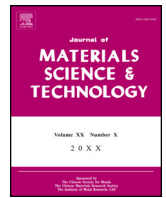




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## Strain-induced formation of a gradient nanostructured surface layer on an ultrahigh strength bearing steel

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### ABSTRACT

In the present work, an ultrahigh strength bearing steel (AISI 52100) was subjected to surface mechanical rolling treatment (SMRT) at room temperature. Microstructural observations showed that martensitic laths, twins and cementite particles in the initial microstructure underwent distinct plastic strains and were gradually refined into nanostructures. Consequently, a gradient nanostructured (GNS) surface layer with a mean grain size of ~24 nm at the top surface was obtained on the bearing steel, resulting in an increment of ~20% in the surface hardness. Analyses based on microstructural evolution, phase constitution and in-depth hardness distribution revealed a mechanically induced formation mechanism of the GNS surface layer. The multiple surface severe plastic deformation under fine lubrication and cooling during SMRT contributed to the formation of a thick hardened surface layer on the bearing steel.

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### 1. Introduction

In recent years, forming a gradient nanostructured (GNS) surface layer, in which the grain size increases gradually from nanometers to micrometers with increasing depth, has been developed to modify various properties and performances of metallic materials. For example, a synergic enhancement in strength and ductility was obtained on different metallic materials with a GNS surface layer, in comparison with the traditional trade-off between strength and ductility in the counterpart materials with homogeneous coarse-grained or nano-grained microstructure [1–4]. In addition, enhancements in fatigue, corrosion and wear resistances have also been achieved on some materials due to the formation of a GNS surface layer [5–10].

Bearing steels in a quenched and tempered (normally at temperatures <300 °C) state possess ultrahigh strength, and are widely used in the manufacture of technologically important engineering components [11]. Forming a GNS surface layer on bearing steels is therefore expected to further promote their performances, such as resistances to wear and fatigue. While some techniques based on surface gradient plastic deformation, namely surface mechanical attrition treatment (SMAT) and surface mechanical grinding

treatment (SMGT), have been developed to produce GNS surface layers on various metals and alloys [12–15], the success employment of these methods on bearing steels is implausible to form a GNS surface layer. This is mostly due to the fact that enough deformation without failure is difficult to be accumulated by these methods to form nanostructures in the surface layer of a material with ultra-high hardness and poor ductility.

In the past decades, a large number of works reported that a thin and hard surface layer could form on a bearing steel by hard turning [16–18], drilling [19] and rolling contact fatigue (RCF) [20–24]. While this layer was observed to be white and featureless by using optical microscopy, it was commonly named “white etching layer” (WEL). Elaborative microstructural characterizations by using scanning electronic microscopy (SEM) and transmission electron microscopy (TEM) demonstrated that it is usually composed of a nano- and sub-micrometer sized grain structure. However, the effects of WELs on properties of bearing steel are still under controversy. This is mostly related with the fact that different mechanical properties (e.g. hardness) were obtained in the WELs, possibly due to their different formation mechanisms. For example, Li et al. [19] found that the WELs of drilled holes in different bearing steel samples consisted of a high content of austenite, and a decreased hardness was detected in the WELs in comparison with base materials. Consequently, they inferred that the WELs were produced by severe plastic deformation accompanied by dynamic phase transformation from martensite to austenite during drilling. Meanwhile,

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after studying WELs induced by hard turning AISI 52100 steel, Hosseini et al. [17] proposed two possible formation mechanisms: one was predominantly mechanically induced and the other was predominantly thermally induced. The former was controlled by dynamic recovery and the latter was controlled by dynamic recrystallization at elevated temperatures due to higher cutting speeds.

It is noted that a sharp transformation in microstructure was typically observed between WEL and matrix in previous works [11,18,19,21], and the above-mentioned formation mechanisms were mainly inferred from the phase constitutions in the WEL while no enough information could be obtained from microstructural observations in the transformation layer. Meanwhile, the materials were prone to premature failure during service (e.g. fatigue and wear) due to a higher stress concentration state in the layer with sharp transitions in microstructure and mechanical properties [25]. By using a newly developed technique, i.e. surface mechanical rolling treatment (SMRT), a GNS surface layer has been generated on AISI 52100 bearing steel in a quenched and tempered state in a previous work [26]. And significantly enhanced fatigue property and wear resistance have been achieved on the SMRT sample. In comparison with the pre-existing surface gradient plastic deformation techniques, e.g. SMAT and SMGT, SMRT is able to produce a thicker GNS surface layer with a much lower surface roughness [6].

In the present work, microstructure evolutions in the surface layer of the SMRT bearing steel sample were systematically investigated. Subsequently, the formation mechanism of the GNS layer was analyzed in comparison with those of WELs in literatures, and effects of processing technologies of SMRT on the formation of GNS surface layer were discussed.

## 2. Experimental

The material studied in this work was commercial AISI 52100 steel with chemical compositions (wt%) of 0.97C, 1.55 Cr, 0.40 Mn, 0.27 Si, 0.02 Cu, 0.01 Ni and balance Fe. The as-received material was austenitized at 845 °C for 30 min, oil-quenched and tempered at 150 °C for 120 min. Hereafter, an initial hardness of  $8.5 \pm 0.2$  GPa was obtained.

Rod samples with a diameter of 10 mm were cut by electro-spark discharge machining and submitted to SMRT processing to form a GNS surface layer. Details for the set-up and processing of SMRT are available in Refs. [6,26]. In brief, a polished and rotatable WC-Co cermet ball (8 mm in diameter) was pressed into the sample surface during SMRT, while the sample turned around its axis at a velocity ( $v_1$ ) of 600 revolutions per minute and the cermet ball moved along the axis from one side to the other side at a velocity ( $v_2$ ) of  $0.1 \text{ mm s}^{-1}$ . This procedure was repeated 3 times, while  $v_1$  and  $v_2$  kept the same and the preset pressing depth of the ball into the sample ( $a_p$ ) increased  $50 \mu\text{m}$  each time. The processing was performed at ambient temperature, with cycling oil to lubricate and cool the ball and the sample.

Morphologies of the initial sample were observed using SEM on an FEI Nova NanoSEM 430 unit after etching by Nital etchant. Detailed microstructural characterizations of the initial sample, as well as the SMRT surface layer, were observed by using an FEI TENCAI F20 TEM operated at 200 kV. Foils for TEM observations at different depths in the SMRT surface layer were prepared by following steps: (i) cutting a surface layer ( $\sim 1$  mm in thickness) from the treated sample by electro-spark discharge machining; (ii) mechanically removing a surface layer with a certain thickness (i.e. the desired depth) from the treated side, and then grinding the foil to a thickness of  $20 \mu\text{m}$  from the untreated side; (iii) dimpling and ion-milling the foil from the untreated side. Meanwhile, phase constitutions at different depths were characterized by using

a Rigaku D/max-2400 X-ray diffractometer with  $\text{Cu-K}\alpha$  radiation using a small scanning step ( $2\theta = 0.02^\circ$ ).

Variations of microhardness along depth in the GNS surface layer were determined on the SMRT sample by combining measurements from both the planar view and the cross-sectional view, i.e. on the surfaces vertical and parallel to the normal direction of the rod, respectively. Iterative electrolytic polishing was applied to prepare the planar samples by removing surface layers with different thicknesses, and the microhardness was measured on the exposed surface each time. The microhardness measurements were performed using a Qness Q10 A+ tester fitted with a Vickers indenter, with the maximum load of 100 g and a holding duration of 10 s. The distance between any two neighboring indentations was larger than  $50 \mu\text{m}$  to minimize the interference between different measurements.

## 3. Results

### 3.1. Microstructural evolutions in the subsurface layer

The initial material before SMRT is characterized by a typical martensitic structure of bearing steel in a quenched and tempered state, i.e., the martensite matrix embedded with numerous cementite particles, as shown by the SEM morphology in Fig. 1(a). Further TEM observations indicate that the martensite matrix is mostly composed of lath martensite (or dislocation type, Fig. 1(b)), in which dense dislocations exist. In addition, few plate martensite (or twin type,  $\sim 8\%$  in volume fraction) co-exists in the matrix, as shown in Fig. 1(c) with the corresponding selected area electron diffraction (SAED) pattern. Statistical measurements show that the mean size of cementite particles, the average width of martensitic laths, and the average thickness of martensitic twin/matrix lamellae in the initial sample are about 450 nm, 196 nm and 17 nm, respectively. It is noticed that a small amount of retained austenite phase can also be detected in the initial sample, as shown by the XRD pattern of matrix in Fig. 1(d). The volume fraction is estimated to be  $\sim 15\%$  from the pattern by considering the integrated intensity of diffraction peaks and corresponding material scattering factors of austenite and martensite [6].

Clear evidences of plastic deformation and microstructure refinement have been observed in the subsurface layer of the SMRT sample, as revealed by TEM observations on the microstructural evolution processes of martensitic laths, twins, and cementite particles at different depths from the treated surface.

As shown in Fig. 2, the sizes of martensitic laths gradually decrease along both the short and long axes and the dislocation density increases with decreasing depth in the subsurface layer. For example, the average width of the martensitic laths decreases to  $\sim 121$  nm at the depth of  $\sim 40 \mu\text{m}$ , and the boundaries between different laths are relatively blurred due to interactions with dense dislocations and shear bands in them. Furthermore, it is noticed that lath boundaries vanish and are replaced by tangled dislocations in some regions at a larger depth ( $\sim 140 \mu\text{m}$ ), as circled in Fig. 3(a). With decreasing depth, the dislocation density increases significantly and dislocation cells with a mean size of  $\sim 150$  nm are formed at the depth of  $\sim 80 \mu\text{m}$ , as shown in Fig. 3(b).

Accompanying the plastic deformation and microstructural evolution in martensitic laths, the twins are also significantly strained and refined in the subsurface layer. As shown in Fig. 4(a) and (b), a bundle of twins are broken by shear bands with the thickness of  $\sim 200$  nm at the depth of  $\sim 40 \mu\text{m}$ . Meanwhile, remarkable interactions of twins with dislocations are also revealed outside the shear bands, so that the twins are cut into the nanosized grains. Furthermore, interactions of twins within two different orientations are observed at the depth of  $\sim 20 \mu\text{m}$  in the subsurface layer,

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