Experimental Investigation on Mechanical Properties of Carbon Nanotube-Reinforced Epoxy Composites for Automobile Application

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Carbon nanotubes are established as a superior form of carbon. These have superior characteristics in terms of mechanical and chemical properties when compared to the other fibres available. High-strength fibres can be employed in a composite in a short form and mass-produced to fulfil high demands in composite applications. These composites can meet the strength requirements of nonstructural and structural components in a wide range of industries. Because of their light weight and excellent strength-to-weight ratio, these composites can be used in a wide range of applications. With Young’s modulus as high as 1 TPa and tensile strength up to 63 GPa, they are among the stiffest and strongest fibres. There is currently a lot of interest in using carbon nanotubes in a matrix to take advantage of these features. There have been a variety of polymer matrices used, and nanotube/ceramic and nanotube/metal composites are gaining popularity. The study of these materials is an ongoing process, as researchers and design engineers have yet to realize their full potential. Carbon nanotubes (CNTs) are used in this study to create the composite with the resin. The percentage of CNT used as a filler material in the composite is varied from 1 to 4 percent, with the best percentage chosen for optimal mechanical properties.

1. Introduction

Carbon nanofibres (CNF) and carbon nanotubes (CNT) have had increasing potential in recent decades. Researchers from all around the world are attempting to apply the better qualities of these nanocomponents to a variety of applications [1]. The application range between biosensors and batteries of the new age CNTs has outstanding mechanical properties due to the two-dimensional arrangement of carbon atoms in a graphene sheet [2]. Due to this, massive out-of-plane distortions will happen while the strength of carbon-carbon in-plane bonds keeps the graphene sheet extremely strong against any in-plane distortion or fracture [3]. CNTs have a high aspect ratio and excellent electrical
and physical properties [4]. CNTs also have a high aspect ratio [5], as well as good electrical and physical properties [6]. Because of its unique characteristics, carbon nanotubes have been used in nanoscale electron devices namely field emission displays [7], atomic force microscope tips, and hydrogen storage cells [8]. According to both experimental and theoretical research, CNTs (both single walled and multiwalled) have remarkable mechanical properties, such as a high aspect ratio, lightweight [9], high thermal properties [10], excellent waviness characteristics, high melting point, low density, good electrical conductivity [11], larger surface area, remarkable biological properties, excellent hydrogen storage, and higher corrosion resistance [12]. Because of its symmetric structure, a typical CNT has a hexagonal arrangement of carbon atoms in the shape of tubes and offers extraordinary features [13]. Their performance is fully determined by their helical nature, and as a result, they operate as either a semiconductor or a metal [14].

Delogu et al. observed that a hybrid composite material can be formed by compression moulding of advanced sheet metal (ASMC) and unidirectional (UD) prepregs [15]. Ma et al. investigated the impact of nylon6 on cure behavior and mechanical properties of CF-nylon6 epoxy composites, produced by the hand lay-up process [16]. Kupski et al. manufactured two distinct configurations of the carbon/epoxy system; tests are carried out under quasistatic tension and it was found that mechanical properties vary with the thickness and length of the overlapping ply blocks [17]. Fotouhi et al. observed that in the case of UD all-carbon epoxy hybrid composites, delamination occurs from the outer high strength carbon layers subjected to uniaxial tensile loading [18]. Yogeshwaran et al. prepared a laminated CF-reinforced epoxy composite by hand lay-up and autoclave process, and the residual stresses are calculated using the incremental slitting method [19]. Based upon the build-up of the nanotubes, these are classified as two types [20], the single-walled carbon nanotube and the multiwalled carbon nanotube [21]. The single-walled carbon nanotube has one layer of carbon atoms as the wall of the tube, whereas in the multiwalled carbon nanotube, there are multiple layers of carbon as walls of the nanotube [22]. These multiwalled nanotubes can be considered as single-walled nanotubes inserted in one another [23]. Depending on the number of layers and the shape of the carbon tubes, the properties of the nanotubes vary [24]. There are numerous research publications on the synthesis of CNTs using diverse methodologies [25]. Currently, a wide range of carbon precursors, catalyst nanoparticles, carrier gases, and substrates may be used to produce CNTs in various crystallographic configurations [26].

CNTs have been used as extremely strong nanoreinforcements for composites, which possess extremely high strength with low weight and moderate electrostatic discharge properties [27]. Due to its highly adhesive, low weight, and good chemical resistance, epoxy-based composites are increasingly used in aerospace and car manufacturing as structural components [28]. The relatively weak mechanical characteristics of epoxy have however prevented its application in components which require high mechanical stability [29]. Recently, the use of MWNT as the polymer matrix filler has attracted considerable concern due to its mechanical, thermal, and electrical characteristics. Epoxy composite nanotubes were manufactured through different processes of purification and dispersion [30]. The amount of carbon nanotubes (CNT) in the present study composite varies from 1 to 4% of the total weight of the phenolic resin. This research is aimed at carrying out the fraction of CNT to use in CNT-based epoxy composites to get the greatest mechanical performance. The tensile strength and toughness of the prepared samples are then tested, and SEM micrographs are obtained to study the characteristics of CNT composites.

2. Methodology

Multiwalled CNTs have been procured from Ilijin Nanotech Co. The chemical vapour deposition process synthesizes this MWCNT. The procured MWCNT have an average diameter of 25 nm, and the size is 10 microns. The SEM micrograph of MWCNT is shown in Figure 1. These MWCNT are cleaned for removing carbon particles, graphite, and other particles. A solution of 65% H₂SO₄/HNO₃ with 3:1 ratio is used for 30 min at 100°C to purify CNT.

CNTs with a large L/D ratio are bound to agglomerate together and make it difficult to disperse uniformly in a polymer matrix. The properties of the composite cannot be enhanced as the CNTs are not uniformly distributed, and on top of that, the agglomerated CNT acts as a defect and initiates a crack in the composite. SnCl₂ and PdCl₂ solutions were used to sensitize and activate the dispersed CNTs at room temperature. The CNTs were then filtered and thoroughly washed in distilled water after being pretreated. Before creating the CNT composite, CNT powder was mixed with polyglycol-dissolved phenolic resin. The phenolic resin was a thermosetting polymer created from phenol and formaldehyde with a catalyst of ammonia. The CNT weight ratio in the billets varies between 1 percent, 2 percent, 3 percent, and 4 percent. Multiple billets are prepared for each % of CNT without modifying the specifications. To obtain a solidified billet sample, the combined CNT and phenolic resin is cured in an oven at 160°C. Microhardness is determined using a microindenter, tensile strength is computed using UTM, and SEM micrographs are generated using a
FE-SEM. XRD analysis is used to analyze the chemical composition of the CNT composite.

3. Results and Discussion

3.1. Micrographs.
To determine the CNT dispersion in the composite, the generated CNT composites are examined using a FE-SEM apparatus. The SEM micrographs that have been developed are shown below. It signifies that the CNTs have been evenly distributed across the matrix. Due to ultrasonic mixing before adding the CNTs to the resin, all of the CNT content percentages are dissolved uniformly in the composite. Milling before combining with resin dropped the CNT ratio and enhanced the wettability of the fibres [31]. For any of the produced composites, no aggregation of CNT in the matrix has been discovered. There is no fracture of the matrix at the interface of the CNT and matrix in the composite. The CNTs have a good bond with the matrix, and there is no slipping of the fibre with the matrix.

The SEM micrographs of all the composites have shown the same pattern without any significant difference to observe. The section that has been sliced for the composites has shown CNT at the cross-section and has a similar tendency of interaction between CNT and the matrix. Hence, a single micrograph is presented for all the four grades of the composites tested in Figure 2. The matrix is also uniform throughout the composite without any air bubbles trapped or any cracks detected. There is no slippage between CNT and the matrix. As the ultrasonic mixing is employed in the mixing of CNT, there is no agglomeration of CNT detected in the composite, and hence, the CNT is uniformly distributed. This gives the composite fine strength.

3.2. XRD Analysis.
The graphitic-like carbon peaks can be seen in the CNT composite XRD diffraction pattern in Figure 3. The CNT composite closely resembles CNTs in terms of XRD patterns, implying that the graphite-like structure is conserved after the CNT powder is produced with a polymer binder and then carbonized at high temperatures. The contribution of polymer-derived carbon in the composite is undetectable in the XRD diffraction spectrum. Due to the surface graphitization of thermosetting resin during carbonization results in a graphite-like layer separate from the

### Table 1: Microhardness (HV) of CNT composites.

| Sample | Trial 1 | Trial 2 | Trial 3 |
|--------|---------|---------|---------|
| CNF 1% | 29.5    | 25.2    | 25.4    |
| CNF 2% | 33.4    | 34.5    | 29.9    |
| CNF 3% | 34.6    | 36.4    | 36.6    |
| CNF 4% | 38.4    | 38.8    | 40.1    |

Figure 2: SEM micrograph of CNT composite.

Figure 3: XRD graph of the CNT composite.

5.0 kV 9.1 mm ×100 k SE (M) 5.0 kV 9.1 mm ×80.0 k SE (M)
bulk glassy carbon. There is no mix-up of CNT and matrix polymer during curing, and no unwanted material has been observed. The XRD diffraction graphs obtained for the all samples developed gave the similar pattern and have no significant difference to be discussed individually, and hence, only one graph is provided for all the samples. The peak is consistent for all the samples, and no peak of the secondary carbon is detected consistently for all the samples detected.

3.3. Mechanical Properties

3.3.1. Hardness. When a load is applied to a softer material, the substrates deform elastically, resulting in underreporting of hardness. As a result, the matrix and CNT collective hardness was obtained in this situation, significantly reducing the composite characteristics. Every sample has been put through a series of tests, yielding an average hardness value. The composite hardness is determined using a microindenter and a 100 g force for each indentation. Shimadzu's Vickers indentation was employed for the evaluation. The hardness of the composites increases as the percentage of CNT in the composite increases. The hardness of the 1% CNT composite is lower than the other composites, whereas the hardness of the 4% CNT composite is higher. The hardness values obtained for each sample are listed in Table 1. For a 15-second indentation time, all values are recorded. The hardness of CNT composites is seen in Figure 4. The variation in hardness values can be explained by the fact that the distribution of CNT at the surface layers of the composite is high for composites with a higher CNT percentage; hardness is higher for composites with a 4-percent CNT percentage and gradually decreases for composites with a lower CNT percentage.

3.3.2. Tensile Strength. Table 2 shows the tensile strength of the composites assessed by UTM. Compared to other produced composites, the composite with a higher CNT concentration has a higher tensile strength value. Because the connection between matrix and CNT is sufficient and there are no vacancies, the higher CNT in the composite absorbs the load. The tensile strength of CNT composites is shown in Figure 5. When the CNT concentration in the composite is increased from 1% to 4%, the tensile strength of the composite increases by 27%. When CNT is increased from 1% to 2% in the composite, the percentage increase is 13 percent. When CNT is increased to 3 percent from 2 percent, the percentage for tensile strength increment is reduced to 9%. This indicates that increasing CNT content enhances tensile strength, but the increment level decreases over time. Figure 6 shows the graphical presentation of the tensile strength of the CNT composites developed.

3.3.3. Elastic Modulus. The elastic modulus of the samples has been determined while testing for the tensile strength of the samples on UTM. While the tensile strength has increased almost linearly after addition of MWCNT, at the same time, it has reduced the elastic modulus of the composite. The least modulus has been registered for the 4% CNT composite while the highest modulus is registered for the 1% CNT composite. The total measurements of the modulus for the samples are given in Table 3 below. The highest modulus registered is for 1% of CNT at 1025 MPa and the lowest recorded for 4% CNT is 952 MPa. There is a total of 7% reduction in Young’s modulus when CNT is increased from 1% to 4%. The graphical presentation of elastic modulus is given Figure 5.

4. Conclusions

Phenolic resin with different wt% of multiwalled carbon nanotube composite was fabricated successfully. Among the composites, the one with higher CNT content has
superior properties in terms of mechanical properties. The superior mechanical properties of CNT have influenced greatly testing composites.

(i) The XRD examination confirmed that the composites sample contains no impurities or other materials. CNT and resin produced XRD peaks

(ii) The tensile strength increases as the CNT concentration increases, but the rate of increase decreases over time. CNT with 4 wt% composite tensile strength results were increased 27% compared to 1% of CNF composite

(iii) The hardness of the composite is increased when the CNT content is increased from 1% to 4%

(iv) The elastic modulus of the composite is reduced from 1% CNT to 4% CNT by over 7%

(v) The gradual increase in the CNT content in the composite will increase the strength of the composite and hence is useful in the automotive industry

Data Availability

The data used to support the findings of this study are included in the article. Should further data or information be required, these are available from the corresponding author upon request.
Disclosure
This study was performed as a part of the Employment of Mettu University, Ethiopia.

Conflicts of Interest
The authors declare that there are no conflicts of interest regarding the publication of this paper.

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