# Evaluation of mechanical properties of functionalized carbon nanotube reinforced PMMA polymer nanocomposite

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## Abstract

In this paper, the multi-walled carbon nanotubes (MWCNT) were functionalized by chemical treatment for surface modification to create a better interfacial adhesion between polymer and nanotubes. Functionalization has proved to be an effective method to modulate different physical and chemical properties of the carbon nanotubes, facilitates dispersion and processing. The goal of this study is to determine the mechanical properties of the nanocomposite using experimental methods. Various mechanical tests such as tensile strength and impact strength were carried out to study the effect of functionalized filler content in the nanotube-reinforced Poly (methyl methacrylate) (PMMA) nanocomposite. The surface morphology of MWCNT and of the fractured surface of the fabricated MWCNT/PMMA were analyzed by Scanning Electron Microscope (SEM).
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## 1. Introduction

Composite materials are the main substitute for the conventional engineering materials due to its good characteristics of strength to density, low-cost, eco-friendly manufacturing processes [1–3]. Composites are light and have comparatively enhanced physical properties than their constituent materials [4]. Since the discovery of the carbon nanotube (CNT) [5], it has been the center of attractions due to its interesting properties. It has managed to capture the attention of researchers for its wide range of applications, including field emission, energy storage, molecular electronics and so on [6–8]. Nanocomposite with good CNT dispersion exhibits an exceptional combination of mechanical, electrical, thermal and tribological properties [9,10]. Polymer nanocomposites, which are endowed with many important properties such as nonlinear optical properties, electrical conductivity and luminescence, represent a new alternative to conventionally filled polymer composites. These have been proposed for their use in various applications including chemical sensors, electroluminescent devices, electro catalysis, batteries, biosensors, photovoltaic devices, smart windows and memory devices. CNTs have many advantages over other carbon materials in terms of

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electrical conductivity and thermal properties for which these have numerous applications in electronics and advanced materials. However, nanocomposites have had dispersion problems, which affect the inherent properties of the composites. The electron movement and the dipole−dipole interactions are influenced by the C−C bond structure of the carbon nanoparticles. But, the readily entangled carbon nanoparticles develop attractive forces among themselves, which eventually needs physical and chemical dispersion to break-up and reduce agglomeration [11,12,4]. The poor dispersion of CNTs in the polymer matrix results in poorer interfacial interaction. Nanotubes are functionalized to activate its surfaces to form a better adhesion between polymer and CNTs via the interface. The CNTs have found their applications as one of the best reinforcements in the composites, providing excellent mechanical, thermal and electrical properties [13−20]. In literature, various composite fabrication techniques have been reported for polymeric nanocomposites. Melt-mixing [21] and solution processing [22] are common for thermoplastics and thermosetting polymer. To have a better dispersion shear mixing [23] or ultrasonication method is used. To improve the bonding between the phases techniques like in situ polymerization [24], attachment of functional group [25] or surfactant application [26] may be employed. The process of fabrication affects much more than the grade of CNTs and polymer [27−32]. Better fabrication techniques enhance the above properties tremendously. Nowadays, researchers are much more focused on discovering the novel approaches to fabricate nanocomposite with good dispersion of reinforcements [33−36]. However, the controlling parameters can be optimized to get the best set of factors to prepare a composite. In this work, a fibrous composite has been prepared by taking multi-wall carbon nanotube as reinforcement and polymethyl methacrylate as a matrix. Many specimens have been prepared by taking different compositions of MWCNT varying from 0.3 to 1.5% weight. The tensile properties have been studied. Finally, the scanning electron microscope image is taken to study the microstructure of the composite samples.

## 2. Experimental method

This section describes the manufacturing and testing procedures. The tensile modulus E, tensile strength, and impact strength are measured. Specimens fabricated by injection molding taking PMMA with different weight percentages of MWCNTs were tested.

### 2.1. Materials

#### 2.1.1. Poly (methyl methacrylate) (PMMA)

The thermoplastic polymer, PMMA is highly hygroscopic for which it must be dehydrated prior to micro compounding. An appropriate amount of PMMA is preheated in a vacuum oven at $60\,°C$ for 1 h. This heating process extracts all the moisture from the PMMA otherwise this will react with MWCNT and form voids inside the composite. This process is done in a vacuum medium to avoid reaction with atmospheric moisture.

#### 2.1.2. Functionalization of CNT

The carbon nanotubes produced in the laboratory were chemically treated for the functionalization of tubes. Chemical treatment makes the end of the tubes fragmented and facilitates the attachment of functional groups to the tube surface. This results in a better adhesion between the tube surface to the matrix occurs. The end caps of the nanotubes are supposed to be composed of the highly reactive fullerenes-like hemisphere in contrast with the side walls. The side walls themselves have a pentagon−heptagon pair called Stone-Walls defect, $sp^3$-hybridized defects and vacancies in the carbon nanotube lattice as shown in Fig. 1. This results in improved mechanical properties by effective load transfer from the matrix to the tube. The electrical conductivity also depends on the good interconnection of the tube in the matrix. The carbon nanotubes are treated with concentrated $H_2SO_4$ and $HNO_3$ in the ratio of 3:1 by volume. Then the solution is stirred to disperse the hydrophobic f-MWCNTs completely using a magnetic stirrer at room temperature with a low stirring speed.
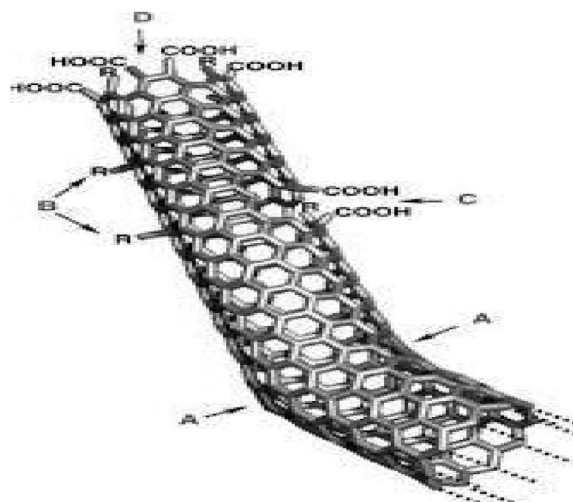


Fig. 1. Various defects in a Single-walled nanotube.

Ultra-sonication is the process of using ultrasound energy to excite and agitate particles in a solution by a sonicator. This is a preferred principle for nanoparticle dispersion in a solution as in the biological cases and material science field application of nanoparticle. The ultrasound wave propagates through a series of compression and expansion; these attenuated waves are induced in the molecules of the medium produce shock waves and disintegrate individual nanoparticle in the bundle, in the agglomerates and separate from the bundles. In this process CNTs in liquids having a low viscosity, such as water, acetone and ethanol are effectively dispersed. Sonication is to be carried out at 40 °C for 24 h.

Afterwards, the adequate amount of distilled water is added into the solution and allowed for settling down of the nanotube particles on the bottom of the container. After that, it was filtrated by Whatman 42 filter paper then treated with the oxidizing agent sulphuric acid ($H_2SO_4$) and hydrogen peroxide ($H_2O_2$) in the ratio of 4:1 by volume. The $H_2SO_4$ has the oxidization capacity, cuts the edges of CNTs and $H_2O_2$ add the carboxyl group or oxygen groups to the surface of CNTs for proper bonding and binding. After treating with these oxidants, the CNTs become hydrophobic to hydrophilic in nature by the attachment of polar groups on its surface. Then the solution is kept on the magnetic stirrer at 70 °C and stirred at low rpm for 1 h to avoid spilling out of the solution from the flask. Then it is taken out and diluted by 200−250 volumes of distilled water and kept aside to settle down the colloidal particles of CNTs. Then an acid solution of $H_2SO_4$ and $H_2O_2$ are decanted by pipette tube.

The colloidal particles of CNTs are taken into the centrifuge rotating at a high-speed of about 9300 rpm at 20 °C for 15−20 min. The CNTs are attached to the wall of the tubes being separated from the acid and bases due to the centrifugal forces. Then it is collected. The acidic and base property of the solution should be checked at a regular interval of time by PH paper. So, it is diluted by adding distilled water until a neutral PH is obtained.

The extracted CNTs are semisolid in nature and more in volume. So, these nanoparticles are dehydrated in an oven at 80 °C for 24 h. The nanotubes now obtained are functionalized carbon nanotubes. The dehydrated MWCNTs are in the form of flakes. So, this further needs to be churned into powder form using cryomilling.

## 2.2. Cryomilling

The process of converting the CNTs into powder requires milling. This is obtained either by ball milling process or the cryo-milling process. Cryomilling is the low-temperature milling process in which the solid lump or crystal shaped materials are churned into powder form by vibration and impact force. The cryomilling machine RETSCH is employed to churn the lump of CNTs effectively. To obtain a very low-temperature environment of around −196 °C, the liquid nitrogen is supplied to the milling machine at around a steady pressure of 0.2 bar. At low temperatures, the ductility of the material decreases, and brittleness increases so that at a high frequency of vibration the agglomerated CNT particles break into the powder form. So, the CNT particles are poured into the cylinder with an alloy ball inside it and properly tightened and placed firmly in the machine. A frequency of 3 Hz was applied to initiate the vibration. Further, at a frequency of 15 Hz, the milling was carried out for only 2−3 min and checked regularly to avoid the formation of flakes by excessive milling. A very high frequency of vibration may lead to breakage of carbon nanotube so low frequency of vibration is maintained.

## 2.3. Method

The composite was fabricated by compounding PMMA with various loadings of MWCNTs filler. The process follows.

### 2.3.1. Micro compounding

The DSM XPLORE 5 & 15 micro compounder is used for mixing small quantities of material. This has the unique vary-batch TM concept, which helps to choose a batch volume from 3 ml to 15 ml. Hopper of the compounder houses a funnel, plunger and hollow pipe. The mixture is fed into the hopper and then it moves towards the barrel maintained at a temperature of 220 °C and, melts before reaching the bottom of the barrel. The mixture is fed into the barrel which houses a twin co-rotating screw rotating at a speed of 1000 rpm. But, the speed of the co-rotating screw decreases to 13−15 rpm because of high viscosity of the polymer. It has made sure that the drain valve is closed during the compounding. The barrel is supplied by an inert gas (argon or nitrogen) to prevent the oxidation of the mixture and the machine is water cooled to avoid the heat generation. The compounding time is set for 15−20 min, during this process, the materials melt and create a viscous solution of CNT and PMMA. To ensure circulation of the viscous materials pressure must be developed at the bottom part of the barrel. After compounding process, the drain valve is opened, and the compounded material is collected in the mini injection

molding machine and extruded in the desired ASTM standard shape. Table 1 shows different batches.

## 2.4. Fabrication of composite specimens

There are many specimen preparations with varying wt% of filler material required before the testing. These include measuring and cutting of the specimen gauge. DSM Micro 10 cc mini injection molding machine is used for molding small volume of materials. As compared to the conventional molding, mini injection molding processes are right and reliable.

Mini injection molding machine with a capacity of 10 cc has been used and its different parts are mold, mold holder, nozzle and pressure cylinder. The compounded materials discharged from the micro-compounder are accumulated inside the cylinder (nozzle). The nozzle has heating oil inside it, which maintains the temperature of the compounded melt at around 220 °C as in the micro-compounder. The injection is done by means of the compressed air in a cylinder that forces the melt mixture into the mold at a

desired temperature. The injection temperature has been kept at 0.8 bar. The pressure gradually increases from zero to maximum varying with time and decreases suddenly after injection. The process is run by setting up of desired pressure in the pneumatic chamber. After the process we get the desired specimen in the mold as shown in Fig. 2a and b.

The mold is covered by a funnel kept at 220 °C, same as micro compounding temperature to avoid solidification of the liquid composite during injecting process. After the injection process, the mold is cooled at room temperature.

## 3. Result and discussion

In the earlier chapter, the fabrication process of the f-MWCNT/PMMA composite samples by Mini Injection followed by Micro-compounding after the functionalization of the CNTs was discussed. The mechanical properties of the prepared composites were measured by Instron and Impact Test Machine Tinious Olsen as per the ASTM standards.

Table 1
Various percentages of f-MWCNT in the composites (Tensile Specimen).

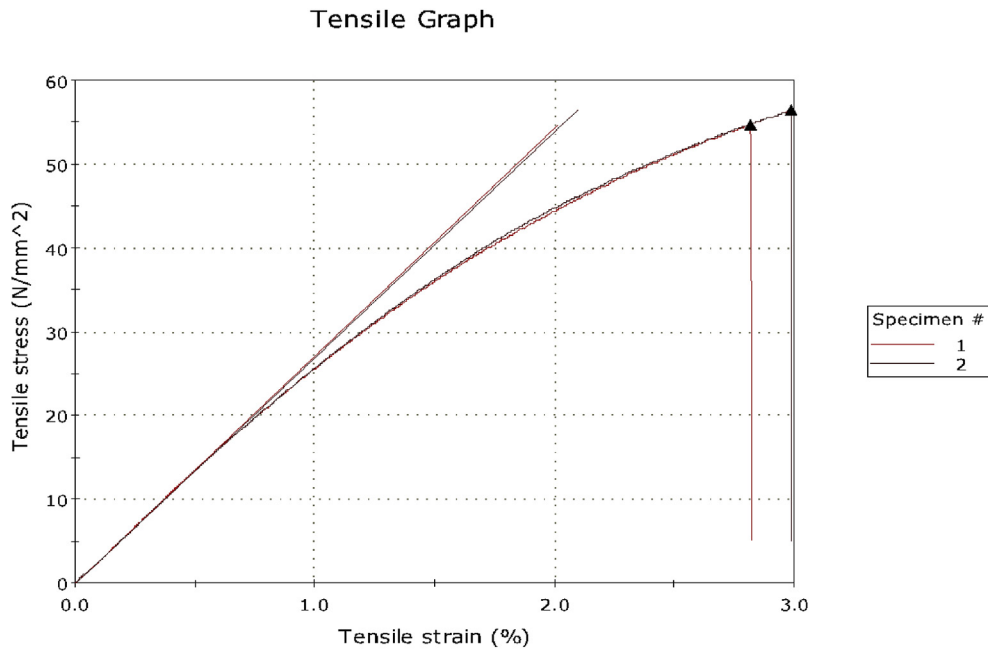| Batch no | CNT/PMMA to be taken in each batch in grams. | No of samples tensile specimen in each batch | CNT% each batch in gram. | PMMA in gram. | Total weight of composite in gram. |
|---|---|---|---|---|---|
| 1 | 11.5 | 1 | 0 | 11.5 | 11.5 |
| 2 | 11.5 | 1 | 0.3% = 0.0345 | 11.4655 | 11.5 |
| 3 | 11.5 | 1 | 0.5% = 0.0575 | 11.4425 | 11.5 |
| 4 | 11.5 | 1 | 1.0% = 0.115 | 11.385 | 11.5 |
| 5 | 11.5 | 1 | 1.5% = 0.1725 | 11.3275 | 11.5 |
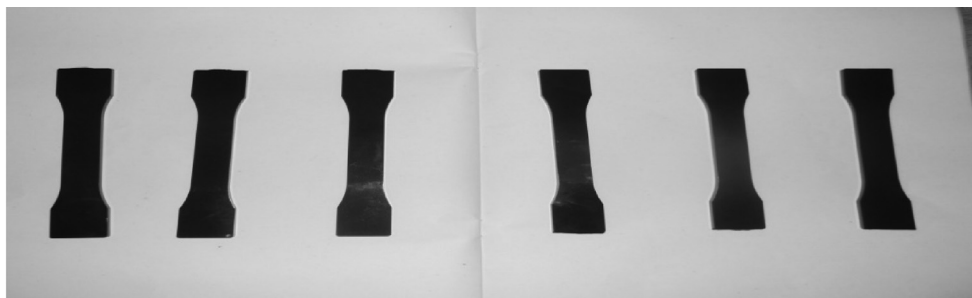


(a)


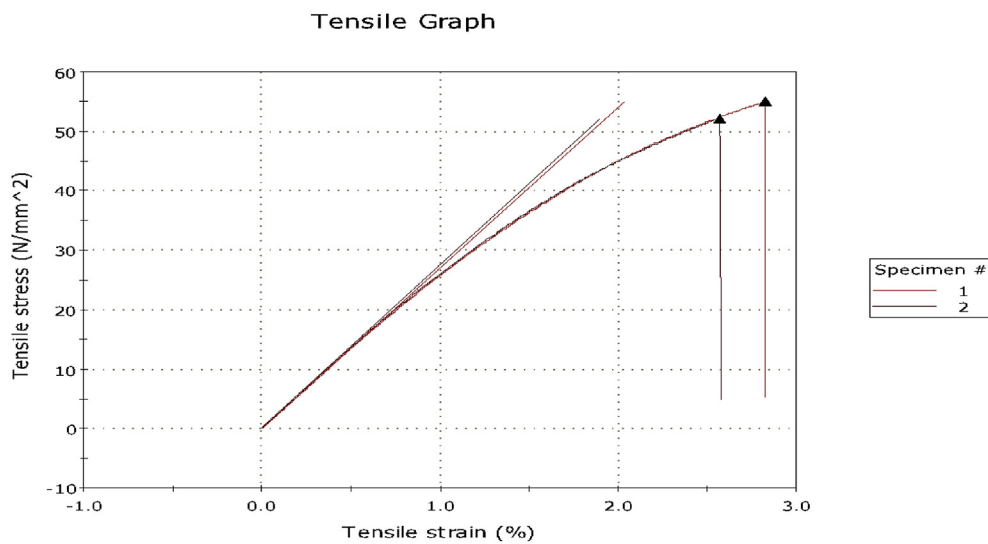
(b)

Fig. 2. Specimens for tensile and impact testing.

Fig. 3. Stress−strain curve (a) 0.3 wt%, and (b) 1 wt%.

## 3.1. Tensile stress test

Fig. 3a shows the stress−strain curves of f-MWCNT/PMMA composite for 0.3wt% of f-MWCNT. As shown in Table 2 two observations are taken for the same percentage composition. The tensile stress plot at a various weight percentages of filler

material with taking standard error into consideration is shown in Fig. 4. From the plots, it is observed that as tensile stress increases the strain increases. At the breaking point, the tensile stress is maximum, and the mean value is 55.602 MPa. From Table 3 and the graph, it is found that initially, the tensile stress goes on increasing up to 0.5 wt% of filler content then there

Table 2
Tensile stress, strain and extension at maximum load.

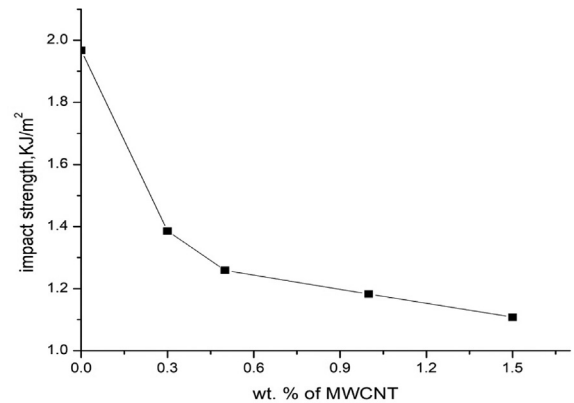| wt% of MWCNT | Specimen no. | Tensile stress at maximum load (MPa) | Tensile strain at maximum load (%) | Tensile extension at maximum load (mm) |
|---|---|---|---|---|
| 0.3 | 1 | 54.751 | 2.818 | 2.81836 |
| | 2 | 56.453 | 2.987 | 2.98672 |
| | Mean | 55.602 | 2.903 | 2.90254 |
| 1 | 1 | 55.029 | 2.823 | 2.82332 |
| | 2 | 52.212 | 2.573 | 2.57340 |
| | Mean | 53.620 | 2.698 | 2.69836 |
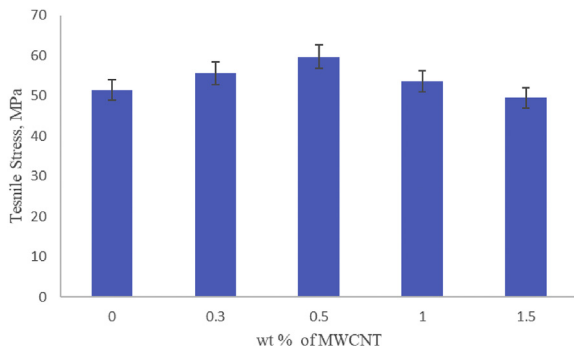


Fig. 4. Tensile stress vs. wt% of MWCNT.

Table 3
Tensile stress and Tensile modulus with a variation of f-MWCNT content.

| Percentage of CNT | 0.0 | 0.3 | 0.5 | 1.0 | 1.5 |
|---|---|---|---|---|---|
| Tensile stress in MPa | 51.466 | 55.602 | 59.755 | 53.620 | 49.503 |

has been a steep decline. Fig. 3b shows the stress–strain curves of f-MWCNT/PMMA composite for 1wt%. Two observations are taken for the same percentage composition.

### 3.2. Impact test results

Table 4 represents the energy absorbed by the composite during impact load from the pendulum. Moreover, it is found that the impact strength decreases with an increase in nanotube fillers as shown in Fig. 5.

Table 4
Impact strength of f-MWCNT/PMMA composite.

| Sl. no. | wt% of f-MWCNT | Strength in KJ/m$^2$ |
|---|---|---|
| 1 | 0 | 1.96746 |
| 2 | 0.3 | 1.38603 |
| 3 | 0.5 | 1.25905 |
| 4 | 1.0 | 1.18284 |
| 5 | 1.5 | 1.10823 |



Fig. 5. Impact strength vs. wt% of MWCNT.

The cause for the decrease in impact strength can be attributed to the increment in filler loading and the improper adhesion between filler and the matrix. The increase in f-MWCNT content affects the adhesion and makes the composite increasingly brittle. The set of different parameters for Izod test are pendulum energy, notch depth, notch radius, specimen width and specimen thickness and their values are taken as 2.7475 J, 2.54 mm, 0.25 mm, 10.16 mm, 3.2 mm respectively.

### 3.3. Characterization

To maximize composite performance for a given CNT wt%, the carbon nanotubes must be evenly dispersed within the polymer matrix. This was achieved by functionalization and micro-compounding. The SEM micrographs show the surface morphology of the MWCNT and the texture of the fractured surface. Fig. 6a shows the SEM image of MWCNT powder. The microstructure of MWCNT is the fibrous type. The microphotographs of the fractured surfaces of the MWCNT/PMMA nanocomposite were taken to study the dispersion of materials as exhibited in Fig. 6b and c. Fig. 6d and e shows the TEM micrograph of
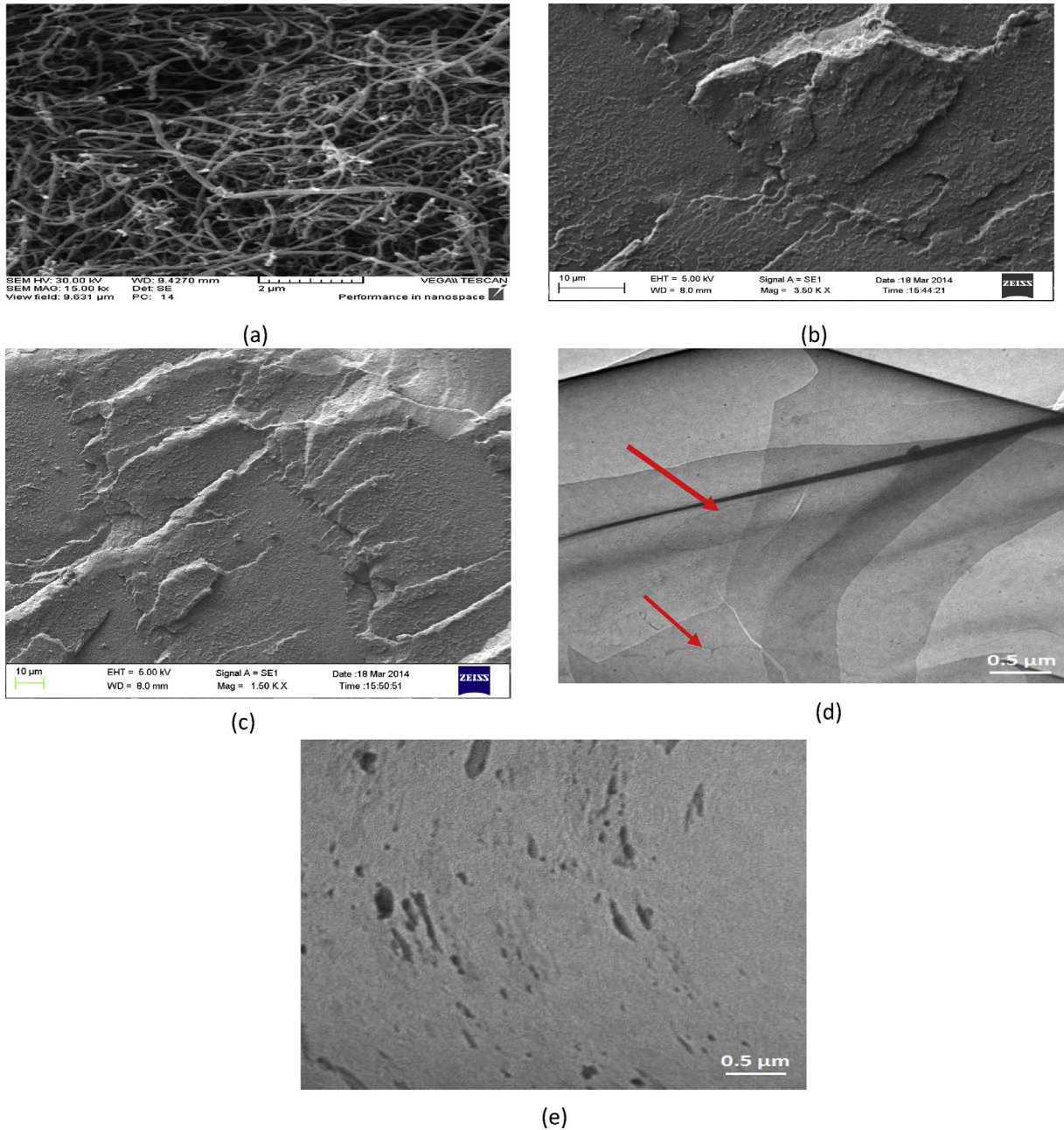
Fig. 6. SEM micrograph of (a) MWCNT, (b), (c) fractured graph, and (d), (e) TEM micrograph of PMMA/0.5 wt% and PMMA/1.5 wt%.

PMMA composites with 0.5 wt% and 1.5 wt% of MWCNT. The dark spots are the nanoparticles in the PMMA matrix. As shown in Fig. 6d, the nanoparticles are properly interspersed in the polymer matrix. It shows the homogeneous dispersion of MWCNT in PMMA composite with no significant aggregation of nanotubes which suggests that ultrasonication is useful in the dispersion. The marked portions in Fig. 6d represent the starting of nanoclustering around the crack possibly due to stress concentration [37]. In Fig. 6e there seems a noticeable aggregation at 1.5 wt % of MWCNT.

## 4. Conclusion

Functionalization was done to increase the effective dispersion of the nanofiller in the matrix content. Mechanical properties such as tensile and impact loading tests were carried out. SEM and TEM were performed for morphological study of the composite. The tensile strength of MWCNT/PMMA composite increases with an increase in weight percentages of MWCNT up to 0.5 wt% and then it decreases. With the addition of MWCNT, there is a 16% increase in the tensile stress. The reason behind this fluctuation could be insufficient melt and mixture time for injection molding process. Moreover, with an increase in filler content, the high viscosity of the polymer in melt state making it tougher for f-MWCNT to be dispersed efficiently in it. The lack of flexibility causes a further decrease in tensile strength. The impact strength decreases sharply up to 0.3wt% of MWCNT then it decreases very slowly.

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