Wear 316 (2014) 30–36

Contents lists available at ScienceDirect

Wear

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

Effect of proton irradiation on the friction and wear properties of polyimide

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ARTICLE INFO

Article history:
Received 26 January 2014
Received in revised form
15 April 2014
Accepted 16 April 2014
Available online 26 April 2014

Keywords:
Polymers
Sliding friction
Proton irradiation
Three-body abrasion

ABSTRACT

Effect of 25 keV proton irradiation with fluence of 2.25×10^{17} ion/cm^2 on the structural and tribological properties of polyimide blocks were investigated in a ground-based simulation facility. The changes in surface structure were characterized by FTIR-ATR, laser micro-Raman, contact angle measurement and nanoindentation. The experimental results indicated that the proton irradiation induced bond breaking to form the carbon-enriched structure on polyimide surface, and then increased the surface hardness and the surface energy. The irradiation depth was restricted within 514 nm from the surface by TRIM simulation.

The carbonized layer induced by irradiation was worn out in friction test, which was proved through Raman spectra analysis of the wear track. Proton irradiation increased the initial friction coefficient and decreased the steady friction coefficient of polyimide. In the initial stage, the friction coefficient was closely related to surface hardness of material, and the main wear mechanism was adhesive wear. In the steady stage, the main wear mechanism was three-body abrasion wear, three-body abrasion and the low surface energy could reduce the wear rate and the friction coefficient.

1. Introduction

There exist many complicated and changeable environmental factors in the cosmic space, including high vacuum, ultraviolet rays, atomic oxygen, and charged particle radiation, etc., especially the irradiation of protons and electrons in the earth radiation belt [1–3]. Polymers are widely applied in space systems as thermal blankets, structural materials, thermal control coatings, adhesives and solid lubricants owing to some unusual properties, such as high strength-to-weight ratio, good mechanical properties, excellent thermal stability, and chemical inertness [4]. However, polymers are vulnerable to be influenced by harsh space environment, especially proton irradiation, which will affect the life-span and reliability of spacecraft [5,6].

Much attention has been paid to the degradation of polymer thermal control material due to proton irradiation over the past decades. Such degradation could destroy the thermal balance of spacecraft [7–9]. Besides polymer applied in thermal control coatings, it is also one of the vital tribological materials in space environment. Existing and future moving mechanical assemblies of spacecraft mainly depend on the maintenance free, low friction and low wear [10]. The tribological fault caused by irradiation is one of the primary reasons for the spacecraft malfunctions, which often arises catastrophic results. In addition, there is no lubrication system as back-up for such tribological failure in a large number of cases [11]. Therefore, in order to improve the service reliability and long life of spacecraft, it is very necessary to study on the damage mechanism of tribological materials caused by irradiation through ground simulation experiments.

Polyimide (PI) is a kind of important engineering plastic which is widely used as friction material in space science. It is well known that PI exhibit superior friction and wear characteristics in vacuum [12,13]. Recently, the evolution of properties and damage mechanism of polymer tribological materials by ion irradiation have been reported. Some researchers found that ion irradiation increased the friction of polymer materials [14,15], others found the contrary results [16]. However, these researches did not refer to the irradiation depth in polymer. During the friction test, the irradiation layer may be worn out, which would directly affect the change of tribological properties. At present, to the author's knowledge, there are no reports on revealing the relationship of proton irradiation depth and the evolution of wear mechanism. This work is an attempt to fill this gap.

In this paper, we obtained the proton irradiation depth in PI blocks using the well-known Monte Carlo simulation method

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http://dx.doi.org/10.1016/j.wear.2014.04.011
0043-1648/

(TRIM program), which are successfully applied to irradiation damage, ion implantation, surface scattering, and so on [17,18]. The tribological properties of irradiated PI against GCr15 steel ball were performed on ball-on-disc tribometer in the ground simulation facility. The changes in microstructure and the ingredients of wear debris were detected by FTIR-ATR and laser micro-Raman. The morphologies of wear track were examined by scanning electron microscopy.

2. Experimental

2.1. Materials

The polyimide (ODA-ODPA) powder (shown in Fig. 1) was purchased from Shanghai Research Institute of Synthetic Resins (Shanghai, China). To produce samples for testing, PI powder was heated from room temperature to a maximum temperature of 375 °C at a rate of 2 °C min^{-1} in mold, and then held at 375 °C and 20 MPa for 90 min to allow full compression sintering with intermittent bumping to release the trapped moisture. Finally, the mold in the press was cooled to room temperature in air. The thermoforming PI were cut into 18 mm × 18 mm × 2 mm blocks for irradiation and wear tests. Before proton irradiated, the sample surface was polished to the roughness Ra ≤ 0.2 μm and Rz ≤ 0.4 μm, and cleaned in acetone by ultrasonic.

2.2. Proton irradiation on polyimide blocks

In this study, proton irradiation was carried out in a ground-based simulation facility in the Lanzhou Institute of Chemical Physics, Chinese Academy of Sciences. The schematic view of the irradiation chamber was shown in Fig. 2. The 25 keV H^+ ions were produced by ECR microwave plasma technology. The beam was 50 mm in diameter and the beam current was 1.0 mA. The irradiation fluence was 2.25×10^{17} ion/cm^2 and the irradiation

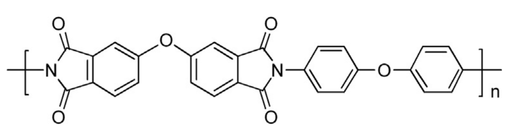

Fig. 1. Chemical structure of polyimide.

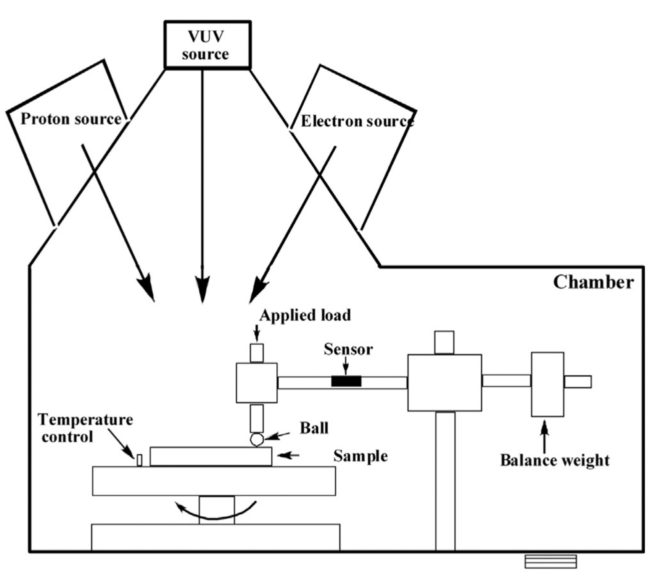

Fig. 2. The schematic view of the irradiation chamber.

was done in high vacuum environment (10^{-4} Pa). The irradiation resulted in a visible color change. After irradiation, the color of PI blocks changed to black from original yellow. However, the irradiated black surface was easily polished off with metallographic sandpaper. In this study, the temperature of PI surface after proton irradiation was measured by a thermocouple situated beside the sample and in the range of the ion beam (Fig. 2). The proton irradiation depth in PI blocks was calculated by TRIM code which is the part of the SRIM program package. On the basis of quantum mechanical Monte Carlo simulation, this program could estimate the stopping power and the range of ions in matter [19,20]. The mechanisms of ion stopping in matters are divided into nuclear and electronic collisions, and the electronic collision dominates for the light incident ions (i.e., H^+, He^+) [19,21]. Fig. 3 shows relationship between stopping power and proton irradiation depth in PI block.

2.3. Surface characterization of polyimide

The infrared spectroscopic measurements were carried out subsequent to the irradiation and outside of the high vacuum chamber with a Nexus 870 infrared spectrometer using an attenuated total reflection accessory (FTIR-ATR). The changes in microstructure and the ingredients of wear debris were recorded by a laser micro-Raman microscope (LabRam HR800, Japan) at an excitation wavelength of 633 nm. The worn surface morphologies and the width of the wear track were observed using a JEM-5600LV scanning electron microscope (JEOL, Japan). The roughness was determined using a three-dimensional non-contact surface mapping profiler (MicroXAM, ADE Corporation Inc.). A Hysitron TI 950 TriboIndenter was used to obtain the nanomechanical properties of sample surface, and the indentation depth was 500 nm, and the testing standard is general rules of instrumented nanoindentation test (GB/T 22458-2008). Contact angle measurements were performed by the static sessile drop method using a DSA-100 optical contact-angle meter (Kruss Company Ltd., Germany) at room temperature (23 °C). The average contact angle values were obtained by measuring the same sample at five different positions with 5 μL double distilled water or diiodomethane. Images were captured with a Sony Digital Camera (Sony Ltd., Japan). The method of Owens and Wendt was used for the calculation of the total surface energy and its polar and dispersive components [22].

2.4. Wear tests

Friction and wear data were collected using a ball-on-disc tribometer in the ground simulation facility at room temperature in a vacuum level of 3×10^{-4} Pa. In this study, the wear tests were performed after the proton irradiation. The counterpart ball, made of the GCr15 stainless with the diameter of 3.175 mm, slid on a disk rotating at a speed of 0.126 m/s under a load of 1 N for 1200 s. And the starting stress levels in Hertz-contact were calculated to be 67 MPa for the untreated PI and 106 MPa for the irradiated one, respectively. The rotatory diameter was 12 mm. Before each test, the steel balls were cleaned in acetone by ultrasonic.

Fig. 4 shows the calculation for depth of the wear track and wear rate. Where b is the width of the wear track measured by SEM, r is the radius of the steel ball (1.588 mm), d is the diameter of the disk (12 mm), D is the depth of wear track (mm), V is the wear volume loss (mm^3), K (mm^3 Nm^{-1}) is wear rate value, P is the applied (N) load and L is the sliding distance (m).

In this work, three specimens were tested under each condition, and the average wear rate of three replicate samples was taken to evaluate the wear resistance of PI.