MECHANICAL AND PHYSICAL PROPERTIES OF POLYETHERIMIDE/CNF COMPOSITES FABRICATED BY DIFFERENT METHODS

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Introduction

Polymer nanocomposites with carbon nanofillers have attracted great interests due to their superior mechanical and physical properties. One particular kind of nanofiller that offers excellent performance and low cost commercial availability is carbon nanofibers (CNFs). With only small loading of CNFs, improvements in mechanical and physical properties such as electrical, dielectric and thermal properties of polymers have been demonstrated [1].

Polyetherimide (PEI) is a derivative of polyimide by introducing flexible phenylene ether groups to polyimide chains. PEI has exceptional thermal stability and mechanical properties, comparable to those of polyimide [2], but with superior solubility and processibility. Thus, PEI is a desirable candidate for application in microelectronic and aerospace industries. Further enhancements in the mechanical and physical properties of PEI through the incorporation of nanofibers would not only benefit current applications, but would broaden the relevance to many industries.

In our study, both as-received and ultrasonically treated CNFs were used to make PEI nanocomposites. The nanocomposites were prepared by two different fabrication methods. Their mechanical properties and physical properties were investigated.

Experiments

Materials

PEI (ULTEM 1000) was supplied by Sabic Innovative Plastic Inc. Carbon nanofibers (Pyrograf® III, PR-24-HHT) were purchased from Applied Science Inc. Some of the as-received CNFs were treated by high power ultrasonicator (Branson W385) for 1 hour to break big agglomerates and shorten long CNFs.

Preparation of nanocomposites samples

The 1-step method involved only a direct dry mixing of as-received or treated CNFs to PEI on a twin screw extruder (Leistritz) at a temperature of 370 ºC.

The 2-step method started with the pre-dispersion of both as-received and treated CNFs in PEI solution in the presence of dichloromethane as a solvent by low power ultrasonication (Branson 1510). The solution was then poured into an aluminum frame and set into a panel form. Once dried, the panel was cut into small pieces and then dry mixed with PEI under the same processing conditions as the 1-step method.

The extrudates for each nanocmposite were compression molded using hot-press (Carver, Inc.) to ca. 3 mm thick nanocomposite panels at 216 ºC. The 50 mm × 10 mm × 3 mm testing samples were cut from these panels to be used for mechanical tests.

The film samples for electrical measurement were prepared by a cast coating method, and the solution used for film coating was obtained by dissolving the extrudates in Dichloromethane.

Mechanical Tests

3-point flexural testing was conducted on an Instron 4466 2 kip electromechanical universal test machine with a loading rate of 2.032 mm/min (0.08 in/min). The dynamic mechanical analysis was done on a Tritec 2000 DMA test machine in 3-point bending mode. The heating rate was 5 °C/min ranging from 30 °C to 300 °C.

Electric and dielectric tests

The electrical conductivity was measured on a Keithley 6517A with 8009 test chamber at a DC voltage of 50V at room temperature.

Results and Discussion

Table 1 Flexural properties of CNF reinforced PEI nanocomposites

Concentration	Flexural Modulus (GPa)	Flexural stress (MPa)	Flexural strain %
Pure PEI	3.81 ± 0.70	161.2 ± 15.8	5.36 ± 0. 8
0.5% CNF (as-received),1-step	3.68 ± 0.31	88.7 ± 17.1	3.29 ± 0. 1
1.0% CNF (as-received),1-step	4.01 ± 0.26	> 170.6 ± 45.1	> 14.6 ± 0. 4
3.0% CNF (as-received), 1-step	4.15 ± 0.11	144.0 ± 16.8	4.10 ± 0.4
1.0% CNF (1hr-treated),1-step	4.03 ± 0.29	170.6 ± 45.1	4.62 ± 0.8
1.0% CNF (as-received), 2-step	4.17 ± 0.16	160.5 ± 27.6	4.60 ± 0.7
1.0% CNF (1hr-treated), 2-step	4.22 ± 0.15	125.3 ± 15.3	3.63 ± 0.4

In the PEI/CNF nanocomposites prepared by the 1-step method, the PEI/1.0wt% CNF (as-received) nanocomposite exhibited greatly improved flexural properties. The bending samples did not break even when the flexural testing was at ultimate displacement. This may indicate that at certain loading level (1.0 wt%), the interaction among long CNFs (Fig. 1) resulted in the formation of an effective CNFs framework throughout the sample and increased the loading capacity, which could restrain the negative effect of poor interfacial bonding.

The high power ultrasonication can effectively shorten the length of CNFs (Fig. 2), and prevent the formation of CNF framework, so that the flexural properties of PEI/1.0wt% CNF (1hr-treated) nanocomposites are inferior to those of PEI/1.0wt%CNF (as-received) nanocomposite.

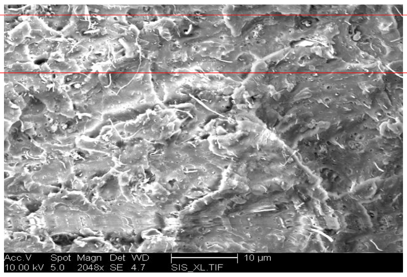

Fig. 1 SEM image of PEI/1.0 wt% CNF (as-received) nanocomposite by 1-step method

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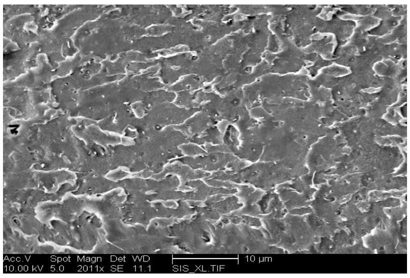


Fig. 2 SEM image of PEI/1.0 wt% CNF (1hr-treated) nanocomposite by 1-step method

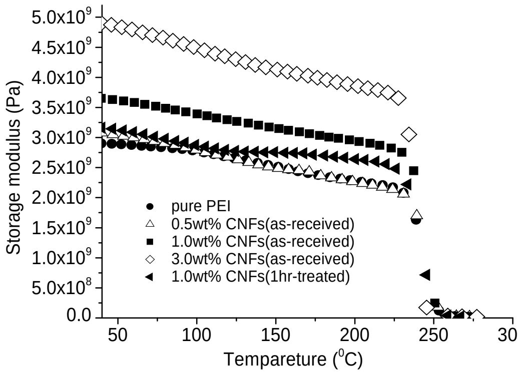
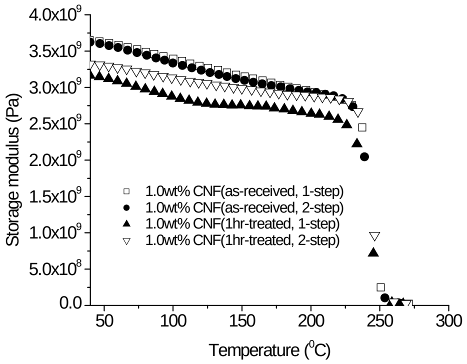


Fig. 3 Storage modulus of PEI/CNF nanocomposites prepared by 1-step method

According to Fig. 3, the increasing CNF loading in PEI/CNF nanocomposites can effectively improve the storage modulus. At 50^{0}C, with 3.0wt% CNF loading, there is a ca. 65.5% increase in storage modulus. However, the ultrasonically treated CNFs did not show obvious enhancement effect, indicating the effect of length of CNFs.

Compared with the 1-step method, the 2-step method exerts greater effect on the ultrasonically treated CNFs filled PEI nanocomposites (Fig. 4). The storage moduli of PEI/1.0wt% CNF (as-received) nanocomposites fabricated by two methods are almost equivalent. But for the PEI/1.0wt% CNF (1hr-treated) nanocomposites, 2-step method can endow the nanocomposites with higher storage modulus, which indicates that the 2-step processing method provided compensation in the loss of storage modulus caused by shortened CNFs.



The processing method also has great effect on electrical conductivity. The nanocomposites fabricated by the 2-step method shows much higher electrical conductivity (Table 2).

Table 2 Electrical conductivity (S/cm) of PEI/1.0 wt% CNF nanocomposites by different fabrication methods

Fabrication methods	As-received CNFs	1hr-treated CNFs
1-step	7.29×10^{-16}	2.15×10^{-16}
2-step	6.67×10^{-9}	5.05×10^{-8}

Conclusions

The PEI/1.0wt% CNF (as-received) nanocomposite fabricated by 1-step method showed the best flexural properties, presumably because of the formation of an effective CNF framework. The results implied that the different fabrication methods can result in different mechanical and electrical properties of PEI nanocomposites by influencing the dispersion and distribution of CNFs in PEI matrix.

Reference

1. G. G. Tibbetts, M. L. Lake, K. L. Strong, B. P. Rice, A review of the fabrication and properties of vapor-grown carbon nanofiber-polymer composites, *Compos. Sci. Tech*, 67 (2007) 1709-1718.

2. B. K. Chen, Y. T. Fang, J. R. Cheng, Synthesis of low dielectric constant polyetherimide films, *Macromol. Symp.* 242 (2006) 34-39.

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