

## Research Article

# Effect of Acid- and Ultraviolet/Ozonolysis-Treated MWCNTs on the Electrical and Mechanical Properties of Epoxy Nanocomposites as Bipolar Plate Applications

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Carbon nanotubes (CNTs) have a huge potential as conductive fillers in conductive polymer composites (CPCs), particularly for bipolar plate applications. These composites are prepared using singlefiller and multifiller reinforced multiwalled carbon nanotubes (MWCNTs) that have undergone a chemical functionalization process. The electrical conductivity and mechanical properties of these composites are determined and compared between the different functionalization processes. The results show that UV/O<sub>3</sub>-treated functionalization is capable of introducing carboxylic functional groups on CNTs. Acid-treated CNT composites give low electrical conductivity, compared with UV/O<sub>3</sub>-treated and As-produced CNTs. The in- and through-plane electrical conductivities and flexural strength of multifiller EP/G/MWCNTs (As-produced and UV/O<sub>3</sub>-treated) achieved the US Department of Energy targets. Acid-treated CNT composites affect the electrical conductivity and mechanical properties of the nanocomposites. These data indicate that the nanocomposites developed in this work may be alternative attributers of bipolar plate requirements.

## 1. Introduction

Bipolar plates are the key components in a proton exchange membrane fuel cell (PEMFC) and perform as current conductors between cells, provide conduits for reactant gases flow, and constitute the backbone of a power stack [1]. Conventional pure graphite (G) bipolar plates contribute significantly to the cost and weight of PEMFC stacks. Metals, such as stainless steel and metal alloys, are not preferable because of corrosion-related issues [1, 2]. The development of lightweight, low-cost, and highly conductive polymer composite bipolar plates with a scope for mass production can aid the rapid commercialization of PEM fuel cells [3–5]. Therefore, polymer composite bipolar plates can replace heavier G bipolar plates in next-generation PEMFCs [6]. The materials chosen in the fabrication of bipolar plates have

caught the attention of many researchers. One of the branches being studied is the polymer nanocomposite-based type. The reinforcing and/or electrically conducting fillers commonly used, including G, carbon fiber, and carbon black, have been reported to enhance the overall performance of composite plates formed using the conventional polymer processing technique [7–13]. Since the discovery of carbon nanotubes and their exceptional mechanical properties by Iijima, the idea of using them as reinforcing fibers in composite materials has been the driving force in composite design [14]. Theoretical and experimental results show the superior electrical properties of CNTs. Multiwalled carbon nanotubes (MWCNTs) are reported to be always electrically conductive and to have an electrical conductivity of approximately  $1.85 \times 10^3 \text{ S cm}^{-1}$  [15]. Despite the MWCNTs' wide range of potential applications, their use often remains problematic because

of the agglomeration caused by strong intertube van der Waals forces and their high surface energy due to the strong curvature of the thin nanotubes [16]. The functionalization of CNTs involves the generation of chemical moieties on their surface, which could improve solubility and process ability. These attributes allow CNTs to be combined with other types of materials, particularly polymer matrix [17]. Oxidation is the most common method of purifying CNTs via reflux in a concentrated acidic solution. The introduction of active functional groups, damage to the CNTs, and opening of caps may also be induced by oxidation. The extent of each of these effects depends on the oxidation conditions and has much influence on the physical and electrical properties of the composites [18, 19]. The oxidation process has been found to contain strong acids that create significant physical damages to the tube chirality, helicity, and micropathway of CNTs. These damages severely degrade the originally desirable properties of CNTs [20–26]. The dry oxidation process using ozone in the presence of ultraviolet ozonolysis (UV/O<sub>3</sub>) has been an alternative treatment to issues associated with wet oxidation [27–30].

This study mainly aims to investigate the effect of functionalized MWCNTs on the electrical and mechanical properties of nanocomposites. Functionalization here refers to the acid and UV/O<sub>3</sub> treatments. The scope of this study is then followed by the fabrication of multifiller polymer nanocomposites to achieve good electrical and mechanical properties that had been set by the US Department of Energy targets as shown in Table 1 [31]. Lastly, the comparative study between the acid- and UV/O<sub>3</sub>-treated oxidation treatments on the hybrid MWCNTs composites is discussed.

## 2. Experimental

**2.1. Materials.** MWCNTs were used as nanofillers in this study. NC 7000 was purchased from Nanocyl, Belgium. Its diameter and length were 9.5 nm and 1.5 μm, respectively, with purity of ≥90%, as reported by the manufacturer.

The synthetic G powder used in this study had an electrical resistivity of 0.03 Ωcm, a surface ratio of 1.5 m<sup>2</sup>g<sup>-1</sup>, and an average particle size of 44 μm. The G powder was purchased from a local agent of Asbury Carbons, New Jersey. The epoxy resin (EP) used in this research was bisphenol-A-based epoxy resin (635), with a viscosity of 6 poise obtained from US Composites, Inc. A lower viscosity of the epoxy matrix was selected to obtain better wetting conditions with reinforcement fillers.

**2.2. Preparation of MWCNTs.** Two different functionalization treatments were done in this study, namely, the well-established acid-treated oxidation and the latest UV/O<sub>3</sub>-treated oxidation. MWCNTs were treated with sulfuric and nitric acids at 3:1 (v/v) ratio to produce a high-concentration acid solution. The MWCNTs were suspended in a dilute solution and refluxed under a given set of oxidation conditions. The oxidized MWCNTs were subsequently filtered and washed with distilled water until they reached pH 7. The functionalized MWCNTs were dried in an oven at 40°C for 48 h. For UV/O<sub>3</sub>-treated functionalization, the MWCNTs were

TABLE 1: US Department of Energy targets of a bipolar plate.

Property	Value
Electrical conductivity	>100 S cm <sup>-1</sup>
Thermal conductivity	>10 W (mK) <sup>-1</sup>
Flexural strength	>25 MPa
Corrosion resistance	<1 μA cm <sup>-2</sup>
Weight	<0.4 kg kW <sup>-1</sup>
Gas permeability	<2 × 10 <sup>-6</sup> cm <sup>3</sup> cm <sup>-2</sup> s <sup>-1</sup> at 80°C and 3 atm

exposed to a self-made in-house UV/O<sub>3</sub> system for 95 min exposure time at 18 cm exposure distance and 10 Lmg<sup>-1</sup> ozone flow rate. These parameters were set according to the pilot and optimization experiments using the response surface method (RSM) technique in earlier studies.

**2.3. Preparation of Composites.** For the fabrication of single-filler EP/MWCNT nanocomposites, the epoxy and curing agent were mixed at 3:1 ratio in wt%. The mixture was mixed using a high-speed mechanical mixer (RW 20-KIKAWERK) at 1200 rpm for 40 s. MWCNTs with different loading concentrations were added into the epoxy and curing agent mixture. The suspension was mixed again at 800 rpm for 3 min. The composite mixture was poured into a steel mold for compression molding at 130°C for 1.5 h. On the other hand, the multifiller EP/G/MWCNT nanocomposites were fabricated in three stages. In the first stage, the CNTs (5 wt%) and G (75 wt%) concentrations were mixed using a planetary ball mill to obtain a homogeneous mixture. The ball-to-powder ratio used was 4:1, with stainless balls of 10 mm in diameter and at a rotating speed of 200 rpm for 1 h. In the second stage, the EP and curing agent were mixed using the high-speed mechanical mixer at 1200 rpm for 40 s. Finally, the G/CNTs are mixed further using an internal mixer with a Haake torque rheometer at a temperature of 30°C. The rotational speed and mixing time were set at 20 rpm and 5 min, respectively. The composite mixtures were poured into a steel mold (100 mm × 100 mm × 3 mm) at 130°C curing temperature. The curing pressure was maintained at 10 MPa for 1.5 h.

## 3. Characterization

**3.1. Electrical Conductivity Measurement.** The electrical conductivities of the nanocomposites were determined using two different methods, namely, the in- and through-plane conductivities. The in-plane electrical conductivity of the composite was measured using a Jandel Multi Height Four-Point Probe and an RM3 Test Unit, which has a constant-current source and digital voltmeter. This equipment can measure sheet resistance ranging from 1 mΩcm<sup>-1</sup> to 5 × 10<sup>8</sup> Ωcm<sup>-1</sup> and volume resistivity ranging from 10<sup>-3</sup> to 10<sup>6</sup> Ωcm. Meanwhile, through-plane electrical conductivity was measured using CTU-01 from ZBT GmbH Duisburg.

**3.2. Mechanical Property Measurement.** Three-point flexural testing was performed using a Universal Testing Machine (Instron 5567). Testing was based on an ASTM D790-03

standard with a sample dimension of  $100 \times 12.7 \times 2.5 \text{ mm}^3$  and supported range distance of 50.0 mm. The hardness of composites was performed using a dynamic ultramicrohardness tester using a Vickers-type diamond indenter. Dwell load is 0.98 N and dwell time is 10 sec which were used as test parameters.

**3.3. Fracture Surface Morphology.** The fractured surfaces of nanocomposites were observed using a field emission scanning electron microscope (SUPRA 55VPSEM) to study the dispersion and intercalation mechanisms of conductive fillers in the polymer matrix.

## 4. Results and Discussion

### 4.1. Single Filler

**4.1.1. In-Plane Electrical Conductivity.** The in-plane electrical conductivity of polymer composites depends on the arrangement of reinforcement materials and the dispersion and distribution of the reinforcing materials in the polymer matrix. The reinforcement materials for CNTs in the composite produced a conductive network that helps the movement of electrons and indirectly becomes a conductive composite. Figure 1 shows the in-plane electrical conductivity of the composites produced using three types of CNTs, namely, the As-produced MWCNTs, acid-treated MWCNTs, and UV/O<sub>3</sub>-treated MWCNTs. The in-plane electrical conductivity of all treatments of MWCNTs/epoxy nanocomposites, with the addition of a small amount of MWCNTs, increased with the increasing weight loading of the reinforcing material composition, the CNTs [32–35]. A comparison between the three types of composites shows that the composites with As-produced MWCNTs achieved the highest in-plane electrical conductivity of  $2.84 \text{ S cm}^{-1}$ , whereas the composites with UV/O<sub>3</sub>- and acid-treated MWCNTs gave electrical conductivity values of 2.2 and  $1.1 \text{ S cm}^{-1}$ , respectively. The composite with acid-treated MWCNTs showed the lowest in-plane electrical conductivity at a weight percentage loading composition of 10 wt% MWCNTs. This phenomenon is due to the treatment of strong acids during wet oxidation, which causes structural damage to the wall of MWCNTs and hence reduces the overall electrical conductivity of MWCNTs [20, 21, 33]. However, the composite with UV/O<sub>3</sub>-treated MWCNTs showed an upward trend after the composition of 10 wt%. Even though it shows increasing trend for all types of CNTs, addition CNTs more than 10 wt% caused improper mixing process occurred, epoxy as a matrix not properly wetted with the conductive fillers due to high surface area of CNTs. Moreover compression molded plate at more than 10 wt% MWCNTs has significant voids which are not suitable for bipolar plate application.

**4.1.2. Mechanical Test.** The hardness properties of EP/MWCNT composites are shown in Figure 2. The test was performed for MWCNT loadings of 0 wt%, 2.5 wt%, 5.0 wt%, 7.5 wt%, and 10 wt% for the three types of treatments, namely, the As-produced, acid-treated, and UV/O<sub>3</sub>-treated MWCNTs. The hardness of pure epoxy is increased with additional

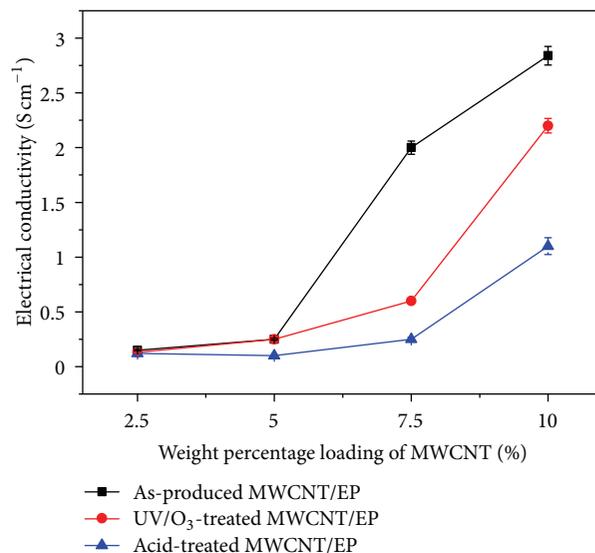


FIGURE 1: In-plane electrical properties of As-produced, acid-treated, and UV/O<sub>3</sub>-treated MWCNT composites.

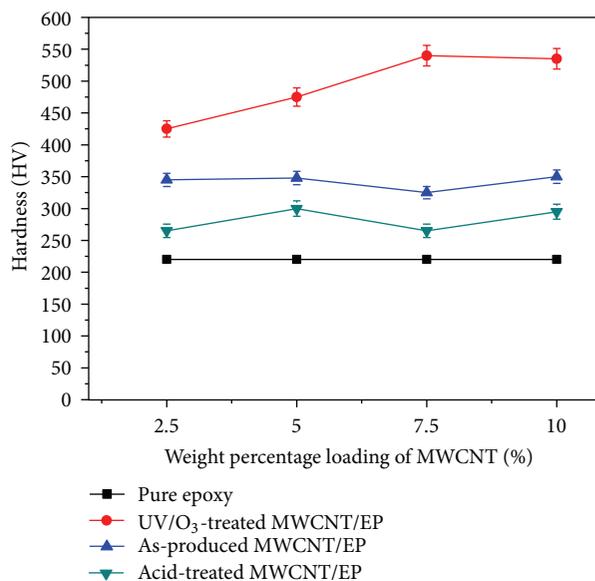


FIGURE 2: Mechanical properties of As-produced, UV/O<sub>3</sub>-treated, and acid-treated MWCNTs compared with pure epoxy.

of MWCNTs in polymer matrix. Figure 2 shows that UV/O<sub>3</sub>-treated MWCNTs exhibited the highest hardness, follows by As-produced and acid-treated MWCNTs/EP composites. This finding supports the results of previous studies that UV/O<sub>3</sub> treatment gives better mechanical properties than acid treatment because of the defect formed on the surface of MWCNTs during acid treatments [20, 22–26]. The hardness of the UV/O<sub>3</sub>-treated MWCNTs is increasing until 7.5 wt% of CNTs and then becomes stable until 10 wt%. However, acid-treated MWCNTs and as-produced MWCNTs/EP show stable manner despite the increase in MWCNTs loading concentration until 10 wt%.

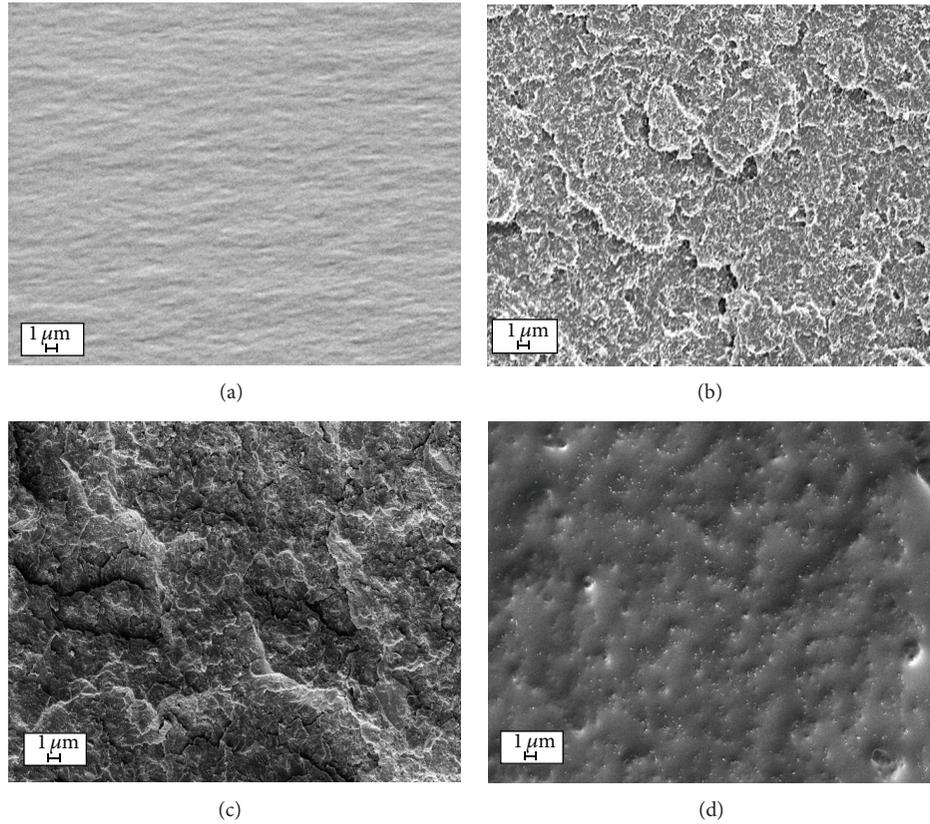


FIGURE 3: Fracture surface morphology of (a) pure epoxy, (b) As-produced MWCNTs, (c) acid-treated MWCNTs, and (d) UV/O<sub>3</sub>-treated MWCNT/epoxy composites under FE-SEM.

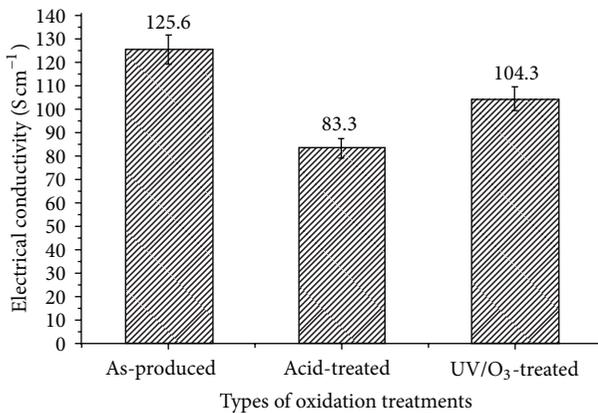


FIGURE 4: In-plane electrical conductivity of EP/G/MWCNTs.

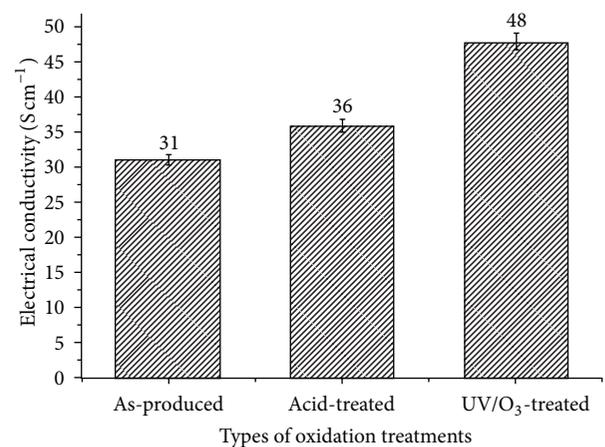


FIGURE 5: Through-plane electrical conductivity of EP/G/MWCNTs.

**4.1.3. Surface Fracture Morphology.** The scanning electron micrographs of the pure epoxy and MWCNT/epoxy nanocomposites for the three types of treatments are shown in Figure 3. The composition was fixed to a weight loading of 10 wt%. The MWCNTs were reasonably well dispersed and evenly distributed over the whole area. Moreover, the UV/O<sub>3</sub>-treated MWCNTs seemed shorter in length due to being firmly embedded in polymer matrix. This indicates good adhesion between filler and polymer matrix. Higher

amounts of MWCNTs loading concentration in polymer matrix MWCNT content resulted in more networks and increased the in-plane electrical conductivity properties. Compared with chemical functionalization, UV/O<sub>3</sub> treatment showed better dispersion and even better distribution than pure acid treatment [33]. For electrical conductivity behavior, at high electrical conductive polymer composite

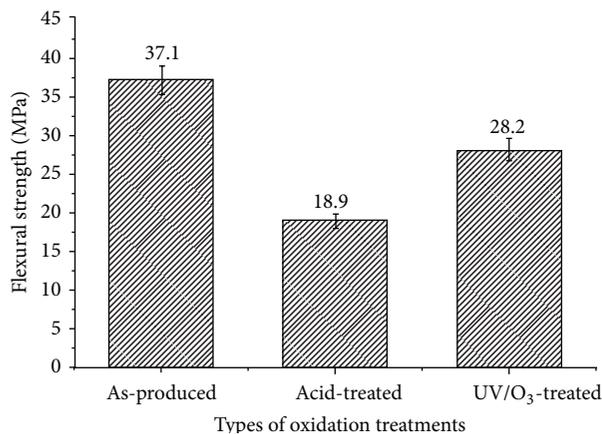


FIGURE 6: Flexural strength of EP/G/MWCNTs.

where more than  $20 \text{ S cm}^{-1}$  is required, distribution of MWCNTs with continuous electrical conductive network through matrix composite plays a main role rather than dispersion of MWCNTs without continuous electrical conductive network [8]. Thus, in-plane conductivity results in Figure 3 which is in parallel with this behavior [8].

In this single-filler study, we could not achieve the DOE target for the bipolar plate application [31]. Therefore, further studies on the multifiller or hybrid conductive composite plates were made. A combination of G and CNTs was said to have synergistic effects that could obtain desired in- and through-plane conductivities [6, 12, 13]. The effect of acid- and UV/O<sub>3</sub>-treated MWCNTs was also discussed in this study.

## 4.2. Multifiller

### 4.2.1. Electrical Conductivity

*In-Plane Electrical Conductivity.* Based on previous results reported by Suherman et al. which study effect of different combination concentration of MWCNTs and graphite in epoxy matrix. Their findings found that the highest values of in-plane electrical conductivity and through-plane conductivity for multifillers composite plates were at 5 wt.% of As-produced MWCNTs and 75 wt.% graphite in epoxy matrix [13]. Therefore, this study used composition of EP/G/MWCNTs at (20/75/5) wt.% with different type of MWCNTs treatment. The in-plane electrical conductivity of the EP/G/MWCNTs nanocomposites for the three types of MWCNT treatments is shown in Figure 4. The hybrid composite plates with As-produced, acid-treated, and UV/O<sub>3</sub>-treated MWCNTs showed electrical conductivity values of 125.6, 83.3, and 104.3  $\text{S cm}^{-1}$ , respectively. The in-plane electrical conductivity of the EP/G composite at a composition of (20/80) wt.% was reported as 50  $\text{S cm}^{-1}$  [13]. Thus it indicates that additional CNTs are able to improve electrical conductivity of EP/G composites. The addition of MWCNTs to these hybrid composites increased the electrical conductivity properties to values higher than those of

single-filler reinforcement composites. The spaces between the G particles were filled with small-sized MWCNTs. Thus, networks may be formed in the EP/G/MWCNT composites. However, the type of MWCNT plays an important role in the formation of a conductive network. In this context, this study shows the effect of chemical functionalization on electrical conductivity. The present results clearly show that acid-treated MWCNT composites have low electrical conductivity. The oxidation process using strong acid damages the chirality of the tube, helicity, and micro-CNT flow because of the defects formed during the acid treatment on the CNT surfaces [20–26]. The hybrid composite plate with UV/O<sub>3</sub>-treated and As-produced MWCNTs achieved the electrical conductivity target set by the US DOE for bipolar plates in fuel cells. However, the UV/O<sub>3</sub>-treated MWCNTs obtained an electrical conductivity value of ~17% less than that of the As-produced MWCNTs.

*Through-Plane Electrical Conductivity.* Figure 5 shows the results obtained by through-plane electrical conductivity. The hybrid composite plates with As-produced, acid-treated, and UV/O<sub>3</sub>-treated MWCNTs showed conductivity values of 31, 36, and 48  $\text{S cm}^{-1}$ , respectively. The through-plane electrical conductivity for the conductive composite polymer was much lower than the in-plane electrical conductivity. This difference was caused by the orientation of G particles in the composite, that is, perpendicular to the compression direction. Therefore, only a small amount of electrical conductivity path flow formed and lowered the through-plane electrical conductivity [36].

*4.2.2. Mechanical Test.* Good mechanical properties are required for composite materials used in PEMFC bipolar plates. A bipolar plate is required to support thin membranes and electrodes and withstand high clamping forces for the stack assembly. Obtaining high electrical conductivity and sufficient mechanical strength simultaneously from the same material is difficult [6]. In this study, three types of samples were prepared using As-produced, acid-treated, and UV/O<sub>3</sub>-treated MWCNTs. Figure 6 shows the flexural strengths of these three types of samples. The flexural strength of the EP/G/As-produced MWCNT composite plate was highest at 37 MPa, followed by the EP/G/UV/O<sub>3</sub>-treated MWCNT composite plate at 28 MPa and, finally, the EP/G/acid-treated MWCNT composite plate at 19 MPa. The average ratio and high strength of the CNTs, their dispersion, and the good interfacial bonding between CNTs and the polymer matrix contributed to the increase in the flexural properties of the composite produced. In addition, the 75 wt.% G content in the composite also contributed to the flexural strength. Therefore, the strength of the EP/G/As-produced MWCNT composite plate reached the DOE target. The EP/G/UV/O<sub>3</sub>-treated MWCNT plate also reached the minimum value set by the US DOE. However, the acid-treated MWCNT composites gave the lowest value and, therefore, did not reach the minimum target value. These results indicate that UV/O<sub>3</sub> treatment provided better mechanical properties than acid treatment.

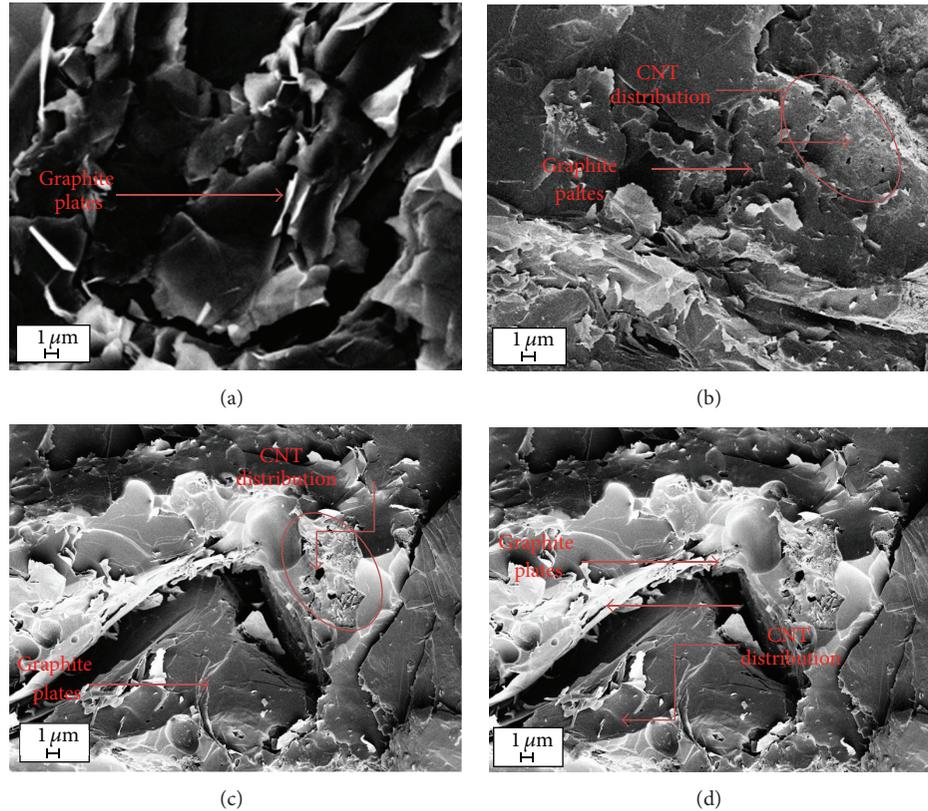


FIGURE 7: Surface fracture of (a) EP/G composite, (b) EP/G/As Produced MWCNTs, (c) EP/G/Acid-treated MWCNTs, (d) EP/G/UV/O<sub>3</sub> Treated MWCNTs.

**4.2.3. Surface Fracture Morphology.** The CNT powder was measured by examining the distribution of CNTs in the composite powder using a scanning electron microscope (FE-SEM). G can be seen in the form of large gray flakes, whereas the MWCNT powder looks like light capillaries (white) because of the gold coating process. The MWCNTs were small; hence, they could fill the voids between the pieces of G and form a network of conductivity in the composite plate. From Figure 7, the MWCNT distributions in the three types of treatments were good and even. Moreover, the As-produced MWCNT composite plates obtained better electrical conductivity and mechanical properties compared with the functionalized MWCNTs. The comparison among the treatment processes showed that UV/O<sub>3</sub>-treated MWCNTs are more efficient than acid-treated MWCNTs.

## 5. Conclusion

The study showed that UV/O<sub>3</sub>-treated MWCNTs have higher electrical conductivity and better mechanical properties than acid-treated MWCNTs for single-filler epoxy nanocomposites. However, highly conductive epoxy composites may be developed using synergistic combinations of G and CNTs and not single-filler CNTs for bipolar plate applications. The EP/G/As-produced MWCNTs had higher in-plane electrical conductivity than functionalized MWCNTs. However, the

EP/G/UV/O<sub>3</sub>-treated MWCNTs had the highest through-plane conductivity. Therefore, dry oxidation treatment can be an alternative functionalization method for improving the dispersion and distribution of nanomaterials in a polymer matrix to achieve high electrical conductivity and good mechanical strength simultaneously.

## Conflict of Interests

There is no conflict of interests between the authors and material suppliers mention in this paper (Nanocyl, Asbury Carbon, and US Composites, Inc).

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