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Morphology and electrical resistivity of melt mixed blends of polyethylene and carbon nanotube filled polycarbonate

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Abstract

Blends were prepared from a high density polyethylene (PE) material and a conductive polycarbonate (PC) composite containing multiwalled carbon nanotubes (MWNT). The PC composite contained 2 wt% MWNT and was prepared by diluting a PC masterbatch consisting of 15 wt% MWNT by melt mixing in an extruder. The aim of blending a conductive PC composite with PE was to obtain a conductive blend at lower amounts of MWNT than required for the pure PC component. The blend morphology was found to be co-continuous over a broad composition range of 30–80 vol.% of the filled PC phase as verified by selective extraction of PC and morphological investigations. The co-continuous structure is very fine with ligaments in the range of 1 μm. Significantly reduced volume resistivity values, i.e. in the range of 10^7 Ω cm, could be achieved in the same composition range of that of the continuous PC phase, i.e. starting at compositions of 30 vol.% filled PC. Here, the total MWNT content in the blend was only 0.41 vol.%. Interestingly, even if the MWNT had been incorporated in the PC phase, the tubes are still visible in the blends after selective extraction of PC. Scanning electron microscopy investigations revealed that the MWNT bridge the PC and PE phases, at least with their ends. This can be understood by the length dimensions of the tubes, which is higher than the phase sizes in the blends.

Keywords: Blends; Multiwalled carbon nanotube composites; Electrical resistivity

1. Introduction

Carbon nanotubes (CNT) have been shown to be an attractive filler for increasing the electrical conductivity of polymers at relatively low CNT contents. This efficient behavior is caused by the excellent electrical properties (CNT are as conductive as copper) and the very high aspect ratios, p ($p = L/D$, where $L =$ length, 1–50 μm and $d =$ diameter, 1–50 nm) of the fibrous CNT. The percolation concentration, i.e. the filler concentration at which a pathway of conductive filler is formed through the matrix, which is manifested in changes of resistivity over decades, is inversely proportional to the aspect ratio of the CNTs. According to theoretical calculations or cylindrically shaped fillers [1], the onset of percolation of polymer/MWNT composites is expected to begin at ~0.05 vol.% filler (assuming an aspect ratio of 1000). The lowest percolation concentrations reported in the literature are 0.04 wt% for an epoxy/multiwalled carbon nanotube (MWNT) composites [2] and 0.05 wt% found for polypropylene/MWNT films [3]. Most experimental studies on polymer/MWNT composites, however, report percolation between 1–5 vol.% [4–8] which is still much lower than the filler concentration necessary for percolation in systems with carbon black (CB) or metallic fillers. In addition, mechanical enhancement is expected by using CNTs which exhibit very high moduli and strength [9–11].

Based on the above characteristics of CNT, it is also promising to use them as additives in polymer blends for electrically conductive or antistatic applications and achieving, at the same time, the well-known advantages of polymer blending for tailor-made material properties. When dispersing CNTs in the blends of two or more polymers that are immiscible, the conductive filler can be located either in one or both of the phases or at the interface. The resulting electrical properties will depend on the location of the conductive filler as well as on the blend morphology; this has been shown extensively for carbon

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black filled polymer systems. For CB filled blends, Sumita et al. [12] first introduced the concept of 'double percolation' in which the percolation of the carbon black occurs in a phase that is also continuous thus creating a second percolation. Gubbels et al. and Calberg et al. [13–15] showed that the selective localization of CB at the interface between PE and PS that form a co-continuous blend can dramatically reduce the amount of CB needed for percolation. For example, a PE/PS = 45/55 wt% blend requires only 0.5 wt% CB to achieve resistivities lower than $10^4\ \Omega$ cm [13]. This selective localization at the interface depends on the polymer/polymer interfacial tension relative to the interfacial tensions of CB and the individual polymers. On the other hand, the composition range in which co-continuous structures are found as well as the stability of this structure are enhanced by the CB filler [14]. This effect was explained by enhancement of melt viscosity leading to a reduction of coalescence in the filled polymer phase(s). Investigations on short carbon fiber filled polymer blends were reported by Zhang et al. [16]. Vapor grown carbon fibers having an average diameter of 200 nm and lengths on the order of 10 μm were added to premixed blends of HDPE and PMMA. The filler content needed for percolation in a 50/50 blends was 1.25 phr which was much lower than the percolation thresholds of the individual components. This was attributed to a segregation of carbon fibers in the PE phase due to their higher affinity for PE relative to PMMA.

Recently, GE Plastics introduced a conductive nanotube filled, impact-toughened polyphenylene ether (PPE)/polyamide (PA) blend (grade name NORYL GTX 990EP resin) in which PA 6,6 forms the matrix and PPE the dispersed phase [17]. Addition of MWNT to this blend system requires relatively low nanotube loadings to achieve resistivities lower than $10^6\ \Omega$ cm, thus making this material suitable for electrostatic painting applications [17]. Aside from this particular blend system, few reports are available in the literature involving the use of carbon nanotubes in polymer blends. Valentini et al. [18] showed results concerning the addition of SWNT into PP-EPDM blends and studied the influence of the nanotubes on the crystallization behavior of the components and the dynamic mechanical behavior at a given composition of PP/EPDM = 70/30. At this composition the blends exhibit matrix-dispersed particle structures. Electrical properties were not investigated.

This paper deals with blends of polyethylene (PE) and polycarbonate (PC) by using carbon nanotubes as a conductive filler. Carbon nanotubes are first introduced into polycarbonate using a masterbatch technique, which are expected to remain in this phase during melt mixing with PE. This blending strategy is indented to produce conductive blends containing much lower contents of CNT than required for the pure PC component. It is of interest here to also investigate the applicability the concept of double percolation for the given blend. In order to get conductive blends as a result of mixing, a continuous structure of the filled PC phase is necessary. In other words, the filled PC should form the matrix of a dispersed blend or exhibit a continuous phase in a co-continuous blend. PE was selected as a model component because of its immiscibility with PC.

2. Experimental

2.1. Materials and blend preparation

High density polyethylene (PE, Lupolen® 4261A, BASF AG, Germany) was blended with a PC composite containing 2 wt% MWNT (PC-2NT) to produce a full range of blend compositions. Compounding of these blends was carried out with a 4 g capacity DACA Micro Compounder (DACA Instruments, Goleta, USA) operating at 260 °C, 50 rpm, and a mixing time of 5 min. Additional experiments were conducted using longer mixing times (up to 15 min) and higher mixing speeds (up to 150 rpm). The original PC-2NT composite was produced by diluting a masterbatch of 15 wt% MWNT in polycarbonate, supplied by Hyperion Catalysis International, Inc. (Cambridge, MA, USA), with pure PC (Iupilon E-2000, Mitsubishi Engineering Plastics) using a Haake co-rotating, intermeshing twin-screw extruder ($D = 30$, $L/D = 10$) as previously described [7]. The final blends obtained after melt compounding in the DACA Micro Compounder were formed into a continuous strand using a heated cylindrical die (2 mm diameter, length 35 mm). The strands were placed on an aluminium plate without additional cooling or drawing. An additional blend using PC with 5 wt% (PC-5NT) in a PC-5NT/PE = 45/55 vol.% composition was prepared at 260 °C, 150 rpm, and a mixing time of 5 min.

2.2. Rheological measurements

Melt rheological properties were obtained using an ARES oscillatory rheometer (Rheometric Scientific) at 260 °C in nitrogen atmosphere using a parallel plate geometry (plates 25 mm diameter, gap of 1–2 mm). Granules and/or small pieces of extrudate strands were placed between the preheated plates and allowed to equilibrate for about 3 min. Frequency sweeps were performed between 0.1 and 100 rad/s at strains within the linear viscoelastic range. Repeated sweeps with increasing and decreasing frequency were performed in order to check the stability of the sample over time (each sweep requires approximately 8 min). In most cases, the second sweep was interpretated.

2.3. Electrical resistivity

The volume resistivity was measured on compression molded samples (diameter 60 mm, thickness 0.35 mm) which were prepared by pressing extrudate strand pieces using a Voit press at 260 °C. A Keithley electrometer Model 6517 equipped with a 8002A High Resistance Test Fixture was used to measure high resistivity samples. For lower resistivity blends and PC-2NT samples of 20 mm in length and 3 mm in width were cut from the pressed films and measured with a 4-point test fixture equipped with gold contact wires (distance between the source electrodes 20 mm and between the measuring electrodes 7.1 mm) using a Keithley electrometer Model 2000. The samples were thoroughly cleaned with ethanol prior to measurements. The measured volume resistance, R_v, was converted to volume resistivity, ρ_v, according to ASTM D4496 and D257 using the formula

$$\rho_v = R_v A/t \tag{1}$$

(where $A =$ effective area of the measuring electrode and $t =$ specimen thickness).

2.4. Microscopic characterization

Test specimens for atomic force microscopy (AFM) were prepared by cutting the strand perpendicular to the extrusion direction at room temperature using a Leica RM 2155 microtome (Leica, Germany) equipped with a diamond knife. Blends with PE were cut at approximately −60 °C. The AFM measurements were carried out in the tapping mode using a Dimension 3100 NanoScope IV (Veeco, USA) [19].

Scanning electron microscopy (SEM) was performed on samples coated with gold using a LEO VP 435 scanning electron microscope (Leo Elektronenmikroskopie, Oberkochen, Germany). For select samples, a low voltage Zeiss Gemini DSM 982 scanning electron microscope (Zeiss, Germany) was used on uncoated samples. Photomicrographs were taken of cryofractured extruded strands. In some cases the strands were selectively dissolved with chloroform for 4 days at room temperature to remove the PC portion of the blend prior to cryofracturing.

2.5. Determination of PC phase continuity by selective extraction

Extruded strands of blends, ~2 cm length, were dried for 8 h at 80 °C and subsequently immersed in 50 ml of chloroform at room temperature for 4 days. After the first day the chloroform-PC solution was replaced by fresh chloroform. The extracted strands were then rinsed in fresh chloroform and dried at 105 °C to remove the chloroform. The phase continuity was calculated using the ratio of the dissolvable PC part to that of PC in the blend, where the dissolvable PC part is the weight difference between the sample before and after extraction and the PC part in the blend is calculated from the sample weight before extraction and the weight percentage of PC. The continuity gives a measure of the fraction of PC associated with the continuous PC phase. The reported values are the average of two samples.

3. Results and discussion

3.1. Blend morphology and co-continuous composition range

The aim of mixing PE with a carbon nanotube filled PC material was to form a co-continuous phase structure between both polymeric components such that the filled PC forms a continuous phase acting as electrical pathway through the sample volume. The formation of co-continuous structures in melt mixed blends primarily depends on the viscosity and elasticity ratios between the blend components at the mixing conditions, whereas the interfacial tension is mainly related to the stability of the morphology [20]. To achieve continuity of the filled PC at low concentrations, the PC phase should have a melt viscosity lower than PE under the given mixing conditions. However, as it was shown in a previous paper [7] the melt viscosity of nanotube filled composites is increasing significantly above the filler percolation threshold. This is much more pronounced at low oscillation frequencies but also found at shear rates as applied during mixing. Thus a high viscosity PE material was selected to ensure that its viscosity was higher than that of the filled PC over a broad range of frequencies. The viscosity curves of the PC-2NT, neat PC, neat PE, and a blend of PC-2NT with PE are shown in Fig. 1 at 260 °C. Comparing pure PC with the extruded PC-2NT composite clearly shows that a qualitative change in the viscosity vs. frequency behavior has taken place. At low frequencies, the PC shows a Newtonian plateau leading to a zero shear viscosity of 2670 Pa s, while the filled PC shows an increasing viscosity with decreasing the frequency without reaching a Newtonian plateau. This behavior is caused by the percolated structure of the MWNT within this sample [7]. The PE selected shows a linear decrease in viscosity vs.

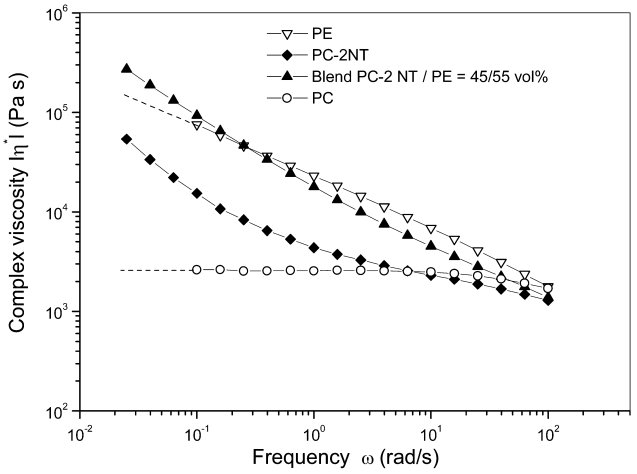


Fig. 1. Complex viscosity vs. oscillation frequency at 260 °C.

frequency and is absent of a Newtonian plateau under the measurement conditions. The higher melt viscosity of the PC-2NT/PE = 45/55 vol.% blend at very low oscillation frequencies relative to its constituting components is already indicative of the formation of a co-continuous structure [20,23]. The mean shear rate at 50 rpm inside the DACA Micro Compounder is estimated to be $\sim 50\ \mathrm{s}^{-1}$ using the approach proposed by Dedecker and Groeninckx [21]. At the shear stress of PE corresponding to this shear rate the ratio of PC-2NT viscosity to that of PE is 0.45. The phase inversion (PI) for these materials is expected to occur at a concentration ($\phi_{\text{PC-2NT, PI}}$) between 18 and 52 vol.% of PC-2NT in PE, according to the Metelkin and Luciani models, respectively [20] (Fig. 2). The most widely used model for phase inversion by Utracki (see Ref. [20]) predicts a phase inversion composition at 41 vol.%. However, these models do not give any information about the co-continuous composition range which is expected to encompass the phase inversion composition.

A viable method to investigate the co-continuous structure composition range is selective extraction. For the blend system used, PC can be extracted using chloroform, which does not dissolve, swell, or penetrate the polyethylene. Thus, the part of PC which can be extracted from the sample originates from the part of PC taking place in a continuous PC structure. An important criterion in selective extraction is that the sample after extraction must be self-supporting. Extraction experiments on strand samples met this criterion for concentrations up to 80 vol.% PC-2NT. In addition, the strand samples did not change in shape or size following extraction. However, even if the MWNT were incorporated into PC, the chloroform-PC solution obtained during extraction was clear and the strand pieces remained the black color typical for all nanotube filled blends.

Fig. 3 shows the continuity index of PC as measured by the selective extraction experiments. This figure indicates that the PC forms a fully continuous structure between 30 and 80 vol.% PC-2NT. At 20 vol.% PC-2NT, 81 wt% of the PC takes place in a continuous structure while 19 wt% exists in PE as dispersed particles. Interestingly, this co-continuous composition range is very broad as compared to values reported in the literature (see references within [20]).

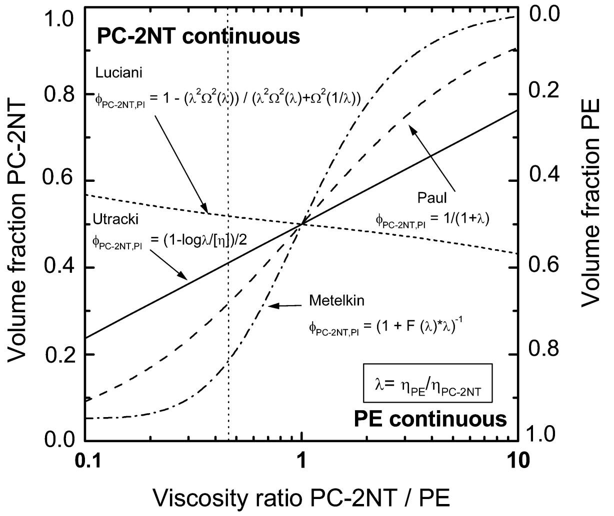


Fig. 2. Expected phase inversion composition according to different theoretical models [20] for PC-2NT/PE blends; the viscosity ratio of the blend system can be assumed to be 0.45. The symbol *Ω* represents the Tomotika function.

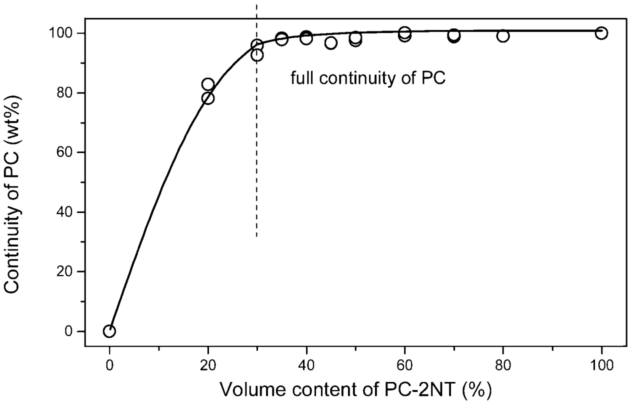
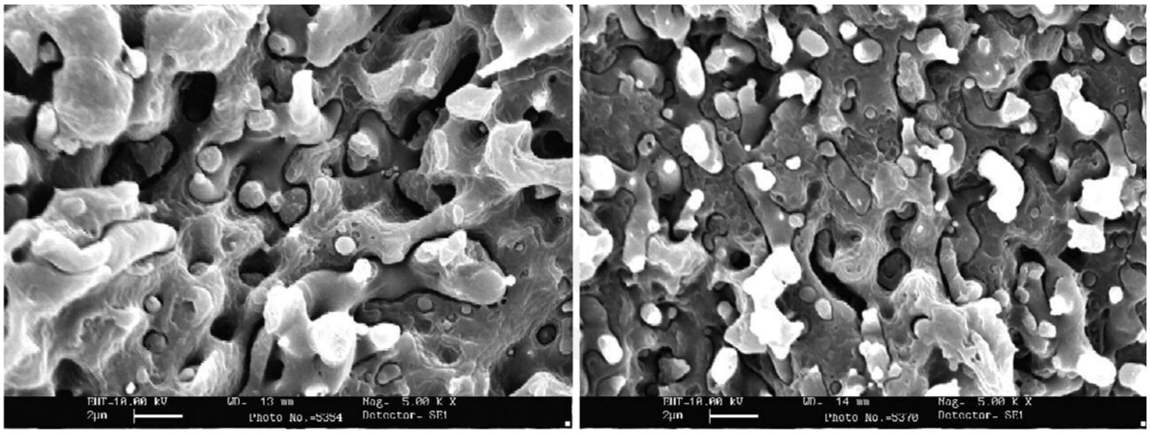


Fig. 3. Continuity of PC as measured by selective extraction of PC using chloroform.

SEM micrographs of cryofractured samples shown in Fig. 4 provide visible evidence of a co-continuous structure, in addition, illustrate the poor adhesion between the two polymer phases. From AFM micrographs (Fig. 5) we can conclude that the phase having higher stiffness and smoother surface corresponds to the phase containing PC.

Fig. 6 shows SEM micrographs taken of different blend compositions that were cryofractured after selective extraction of PC. Each micrograph reveals a co-continuous structure in which PE forms a continuous phase. At 20 vol.% PC-2NT, some spherical inclusions of PC in the remaining PE phase are visible. These particles were not dissolved during the selective PC extraction procedure since they were isolated within the PE and not accessible by the solvent. The co-continuous structure is very fine having ligaments in the range of 1 μm and varies only little in size over the entire range of compositions. Interestingly, above approximately 40 vol.% PC-2NT the appearance of the structure changes from predominantly concave PE surfaces to predominantly convex PE surfaces. This composition coincides with the phase inversion composition predicted using the Utracki model. Starting at this composition, the structure contains a higher number of dispersed elongated PE particles connected by thin ligaments with increasing PC-2NT concentration. This effect is especially pronounced at 80 vol.% PC-2NT (see also Fig. 8(b)) where according to the volume ratio of PE and PC-2NT particles of PE are expected to be dispersed within the PC phase. However, it should be noted that at this composition the strand shape and size was unchanged after selective PC extraction indicating that the PE forms a continuous phase.

Variations in melt mixing conditions lead to minor changes in the structure as seen in Figs. 4 and 7 for blends



a)

b)

Fig. 4. SEM micrographs of cryfractured surfaces of PC-2NT/PE = 35/ 65 vol.% blends; (a) screw speed 50 rpm; (b) screw speed 150 rpm.

containing 45 vol.% PC-2NT. Increasing the screw speed from 50 to 150 rpm leads to a finer co-continuous morphology (see Fig. 4(a) and (b)), while increasing mixing time from 5 to 15 min (compare Figs. 6 and 7(a)) has little effect on the morphology, thus, revealing the stable nature of the co-continuous morphology. In Fig. 7(b) the morphology of a blend containing PC-5NT is shown at the same blend composition. The morphology is quite similar to that of the corresponding blend with PC-2NT.

3.2. The effect of nanotube bridging

The blends described in this paper exhibit a stable co-continuous structure over an unusually large composition range. A reason for this behavior can be found by taking a closer look at the morphology. SEM on the extracted samples reveals that the nanotubes remain inside the sample after dissolving the PC phase and seem to connect the structure elements of the PE phase and/or different PE particles. This phenomenon, visible in Figs. 5–7, is shown in greater detail in Fig. 8. In the extracted blend samples, tubes are visible at the surface of the cryofractured strand and in some cases they connect PE domains. In Fig. 8(a) and (b), the tubes are partially visible along their length and appear to be stretched. The actual length of the nanotubes is more easily viewed in these micrographs of blends than those taken previously on PC-MWNT composites [19]. Fig. 8(c) shows that the observed tubes are not an artifact produced by cryofracturing the extracted strands. The AFM micrograph of the cryocut (non-extracted) surface also reveals bridging between the phases, as indicated by circled regions. This bridging behavior indicates that the tubes were not aligned within the continuous PC phase during the processing of the co-continuous blends. Previous studies from this laboratory have shown that nanotubes within PC-MWNT composite strands are randomly oriented in the PC and do not align in flow direction under the extrusion conditions selected [19]. Even if the phase size of the PC is much smaller in the co-continuous blends with PE (about 1 μm) as compared to the strand diameter (2 mm) in PC-MWNT composites, we may also assume that MWNT fail to align within the PC phase in the co-continuous blends. In fact, the nanotubes may tend to migrate to the PE phase owing to their good affinity for polyolefins [12]. Such behavior has been observed in blends of polyethylene filled with carbon black [12,13,22]. The level of migration, however, may be sizably less owing to the nanotube shape.

According to investigations on the mechanism of formation of co-continuous structures by melt mixing, the process of morphology development likely involves the breakup processes of thin sheets of both components into a lacy-like structure and to fibrous filaments which can break up to droplets (see Ref. [20]). These breakup mechanisms seem to be delayed by the nanotubes since they enhance the viscosity of the filled component, as well as hinder the breakup of sheets or filaments mechanically. The latter effect may be explained by the length dimensions of the tubes, which are larger in size than the phase sizes (ligament sizes) of the PC phase in the blends. This stabilizing effect for the co-continuous morphology could be a major reason

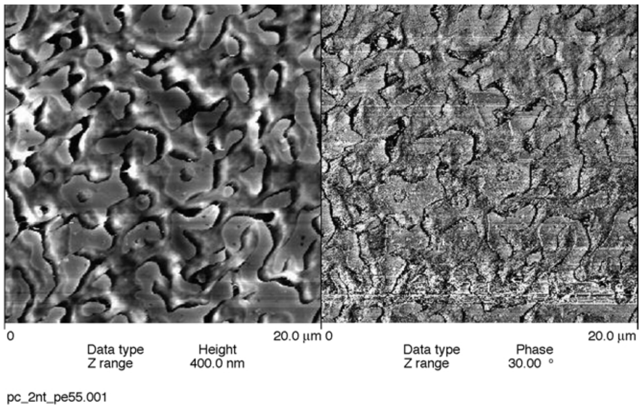
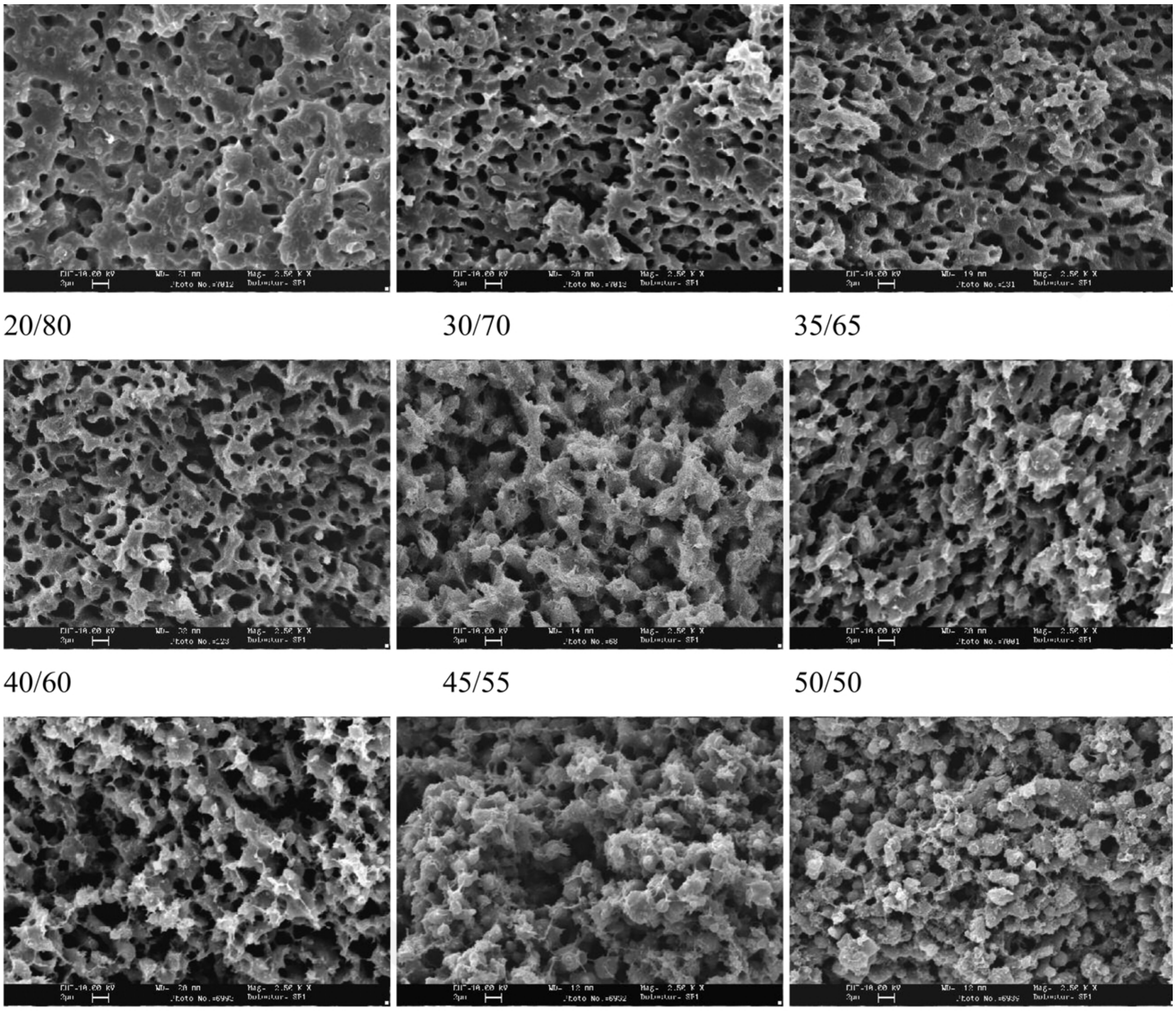


Fig. 5. AFM micrograph of the blend PC-2NT/PE = 45/55 vol.% (left height image, right phase image).



20/80

30/70

35/65

40/60

45/55

50/50

60/40

70/30

80/20

Fig. 6. SEM micrographs of cryofractured extracted strands of different PC-2NT/PE (vol.%) blends.

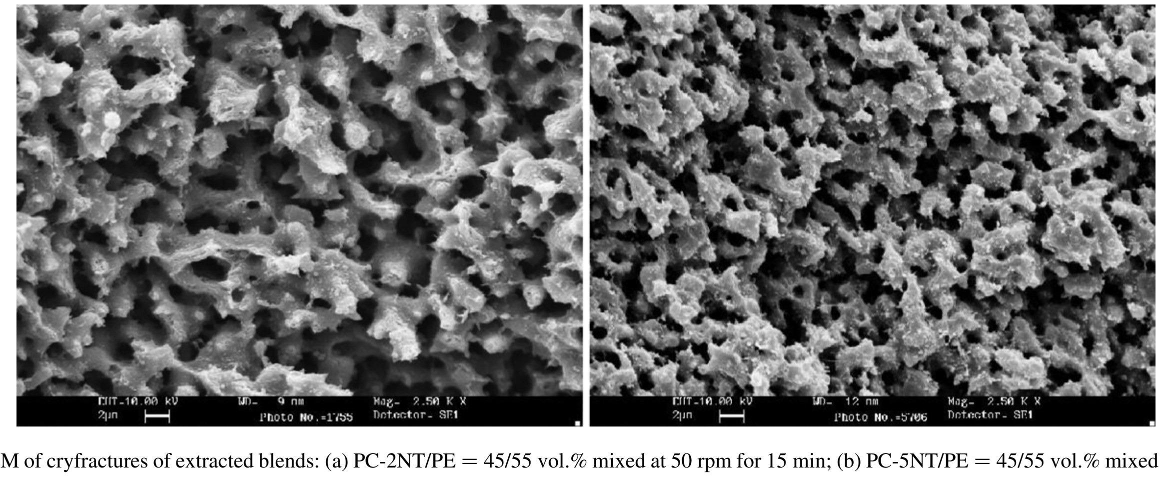
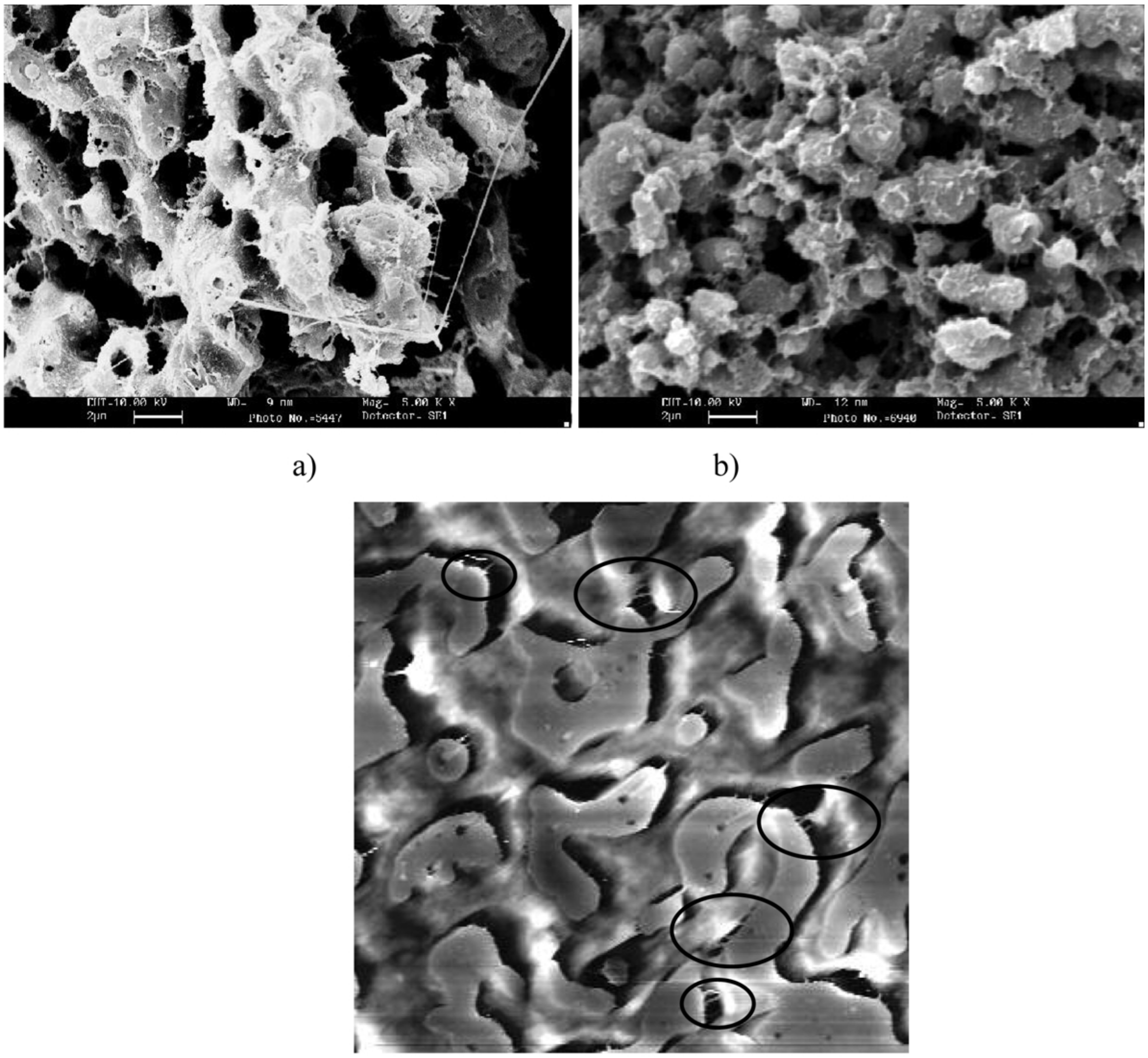


Fig. 7. SEM of cryfractures of extracted blends: (a) PC-2NT/PE = 45/55 vol.% mixed at 50 rpm for 15 min; (b) PC-5NT/PE = 45/55 vol.% mixed at 150 rpm for 5 min.



a)

b)

c)

Fig. 8. Nanotubes bridging the PE and PC phases: (a) SEM micrograph of cryofractured extracted sample of (a) PC-2NT/PE = 45/55 vol.%, (b) PC-2NT/PE = 80/20 vol.%, (c) AFM micrograph of cut surface before extraction; height image, picture frame 12 × 12 µm.

that the co-continuous composition range is very broad in this system and that the morphology fineness does not differ very much at different compositions.

In this context, it seems unlikely that the nanotubes, owing to their dimensions, can be located at the interface between both phases, as it was shown for carbon black.

3.3. Electrical resistivity

Electrical resistivity measurements reveal that conductivity of the blends is strongly related to morphology. Fig. 9 shows a significant drop in resistivity by about seven decades starting at 30 vol.% PC-2NT which correlates to the lowest PC-2NT composition of the fully co-continuous range. The PC-2NT phase still exhibit a percolated structure, however, the resistivity values are not as low as in the PC-2NT phase itself. This is due to the localization of parts of nanotubes within the PE phase reducing the number

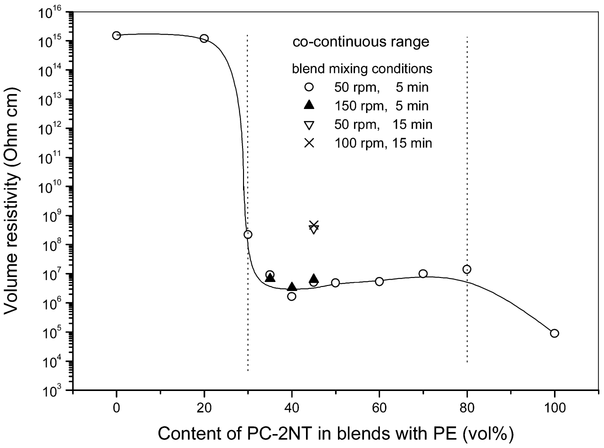


Fig. 9. Volume resistivity vs. PC-2NT content in blends with PE.

of nanotube–nanotube contacts. The higher resistivity values of the pure PC-2NT material shown here as compared to those presented in Ref. [7] is due to the additional second extrusion step of the filled material which seems to cause attrition of the MWNT as discussed in Ref. [24]. For the blends, increasing mixing speed leads to comparable resistivity values despite finer structures observed at higher speed (compare Fig. 7). Longer mixing time enhances the resistivity slightly, which may also be attributed to reduced nanotube lengths. The PC-5NT/PE = 45/55 vol.% blend prepared at 150 rpm for 5 min exhibited a volume resistivity of $5 \times 10^6\ \Omega$ cm that exhibits a considerably higher resistivity than that of the extruded PC-5NT sample whose value is 35 Ω cm. The attrition of nanotubes at high loadings and with mixing time may be more pronounced due to the nanotube bridging.

Another interesting point is that blends of selected compositions retain low resistivity values even after extraction of the PC phase. PC-2NT/PE = 45/55 vol.% blend strand pieces were pressed to sheets (diameter 60 mm, thickness 0.35 mm) and their resistivity were compared with those of extracted sheets after dissolving the PC part. The volume resistivity was nearly unchanged in the range of $10^8\ \Omega$ cm. This result demonstrates the potential use of such co-continuous blends as basic material for the production of conductive/antistatic membranes.

Even though the blends resistivity values are not in the range of 'conductive materials', their resistivity is in the range of dissipative materials. At a composition of 30 vol.% filled PC, the total content of MWNT in the blend is only 0.41 vol.% (based on a volume concentration of 1.37 vol.% in the PC-2NT composite [7]).

4. Summary and conclusions

Blends of PC filled with MWNT and polyethylene were prepared. The aim of blending a conductive PC composite with PE was to obtain a conductive blend at low contents of MWNT by using the concept of double percolation through the formation of a co-continuous morphology. For this purpose, a PC-2 wt% MWNT composite was selected due to its conductive nature. In the melt mixed blends, fully co-continuous morphologies could be obtained in the composition range between 30 and 80 vol.% of the filled PC phase, which was verified by selective extraction of the PC from the extruded strands. The co-continuous structure is very fine with ligaments in the range of 1 μm and shows only marginal differences in phase size with blend composition. Significantly reduced resistivity of the blends can be achieved in the same composition range in that PC forms a continuous phase, i.e. starting at compositions of 30 vol.% filled PC. Here, the total MWNT content in the blend was only 0.41 vol.%. The resistivity values depend on the mixing conditions, e.g. screw speed and mixing time. Nanotube attrition seems to be the primary reason why resistivity values increase with mixing time.

Interestingly, even if the MWNT had been incorporated in the PC phase, the tubes are still visible in samples after extraction of PC. SEM investigations revealed that the MWNT bridge the PC and PE phases at least with their ends. This is likely attributed to the large length dimensions of the tubes, which are larger than the dimensions of phases in the blends. This bridging effect seems to be responsible for the excellent blend stability and the broad co-continuous composition range.

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