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Comparisons Among Electrical and Rheological Properties of Melt-Mixed Composites Containing Various Carbon Nanostructures

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The present investigation compares different carbon-based nanoscaled materials with regard to their effectiveness in producing thermoplastic polymers with antistatic and electrically conductive behavior. The dispersed phases are carbon black (CB) as spherical particles, multiwalled carbon nanotubes (MWNT) as fiber-like filler, and expanded graphite (EG) as platelet-like filler. Each was incorporated into polycarbonate by small-scale melt mixing. The electrical percolation concentrations were found to be 2 wt% for MWNT, 4 wt% for EG, and 8.75 wt% for CB which parallels the aspect ratios of the fillers. For EG a strong dependence of morphology and electrical resistivity on mixing time was observed, indicating a structural change/destruction during intensive shear mixing. Rheological percolation thresholds were found to be lower than electrical percolation threshold for the MWNT and CB, but similar in the case of EG. The general impact on complex melt viscosity decreases in the order MWNT, CB, EG. For EG, at higher loadings (above 4wt%) the viscosity increase with filler content is delayed as is the decrease in resistivity.

Keywords: nanocomposites, carbon black, expanded graphite, carbon nanotubes, melt mixing, electrical resistivity, melt rheology

1 Introduction

The state of the art in producing polymer-based materials with electrostatic dissipative or conductive behavior involves introduction of conductive fillers by melt mixing. Carbon black (CB) has been used for decades as a convenient and cheap additive for thermoplastics as well as for rubbers, for example in tire applications (1–7). It is known that the primary particles being in the nanometer size range form aggregates and agglomerates, and thus, fractal structures. The percolation of the conductive fillers, responsible for the conduction mechanism of the composite material having an insulating polymer as matrix system, is reached by percolation of such fractal aggregates. By this

mechanism, CB represents a fractal-like spectral dimension, d_s , between 1 and 2. In the last few years, special high-structured CBs were developed to produce antistatic and electrically conductive materials at concentrations much lower than expected from the geometrical percolation of spheres at 16 vol%. The spectral dimension describes effectively the connectivity of the carbon objects we compare in our study (8). During the last two decades, the potential of carbon nanotubes (CNT), as another allotropic modification of carbon, was explored (9). Here, the content necessary to get conductive pathways through the insulating matrices is much lower than that for CB, even when CB can form agglomerated structures. This is due to the fibrous shape with a very high aspect ratio (in the range of 1000 and higher) as compared to the approximately spherical shape in the case of the CB fillers. These different filler contents necessary for percolation can lead to different effects on other desired composite properties such as processability, gloss, and mechanical properties. In addition, CNT can result in mechanical enhancement due to the reinforcement effect of the fibrous filler with exceptional good mechanical properties. CNT can be regarded as a representative of a linear object with a spectral dimension of $d_s \approx 1$. However,

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even if costs are significantly reduced, CNT are still much more expensive than CB.

Additionally, there has now been interest in graphites or even graphene molecular sheets as fillers that combine high performance with moderate prices (10). Therefore, in addition to the already mentioned conductive fillers, expanded graphite (EG) was incorporated in nanocomposites as a representative of a platelet-like filler with a spectral dimension of $d_s \approx 2$. This material has gained increasing interest in scientific research for composite applications in the last few years. One can expect that it behave more effectively than CB with regard to percolation behavior. On the other hand, EG is much easier to produce as compared to CNT and has much lower prices.

Because of its industrial relevance, melt mixing was used here to incorporate the described nanoscaled fillers into a thermoplastic polymer. From the large variety of available CBs, a standard one was chosen. For CNT, a purified multiwalled carbon nanotube (MWNT) was selected. The expanded graphite was prepared at the University of Cincinnati. As matrix material, polycarbonate (PC) as an amorphous matrix material was chosen in order to exclude changes in crystallization that could be caused by the nanofillers.

In this way, the present study should clarify the potential of these fillers to achieve electrical percolation at low loadings. In addition, the rheological responses as indirect measure of the nanofiller dispersion and melt reinforcing effect will be compared and the relationships between rheology and nanocomposite morphology will be evaluated. A recent study (11) did compare electrical and mechanical effects on the stress-strain behaviour of CB, MWNT, and thermally-reduced graphite oxide (TrGO) in nanocomposites. This was done, however, in *different* polymeric matrices, such as styrene-acrylonitrile copolymer, PC, polypropylene, and polyamide 6 and no systematic investigation was done on percolation and rheological properties. In addition, the graphite oxide had been premixed using a solvent step before melt compounding, which limits direct comparisons. In any case, the TrGO was found to be percolating at lower or similar amounts than was found for the CB, whereas the MWNT material used showed higher or similar percolation thresholds than TrGO (11). In most studies on MWNT, in which CB was added as a benchmark, the percolation threshold of the nanotubes was much lower than that of the CB, as expected.

The present investigation should remove these uncertainties by comparing these fillers in the same polymer matrix, specifically PC.

2 Experimental

2.1 Materials and Processing

The CB chosen was Vulcan XC72 from the Cabot Corporation (USA). According to the supplier, this CB has a

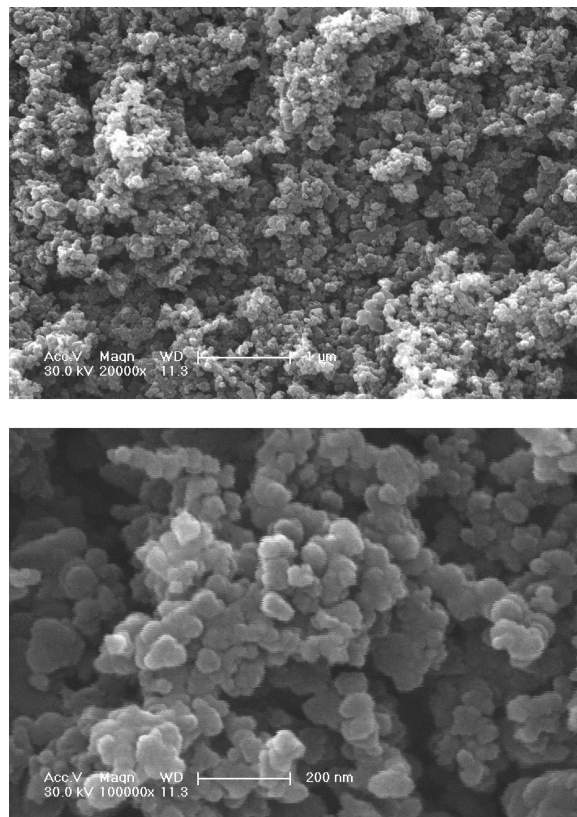


Fig. 1. SEM of carbon black Vulcan XC 72.

density of 270 g/l and BET measurements indicated a specific surface area (in N_2) of 254 m^2/g . The volatile content was 1.5%. As seen in Figure 1, the primary particles have sizes in the vicinity of 30 nm and show isolated compact structures of high apparent density without the branches that are typical for a less structured CB (12). This material has been the industry standard for imparting electrical conductivity in plastics for many years. In addition, it is the most widely used support material for direct methanol fuel cell electrodes (13).

Purified CNT multiwalled carbon nanotubes (MWNT) were obtained from Nanocyl S.A. (Sambreville, Belgium) (very thin straight and coiled nanotubes, purity >95%). A scanning electron microscopy (SEM) image is shown in Figure 2a, whereas Figure 2b shows a transmission electron microscopy (TEM) image. According to the supplier, the MWNT had been produced by chemical vapor deposition and had outer diameters between 5–15 nm, and lengths up to 50 μm .

The expanded graphite (EG) was prepared at the University of Cincinnati starting from flakes with an average size of 500 μm provided by Asbury Graphite Mills Inc. (USA). The expansion was performed by heating the acid-pretreated graphite flakes at 1000°C for 30–60 s in a manner similar to that reported in the literature (14). The structure obtained is shown in Figure 3. The SEM image illustrates the expanded character and the sheet-like structures

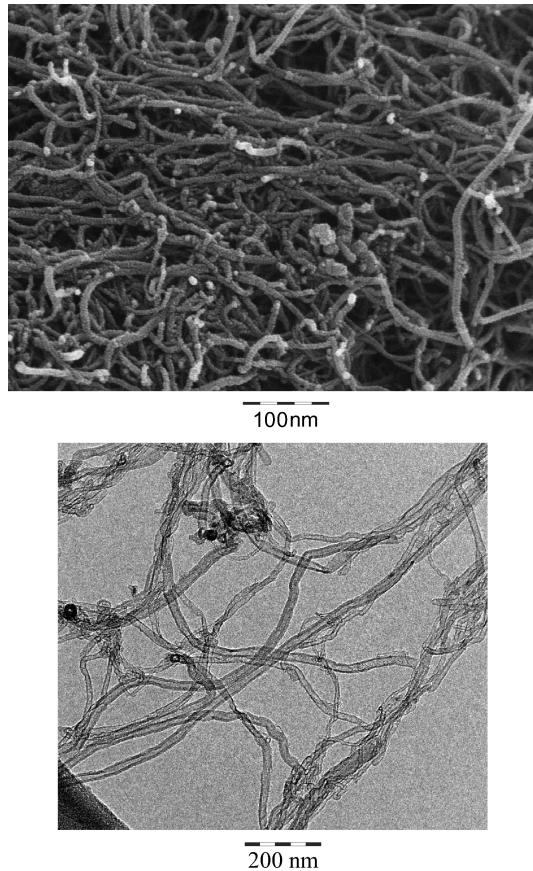


Fig. 2. SEM and TEM of MWNT (courtesy of Bayer Technology Services).

produced during the expansion. The sheet thickness is seen to be in the range below 100 nm, whereas the sheet areas are in the range of $20 \times 20 \mu\text{m}^2$ and about $200 \times 200 \mu\text{m}^2$. From the SEM images, the sheets appear to be very fragile.

Polycarbonate Iupilon E2000 (powder, Mitsubishi Engineering Plastics, Japan) was used as matrix polymer and was pre-dried at 120°C for at least 4 h under vacuum. The nanofillers were dried under vacuum at 120°C for at least 1 h. The EG and the PC powder were premixed in

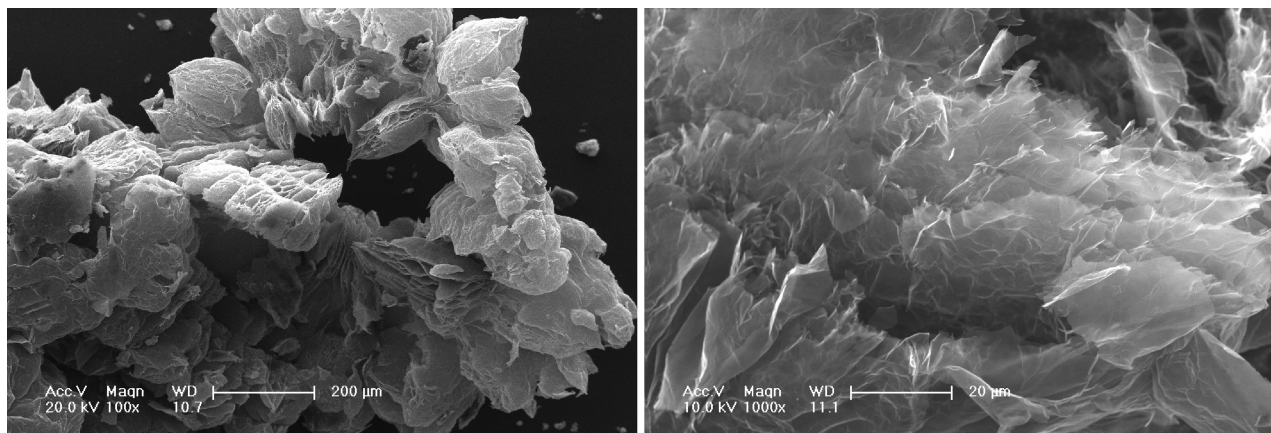


Fig. 3. SEM images of the expanded graphite.

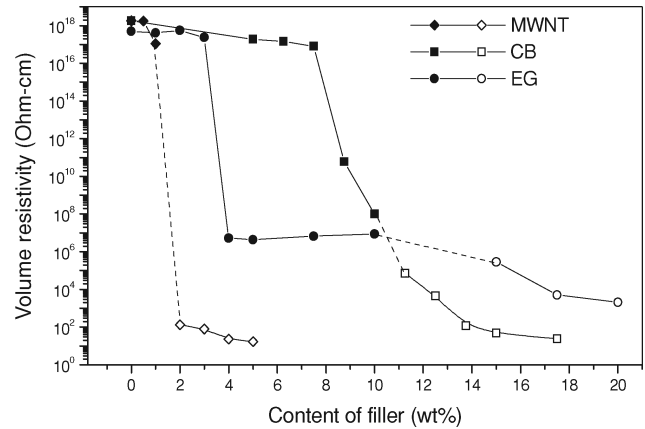


Fig. 4. Percolation of MWNT, CB, and EG in PC (filled symbols measured on plates, open symbols measured on strips).

the dry state, at various compositions, before being fed into an already-running mixer. The mixer was a DACA-Microcompounder (small-scale conical co-rotating twin screw compounder with a capacity of 4.5 cm^3 , from DACA Instruments, Goleta, USA). It was operated at a temperature of 280°C , a mixing speed of 50 rpm, and a mixing time of 5 (EG) or 15 min (MWNT, CB, and again EG). Sample plates (thickness 0.35 mm, diameter about 70 mm) of each composite were pressed at 280°C using a Vogt press under comparable conditions.

2.2 Composite Characterization

The electrical volume resistivity was measured using a Keithley 8009 test chamber (ring electrodes) combined with a 6517A electrometer on pressed plates (filled symbols in the plot in Figures 4 and 5). Samples with lower resistivity were measured using a 4-point fixture combined with a Keithley DMM 2000 electrometer on strips ($3 \times 20 \text{ mm}^2$) cut from the plates (open symbols). The values shown are mean values of 4–6 measurements at different positions, or on different strips.

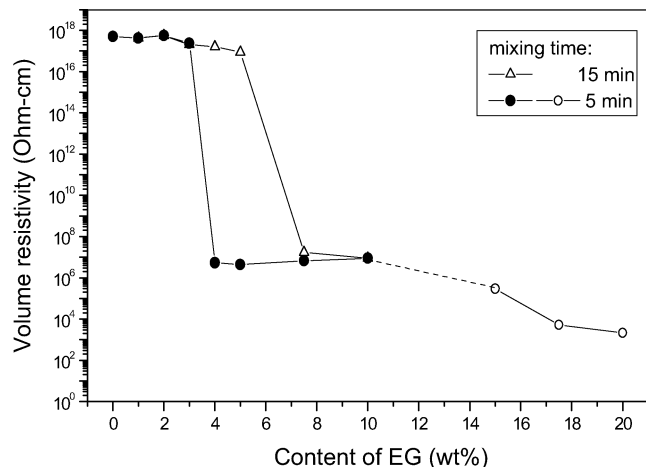


Fig. 5. Percolation of PC/EG at 5 and 15 min mixing.

SEM investigations on the nanofillers were performed using a FESEM (field emission environmental scanning microscope, XL30 ESEM-FEG Philips) in high vacuum mode at various magnifications. The nanocomposites were investigated using gold sputtered cryofractured samples of the pressed plates by means of a LEO VP 435 (Zeiss, Germany) microscope (MWNT and EG) or a XL30 ESEM-FEG Philips (CB).

Wide-angle X-ray scattering (WAXS) was executed on plates by a 4-circle diffractometer P4 (Bruker axs Karlsruhe) using $\text{Cu}_\alpha\text{-K}\alpha$ radiation in transmission mode. The radial diffraction was recorded between $2\Theta = 1.5$ and 40.5° using a HiStar/GADDS area detector. The samples were mounted in two manners: X-ray beam normal (perpendicular) as well as parallel to the plate surface.

Melt rheology was studied using an ARES rheometer (Rheometrics Sci., USA) in plate-plate geometry (25 mm diameter) under nitrogen atmosphere at 280°C by performing frequency sweeps at strains within the linear viscoelastic range. The second sweep ranging from 100 rad/s to 0.02 rad/s was used for interpretation. In preparation for these tests, the extruded materials were molded at processing temperatures into sheets with a thickness of 0.9 mm, from which 8 mm diameter discs were punched.

3 Results and Discussion

3.1 Electrical Resistivity

Figure 4 shows the electrical percolation curves for the three fillers in PC. The MWNT exhibits a sharp decrease in volume resistivity between 1 and 2 wt%, which is in agreement with results in previous investigations on similar samples (14). For CB the percolation is visible starting at 8.75 wt% (in accordance with the CB materials data sheet), whereas for EG (mixing time 5 min) the percolation starts at 4 wt%. These orders follow the expectation according to the aspect ratio of the nanofillers which is the highest in case of

MWNT (between about 3,000 and 10,000), but also high in the case of EG (about 200 to 2,000). The percolation value of CB reflects the structures of the CB primary particles within the fractal agglomerate structures. The importance of the degree of anisometric structure is clearly shown by these results.

Materials with resistivity values lower than $10^4 \Omega \text{ cm}$ can be regarded as electrically conductive, which is the case for the MWNT filled composites starting at 2 wt% and for the CB filled composites starting at 12.5 wt%. The composites with EG reach this resistivity level only at concentrations of 17.5 wt%, even if percolation occurred at much lower loadings. The resistivity values between 4 and 10 wt% are in the vicinity of $10^6 \Omega \text{ cm}$ and do not decrease significantly. Above 10 wt% a slight decrease up to $2 \cdot 10^3 \Omega \text{ cm}$ was observed, but these values still exceed the much lower values for MWNT ($17 \Omega \text{ cm}$ at 5 wt%) and CB ($24 \Omega \text{ cm}$ at 17.5 wt%).

The percolation thresholds found for the different fillers vary much more than those reported earlier (11), but this involved a different grade of PC. In this case, percolation was found at 2.5 wt%, independent of the type of filler (TrGO, CB, MWNT). The differences may also be due to be the different grades of nanofillers used, different pressing conditions and sample thicknesses, and different measurement devices and testing directions.

3.2 Influence of Mixing Conditions in Composites with Expanded Graphite

For the composites with EG the mixing time was found to play an important role on the resistivity obtained. Using a mixing time of 15 min, as used for MWNT and CB, percolation occurs at higher concentration, namely between 5 and 7.5 wt% EG, as can be seen in Figure 5. The dependence of resistivity on mixing time at 4 wt% indicated a sharp transition from values in the vicinity of $10^6 \Omega \text{ cm}$, to insulating materials at a mixing time of 7.5 min. This is shown in Figure 6. Therefore, it may be assumed that some

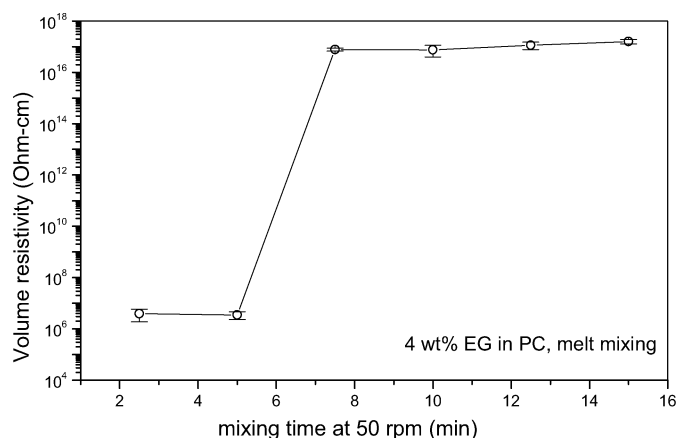


Fig. 6. Electrical volume resistivity vs. mixing time for 4 wt% EG in PC.

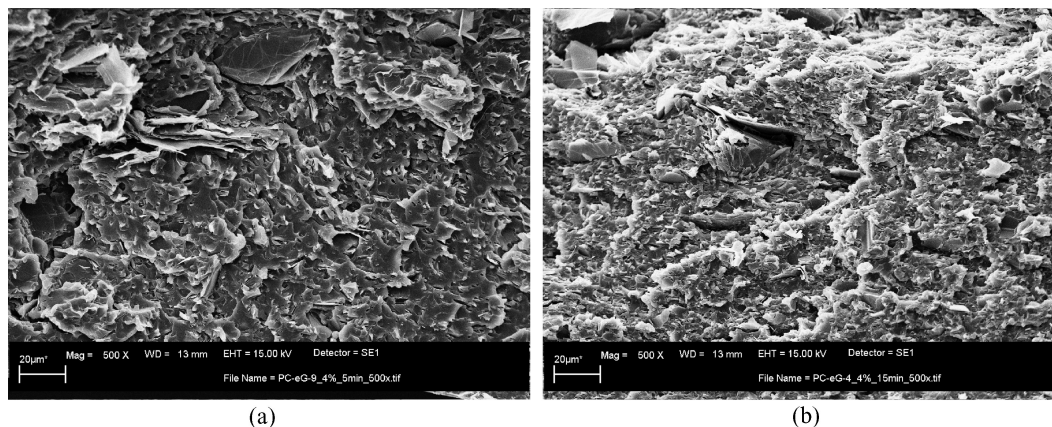


Fig. 7. SEM of cryofractured surfaces of PC/4wt% EG mixed at a) 5 min, b) 15 min.

changes in the structure of these composites occur during mixing. As demonstrated in Figure 2, the EG material consists of sheets; however they are not completely separated and seem to be quite fragile. SEM investigations on the composites were performed on compression-molded plates as used for resistivity measurements (Fig. 7). It is clearly evident that there are still stacks of EG layers in the material and that the stacks and sheets are mainly oriented in the plate direction. The planar size of the EG layers and thus the aspect ratio is significantly lowered by this, when comparing composites with 4 wt% EG prepared at 15 min vs. 5 min. It seems that the EG structure within the composites is very sensitive to the mixing process and that perhaps for some purposes melt mixing is not the method of choice due to the high shear forces typically involved.

3.3 Nanocomposite Morphology

SEM cryofractures of pressed sheets of selected samples are shown in Figures 7–9. In general, quite homogeneous distributions and dispersions can be observed for all composites. In the case of the EG (Fig. 7), which very thin sheets and small pieces of graphite layers are visible next to areas

containing relatively large stacks of nonexfoliated graphite sheets. This leads to the highest inhomogeneity among the fillers investigated. In addition, an orientation of the stacks and platelets parallel to the surface of the pressed plates is visible and this may be caused by the pressing process, especially through compression flows. This also induces an anisotropy in the resistivity. Up to 10 wt% filling the resistivity values lie in a range that required the measurements to be performed using ring electrodes, thus measuring through the thickness of the sample and perpendicular to the stacks and sheets of EG. Measurements in the sheet direction using the strip cell were not successful since the values were above the measuring range of the combination of this cell and the electrometer used. This may explain the plateau in resistivity between 4 and 10 wt% EG loadings. This anisotropy was investigated more in detail using WAXS, here presented for pure EG and composites with 4 and 20 wt% EG (Fig. 10). The WAXS signal intensity at $2\theta = 26.4^\circ$ ((002) reflection of the known hexagonal structure) scales with the amount of EG and the measurements in the plate direction show higher values than those perpendicular to the plate. In addition, the peak is slightly shifted to higher values and higher asymmetry when measured

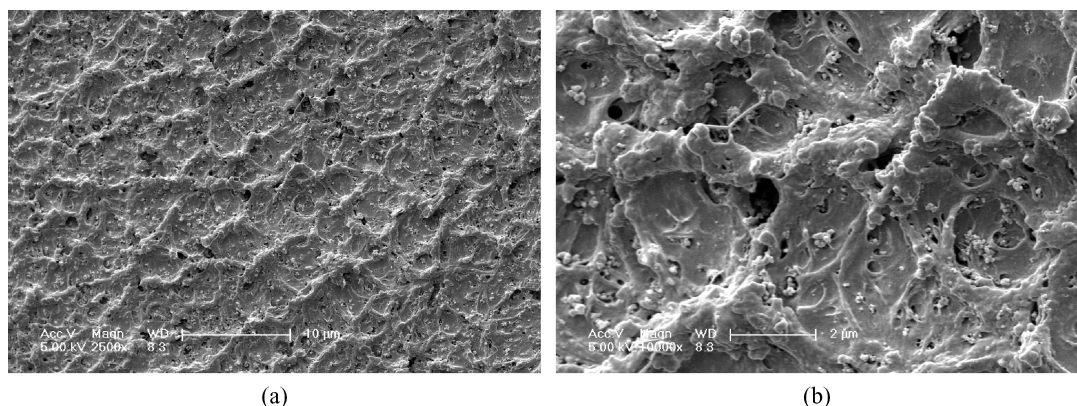


Fig. 8. SEM of cryofractured surfaces of PC/4 wt% CB.

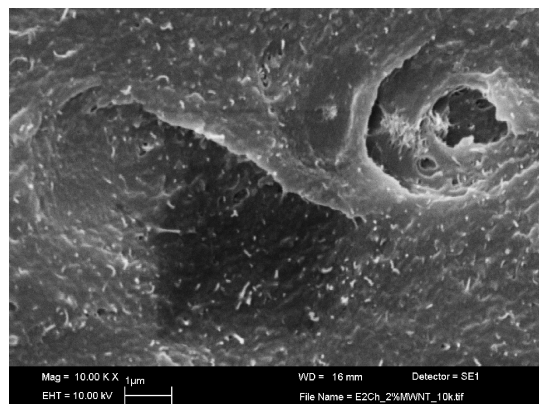


Fig. 9. SEM of cryofractured surface of PC with 2 wt% MWNT.

perpendicular to the plate axis. These effects were more pronounced at 20 wt% EG.

The 4 wt% CB sample showed a homogeneous distribution within the PC matrix as observed on the cryofractures, with a clustered structure within the CB being visible (Fig. 8). The sample with 2 wt% MWNT (Fig. 9) showed a very good dispersion into single nanotubes and a homogeneous distribution of nanotube parts visible at the surface of the fracture.

3.4 Melt Rheological Characterization

Melt rheological investigations are known to be suitable tools for studying the impact of the nanofillers in creating a combined polymer-filler network (15–18). Oscillatory melt rheology is particularly relevant here since polymer-mediated interconnected structures of anisometric fillers lead to marked qualitative changes in the spectra of dynamic moduli and viscosity. Specifically, the complex viscosity changes from a Newtonian plateau for pure polymers showing the shear thinning behavior to a continuous increase with decreasing frequency, whereas the storage modulus G' and loss modulus G'' flatten significantly and merge into a secondary plateau at low frequencies indicating the change from liquid-like to solid-like behavior due to the development of the elastic component. The melt rheological investigations on the different nanocomposites, as illustrated in Figure 11, indicated that MWNT have the highest impact on the complex viscosity, followed by CB and then the EG. The described change in the shape of the viscosity-frequency curve is observed for CB starting at 4 wt% and for MWNT already at 2 wt%. These values can be regarded as rheological percolation thresholds and are lower than the measured electrical ones, as is frequently reported in the literature when concentration steps are sufficiently small. This may be explained by the much smaller tube-tube distances required for electrical percolation than for a visible network effect in rheology,

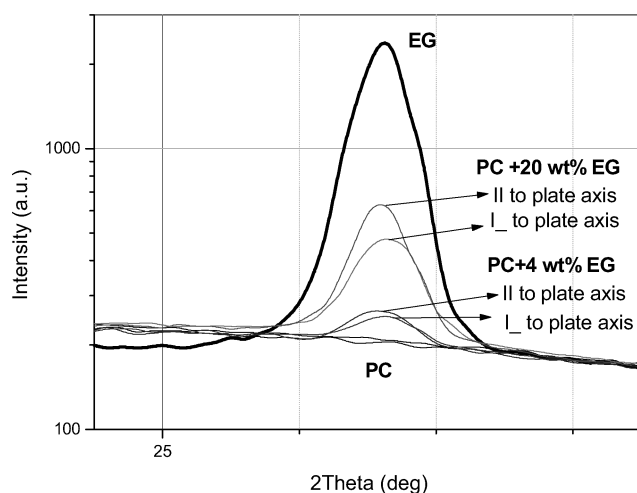
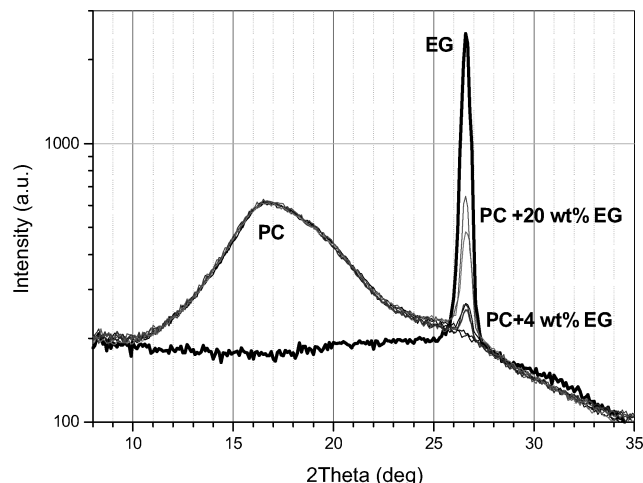


Fig. 10. WAXS curves of PC, PC with 4 wt% EG, and PC with 20 wt% EG measured in different directions, a) full pattern, b) detail graphite peak.

which results from combined effects of nanotubes and connecting polymer chains. This can be seen in illustrations presented elsewhere (17,18).

For EG, the general changes in melt viscosity upon filler addition are the lowest in these comparisons. The effect of increasing viscosity by lowering the shear rate is less pronounced, however as clearly shown earlier (19) using a van Gorp-Palmen plot rheological percolation could be detected starting at 4 wt% which coincides with the electrical percolation concentration. Up to 4 wt%, the effects in enhancing complex viscosity are nearly in the same range as in the CB composites; however, at higher concentrations the EG shows much lower viscosity increases than does the CB. This again may be an indication that the structure of the EG platelets inside the matrix has been changed, i.e., destroyed when filler content is increased, thus increasing the shear forces resulting during the mixing.

The effect of increasing viscosity at low frequencies as observed with 5 wt% MWNT, requires 15 wt% CB and

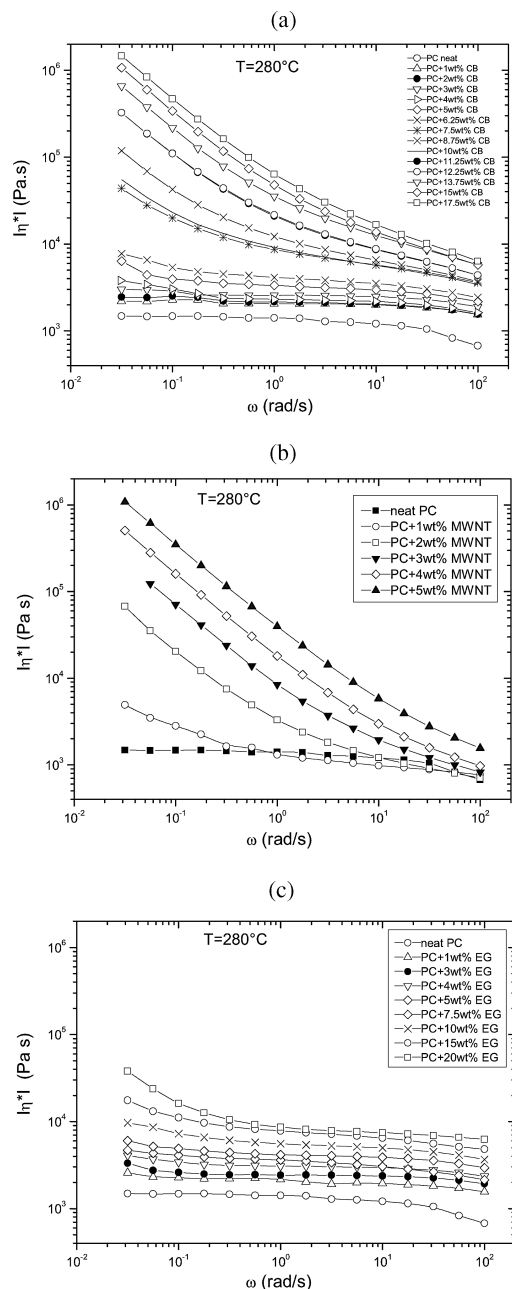


Fig. 11. Complex viscosity vs. measurement frequency for composites of PC with a) CB, b) MWNT, and c) EG.

can be not reached by EG until a loading of 20 wt%. Interestingly, at higher frequencies CB leads to higher viscosity enhancement than MWNT, as clearly seen when comparing both composites with 4 wt% filler content. This illustrates the higher shear thinning effect in the MWNT composites. A direct comparison of the complex viscosity $|\eta^*|$ and the storage modulus G' for composites with 4 wt% filler is shown in Figure 12. This illustrates again the largest impact for the MWNT, followed by the EG and CB.

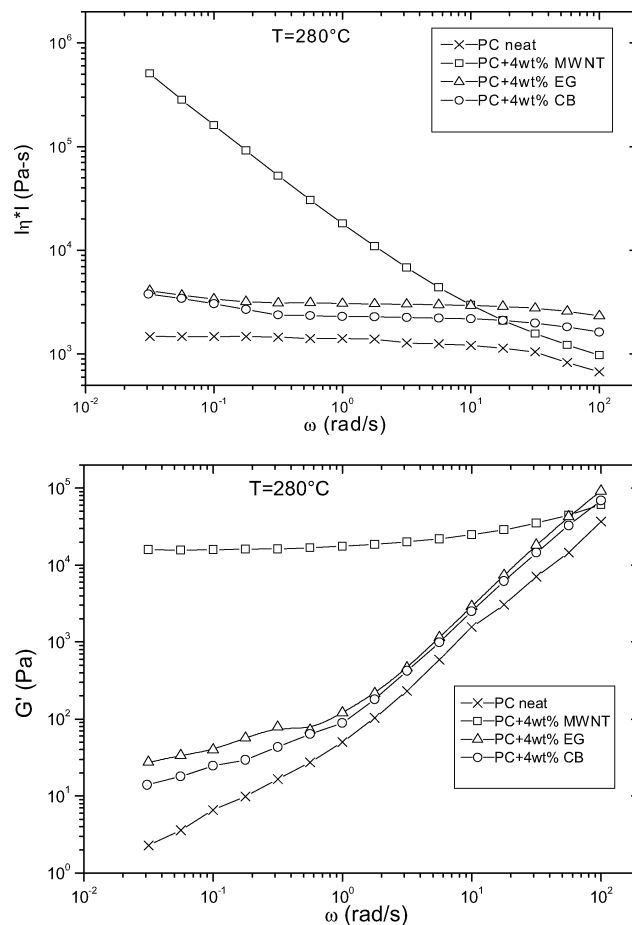


Fig. 12. Complex viscosity and storage modulus G' vs. measurement frequency for composites of PC with 4 wt% of the three carbon nanofillers.

4 Conclusions

The different conductive nanofillers under investigation in this study lead to quite different percolation behaviors. The selected high-purity MWNT with relatively low diameters percolated between 1 and 2 wt% and showed a steep decrease in resistivity by about 17 orders of magnitude! This is accompanied by significant changes in the complex melt viscosity at low measurement frequencies, starting as low as 1 wt%. The EG used in the investigations was found to percolate electrically between 3 and 4 wt%; however, the decrease in resistivity is only 11 decades and leads to values in the antistatic range. This filler showed a strong dependence of morphology and resistivity on mixing conditions. In particular, mixing time was found to increase the percolation threshold. The increase in viscosity with loading is the lowest in these comparisons, suggesting an alteration of the platelet structure occurs during long and intensive mixing, or at high filling levels. The CB showed the highest percolation concentration, between 7.5 and 8.75 wt%, and a more gradual decrease in resistivity as compared to MWNT but

reaching similarly low values at high loadings. The impact on melt viscosity is much lower than for MWNT. Near the percolation concentration EG shows a slightly higher effect in melt rheological properties but above this concentration, CB shows larger effects.

The results indicate that EG is a good candidate for cost-effective conductive filler materials for antistatic applications. Its percolation concentration is significantly lower than that of CB, is less expensive, and is established in industrial productions. The better performance of MWNT still does not justify its much higher price if one looks only for electrical properties. However, the multifunctional properties in also enhancing other properties such as mechanical behavior, fire retardancy, dimension stability, etc. should be taken into account. The results also indicate possible difficulties in melt processing expanded graphite but such problems can be reduced when using graphite platelets obtained by other strategies such as thermally reduced graphite oxides (11).

Acknowledgments

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