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Structure and micro-tribological properties of PTFE/ Al_2O_3 micro-assembling film ①

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Abstract: In order to improve the wear resistance of elastic metallic-plastic thrust bearing pad, micro-assembling PTFE/ Al_2O_3 multi-layer film was developed by alternating radio frequency(RF) magnetron sputtering PTFE and Al_2O_3 targets. For enhancing the adhesion of the interfaces between PTFE and Al_2O_3 film, N^+ implantation was employed. The structure, mechanical and micro-tribological properties were studied by XPS, X-ray photoelectron spectrometer and atomic force and friction force microscope (AFM/FFM). The results show that the multi-layer consists of Al_2O_3 component and crystalline PTFE. The hardness of the multi-layer modified by ion implantation is less than that of Al_2O_3, but its toughness is greatly improved. The friction coefficient of PTFE/ Al_2O_3 multi-layer modified by ion implantation is much lower than that of Al_2O_3 film, and its resistance to wear is much greater than that of PTFE film. Therefore the wear resistance of elastic metallic-plastic thrust bearing pad is greatly improved.

Key words: multi-layer film; PTFE; ion implantation; Al_2O_3

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1 INTRODUCTION

Compared with Babbitt pads of thrust bearing, the elastic metallic-plastic pads possess the combination of reliable running, excellent performance, flexible operation, convenient installation and long service life. Therefore the elastic metallic-plastic pads are widely employed in vertical electric machines of hydropower plants. Polytetrafluoroethylene (PTFE) has been used as the surface material of the elastic metallic-plastic pads because of its low friction coefficient and electrical insulation[1]; but its strength and wear resistance are not good enough. For solving this problem, several methods have been proposed, based directly on physical intermingle. To improve the compressibility, creepocity and wear resistance, a method of filling glass fiber into PTFE has been developed in Toshiba Company[2] of Japan. As a result, the friction coefficient of elastic metallic-plastic pads is higher than that of the former ones. And in order to lower the friction coefficient, MoS_2 is filled into these pads. MoS_2 is a nice solid lubricant with low shear strength due to its HCP structure. The friction and wear of the mechanical elements coated with MoS_2 films can be reduced effectively; especially in vacuum or in dry air environments. However, the function of MoS_2 in humid environments usually decreases greatly, which results in the limitation of its application. Al_2O_3 is a hard material with good wear resistance. According to the characteristics of PTFE and Al_2O_3, the idea of laminate structure is chosen to design a multi-layer with relatively low friction coefficient and high wear resistance. The micro-assembling PTFE/ Al_2O_3 multi-layer film was prepared by ion beam alternatively sputtering PTFE and Al_2O_3 targets, and the PTEE film is modified by N^+ implantation. The structure and micro-tribological properties of the multi-layer film are investigated.

2 EXPERIMENTAL

The micro-assembling PTFE/ Al_2O_3 multi-layer was prepared by Ar^+ beam alternatively sputtering pure PTFE and Al_2O_3 targets using an alternating radio frequency(RF) magnetron sputtering system[3-5]. The elastic metallic-plastic pad was used as substrate. The multi-layer was designed to 10 layers with alternative PTFE and Al_2O_3 layer. The inner was Al_2O_3 layer and the outer was PTFE layer. The deposition rate of PTFE and Al_2O_3 layer was 15 nm/min and 8 nm/min respectively, so the sputtering time was chosen 5 min for each PTFE layer and 10min for each Al_2O_3 layer. The thickness of each PTFE layer was almost the same as that of Al_2O_3 layer (80 nm). In order to enhance adhesion of interfaces, N^+ implantation[6-8] was employed for each PTFE layer and not for Al_2O_3 layer. The implantation was carried out with 50 keV and a dose of $1\times10^{17}N^+/cm^2$. Adjusting the acceleration energy and the ion current during the implantation can control the penetration depth and the dopant dose. Detail parameters of the sputtering process are listed in Table 1.

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Table 1 Parameters of magnetron sputtering

Samples	Base pressure/ 10^{-3} Pa	Sputtering energy/ keV	Sputtering current/ mA	Pressure of argon/ 10^{-3} Pa	Pressure for deposition/ 10^{-3} Pa	Temperature/ ℃
PTFE	1.5	1.5	35	5.1	1.4－1.8	35
Al_2O_3	1.5	3.2	110	5.1	1.4－1.8	35

In order to compare with the PTFE/ Al_2O_3 multi-layer, pure PTFE and pure Al_2O_3 films are also prepared using the same deposition parameters as listed in Table 1. The deposition time is 50 min for pure PTFE film and 95 min for Al_2O_3 film. The film thickness of pure PTFE and pure Al_2O_3 is almost the same as that of PTFE/ Al_2O_3 multi-layer.

3 RESULTS AND DISCUSSION

3.1 Structure analysis

The chemical bond and phase composition are determined by PHI-5300 X-ray photoelectron spectrometry and X-ray diffractometry. The C 1s spectrum of the PTFE film is shown in Fig. 1.

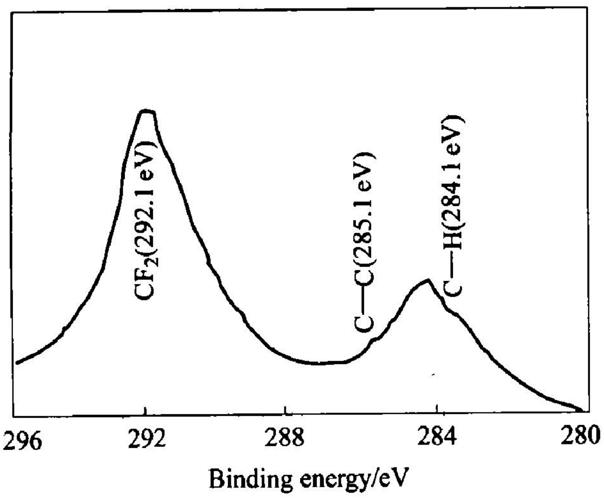

Fig. 1 C 1s spectrum of as-deposited PTFE film

It can be observed that the film is mainly composed of C- F bond ((292.1 ±0.1) eV). Meanwhile there are small amounts of C- C ((285.1 ±0.2) eV) and C- H ((284.2 ±0.2) eV) bonds. The results show that the chemical bonds of the deposited film coincide with those of the bulk PTFE.

The X-ray diffraction patterns[9] of the pure PTFE, pure Al_2O_3, PTFE/ Al_2O_3 multi-layer and PTFE/ Al_2O_3 multi-layer modified by ion implantation are shown in Fig. 2. In the X-ray diffraction pattern of PTFE/ Al_2O_3 multi-layer, there are not only pure PTFE's diffraction peak at $2\theta \approx 25°$ but also pure Al_2O_3 peak at $2\theta \approx 25°$. Apart from these two peaks, there are no other peaks in the multi-layer. PTFE component in pure PTFE film and PTFE/ Al_2O_3 multi-layer is in the crystalline state, but Al_2O_3 component in pure Al_2O_3 film and PTFE/ Al_2O_3 multi-layer appears to be in the diffused scattering pattern. This proves that the multi-layer consists of Al_2O_3 component and crystalline PTFE. The ESCA analysis shows that there are some Al and N compound in the outer layer PTFE of PTFE/ Al_2O_3 multi-layer modified by ion implantation. Therefore, in the interfaces of PTFE and Al_2O_3, the atomic intermingle[10-12] is involved by N^+ implantation, and adhesion is enhanced between the interfaces of PTFE and Al_2O_3.

3.2 Mechanical properties

Table 2 shows the film thickness, micro-hardness and toughness of pure PTFE, pure Al_2O_3, PTFE/ Al_2O_3 multi-layer and PTFE/ Al_2O_3 multi-layer modified by N^+ implantation. The thickness of PTFE/ Al_2O_3 multi-layer is almost the same as that of pure PTFE and pure Al_2O_3 films. The hardness of the PTFE film is very low, but its critical load is very high. The Al_2O_3 film have high hardness and low toughness different from PTFE film. Through assembling of PTFE and Al_2O_3 films, the PTFE/ Al_2O_3 multi-layer not only has high hardness but also has relatively high toughness, i. e. the comprehensive properties of multi-layer are superior to those of pure PTFE film or pure Al_2O_3 film. The PTFE/ Al_2O_3 multi-layer modified by ion implantation is superior to the others.

Table 2 Film thickness and mechanical properties of deposited film

Samples	Film thickness/ nm	Micro-hardness/ MPa	Critical load/ N
PTFE	750	60	> 0.98
Al_2O_3	750	2 128	0.127
PTFE/ Al_2O_3	770	1 437	0.441
PTFE/ Al_2O_3 assisted by ion implantation	770	1 856	0.343

A series of experimental results show that the strength and toughness of the PTFE/ Al_2O_3 multi-layer can be varied with the thickness and number of PTFE and Al_2O_3 films and the adhesion of N^+ implanted in PTFE layer of PTFE/ Al_2O_3 multi-layer.

3.3 Characteristics of micro-friction and wear

The observation of micro-tribological behavior is carried out in an atomic force and friction force

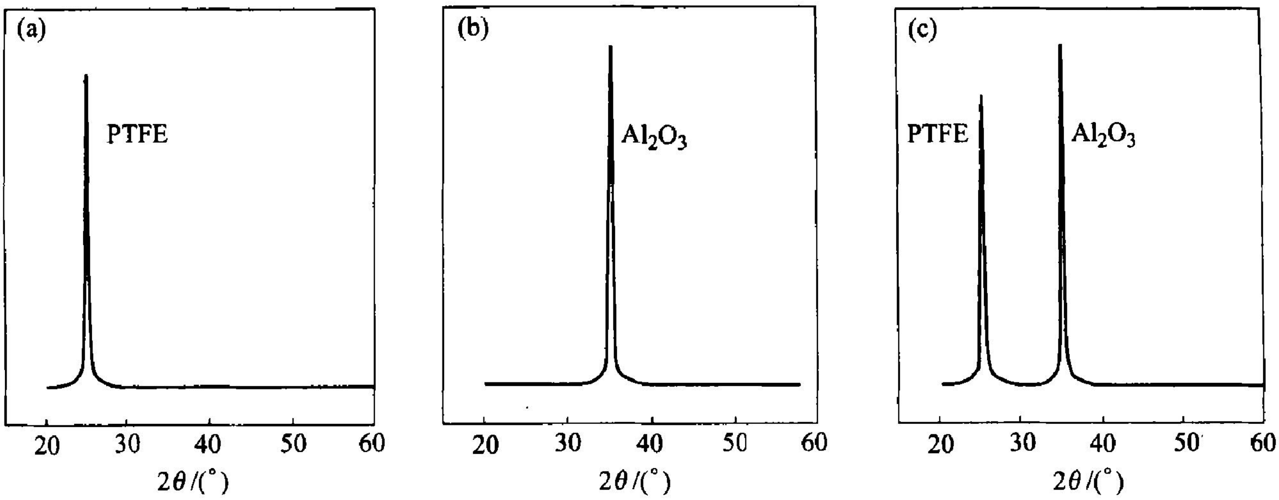

Fig. 2 X-ray diffraction patterns of PTFE, Al_2O_3 and PTFE/ Al_2O_3 film

microscope (AFM/FFM). Fig. 3 shows the dependence of micro-friction force signal of pure PTFE film, pure Al_2O_3 film, PTFE/ Al_2O_3 multi-layer and PTFE/ Al_2O_3 multi-layer modified by N^+ implanted on load. It can be observed that the micro-friction force signals of Al_2O_3 film and PTFE/ Al_2O_3 multi-layer are almost linear with load. For pure PTFE film, when the load is less than 70 nN, the micro-friction force signal increases linearly with the load. When the load is greater than 70 nN, the micro-friction force signal does not increase with the load, while remains about constant. When the load is above 70 nN, the surface morphologies of the pure PTFE film and PTFE/ Al_2O_3 multi-layer are scratched, but there are no worn marks on the surfaces of pure Al_2O_3 film and PTFE/ Al_2O_3 multi-layer modified by ion implantation. According to the friction theory[13] and the relationship between the friction force and friction force signal, the friction coefficient factor of pure PTFE film is the smallest (0.057), the second is the PTFE/ Al_2O_3 multi-layer modified by ion implantation (0.088), the third is PTFE/ Al_2O_3 multi-layer (0.108) and the largest is pure Al_2O_3 film (0.162).

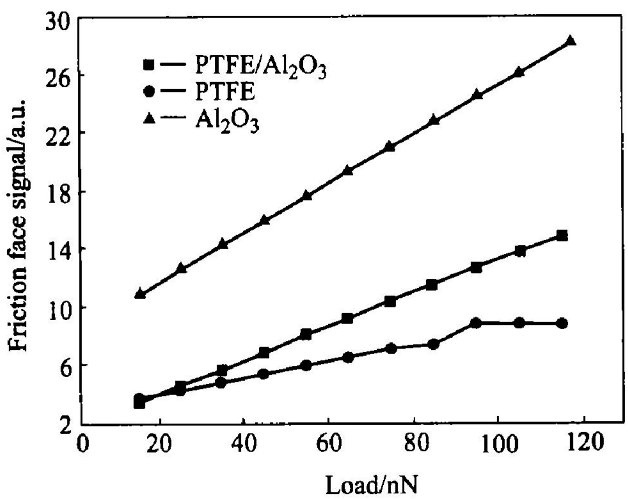

Fig. 3 Dependence of friction on load of PTFE, Al_2O_3 and PTFE/ Al_2O_3 film

When the load is greater than 70 nN, the worn mark is started to form on pure PTFE and PTFE/ Al_2O_3 films, but there is no obvious worn mark on pure Al_2O_3 film. The dependence of worn depth of PTFE and PTFE/ Al_2O_3 films on load is shown in Fig. 4. The worn depths of PTFE and PTFE/ Al_2O_3 film are also in nano-scale; the worn depth of PTFE/ Al_2O_3 multi-layer is about one tenth of that of PTFE film at the same load, and the worn depth of PTFE/ Al_2O_3 multi-layer modified by ion implantation is about one fifteenth of that of PTFE film at the same load. Although the wear resistance of PTFE/ Al_2O_3 multi-layer modified by ion implantation is less than that of Al_2O_3, it is much greater than that of PTFE film.

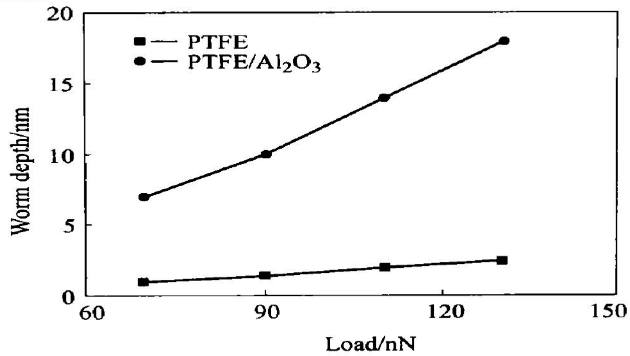

Fig. 4 Dependence of worn depth on load of PTFE and PTFE/ Al_2O_3 film

4 CONCLUSIONS

1) The deposition PTFE components of pure PTFE film and PTFE/ Al_2O_3 multi-layer are in crystalline state, and their structures are the same as that of bulk PTFE. In the friction and wear process, the pure PTFE film has high lubricity, low friction coefficient and low wear resistance.

2) The PTFE layer in PTFE/ Al_2O_3 multi-layer

is located between two Al_2O_3 layers. Because of the interaction between PTFE and Al_2O_3 layer, the multi-layer combines the superior characteristics of PTFE and Al_2O_3 films.

3) The multi-layer modified by ion implantation has high comprehensive properties, i. e. the combination of high hardness, high toughness, low friction coefficient and high wear resistance. So this surface modification technique for enhancing properties such as wear resistance and wear surface hardness is effective for elastic metallic-plastic pads.

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