



Microstructure and vacuum tribological properties of 1Cr18Ni9Ti steel with combined surface treatments

WangHai-dou^{*}, MaGuo-zheng, XuBin-shi, SiHong-juan, YangDa-xiang

National Key Laboratory for Remanufacturing, Academy of Armored Forces Engineering, Beijing 100072, China

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ABSTRACT

A sulfide film was fabricated on the nanocrystalline layer of 1Cr18Ni9Ti stainless steel by a two-step method of supersonic fine particles bombarding (SFPB) and low temperature ion sulfurization treatments. The microstructure and mechanical properties of the nanocrystallized surface and the sulfide film were characterized by X-ray diffraction, transmission electron microscopy, scanning electron microscopy (equipped with EDS), auger energy spectroscopy, X-ray photoelectron spectroscopy, and nano-indenter. The tribological behaviour of the treated (after SFPB and sulfurizing treatments) 1Cr18Ni9Ti steel in vacuum was investigated on a ball-on-disk tribometer. The results showed that, randomly oriented equiaxial nanograins with the mean grain size less than 30 nm were formed in the top surface layer of the SFPB treated sample, and a compact and uniform sulfide film mainly composed of FeS was obtained after the succedent sulfurizing treatment. Compared to the original 1Cr18Ni9Ti steel, the treated surface revealed lower friction coefficient and better wear resistance in vacuum, and the variation of tribological properties with atmospheric pressure of the treated samples was not significant. The dominant wear mechanisms of the treated 1Cr18Ni9Ti in vacuum were abrasive wear and fatigue wear.

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1. Introduction

Nanomaterials are being paid much attention for its excellent physical and chemical properties. According to the current technical level, it is difficult to prepare three-dimensional bulk nanomaterials with no defects, no interface pollution and various shapes at low cost [1]. Considering most of the failures (corrosion, wear, fatigue, etc.) of engineering equipments and materials starting from local surface, if a nanocrystalline surface layer can be introduced to the structural materials' surface, that is surface nanocrystallization, the overall performance of the block materials can also be improved by the excellent properties of the nano-structure layer [2]. Surface self-nanocrystallization is a new nano-technology based on conventional surface modification technique, to improve the materials' overall service performance via the optimization of surface microstructure and properties. The nanocrystallized surface layer of metal material not only has excellent mechanical and tribological properties, its high chemical activity and high density of crystal defects are also helpful for the traditional surface chemical heat treatments greatly [3].

Low temperature ion sulfurization process is an effective method to meliorate the friction between the moving steel parts, which is widely used in tools, dies, gears, cams and other friction parts [4]. The existing research about the effects of surface nanocrystallization and

sulfurization treatment on tribological properties of materials was mainly tested in aerosphere environment at room temperature [5–7], while the documentations about the tribological properties of nanocrystallized surface layer and sulfide film in vacuum are rare.

In the present work, the 1Cr18Ni9Ti sample was surface nanocrystallized by supersonic fine particles bombarding (SFPB) process, and followed by the low temperature ion sulfurization treatment to form FeS solid lubrication film. The vacuum tribological properties of the treated (after SFPB and sulfurizing treatments) 1Cr18Ni9Ti steel were investigated. The research results would provide a novel surface modification method for the friction pairs working in space vacuum environment.

2. Experimental details

The material of the sample used in this work is the hot rolled 1Cr18Ni9Ti stainless steel with the chemical composition (in wt.%) of 0.1C, 1Si, 1Mn, 0.003S, 0.035P, 17.5Cr, 8.5Ni, 0.40Ti and balance Fe. The samples were machined to circular disks with the dimension of ϕ 50 mm $\times$ 8 mm. The surface roughness of the original samples is Ra0.08 μ m.

The SFPB technology was employed to obtain a surface nanocrystalline layer on 1Cr18Ni9Ti steel sample. The equipment and process of SFPB are similar to the blasting shot technology and can be found in Ref. [8]. In the present work, the samples were bombarded by Al₂O₃ particles (20–30 μ m in diameter) at 50 °C for 6 min, the pressure of airflow was about 1.5 MPa, and the distance and angle of

^{*} Corresponding author. Present address: National Key Laboratory for Remanufacturing, Beijing 100072, China. Tel.: +86 10 66718541; fax: +86 10 66717144.

E-mail address: wanghaidou@tsinghua.org.cn (H. Wang).

bombardments were 15 mm and 90°, respectively. The surface of the SFPBed (SFPB treated) sample was re-polished to the roughness of the original sample. The following sulfurizing treatment for SFPBed sample was carried out at 200 °C for 1.5 h, and the technology of low-temperature ion sulfurization is illustrated in detail in Ref. [9].

The sliding friction and wear tests were performed on the YTI-1000 type ball-on-disk vacuum tribometer (Ukraine). Its schematic diagram is shown in Fig. 1. In this experiment, the upper sample was a 440 C steel ball with a diameter of 9.525 mm, hardness of HRC58 and surface roughness of Ra0.032 μm . The lower samples were the cylindrical 1Cr18Ni9Ti disks. In operation, the upper ball sample was fixed, and the lower disk rotated. The friction forces were measured by the dynamometer and then converted into friction coefficients. A Sartorius MC210S type analytic balance (with an accuracy of 0.1 mg) was utilized to weigh the lower samples before and after the wearing tests to obtain their respective wear losses.

The tribology tests were performed in air, low vacuum (1 Pa) and high vacuum (1×10^{-5} Pa), respectively. A normal load of 4 N, a sliding velocity of 0.2 m/s and a sliding time of 20 min were chosen for each test.

The microstructure of the surface nanocrystalline layer on the SFPBed sample was characterized by an H-800 transmission electron microscopy (TEM) operated at 150 kV. The thin foil samples for TEM observations were cut from the SFPBed surface layer by electro spark discharge technique, and then were thinned by ion-beam milling at low temperature.

A scanning electron microscope (SEM) equipped with energy dispersion spectrum (EDS) was employed to observe the morphologies of sulfide films and wearing tracks. The cross-sectional profile and 3D pattern of the typical wear grooves were observed using a VK-9710 laser scanning microscope. A Rigaku D/MAX 2400 type X-ray diffraction (XRD) was adopted to analyse the phase constituents of the sulfurized layer. The distribution of elements along the depth of the treated sample was measured by augur energy spectroscopy (AES). The chemical valence and composition of the sulfide films and wear scars were analysed by X-ray photoelectron spectroscopy (XPS). The surface hardness of the original and SFPBed sample were measured by a MML-Nano Test 600 type nano indenter.

3. Results and discussion

3.1. Microstructure and mechanical properties

The dark-field TEM images and the corresponding selected area electron diffraction (SAED) patterns of the top surface layer and of the layers about 10 μm and 20 μm in depth on the SFPBed sample are shown in Fig. 2. It can be seen that the microstructure of the SFPBed top surface layer consists of a uniform distribution of nano-sized

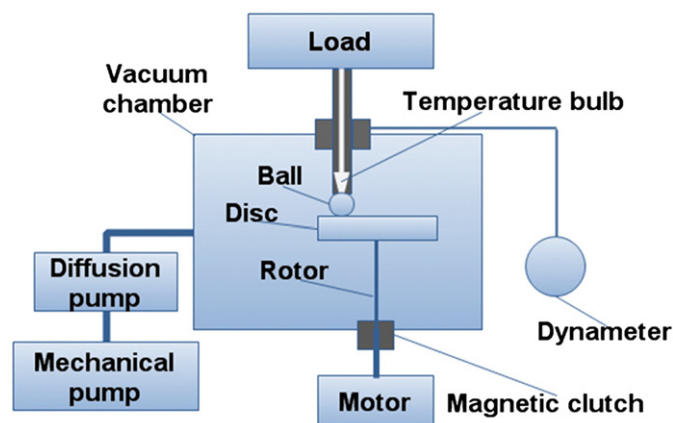


Fig. 1. Schematic diagram of the vacuum tribometer.

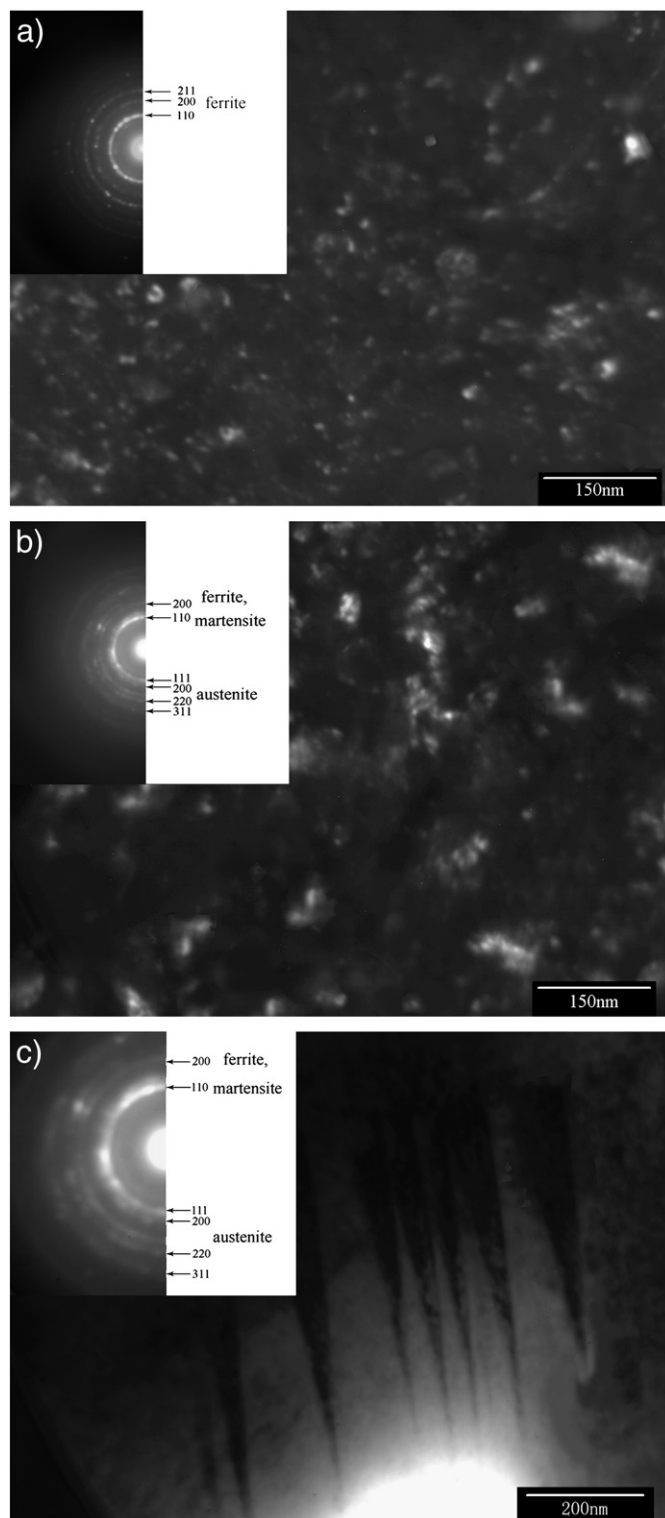


Fig. 2. Dark field TEM images and the corresponding selected area electron diffraction (SAED) patterns of the surface layers on the SFPBed sample: (a) top surface; (b) 10 μm in depth; (c) 20 μm in depth.

equiaxial ferrite grains with random crystallographic orientations, as indicated by Fig. 2(a). From the image and SAED pattern of the top surface layer, the grain size was estimated to be 30 nm, and all the diffraction rings corresponded to ferrite phase.

As can be seen from Fig. 2(b), at the depth of 10 μm from the SFPBed surface, the grain size is still in the nanometer range, but the grain size increased significantly (about 80 nm) and the grain refinement is inconsistent and incomplete, so some large grains can

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