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Fast pulsing plasma immersion ion implantation for tribological applications

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Abstract

Fast pulsing, low voltage plasma immersion ion implantation (FLPIII) is an effective surface modification technique for metals. Low voltage PIII features high ion current density and the ability to treat components possessing an irregular shape. In this process, a high temperature can be attained at an average to high ion flux enabling fast diffusion of the implanted species and the formation of a thick modified layer. An experimental investigation of FLPIII into AISI304 and mild steels is described. The resulting surface microhardness is much higher, and the friction coefficient as well as wear rate are dramatically reduced on account of the formation of new phases in the near surface. Our experimental results also demonstrate that FLPIII increases the critical load of the materials. Combining the non-line-of-sight advantage that enables efficient processing of samples with an irregular geometry, this method has large commercial potential. © 2000 Elsevier Science S.A. All rights reserved.

Keywords: Fast pulsing plasma immersion ion implantation; Tribology; Stainless steel

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1. Introduction

Low energy, high current density ion implantation at elevated temperatures has been shown to improve the tribological properties of various materials [1–5]. Compared to conventional low temperature, high voltage ion implantation, low energy (several keV) ion implantation at room temperature generates implanted layers only a few nanometers thick, but when this is done at an elevated temperature, significant diffusion of the implanted species can take place. As a result, low energy ion processing can be used to produce microstructures and tribological performance in metals rivaling those by high energy processing.

The major disadvantage of conventional low energy ion implantation is that it is a line-of-sight process. It is thus difficult to process multiple samples with complicated geometry simultaneously. We have developed a novel plasma-based surface modification method utilizing fast pulsing, low voltage plasma immersion ion implantation (FLPIII), also referred to as high frequency, low voltage plasma immersion ion implantation (HLPIII) [6]. In this paper, we report our FLPIII work on AISI304 and mild steels.

2. Experimental

The special modulator for fast pulsing, low voltage, and high current PIII has recently been developed in our laboratory [7]. A detailed description of our plasma immersion ion implanter and plasma sources can also be found elsewhere [8]. A thermocouple is attached to the back of the target to monitor the sample temperature directly during the implantation process [9]. In our process, the temperature was kept constant by adjusting the plasma density, pulse frequency, and/or pulse duration.

In order to facilitate the comparison of different

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experimental conditions, an argon plasma was used to preheat and clean the samples. The FLPIII process commenced after the nitrogen plasma was triggered by hot filament glow discharge. AISI304 stainless steel (SS) discs 30 mm in diameter and 5-mm thick and mild steel (MS) discs 25 mm in diameter and 5-mm thick were used in our experiments. Before the treatment, the specimens were mechanically polished to a roughness R_a of 0.02 μm. The sample temperature was kept at 400°C during PIII. The implantation time was 2.5 h and the implantation voltage was 1 kV.

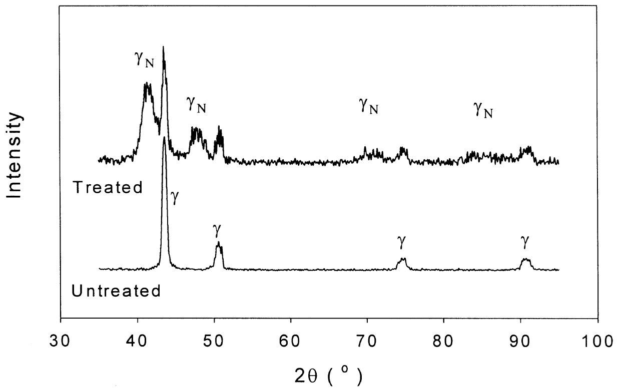

Fig. 1. X-Ray diffractograms of the AISI304 stainless steel samples.

3. Results

3.1. AISI304

After nitrogen PIII, the surface of the samples is quite rough, not only because of etching of the grain boundaries but also deformation of the grains due to the high N content [10]. The phase transformation in the near surface is responsible for the change of the sample appearance, as indicated by XRD results in Fig. 1. The broad peaks labeled γ_N at a smaller 2θ angle are associated with the nitrogen-rich expanded austenite phase. γ_N is a metastable phase consisting of supersaturated nitrogen in a solid solution. The relative intensity of the γ_N phase is similar to and sometimes greater than that of primary austenite indicating that γ_N is thick and abundant in the near surface layer.

The thickness of the modified layer is estimated by cross-sectional metallography. The scanning electron micrograph in Fig. 2 shows that the thickness is approximately 1.5 μm and the modified layer is very homogeneous and uniform in thickness. Its superior corrosion properties are also demonstrated by its greater resistance to etching.

The microhardness measurement results shown in Fig. 3 indicate a significant increase in the surface microhardness. The surface microhardness diminishes with increasing indentor load. However, a smooth decline of the curve means that the microhardness value is not very sensitive to the load and suggests the presence of a thick modified layer.

The pin-on-disc tests demonstrate that the tribological properties have been substantially improved. The AISI304 samples are wear-tested employing a load of 100 g with a rotation speed of 100 turns/min against a 6-mm diameter silicon nitride ball. As shown in Fig. 4, the friction coefficient is lower for the treated sample. The coefficient of friction is only 0.3–0.4 and much less than that of the untreated specimen. For the treated samples, the layer may have a critical load of more

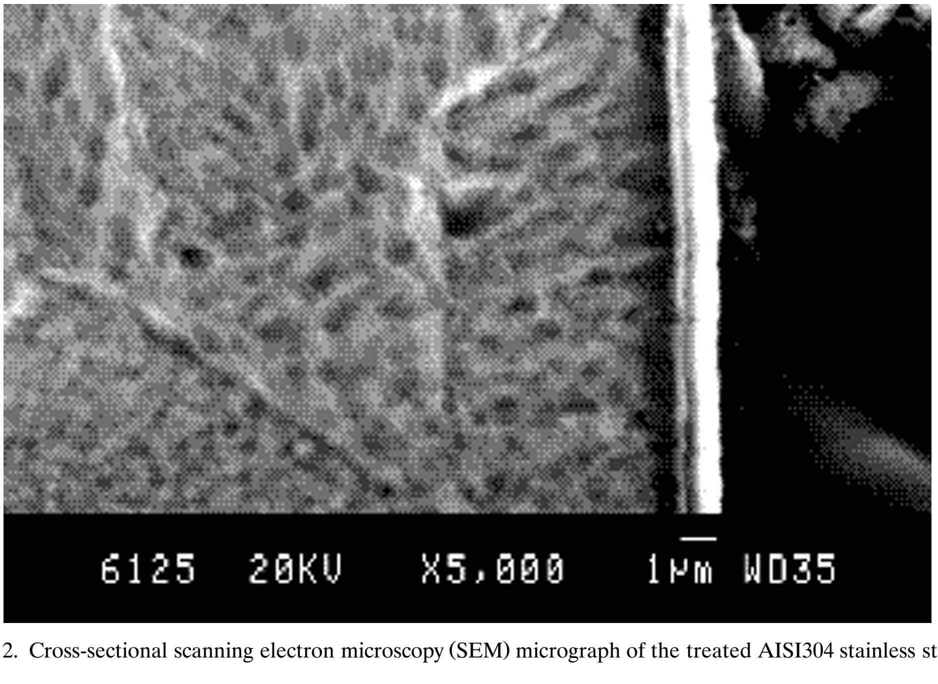

Fig. 2. Cross-sectional scanning electron microscopy (SEM) micrograph of the treated AISI304 stainless steel.

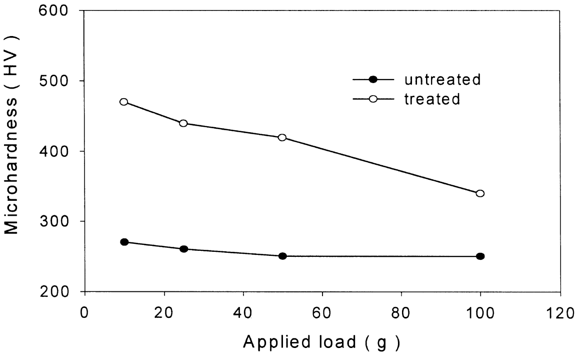

Fig. 3. Relationship of surface microhardness with indentation load for the treated AISI304 stainless steel.

than 100 g and the pin cannot penetrate into the modified layer. Hence, the wear track on the treated sample is small and not clear even under high magnification SEM (Fig. 5). In contrast, the untreated sample shows severe wear as well as adhesion, abrasion and plastic deformation in and around the wear track.

3.2. Mild steel

As illustrated in Fig. 6, the appearance of the mild steel is very different from that of the AISI304 samples indicating dependence on the materials. The surface of the mild steel is characterized by grain configuration and needle-like morphology. The original grain size of the sample is outlined by preferential precipitation of the nitrides. A higher magnification view shows that most of the precipitates are needle-like and the formation occurs along specific crystallographic directions of each grain. The size of the nitride needles is fairly uniform among the grains.

Compared to the AISI304 sample, more new phases are formed after the treatment. As shown in Fig. 7, new peaks appear besides the original peaks of the untreated sample. Strong α-Fe peaks from the matrix become weaker due to the formation of nitrides. The diffraction of the γ'-Fe_4N and ε-Fe_2N_{1-x} phases is strong. These phases produce the needle-like mor-

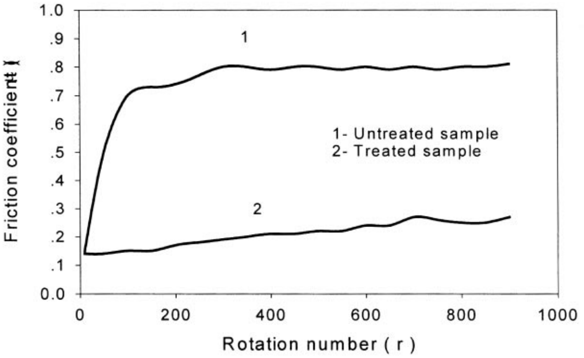

Fig. 4. Friction coefficients of the treated and untreated AISI304 samples vs. the number of rotations.

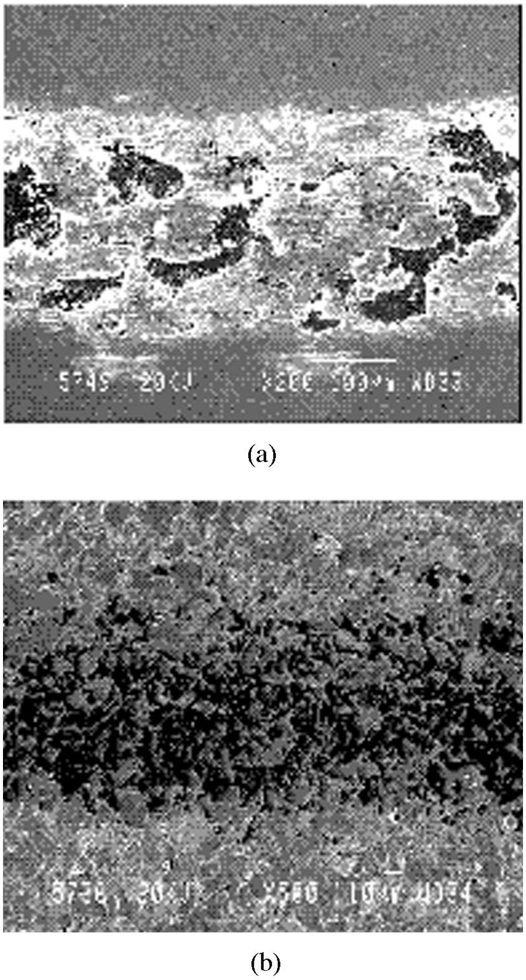

Fig. 5. SEM micrographs of the wear track after the pin-on-disc wear tests: (a) for untreated sample; (b) for treated sample.

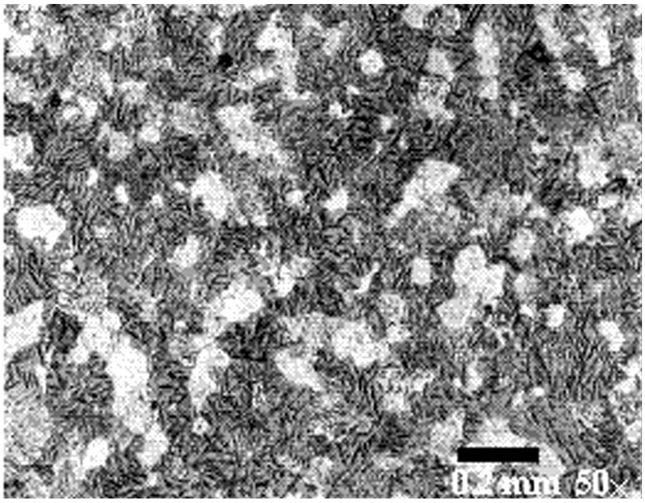

Fig. 6. Optical photography (50 ×) of the treated mild steel sample surface.

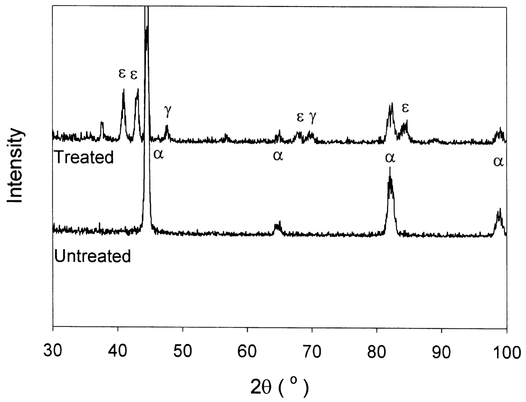

Fig. 7. X-Ray diffractograms of the treated (top) and untreated (bottom) mild steel samples.

phology illustrated in Fig. 6. In general, the needle-like γ'-Fe_4N dominates precipitation in the initial treatments, and then the ε-Fe_2N_{1-x} phases evolve right below the γ'-Fe_4N layer.

The microhardness of the treated sample is increased from HV126 (untreated) to HV170 showing an improvement of 34%. This can be attributed to the formation of the new phases in the near surface as confirmed by X-ray diffraction (XRD). The pin-on-disc tests are conducted using a load of 25 g and the pin can only grind against the modified layer owing to a small load leading to small friction coefficient for the treated/sample (Fig. 8). Although the friction coefficient increases gradually, the wear track is very blurry even until the end of the test since the critical load is speculated to be more than 25 g.

4. Discussion

Our results unequivocally show that the tribological properties of samples treated by our FLPIII process are improved significantly. The surface microhardness is increased, and the friction coefficient and wear rate are reduced. The experimental results indicate that synergistic effects of the harder phases and thicker modified layer are responsible for the considerable improvement in the surface tribological behavior. The thick modified layers generated by FLPIII stem from the high ion current density. High flux ion implantation improves the modification efficiency overcoming surface barriers and building up a high nitrogen concentration rapidly to facilitate dopant diffusion. Different mechanisms have been proposed [2,11–13].

FLPIII features a high ion flux and non-line-of-sight plasma immersion. Therefore, it is more effective for the treatment of irregularly-shaped components compared to the conventional low energy, high current ion beam techniques. Our results are similar to those obtained by Lei et al. with an ECR plasma source [14] and Mukherjee [15] who observed a significant increase in the surface microhardness. Our data are also consistent with those on conventional elevated temperature plasma immersion ion implantation [16,17]. To some extent, the energy of the ions is not the most decisive factor in determining the nitrided structure, and the total ion dose is more critical in determining the structure than the ion energy. Hence, there are technological reasons favoring the use of as low a voltage as possible. Although high energy ion bombardment may have same advantages in removing oxide layers which prevent nitrogen uptake as well as implanting nitrogen ions through any surface layers, a lower voltage gives rise to a thinner plasma sheath which is more advantageous in the processing of specimens possessing an irregular geometry. Consequently, FLPIII is more efficient and preferred in many tribological applications, particularly for large and odd-shape industrial components.

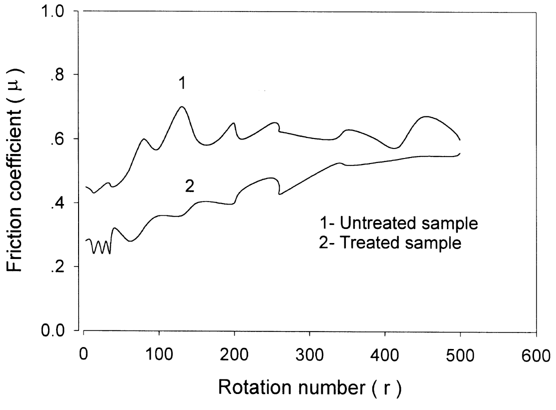

Fig. 8. Friction coefficient of the treated and untreated mild steel samples vs. the number of rotations.

5. Conclusion

Fast pulsing, low voltage plasma immersion ion implantation is an effective surface modification method and yields superior tribological properties such as surface microhardness, friction coefficient, and wear. XRD data indicate that the AISI304 sample surface has a uniform layer composed of expanded austenite, and iron nitride is rich in the mild steel sample surface. High flux ion implantation improves the modification efficiency overcoming surface barriers and building up a high nitrogen concentration rapidly to facilitate dopant diffusion. The method combines high current density ion implantation and non-line-of-sight plasma immersion enabling the treatment of samples that are large or have irregular geometries. It is noteworthy that because the critical load can be improved by this process, there is great industrial potential.

Acknowledgements

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