Dispersion of carbon nanotubes in poly(ether ether ketone) and its effects on rheological and electrical properties

A range of multi-wall carbon nanotubes (MWNTs) and single-wall carbon nanotubes (SWNTs) reinforced high temperature semi-crystalline poly (ether ether ketone) (PEEK) were prepared by melt compounding process. The composites with high degree of nanotube dispersion show nearly five orders of magnitude increase in storage modulus and an abrupt increase of ten orders magnitude in electrical conductivity by adding only 2 wt% of nanotubes. Additionally, both the melt strength and the elongational viscosity can significantly increase by incorporating nanotubes. However, this increment effect strongly depends on the degree of dispersion of nanotubes in the polymer matrix.

Dispergierung von carbon nanotubes in poly(ether ether keton) und deren Effekte auf die rheologischen und elektrischen Eigenschaften

Dispersion of carbon nanotubes in poly(ether ether ketone) and its effects on rheological and electrical properties.

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1 INTRODUCTION

Carbon nanotubes (CNTs) having diameters in the range of 1-50 nm and lengths of several microns offer a great potential for fabricating composites with substantially enhanced properties at low nanotube loading, including conductivity, strength, elasticity, toughness and durability [1-3]. The combination of these properties with very low densities suggests that CNTs are an ideal candidate for high-performance polymer composites. However, the effective utilization of carbon nanotubes in composite applications strongly depends on the ability to disperse the nanotubes homogeneously throughout the matrix and to achieve good interfacial bonding for load transfer across the CNT-matrix interface.

Many researchers have incorporated CNTs into a variety of polymer matrices by applying several methods including solution-processing [4,5], in-situ polymerization of CNT/polymer monomer mixes [6], and melt-mixing of CNTs with polymers [7,8]. Although solution-blending can result in a comparatively fine dispersion of nanotubes, melt-mixing is a more practical and industrially-relevant process as it allows the manufacture of either semi-finished (extrusion) or finished (injection-molding) parts independent of volume and complexity. However, the achievement of a fully dispersed state of the nanotubes in a given polymer by melt-mixing poses as yet unresolved challenges. Depending on the particular type of starting nanotube material, the processing equipment as well as the polymer matrix, such melt-mixing operations often lead to remaining nanotubes agglomerates. In order to further break down these agglomerates, very high shear stresses and prolonged mixing times are required to overcome both the van der Waals interactions of individual nanotubes and the growth-related mechanical entanglements. However, it is important to note that such high shear force can also damage the nanotubes (reduction of aspect ratio) [9], an effect detrimental for both mechanical reinforcement as well as the resulting electrical conductivity of the composites.

When dispersing nanoparticles in a polymer matrix, particle-particle and particle-matrix interaction forces play an important role in the dispersion process. It is not only the absolute filler size but rather the specific surface area and the resulting interfacial area which significantly influence the filler matrix interactions during processing. In turn, one would expect these interactions to induce variations in the microstructure of the polymer matrix which, in the case
of semi-crystalline matrices for example, can have significant implications on the resulting mechanical behavior of the composites in themselves. For these applications, understanding the rheology and microstructure is of key importance, as this would allow for processing nanofillers into macroscopically usable form with controlled properties.

Although the high aspect ratio of carbon nanotubes generally appears to be a clear benefit for the exploitation of their mechanical as well as physical properties, it is as yet not established which of the many different types of nanotubes will yield the ultimate performance in a polymer composite and only few work [8-10] has been undertaken to apply rheological and electrical data to characterize the state of dispersion directly. Therefore, the aim of the present study is to evaluate the dispersion of multi-wall and single wall nanotubes in a semi-crystalline polymer matrix by electron microscopy and to correlate the observed structural features to the rheological as well as electrical behavior of such nanocomposites.

2 EXPERIMENTAL PROCEDURE

2.1 Materials and preparation

A high-temperature, semi-crystalline poly(ether ether ketone) (PEEK), Victrex PEEK 151 grade, (molecular weight 27,000 g/mol) was used as a matrix system. Two types of multi-wall nanotubes (MWNTs) were used in this study. Nanocyl®-7000, with an average outer diameter of 9.5 nm (10 graphitic shells), length of up to 1.5 μm and density of 1.66 g/cm³, as stated by the manufacturer, were obtained from Nanocyl Co., Belgium. According to the supplier, these nanotubes are produced via the catalytic carbon vapor deposition (CCVD) process with a carbon purity of 90% and a surface area of 250-300 m²/g. A master batch containing 5 wt% carbon MWNT in PEEK (Hyperion MB9015) was obtained from Hyperion Catalysis. These nanotubes have a typical diameter range from 10 to 15 nm with 8-15 graphitic layers, lengths between 1 and 10 μm and surface area of 250 m²/g [8]. Purified single-wall carbon nanotubes (SWNTs), Elicarb P925 with an average diameter of 1.4 nm and length up to 10 μm was obtained from Thomas Swan & Co., UK.

Poly(ether ether ketone)/carbon nanotubes composites were prepared by melt compounding using a twin screw extruder. PEEK containing 2 and 5 wt% of nanocyl MWNTs and 2 wt% SWNTs were prepared using a MAPRE co-rotating twin screw extruder equipped with a gravimetric metering device from Colortronic. The screw has a diameter of 30 mm with L/D = 33. Compounding was carried out at a screw speed of 200 rpm with the maximum barrel temperature set at 360°C and a flow rate of 10 kg/h. The blend compositions investigated in this study is listed in Table 1.
Bangarusampath et al. Rheology and electrical properties of PEEK/CNT

<table>
<thead>
<tr>
<th>Carbon nanotubes (CNTs)</th>
<th>Supplier</th>
<th>Nanotube content (wt%)</th>
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<tr>
<td>Multi-walled carbon nanotubes (MWNTs)</td>
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<td>Single-wall carbon nanotubes (SWNTs)</td>
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Table 1: Compositional range of investigated PEEK nanocomposites

2.2 Shear rheological measurements

The rheological behavior both under shear and elongational flow were investigated, in order to probe the role of nanotubes on viscoelasticity of PEEK matrix. Shear rheological properties of nanotube filled PEEK were performed over a large range of shear rate ($10^{-2}$ to $10^{4}$ s$^{-1}$) using a rotational rheometer as well as a capillary rheometer operating at 360 °C. An Advanced Rheometric Expansion System (ARES) with a 25 mm parallel plate geometry at a fixed gap of 1 mm was used to measure the shear viscosities at low to middle shear rates (up to 500 s$^{-1}$). All measurements were performed in linear viscoelastic regime and under nitrogen atmosphere. A Göttfert Rheograph 6000 high-pressure capillary rheometer with die diameter of 1 mm (L/D= 30) was used to measure the shear viscosity at very high shear rates (up to $10^{4}$ s$^{-1}$), typically occurring at processing techniques such as extrusion and injection-molding.

2.3 Uni-axial elongational measurements

Uniaxial melt elongational measurements were carried out using a Göttfert Rheotens apparatus. The Rheotens unit consists of a pair of counter-rotating copper wheels with a diameter of 40 mm which are mounted on a balanced beam. A high-pressure capillary rheometer, fixed with a die of 2 mm diameter, provides a polymer melt strand at a constant volume flow rate at 360°C. The melt strand was fed between the two draw-down wheels, placed 96 mm below the die, and the speed of the wheel was then accelerated linearly at 24 mm/s$^2$. The maximum force required to rupture the strand is commonly defined as the ‘melt strength’, and the maximum draw-down velocity is defined as the ‘drawability’. Each measurement was repeated five times, and the data were fitted using a rheological model proposed by Wagner and a Levenberg-Marquardt procedure [11].
2.4 Electrical conductivity

The AC conductivity measurements were performed in the frequency range from $10^{-1}$ to $10^6$ Hz using a Novocontrol Alpha high-resolution impedance analyzer at room temperature (23°C). Rectangular bars (10 x 4 x 1.3 mm$^3$) were cut from the compression-moulded specimens. The cross-sectional areas were coated with conductive silver in order to provide good contact to the specimen. The specific conductivity of the nanocomposites as a function of frequency, $\sigma(\omega)$, is calculated from the modulus of the complex admittance, $|Y^*|$, [12]:

$$\sigma(\omega) = |Y^*(\omega)| [\Omega^{-1}]$$  \hspace{1cm} (1)

where $A$ is the cross-sectional area and $t$ is the thickness of the composite.

2.5 Morphological characterization

The state of nanotube dispersion within the PEEK matrix was evaluated using field-emission scanning electron microscopy (FE-SEM) and transmission electron microscopy (TEM). SEM micrographs of cryo-fractured, extruded pellets were taken using a Zeiss 1530 microscope. Ultrathin sections of the nanocomposites (thickness of approximately 60-100 nm) were cut using a Leica Ultracut E microtome equipped with a diamond knife. TEM was carried out using using a Zeiss CEM 902 apparatus at an acceleration voltage of 80 kV.

3 RESULTS AND DISCUSSION

3.1 Nanotube dispersion in PEEK matrix

The homogeneous dispersion of nanotubes in the polymer matrix is one of the most important requisites to observe any improvement in properties because the inhomogeneities lead eventually to structural defects in the composite materials. Fig. 1 shows SEM micrographs of PEEK nanocomposites filled with different types of nanotubes. The bright dots and lines in the SEM images correspond to the nanotubes. Due to the fibrillation of the polymer and complexity of the nanotube network, it is not possible to get a detail in-sight on the state of dispersion of MWNTs. TEM micrographs (Fig. 2) provide a clearer view that the MWNT-Ncyl appear to be homogenously dispersed in the PEEK matrix without any large aggregation (Fig. 2a), while some aggregates are observed for MWNT-Hyp/PEEK composites (Fig. 2b). In contrast, the SWNTs show a very poor dispersion in the PEEK matrix (Fig. 2c). It is known from literature [13] that SWNTs are produced as a rope and are difficult to disperse, due to very high inter-particle van der Waals forces.
Figure 1: SEM micrographs of carbon nanotube filled PEEK composites
(a) 5 wt% MWNTs – Ncyl (b) 5 wt% MWNTs – Hyp (c) 2 wt% SWNTs
Figure 2: TEM micrographs of carbon nanotube filled PEEK composites
(a) 5 wt% MWNTs – Ncyl (b) 5 wt% MWNTs – Hyp (c) 2 wt% SWNTs
Most MWNTs-Ncyl were separated into individual tubes by the shear force during melt mixing and thus evenly dispersed in the PEEK matrix which was a great practical importance for making nanotube reinforced polymer composites. The high shear mixing can also result in some breakage of the nanotubes. For example, Kharchenko et al. [9] reported that the aspect ratio of MWNTs in a polypropylene matrix was reduced from 1000 to 300-400 after melt processing. Therefore, good dispersion can be achieved only at the expense of an acceptable reduction in tube length.

As can been seen in Fig.3, the individual nanotubes are recognized as long hollow fibers and they are randomly dispersed in the PEEK matrix without preferred alignment. It can also be seen that the nanotubes are very flexible (diameter between 10-15 nm) and often characterized by a certain degree of waviness along their axial dimension, which in-turn reduce the structural reinforcement of the nanotubes. Additionally, a smooth interface between the nanotube and the PEEK matrix can also be observed.

Electron microscopy is the most common method to visualize the dispersion of nanotubes in a polymer matrix, but only provides information of very distinct regions of the specimen which may or may not be representative for the overall composite. Moreover, sample preparation for TEM analysis is time-consuming and expensive for routine characterization of the nanocomposites. In order to overcome this drawback, rheological characterization has developed as an alternative quantitative method for monitoring the dispersion [14].

Figure 3: HR-TEM micrographs of carbon nanotube filled PEEK composites.  
Left: Dispersion of Multiwall carbon nanotube (Nanocyl) in PEEK  
Right: Individual carbon nanotube showing their flexibility
3.2 Shear flow behavior of PEEK/CNT nanocomposite

Fig. 4 shows the double logarithmic plots of the complex viscosity, $|\eta^*|$, vs. Frequency, $\omega$ and shear viscosity, $\eta$ versus shear rate, $\dot{\gamma}$, for pure PEEK and their nanocomposites. The rheological properties were obtained using the parallel plate and capillary rheometer at 360°C, respectively. Evaluation of these flow curves reveals a significant difference between the different nano–composite materials, particularly at low frequencies. For pure PEEK, two distinct regions are present: (1) a Newtonian region at low frequency and (2) shear thinning behavior at high frequency. The addition of only 2 wt% of MWNT-Ncyl shows an increase in viscosity of almost four orders of magnitude, but also enhanced shear thinning characteristics. This dramatic increase is attributed to the high aspect ratio of MWNT-Ncyl and indicates a good dispersion and strong PEEK-nanotube interaction. As expected, the viscosity increases with the content of MWNT-Ncyl. Addition of 5 wt% MWNT-Hyp and 2 wt% SWNTs shows lower viscosity increase especially at low frequency. When subjected to shear flow, the presence of carbon nanotube aggregates in MWNT-Hyp (Fig.2b) and SWNT (Fig.2c) composites is expected to offer lower flow resistance (low viscosity) as compared to the well-dispersed MWNT-Ncyl (Fig. 2a) with similar nanotube concentration [15]. The well-separated nanotube could effectively restrict the polymer mobility and had higher probability of nanotube entanglements, which in turn might offer higher flow resistance.

![Figure 4: Complex and steady shear viscosities of pure PEEK and its nanocomposites containing various weight contents of nanotubes](image)

Earlier investigations on the rheological properties of polymer melts have shown that the complex viscosity, $\eta^*$, and the steady shear viscosity, $\eta$, are closely super-imposable for numerically equivalent values of frequency and shear rate (Cox-Merz rule) [16].

$$|\eta^*(\omega)| = \sqrt{\eta'(\omega)^2 + \eta''(\omega)^2} = \eta(\dot{\gamma})$$ \hspace{1cm} \text{for} \hspace{1cm} \dot{\gamma} = \omega \hspace{1cm} (2)$$

As can be seen in Fig. 4, only the homogeneous PEEK melt obeys the rule, whereas the heterogeneous nanotube filled composites fail. The observed lower steady viscosity than the complex viscosity might be due to the break down of the nanotube network and interaction of nanotubes [17]. As a result, one is unable to elucidate the processing behavior of the nanocomposites directly from the dynamic tests.

It is known from literature [8-10] that the modulus is very sensitive to microstructure formation. Fig. 5 shows the magnitude of both storage and loss moduli increases monotonically with increasing nanofibre content. At higher frequencies, the rheological response of the PEEK composites is dominated by the polymer matrix, whereas the presence of nanotubes influences the low frequency regime and so, the increase is more dramatic especially at the low frequencies. This increase is more pronounced in $G'$ as compared to $G''$.

All nanocomposites tend to form a plateau at low frequencies indicating a different elastic behavior, which is often referred to as solid-like behavior. Such behavior is mainly attributed to the tube-tube interactions and to a network formation, as the interparticle distance decreases [8-10]. In accordance to the shear-rheological results, the well-dispersed MWNT-Ncyl shows nearly four orders of magnitude increase in $G'$, whereas the moderately-dispersed MWNT-Hyp as well as the poorly-dispersed SWNT reveal lower values of $G'$. 
Fig. 5: (a) Storage modulus and (b) Loss modulus of pure PEEK and its nanocomposites containing various weight content of nanotubes.
3.3 Electrical properties of PEEK nanocomposites

Due to relatively high electric conductivity, CNT can be exploited as a filler for improving the conductivity of composite materials. Electrical conductivity occurs when the nanotubes form an infinite conductive pathways at a critical concentration; also dependent on the dispersion and aspect ratio of the nanofiller [18]. The frequency dependence electrical conductivity for PEEK/CNT composites with different weight contents of MWNTs and SWNTs at room temperature (23 °C) is depicted in Fig. 6a. As expected for insulating materials, the bulk conductivity of pure PEEK has a value of about $10^{-13} \text{Sm}^{-1}$ at 0.1 Hz. An abrupt increase in conductivity of nearly ten orders of magnitude is observed by adding only 2 wt% MWNT-Ncyl, and the conductivity furthermore remains independent of frequency over a wide range. An increase in MWNT loading above 2 wt% yields a more gradual increase in electrical conductivity, with values of about 0.01 S m⁻¹ at 5 wt%. Fig. 6b shows the electrical conductivity of composites at a fixed frequency of 0.1 Hz. It is apparent that by loading less than 2 wt% MWNT-Ncyl, the conductivity of the composite already satisfies the antistatic criterion ($10^{-8} \text{Sm}^{-1}$).

Du et al [10] and few other researchers related the rheological percolation threshold to the electrically measured percolation threshold. They reported that a denser nanotube network is required for electrical threshold, while a less dense nanotube network sufficiently restricts the polymer chain mobility related to the rheological percolation threshold. Interestingly, the percolation threshold value of 2 wt% (MWNT-Ncyl) observed in this study is one of the lowest values reported in literature for melt processed thermoplastics. In our on-going studies, we prepared several small steps of lower MWNT-Ncyl concentration in PEEK to exactly determine the percolation value which we expect to be lower than 2 wt% nanotube.

As expected, the inhomogenously dispersed MWNT-Hyp and poorly dispersed SWNT show moderate and poor electrical conductivity, respectively. In summary, only the effective dispersion of nanotubes is able to provide a conductive path throughout the specimen even at very low concentrations, without deteriorating the other physical properties of the matrix.
Fig. 6: Conductivity of PEEK nanocomposite (a) as a function of frequency (b) as a function of nanotube content (at $f = 0.1$ Hz).
3.4 Melt elongational properties of PEEK nanocomposites

Many polymer processing routes such as film blowing, fibre spinning and foaming [19,20] involve not only shear but also significant elongational flows, where the process involves large and rapid stretching deformation. The results of the uniaxial elongational experiments, using the rheotens method at 360 °C, are shown in Fig. 7, as a plot of draw-down force, F, vs. draw-down ratio, V (draw-down velocity of the wheel / die exit velocity of the melt, \( \nu_r / \nu_o \)). As can be seen, the addition of nanotubes substantially increases the melt strength, however the drawability decreases significantly with increasing MWNT-Ncyl content. The increase in melt strength can be attributed to positive interactions between polymer chains and the filler and to partial load-bearing by the dispersed nanotubes in the PEEK matrix. In contrast, the inhomogeneously dispersed MWNT-Hyp and SWNTs show only moderate increases in melt strength, while the drawability remains unchanged.

![Experimental average Rheotens curves for pure PEEK and its nanocomposites at 360°C fitted using Lavenberg-Maquardt procedure [11].](image)

A direct conversion of the measured Rheotens data (draw-down force vs. draw-down velocity or ratio) into an elongational viscosity is complex, as a non-uniform strain rate is applied along the melt strand. Nevertheless, by following a detailed analysis of the complex deformation of the polymer melt strand during the rheotens
test, an analytical model was suggested by Wagner et al. [11]. Following this approach, the measured draw-down force curve as a function of the draw-down ratio was fitted using the Levenberg-Marquardt procedure and from the obtained fitting parameters, the elongational viscosity can be calculated.

Fig. 8: Apparent elongational viscosity of PEEK nanocomposites as function of the elongational rate calculated using the Wagner analytical model.

Fig. 8 shows the elongational viscosity as a function of the elongational rate, obtained using an analytical Wagner model. The plot shows that the pure PEEK exhibits a little strain hardening behavior up to 0.6 s⁻¹, where the elongational viscosity reaches a maximum and subsequently continuously decreases at higher strain rates, according to a viscous power-law model. Addition of nanotubes increases the elongational viscosity significantly at low elongational rate; however, elongational thinning at higher shear rates becomes more pronounced. A well-dispersed MWNT-Ncyl/PEEK composite shows the maximum increment in elongational viscosity as compared to MWNT-Hyp and SWNT composites. It should also be noted that addition of 5 wt% MWNTs suppresses the strain hardening and shifts the maximum to the lower elongational rate. Thus the addition of the nanofillers offer a unique way of modifying the elongational behavior of polymer melts which is important for processes involving elongational flows. For example, during foaming, higher elongational viscosity/melt strength is required to prevent bubble coalescence. These beneficial effects are especially significant in the case of semicrystalline
melts as the viscosity cannot always be increased by decreasing the temperature due to the onset of crystallisation of the matrix.

4 CONCLUSIONS

High-temperature poly(ether ether ketone) nanocomposites with different types of MWNTs and SWNTs were prepared by melt-compounding. The state of dispersion and the resulting microstructure were critically assessed through electron microscopy and by rheological as well by electrical measurements. MWNTs were well-dispersed in PEEK matrix, whereas SWNTs show poor dispersion. Nanocomposites with the homogeneous dispersion of nanotubes exhibit a higher melt elasticity, conductivity and melt strength, whereas a poor nanotube dispersion results only in minor improvements. Linear viscoelastic and conductivity measurements reveal that a threshold of 2 wt% of MWNTs is required to form a nanotube network within the PEEK matrix. These experimental results highlight the fact that the use of proper processing techniques to disperse the nanofillers in the polymer matrix and of appropriate carbon nanostructures can thus beneficially enhance both the processing behavior as well as enhance the final properties of the polymer composite.

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