Enhanced Flexural Strength of Tellurium Nanowires/epoxy Composites with the Reinforcement Effect of Nanowires

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Abstract. Investigating the mechanical properties of polymer nanocomposite materials has been greatly increased in the last decade. In particular, flexural strength plays a major role in resisting bending and shear loads of a composite material. Here, one dimensional (1D) tellurium nanowires (TeNWs) reinforced epoxy composites have been prepared and the flexural properties of resulted TeNWs/epoxy nanocomposites are studied. The diameter and length of the TeNWs used to make TeNWs/epoxy nanocomposites are 21±2.5 nm and 697±87 nm, respectively. Plain and TeNWs/epoxy nanocomposites are characterized by X-ray diffraction (XRD), thermogravimetric analysis (TGA), and differential thermal analysis (DTA). Furthermore, significant enhancement in the flexural strength of TeNWs/epoxy nanocomposite is observed in comparison to plain epoxy composite, i.e. flexural strength is increased by 65% with the addition of very little amount of TeNWs content (0.05 wt.%) to epoxy polymer. Structural details of plain and TeNWs/epoxy at micrometer scale were examined by scanning electron microscopy (SEM). We believe that our results provide a new type of semiconductor nanowires based high strength epoxy polymer nanocomposites.

1. Introduction

Development of advanced composite materials is one of the attracted areas of research to scientists and engineers due to their significant technological importance and excessive demand for competent working and superior mechanical properties [1]. Combination of soft materials such as polymers and nanostructured materials is an active example to advanced composite material. Development of the stronger, tougher and lightweight polymer matrix composites (PMCs) to support complex shaped structures like aircraft, automotive structures, and large wind turbine blades is one of the prime challenges for the composite engineers. PMCs are newly emerging advanced structural composites whose usage has been greatly increasing during the last decade due to their exceptional mechanical, thermal and chemical properties with minimized weight [2].

Among many available polymers for PMCs, epoxy has been the choice for several structural applications due to its properties of irreversible cure, strong mechanical, thermal properties as well as chemical resistance [3]. These properties of epoxy can be further improved by the reinforcement of inorganic nanofillers, called epoxy nanocomposites. Optical, thermal, electrical, magnetic and anticorrosive properties of the epoxy got improved because of the inorganic fillers [4-11]. Several nanomaterials such as Ag [4], Cu [5] nanoparticles, Fe₃O₄ nanoparticles [6], TiO₂ nanoparticles [7,8],
carbon nanostructures [9-11] and etc. have been used as nanofillers to improve the properties of conventional epoxy composites. For example, thermal and electrical conductivity of the epoxy nanocomposites are improved by the use of silver [4] and copper nanoparticles as fillers [5]. Magnetic nature in epoxy polymer has been successfully introduced by the reinforcement of magnetic nanoparticles as fillers [6]. Semiconducting properties and the subsequent applications in the area of photovoltaics have been imputed by the insertion of TiO$_2$ nanoparticles as well as TiO$_2$ nanotubes [7, 8] in epoxy polymers. Furthermore, carbon nanotubes based epoxy composites are extremely useful in light weight and high strength applications [9-11].

On the other hand, nanowires (NWs) are the subject of recent research began in 1998. Morales and Lieber [12] demonstrated for the first time the bottom up synthesis of uniform single-crystal Silicon and Germanium 1D nanostructures. Most of the NWs growth process is relatively simple as a result they occupied a large portion in nanoscience. 1D nanostructures are no longer only at the testbeds but excellent applications as a result of their unique dimensionality, size, electronic, mechanical, and optical properties [13, 14]. The unique applications of NWs are extended toward advanced polymer nanocomposites. Carbon nanotubes are being used extensively to improve the mechanical properties of epoxy polymers [14, 15]. Other 1D nanostructures such as copper silver, nickel, Bi$_2$Te$_3$, chitin (a long chain polymer) NWs has been used to improve thermal, electrical conductivities with enhanced mechanical strength in epoxy composites [16-20]. These studies showed that the uniform reinforcement of one dimensional nanomaterials in the polymer matrix can substantially improve the conductivity, absorption energy, toughness, elasticity, and strength of the polymer material. Furthermore, the advanced research involves the structural control by manipulating the arrangement of the NWs within the epoxy polymer matrix [10]. Mostly the vertical and horizontal arrangements of NWs in epoxy composites are controlled by the interplay between the magnetic nanowires the external magnetic field. Due to the peculiarity over spherical nanostructures, these NWs can be excellent nanofiller materials to create good interfacial bonding between nanowire-polymer interfaces. This good interfacial bonding is crucial to enhance the mechanical and thermal properties of polymer nanocomposites. As a result, these nanocomposites are widely useful in defence, automobile and aero applications [21].

In our current study, we specially focused on flexural properties of semiconductor nanowires reinforced epoxy composites. In particular, TeNWs are selected as nanofillers for the following reasons: Te is well-known p-type narrow band-gap semiconductor with a direct band gap of 0.35 eV at room temperature. This unique electronic structure of TeNWs offers potential candidature for photoconductivity, nonlinear optical response, high thermoelectric performance and piezoelectric responsive materials. TeNWs and their derivatives are also suggested as platforms for gas sensing probes, optoelectronic devices, photonic crystals, field-effect devices, self-developing holographic recording devices, radiative cooling devices, topological insulators, sensors, and magnetic memories [22-28]. In view of the importance of this material, in our previous work, we focused on the reinforcement effect of TeNWs on the impact and tensile strength of epoxy composites. Our previous results shows the impact and tensile strengths are increased by 100 % and 10.6 % with the addition of very little Te NWs content (0.05 wt. %) [29].

Here, we report the synthesis and flexural properties of TeNWs reinforced epoxy composites. The synthesis of 1D TeNWs is facile, highly reproducible and possible to get them in large quantities. The growth of Te seed in one direction is due to its unique crystal structure. Te maintains a polymeric structure, consisting of zig-zag hexagonal lattice chains of Te atoms. The distance between each adjacent chains are more than covalent bond and less than van der Waals interaction. Overlapping between the antibonding orbitals and the lone-pair orbitals on adjacent chains induces the inter chain interactions. Above reasons helps to grow Te in one-dimensional structures [30]. Several synthetic protocols such as thermal deposition, hydrothermal [25], microwave-assisted [26], and biomolecule-assisted [27] and chemical vapour deposition [28] are available for the synthesis of TeNWs. Some of the synthetic methods need harsh conditions such a high temperatures. However,
for facile, monodisperse and high-yield synthesis of TeNWs, we used a room-temperature protocol reported by Lin et al [22] and the follow-up protocols [23].

2. Experimental section

2.1. Materials

The matrix material used is epoxy system (Araldite LY556) and the curing agent is Aradur HY951 by Huntsman, India. TeNWs are synthesized at ambient laboratory conditions. All the chemicals used for the synthesis of TeNWs are commercially available and are used without further purification. Sodium Dodecyl Sulfate (SDS, C12H25O4SNa, 99%) is obtained from Acros. Tellurium dioxide (TeO2, 99.9%) powder by Alfa Aesar. Hydrazine mono hydrate (N2H4·H2O, 99-100%) by SD Fine Chemicals, India. Deionized water is used throughout the experiment. All glass wares are thoroughly cleansed with aqua regia (3:1vol% of HCl:HNO3), rinsed with plenty of deionized water, and then dried in oven at 120 °C prior to use.

2.2. Instrumentation

UV-vis spectra are measured with a Perkin Elmer Lambda 25 instrument in the range of 200-1100 nm. Transmission electron microscopy of TeNWs is carried out with a JEOL3010, a 300kV instrument. The samples are drop cast on carbon-coated copper grids and allowed to dry under ambient conditions. Scanning electron microscopic (SEM) analyses is done in a FEI QUANTA-200 SEM. For measurements, samples are drop-cast on an indium tin oxide coated conducting glass and dried in vacuum. Powder XRD patterns of the samples are recorded using a PANalytical-X’pertPro diffractometer. The powder samples of broken piece as well as the powder samples of plain and TeNWs/epoxy are subjected to X-ray diffractogram and are collected from 5 to 100 degrees in 2 theta using CuKα radiation. Differential scanning calorimetry (DSC) is carried out using a NETZSCHDSC 204 instrument under nitrogen atmosphere, at a heating rate of 10 °C/min.

Scheme 1. A) Synthesis of Te NWs started with a colorless hydrazine solution which acts as a reducing agent. B) TeO2 powder is slowly added to the hydrazine solution. Upon vagarious mixing of the components, color changes from colorless to pale yellow, purple and finally to blue within an hour. C) Growth of the TeNWs is arrested by the addition of SDS surfactant. D) TeNWs solution is centrifuged repeatedly. Supernatant contains the excess SDS, reducing agent. The solid that precipitate out of solution is TeNWs.

2.3. Synthesis of TeNWs

The synthesis procedure is shown in Scheme 1 (taken from the ref. 29). TeNWs are prepared by the synthetic protocol reported by Lin et al [22]. In the reaction, 16 mg of TeO2 powder is slowly added to a round bottom flask containing 10 mL of hydrazine hydrate. The reaction is continuously stirred at room temperature. The TeO2 powder got completely dissolved, and the color of the solution changed from colorless to pale yellow, purple and to blue about an hour. The blue color indicates the formation
of TeNWs. Then the solution is diluted 10-fold with SDS solution (10 mM), to control the length of the NWs. SDS is organo sulfate consisting of a 12-carbon tail attached to a sulfate group. The as-prepared solution is purified by centrifugation at 15,000 rpm for 15 minutes to remove excess hydrazine and SDS. The residue is redispersed in deionized water and centrifuged twice for the complete removal of unreacted species and excess and loosely bound SDS. Removal of excess surfactant is important to improve the mechanical properties of the TeNW/epoxy nanocomposites.

2.4. Preparation of Specimens
The fabrication of the composites is carried out through hand lay-up technique. The mould used to prepare epoxy nanocomposites consists of two soft rectangular aluminum alloy sheets and a single rubber spacer arranged in between them. The thickness of the epoxy specimens is controlled by the thickness of the rubber spacer which is 3 mm. The inner surfaces of aluminum plates are polished to achieve smooth sample surface and every time coated with a very thin layer of wax as a releasing agent.

Epoxy resin and hardener mixture is at ratio of 1.0:0.1 wt%. Specimens with loading concentration of 0.05% TeNWs are prepared. The sample is subjected to gentle mechanical stirring in a beaker at ambient conditions for overnight. This long stirring process can help to produce uniform TeNW-epoxy slurry and allow the epoxy to attach TeNWs surface for better interface. It is known that epoxy is rich in hydroxyl (-OH) groups, have much chemical affinity toward metallic surface of active TeNWs surface. Hence -OH groups get attached to the bare surface of TeNWs and helps to create good interfacial bonding between TeNWs and epoxy. These nanowires can be visualized as small iron rods in concrete, so it is expected to improve the strength. During the stirring, the color of epoxy changes from colorless to pale blue color indicates uniform mixing of TeNWs with epoxy resin. By placing the mixture in the vacuum for 20 minutes, the gas bubbles generated during the mixing process are removed. Then, the hardener is added and the mixture is stirred well with glass rod. The resulting epoxy mixture is then poured into the mould uniformly and the mould is placed at room temperature for 24 hours. After that, the cured epoxy composite plate is removed from the mould and the required specimens are cut.

2.5. Mechanical test
After fabrication, the test specimens are subjected to three point bending as per ASTM D 790 standards. The flexural test is conducted at a speed of 2 mm/min at room temperature (303K) using Instron UTM with 5 kN load cell.

3. Results and Discussion
3.1. Characterization of TeNWs
The formation of TeNWs is confirmed by optical extinction spectrum. Figure 1 shows the absorption spectrum of TeNWs after the removal of excess surfactant, impurities and again re-dispersion in distilled water. Extinction spectrum shows the two characteristic TeNW peaks in the range of 250 to 900 nm. According to the Hartree–Fock, peak I located in the range of 280–300 nm (4.1–4.4eV) is due to the transition from the valence band (p-bonding VB2) to the conduction band (p-antibonding CB1), and peak II located in the range of 650-750 nm (1.6–1.9eV) is assigned to be the transition from the valence band (p-lone-pair VB3) to the conduction band (p-anti bonding CB1). Peak II is characteristic peak of length of the NW. Increasing the length of the TeNWs will shift to the peak II to a higher wavelength region. In this case, peak I is centered at 293 nm and peak II is centered at 720 nm. From the previous reports [22, 23, 30], the peak I and II positions around 290 nm and 720 nm reveals that the length and width of TeNWs are around 690 nm and 20 nm.
The TeNWs are further analyzed by SEM and TEM to find the exact length and width of the TeNWs. The sizes of the TeNWs are in agreement with the length obtained from the extinction spectrum. Figure 2 A&B shows the large-area SEM and TEM images of TeNWs, respectively. They show that the TeNWs are monodispersed, straight, pretty clean and free from extra surfactant. TeNWs are structurally uniform and there are no obvious defects, indicating that the TeNWs are of single crystals. Figure 2 C &D shows the length and width histogram of the TeNWs distributions, respectively. Length of the TeNWs is 697±87 nm and diameter of TeNWs is 21±2.5 nm. From the literature, it is known that three representative planes of t-TeNWs, with interplanar spacings of 0.59, 0.39, and 0.22 nm corresponding to {001}, {010}, and {110}, respectively [22, 23].

Figure 3. A) Schematic representation of epoxy composite formation. B) Photographs of one set of specimens of plain epoxy and TeNWs/epoxy used for flexural tests. Note that TeNWs/epoxy specimens are uniformly
transparent with pale blue color of TeNWs indicates chemisorption of epoxy polymer through –OH groups on active TeNWs surface is represented in Scheme C.

Mechanism of formation of epoxy composites are given in Figure 3A. Rectangular shaped specimens are used for flexural test (Figure 3B). Even at 0.05 wt.%, the TeNW/epoxy specimens are uniformly transparent with pale blue color showing the uniform mixing of TeNWs with epoxy resin. Epoxy can strongly interact with TeNWs through the chemisorption of epoxy –OH groups on active TeNW surface.

We have performed wide-angle XRD and TGA-DTA analysis methods on these samples. Figure 4 shows the XRD pattern of plain epoxy powder and the TeNWs based epoxy nanocomposites, respectively. As illustrated in Figure 4A, the cured networks of plain and TeNWs/epoxy samples are amorphous in nature due to the highly cross-linked structures. For the pure epoxy samples, a broad peak centered at 2θ=18.5˚is observed which is assigned to the amorphous structure of the epoxy. After addition of TeNWs into the epoxy matrix, this peak shifted towards lower values and shifted to 2θ=17.3˚ for the TeNWs/epoxy composites. The downward shifting of this peak in the TeNWs/epoxy composites clearly shows that the embedding of TeNWs into epoxy resin influences the microstructure of the composites by the means of interfacial interaction between the TeNWs and epoxy matrix. Importantly the peak at 2θ=17.3 in TeNW epoxy sample is sharp (full width at half maximum (FWHM) for plain epoxy and TeNWs/epoxy are around ≈ 10 and ≈ 6, respectively) in comparison to plain epoxy sample indicates the increase in the crystallinity of epoxy structure upon the TeNW insertion.

Figure 5 shows the thermograms obtained by thermal decomposition of plain and TeNWs/epoxy specimens. Both samples show similar weight loss (around 95%) irrespective of the Te content. This result confirms that the amount of TeNW used in epoxy is at very low amount (negligible amount). However, the presence of Te is confirmed by very sensitive technique such as SEM/EDAX (see latter section). DTA data shows the sharp decomposition or changes in epoxy polymer for plain and TeNW/epoxy is at 385.2 °C and 378.87 °C. It shows TeNWs reinforced polymer slightly higher thermal stability than plain epoxy. We believe that the usage of higher TeNWs content can improve the thermal stability of TeNWs/epoxy nanocomposite substantially.
Figure 6. FT-IR spectrums of plain and TeNWs/epoxy polymer.

Figure 6. shows the FR-IR spectra of plain and TeNWs/epoxy polymer. The main bands of epoxy and TeNWs/epoxy are at 3045 cm⁻¹ and 1630 cm⁻¹ were assigned to the characteristic peak of the double bond group. Broad band at 3400 cm⁻¹ (OH), 2962 cm⁻¹ and 2930 cm⁻¹ (C-H), and 1110 cm⁻¹ (C-O). The main bands are 2958, 2825 and 2780 cm⁻¹ (C-H), 1240 cm⁻¹ (C-N), and 800 cm⁻¹ (NH₂) similar in both samples.

3.2. Mechanical Properties
The flexural strength values of the plain epoxy and TeNWs/epoxy composites are tabulated in Table 1 and Figure 7. All measures of the strength reported here show that the TeNWs/epoxy specimens are significantly stronger than the plain epoxy specimens.

3.2.1. Flexural properties
We have conducted three point bending test on epoxy and TeNWs/epoxy composites as per ASTM D790 standards. The specimens of 13 mm (width)×3 mm (thick)×127 mm (length) are tested. The flexural strength of the material is the property to resist deformation under bending loads. The flexural strength obtained from epoxy and TeNWs/epoxy specimens is studied by Instron Universal Testing Machine. It is observed that the flexural strength of TeNWs/epoxy composite tremendously increased compared to plain epoxy matrix. The flexural properties are shown in Table 1. TeNWs/epoxy composite material showed flexural strength of 110.7 MPa, while plain epoxy composite’s is 66.9 MPa. From this study, it is observed that even at low amounts of TeNWs (0.05 wt.%) the flexural strength is increased by 65%. The experimental results show that the presence of TeNWs helps to resist bending load and efficient stress transfer between epoxy polymer and TeNWs, which results in increased strength. These results are in consistent with our previous study, where we showed a remarkable enhancement on the tensile and impact properties of the Te NW epoxy polymer [29].

| Specimen     | Flexural strength (MPa) |
|--------------|-------------------------|
| TeNWs/epoxy  | 110.7                   |
| Plain epoxy  | 66.9                    |

Table 1. Flexural test results of TeNWs/epoxy composite and plain epoxy respectively.

Figure 7. Comparison of flexural strength of TeNWs/epoxy composite and plain epoxy.
3.3. SEM characterization

In order to understand the interfacial adhesion between TeNWs and epoxy matrix, fractured surface of epoxy samples containing TeNWs and pure epoxy is further analyzed using SEM imaging (Figure 8). Fractured surface images of the plain epoxy are taken at different magnifications to see the surface property. However, we haven’t seen noticeable differences in the surface smoothness of pure epoxy and TeNWs/epoxy composites as shown in Figure 8.

![SEM images of TeNW/epoxy](image)

**Figure 8.** A, B, D are SEM images of TeNW/epoxy collected for the specimen at different magnifications. The presence of Te is confirmed by SEM/EDAX spectrum C, collected from the TeNWs/epoxy specimen.

From the microstructure examination of TeNWs/epoxy nanocomposites by SEM showed the possibility of side by side and end to end self-assembly of TeNWs into linear structures inside the epoxy composite (shown in Figure 8D). The reason behind the improved flexural properties of TeNWs/epoxy composites is attributed to 1) stronger interaction between the TeNWs and epoxy, and 2) the self-assembly of a group of stacked TeNWs into linear fashion, also called unity effect [29].

4. Conclusions:

This study provided the reinforcing influences of TeNWs on flexural strength of epoxy polymer nanocomposites. Impressively, even at lower percentages of TeNWs (0.05 wt.%), the TeNWs/epoxy nanocomposites show remarkable enhancement in the flexural strength. Flexural strength of TeNWs/epoxy nanocomposites shows that TeNWs are good fillers to enhance the mechanical properties of epoxy polymer. SEM results showed that these TeNWs are self-assembled inside epoxy polymer. These results could provide guidance to design future semiconductor (TeNWs) based light weight and strong polymer nanocomposites.

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Description of corrigendum

Page 7: In the Figure 6. FT-IR spectrums of plain and TeNWs/epoxy polymer, Y-axis label is given as “Intensity”

This should read:

"Transmittance (%T)" as shown in the below figure 6

![Figure 6. FT-IR spectrums of plain and TeNWs/epoxy polymer](image-url)