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Effects of single- and dual-element ion implantation on tribomechanical properties of Cronidur 30 bearing steel

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ABSTRACT

This paper presents the results of a study on the effects of Ti ion and Ti + Cr ion implantation on the hardness and wear resistance of Cronidur 30 bearing steel. The mechanical properties of the samples implanted and nonimplanted were investigated by nanoindentation, friction and wear tests; The microstructures and phase transformations were also studied by grazing incidence X-ray diffraction (GIXRD) analysis, X-ray photoelectron spectroscopy (XPS), and transmission electron microscopy (TEM). The surface nanohardness improved significantly, changing from 11 GPa (non-implanted substrate) to 13 GPa (Ti ion sample) and 14 GPa (Ti + Cr ion sample). The increased wear resistance of Cronidur 30 after Ti + Cr ion implantation was twice that after Ti ion implantation and more than 10 times higher than that of the untreated sample. The TEM analysis confirmed the presence of amorphous and nanocrystalline phases in the samples. The XPS and GIXRD analyses confirmed new ceramic solutes. The tribomechanical improvement induced by implantation is attributed to the substructure strengthening and reinforcement of ceramic solutes.

1. Introduction

With the development of aviation engine technology, the working conditions for the bearings used in the main shaft bearings of aircraft engines are becoming increasingly complex, such that the bearings are subjected to severe corrosion, high loads, and elevated working temperatures [1,2]. Improvements in the performance of engine components require the development of high-temperature bearing alloys that exhibit high surface hardness for good wear resistance while maintaining a core with good fracture toughness, high ductility, and high impact toughness. Cronidur 30 is a recent nitrogen alloyed martensitic stainless steel for rolling bearing and ball screw applications applied in the field of aviation and aerospace [3]. As reported [4], its corrosion resistance is 100 times that of AISI 440C and it has a 5 times improvement in bearing life over M50 steel. This steel uniquely differs from the commercially available bearing steels because of high contents of chromium and nitrogen [5]. However, a high nitrogen content is unsuitable as it readily leads to segregation and precipitation, which cause defects during the forming process [6].

Based on consideration of further improvement on mechanical properties of Cronidur 30, surface modification techniques are feasible and can be used for the nitrogen bearing steel. For example, borides of transition metals as new coatings are relatively new promising materials for tribological applications owing to their high hardness, low friction, and chemical inertness [7]. However, the applicability of these coatings is limited owing to their high contact stress and adhesive problems [8]. The ion implantation method of surface modification is free from these adhesive or stress problems due to its merits below [9,10]. It is known that the treatment of bearing steel with a high-energy ion beam can considerably modify the properties of the subsurface layer of the steel. The formation of a hardened thin layer by ion irradiation does not result in sharp boundaries on the substrate. Further, it does not lead to changes in the basic dimensions or roughness of the substrate. Thus, this treatment can be used with bearings in the cases where the performance of conventional wear-resistance coatings is less than desirable [11,12].

The effectiveness of ion implantation method depends mainly on the ion implantation regime and the composition of the substrate being treated. Further, it was reported that, in many cases, the irradiation of a material with two or more distinct types of ions (for example, gas and metal ions) brings about more significant changes in the material properties [13–15]. Results from previous work [16] demonstrated further improvement on dual implantation, like hardness, wear resistance and corrosion resistance, in comparison with single implantation. However, there are still some ambiguities remained, particularly the effect on the modification of tribological properties [17,18]. Also, it is noted that the improvement depends on the kinds of elements and base metals [12]. In the previous study [19], researchers implanted

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Zr + N ions into M50NiL substrate and the implanted sample exhibited higher surface hardness, anti-friction and wear-resistance properties and resistance to hot contact fatigue. However, the strengthening effect is restricted by the single ceramic phase, ZrN. Moreover, the strengthening mechanism was also not discussed by comparison with single element. Also Cronidur 30 is different from M50NiL, especially the components and structures. As known, Cronidur 30 is high-nitrogen steels while there is no nitrogen in M50NiL. In addition, few researchers have reported the improvement of mechanic properties of Cronidur 30 with ion implantation. It is necessary to investigate whether pure metal ions implantation can induce ceramic phases and structural modification for mechanical improvement in this high-nitrogen steels.

Thus, to meet the demand for improving nanohardness and friction and wear resistance of Cronidur 30 bearing steel, the ion implantation is used to improve the hardness and tribological properties. Yet few papers present effects of the single or dual ion implantation on Cronidur 30 steel. Thus, Cr + Ti dual implantation is applied in this study and, by comparison with single Ti implantation, the changes in the structures, mechanical properties and tribological performance of dual implantation were investigated. Finally, the strengthening mechanism is further investigated.

2. Materials and methods

2.1. Materials

Cronidur 30 bearing stainless steel (0.33 wt% C, 0.8 wt% Si, 0.41 wt % Mn, 15.6 wt% Cr, 0.25 wt% Ni, and 0.93 wt% Mo) was cut into disc-like samples, Φ 5 mm × 10 mm. Before the ion implantation process, these discs were firstly polished with a series of WC abrasive papers with grits of 600# to 2000# and then polished with W2.5 diamond grit paste, and finally cleaned ultrasonically with acetone and ethanol for 10 min.

Ion implantation was carried out using a metal evaporation vacuum arc (MEVVA) ion source for the implantation of transition metal elements. The purity substrate is noted as P0. And samples implanted with Ti and Ti + Cr ions are noted as P1 and P2, respectively. The implantation parameters are listed in Table 1.

2.2. Surface characterization

To elucidate the structure of the implantation zone at different depths, the cross-sections of non-implanted and implanted samples were examined using high-resolution transmission electron microscopy (HRTEM) technique on JEOL JEM 2010F equipment with an energy-dispersive X-ray spectroscopy (EDS) attachment (Oxford Instruments). Cross-sectional specimens were prepared using a focused ion beam system (TESCAN LYRA 3 FEG-SEM/FIB). The samples were trimmed to dimensions of 10 μm × 5 μm using a gallium ion sputtering gun with various beam energy of 30∼ 50 keV, followed by fine milling for HRTEM by sputtering at a series of lower energy levels. To protect the sample surface, a thin platinum (Pt) film was deposited.

X-ray photoelectron spectroscopy (XPS) was performed using a Quantera SXM (PHI) instrument with a monochromatic Al X-ray source (energy = 55 eV). The incident angle of the X-rays was 45°. To analyze the elemental distribution of the samples with depths, a sputtering argon ion gun was used for etching; accelerating voltage was 4 kV; the etching rate for a standard SiO_2/Si sample was 5 nm/min.

To analyze the phase compositions of the samples, grazing incidence X-ray diffraction (GIXRD) analysis was performed using a D-MAX-2500 (Rigaku) system with a Cu-Kα1 (k = 0.154 nm) radiation source. The angle between the sample surface and the incident X-rays was set to 0.5 degree, and the detector scanned from 10 degree to 90 degree at 3 degree/s. The data were compared with the corresponding ICDD-JCPDS standard cards.

Table 1
Ion implantation parameters for different samples.

Sample	P0	P1	P2
Base pressure (Pa)	-	3.0×10^{-4}	3.0×10^{-4}
Sample temperature (°C) in ion implantation process	-	18–40	18–40
Energy (keV)	-	88	88
Total implantation dose ($\times 10^{17}$ ions/cm^2)	-	2	2
Element	-	Ti	Ti + Cr
Process time (number of cycles)	-	1	2

2.3. Mechanical properties

The nanohardness was tested by using an Agilent G200 nanoindentor in accordance with the literature [20]. The nanohardness of each sample was tested at depths ranging from 50 nm to approximately 500 nm using different loads (5-50 mN). The indented speed of the indenter was adopted as 10 nm/s and the transverse strain rate was limited to 0.05 s^{-1}.

The tribological properties of the samples were investigated using a reciprocating friction-and-wear tester (CETR-UMT-5, Bruker). The applied load was 2 N, and the frequency was 5 Hz at a stroke of 3 mm. A Si_3N_4 ball with a diameter of 4 mm was used as the counterpart. All the measurements were performed at least three times at room temperature (23 °C) and a relative humidity of 20%∼ 30%. The wear volumes were tested by a three-dimensional white-light interfering profilometer (NeXView, ZYGOLamda) and the average values with error bars were provided in the paper.

3. Results

3.1. Surface structure and composition evaluation

3.1.1. HRTEM analysis

Fig. 1 shows the cross-sectional HRTEM images of Cronidur 30 (P0), Ti implanted sample (P1) and Ti + Cr dual implanted sample (P2). The lattice boundaries of body-centered cubic (bcc) martensite can be seen arranged in an orderly manner in the image of the substrate (P0), indicating that it had a near-perfect crystalline structure. The Pt protective layer consists of an amorphous phase. Nevertheless, an amorphous sublayer can be seen at a depth of approximately 15–20 nm between the crystalline structure of the substrate and the amorphous Pt layer in the case of the ion-implanted samples P1 and P2. As shown in Fig. 1(c), after P0 was implanted with Ti ions, the lath martensite grains gradually became finer and twins grew in the implantation zone [21,22]. The HRTEM image of the refined substrate layer (Fig. 1(d)) shows lattice fringes that are indicative of the presence of defects. In addition, in the amorphous layer, a mixed zone consisting of a nanocrystalline phase and an amorphous phase, wherein the crystal planes are disordered and the degree of atomic arrangement is low, is also present. The nanocrystalline or amorphous phase is in keeping with the broadened halo seen in the selected area electron diffraction pattern. Compared to P1, sample P2, which was subjected to dual-element ion implantation, exhibits a more refined but graded structure, with a greater number of twins being present in the implantation zone (Fig. 1(e)). The HRTEM image of the interface shows that an amorphous layer is formed by nanocrystalline/amorphous phases, as evidenced by the presence of several broadened halos (Fig. 1(f)).

It can be seen that a sharp and plane interface is formed between the substrate and the Pt film in the case of P0 sample (see yellow oval loop in Fig. 1(b)). However, the “structural interface” between the substrate and the amorphous layer is not very smooth in samples P1 and P2; in these samples, the interface is wavy and concavo-convex, owing to the bonding of the two layers (see yellow oval loop in Fig. 1(d)). The elemental distribution maps of the cross-sections of the samples were also