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CARBON NANOTUBES FILLED POLYETHERETHERKETONE (PEEK) COMPOSITES

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Abstract

The addition of carbon-based nanofillers to polymer matrix has widely used during the last years to increase the mechanical, thermal and electrical properties of the polymers. One of the biggest challenges in the use of nanocomposites has been to scale up the fabrication from laboratory to industrial scale. In this work, PEEK/CNT nanocomposites have been fabricated by semi-industrial melt-compounding and injection-moulding techniques. The addition of CNT increased the mechanical properties of the nanocomposite. Well dispersion of CNT was observed by SEM, indicating that industrial techniques can be used to obtain these materials.

1. Introduction

In the last years, carbon-based nanofillers such as graphene or carbon nanotubes (CNT), have been widely studied as part of a novel generation of nanocomposite materials (1–3). CNT show an excellent mechanical, thermal, and electrical properties, which make CNT very interesting to use as nanofiller in polymer nanocomposites (2). At the same time, aerospace and electronic industries have motivated the development of high-performance polymers, due to the need to find materials that can undergo extreme thermal conditions with high mechanical properties, high thermal and chemical stability, and low density (4,5). Nowadays to increase the properties of high-performance polymers is even more important for these industries. A possible solution could be to add nanofillers to high-performance polymers in order to improve the mechanical properties or to add other properties such as thermal and electrical conductivity (6).

In this way, different loadings of CNT (0.5, 1, 5, and 10 wt.%) have been added within polyetheretherketone (PEEK) matrix, which is a high-performance thermoplastic polymer to obtain a nanocomposite material with new and improved properties.

2. Materials and Methods

2.1. Materials

The PEEK matrix (PEEK-150G) was provided in pellets by Victrex. This grade of PEEK presents the following physical characteristics: Glass transition temperature (T_g) = 143 °C, melting temperature (T_m) = 343 °C, density ($d_{25°C}$) = 1.3 g/cm3, viscosity ($\eta_{400°C}$) = 90 Pa·s. Multiwall carbon nanotubes (CNT, NC7000) were purchased from Nanocyl.

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2.2. Extrusion-compounding and injection-moulding

Melt-blending of PEEK nanocomposites was performed in an semi-industrial extrusion-compounding machine (Coperion ZSK 26) equipped with a 26 mm diameter co-rotating twin-screw and with two Brabender gravimetric feeders. The temperature profile of the extruder was set at 330 °C at the feeder increasing to 360 °C at the nozzle. A specifically designed, high shear rate screw profile was used to ensure proper dispersion. A rotor speed of 250 rpm was used to process all the nanocomposites. The molten material was extruded through a 2 mm diameter die at a constant output rate to give the different compositions. The extrudate strand was quenched immediately in a water bath at room temperature, dried and cut into small pellets. Pure PEEK material was processed under the same conditions as a reference material.

Dog-bone shaped specimens (type 1A) for tensile tests, under ASTM D638-02a were injected in a mould made of tool steel. The pellets were dried at 150°C for 3 hours prior to processing. The specimens were produced by injection moulding through a JSW 85 EL II injection machine with a 35 mm diameter reciprocating screw at a screw speed of 120 rpm. Neat PEEK and specimens that contained different loadings of CNT were prepared by following this protocol. At least 15 specimens were produced for each composition.

2.3. Differential scanning calorimetry

Differential Scanning Calorimetry (DSC; Q200, TA instruments) was used to obtain information about the thermal properties of the nanocomposites. The samples (5–10 mg) of PEEK with different percentages of CNT (0.5, 1, 5, and 10 wt.%) were heated from 20 to 400°C at 10°C/min, and held at 400°C for 5 min to remove the thermal history of the samples. Then the samples were cooled to 20°C at 10°C/min, held for 0.5 min and heated again to 400°C at 10°C/min. The DSC samples were cut from the bulk of the injection specimen (dog-bone) by impacting them with a hammer and using a knife. One test per sample was performed.

2.4. Scanning electron microscopy

Dispersion of CNT within the PEEK matrix was analysed by SEM (Helios NanoLab 600i, 2 KeV and 0.17 mA). The specimens were cooled in liquid nitrogen and immediately broken by using a razor blade and a hammer. A thin layer of gold (3 nm) was sputter-coated onto the surfaces.

2.5. Tensile tests

Tensile tests were carried out under ambient conditions with an Instron 3384 by using a load cell of 10 kN by using type V specimens at 1 mm/min of crosshead speed (ASTM D638-02a). The axial and transverse displacement (ε_1 and ε_2) of each specimen during the test was performed by digital image correlation (DIC). One side of every ´dog bone´ specimen was painted white and then carefully and lightly sprayed with black paint to get the random speckle pattern required for DIC analysis. The images (36 x 4.5 mm^2) were taken every 2 s during the test (3,7). Finally, the images were evaluated by using Vic-2D 2009 DIC software (VicSNAP, Correlation Solutions Inc., Columbia, SC, USA).

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3. Results

3.1. Dispersion of CNT within PEEK matrix

The cryogenically fracture of the injection samples was analysed by SEM. The fractured images showed that CNT were dispersed homogeneously within PEEK matrix. No big agglomerates of CNT could be found. A fractured surface of neat PEEK and PEEK with 5 wt.% of CNT are showed in Figure 1 a) and b), respectively. The addition of higher percentages of CNT increased the number of CNT agglomerates observed in the samples, as expected.

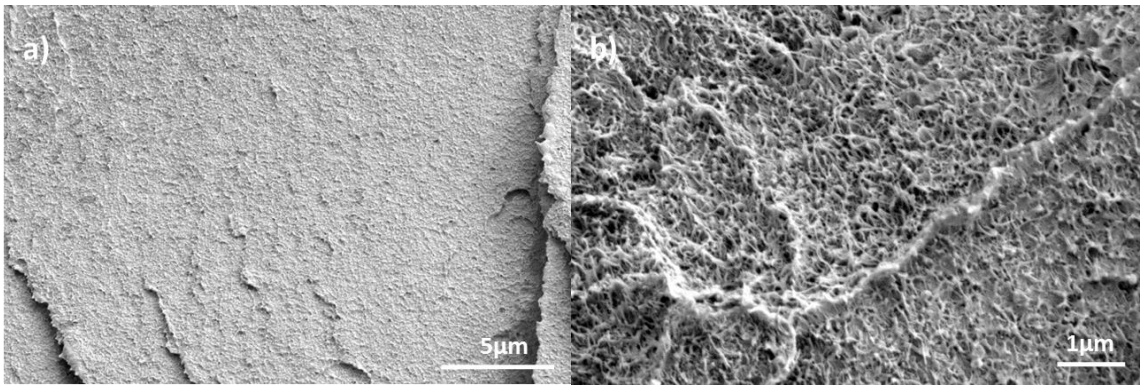

Figure 1. SEM images a) PEEK and b) PEEK with 5wt.% of CNT

3.2. DSC measurements

The thermal behaviour of the nanocomposites was analysed by DSC. The melting temperature did not varied with the addition of CNT and was around 340 ºC. On the other hand, an increase in the crystallisation temperature of the nanocomposites was observed. The increase was higher adding higher loadings of CNT. This behaviour indicated that CNT had a nucleation effect in the crystallisation of PEEK. This behaviour was reported previously with the addition of CNT and other carbon based nanofillers as graphene nanoplatelets (8).

3.3. Mechanical properties

The mechanical properties of the injection samples were analyzed through the tensile tests. The addition of CNT increased the tensile modulus and strength of PEEK. The higher enhancement was found adding 10 wt.% of CNT, reaching 13% and 48% of improvement in tensile strength and tensile modulus, respectively. The results present in this work were higher than previously reported in PEEK/CNT nanocomposite samples made through injection-moulding machine (9,10).

On the other hand, the addition of CNT decreased slightly the strain at break of the samples. This effect could be observed especially in the samples with 5 and 10 wt.% of CNT.

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