Rapid Communication

316L Austenite Stainless Steels Strengthened by Means of Nano-scale Twins

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By means of dynamic plastic deformation (DPD) followed by thermal annealing, a mixed structure of micro-sized austenite grains embedded with nano-scale twin bundles (of about 20% in volume) has been synthesized in a 316L stainless steel (SS). Such a 316L SS sample exhibits a tensile strength as high as 1001 MPa and an elongation-to-failure of about 23%. The much elevated strength originates from the presence of a considerable number of strengthening nano-twin bundles, while the ductility from the recrystallized grains. The superior strength-ductility combination achieved in the nano-twins-strengthened austenite steel demonstrates a novel approach for optimizing the mechanical properties in engineering materials.

KEY WORDS: Nano-scale twins; Strength-ductility combination; Dynamic plastic deformation (DPD); Stainless steels

1. Introduction

The 300 series austenitic stainless steels provide high resistance to corrosion, oxidation and good formability over a wide temperature range[1–3]. However, their low mechanical strength and poor anti-friction properties restrict them from engineering applications with higher requirements. Grain refinement has been considered for strengthening the steels according to the Hall-Petch relationship[4–5]. For example, when grain sizes of 316L stainless steel (316L SS) decrease to about 40 nm, tensile yield strength increases to 1450 MPa, about 6 times higher than that of the coarse-grained counterpart (250 MPa)[6]. But such a grain refinement strengthening is achieved at an expense of ductility and toughness inevitably. The uniform elongation in tensile tests of the nanocrystalline 316 SS is less than 3%[6].

Twin boundary (TB) is a special kind of internal interfaces with low excess energies, which may serve as effective barriers against dislocation transmission, similar to conventional grain boundaries[7–8]. Recently, Lu et al. showed that an excellent combination of yield strength and ductility can be achieved by introducing a high density of TBs in coarse grains of polycrystalline pure copper samples prepared by using electro-deposition[9–10]. The strengthening mechanism is associated with the interaction between dislocations with plenty of TBs which block dislocation motions effectively.

It has also been demonstrated that dynamic plastic deformation (DPD) (i.e., plastic deformation at high strain rates) provides another practical approach for generating nano-scale twins in metals with low- or medium- stacking fault energies in bulk formation[11–14]. Thereby, it is a consequent development to produce nano-twins in engineering alloys such as austenite stainless steels by means of the DPD process. In this work, a commercial 316L SS is processed by DPD followed by subsequent thermal annealing, for synthesizing nano-scale twins to
strengthen the steel.

2. Experimental

The chemical composition (wt pct) of AISI 316L SS studied in the present work is 16.42Cr, 11.24Ni, 2.12Mo, 0.02C, 0.37Si, 1.42Mn, 0.011S, 0.04P and Fe balance. The as-received 316L SS was annealed at 1200°C for 60 min to obtain a uniform face-centered cubic (fcc) austenite structure with an average grain size of 85 µm. The DPD set-up and procedures are described in literature [12]. 316L SS cylinder samples, 12 mm in diameter and 15 mm in height, were treated at room temperature on a DPD facility with a strain rate of about 10^3 s^{-1}, forming a disc with a total strain of 1.6, where the strain is defined as \( \varepsilon = \ln(L_0/L_f) \) (\( L_0 \) and \( L_f \) are the initial and the final thicknesses of the treated sample, respectively). For a total strain of 1.6, the final DPD sample dimensions are 27 mm in diameter and 3 mm in height.

Uniaxial tensile tests were carried out on an Instron 5848 MicroTester (2 kN) at a strain rate of 6×10^{-3} s^{-1} at room temperature. The gauge section of dog-bone shaped tensile specimens from the DPD disc samples is 5 mm in length and 1 mm in width. The tensile samples were electro-polished to a mirror surfaces with a final thickness of 0.5 mm. A contactless MTS LX300 laser extensometer was used to calibrate and measure the sample strain upon loading. To ensure the reliability of the tensile data, three repeated tensile tests were performed on the tested samples. The microstructures of the DPD 316L SS samples were characterized by transmission electron microscopy (TEM) performed on a JEOL 2010 TEM operating at 200 kV.

3. Results and Discussion

The transverse microstructure of the as-DPD 316L SS sample is shown in Fig. 1(a). Obviously, original coarse grains were severely refined into a mixed nanostructure by DPD process. Nano-sized grains were formed and most of them are elongated with an aspect ratio of \( \sim 3 \). From statistic TEM measurements, the short-axis grain sizes are in a range from 15 nm to 80 nm, with a peak value of about 36 nm. There are a considerable number of deformation twins in the mixed nanostructure. The twins are in form of isolated bundles with sizes of a few micrometers thick and several micrometers long, surrounded by nano-sized grains. The twin bundles are roughly perpendicular to the loading direction. Detailed observations revealed that most deformation TBs are stepped or curved due to the presence of a high density of dislocations. The statistical measurements show that the volume fraction of twin bundles is about 38% and the average TB spacing is about 17 nm with a distribution from several nanometers to 90 nm. In both X-ray diffraction analysis and selected area electron diffraction (SAED) patterns in TEM, no strain-induced martensite phase was detected in the as-DPD 316L SS sample.

High-rate strain can induce high density of deformation twins in 316L SS during DPD because twinning is more favorable than slip in low stacking fault energy materials. When twin density is saturated, further twinning becomes difficult to accommodate subsequent strain so that shear banding is usually activated in the area of high density nano-scale twins, of which the kinetic mechanism was studied in a DPD Cu-Al alloy[14]. As twin structure is deformed and destroyed within a narrow band, elongated dislocation cells can form along shear direction. With further straining, these elongated cells would break up into roughly equiaxed subgrains accompanied by increasing misorientations, and eventually evolve into randomly oriented nano-sized grains.

Thermal annealing of the as-DPD sample at 750°C for 25 min in vacuum led to a partial recrystallization of the deformed structure, forming a mixed structure consisting of nano-scale twin bundles embedded in ultrafine grained matrix, as shown in Fig. 1(b). The recrystallized (RX) grains are dislocation-free, constituting roughly 41% in volume. The grain size of static recrystallization (SRX) grains ranges from about 0.5 µm to about 3.5 µm from the SEM observations, with an average grain size of about 1.67 µm. There is a slight change in the average TB spacing (~18 nm) in the remaining nano-twin bundles compared with the as-DPD state, but the dislocation density inside
the twin bundles becomes lower as indicated by TEM images. The volume fraction of survived nano-twin bundles was decreased to about 24%. The remaining nano-sized grains slightly grow and the grain size ranges from 20 to 200 nm with an average size of 89 nm.

Since twin structure has a higher thermal stability in comparison with nano-sized grains, SRX may occur preferably in the nano-sized grains instead of the nano-twin bundles. Therefore, during thermal annealing most nano-sized grains were recrystallized into ultrafine grains while the nano-twin bundles are remaining. Hence, the un-recrystallized region consists of mostly nano-twin bundles and some minor nano-sized grains.

Figure 2 shows typical tensile engineering stress-strain curves for both as-DPD and the annealed DPD 316L SS samples, in comparison with that of the original coarse-grained sample. Obviously, the as-DPD 316L SS exhibits much enhanced strength: yield strength (0.2% offset) is 1345±31 MPa, about 4.6 times that of the coarse grained samples and is comparable to that of the nanocrystalline 316L SS samples[6]. The ultimate tensile strength is 1432±47 MPa. The plasticity of the as-DPD 316L SS samples is very limited, with an elongation-to-failure of 6%. However, the annealed DPD sample exhibits much enhanced elongation-to-failure, being as high as 23%, with an obvious strain hardening. The uniform elongation exceeds 15%. Meanwhile, both yield strength (901 MPa) and the ultimate tensile strength (1001 MPa) are still in a high level.

Strength of DPD 316L SS increases obviously with a decreasing ductility upon straining, as shown in Fig. 3. Such a trend is consistent with the general strength-ductility trade-off of deformed metals and alloys[6, 15–16]. In order to improve the ductility of deformed materials, subsequent annealing is usually performed. It is noted that the annealed DPD sample simultaneously exhibits high strength and high ductility in comparison with the as-DPD samples.

For example, the elongation-to-failure of the annealed DPD sample (indicated by red solid circle) is about two times higher than that of as-DPD sample with strain of 0.41 and the strength of the former is higher than that of the latter. Alternatively, it can be said that the strength of the annealed DPD samples is much higher than that of the as-DPD sample when their elongation-to-failure values are comparable. Clearly, the strength-ductility combination of the annealed DPD sample is superior to that of the as-DPD samples. This phenomenon was also observed in the annealed DPD Cu samples[17].

The strengthening of the annealed DPD 316L SS can be attributed to the following several factors. Since the average thickness of twin/matrix lamellae is still very small (18 nm) and the volume fraction of the survived twins is about 24% after annealing, the contribution of the survived twins is about 24% after annealing, the contribution of twin boundaries is estimated to be dominant according to the TB strengthening[18]. TBs can act as barriers to dislocation motion during plastic deformation, so that dislocations pile up and propagate across the twins when they are dissociated into partial dislocations. Stress concentrations at twin-slip band intersections are required to activate the slip transmission across TBs, which leads to strengthening. Especially for very thin twin lamellae, an extremely high stress is needed to activate a single dislocation to penetrate the TB[18]. In addition, the survived nano-sized grains with average size of 89 nm and volume fraction of 35% also contribute to the high strength. The SRX grains are much smaller than that of the initial sample, from which the grain-boundary-strengthening contribution is considerable due to their large volume fraction in the annealed sample. Because the DPD process at room temperature and subsequent annealing did not introduce any martensite phase transformation or other impurities, the strengthening effect of martensite in the annealed

![Fig. 2 Tensile engineering stress-strain curves of the as-DPD and the annealed DPD 316L SS samples. For comparison, the tensile curve for the coarse-grained counterpart is also included](image1)

![Fig. 3 Elongation to failure vs. ultimate tensile strength for the as-DPD and the annealed DPD 316L SS samples. For comparison, other data reported in literature are also shown: ▽ SMAT 316L[6]; △ cold rolling 316L[15]; ○ annealing after cold rolling 316L[16]](image2)
DPD 316L SS can be excluded.

Besides the high strength of the annealed DPD sample, its ductility is also attractive. Compared with the limited plastic strain of the as-DPD samples, a much enhanced plastic strain as well as the strain hardening ability in the annealed DPD 316L SS were found, indicating that a large amount of dislocations accumulated during the plastic deformation prior to failure. Indubitably, the relatively large SRX grains provide a substantial room for lattice dislocation pile-up and accommodation, which contribute to an obvious strain hardening. Microstructural observations indicated that the twin boundaries in the annealed DPD 316L have a lower initial dislocation density in comparison with the as-DPD samples. This suggested that the numerous twin boundaries in the annealed samples could also serve as the locations where more dislocations can be accommodated. Hence, the enhanced ductility of annealed DPD 316L SS could be contributed to the large SRX grains as well as the twin boundaries with a low dislocation density.

4. Summary

A mixed structure consisting of nano-scale twin bundles embedded in micro-sized grains has been synthesized in a 316L SS by means of DPD followed by thermal annealing. This 316L SS exhibits a high tensile strength of 1001 MPa and a high elongation-to-failure (~23%). The elevated strength originates from the presence of a considerable number of the nano-twin bundles which possess a much high strength due to the significant effect of the nano-scale TB strengthening. The high ductility can be attributed primarily to the recrystallized grains and TBs with a low dislocation density. The superior strength-ductility combination achieved in the nano-twins-strengthened austenite steel demonstrates a novel approach for optimizing the mechanical properties in engineering materials.

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