Annealing Effect on Wear Resistance of Nanostructured 316L Stainless Steel Subjected to Dynamic Plastic Deformation

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Bulk nanostructured 316L austenitic stainless steel (SS) samples with nano-scale twin bundles embedded in nano-sized grains were synthesized by using dynamic plastic deformation (DPD). Subsequent thermal annealing of the as-DPD sample leads to a single austenitic structure with static recrystallized (SRX) grains in nanostructured matrix. Oil-lubricated sliding tests of ball-on-disc type were carried out for the as-DPD and the as-annealed DPD steel samples in comparison with coarse grained (CG) steel samples. Experimental results show that the as-DPD 316L steel exhibits a little enhanced wear resistance under a load of 10 N, and nearly identical wear resistance under a load of 30 N relative to that of the CG sample. After annealing, the wear resistance roughly follows the Archard equation under a load of 10 N. However, the wear resistance increases with increasing hardness, and decreases with a further increase in hardness under a load of 30 N. The highest wear resistance can be found in the DPD sample annealed at 750°C for about 20 min, which is more than 46% higher than that of the CG steel sample. This phenomenon is originated from the microstructure with an optimized combination of strength and ductility as a result of moderate plastic deformation in SRX grained regions.

KEY WORDS: Nanostructured metals; Stainless steel; Wear resistance; Annealing; Subsurface microstructure

1. Introduction

Austenitic stainless steels (SS) have widespread engineering applications for their excellent resistance to corrosion and good formability[1,2], but their usage in tribological application is limited due to their relatively low hardness and poor wear performance. Wear causes about 50% loss of all materials in service especially in contacting deformation and damage situation[3]. For many aspects of applications, it needs primary consideration for investigating wear resistance and tribological behavior.

Grain refinement is a well-known strengthening technique to meet further comprehensive industry requirements. For instance, mechanical alloying[4], equal channel angular pressing[5], cold rolling[6] and surface mechanical attrition treatment[7] are different plastic deformation techniques to produce nanostructured metals. Moreover, according to Hall-Petch relationship[8], both hardness and strength increase linearly with decreasing grain size. As described in classical Archard equation[9,10], wear volume is reversely proportional to the hardness of the materials. Therefore, grain refinement also makes a contribution to the enhancement of wear resistance and lots of related studies have been reported[11,12]. However, wear resistance of material is not only determined by its intrinsic hardness related to microstructure, but also decided by its extrinsic frictional conditions. Nanostructured metals are usually brittle, especially for steel, ductility loss is more pronounced than the strength gain for the austenitic stainless steel[13], which could impose an influence on wear coefficient (K) in Archard equation. Zhou et al.[14] found that a maximum wear resistance in nanostructured AISI52100 steel did
not correspond to the finest grain with the highest hardness, but corresponded to an optimized hardness-plasticity combination at a critical grain size.

Dynamic plastic deformation (DPD) is a novel synthesis method to get bulk nanostructured metals due to its high strain rate\cite{15,10}. It has been carried out to produce 316L SS consisting of nano-scale twin bundles embedded in nano-sized grains\cite{17}. Meanwhile, after subsequent thermal annealing of the as-DPD samples, a single-phased austenitic structure in static recrystallized (SRX) grains embedded with retaining nano-twin bundles and nano-grains was developed, which exhibits an enhanced strength-ductility combination\cite{18}. With this excellent and comprehensive mechanical properties, what the wear properties of the as-DPD and as-annealed DPD 316L SS samples will exhibit is our main focus of the present work.

In this work, both the as-DPD 316L SS samples with nano-twin bundles embedded in nano-grains and the DPD samples annealed in a temperature range of 730–770 °C with SRX grains in retaining nanostructures are investigated in lubricated-sliding tests. Coarse grained (CG) AISI 316L SS is also involved for comparison. The subsurface microstructure and micro-hardness variations underneath the worn surface are discussed as well.

2. Experimental

The materials studied in the present work is a commercial AISI 316L austenitic SS. Prior to the DPD treatment at room temperature, the as-received CG AISI 316L SS samples with a dimension of 10 mm in diameter and 15 mm in length were annealed at 1200 °C for 1 h to reduce the effect of mechanical processing and obtain homogeneous coarse grains.

The DPD facility set-up and processing procedures are described in literature\cite{15,16}. 316L SS samples were processed on a DPD facility at room temperature with a strain rate of \(10^2–10^3 \text{s}^{-1}\). Multiple times of impacts were applied to deform the steel samples, forming a disc with a total strain of 1.65, where the strain of the as-DPD samples is defined as \(\varepsilon=\ln (L_o/L_f)\), where \(L_o\) and \(L_f\) are the initial and final thickness of the treated samples, respectively. To investigate microstructure effect on the wear properties, the as-DPD 316L SS samples were annealed at 730, 750 and 770 °C for 20 min in order to modify microstructures via partial SRX, respectively. And some samples are annealed at 750 °C for different time (5, 20 and 60 min).

The microstructures of the as-DPD and the as-annealed DPD 316L SS samples were characterized by scanning electron microscopy (SEM) on a FEI Nova Nano-SEM system at an operating voltage of 15 kV. The micro-hardness of the different samples was measured by using a Vicker’s hardness tester with a load of 50 g and duration of 10 s.

Sliding wear tests of the as-DPD and the as-annealed DPD 316L SS samples were conducted by using an Optimal SRV III oscillating friction and wear tester in a ball-on-plate contact configuration. The specimens, with a dimension of 7 mm×7 mm×2.7 mm, slid against WC-Co balls of 10 mm in diameter with a hardness of 17.5 GPa. The sliding contact surface of the specimens is perpendicular to the impact direction of the DPD treatment. Before the wear tests, the top surface layer of the specimens was removed by electrochemical polishing carefully in order to eliminate the surface roughness effect on the tribological behavior. The wear tests were carried out in a relative humidity of 40%–50% and at room temperature of 20–25 °C. Oil lubricated sliding tests were conducted in this experiment, at an oscillating stroke of 1 mm, normal loads of 10 and 30 N, a frequency of 5 Hz and a duration of 30 min. The oil used is mineral oil without any additives. The wear tests of the CG samples were also conducted under the same conditions for comparison.

Profiles of the worn surfaces were measured by using a MicroXAM 3D surface profiler system so as to determine the worn volume. 3D surface profiles are imaged by the interference of two white lights. To quantify the volume of wear scar, a reference surface is needed to be determined, below which the volume of material was taken as the worn volume.

The morphologies of the worn surfaces under different wear conditions were observed by SEM. Before SEM observation, the specimens were cleaned ultrasonically in acetone for about 5 min. The composition of the worn surface was analyzed by energy dispersive spectroscopy (EDS) in SEM. A protective layer of nickel was electrodeposited on the worn surface for the subsurface structure investigation. The cross-sectional wear scar samples perpendicular to sliding direction were prepared. The worn subsurface structure of specimens was investigated by SEM in electron channeling contrast (ECC) mode. The micro-hardness variations along the depth from the worn surface were measured by using a dynamic ultra micro hardness tester on a DUH-211S as well.

3. Results and Discussion

3.1 Microstructure and hardness

Fig. 1 shows the microstructure characterization of the as-DPD and the as-annealed DPD 316L SS samples by SEM. From Fig. 1(a), the microstructure of the as-DPD samples with a saturated strain is more or less blurred; “dark-etching” and wavy bands of shear bands can be observed. Under SEM it is not clear due to severe plastic deformation, which consists of nanoscale twin bundles embedded in nano-sized grains matrix. The twin/matrix (T/M) lamellar thickness mostly ranges from a few nm to 90 nm, with an average value of 20 nm. More detailed TEM microstructure information can be found in literature\cite{17,18}.\[872\]
The structural evolution upon thermal annealing of the as-DPD samples is shown in Fig. 1(b–f). The higher temperature and the longer annealing duration may correspond to the more volume fraction and the larger size of SRX grains\textsuperscript{[18]}. After annealing at 730 °C for 20 min, a small amount of SRX grains was found, and the unrecrystallized regions are still composed of T/M lamellar and nano-grains (Fig. 1(b)). With the increase of annealing temperature, obvious SRX grains of $\sim$50% in volume were observed in the samples annealed at 750 °C, they become larger and account for a larger volume fraction (Fig. 1(c)). No obvious size and orientation change occurs in recrystallized areas, while SRX grains grow to a size of a few microns with random orientations. With the further increase of temperature, SRX grains grow to a dominant volume fraction of above 90% after annealing at 770 °C (Fig. 1(d)). Obviously, the sizes of SRX grains increase to a certain extent and become nonuniform. For isothermal annealing at 750 °C for different durations, a similar situation was observed. After annealing for 5 min, SRX is still in an incubation period. No obvious SRX grains were found under SEM ((Fig. 1(e)). With increasing annealing time, SRX occurs obviously, as described above in the situation for 20 min. When the duration is up to 60 min, both the volume fraction and the sizes of SRX grains increase significantly, as shown in Fig. 1(f). Either nano-grained or nano-twined regions could hardly be observed, the
situation looks quite like the ones annealed at 770 °C for 20 min.

Variations of micro-hardness for the as-DPD samples and the as-annealed DPD 316L samples are shown in Table 1. For the as-DPD samples with a saturated strain of 1.65, the hardness goes up to 4.15 GPa, which is much higher than that of the CG samples (1.55 GPa). After annealing, the hardness of samples decreases with increasing annealing time and annealing temperature. Among the annealed conditions used, the hardness of the as-annealed DPD samples is in a range of 2.26–3.67 GPa. This is due to SRX partly and grain growth, which is consistent with the microstructure characterization.

### 3.2 Wear behavior

Wear volumes of the CG, the as-DPD and the as-annealed DPD samples under loads of 10 N and 30 N are given in Table 2. At an applied load of 10 N, the wear volume of the CG samples is the highest one of about $2.20 \times 10^{-3}$ mm$^3$ among three kinds of steel samples, while wear volume of the as-DPD samples is the lowest of about $1.58 \times 10^{-3}$ mm$^3$, which is 25% lower than that of the CG samples. For the as-annealed DPD samples, the value of wear volume is between that of the CG and the as-DPD samples, ranging from 1.65 to $2.21 \times 10^{-3}$ mm$^3$. Compared with the hardness in Table 1, it can be found that the as-DPD steel samples with the highest hardness exhibit the lowest worn volume. As shown in Fig. 2(a), it seems that the wear volume decreases with increasing hardness of the samples. It is clear that the wear volume-hardness relationship roughly follows Archard equation, i.e., the harder the samples are, the higher wear resistance the samples exhibit.

However, when sliding at a higher applied load of 30 N, the results are quite different. Wear volume of the as-DPD samples is $3.09 \times 10^{-3}$ mm$^3$, which is nearly as large as that of the CG samples ($3.28 \times 10^{-3}$ mm$^3$). The samples with the highest hardness did not exhibit the best wear resistance as expected in Archard equation. After annealing, wear volume is lower than that of the CG and the as-DPD samples, ranging from 1.76 to $2.68 \times 10^{-3}$ mm$^3$. It is interesting to note that the DPD sample annealed at 750 °C for 20 min shows the lowest wear volume value of $1.76 \times 10^{-3}$ mm$^3$, which is 46% lower than that of the CG samples and 43% lower than that of the as-DPD samples. To plot the variation of wear volume with the hardness of the samples, as shown in Fig. 2(b), it can be found that wear volume decreases with increasing hardness and then increases with the further increase in hardness. Obviously, the best wear resistance of 316L SS samples corresponds to a moderate hardness, not the highest hardness among the different samples. This phenomenon is contradicted to Archard equation, and it suggests that hardness should not be the only factor to impose the influence on wear properties of the materials.

SEM micrographs of the worn surfaces of the CG,
the as-DPD, the as-annealed (750 °C, 20 min) DPD samples under a load of 30 N are shown in Fig. 3(a–c). The worn surfaces for different samples exhibit similar morphologies of the parallel grooves. As austenitic stainless steel is lack of ability to form an effective anti-wear boundary film under oil lubrication\cite{19}, the dominant wear loss is ploughing caused by the contacting between the WC-Co balls and the samples. More rough grooves appears on the worn surface of the CG sample and some of them give rise to long cracks. This may be ascribed to the softer CG matrix, which is easier to be removed by harder ball. For the as-DPD sample there are more intense grooves parallel to the wear direction on the worn surface, and no obvious peeling and cracks are formed. Compared with two former samples, the as-annealed DPD sample exhibits smoother worn surface and fewer grooves. Fig. 3(d) shows 2D cross-sectional profiles of the wear scars for the CG, the as-DPD and the as-annealed (750 °C, 20 min) DPD samples under a load of 30 N. The worn surface profiles of the CG and the as-DPD samples look like quite fluctuant and quite similar in depth. The width of the wear scars on the as-DPD sample is about 20 µm smaller than that of the CG sample. But for the as-annealed DPD sample, the profile is much smoother compared with two former samples. The depth of wear scar is about 3 µm shallower and the width is 50 µm thinner than that of the CG sample. The results of the morphologies and profiles of the wear scars are consist with that of wear volume. Furthermore, EDS analysis of the worn surfaces also reveals that there is no obvious amount of O and W on the worn surface after lubricated-sliding tests. It is demonstrated that no transfer layers and surface oxidation happened.

3.3 Worn subsurface

To explore the controlling factor on the wear resistance apart from hardness, the worn subsurface structure of three typical samples was observed by using SEM in ECC mode. In lubricated sliding tests, the degree of the wear is milder than that in dry sliding tests. Therefore, the deformed layer that sliding induced in the subsurface is not large, just about 20–30 µm in depth, as seen in Fig. 4, the microstructure in this layer underneath the worn surface shows a contrast with the matrix. For the CG samples (Fig. 4(a)), under the worn surface significant grain refinement happened in the deformed layer of about
20 \mu m as a result of repetitive loading. The grain size decreases with decreasing distance away from the worn surface, the involved deformation mechanism may be mechanical twinning for 316L SS with low stacking-fault energy\cite{20}. As described in many literature\cite{21,22}, the top surface in the deformed layer consists of nano-grains. Unlike the CG sample, grain coarsening happened in the worn subsurface region for the as-DPD sample (Fig. 4(b)). Because of nanostructure in the hard matrix of the as-DPD sample with a saturated strain, plastic deformation was restricted by high density of dislocations, grain boundaries and twin boundaries, grains could not be refined further and the deformed layer is less than 20 \mu m. On the contrary, under high plastic strain during sliding, for the as-DPD sample with high stored energy, grain growth occurs accompanied by plastic deformation. No obvious dynamic recrystallization occurred (see Fig. 5(a)), the grains in the worn subsurface remained in a size range of submicrons to nanometers, which is quite different from the recrystallized structure in the as-DPD Cu samples after sliding\cite{23}.

However, the situation of the as-annealed DPD sample (750 °C, 20 min), the situation differs from two former samples, which exhibits a distinct subsurface structure as shown in Fig. 4(c). The deformed layer is more than 30 \mu m in thickness, which is much deeper compared with the as-DPD sample. It implies that plastic deformation-bearing ability significantly increase after annealing. Careful observation (Fig. 5(b)) shows that an ultrafine grained ability occurs between the top nanostructured layer and the matrix. Considering the microstructure characteristic of the as-annealed DPD samples, SRX grains embedded in nanostructured matrix are ductile enough to bear plastic deformation, which can be refined from a few microns to submicrons. It is understandable that ductile SRX grains provided the plasticity to undergo moderate plastic deformation in the hard matrix, which consumed the frictional work and inhibited peeling of materials. Meanwhile, grain growth may occur in the original nano-grained regions like the situation in the as-DPD samples. So, for the as-annealed DPD sample, ultrafine grained regions in the worn subsurface developed during sliding are responsible for the highest wear resistance, which originates from an optimized mixed structure of SRX grains embedded in nano-grains and nano-twin bundles. As proved in tensile tests, the sample with this mixed structure exhibits the best strength-ductility combination\cite{18}, which leads to the highest wear resistance.

To further understand the difference in the worn subsurface structure, the variations of micro-hardness with the distance away from the worn surface for dif-
ferent samples were shown in Fig. 6. A gradually decreased hardness distribution was found in the CG sample along the depth from the worn surface, as shown in Fig. 6(a). The hardness varies from more than 2.8 GPa of 5 µm below the worn surface to 1.6 GPa of the CG matrix. The as-DPD sample exhibits an opposite tendency, the hardness increases with the increase of the distance from the worn surface (see Fig. 6(c)). The hardness changes from more than 3 GPa near the worn surface to more than 4 GPa of the matrix. The profile in the as-annealed DPD sample looks like that in the CG sample, as shown in Fig. 6(b). However, the hardness varies from more than 4 GPa near the surface to the matrix of nearly 3 GPa, which gives an evidence of an optimized combination of a high hardness and a moderate plasticity. All hardness results are consistent with the worn subsurface structure which determines the wear resistance of the samples.

4. Conclusions

(1) Bulk nanostructured 316L austenitic SS with nano-scaled twins embedded in nano-grains was synthesized by means of DPD. After thermal annealing treatment, the as-annealed DPD sample shows a mixed structure with SRX grains in remained nanostructured matrix. The hardness of the as-DPD sample is much higher than that of the CG sample, while the hardness of the as-annealed DPD sample is more or less lower than that of the as-DPD one.

(2) Under oil lubricated sliding condition, the wear resistance of the samples roughly follows Archard equation at a load of 10 N. For a relatively high applied load of 30 N, the wear resistance increases with an increase in hardness and decreases with the further increase in hardness. The highest wear resistance was found in the DPD sample annealed at 750 °C for 20 min, which is more than 46% higher than that of the CG sample.

(3) The highest wear resistance originates from the microstructure with an optimized combination of strength and ductility as a result of moderate plastic deformation in SRX grained regions.

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