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Effects of Individual and Sequential Irradiation with Atomic Oxygen and Proton on the Surface Structure and Tribological Performance of Polyetheretherketone in Simulated Space Environment

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Abstract

The changes in the surface structure and the tribological performance of polyetheretherketone (PEEK) induced by individual and sequential irradiations with atomic oxygen (AO) and proton (Pr) were investigated in a space environment simulation facility. The experimental results showed that Pr irradiation induced the surface carbonization of PEEK which induced the greatest degree of decreasing in the surface roughness from 29.61 nm to 16.15 nm, surface energy from 49.16 mJ/m^2 to 46.96 mJ/m^2, friction coefficient from 0.28 to 0.08 and wear rate from 10.28 × 10^{-5} mm^3/Nm to 5.45 × 10^{-5} mm^3/Nm. AO irradiation induced the surface oxidation of PEEK, and then increased the surface roughness from 29.61 nm to 58.77 nm, surface energy from 49.16 mJ/m^2 to 73.75 mJ/m^2, friction coefficient from 0.28 to 0.35 and wear rate from 10.28 × 10^{-5} mm^3/Nm to 18.22 × 10^{-5} mm^3/Nm. The surface structural variations and tribological performance of PEEK induced by sequential Pr–AO and AO–Pr irradiations were respectively similar to the results of individual AO and Pr irradiation, and the final form of irradiation has a bigger effect on the changes in surface structure and tribological performance during the sequential irradiation tests. The erosion stacking effect of sequential irradiations was observed, and the AO–Pr irradiations caused the biggest changes in infrared spectra and the surface composition of C and O elements in X–ray photoelectron spectroscopy. Pr–AO irradiations have the biggest increment in surface energy from 49.16 mJ/m^2 to 74.03 mJ/m^2 and wear rate from 10.28 × 10^{-5} mm^3/Nm to 24.07 × 10^{-5} mm^3/Nm.

KEYWORDS: Polyetheretherketone; Atomic oxygen and proton irradiations; Surface structure; Sliding friction; Tribological performance.

Introduction

It is known that polymer materials are widely applied for friction material as critical move assemblies of satellites and spacecrafts in space systems owing to good mechanical performance, low weight, high wear resistance, easy manufacturing processes, resistance to irradiation, self–lubrication properties and chemical inertness.[1, 2] Polyetheretherketone (PEEK) as one of the high performance engineering thermoplastic polymers has attracted increasing interests due to its attractive physical and chemical properties, such as excellent mechanical properties, good chemical resistance and high long–term working temperatures, which is considered to be one of the most prospective applications in biomedical applications, the automotive industry, electronics and spacecraft design among the various polymers.[3-7]

The cosmic space exist many rigorous environmental factors including high vacuum, thermal cycles, ultraviolet rays, atomic oxygen, and proton irradiation, electron irradiation and so on, which could severely affect the service life and reliability of mechanical equipment.[8-10] Thereby the materials used in satellites or spacecrafts would be proposed higher demand to defense rigorous space environment. However, another important research subject on the damage to polymer materials in cosmic space environment also induced the broad interests.

In our previous work, the changes in surface structure and tribological performance of the polytetrafluoroethylene, polyimide, phenolphthalein poly (ether sulfone) and their composites enforced with various fibers or nano–oxides have been investigted under the single form of irradiation with proton (Pr), electron, atomic oxygen (AO) or ultraviolet rays in simulated space environment.[11-15] With the design improvements of materials, a kind of porous polyimide material with highly stable tribological performance and hydrophobicity in a simulated space environment has been reported.[16] The experimental reuslts showed that the Pr or AO irradiation induced more effects on the surface performance of these polymers compared to the other forms of single irradiation especially for the tribological performance, and the order of irradiations have big effect on tribological performance and surface properties of polymer materials. PEEK as one of important space materials, the changes in the surface structure and tribological performance are particularly important to design the airspace parts in simulated space environment. Moreover, the AO and Pr as most destructive irradiations may result in different impact on the properties of PEEK. To the best of our knowledge, there has been no attempt on the effect of the sequential irradiation with AO and Pr on the polymer materials, which makes a very valuable research content. More importantly, the study of sequential irradiation on polymer material is very meaningful from both the basic research and practical application. On the one hand, the irradiation damage mechanism of polyer materials can be further revealed by subquential irradiation. On the other hand, it is contribute to improve and design the service reliability and long life of spacecraft.

In this paper, the effect of individual and sequential irradiations with Pr and AO on the surface properties and tribological performance of PEEK were investigated in the ground simulation facility. The changes in the surface structure before and after irradiations were detected by attenuated total reflectance infrared spectroscopy (ATR–FTIR), X–ray photoelectron spectroscopy (XPS), contact angle measurements and X–ray Diffractometer (XRD). The changes in the tribological performance of PEEK before and after irradiations were investigated on ball–on–disc tribometer that was used for tribological test because of its low cost, small space and the ease of handling little samples. The morphologies of wear track were observed by scanning electron microscopy (SEM).

Experimental

Materials

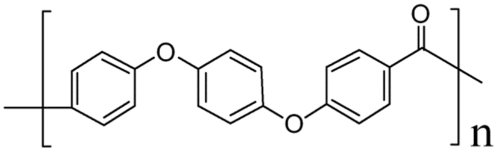


Fig. 1. The chemical repeat unit of PEEK.

The PEEK powder (450P, molecular structure was shown in Fig. 1) used in this study was supplied by Victrex (Lancashire, UK). The density of PEEK is 1.32 g/cm^3, and its glass transition temperature, the melting point, and the decomposition temperature are 143, 334 and 590 °C, respectively. PEEK powder were pressed in mold and heated to 375 °C, and held at 20 MPa for 120 min to form a 50 × 60 × 8 mm^3 block. The thermoforming PEEK were then cut into 18 × 18 × 2 mm^3 blocks for irradiation and wear test. Every sample surface was polished carefully to the roughness Ra $\leq$ 0.2 μm. All the samples were cleaned with ultrasonic in acetone before irradiation test.

Irradiation test

The experiments of AO and Pr irradiations were performed in a space simulation facility in Lanzhou Institute of Chemical Physics, Chinese Academy of Sciences. The basic principle schematic illustration of the irradiations was referred in our previous papers.[17, 18] As for the AO irradiation, a microwave power source with electron convolute resonance technique was used to excite O_2 to produce oxygen plasma which would become a beam and be accelerated towards a molybdenum plate with a negatively charged electric field. As colliding with the plate the accelerated oxygen positive ions are neutralized by the negative charges and rebounded to form a neutral AO beam with a mean kinetic energy of about 5 eV that is similar to the energy of AO impinging on the surface of spacecraft in space environment.[19, 20] The flux of AO beam was determined to be 6.0×10^{15} atoms cm^{-2} s^{-1} by the standard method of Kapton mass loss.[19, 21] The Pr irradiation was carried out at an accelerative voltage of 25 kV and the flux of protons was determined to be about 6.25×10^{15} ions/$cm^2 \cdot s$. The tests of AO and Pr irradiations were performed in individually and sequentially ways, respectively. The individual irradiation time of AO and Pr irradiations was controlled about 180 min and 5 min, respectively. For Pr irradiation, 5 min was selected due to Pr possessing higher energy for corroding polymer molecules compared with AO.[22] The experimental procedure for the sequential irradiation is that the sample was first irradiated with Pr for 5 min and then with AO irradiation for 180 min (or AO for 180 min and then Pr irradiation for 5min).

Surface characterization of PEEK

The infrared spectroscopic measurements of PEEK samples before and after irradiations were performed on a Nexus 870 FTIR spectrometer (Nicolet, America) using an attenuated total reflection accessory (ATR) technique with a germanium crystal. The surface chemical composition before and after irradiations were analyzed using an ESCALAB 250Xi X–ray photoelectron spectroscopy instrument (ThermoFisher, America). All spectra were acquired using Al–Kα X–ray source (1391 eV) with a binding energy range of 0−1400 eV. All binding energy were referenced to the C1s hydrocarbon peak at 284.6 eV. 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 (25 °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 total surface energy and its polar and dispersive components were calculated using the method of Owens and Wendt.[23] The surface morphologies and the width of the wear track were observed using a JEM–5600LV scanning electron microscope (JEOL, Japan). The average roughness (Ra) of samples was measured on a MicroXAM 3D non–contact surface mapping profiler (ADE Corporation, America). The three–dimensional (3D) images and root–mean–square roughness values (RMS) of the samples before and after irradiations have been acquired using scanning probe microscope integrated in a Hysitron Triboindenter TI–950 system (Hysitron, America).

Friction and wear test

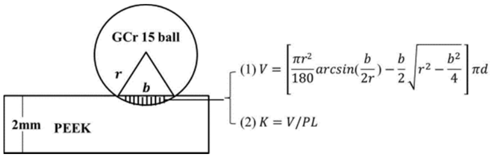


Fig. 2. Calculation formulas of the wear rate.

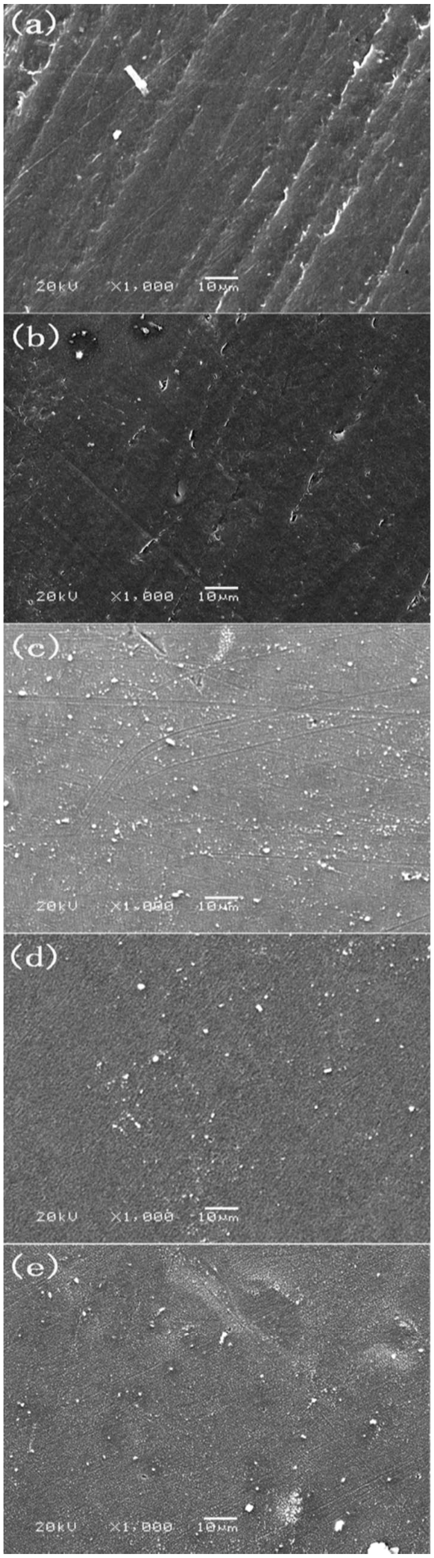
The friction and wear behaviors of the PEEK before and after Pr irradiation against GCr15 steel ball were tested on a ball–on–disk tribometer in a vacuum level of 3×10^{-4} Pa. The GCr15 steel ball has a standard 3 mm diameter with the chemical composition (in wt%): Mn (0.20–0.40), Si (0.15–0.35), Cr (1.30–1.65), C (0.75–0.85), P (≤0.026), S (≤0.020) and Fe balance.

The steel ball slid on sample disk that rotate at a speed of 0.126 m/s under the load of 0.5 N for 1800 s with a rotational diameter of 12 mm. The corresponding starting stress level is about 100 MPa by Hertz-contact formulae. The calculation for wear rate was shown in Fig. 2. Where b and d respectively refers to the width and the diameter of the wear track (12 mm), r denote the radius of the counterpart steel ball, V correspond to the wear volume loss (mm^3), K ($mm^3 \cdot Nm^{-1}$) is the wear rate value, L is the sliding distance (m) and P is the applied load (N). In order to minimize the error, three specimens were tested under each condition to get the average wear rate of samples.

Results and discussion

Surface morphologies

The surface morphologies of PEEK samples before and after individual and sequential irradiations with AO and Pr were studied by SEM, and the results were given in Fig. 3. The surface morphologies of the untreated and Pr irradiated PEEK were relatively plat in Fig. 3a and b. However, the surface morphologies of AO, Pr–AO and AO–Pr irradiated PEEK had significantly changed and exhibited ‘blanket-like’ structure shown in Fig. 3c, d and e. In order to get a precise analysis of the surface roughness, the 3D images and RMS before and after individual and sequential irradiations with AO and Pr were presented in Fig. 4. The surface roughness of the PEEK was obviously decreased from 29.61 nm to 16.15 nm after Pr irradiation. While the surface roughness of the PEEK was significantly increased from 29.61 nm to 58.77 nm after AO irradiation, which could be ascribed to the numerous larger short cones formed in surface. In comparison with the analytical results, the changes in surface morphologies of PEEK induced by AO and Pr irradiations were opposite process, and the Pr irradiation made the PEEK surface became smooth, AO irradiation made the PEEK surface became coarser. Thus, the surface roughness of PEEK changed a little in sequential AO–Pr and Pr–AO irradiated experiments compared to that of untreated PEEK. However, the short cones became smaller and denser probably due to the combined effect of AO and Pr irradiations and different erosion mechanism.



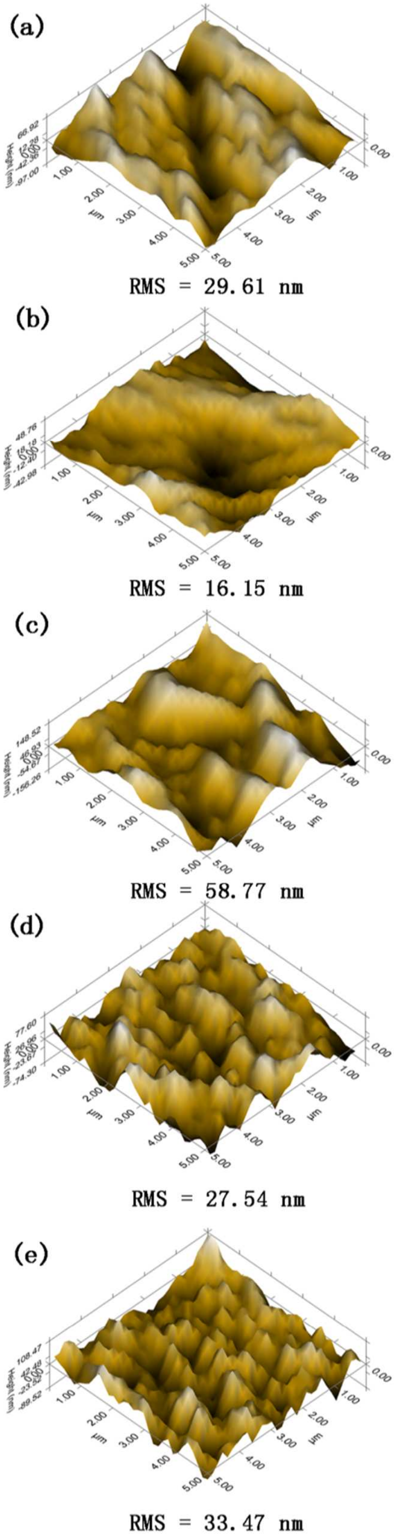


Fig. 3. SEM images of PEEK specimens before

Fig. 4. 3D images (5 μ m × 5 μ m) and RMS of after

and irradiations: (a) untreated PEEK, (b) Pr, (c) AO, (d) Pr–AO, (e) AO–Pr.

PEEK specimens before and after irradiations: (a) untreated PEEK, (b) Pr, (c) AO, (d) Pr–AO, (e) AO–Pr.

XRD analysis

PEEK is a semi–crystalline thermoplastic polymer. The effect of irradiations on the crystallinity variation of the PEEK was investigated by XRD technique, and the results were shown in Fig. 5. The four distinct peaks were observed at about 18.81°, 20.81°, 22.80° and 28.80° that can be assigned to the (110), (111), (200) and (211) planes of crystallized PEEK, respectively, which indicated that the PEEK mainly exhibited an orthorhombic crystalline form.[24, 25] In addition, the sharp and diffuse patterns for PEEK were characteristic of semi–crystalline polymers. All diffraction peak positions have not shift after individual and sequential irradiations with AO and Pr signifying that lattice parameters did not change, which is due to the fact that the Pr and AO irradiations only lead to the degradation of outmost surface of the polymer material.[17, 26] and the microscopic changes on PEEK surface were not observed by XRD, which shows that the irradiations could not affect the performance of main body materials.

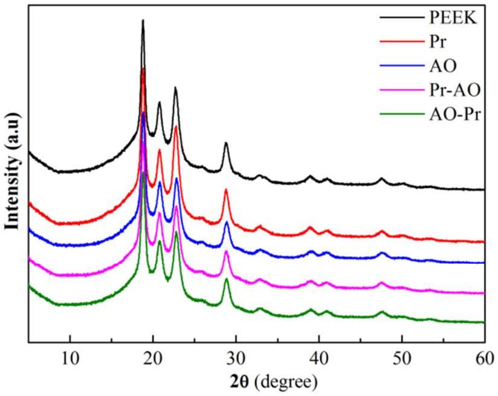


Fig. 5. The XRD profiles of PEEK specimens before and after irradiations.

ATR–FTIR spectra

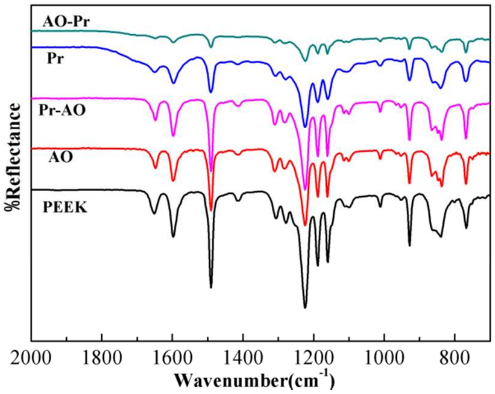


Fig. 6. The ATR–FTIR spectra of the PEEK specimens before and after irradiations.

The changes in chemical structure of PEEK surface induced by individual and sequential irradiations with AO and Pr were studied by ATR–FTIR, and the results were presented in Fig. 6. The typical characteristic peaks of the original PEEK was at 1651 cm^{-1} due to C=O stretching vibration, 1598, 1490 and 1413 cm^{-1} due to the aromatic skeletal vibration, 1306 cm^{-1} ascribed to the bending motion of C–C(=O)–C, 1280 and 1187 cm^{-1} due to the asymmetric stretching of C–O–C, 1157 and 1103 cm^{-1} due to a number of aromatic hydrogens in–plane deformation bands, 927 cm^{-1} due to the diphenyl ketone band, 860 and 841 cm^{-1} attributed to the out of plane bending modes of the aromatic hydrogens, which was consistent with the report of this material.[27, 28] After individual and sequential irradiations with AO and Pr, the intensity of these characteristic peaks for the PEEK samples decreased in a different degree which indicated that irradiations induced a different level of breakage of the molecule chains of PEEK samples, and the complex chemical reaction may take place during the irradiations process. During all irradiated conditions, AO–Pr irradiation caused the signal peaks of PEEK nearly disappeared, which indicated that AO first and then Pr irradiation could give rise to the worst erosion on PEEK surface, and this case also confirmed that the stacking effect could be happened by multiple forms of irradiations.

Changes in surface chemical composition

Table 1 The surface composition of PEEK specimens before and after irradiations by XPS.

Specimens	Surface composition (at.%)	
	C	O
Untreated PEEK	77.64	21.89
Pr irradiated	80.56	19.43
AO irradiated	66.47	33.53
Pr–AO irradiated	67.44	31.96
AO–Pr irradiated	80.64	19.36

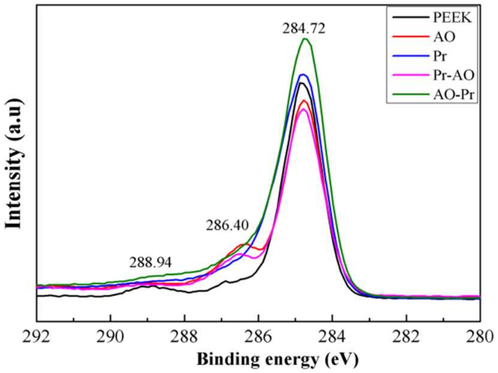


Fig. 7. The HR C1s spectra of PEEK specimens before and after irradiations.

In order to reveal the possible chemical reaction occurring in PEEK surface during various irradiation processes, the changes in surface chemical composition induced by individual and sequential irradiations with AO and Pr were studied by XPS, and the corresponding results were summarized in Table 1. The untreated PEEK has a composition of C 77.64 %, and O 21.89 %. The surface composition has experienced obvious changes after different irradiations in this test. It is found that the surface concentrations of C element were respectively increased to 80.56 % and 80.64 % whereas the concentrations of O element were respectively decreased to 19.43 % and 19.36 % after individual Pr irradiation and sequential AO–Pr irradiations. However, the surface concentrations of C element were respectively decreased to 66.47 % and 67.44 % whereas the concentrations of O element were respectively increased to 33.53 % and 31.96 % after individual AO irradiation and sequential Pr–AO irradiations. These results indicated that both individual Pr irradiation and sequential AO–Pr irradiations could lead to the surface carbonization of PEEK. While individual AO irradiation and sequential Pr–AO irradiations could result in the surface oxidation of the PEEK. As shown chemical structure of PEEK in the Fig. 1, there are three different carbon environments: (1) carbon atoms in the groups of aromatic rings (284.72 eV), (2) carbon atoms in the groups of C–O (286.40 eV), (3) carbon atoms in the groups of carbonyl group (288.94 eV). Fig. 7 depicted the high resolution (HR) C1s XPS spectra of PEEK before and after irradiations. The relative content of C around 284.72 eV increased during the Pr and AO–Pr irradiations process, which may be due to the chain scission occurring during irradiations to form the amorphous carbon species in the surface of the PEEK and the irradiation region was also changed into black. The relative content of C in the groups of C–O obviously increased during the AO and Pr–AO irradiations process, which indicated that the molecular chains of PEEK were destroyed by the oxidation reaction to produce some oxygen–containing functional groups. Moreover, a tendency is that the latter irradiation would occupy dominant effect during the sequential irradiations. The results of two forms of irradiations showed that sequential irradiations induced a more influence than individual irradiation from the changes in elements of C and O, and the AO–Pr irradiations process caused the worst erosion, which is consist with the results of ATR–FTIR.

Changes in Surface energy

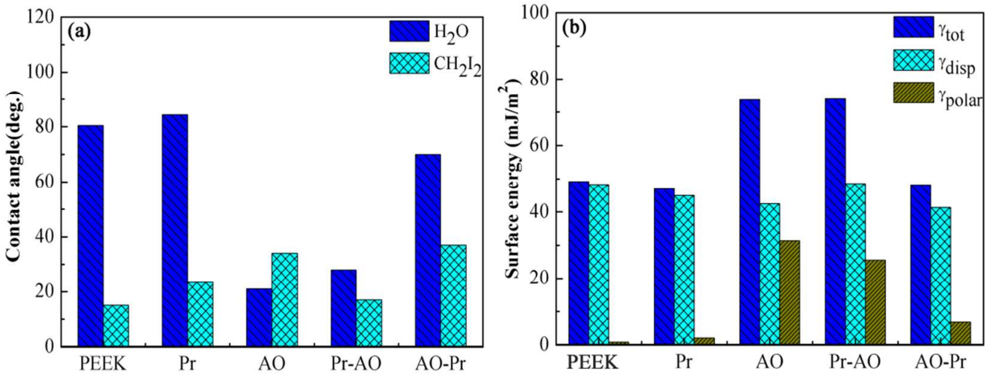


Fig. 8. The changes in contact angles (a) and total, polar and dispersive surface energies (b) of the PEEK specimens before and after irradiations.

The surface energy has different effects on the sliding friction behavior of the polymer.[29, 30] Some researchers have shown that ions irradiation could affect the surface energy of the polymer.[31, 32] In order to evaluate the changes in surface energy of the PEEK samples, the contact angle tests were carried out using the static sessile drop method. The changes of contact angles of PEEK in the water and diiodomethane were shown in Fig. 8a before and after individual and sequential irradiations with Pr and AO. Meanwhile, the changes in surface energy were determined by the evaluation of contact angles according to the method of Owens and Wendt, and the calculated values of the total surface energy (γ_{tot}) and its polar (γ_{polar}) and dispersive components (γ_{disp}) were presented in Fig. 8b. It can be seen that untreated PEEK has a surface energy of 49.16 mJ/m^2 which is very close to the dispersive component because of the negligible polar component. After individual Pr irradiation and sequential AO–Pr irradiations, the total surface energy decreased slightly to 46.96 and 48.20 mJ/m^2 which may be attributed essentially to the formation of carbonized layer on the PEEK surface. After individual AO irradiation and sequential Pr–AO irradiations, the total surface energy obviously increased to 73.75 and 74.03 mJ/m^2 that was mainly attributed to the great increment of the polar component as the formation of oxygen–containing functional groups. The individual and sequential irradiations induced different changes in contact angles and surface energies, and the changing trends were in accord with the results of latter irradiation. These results indicated that the changes in surface energy were related with the changes in the surface composition.

Friction and wear properties

Friction coefficients and wear rates of material are key parameters for the evaluation of tribological performance. The influence of irradiation environment on the friction and wear properties of the PEEK samples was investigated using a ball–on–disk tribometer. The friction coefficient variations of PEEK before and after irradiations were displayed in Fig. 9a. The friction coefficient of the untreated PEEK sample is stable around 0.28. After individual Pr irradiation, the friction coefficient was obviously decreased to 0.08, which is about 3.5 times lower than that of the untreated PEEK. After individual AO irradiation, the friction coefficient was increased to 0.35 that is around 1.25 times higher than that of untreated PEEK. It is worth noting that the friction coefficient of PEEK after the sequential AO–Pr irradiations was also significantly decreased to 0.08 and that after the sequential Pr–AO irradiations was increased to 0.31. The bar charts in Fig. 9b displayed the wear rates of PEEK before and after irradiations. The wear rate of untreated PEEK was 10.28× 10^{-5} mm^3/Nm. After individual Pr irradiation, the wear rate was obviously decreased to 5.45 × 10^{-5} mm^3/Nm, which is about 1.89 times lower than that of the untreated PEEK. After individual AO irradiation, the wear rate was increased to 18.22 × 10^{-5} mm^3/Nm that is about 1.77 times higher than that of the untreated PEEK. Similarly, the wear rate of PEEK after the sequential AO–Pr irradiations was decreased to 6.89 × 10^{-5} mm^3/Nm and that after the sequential Pr–AO irradiations was increased to 24.07× 10^{-5} mm^3/Nm. All the above results indicated that both the individual Pr irradiation and sequential AO–Pr irradiations could obviously decrease the friction coefficient and wear rate of the PEEK. Both the individual AO irradiation and sequential Pr–AO irradiations could induce an increment in friction coefficient and wear rate of the PEEK, and the Pr–AO irradiations have the biggest impact on wear rate of the PEEK, which also confirmed that the latter irradiation would play leading roles in the friction and wear properties of material. Moreover, the changes in the tribological properties induced by individual and sequential irradiations with AO and Pr were consistent with the surface energy and not the surface roughness.

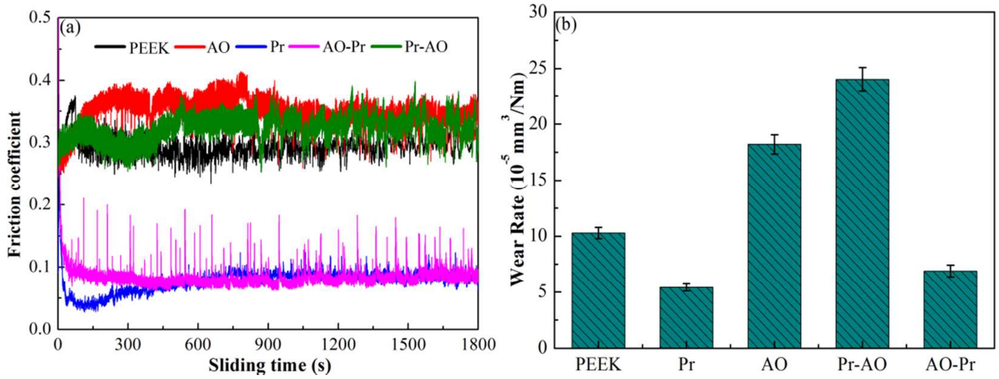


Fig. 9. The friction coefficients (a) and wear rates (b) of the PEEK specimens before and after irradiations.

The damages of the PEEK blocks after friction test before and after irradiations were further investigated by SEM, and the results were shown in Fig. 10. The low magnification images (Fig. 10a-e) mainly presented the wear track whereas the high magnification images (Fig. 10f-j) focused on the worn surface. The worn surface of the untreated PEEK was relatively smooth and mainly presented the plastic deformation (Fig. 10a, f). The individual Pr irradiated PEEK had a relative small wear track width with obvious grooves (Fig. 10b, g), which is because that the carbon–enriched wear debris during the friction process could introduce three–body abrasive wear arising from their high hardness and small size, and three–body abrasion could reduce the wear rate and friction coefficient. The individual AO irradiated PEEK displayed a relative larger wear track width with more serious plastic deformation (Fig. 10c, h), indicating that AO irradiation aggravated the friction and wear, which displayed the higher friction coefficient and wear rate by AO irradiation.

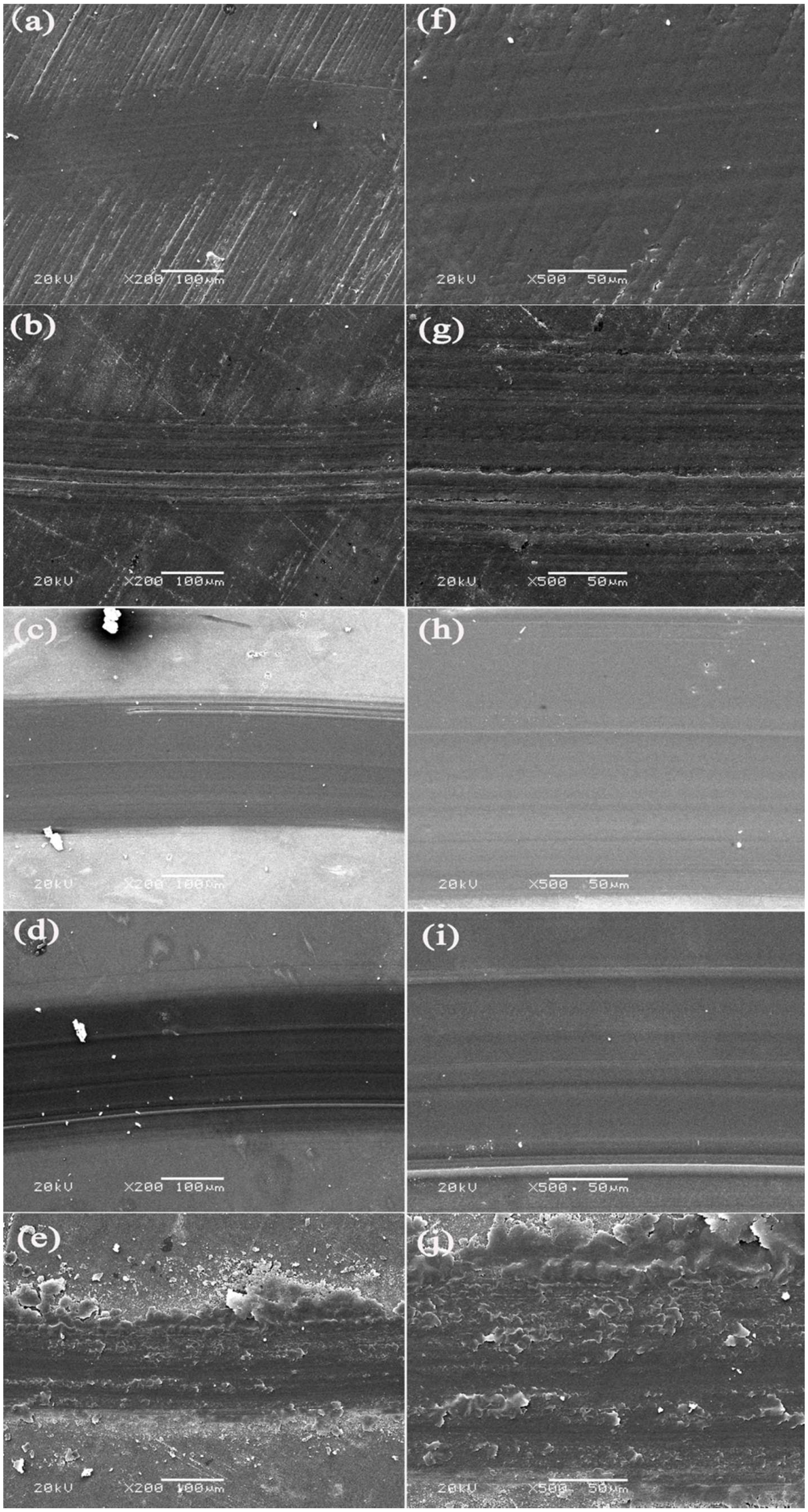


Fig. 10. SEM micrographs of the typical wear scars seen under low (a-e) and high (f-j) magnification: (a,f) PEEK, (b,g) Pr, (e,h) AO, (d,i) Pr–AO, (e,j) AO–Pr.

The sequential Pr–AO irradiated PEEK showed similar wear morphology to that of the AO irradiated PEEK except for the difference of the severer plastic deformation (Fig. 10d, i), which indicated that the sequential Pr–AO irradiations aggravated the friction and wear compared to the individual AO irradiation. Compared with the individual Pr irradiated sample, the sequential AO–Pr irradiated PEEK exhibited similar width of the wear track, but displayed a quite different aspect of production of massive flaky debris instead of the grooves (Fig. 10e, j), which indicated that the sequential AO–Pr irradiation aggravated the friction and wear compared to the individual Pr irradiation.

Conclusions

In this article, the effects of individual and sequential irradiations with AO and Pr on the surface structure and tribological performance of PEEK were evaluated by various characterization techniques. The individual Pr irradiation decreased the surface roughness of PEEK, but the individual AO irradiation greatly increased the surface roughness of PEEK. While the sequential AO–Pr or Pr–AO irradiations had little effect on the surface roughness of PEEK. The results of XRD and ATR–FTIR indicated that individual and sequential irradiations with AO and Pr only led to the degradation of outmost surface of the PEEK material. The individual Pr and sequential AO–Pr irradiations resulted in the carbonization of PEEK surface, which decreased the surface energy, friction coefficient and wear rate. Compared with the individual Pr irradiation, the sequential AO–Pr irradiations displayed the higher surface energy, friction coefficient and wear rate. The individual AO and sequential Pr–AO irradiations led to the surface oxidation, which caused an increment of the surface energy, friction coefficient and wear rate. Compared with the individual AO irradiation, the sequential Pr–AO irradiations presented the higher surface energy, friction coefficient and wear rate. The effect the sequential irradiation with Pr and AO on the surface structure and performance of PEEK was determined by the final form of irradiation.

Acknowledgments

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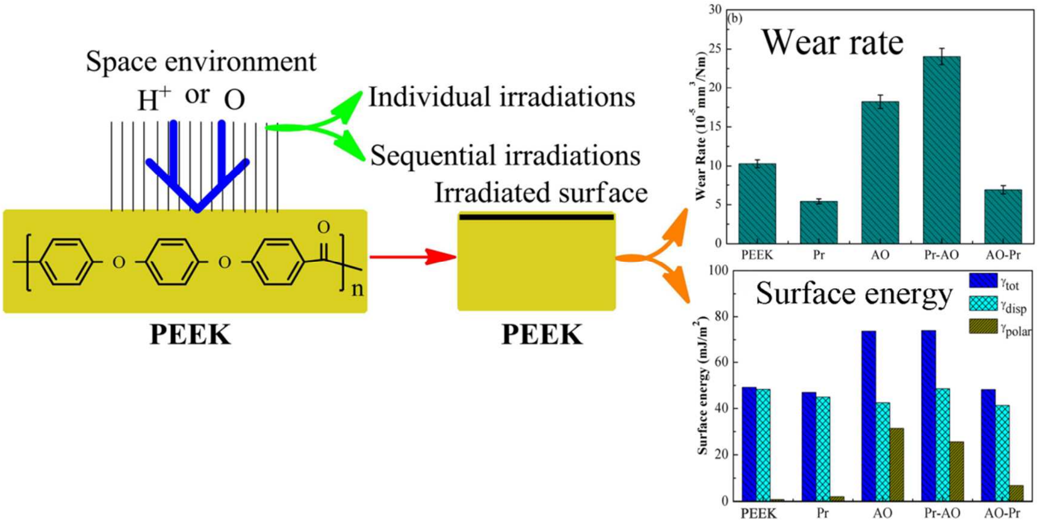
TOC graphic



AO and Pr–AO irradiations induced the higher surface energy and wear rates, Pr and AO-Pr irradiations caused the opposite results.