



Enhanced tribological performance of a gradient nanostructured interstitial-free steel

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ABSTRACT

A gradient nanostructured (GNS) surface layer was fabricated on a commercial interstitial-free (IF) steel by means of surface mechanical grinding treatment (SMGT). Reciprocating dry sliding tests of the GNS IF steel in air at room temperature were carried out in comparison with the coarse-grained (CG) sample. Worn surface morphologies, chemical compositions and worn subsurface microstructures were investigated for both IF steel samples. IF steel with a GNS surface layer exhibits lowered coefficients of friction (COFs) and significantly enhanced wear resistance under high testing loads. The superior tribological performance of the GNS IF steel sample is attributed to the finer dynamic recrystallized grains, and the grain coarsening layer that can accommodate large plastic strain and suppress the formation of cracking vortical structure.

1. Introduction

Due to the high strength/hardness of nanostructured (NS) materials [1], their tribological behaviors have attracted much attention in the past decades for potential industrial applications [2]. Many NS metals exhibit enhanced wear resistance [3–6] in sliding, abrasive as well as fretting wear in comparison with their coarse-grained (CG) counterparts, which is mostly attributed to their positive effect of high hardness on the wear resistance according to the classical Archard's law.

Failures of engineering materials, such as wear, corrosion and fatigue, usually take place on the surfaces, it is reasonable to enhance materials' performance by preparing a NS layer on the surface of materials. In recent years, various techniques were applied to synthesize NS surface layers on metals and alloys, including surface mechanical attrition treatment (SMAT), high-energy shot peening (HESP), supersonic fine particles bombardment (SFPB) and surface rolling (SR) [7–11]. And the tribological behaviors of NS surface layers on metals and alloys have also been frequently reported. Zhang et al. [7] reported that after SMAT, pure copper with a NS surface layer showed an enhanced wear resistance under dry sliding condition due to high hardness and load-bearing ability. But the coefficient of friction (COF) can only be lowered under low contacting loads and the COF reduction disappeared because of the wearing off of the NS surface layer with the increase of testing load. Li et al. [11] reported that medium carbon steel with a NS surface layer, generated by using HESP, exhibited almost the same COF and wear rate as that of the CG counterpart under high

loading condition, which was attributed to the fatigue fracture of hard NS surface layer. Similar results was also reported in 304 stainless steel [8].

Above all, it is often limited to enhance the tribological properties of materials by utilizing NS surface layers treated via above methods. The positive effect of grain refinement on the tribological performance seems not to be as great as expected, especially achieving a COF reduction. Actually, for many NS metals and alloys, reduced COFs were observed only at low loads compared with their CG counterparts and no obvious reduction in COFs was obtained under high loads [12,13]. However, a newly developed surface mechanical grinding treatment (SMGT) has been successfully applied to generate thick gradient nano-grained or nano-laminated surface layers on various metals, showing tremendous advantages such as ductility [14] and thermal stability [15] compared to other techniques. Especially, a Cu-Ag alloy [16] with a gradient nano-grained surface layer acquired by the SMGT processing showed a remarkably decreased COF compared to the CG sample, even under high loads. This novel low friction phenomenon of the gradient nanostructured Cu-Ag alloy attracted us to investigate of the tribological behaviors of other engineering materials with the gradient nanostructure.

Interstitial-free (IF) steels are ferritic steels with low contents of interstitial elements (carbon and nitrogen), and commonly used in the automobile industry. IF steels usually exhibit excellent deformability and high ductility, but low yield strength and poor wear resistance. Investigations on the tribological properties of IF steels are rather rare,

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Table 1
Chemical composition (wt.%) of the IF steel used in this work.

C	N	O	P	Si	S	Ti	Al	Ni	Cu	Mn	Fe
0.037	0.0062	0.0027	0.04	0.04	0.003	< 0.01	0.034	0.016	0.031	0.29	balanced

but improving the wear performance of IF steels are of significance for the automotive industry. In this work, the SMGT technique was applied to generate a gradient nanostructured (GNS) layer on the surface of a commercial IF steel, in order to explore the GNS effect on the friction and wear behaviors of IF steel under dry sliding. Worn surface morphology and worn subsurface microstructure were analyzed for discussing the involved wear mechanisms of the GNS IF steel.

2. Experimental

2.1. Sample preparation

A commercial IF steel rod (see Table 1 for detailed composition) used in this work was annealed at 1173 K for 10 h to obtain a fully recrystallized structure with an average grain size of 27 μm and a Vickers hardness of 1.02 GPa.

During the SMGT processing, a rod sample (with a diameter of 10 mm and a length of 100 mm) was processed at a rotating velocity of 360 rpm against a WC-Co tool tip with a radius of 4 mm. With a fixed indentation depth of 30 μm into the rotating sample, the tool tip moves from one end to another in one pass at a velocity of 10 mm/min. In order to induce large strain on the surface, this process was repeated for 7 passes with a gradually increased indentation depth (30 μm per pass). The treatment was performed at room temperature and cutting oil (ilolcut 481 CN, Castrol) was supplied during the treatment. To ensure the repeatability of wear tests, the SMGT samples were grinded off about 20 μm with emery papers and then mechanically polished to a mirror-like surface with a roughness of $R_a \sim 110$ nm before the wear tests and microstructure characterizations. The CG sample was electro-polished to gain a comparable surface roughness ($R_a \sim 80$ nm) to that of the SMGT sample.

2.2. Microstructure characterization and micro-hardness measurement

The cross-sectional microstructure of the SMGT sample was characterized by using a FEI 430 SEM. A nickel layer about 500 μm in thickness was electro-deposited on the surface to protect against possible damages. The coated sample was mechanically polished and then electro-polished in an electrolyte of 92% ethanol and 8% perchloric acid for 5 s to reveal the microstructure. Cross-sectional micro-hardness of the SMGT sample was measured on an automatic Vickers hardness tester (Qness Q10 A+) using an indentation load of 25 g and a loading duration of 10 s. A preset measuring array was applied to measure the micro-hardness in different depth.

2.3. Wear tests

Reciprocating dry sliding tests were conducted on an Optimol SRV-III tester with a ball-on-plate test module at room temperature in air with a relative humidity of 30%. WC-Co balls with a diameter of 10 mm and a micro-hardness of 17.5 GPa were used as the counterfaces. The friction and wear tests were carried out at a slide stroke of 1 mm, a frequency of 5 Hz, normal loads of 30–90 N and a duration of 300–18000 cycles. Sliding direction was along the rod axis of the SMGT IF steel sample. To compare the increments of wear volumes for the CG and the SMGT samples during sliding, different testing cycles were conducted under a load of 50 N.

To determine the wear volumes, profiles of the worn surfaces were measured by using a MicroXAM 3-dimensional (3D) surface

profilometer system. By adjusting the reference plane to the original level, the void volume below the reference plane was taken as the wear volume. Average roughness (R_a) along the sliding direction and 3D profiles of the worn surfaces were acquired with an Olympus LEXT 4000 confocal laser scanning microscope.

2.4. Worn surface morphology and composition analysis

Worn samples were cleaned ultrasonically with ethanol for 10 min to investigate the worn surface morphology with the FEI 430 SEM. Chemical compositions and chemical states of the products on the worn surfaces were analyzed by using X-ray photoelectron spectroscopy (XPS, Thermo VG, Escalab 250, a monochromatic Al $K\alpha$ X-ray source). Worn surfaces were sputtered by argon ion for 20 s to remove the contaminations. When processing data, all binding energies were referenced to the C 1s peak (284.8 eV) arising from adventitious carbon.

2.5. Worn subsurface microstructure characterization

To characterize the subsurface microstructure along the sliding direction, a nickel layer was electro-deposited to protect the worn surface. The coated samples were then electro-polished to reveal the microstructure. The worn subsurface microstructures were also characterized by using electron back-scattered diffraction (EBSD) technique with a HKL Technology Channel 5 system. The EBSD data was gathered under an acceleration voltage of 20 kV and a tilt angle of 70°. During data processing, the software (HKL Channel 5) cleaned unresolved pixels automatically with the standard noise-reduction procedure, and reconstructed all the grains with a misorientation angle (MA) of 15°. Then the software measured the internal average MA within the reconstructed grains. Grains with an average MA higher than 2° were defined as the deformed grains (Red). If the average MA in a grain was lower than 2°, but the misorientation angle between any sub-grains was higher than 2°, that grain was considered as the sub-grain (Yellow). The grains with an internal average MA below 2° were considered as the fully recrystallized grains (Blue).

3. Results and discussion

3.1. Microstructure and micro-hardness

Fig. 1a shows a cross-sectional SEM image of the SMGT IF steel sample. A gradient nanostructured surface layer was fabricated on the IF steel rod by the SMGT processing. In a depth span of 10 μm from the treated surface, the microstructure is refined to a nano-laminated structure with a mean lamellar thickness of 90 nm, as shown in Fig. 1b. This lamellar size agrees well with the lamellar thickness found in [17] in a depth of 30 μm due to the additional grinding procedure in this work. A relatively sharp and straight lamellar structure is generated due to severe shear deformation imposed by high speed sliding of the WC-Co tool tip. Generating the gradient distributions of strain and strain rate is effective for fabricating gradient nanostructures, as observed in the SMGT Ni [15] and medium carbon steel [18]. Due to the large strain and the high strain rate in the SMGT processing, lamellar structure with much finer size was formed on the surface of the IF steel relative to the traditional severe plastic deformation techniques [19]. With an increasing depth from the surface, the thickness of lamellar structure gradually increases (Fig. 1b-d). The average lamellar thickness is about 340 nm at a depth of 50 μm (Fig. 1c) and 530 nm at a depth of 100 μm

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