Composites Part B 88 (2016) 253–263

Contents lists available at ScienceDirect

Composites Part B

journal homepage: www.elsevier.com/locate/compositesb

Thermal aging and reinforcement type effects on the tribological, thermal, thermomechanical, physical and morphological properties of poly(ether ether ketone) composites

Nevin Gamze Karsli [a], Sadi Demirkol [b], Taner Yilmaz [a, b, *]

[a] Department of Mechanical Engineering, Kocaeli University, 41380 Kocaeli, Turkey
[b] Department of Polymer Science and Technology, Kocaeli University, 41380 Kocaeli, Turkey

A R T I C L E I N F O

Article history:
Received 8 August 2015
Received in revised form
26 October 2015
Accepted 18 November 2015
Available online 2 December 2015

Keywords:
A. Carbon fiber
A. Glass fibers
B. Wear
B. Fiber/matrix bond

A B S T R A C T

In this study, thermal aging process was applied to glass fiber (GF) and carbon fiber (CF) reinforced poly(ether ether ketone) PEEK composites and effects of this process and reinforcement type on thermal, thermomechanical, tribological, physical and morphological properties of PEEK composites were evaluated. All the results demonstrated that a transcrystalline layer was formed between the fiber and matrix in the composites under applied thermal aging condition and this layer affected the fiber–matrix adhesion, reduced the energy dissipation in this region and improved the properties of composites. As a result of this study, it was proved that the thermal aging process is an efficient method to control the thermal, thermomechanical, tribological and physical properties of composites and these properties of composites can be improved by using this method.

1. Introduction

Poly(ether ether ketone) (PEEK) is one of the semi crystalline high performance thermoplastic polymers [1–3]. Relatively high thermal resistance (glass transition temperature 143 °C, melting temperature 343 °C, service temperature 250 °C and decomposition temperature 580 °C), chemical resistance and exceptional mechanical performance have brought the PEEK to the center of the scientific attention for many years and also these properties have allowed for PEEK's use in high performance applications such as automotive, aviation, biomechanics etc [1,4–6]. Due to the these applications require thermal durability in high temperatures and PEEK is one of the few polymers (poly(phenylene sulphide) (PPS), polybenzimidazole (PBI), and polyimide (PI)) which can be compared with metals to provide thermal durability and relatively high performance in high temperatures [1,7]. In addition to this, PEEK is the most proper polymer in this group for tribological and mechanical applications because of its high strength and modulus, excellent toughness, and high wear resistance [7]. Therefore, fiber addition to PEEK is a commonly used method to improve the tribological and mechanical properties of it. For this purpose, glass fiber and carbon fiber are widely used fiber types and in the literature there are so many studies which investigated the various properties (especially mechanical and tribological) of these fibers reinforced PEEK matrix composites [1–22].

Fiber addition to polymeric matrix leads to changes in the fiber–matrix interphase region and these changes are related to the microstructural properties of the composite in this region such as morphology and crystallinity. These fibers act as heterogeneous nucleating agents and nucleate crystallization along the fiber–matrix interface. These nuclei hinder the lateral extension of crystals and force them to grow vertically to the fiber surfaces and result in a crystalline layer and this layer is named as transcrystalline layer (TCL) [23]. The high nucleation density along the fiber resulted in the growth of a thick and uniform TCL on the fiber surface. Formation of this layer has significant influence on the quality of fiber–matrix interface with some other factors (fiber type and topology, physical and chemical properties of fiber surface, the matrix type, thermal history), and correspondingly it affects the mechanical properties of composites. In the literature, while some studies suggest that TCL can improve shear transfer at the interface, and, consequently, the mechanical properties of composites, some studies claims that it has no, or even a negative, effect on these properties [23–26].

* Corresponding author. Current address: Department of Mechanical Engineering, Kocaeli University, 41380 Kocaeli, Turkey. Tel.: +90 0262 303 34 33.
E-mail address: taner.yilmaz@kocaeli.edu.tr (T. Yilmaz).

http://dx.doi.org/10.1016/j.compositesb.2015.11.013
1359-8368/

Based upon these information it can be said that it is possible to control the crystallinity of PEEK composites by choosing the suitable thermal history. Due to the PEEK can crystallize between its glass transition temperature and melting temperature, any thermal treatment between these temperatures can cause crystal morphology changes and correspondingly effect the mechanical and tribological properties [27]. Hence, by using the suitable thermal treatment, effects of TCL on the mechanical, tribological, thermal etc. properties of composites can be evaluated [28].

In the literature there are many studies which investigated and compared the various properties of carbon and glass fiber reinforced PEEK matrix composites. One of these studies was performed by Patel et al. [1]. In their study, the flammability and fire behavior of PEEK and its composites were evaluated by using different techniques. Another study was performed by Sarasua et al. [2]. They investigated the effect of crystallinity differences induced by mold wall temperature and annealing on mechanical behavior of PEEK resin and its glass fiber and carbon fiber reinforced composites. Harsha and Tewari [3] evaluated the tribo performance of PEEK reinforced with short glass, carbon fiber, and solid lubricants in abrasive wear situation.

On the other hand, there are a lot of studies in the literature which investigate the properties of transcrystalline layer and influencing factors of this transcrystalline layer in the fiber reinforced composites [23–26]. Klein et al. [24] studied the interfacial transcrystallinity in aramid fiber reinforced nylon 66 microcomposites by using dynamic mechanical thermal analysis. Wood and Marom [25] investigated the effect of the transcrystalline layer on the interfacial shear strength in carbon fiber–polycarbonate composites by using thermal fragmentation experiments. Nielsen and Pyrz [26] studied the influence of thermal history on the interfacial load transfer efficiency and fiber failure in carbon/polypropylene microcomposites. Their results indicated that thermal history has an important effect on the interfacial load transfer efficiency of the microcomposites. This result was correlated with the influence of thermal history on transcrystallinity and interfacial residual stresses. Because transcrystalline interphase provides a more effective load transfer compared to the non-transcrystalline interphase.

In addition to these studies, in the literature there are some studies about the effects of thermal history processes on the properties of fiber reinforced PEEK composites [27–32]. Buggy and Carew [27] investigated the morphological properties of carbon fiber-reinforced PEEK laminates which were subjected to long-term thermal aging and cycling treatments by using differential scanning calorimetry and wide-angle X-ray diffraction analyses. Sinmazcelik and Yilmaz [28] evaluated the effects of thermal aging on the properties of unfilled and random oriented short fiber reinforced PEEK and its composites.

All the literature survey and obtained results revealed that although there are a lot of studies about the PEEK, fiber reinforced PEEK composites and thermal history effects on the properties of fiber reinforced PEEK composites, there are not any studies which compare the effects of fiber types on the two types of wear (adhesive and abrasive) behavior of composites exposed to same thermal history conditions.

By considering these results, in this study, it is aimed to investigate the effects of microstructural changes, resulted from the thermal aging process, on tribological, thermal, thermomechanical, physical and morphological properties of fiber reinforced PEEK composites for two types of fiber (carbon fiber and glass fiber). Abrasive and adhesive wear tests were performed to evaluate tribological properties, differential scanning calorimetry (DSC), thermogravimetric and dynamic mechanical analyses were performed to investigate the thermal and thermomechanical properties, hardness test and scanning electron microscopy (SEM) analyses were performed to investigate physical and morphological properties of composites, relatively.

2. Experimental

2.1. Materials

Materials were supplied from Victrex with “VICTREX PEEK 450CA30” and “VICTREX PEEK 450GL30” trade names. General properties of these two different materials were given in Table 1.

2.2. Thermal aging process of materials

Carbon fiber and glass fiber reinforced PEEK composites were heated in an oven to 360 °C with 10 °C/min. heating rate and kept at this temperature for 10 min. Composites were removed from the oven after 10 min and were simultaneously immersed into the iced water [28]. This method is named as “quenching” and the composites which applied this method coded are with the letter “Q”. The flow diagram of quenching process was given in Fig. 1.

After the quenching process, half of the quenched composites were stored for characterization tests, the other half of the quenched composites were heated in an oven to 310 °C with 10 °C/min. heating rate and kept at this temperature for 60 min. After that time, composites were cooled to 25 °C with 10 °C/min. cooling rate [28]. This method is named as “thermal aging” and the composites which are applied this method coded with letter “A”. The flow diagram of thermal aging process was given in Fig. 2. In addition to these thermal processes, as received composites were used as reference sample and named as “unaged” during of the study. According to these processes, code names of composites were given in Table 2.

2.3. Characterization

2.3.1. Differential scanning calorimetry analysis

Differential scanning calorimeter (DSC) analyses were performed by using TA Instruments-Q200 model DSC machine in order to obtain glass transition temperature (T_g), melting temperature (T_m) and heat of fusion (ΔH_f) values of composites. Analyses were carried out in the temperature range from 25 °C to 440 °C at a heating rate of 10 °C/min. The relative degree of crystallinity ($X_c(\%)_{rel}$) values of the composites were calculated by using melting enthalpy values and following expression [33]:

$$X_c(\%)_{rel} = \frac{\Delta H_f}{\omega \times \Delta H_0} \times 100 \tag{1}$$

where ΔH_f is the heat of fusion of sample, ω is the weight fraction of PEEK matrix and ΔH_0 is the heat of fusion of the neat PEEK. Heat of fusion of 100% crystalline PEEK was used as 130 J/g [34].

2.3.2. Dynamic mechanical analysis

Dynamic mechanical analysis (DMA) was conducted by using a TA Instruments Q800 model dynamic mechanical analyzer in the three point bending mode. Storage modulus (E′), loss modulus (E″) and tan delta (tan δ) values of composites were determined under the amplitude of 35 μm, at a constant frequency of 1 Hz, in a temperature range of 25 °C–230 °C and at a heating rate of 5 °C/min.

2.3.3. Abrasive wear test

Abrasion wear test was performed by using Ektron EKT-2103 model abrasion testing machine. In this test, 0.9 rpm rotating test