Wear 297 (2013) 736–741

Contents lists available at SciVerse ScienceDirect

Wear

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

Tribological behaviors of hierarchical porous PEEK composites with mesoporous titanium oxide whisker

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A R T I C L E I N F O

Article history:
Received 1 March 2012
Received in revised form
18 August 2012
Accepted 15 October 2012
Available online 26 October 2012

Keywords:
Porous composites
PEEK (polyetheretherketone)
Nano-micro pores
Friction and wear
Mesopore titanium oxide whisker

A B S T R A C T

Hierarchical porous PEEK self-lubricating composites were prepared by mold-leaching and vacuum melting process under high temperature. The tribological behaviors were investigated for the porous PEEK composite and the porous composite after incorporating micro-porogen (NaCl) and mesoporous titanium oxide whiskers. If only micro-porogen was incorporated, the lowest steady state specific wear rate was observed for PEEK composites filled with 30% NaCl. Based on this porous PEEK composite, the effects of mesoporous titanium oxide whiskers and non-perforated titanium oxide whiskers on the friction and wear properties of PEEK composites were studied. Results showed that nano-micro porous PEEK composites with 30 wt% micro-porogen and 5 wt% mesoporous titanium oxide whiskers reached the lowest friction coefficient and specific wear rate, which were recorded as 0.0194 and 2.135×10^{-16} m^3/Nm under the load of 200 N. Compared with 15 wt% carbon fiber-reinforced PEEK composite which is widely used in industry, the wear resistance of the designed hierarchical porous PEEK composite increased by 41 times, showing outstanding wear resistance.

1. Introduction

PEEK (polyetheretherketone) is an important engineering material. As a typical high performance semicrystalline thermoplastic polymer, PEEK has received significant attentions in recent years due to its high mechanical strength and elastic modulus, satisfied combination of thermal and mechanical properties, chemical inertness, high toughness, easy processing, high wear resistance and low friction coefficient [1–3]. Recently, the research on the manufacturing and investigation on friction and wear properties of PEEK and its composites have attracted more interest, and PEEK has become prevalent in the areas of engineering plastics and tribology [4]. Many researchers have modified PEEK to enhance its mechanical and tribological properties by fiber-reinforcing, organic-blending, inorganic micron- and nano-filling. Previous literatures focusing on the tribological properties of PEEK have been mainly done by the groups of Friedrich [5], Burris [6], Bahaduer [7] and Briscoe [8].

With the rapid development of modern technology, high performance PEEK composites with advanced tribological properties are in urgent demand. However, the high friction coefficient and wear rate of pure PEEK and PEEK composites are the main obstacles for their wider applications under high load, velocity and temperature [9]. Nevertheless, porous self-lubricating structural design based on PEEK matrix could be a promising alternative for economical, ecological and technical concerns. The porous structure is able to uptake grease lubricant. During tribological testing, the grease will be squeezed out of the porous structure under the loaded pressure, and will be exuded well on the sliding surface with the effect of generated friction heat. Then, grease lubricating layers can be formed on friction surface for a long working period with low friction and high wear resistance [10]. Some efforts have been made to investigate the effect of internal solid and/or oil lubricants on tribological behaviors of porous polyamide (PA)[11], polyimide (PI) [12] and ceramics [13]. However, to the best of our knowledge, no literature is available regarding hierarchical porous polymers for tribological applications.

In this study, an attempt has been made to develop an effective processing technique and evaluate the feasibility of using NaCl, mesoporous titanium oxide whisker and non-perforated titanium oxide whisker as fillers for PEEK. The aim of this work is to develop a preparation method for a new self-lubricating material—hierarchical porous PEEK material impregnated with lithium-base grease and to study their tribological properties.

2. Experiment work

2.1. Materials and preparation of the PEEK composites

PEEK powder in 160-mesh size (supplied by Jilin University High-Technology CO. LTD, Changchun, China), was used as the polymer matrix material. Sodium chloride was sieved, which

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http://dx.doi.org/10.1016/j.wear.2012.10.008

plays the role of pore-forming agent. Mesoporous titanium oxide whisker (M–TiO_2–W, as shown in Fig. 3) (supplied by the College of Chemical engineering, Nanjing University of Technology) was synthesized from the sintered $K_2Ti_2O_5$, which involves a novel hydrolytic step for the mesoscopic microphase separation [14]. The PEEK powder, NaCl, M–TiO_2–W and non-perforated titanium oxide whisker (N–TiO_2–W) were dried at 120 °C for 4 h, and then mixed mechanically.

The samples were produced by molding in a press mold, followed by sintering at 360 °C for 2 h. Then, the samples were cut into a shape with an external diameter of 32 mm, an inner diameter of 22 mm, and a shoulder height of 2.5–3 mm. The porous polymer structure was obtained via leaching out the porogen in an 80 °C deionized water bath with the assistance of sonication. Finally, the porous structure was filled with lithium-base grease at 150 °C for 2 h under vacuum condition of 0.01–0.2 bar.

2.2. X-ray diffraction(XRD) tests and BET tests

Powder XRD spectra, recorded on a D/max2200 spectrometer (Japan) using Cu Kα radiation, with the samples scanned from 10 to 80° in the steps of 0.02° at each point, were taken to confirm the TiO_2 structure.

Structural properties of samples were determined by N_2 physisorption in a Micromeritics TristarII 3020 apparatus. Surface areas were calculated by the BET method (S_{BET}). Pore volume (V_P) was determined by nitrogen adsorption at a relative pressure of 0.99. Pore size distributions were found from adsorption isotherms by the BJH method.

2.3. Friction and wear tests

The friction and wear tests were run on an MPX-2000 friction and wear tester (Xuanhua Testing Factory, China). Sliding experiments were performed in the ring-on-ring configuration. Tests were conducted under ambient conditions with sliding speeds of 1.40 m/s and normal loads of 100 N and 200 N. The tests duration was 120 min. Before each test, the specimen and counterpart ring were polished to an average roughness of 0.15–0.3 μm with 1000-grit SiC abrasive paper and cleaned with acetone. The samples were put into an oven at 135 °C for 8 h before they were weighed. Computer recorded the frictional torque data once in a second, and the friction coefficient was taken as the average value of the last 60 min. The specific wear rate [Wr (m^3/Nm)] was calculated using the following equation:

$$Wr = \frac{\Delta m}{L\rho F_N} \tag{1}$$

where Δm is the mass loss (g), L is the sliding distance (m), ρ is the density of the composite (g/cm^3), and F_N is the normal load (N). Temperatures in the friction zone were measured by infrared thermometer.

In this work, the friction and wear tests were repeated three times to minimize data scattering, and the average value was reported. The microstructure of the cross-section, worn surface and counterpart surface were investigated with a Quanta 200 scanning electron microscope (FEI Co., Eindhoven, Netherlands).

3. Results and discussion

3.1. XRD and BET comparison of the M–TiO_2–W and N–TiO_2–W

Fig. 1 shows the X-ray diffraction pattern of TiO_2 whiskers. It can be seen that the diffraction peaks of TiO_2 whisker are completely consistent with the characteristic diffraction peaks of the anatase TiO_2. Meanwhile, no diffraction peaks of other crystalline TiO_2 or other substances were found. Therefore, the mesoporous titanium whiskers and non-porous titanium whiskers are mainly anatase TiO_2.

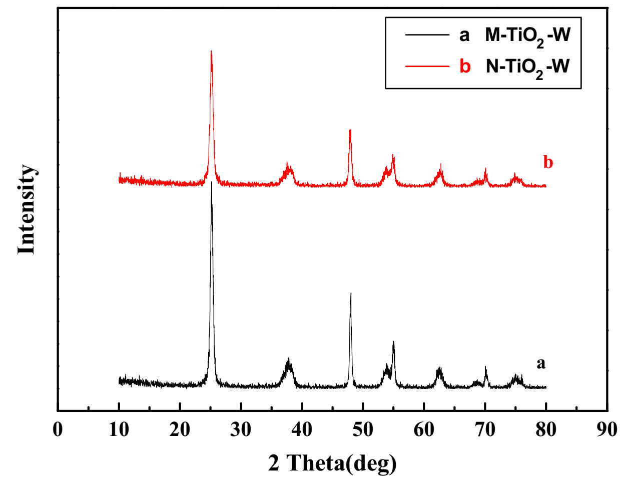

Fig. 1. XRD patterns of the sintered products: (a) M–TiO_2–W and (b) N–TiO_2–W.

Table 1
Pore structure properties of M–TiO_2–W and N–TiO_2–W.

	S_{BET}/m^2 g^{-1}	V_p/m^3 g^{-1}	D_{BJH}/nm
N–TiO_2–W	13	0.02	–
M–TiO_2–W	96	0.35	11.2

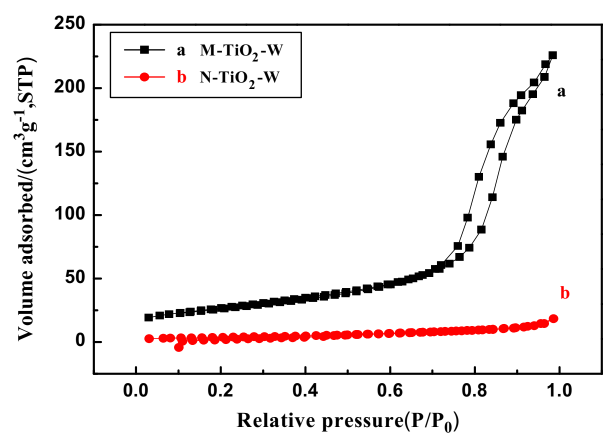

Fig. 2. N_2 adsorption/desorption isotherm of products prepared of M–TiO_2–W and N–TiO_2–W.

The pore structure parameters of the sample such as the surface area, pore size and pore volume are determined from BET results, as listed in Table 1. From the shape and characteristics of curve(a) in Fig. 2, the N_2 adsorption- desorption isotherms of M–TiO_2–W shows typical IUPAC(II) type adsorption characteristics, with a significant hysteresis loop. This proves that the obtained M–TiO_2–W is the mesoporous material. The average pore diameter is 11.2 nm and its surface area is 7.38 times larger than that of N–TiO_2–W. Meanwhile, from Fig. 3 it can be seen obviously that the selected two kinds of whiskers are titanium dioxide whisker with well-defined separated fibrous morphology [14].