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Tribological response and microstructural evolution of nanostructured bainitic steel under repeated frictional sliding

Kritika Singh, Aparna Singh*

Department of Metallurgical Engineering and Materials Science, Indian Institute of Technology Bombay, Mumbai, MH 400076, India

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ABSTRACT

Nanostructured bainitic steels have tremendous potential to be used in structural applications e.g. as rail materials. Strength and toughness in nanostructured bainite can be easily tailored by controlling the lath thickness of bainitic ferrite plates (BF) along with the morphology and volume fraction of retained austenite (RA). However, the wear response under repeated sliding depends on the microstructural evolution with the number of passes and thus is difficult to predict without careful experiments. In the current study, we have made nanobainitic steels with a range of lath thicknesses from a steel of composition Fe-0.89C-1.59Si-1.65Mn-0.37Mo-1Co-0.56Al-0.19Cr (wt%). Austenitization followed by isothermal holding at 250 °C, 300 °C and 350 °C has been used to make specimens with average bainitic lath thicknesses of about 45, 54 and 89 nm respectively. Depth sensing instrumented indenter with a conical tip has been used to make multiple scratches on all the specimens and the damage has been characterized in the form of depth of scratch, scratch thickness and groove volume using a combination of profilometer and scanning electron microscopy. Specimens transformed at lower isothermal temperature show less wear damage evident in the form of thinner scratch profile, shallower scratch depth and hence smaller wear volume. Moreover, the friction coefficient is found to decrease with a reduction in the austempering temperature. However, the dynamic friction coefficient for all the specimens saturates after around 20 passes of sliding. Cross-sectional imaging of the scratches shows loss of blocky RA, refinement and orientation of the RA and BF laths along the shape of the bottom of groove after repeated sliding. Moreover, the sub-surface hardness decreases for all the specimens after sliding fatigue. However, the specimen austempered at 250 °C shows the lowest friction coefficient, the least material removal and smallest sub-surface deformation zone under conditions of repeated frictional sliding.

1. Introduction

Nano structured bainitic (NSB) steels comprise of extremely slender plates of ferrite and regions of carbon enriched austenite. The numerous ferrite-austenite interfaces block dislocation motion effectively imparting significant strength while the retained austenite enhances the impact toughness through transformation induced plasticity [1,2]. The design of the steel composition, prior austenite grain size as well as the austempering temperature for bainitic transformation are potent ways to control the final morphology of the bainitic plates as well as the retained austenite [1,3–5]. The bainitic ferrite lath thickness gets significantly reduced by decreasing the austempering temperature [3,6]. However, the fraction of bainitic ferrite can be increased up to a limit by decreasing the transformation temperature and increasing the holding time. This in turn significantly affects the strength, ductility, fracture toughness, fatigue life, impact toughness and hardness of the steel product [7–9].

Friction and wear is extremely complex as it is governed by a range of experimental parameters like load, speed, environment and contact conditions [10]. Hardness, fracture toughness, ductility and microstructural stability of a material under thermal and mechanical stresses can also significantly govern the tribological response under the exact same experimental conditions. NSB steels are already in use as rail steels and can potentially be used for automobile and defense applications [11–16]. However, most of these applications require continuous rubbing of moving parts under operation in addition to bearing load. Hence, it is critical to study evolution of microstructure at rubbing interfaces. A single asperity contact study simulates the most fundamental and primary contact between components and thus forms the basis for most real-life applications involving friction and wear. The interacting surfaces of mechanical components are rough on microscale and therefore the initial contact occurs only through a few surface asperities. Once the asperities deform plastically under applied load, large number of asperities grow in contact thereby supporting the increasing

* Corresponding author.
E-mail address: aparna_s@iitb.ac.in (A. Singh).

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load [17]. The real contact area is therefore less than the apparent area of contact for real surfaces. This interaction of single asperity affects the deformation regimes as well as the friction behavior. Thus, the microstructure of materials used in continuously rubbing parts, like gears, bearings, cams, mechanical seals and switches, needs to be designed for low friction and enhanced wear resistance under multiple cycles of operation using single asperity contact to reveal the deformation mechanisms [18–20].

NSB steels have a dual phase microstructure where the retained austenite is unstable under high stresses. Thus, it is impossible to predict the friction and wear behavior of NSB steels under contact sliding based on the strength and the initial microstructure alone. A few rolling/sliding studies done on NSB steels using a twin disc system have shown the evolution of subsurface microstructure resulting in a very thin white etching layer (WEL) [16,21,22]. The high compressive stresses under rolling/sliding led to transformation of retained austenite (RA) to martensite leading to enhanced strength close to the surface [23,24]. An increase in hardness has been found to enhance the wear resistance under rolling conditions [24]. Experiments done on NSB steels using a stirring wear tester also resulted in a small deformed layer close to the surface [25]. Presence of a WEL with a concomitant increase in subsurface hardness was also reported when sand was used as an abrasive [16]. Abrasion studies done on NSB steels treated at different austempering temperatures showed the lowest wear resistance for the specimen transformed at the lowest temperature [26–28]. The bainitic laths realigned towards the sliding direction and the deformed sub layer was the thickest for the specimen austempered at the lowest temperature [24,26,29]. Ferritic nano grains with an average size of 18 nm were formed close to the surface [28]. However, no clear trend has been observed between the friction coefficient evolution and the isothermal transformation temperature of NSB steels [24,26]. Twin disc rolling/sliding studies done on NSB steels reported the specimen transformed at the highest temperature to show the highest subsurface hardness and the thickest deformed layer [24] which is in contradiction to the two body abrasion studies done on NSB steels [26].

Thus, while the prior studies on NSB steels clearly point that a decrease in austempering temperature enhances the wear resistance under rolling and sliding conditions, the impact of microstructure on the friction coefficient is still unclear. Moreover, there have been contradictory observations on the effect of initial microstructure on the severity of subsurface microstructural evolution. The aim of the current study is to elucidate the role of austempering temperature and in turn the morphology and volume fraction of bainite and associated retained austenite on the damage evolution, friction coefficient and microstructural change under monotonic and cyclic contact sliding. An instrumented nanoindenter with a conical tip simulating a single asperity contact has been used for the repeated frictional sliding experiments. Due to the high stresses beneath a sharp indenter, the microstructure is unstable thus leading to an evolution of wear response with the number of passes of sliding. The subsurface hardness for all the specimens decreased compared to the bulk hardness. However, the specimen transformed at the lowest temperature exhibited the highest wear resistance, least friction coefficient and was the most stable against microstructural change.

2. Experimental procedure

In the present study, steel of composition Fe-0.89C-1.56Mn-1.59Si-0.56Al-1.0Co-0.37Mo-0.19Cr (wt%) has been examined under repetitive contact sliding. The bainitic start temperature (B_s) of the alloy composition is calculated to be 420 °C using MUCG83. Generous amounts of Al and Co in the steel assist in accelerating the bainitic transformation by increasing the free energy change from austenite to ferrite [2]. The steel was prepared in an induction furnace and cast as an ingot of dimensions $140 \times 70 \times 60\,mm^3$. The cast ingot was homogenized at 1200 °C for 48 h to eliminate macrosegregation. Subsequently, specimens of length 15 mm and diameter of 10 mm were forged and rolled at 1000 °C in a single step to a final thickness of 4 mm using GLEEBLE 3800 thermomechanical simulator. Three specimens of size $10 \times 4 \times 4\,mm^3$ were cut from the deformed region of the rolled specimen. They were subsequently austenitized at a temperature of 900 °C for 1 h and quenched to three different temperatures of 250, 300 and 350 °C followed by austempering for 6 h to get distinct microstructure for each of the specimen. The specimens will be addressed as NB250, NB300 and NB350 in accordance with their austempering temperature.

The specimens were mechanically polished to a 1 µm finish on a velvet cloth with diamond paste for microstructural study and wear testing. Detailed microstructural characterization of the specimens was carried out using AURIGA ZEISS scanning electron microscope (SEM). PANalytical X′pert Pro MPD system with Cu-Kα radiations was used to extract the X-Ray diffraction (XRD) pattern of the three heat-treated specimens. The XRD pattern was subsequently analyzed using X′Pert Highscore software and volume fraction of bainite and retained austenite was calculated from integrated intensities of ferrite {110}, {200}, {211}, {220} peaks and austenite {111}, {200}, {220}, {311} peaks respectively [30].

The initial hardness of the specimens was measured using a Vickers micro-hardness tester at a load of 1 kgf with the contact time of indenter to be 10 s. The hardness values reported are an average of at least 15 indentations taken at various locations sufficiently apart from each other to avoid interaction effects.

The repetitive contact sliding experiments were done using a Triboindenter TI 900, HYSITRON Inc., USA. The heat-treated specimens were cut to a final dimension of $10 \times 4 \times 2\,mm^3$. An axisymmetric conical diamond tip with a half angle of 45° and a radius of 2.5 µm was used to make multiple scratches on the specimens. The process was carried out by making contact between the tip and sample surface with subsequent application of normal load as the stage moved laterally. The normal load was ramped to a maximum value of 500 mN for the initial 50 µm and was kept constant for remaining 450 µm. Repetitive sliding was carried over the same scratch after completion of single scratch in a unidirectional motion. The indenter was programmed to make 1, 30, 60 and 90 pass numbers. The scratches were spaced 500 µm apart to avoid the deformation induced interactions. Tangential loads have been recorded along the entire scratch length by force transducers. The friction coefficient is measured from the ratio of the tangential force and normal force (500 mN).

The specimens were scanned using a DektakXT profilometer with the stylus of 2 µm radius to examine the depth profile of the scratches with increasing pass numbers. The scanned profiles were analyzed by taking five sections along the scratch length to estimate an average depth. The top view of the scratches in the three specimens were examined using SEM to investigate the nature of plastic deformation and wear.

Cross-sectional imaging of scratches with 1st, 30th and 90th passes was done using SEM to examine the strain-induced microstructural evolution under repetitive contact sliding for all the specimens. The specimens were polished such that the cross-section of mid region of scratch is exposed. Size of deformed zone and the features of the microstructure in the deformed zone were quantified using an image analysing software (ImageJ). The fractions of the two forms of RA (film and blocky) were determined in the deformed zone (after different pass numbers) and bulk region using point counting method. Moreover, multiple indents on the cross-section of the scratches were made using a Berkovich tip, fixed to nano-indenter, to evaluate the hardness as a function of the distance from the surface. The indents were made in displacement-controlled mode to a depth of 200 nm. The error bars shown in all plots represent standard deviation.

Finite element modelling (FEM) was used to evaluate the plastic strain developed in the material while being indented by a conical diamond indenter at a load of 500 mN. The plastic strains developed