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Characterization of effects and model of voids induced by high current density tungsten ion implantation

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Abstract

Tungsten ions of low energy and high current densities were implanted into H13 steel to investigate the void effects of heavy ion implantation. The voids induced by ion implantation were examined by high voltage electron microscopy (HVEM). The abnormal tungsten concentration depth profile was characterized by Rutherford backscattering spectroscopy (RBS). Experimental results of wear and hardness indicated that the formation of voids had a great influence on the surface mechanical properties of H13 steel. A new spikes model can be used to qualitatively explain the voids formation and their effects.

Keywords: Heavy ion implantation; Void; Spikes; Wear; RBS; 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, some authors have reported MEVVA ion implantation could improve the wear resistance of some materials [4–6]. The higher an ion current density is, the shorter implantation time is for reaching a certain implantation dose. However, it is not too clear if the high ion current density can induce new implantation effects. In this paper, we have investigated the surface mechanical properties, composition and microstructure of W-implanted H13 steel using a high ion current densities. A model based on spikes was used to explain the relationship among the surface mechanical properties, composition and microstructure.

2. Experiments and methods

Commercially H13 steel was used in this study. All of the samples were polished to a metallgraphic finish and quenched at 1050 °C for an hour and then annealed at 500 °C for an hour and cleaned in an ultrasonic bath with acetone and ethanol, respectively. A MEVVA source can produce multi-charge state ions. The percentages of the tungsten ions with 1 to 5 elementary charges measured by a time of flight (TOF) were 6%, 40%, 36%, 13% and 5%, respectively, when the ion implantation system had no analysis magnet. The tungsten ions were implanted into an H13 steel at an acceleration voltage of 30 kV to a dose of 1×10^{17} cm^{-2}, corresponding to an average ion energy of about 81 keV since the average charge state of tungsten ions measured was about 2.7. The chosen pulsed current density of W ion was between 0.3 and 6 $mA\cdot cm^{-2}$. During the implantation, the samples were fixed on a water-jacketed iron plate in a vacuum chamber to keep their temperature below 200 °C. The hardness was measured by use of a Vickers hardness sclerometer with a load of 2.5 g. Five indentations were made on each sample and the data were averaged. A pin-on-disc apparatus with a pin was used for wear testing at a velocity of 16 $mm\cdot s^{-1}$. The applied normal load of wear testing was 20 g. The wear tests were continued to 800 circles. The average maximal depth (D), the average width (W) and S ($=D\times W/2$) of wear tracks were measured by an optical interference microscopy. Since all the experiment conditions were the same for the unimplanted steel and implanted steel, the S of wear tracks were directly proportional to the wear rate. Specimens for the transmission electron microscopy

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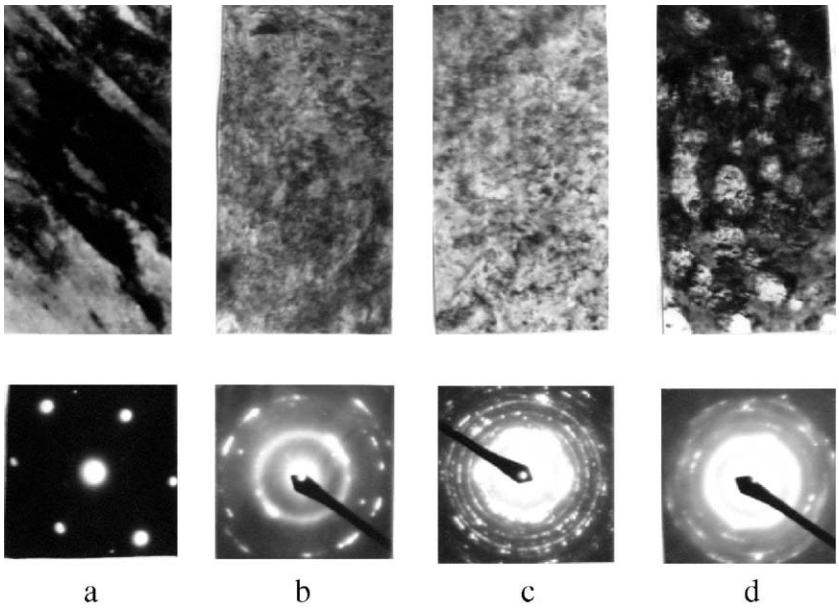

Fig. 1. HVEM images and corresponding diffraction patterns of implanted and unimplanted H13 steel a—unimplanted, b—0.3 mA·cm^{-2}, c—2 mA·cm^{-2}, d—6 mA·cm^{-2}.

(TEM) were prepared by ion milling from the unimplanted side. All of the observations were carried out in a HVEM operating at 1 MV. The phase structure of the implanted specimen was also examined by X-ray photoelectron spectroscopy (XPS). The XPS was taken with XSAM800 using AlK$_\alpha$ radiation at an experimental resolution of 1.0 eV. The electron spectrum was calibrated against carbon in hydrocarbon layers. The XPS of the surface was measured after it was etched by Ar ions in order to remove the contaminative layer of hydrocarbon. The surface compositions of implanted layers were measured by a Rutherford backscattering spectrum (RBS). For the RBS measurement, 2.0 MeV alpha particles were normally incident on the samples, with the backscattering ions being detected at an angle of 165°. The RBS results were compared with that calculated by a dynamic TRIM called as TRIDYN.

3. Experimental results

3.1. HVEM and XPS results

The microstructure of the unimplanted sample as shown by HVEM was a martensite structure, and the corresponding electron diffraction pattern was made up of α-Fe diffraction spots due to big crystal grains of H13 steel (Fig. 1a). A high dense damage layer appeared on the HVEM micrograph instead of the martensite structure after H13 steel was implanted at a pulsed ion current density of 0.3 mA·cm^{-2}, whereas corresponding electron diffraction pattern showed widening diffraction rings corresponding to α-Fe (Fig. 1b). In addition, there were some dotted weak rings corresponding to WC on the electron diffraction pattern, which implied that some WC grains were embedded in the high dense damage layer. Except for a similar intensity of dotted rings corresponding to WC, the HVEM micrograph was quite uniform and the electron diffraction pattern corresponding to α-Fe was similar to that of an amorphous structure at an ion current density of 2 mA·cm^{-2} (Fig. 1c). When the pulsed current density increased to 6 mA·cm^{-2}, some voids at a diameter of about 30 nm appeared on the HVEM micrograph and the corresponding electron diffraction pattern showed widening diffraction halo-rings corresponding to α-Fe, which implied that there were complete amorphous zones surrounding voids. The voids cannot be induced by the sample milling due to having used HVEM to observe the thick foil. The phase structure of implanted steel was also examined by XPS (Fig. 2). The W4s electron spectrum from implanted H13 steel was made up of two peaks, as shown in Fig. 2. They corresponded to W4f$_{7/2}$ and W4f$_{5/2}$ of WC, respectively, according to their binding energy [7]. This XPS result was consistent with that of HVEM.

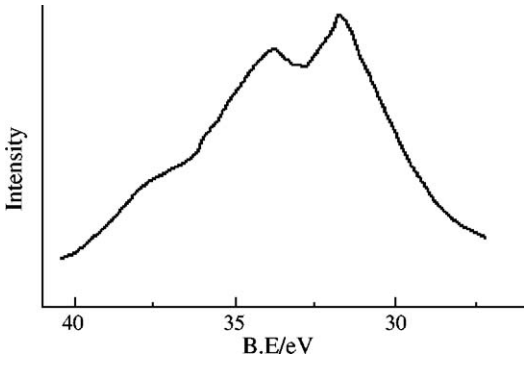

Fig. 2. W4f electron spectrum from an implanted steel.

3.2. Wear resistance measurement

For wear testing, wear track morphologies of implanted and unimplanted H13 steel were determined by an optical interference

Table 1
The values S and hardness of implanted and unimplanted H13 steel

Sample	Ion current density (mA·cm^{-2})	Hardness (HV)	S (× 10^{-4} mm^2)	Target temperature (°C)
a		550	3.8	
b	0.3	750	1.6	150
c	2	800	1.8	160
d	6	1150	4.1	180