composite materials, such as ZnO nanorod pullout and ZnO nanorod breakage. The most significant gains in impact strength, tensile strength, elastic modulus, and in-plane shear were obtained by the ZnO-modified composite at a concentration of 30 mM, with corresponding percentage increases of 39%, 38%, 32%, and 6%. A promising method of functionalization to achieve desired material characteristics for structural applications is the formation of ZnO nanostructures on WCF. Based on these outcomes, the hybrid composites that have been produced offer potential regarding utilization in the development of aviation and automotive industries. The developed composite materials are highly desirable for these industries because they have high impact strength, modulus, lightweight properties, and low void content.

Keywords: ZnO, Carbon fiber, Nanorods, Hydrothermal process, Impact energy absorption, Laminated composite.

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INTRODUCTION

A significant characteristic of composite materials is their impact strength, essential for predicting the harm framework concerning stiffness. However, fiber-reinforced polymer

(FRP) composites exhibited inadequate durability under impact loading due to their low capacity to retain vitality [1]. A sudden load on FRP can cause them to adapt to influence essentiality and recognize distortion. A sudden load on FRP can cause them to adapt to influence essentiality and recognize distortion. Then, the parts form in the lattice if considerable effect essentialness is maintained. The persistent cracks reached the interphase's intermolecular connection, resulting in the most preposterous impact energy ingestion when the interphase was wholly destroyed [2]. Cracks in the structure signal the failure of a plain FRP composite during an impact test, which is followed by differentiation at the area where the lattice and the fortifications meet. The manufacture of hybrid composites, which incorporate secondary reinforcement in addition to conventional miniaturized scale reinforcing filament, is fascinating. Hybrid structures, such as plant cells, creature shells, and skeletons, are broadly seen in nature, demonstrating that high mechanical properties can be obtained, even from genuinely frail constituents, by organizing matter over a scope of length scales [3]. The fundamental reason for adding carbon nanotubes (CNTs) to traditional fiber composites is to mitigate the current impediments to the properties of matrix ruled. For example, CNTs could offer interlaminar and intralaminar fortification, subsequently enhancing the thickness properties and the delamination resistance without trading off in-plane execution [4]. The two main methods used to combine CNTs with regular fiber fortifications in polymer composites are scattering CNTs evenly throughout the matrix and the second one is growing CNTs appropriately onto fiber strands. The first strategy is easy to create and is compatible with conventional mechanical techniques, but it is limited to low loading values.In contrast with the first technique, it is influenced by grid-dominated properties like ILSS (inter-laminar shear strength), which enhance 8-30% more than the fiber-rules in-plane features. For improving the fiber surface region, making mechanical interlocking and local softening at the fiber/grid interface, uniting the CNTs into fiber surfaces is a practical approach. By this, it may progress the load exchange and interfacial properties. Explicit nanoscale impacts on the polymer properties are likewise conceivable as illustrated in Figure 1 [5, 6].

In this way, metal-oxide nanostructures such as nanorods, nanowires, and nanoflowers can be grafted in woven fibers using the surface whiskerization technique to modify the interface properties of hierarchical composites. Metal-oxide material has been one of the critical technologies for over a few years [7, 8]. The non-appearance of a central point of symmetry in its wurtzite structure is exhibited alongside giant electromechanical coupling, which brings about remarkable piezoelectric and pyro-electric features. Zinc-oxide attracts scientists to use its novel properties like vast energy gap of 3.37eV and extensive binding vitality of 60meV. Specialists added its application to microscopy techniques for creating vitality-detecting and acoustic-detecting sensors [9, 10]. Presently, ZnO has been effectively utilized to improve various leveled nanostructures through solvothermal strategies by Lu et al. [11]. A wide assortment of nanostructures of ZnO was created already under controlled development procedures like nanowires, nanorods, nanoflowers, nano flakes, nano brushes, nanoring, nano helices, nanobots, nanobelts and nanocages [12, 13]. Various techniques, for example, thermal decomposition, thermal evaporation, solid-state reaction, self-catalytic development, and solution-based method, out of which the solvothermal approach is more suitable than others due to low

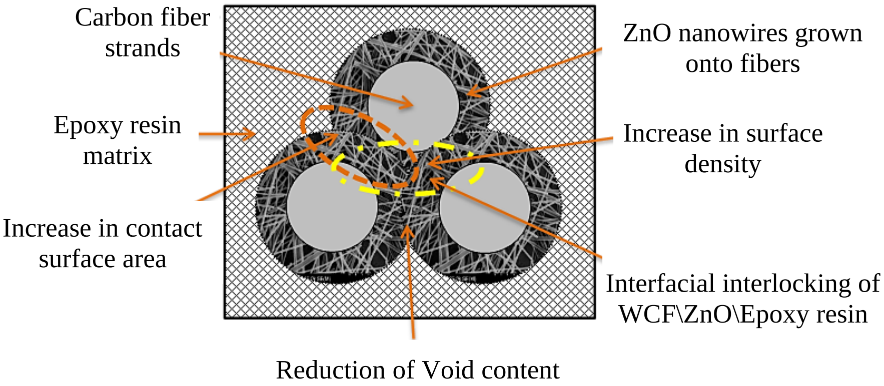


Figure 1. Enhancement of interfacial interaction of ZnO/WCF/Epoxy composites.

wastage of energy and no setup cost required [14–17]. The solvothermal process includes the solution phase synthesis method in the alcoholic medium. In solution phase synthesis of nanostructure, an aqueous solution is generally used; this process is termed solvothermal. The creation of epitaxial and anisotropic development of crystal is managed by solvothermal technique. Usually, this technique does not depend on the surface and allows us to maintain the controlled morphological growth of nanowires [18, 19]. The solvothermal method, first reported by Vayssieres et al. [20], uses equimolar emulsion of HMTA and zinc nitrate hexahydrate on various substrates for the development of ZnO rods on epitaxial layers by fixing resynthesized nanoparticles as seeds. ZnO is less prone to surface damage by impact loading, and it is also reckoned that an increase in the surface territory at the interface provides a good chemical bonding to avoid delamination and enhance mechanical properties. It is dole out by functional groups such as the –OH group and –COOH group, generally made on the surface out of carbon strands, and such groups possess a strong affinity towards ZnO nanowires. A strong ionic bond will propagate while the polar group in the –COOH group of the WCF surface reacts with Zn2+ of nanowires [21]. Deka et al. [22] clarified that enhanced interfacial associations among the nanowires, WCFs, and polyester resin were the essential purpose for the discoveries.

Nonetheless, the cooperation of functions the chemical group presents on the carbon fiber amid the nanowires of CuO and the epoxy resin was a prime parameter in such a manner. Rai et al. [16] successfully grown ZnO nanostructures on WCF through hydrothermal technique and studied the impact of process parameters on the morphology and dimensions of ZnO. Even though the mechanical properties of the interfacial region can improve with the development of ZnO nanowires, such materials are significantly less prone to impact loading [23]. It is assessed that the properties of the materials can be increased due to expanding the interaction zone between nanostructures and polymer matrix by chemical interactions. Additionally, the load exchange can increase [24]. Functional groups such as hydroxyl and carboxyl are the most valuable groups that can be used to develop nanostructures on carbon fibers, which have a strong ability to produce nanowires. These groups form ionic solid bonds with the Zn2+ ion of the ZnO while reacting with the carboxylic acid of the carbon fibers [15]. Rai et al. [25] deposited CuO nanostructures to produce a nanostructured interphase that increased the interfacial strength of CFRP composites.

This paper investigates the solvothermal process on woven carbon fiber surfaces by controlled growth of ZnO nanostructures. The production of ZnO NSs with varied morphologies (nanowires, hexagonal nanorods, and nanoflowers) by treating WCF samples in seed and growth solutions in a hot air oven can be done by solvothermal process. By changing the concentration of ZnO, various morphologies of nanostructures can be developed on WCF. The developed morphologies of ZnO nanostructures on WCF surfaces were examined by FESEM and EDS spectra. Using the vacuum bagging method, the ZnO nanostructured WCF specimens were fabricated into laminates in an epoxy resin matrix. The Charpy impact test technique on the Impact tester, IT-30 (Make: FIE) machine, examined fabricated laminates' energy absorption and toughness.

MATERIAL AND METHODOLOGY

Hexagonal ZnO Nanorods were developed on bi-directional plain weaved carbon fiber after deposition of seed solution, and annealing was done to affix the seed layer on carbon fiber. Nanostructures were affected by seed layer concentration.

The pre-treatment of the seed layer increases the density and morphology of ZnO Nano features. Impurities have a crucial role in the growth and crystallinity of ZnO Nanorods. Seeding also affects the desired morphology that has to be achieved for the required application [16, 24]. Three significant aspects were considered: the amount of seeding, the salt concentration, and the length of the treatment. These parameters were varied in a planned series of studies, probably utilizing different combinations of seeding cycles, growth solution concentrations, and hydrothermal treatment periods. The outcomes of these experiments were noted and reviewed. Table 1 presents the results of the intended test series using the designated process parameters. The sample having ultrafine growth of ZnO nanostructures was taken for fabrication of composite models and further analysis. Laminates were prepared using the vacuum bagging method. In this fabrication method, the WCF on which the ZnO Nanorod was grown was placed on a mold plate. Vacuum bagged along with a vacuum line for injection and ejection of epoxy resins after fabrication of composite laminate, mechanical testing such as impact strength, tensile strength, elastic modulus, and in-plane shear strength were examined to understand their behavior for structural applications.

Table 1. Test conditions and their findings.

S.N.	Seed cycles	Salt concentration (mM)	Time (h)	Results
1	2	10	4	NG (No growth)
			8	NG
2	2	20	4	NG
			8	NG
3	2	30	4	NG
			8	NG
4	4	10	4	AG (Agglomerated growth)
			8	AG
5	4	20	4	AG
			8	LG (Little growth)
6	4	30	4	AG
			8	LG
7	6	10	4	AG
			8	LG
8	6	20	4	AG
			8	LG
9	6	30	4	LG
			8	LG
10	8	10	4	FG (Fine growth)
			8	UFG (Ultra-fine growth)
11	8	20	4	FG
			8	UFG
12	8	30	4	FG
			8	UFG

Solvothermal Process of ZnO Nanorods Synthesis

This method prepared the first seed solution followed by growth solution preparation. To prepare the seed solution, zinc acetate dihydrate (98%) and sodium hydroxide were used as received from suppliers. In this synthesis, initially dissolve 0.35 g of Zn(CH3COO)2.2H2O into 400 of C_2H_5OH and continuously mix at 750°C for 40 min and similarly under an identical condition dissolve 0.2g of NaOH into 100 ml of C_2H_5OH. These prepared solutions were integrated, and an additional 300ml of ethanol was also incorporated using magnetic stirring for 45 minutes to make 800ml of seed solution. The following reactions for seed solution are described:

$$Zn^{2+} + 4OH^- \leftrightarrow Zn(OH)_4^{2-}$$

$$[Zn(OH)_4]^{2-} \leftrightarrow ZnO_2^{2-} + 2H_2O$$

$$ZnO_2^{2-} + H_2O \leftrightarrow ZnO + 2OH^-$$

Next step for preparation of growth solution is mixing of zinc nitrate hexahydrate ($Zn(NO_3)_2.6H_2O$) and Hexamethylenetetramine (HMTA, $C_6H_{12}N_4$) into deionized water in 1:1 molar ratio using magnetic stirrer. The pH values of mixtures were maintained in the range of 8-10. Solution was prepared in varied molar concentration of 0mM, 10mM, 20mM, and 30mM. The chemical reactions involved for growth solution were as follows:

$$C_6H_{12}N_4 + 6H_2O \leftrightarrow 6HCHO + 4NH_3$$

$$C_6H_{12}N_4 + Zn^{2+} \leftrightarrow [Zn(C_6H_{12}N_4)]^{2+}$$

$$NH_3 + H_2O \leftrightarrow NH_4^+ + OH^-$$

$$[Zn(NH_3)_4]^{2+} + Zn^{2+} \leftrightarrow ZnO + 4NH_3 + H_2O$$

It can be concluded from these chemical reactions, the concentration of OH^- plays a vital role in controlling the development of ZnO nanostructures. The process of ZnO development starts with the precipitation of $Zn(OH)_2$ (Wulfingite), which then dehydrates to produce Wurtzite ZnO.

Preparation of ZnO Nanorods on WCF

Three key variables, which are the seeding cycle, molar concentration, temperature, and period of the solvothermal technique of ZnO development on WCF, were considered while defining the design of the experiments. The findings of each investigation are tabulated in Table 1, and the experiments resulting in ultrafine growth of ZnO are considered for further studies. In this work, the effect of molar concentration is studied under 2, 4, 6, and 8 number of seeding cycle, 90°C temperature for 4 and 8 hours of hydrothermal treatment. Different molar concentrations of ZnO growth solution, such as ten mM, 20 mM, and 30 mM, were used. A bi-directional woven carbon fiber was cut into 18cm×18cm pieces and pre-treated with an ethanol-acetone solution. Now, the treated samples were dried at 90°C in a hot air oven for 15 minutes, which allowed the removal of dirt, contaminants, and impurities. The samples were then immersed into previously prepared seed solution for 15 minutes after annealing at 120°C to remove the solvent. This seeding process was performed in 4 cycles, 15 minutes of soaking and 15 minutes of drying. The seed-treated fiber was dipped into a growth solution and kept in a hot air oven furnace at 90°C for 4 hours. After growth treatment, samples were withdrawn and splashed with de-ionized water for 10 minutes to stop the further development of ZnO.

At last, ZnO-grafted WCF samples were dried for 24 hours in ambient conditions. The molar concentrations of growth solution were varied from 0mM to 30 mM to study morphological variations and their effect on the impact resistance of laminates. A schematic diagram for preparation of ZnO/WCF/Epoxy laminated composite using vacuum bagging method as illustrated in Figure 2.

Mechanical Testing of Composite

The impact energy absorption of the composite laminate was tested with the help of an impact test system Model: IT-30 (Make: FIE), which has a servo-electric system for accurate measurement through the Charpy impact test method. The test system had a capacity of 10 tonnes, a dimension of 55 mm×10 mm×2 mm, with a cross-head velocity of 3.5m/s. The data was examined, and results were plotted as impact energy absorption of fabricated laminates. Furthermore, composite samples were tested against the tensile loading on an Instron micro-UTM setup (Model-Instron 8801, UK) having 100 N load cell and strain rate of 1 mm/min. The tensile strength, elastic modulus, and in-plane shear strength of the three samples in each molar concentration were tested, and their average value is plotted in the graphs.

RESULTS AND DISCUSSIONS

ZnO Nano Rods Growth

This paper studied the crystalline growth of ZnO nanostructures through a solvothermal process. Hydroxyl ion concentration plays a vital factor in the development of ZnO nanorods. The morphologies started changing from nanowires to nanorods and nanoflowers while increasing the molar engagement, as depicted in Figure 3. Initially, WCF was made to react with seed solution on which fiber was coated with ZnOOH^-. A growth solution facilitated the growth of ZnO nanorods on

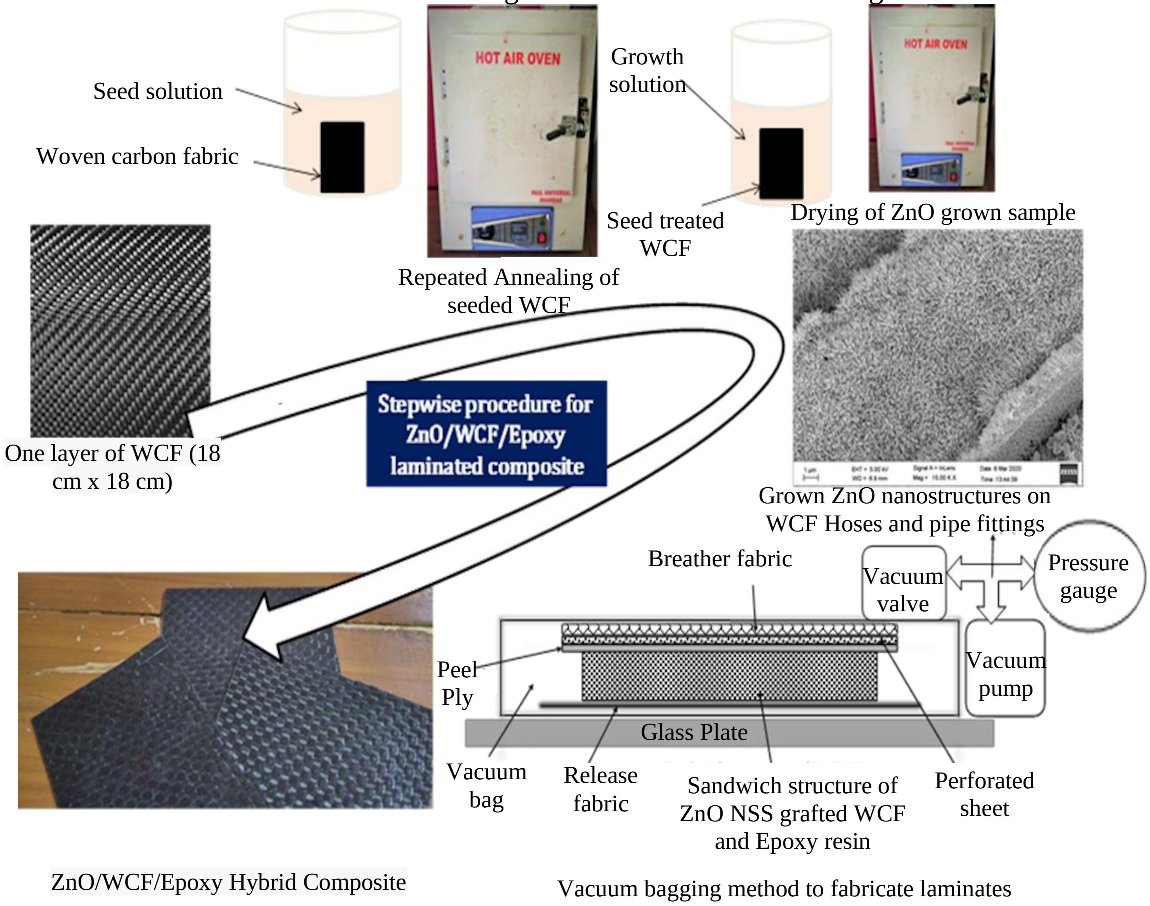
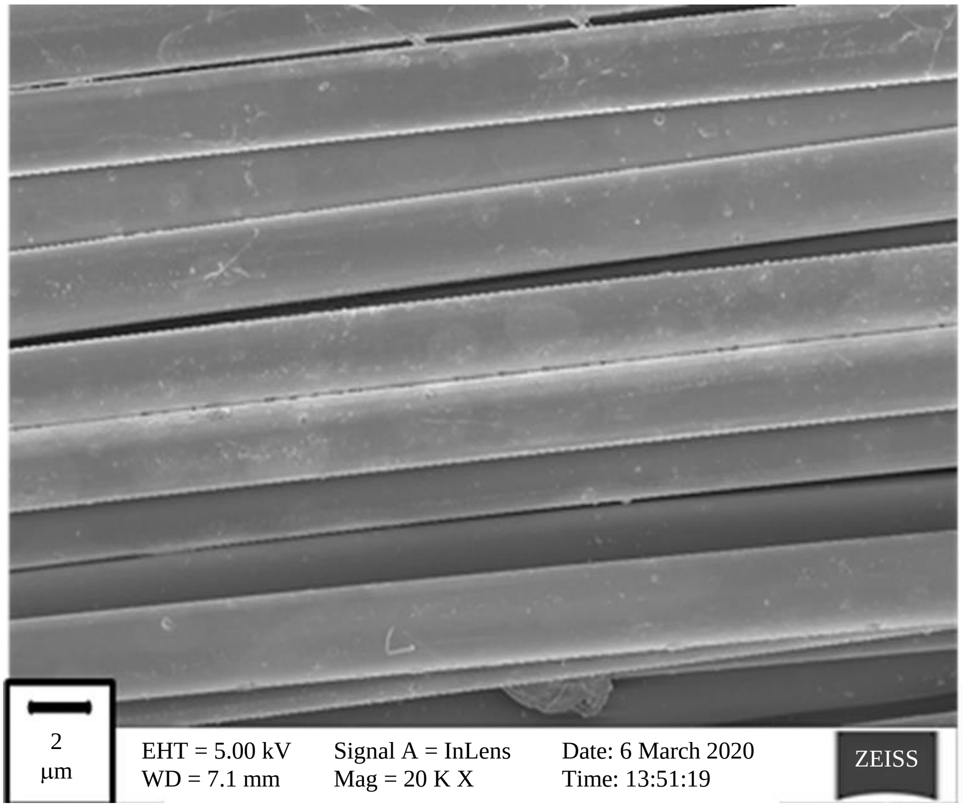


Figure 2. Fabrication of ZnO/WCF/Epoxy hybrid composites.


2
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EHT = 5.00 kV
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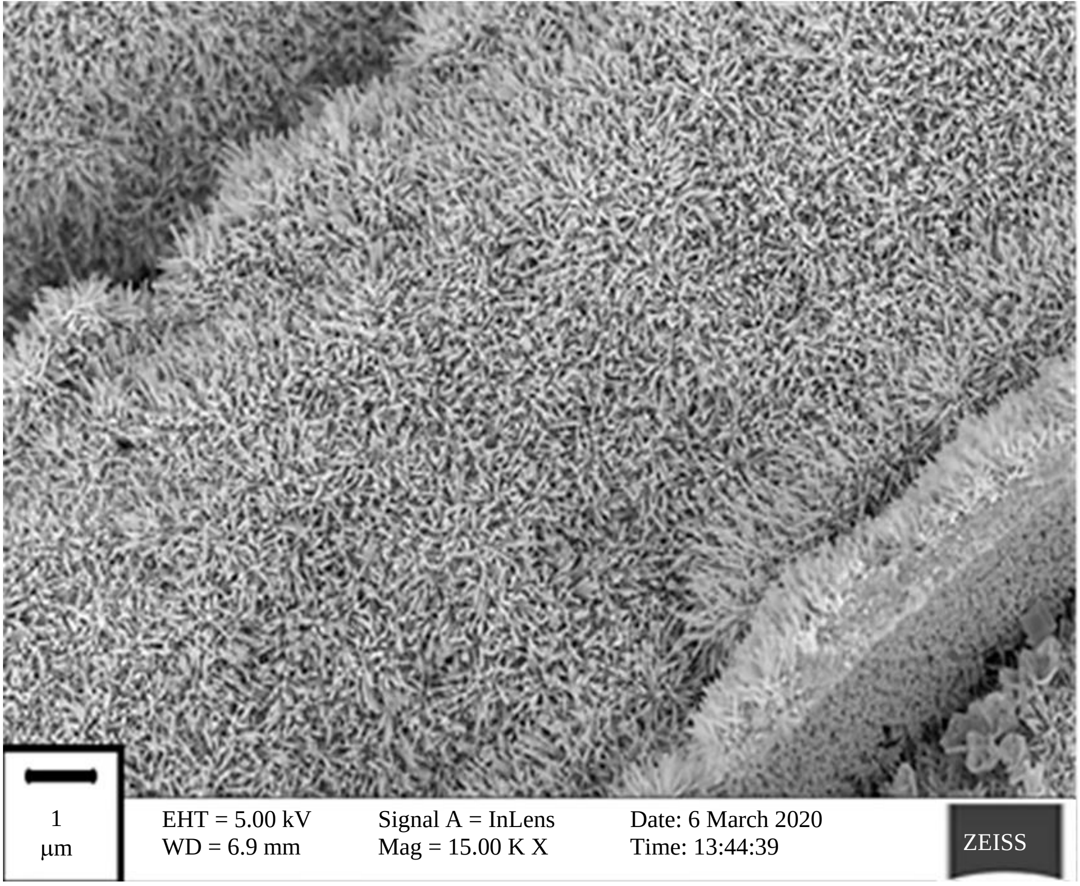
(a)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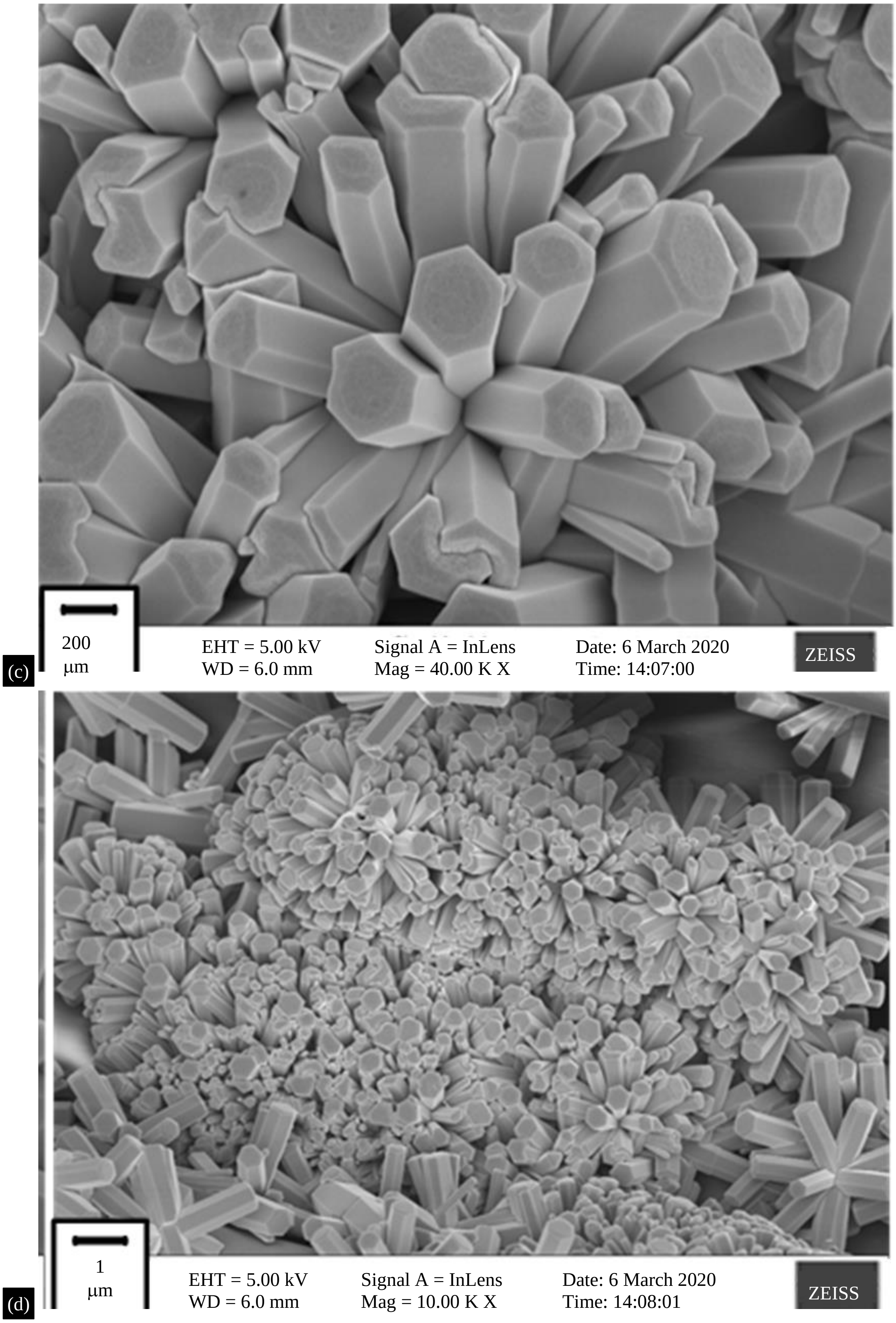
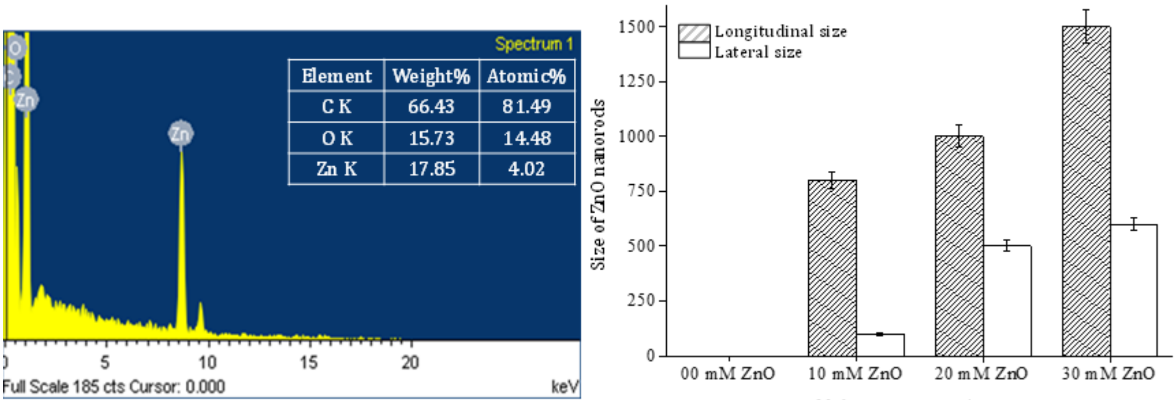


Figure 3. Solvothermally synthesized ZnO on WCF at varying concentration of (a) 0 mM (b) 10 mM (c) 20 mM and (d) 30 mM.



Element	Weight%	Atomic%
C K	66.43	81.49
O K	15.73	14.48
Zn K	17.85	4.02

(a)

(b)

Figure 4. (a) EDS Spectra and (b) Size distribution of synthesized ZnO on WCF

WCF. At lower concentrations, the reaction rate and growth rate are slow. The direction of nanorods was along the c-direction, which is the same as previously published work [26]. Energy-dispersive X-ray spectroscopy analysis of samples reveals the presence of Zn and O on WCF strands, as depicted in Figure 4(a). The EDS spectra demonstrated that the ZnO nanorods are pure and contain no impurities. The physical dimension of grown nanorods at different molar concentrations of growth solution is illustrated in Figure 4(b). The growth rate of nanorods in the axial direction was affected by raising the molar attention of $Zn(NO_3)2.6H_2O$ and HMTA. Still, the nucleation of micron-size rods also led to non-uniformity. The total duration of development of ZnO nanorods plays a crucial role in the solvothermal technique. As the concentration of seed solution and growth solution was increased, the surface density was also changed along with the physical dimension.

Impact Test

The impact test of the hybrid composite was examined by an impact tester using the Charpy impact test method. Three laminated composite samples of each concentration were fabricated and tested for impact loading. The average value of all their test results was analyzed and plotted in this section. The vital fiber/matrix region was found when five layers of ZnO-grown WCF fabricated the samples as reinforcement with epoxy resin as the matrix. The presence of ZnO nanorods (10mM) improves the impact strength of the fabricated nanocomposites by 11% compared to plain WCF composite—the impact energy absorption of nanocomposite increases by increasing the molar concentration of the ZnO nanorods. Due to the rise in the effective surface area of the bond between matrix and fiber, this study shows that ZnO growth can lower the delamination of composite materials. With the increment of the molar concentration, the length of the nanorod increases, which provides a large surface area of bonding between the matrix and fiber. The chappy impact test study of fabricated composites and their energy absorption capacity is illustrated in Figure 5. The highest energy absorption capacity of faked composite is 2.5 Joule, which is 39% more than bare WCF composites due to the most increased interfacial interaction of hexagonal ZnO nanorods with another hexagonal nanorods, WCF and Epoxy resin. Fiber breakage, fiber pullout, crack debris, and other failure modes such as ZnO nanorods pullout and ZnO nanorods breakage define sample fracture mechanisms. The fracture point mainly offers fiber-breaking information. The cracking interaction energy was estimated to be between the ZnO nanorods and the surface of the WCF up until the fracture point. Additionally, the WCF absorbed delamination energy. Interactions between ZnO nanorods and ZnO growing area zones on the surface of the WCFs resulted in variances in impact energy absorption.

Figure 5. (a) Charpy impact test setup (b) Fabricated ZnO-WCF/Epoxy resin hybrid composites and (c) Impact energy absorption of laminated composite samples.

Tensile Test

The tensile test of the hybrid composite was examined on Instron micro-UTM to study their tensile strength, elastic modulus, and in-plane shear strength. Three laminated composite samples of each concentration were fabricated and tested for accurate results. The average value of all their test results was analyzed and plotted in this section. The vital fiber/matrix region was found when five layers of ZnO-grown WCF fabricated the samples as reinforcement with an epoxy resin matrix. The presence of ZnO nanorods (10mM) improves tensile strength, elastic strength, and in-plane shear strength of the fabricated nanocomposites by 17%, 11%, and 3%, respectively, compared to plain WCF composite. Because of enhanced interfacial interaction and chemical bonding of fibers with epoxy resins, this study shows that delamination of the composites can be minimized by developing ZnO nanostructures. The micro-UTM setup for tensile testing based on ASTM D3039 standard along with the fractured composite sample is illustrated in Figure 6. Mechanical properties of the fabricated composites increase on increasing the molar concentration of ZnO nanostructures because of a high content of nanorods and their interaction with polymer matrix. The tensile strength and elastic modulus of composite samples were increased with increasing molar concentration. They achieved the highest variations of 38% and 32%, respectively, compared to plain carbon fiber epoxy composites, as depicted in Figure 6(c,d). The in-plane shear strength of the composite samples was performed at ± 45° to the fiber direction. The test specimens were prepared by cutting the laminate at 45° to the fiber direction and served as per the ASTM D3518 standard. The highest value of in-plane shear was achieved by growing 30 mM molar concentrating of ZnO nanorods on plain woven carbon fiber as illustrated in Figure 7. There is approximately 6% increment in the in-plane shear strength by adding 30 mM of ZnO in the plain carbon fiber polymer composites. The test specimen, volume fraction measurements, and mechanical characteristics of the ZnO nanostructures in the developed nanocomposites are illustrated in Table 2. It can be concluded that the addition of ZnO nanostructures in the carbon fiber polymer composites is beneficial to achieve tailored properties of the composites for structural applications.

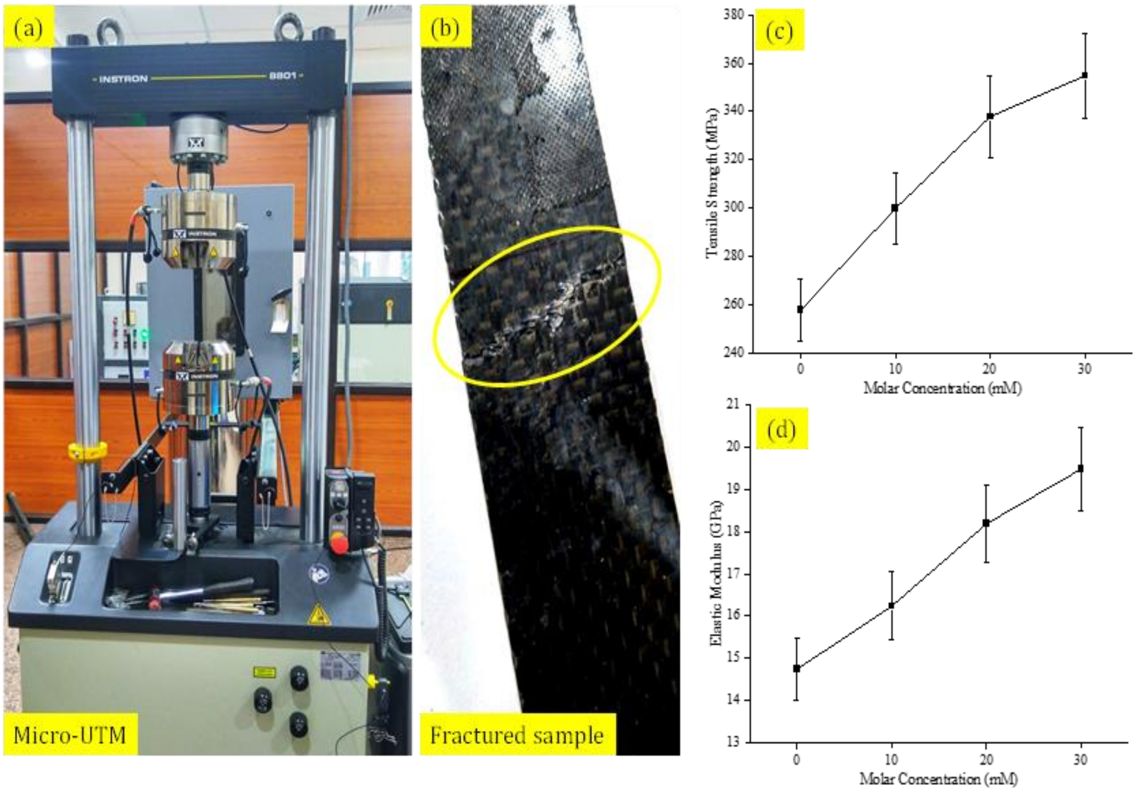


Figure 6. (a) Instron micro-UTM set up for tensile testing, (b) fractured composites sample, (c) variation of tensile strength and (d) elastic modulus with ZnO molar concentration.

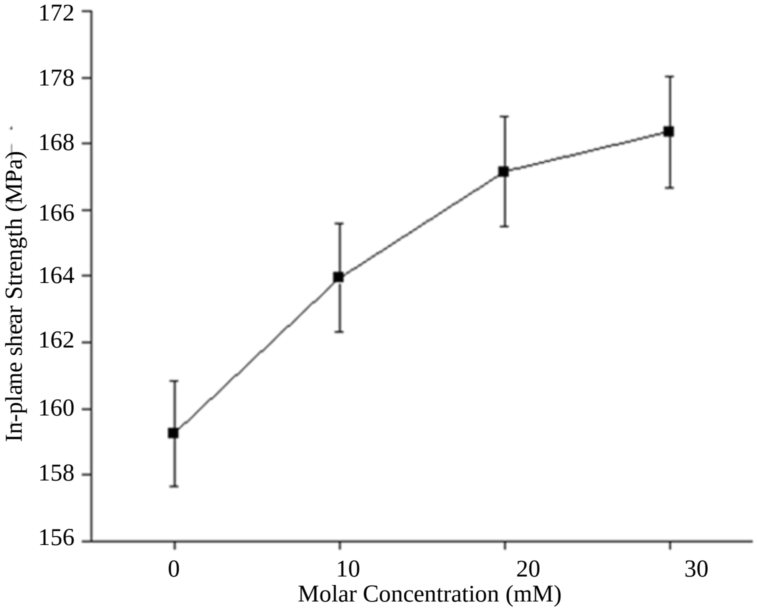


Figure 7. Variation of in-plane shear strength with ZnO molar concentration of composites.

Table 2. Fabricated test specimen and their mechanical characteristics

S.N.	Sample category	Fiber volume	ZnO volume	Void	Tensile	Elastic	In-plane shear

		fraction (%)	**fraction (%)**	**content (%)**	**strength (MPa)**	**Modulus (GPa)**	**strength (MPa)**
1	00 mM ZnO/WCF/BPA Epoxy resin hybrid composite	≈ 48.76 ± 0.80	0.0	≈ 10.84	257.78	14.75	159.42
2	10 mM CuO/WCF/BPA Epoxy resin hybrid composite	≈ 48.76 ± 0.80	≈ 1.62	≈ 8.86	298.69	16.23	164.20
3	20 mM ZnO/WCF/BPA Epoxy resin hybrid composite	≈ 48.76 ± 0.80	≈ 2.64	≈ 6.37	330.31	18.59	167.48
4	30 mM CuO/WCF/BPA Epoxy resin hybrid composite	≈ 48.76 ± 0.80	≈ 3.92	≈ 4.72	354.01	19.23	169.23

CONCLUSIONS

The solvothermal technique was used to develop hexagonal zinc oxide nanorods on the WCFs' surface. Morphological behavior at different concentrations of precursor chemicals was examined through FESEM results. Following are the significant findings of the current work.

- It can be concluded that as the molar concentration increased, the nanorods' size and morphology started varying. ZnO nanowires were developed at lower concentrations, and as concentration changed, they appeared in hexagonal nanorod structures.
- The low seeding cycles and low growth durations cause poor growth of the ZnO nanostructures on the WCF surface.
- Composites of reinforced bare WCF have poor impact behavior because of their brittle nature. Despite that, findings of experimental investigations stipulate that the growth of ZnO nanorods enhances the impact energy absorption due to the cross-linking of nanorods of ZnO at the interface of matrix and reinforcement.
- On higher concentrations (30mM) for the same growth duration, the highest mechanical properties were achieved due to the large amount of ZnO crystals on WCF and their interlocking with the polymer matrix.
- Plain WCF, composite failure mechanism, is explained as fiber breakage, fiber pullout, and crack debris; however, new failure modes such as ZnO nanorods pullout and ZnO nanorods breakage developed due to ZnO nanorods grown on WCF that define fracture mechanism of hybrid composite samples.
- Compared to bare WCF-reinforced polymer composites, the highest impact strength, tensile strength, elastic modulus, and in-plane shear increment were achieved at 30 mM ZnO molar concentration. Their percentage increment is 39%, 38%, 32%, and 6% respectively.
- It can be concluded that the growth of ZnO nanostructures on the WCF surface is a promising method of functionalization to achieve desired materials properties for structural applications.

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