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Effect of the compression molding parameters on the in-plane and through-plane conductivity of carbon nanotubes/graphite/epoxy nanocomposites as bipolar plate material for a polymer electrolyte membrane fuel cell

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Abstract

The main challenges for commercialization of a single-filler graphite (G) polymer-matrix composite as bipolar plates are its low electrical conductivity and flexural strength. The minimum requirements set by the US Department of Energy (DOE) are the electrical conductivity and flexural strength to be greater than 100 S/cm and 25 MPa, respectively. In this study, the electrical conductivity of a G/epoxy (EP) composite (single filler) is only 50 S/cm (in-plane conductivity) at 80 wt% G. However, flexural strength is greater than 25 MPa. Using carbon nanotubes (CNTs) as the second filler at a concentration of 5 wt% in a CNTs/G/EP nanocomposite resulted in the in-plane and through-plane electrical conductivity and flexural strength being 180 S/cm, 75 S/cm, and 45 MPa, respectively. The density of the CNTs/G/EP nanocomposite is also less than that of G/EP composite, which demonstrates that a total weight reduction is achievable.

Keywords: C. Electrical conductivity; B. Nanocomposites; E. Fuel cells

1. Introduction

The bipolar plate is one of the most important components in a polymer electrolyte membrane (PEM) fuel cell, which can contribute up to 80% of the fuel cell stack total weight [1,2]. A cost analysis has shown that the bipolar plate of a PEM fuel cell stack incurs 38% of the total cost, followed by the electrodes, membrane, and catalysts, incurring 32%, 12% and 11% of the total cost, respectively [2]. Various material properties requirements of a bipolar plate have been set by the US Department of Energy (DOE) [3] as shown in Table 1.

Graphite is the most commonly used material for bipolar plates, but its disadvantages are its brittleness and difficulty in machining and thus, a graphite bipolar plate must be made thick, which results in a fuel cell stack that is large and heavy [4]. Polymer–graphite composites have a great potential to replace graphite due to their low cost, low weight, and ease in manufacture. Unfortunately, the primary disadvantages of the composites are their low electrical conductivities and mechanical properties. Therefore, it is important to develop composite materials with high electrical conductivities and suitable mechanical properties by incorporating graphite powder with other types of reinforced carbon fillers [5–7]. Carbon nanotubes (CNTs) have also shown great potential to be used as a secondary conductive filler, but dispersing CNTs in a polymer matrix is a challenge yet to be solved; CNTs tend to agglomerate due to their high surface energy and

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nano-sized diameter. However, incorporating CNTs would be effective if they could be well-dispersed within the polymer matrix. The dispersion of CNTs in a polymer matrix can be achieved using a suitable surface treatment through careful modification [8,9]. However, processing the bipolar plate becomes more complex. Furthermore, using functionalized CNTs as a conductive filler in the polymer matrix could increase the cost of the product. Incorporating pristine CNTs and graphite, which have large particle sizes, is another potential alternative to achieve a higher electrical conductivity and suitable mechanical properties [10,11]. In this study, synthetic graphite was chosen as the primary conductive filler combined with a small amount of electrically conductive carbon nanotubes as the secondary filler to achieve the requirements stated by the DOE for a bipolar plate. The objectives of this research were to study the effect of nano-sized conductive fillers in a graphite/epoxy polymer matrix and develop a fabrication process for a nanocomposite conductive bipolar plate to be used in a PEM fuel cell application.

2. Experimental

2.1. Material

Synthetic graphite (G) type 4102 was used in this study, which has a surface area of 1.5 m^2/g and a particle size of 74 μm. The synthetic graphite was purchased from Asbury Carbons, New Jersey. The multi-walled carbon nanotubes (MWCNTs) NC type 7000 were purchased from Nanocyl, Belgium, which have a surface area of 300 m^2/g, a diameter of 9.5 nm, a length of 1.5 μm, and a purity that is $\geq 90\%$ as reported by the manufacturer. The epoxy resin (EP) was a bisphenol-A based epoxy resin, with a viscosity of 6 P and was obtained from US Composites. The curing temperature of the epoxy recommended by the manufacturer was 80 °C. The curing agent, 4-Aminophenylsulphone, was purchased from New Jersey, USA. The curing agent was a diamine type (tetra functional) to facilitate rapid and dense cross-linking in the epoxy resin. A low-viscosity epoxy matrix was selected for its better wetting conditions with conductive fillers. Fig. 1 shows the SEM images of G and CNTs used in this study. Fig. 1 indicates the G and CNT particles that contained no impurities.

Table 1
US DOE material properties requirements of a bipolar plate.

Property	Value
High electrical conductivity	> 100 S/cm
High flexural strength	> 25 MPa
High thermal conductivity	> 10 W/m K
High corrosion resistance	< 1 $\mu A/cm^2$
Low weight	< 0.4 kg/kW
Low gas permeability	$< 2 \times 10^6$ $cm^3/s\,cm^2$ at 80 °C and 3 atm

2.2. Fabrication of polymer nanocomposites

The liquid epoxy and curing agent were mixed in a 3:1 ratio according to weight percentage (wt%). Mixing G/CNTs in the epoxy was performed in three stages. In the first stage, the CNTs and G mixture was milled at different wt% using a planetary ball mill to attain a homogenous mixture. The wt% compositions of the CNTs, graphite and epoxy are shown in Table 2.

A 4:1 ratio of balls to powder was used, with 10-mm diameter stainless balls rotating at a speed of 200 rpm for 1 h. In the second stage, the EP and the curing agent were mixed using a high-speed mechanical mixer (RW 20-KIKA-WERK) at 1200 rpm for 40 s. In the third stage, G/CNTs

Table 2
Composition of G, CNTs and EP in the composites.

G (wt%)	CNTs (wt%)	EP (wt%)
60	0	40
70	0	30
80	0	20
79	1	20
77.5	2.5	20
75	5	20
72.5	7.5	20
70	10	20

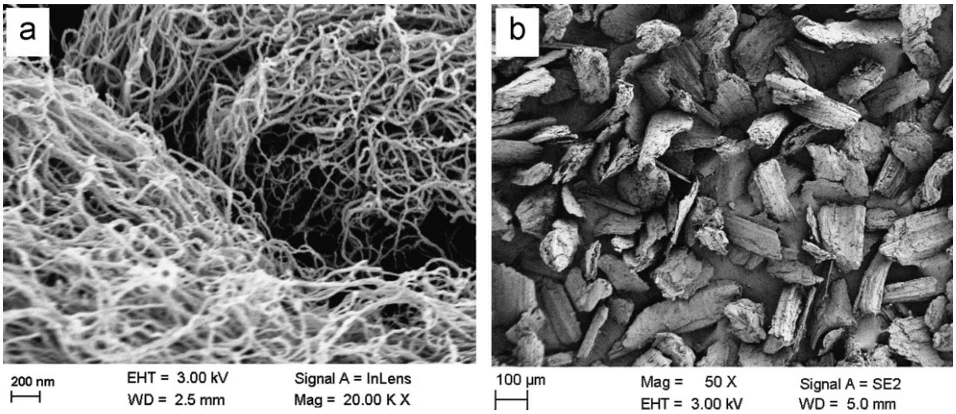

Fig. 1. (a) SEM image of the as-received CNTs and (b) G.

were further mixed using an internal mixer with a Haake torque rheometer at a temperature of 30 °C [12]. The rotational speed and the mixing time were set at a constant 25 rpm and 10 min. The composite mixture was then poured into a steel mold at various curing temperatures (80, 110, 130, 150 °C). Various curing pressures (6.2, 8.3, 10.3 and 12.4 MPa) were applied for 1.5 h.

3. Characterization

The in-plane electrical conductivity of the polymer nanocomposites was measured using a Jandel multi-height four-point. The electrical conductivity of the through-plane of the polymer nanocomposites was measured by a through-plane electrical conductivity tester manufactured at ZBT in Duisburg, Germany [13]. The mechanical properties were measured using a three-point bending test according to the ASTM D790-03 standard at room temperature by a universal testing machine (UTM) Model Instron 5567 at a cross-head speed of 1 mm/min. The dimensions of the specimens were $100 \times 12.7 \times 2.5$ mm^3, and the support span length of the specimens was fixed at 50.8 mm. Fractured surfaces of the nanocomposite plates were observed using scanning electron microscopy (FESEM, Model Supra 55/55VP) to observe the dispersion of the conductive fillers in the polymer matrix and other microscopic features of the fracture surfaces. Thermal degradation of the optimal polymer nanocomposites was measured by thermogravimetric analysis (NETZSCH STA449F3), beginning at room temperature and stopping at 700 °C with a heating rate of 20 °C/min in a nitrogen atmosphere. The density of the nanocomposites was measured according to the ASTM D792 standard by the Archimedes method.

4. Results and discussion

4.1. Electrical conductivity of single filler graphite/epoxy composite

Fig. 2 shows the variations of electrical conductivity of a single-filler G/EP composite as a function of loading concentration. The results show that the in-plane and through-plane electrical conductivities obey the percolation threshold law; the electrical conductivity increased as the loading concentration of the conductive filler increased. The results showed that the electrical conductivity increased from 14.5 S/cm (60 wt%) to 50 S/cm (80 wt%) for the in-plane direction and 9 S/cm to 40 S/cm for the through-plane direction. However, the electrical conductivity of the G/EP composite was still less than the requirements set by the DOE, reaching only 50 S/cm at the higher loading concentration, 80 wt% of G. A higher loading concentration of G is required to create a conductive path throughout the entire composite. However, increasing the G filler loading to 90 wt% decreased the processability of the epoxy composite due to the tremendous increase in viscosity. This result showed that a single-filler composite with G as the conductive filler is not suitable for a bipolar plate. Therefore, using CNTs as the second filler with a high surface area and a nano-sized diameter was important for the next step.

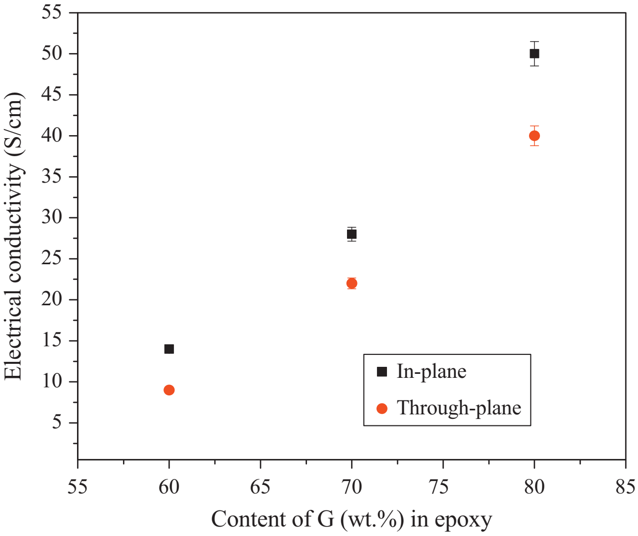

Fig. 2. Effect of the graphite concentration on the in-plane and through-plane electrical conductivity of G/EP composites.

The through-plane electrical conductivities of thermoplastic composites have been reported in other studies [13,14], and there are several studies on the measured in-plane electrical conductivity of thermoset and thermoplastic composites [15–19]. It was reported that the in-plane electrical conductivity of the composite, polypropylene/G, was only 7 S/cm at 80 wt% of G [19]. Different polymer matrices will result in the different bulk electrical conductivities of the composite. Polypropylene (PP) has a lower electrical conductivity (10^{-16} S/cm) compared with epoxy (10^{-14} S/cm) due to the absence of polar groups in the PP backbone, which makes the dispersion of the conductive filler in the polymer matrix more difficult [20].

4.2. Electrical conductivity of CNTs/G/EP nanocomposites

As can be seen from Fig. 2, the results showed that 20 wt% epoxy was the optimal polymer matrix concentration. The in-plane and the through-plane electrical conductivities of the nanocomposites at a fixed composition of epoxy (20 wt%) with different loading concentrations of synthetic graphite (70, 72.5, 75, 77.5, 79 wt%) and CNTs (1, 2.5, 5, 7.5, 10 wt%) are shown in Fig. 3. The in-plane and the through-plane electrical conductivity sharply increased as the CNTs loading went from 1 wt% to 5 wt%. Then, the conductivities began to decrease at 7.5 and 10 wt% of CNTs. These results are explained by the synergistic effect of combining CNTs and G, which produces a higher electrical conductivity. However, the electrical conductivity decreases as the CNTs filler loading concentration increases because the epoxy resin cannot be wetted well; with an excess of CNTs, agglomeration occurs, which deteriorates the electrical conductivity of the nanocomposites. When used as a second filler at 5 wt%, CNTs significantly increase the electrical conductivity by more than 260% compared with the single-filler G/EP composite at the same filler loading (80 wt%). Gaps between the synthetic graphite particles, which exhibit a flake-like geometry, were filled effectively with CNTs, which have a smaller diameter and a tubular geometry and thus, conducting networks were formed between the CNTs/G and epoxy matrix. Fig. 3 confirms that incorporating different conductive fillers with different geometries and sizes can create synergistic effects and reduce the percolation threshold of hybrid filler polymer composites of the in-plane electrical conductivity of nanocomposites [21,22].

Fig. 4 shows SEM images of the fractured surfaces of the CNTs/G/EP nanocomposites. The images reveal that the CNTs were well-dispersed over the entire area between the G particles at 5 wt%, but at 10 wt%, there were aggregates of CNTs. As the CNT loading increased, more aggregations of CNTs were observed. These aggregations were responsible for electrical conductivity decreasing at the higher loadings.

4.3. Effect of CNTs on flexural strength

Fig. 5 shows the effect of increasing the CNT filler loading concentration on the flexural strength. Fig. 5 shows that the flexural strength increased as the CNT filler loading concentration increased. The highest flexural strength obtained was 45 MPa at 5 wt% CNTs; adding CNTs at a loading greater than 5 wt% sharply decreased the flexural strength. The decreasing flexural strength above a critical filler loading concentration (5 wt%) can be attributed to the poor dispersion of CNTs at the higher filler loading concentration and a lack of epoxy resin to bind the conductive fillers, as shown in Fig. 5 [23,24].

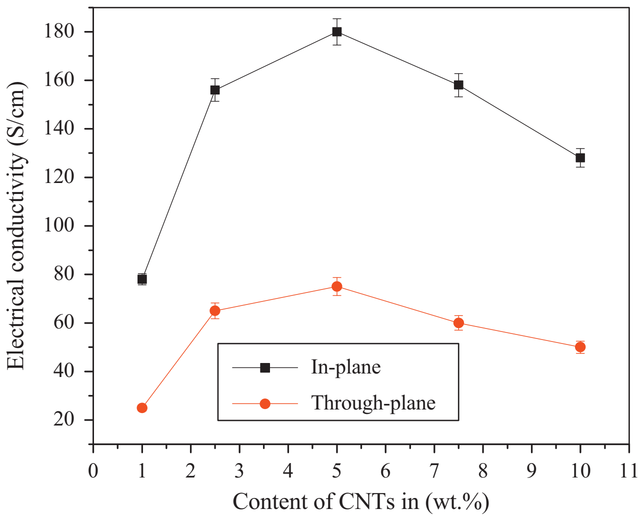

Fig. 3. Effect of the weight percentage on the in-plane and through-plane electrical conductivity of CNTs/G/EP nanocomposites.

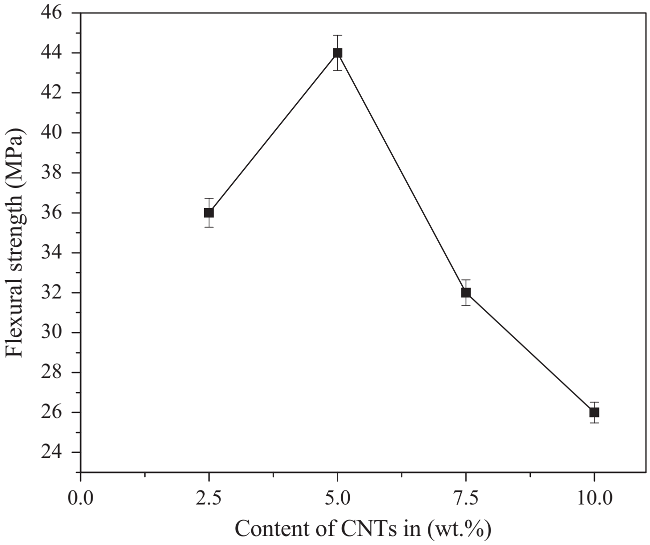

Fig. 5. Effect of the weight percentage on the flexural strength of the CNTs/G/EP nanocomposites.

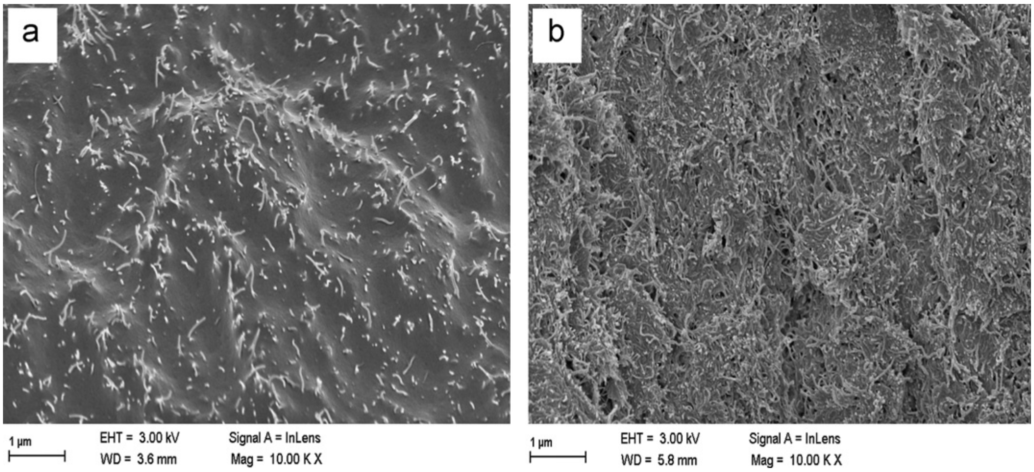

Fig. 4. SEM fracture surface images of the CNTs/G/EP nanocomposites: (a) 5 wt% CNTs and (b) 10 wt% CNTs.

Based on the optimal composition of the CNTs/G/EP nanocomposites (5/75/20 wt%), the curing temperature and the curing pressure were investigated at a fixed 5/75/20 wt% composition.

4.4. Effect of curing temperature on in-plane and through-plane electrical conductivity

To increase the mobility of CNTs (the second filler) and graphite (the primary filler) in the polymer matrix during the manufacturing process, a high curing temperature during the compression process is required to reduce the viscosity of the epoxy resin. This condition increases the tendency of the CNTs and graphite to interact with each other and form conductive filler networks in the polymer matrix. Fig. 6 shows the variation of the electrical conductivity as the curing temperature increased at a 5/75/20 wt% composition of CNTs/G/EP. The results from Fig. 6 show that the electrical conductivity of the nanocomposites gradually increased with an increasing curing temperature (80–150 °C). The highest electrical conductivity for the in-plane and through-plane of the CNTs/G/EP nanocomposite was achieved at a curing temperature of 150 °C. The highest electrical conductivities for the in-plane and through-plane of the CNTs/G/EP nanocomposite were 180 S/cm and 75 S/cm, respectively. The higher the curing temperature, the lower the initial viscosity. Particle movement in the dispersion was progressively enhanced as the curing temperature increased from 80 °C to 150 °C [25]. A high loading of the filler caused dissipation of the epoxy into the filler. Therefore, a higher curing temperature is required for the epoxy to begin cross-linking with the curing agent. The dependence of the curing temperature on the network formation can be explained by considering the curing behavior of the epoxy.

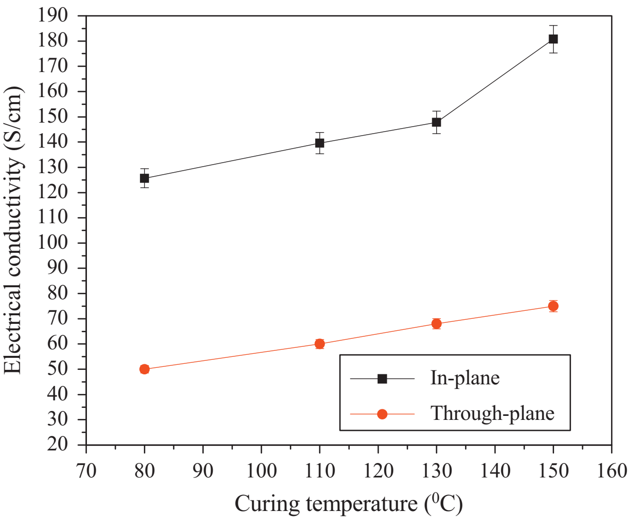

Fig. 6. Effect of the curing temperature on the in-plane and through-plane electrical conductivity.

4.5. Effect of curing pressure on in-plane and through-plane electrical conductivity

A high filler loading concentration in the range 60–80 wt% has been reported to be required to obtain a high electrical conductivity [26]. However, a high viscosity of the resin mixture and the conductive fillers may contribute to the difficulties in processing and may cause formation of voids (air bubbles) in the final bulk nanocomposite. Increasing the curing pressure during the compression molding of the mixture causes the distances between the fillers to decrease, which eliminates voids and should theoretically increase the electrical conductivity [27,28]. Fig. 7 shows the in-plane and through-plane electrical conductivity of the CNTs/G/EP at the composition 5/75/20 wt% as the curing pressure increased. The results showed that the in-plane and through-plane electrical conductivity gradually increased as the curing pressure increased. The in-plane electrical conductivity increased approximately 50% at a curing pressure of 12.4 MPa compared with the nanocomposites fabricated at a curing pressure of 6.2 MPa. The electrical conductivity increased as curing pressure increased because at a higher pressure, the contact distance between the CNTs and the synthetic graphite decreased. A high curing pressure is needed to reduce the distance between the conductive filler at a higher viscosity (80 wt% filler concentration). It can be seen that the optimal curing pressure for the 5/75/20 wt% CNTs/G/EP nanocomposite was 12.4 MPa. Air bubbles or voids produced during the fabrication process will increase as more nanoparticles are added. A low viscosity allows the epoxy resin and the curing agent to penetrate into the graphite and fill the micro-pores between the graphite particles. A large pressure applied during the curing process reduces the number of voids. Boey and Lye [28] used a compressive pressure to reduce the void content. Increasing the curing pressure during the fabrication process could

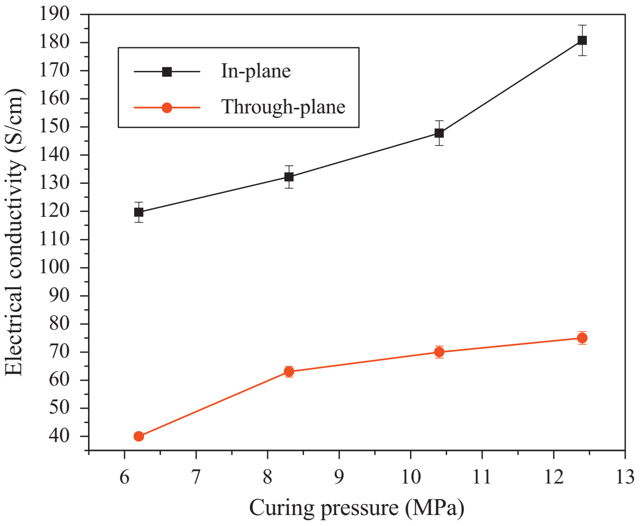

Fig. 7. Effect of the curing pressure on the in-plane and through-plane electrical conductivity.

reduce the void content within the composite specimens [28,29].

Fig. 8 shows the SEM images of the surfaces of CNTs/G/EP nanocomposites. Fig. 8 shows that the number of voids decreased as the curing pressure increased. This decrease in void content at higher curing pressures was due to the nano-sized CNTs being the second filler and being able to fill the micro-pores between the graphite particles in the polymer matrix. Thus, the CNTs acted as a conductive bridge between the graphite particles.

4.6. *Effect of CNTs on density and thermogravimetric analysis (TGA)*

Fig. 9 shows the effect of adding CNTs on the density of the bulk nanocomposite. The density of the CNTs/G/EP nanocomposite at 2.5 wt% (1.75 g/cm^3) was lower than that of the G/EP (1.76 g/cm^3) composite, which demonstrates the potential of CNTs to help reduce the total bulk weight. The CNTs/G/EP nanocomposite densities gradually decreased as the CNT loading increased. The density of the CNTs/G/EP nanocomposite decreased from 1.75 g/cm^3 (2.5 wt% CNTs) to 1.65 g/cm^3 (10 wt% CNTs) because the density of CNTs (1.34 g/cm^3) [30] is lower than that of graphite (1.74 g/cm^3) [31]. The optimal requirement of the bipolar plate application is for the 5/75/20 wt% composition,

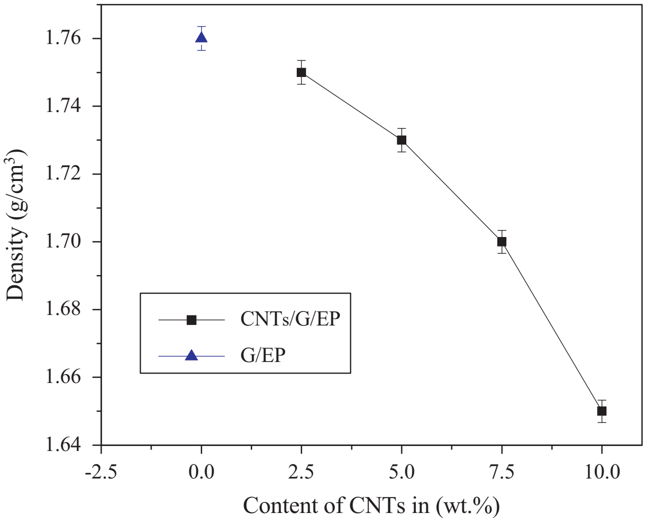

Fig. 9. Effect of weight percentage on density of the CNTs/G/EP nanocomposites.

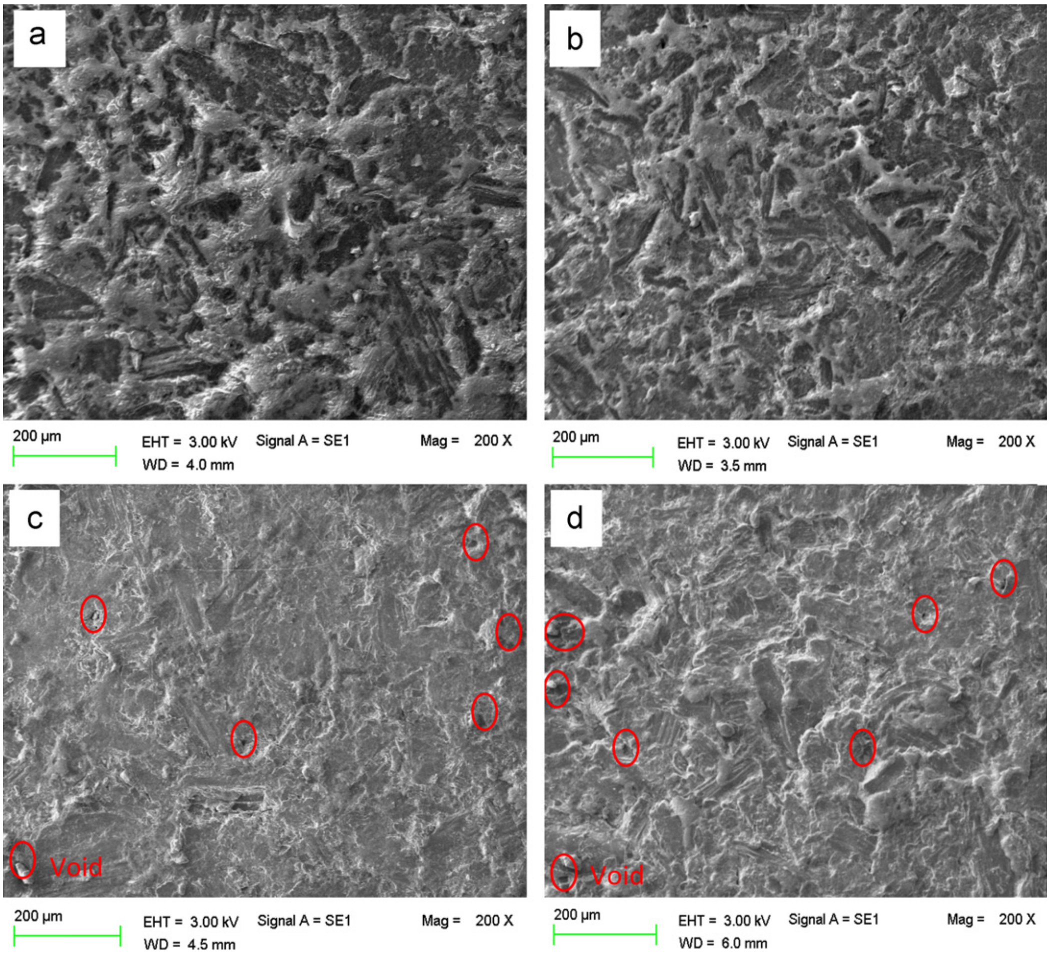

Fig. 8. SEM surface images of the CNTs/G/EP nanocomposites at curing pressures (a) 12.4 MPa, (b) 10.3 MPa, (c) 8.3 MPa and (d) 6.2 MPa.

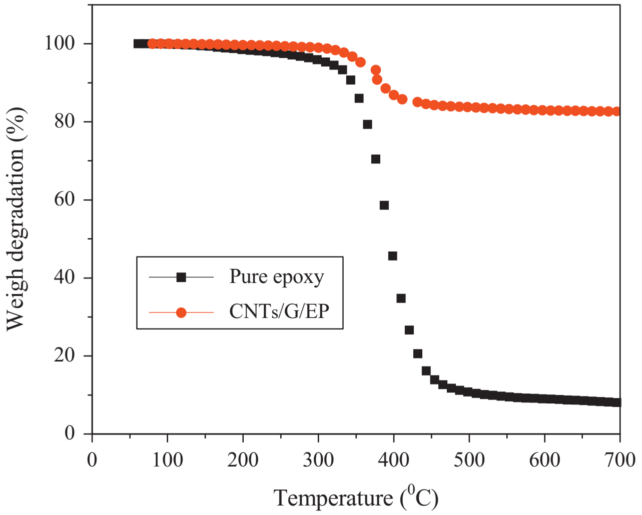

Fig. 10. TGA curve of the polymer nanocomposites at the optimal concentration.

which results in a CNTs/G/EP nanocomposite density of 1.73 g/cm^3. The lower density of the nanocomposite plate would help to reduce the fuel cell stack weight, because 80% of the weight of a PEM fuel cell stack is from the bipolar plate.

Fig. 10 shows the TGA curve for the nanocomposite and the pure epoxy polymer. The thermogravimetric temperature shift of the pure epoxy was approximately 280 °C and increased up to 380 °C for the CNTs/G/EP nanocomposite. A higher thermal stability improves the overall performance of the bulk composite as a bipolar plate as this would allow the component to operate either in a low-temperature PEM fuel cell (80 °C) or in a high-temperature PEM fuel cell (130–200 °C) [14,26]. Fig. 10 shows that the fabricated nanocomposite plate has the potential to be used in a low- and high-temperature PEM fuel cell. The pure epoxy exhibited essentially the same behavior as the nanocomposite until approximately 150 °C, which is when the pure epoxy weight degraded, whereas the nanocomposite plate was essentially at a stable weight degradation until 300 °C. Fig. 10 shows that the weight degradation at 100 °C was only 0.2%; however, Hui et al. reported a 0.6% weight loss degradation of an epoxy/graphite composite bipolar plate at the same temperature [32]. The low weight degradation of our nanocomposite plate was due to the small amount CNTs as the second filler, which increased the thermal stability of the nanocomposite plate. This result confirms that the CNTs/G/EP nanocomposite plate with a 5/75/20 wt% composition exhibits good thermal stability and is suitable for bipolar plate applications in a low- and high-temperature PEM fuel cell.

5. Conclusions

The effects of CNTs on the properties and fabrication process of a polymer nanocomposite plate were investigated, where the focus was on the feasibility of the plate being used as a bipolar plate in a PEM fuel cell. Based on the results, the following conclusions were reached:

1. Incorporating CNTs as the second filler in a hybrid nanocomposite produces a synergistic effect that enhances the flexural strength properties and the electrical conductivity (in-plane and through-plane) compared with those of a single-filler composite.
2. The electrical conductivity of the nanocomposites increased as the curing temperature increased. A high curing temperature reduces the resin viscosity, which enhances the mobility of the conductive filler and results in an increase in the electrical conductive pathway network.
3. It was observed that the critical weight percentages of the CNTs/G/EP nanocomposite was at a 5 wt% CNT concentration as the second filler and a 20 wt% concentration of epoxy resin as the matrix.

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