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The annealing behavior of H13 steel implanted with molybdenum ions

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Abstract

H13 steel was implanted with molybdenum to a dose of 5×10^{17} ions cm^{-2} to investigate the annealing effect on the wear resistance of the implanted steel. The molybdenum ion implantation was done using a metal vapor vacuum arc (MEVVA) ion source. The annealing effect was examined by Rutherford backscattering spectroscopy (RBS), high voltage electron microscopy (HVEM) and grazing-angle X-ray diffraction (XRD). The experimental results have showed that the wear resistance of the implanted steel was not changed significantly by thermal anneal though annealing has made the amorphous layer, which was induced by Mo ion implantation, re-crystallize. The phenomena observed in the experiment are interpreted and the annealing effect on the wear resistance of Mo-implanted surface is discussed by collision theory.

Keywords: Ion implantation; Annealing; HVEM

1. Introduction

Ion implantation is a kind of excellent technology for materials modification [1–3]. Since the development of the metal vapor vacuum arc (MEVVA) source in the late 1980s [4–6], some authors have reported Mo ion implantation could improve the wear resistance of some materials [7,8]. But the thermal stability of the implanted layer is always under suspicion. The present study is concerned with the annealing effect on the wear resistance of Mo-implanted H13 steel. For a well understanding of the annealing effect of the implanted steel, we need to choose a direct way of showing the microstructure evolution with increasing temperature. In situ observation in high voltage electron microscope (HVEM) is suitable, because we can examine a thicker film with it conveniently. Comparing to a normal transmission electron microscopy (TEM), much more information about the microstructure and phase formation in Mo-implanted layer can be obtained simultaneously.

2. Experiment and method

The experimental samples were made from H13 steel (0.36 wt.% C, 5.7 wt.% Cr, 1.6 wt.% Mo, 1.1 wt.% V, 0.8 wt.% Si and 0.2 wt.% Mn) in the size of 15 mm × 15 mm × 1 mm and 15 mm × 15 mm × 0.1 mm. All samples were polished to a mirror finish, heat-treated in a vacuum furnace and cleaned in an ultrasonic bath with acetone and ethanol. Molybdenum ions from a MEVVA source were implanted into H13 steel at a voltage of 48 kV to a dose of 5×10^{17} ions cm^{-2} because the wear resistance of Mo-implanted H13 steel could be improved at the implantation parameters [7]. The target temperature measured was about 220 °C. Some implanted samples were annealed in a vacuum furnace at 350 and 600 °C after ion implantation, respectively. The pin-on-disc apparatus with a pin was used for

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wear testing. The applied normal load of wear testing was 0.7 N. The wear tests were continued up to 20 passes. The maximum depth (D) and the mean width (W) of wear tracks for getting S ($=D \times W/2$) were measured by an optical interference microscope. E ($=S_{\text{unimplanted}}/S_{\text{implanted}}$) was used for evaluating the wear resistance of the implanted sample relative to the unimplanted one (E is equal to 1 for the unimplanted one). The surface compositions of implanted layers were measured by Rutherford backscattering spectrometry (RBS). For the RBS measurement, 2.0 MeV alpha particles were incident on the samples normally. The backscattering ions were detected at the angle of 165°. For HVEM, the samples of 0.1 mm thickness were mechanically polished to about 0.01 mm thickness on the reverse side of the implanted surface. The implanted side was protected from etching with a kind of special glue during the following electro-polishing. The XRD was carried out on a Rigaku D/max-RB diffractometer, with the grazing incidence geometry, using Cu Kα radiation. The incident beam was 5° with respect to the sample surface.

3. Experimental results

3.1. Wear resistance measurement

For wear testing, the interferograms of the wear tracks were obtained by an optical interference microscope. The average value of E was 2.5 for the implanted and un-annealed sample. The annealing of Mo-implanted sample made its wear resistance deteriorate slightly with increasing annealing-temperature. The average values of E were 2.3 for the Mo-implanted one of annealing at 350 °C, 1.7 for that of annealing at 600 °C. According to the relative interferograms, the maximum depth (D) of wear tracks was about 0.5 μm for the implanted and un-annealed sample. At the same time, the molybdenum ion implantation significantly reduced the friction coefficient of H13 steel. The friction coefficients for the unimplanted sample increased rapidly to 0.7 during the first 40 passes, whereas a very low friction coefficient was maintained for the implanted and un-annealed one in entire 350 passes. The friction coefficients for the Mo-implanted one of annealing at 350 °C showed a big jump comparing with that of the implanted and un-annealed one. This jump increased with increasing annealing-temperature. In a word, the annealing of the Mo-implanted H13 steel increased its friction coefficient and deteriorated slightly the wear resistance of the implanted sample according to Figs. 1 and 2.

3.2. HVEM results

The microstructure of the unimplanted sample as shown by HVEM was a kind of martensite structure, and the corresponding electron diffraction pattern was made up of α-Fe diffraction spots due to the big crystal grains of H13 steel (Fig. 3a). A layer with high-density damage appeared on the HVEM micrograph instead of the martensite structure when H13 steel was implanted with Mo ions. The widening diffraction rings should correspond to forming of an amorphous layer. In addition, there were some weak rings corresponding to Mo_2C on the electron diffraction pattern (Fig. 3b). Annealing at 350 °C only made the micrograph of Mo-implanted H13 steel change slightly (Fig. 3c), whereas the implanted layer re-crystallized and some precipitates like needle appeared at 600 °C annealing (Fig. 3d). The corresponding diffraction pattern was made up of un-continue rings and spots, which corresponded to α-Fe and Mo_2C, respectively.

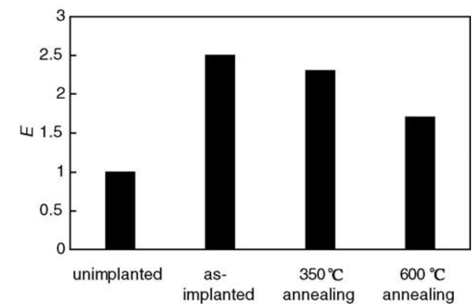

Fig. 1. The wear resistance of implanted and unimplanted H13 steel.

3.3. XRD analysis

The XRD spectra from variously treated samples were shown in Figs. 4–7. Strong diffraction peaks belonging to α-Fe(1 1 0) and α-Fe(2 0 0) were observed in all implanted and unimplanted samples. It was shown that some new peaks, which belong to compound Mo_2C, were detected apart from the peaks belong to α-Fe(1 1 0) and α-Fe(2 0 0) after Mo ion implantation (Fig. 5) though the intensities of the diffraction peaks were much lower than that of the Fe(1 1 0) peak. The XRD of the steel was similar to that of the un-annealed one after it was implanted and annealed at 350 °C. The peak strength of the XRD corresponding to Mo_2C obviously increased when annealing-temperature increased to 600 °C. The Miller indexes corresponding to the diffraction peaks

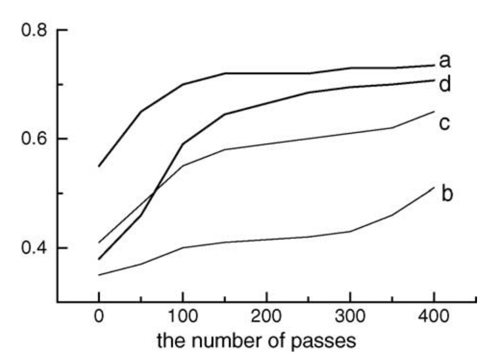

Fig. 2. Friction coefficient vs. the number of passes: (a) unimplanted; (b) as-implanted; (c) implanted + 350 °C annealing; (d) implanted + 600 °C annealing.