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Tensile ductility of nanotwinned austenitic grains in an austenitic steel

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Dynamic plastic deformation followed by recovery annealing of an austenitic stainless steel results in the formation of a hierarchical microstructure consisting of nanotwinned austenitic grains (>55 vol.%) mixed with nanograins and dislocation structures. The sample exhibits a yield strength of 1055 MPa and a uniform elongation of ~5.2% with a considerable work hardening. Such a remarkable tensile ductility originates from the intrinsic plasticity of the nanotwinned austenitic grains in which dislocation density is reduced after the recovery annealing.

Keywords: Nanotwins; Austenitic steel; Ductility; Recovery; Stainless steel

Over the past few years, nanotwinned metals and alloys have attracted considerable attention due to their excellent mechanical properties [1–4]. Electrodeposited nanotwinned copper with a twin thickness of 15 nm exhibits an ultra-high strength of 1 GPa together with a uniform elongation of ~8.0% [1]. This originates from the fact that twin boundaries (TBs) not only serve as effective barriers to dislocation motion, but also provide ample space for dislocation storage to accommodate plastic strains.

Based on the nanotwin strengthening mechanism, a novel approach is proposed for strengthening austenitic steels by introducing nanotwinned austenite (nt-γ) grains by means of plastic deformation followed by thermal annealing [5–8]. Compared with martensitic steels and dual-phase steels, the nanotwinned austenitic steels exhibit a superior strength–ductility synergy [7]. The strength and ductility of nt-γ grains are governing factors determining the mechanical properties of the nanotwinned steels. A previous study indicated a very high yield strength up to 2.0 GPa for nt-γ grains (even stronger than martensite) [5,6], but their ductility is not yet known.

In this work, we synthesized a bulk nanostructured AISI 316L stainless steel (SS) sample containing a large volume fraction nt-γ grains by means of dynamic plastic deformation (DPD). The sample presents a considerable improvement in ductility and work-hardening ability without a pronounced loss of strength after a recovery annealing. The objective of this work is to study the ductility of nt-γ grains under tensile testing.

The material used in this work is a commercial AISI 316L SS with a composition of Fe–16.42Cr–11.24Ni–2.12Mo–0.02C–0.37Si–1.42Mn–0.011S–0.040P (wt.%). The as-received steel samples are annealed at 1200 °C for 1 h to obtain fully austenitic coarse grains (average size ~100 μm). The cylindrical samples were processed by using DPD at room temperature with a strain of $\varepsilon = 0.8$. The DPD setup and processing parameters are described elsewhere [9]. Microstructure characterization was performed by field emission gun scanning electron microscopy (SEM) in a FEI Nova NanoSEM 430 microscope with electron channeling contrast (ECC) imaging, and by transmission electron microscopy (TEM) using a JEOL 2010 high-resolution transmission electron microscope operating at 200 kV.

The microstructure of as-DPD 316L samples is mainly composed of deformation twins. As shown in Figure 1a, numerous parallel strips are found in most grains. Some parallel strips are cut by distinct bands.

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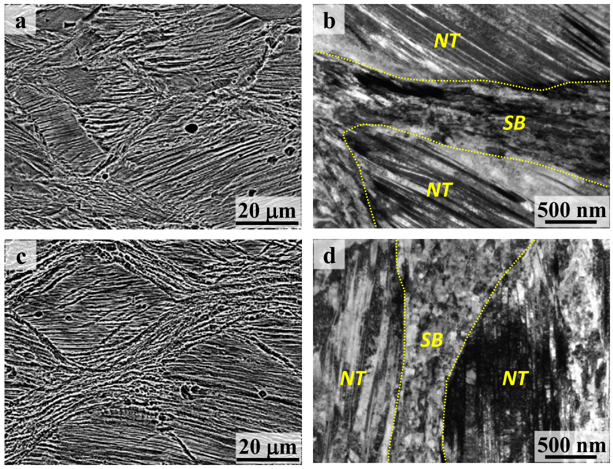

Fig. 1. Typical cross-sectional microstructures of the as-DPD 316L samples: (a) SEM-ECC image; (b) TEM image. The typical microstructures of the recovered DPD 316L samples at 700 °C for 30 min: (c) SEM-ECC image and (d) TEM image. The nanotwins and shear bands are labeled "NT" and "SB", respectively, in the TEM images.

TEM observations (Fig. 1b) reveal that these parallel strips are nanoscale deformation twins in the form of bundles in the austenitic grains which are referred to as nt-γ grains. The intersecting bands are shear bands, resulting in the formation of nanosized grains [10]. Some nt-γ grains are cut into rhombic blocks with sizes ranging from several micrometers to 100 μm (comparable to the original grain sizes). Statistical TEM measurements show that the samples are composed of 56 ± 4 vol.% nt-γ grains mixed with 24 ± 4 vol.% dislocation structures and ~20 vol.% nanosized grains. The average twin/matrix (T/M) lamellar thickness is 22 nm and the mean transverse size of the nanograins is ~40 nm. Most dislocation structures are typical dislocation tangles and dislocation cells which are not uncommon in deformed 316L SSs [11].

The as-DPD samples are annealed for recovery at 700 °C for 30 min. The annealing temperature and duration were determined from the differential scanning calorimetry results of as-DPD samples and our previous work [5]. The recovered microstructure (Fig. 1c) is analogous to that in the deformed state, without forming any static recrystallized (SRX) grains, even in the shear bands (Fig. 1d), where SRX grains are preferentially nucleated [5,12]. Statistical TEM measurements (Table 1) indicate almost no change in constitution and characteristic size between the recovered DPD samples and the as-DPD ones.

The primary microstructure difference between the as-DPD and the recovered samples is an obvious reduction in dislocation density. In terms of the quantitative X-ray diffraction analysis, the microstrain calculated from line broadening of the diffraction peaks by the Scherrer and Wilson methods [13] is about $0.132 \pm 0.004\%$ in the recovered sample, which is much lower than that in the as-DPD sample, $0.247 \pm 0.011\%$. The dislocation density ρ can be calculated in terms of grain size d_{XRD} and microstrain $<\varepsilon^2>^{1/2}$ as follows [13]:

$$\rho = 2\sqrt{3} \cdot \frac{<\varepsilon^2>^{\frac{1}{2}}}{d_{XRD} \cdot \mathbf{b}}$$

where **b** is the Burgers vector (0.258 nm for the present alloy [14]). The estimated dislocation density is $\sim 1.68 \times 10^{15}\ \mathrm{m}^{-2}$ in the as-DPD samples, and $\sim 5.49 \times 10^{14}\ \mathrm{m}^{-2}$ after recovery annealing. The significant decrease in total dislocation density after recovery annealing may originate from an annihilation of dislocations in the nt-γ grains as well as in the dislocation structures and/or nanograins.

The decrease in dislocation density in nt-γ grains after recovery annealing can be demonstrated by TEM observations, as shown in Figure 2. The dislocation density is too high to image in the nanoscale twins in the as-DPD state by bright-field TEM observations (Fig. 2a). In the recovered samples, however, the TBs become clearer due to an obvious reduction in dislocation density in the T/M lamella and at TBs (Fig. 2b). As nt-γ grains constitute the majority (56 vol.%) of the sample, the drop in dislocation density in the nt-γ grains may play a dominant part in the overall decrease in dislocation density. Annihilation of dislocations inside the nt-γ grains can occur via interactions between mobile dislocations confined within fine T/M lamella (especially Shockley partials) and the high density of TBs or sessile dislocations [15].

Uniaxial tensile tests at a strain rate of 5×10^{-3} at room temperature were performed on an Instron 5848 microtester system equipped with a contactless MTS LX 300 laser extensometer to measure the tensile strain in the sample gauge upon loading. The tensile specimens were cut into a dog-bone shape with a gauge section of $5 \times 1 \times 0.5\ \mathrm{mm}^3$. As shown in Figure 3, an obvious increase in uniform elongation (from 1.4 to $5.2 \pm 0.5\%$) is observed after recovery annealing of the as-DPD samples. However, a slight drop in yield strength (from 1186 to 1055 MPa) is induced by the annealing, which is mainly due to the reduction of dislocation density and the coarsened nanograins in the recovered samples. That is, the tensile uniform elongation is elevated several-fold at the expense of a slight loss in yield strength.

Such a large increment in tensile ductility after recovery annealing is distinct from that in ultrafine or nanograined metals formed via plastic deformation [16–18]. It is known that recovered ultrafine or nanosized grains cannot sustain a uniform tensile ductility due to their limited work-hardening capacity [16,17]. Our previous study [5] indicated that the recovered DPD 316L samples (with a DPD strain of 1.6) with ~20 vol.% nt-γ grains and ~70 vol.% nanograins and dislocation structures exhibited no increment in uniform elongation, relative to the as-DPD samples. This implies that the recovery annealing may not regain tensile ductility and work hardening in the nanograins and dislocation structures. In contrast, nt-γ grains can sustain considerable ductility after recovery annealing.

Generally, nanotwinned structures generated via plastic deformation exhibit very limited tensile ductility due to the high density of dislocations within them (e.g. $\sim 1.7 \times 10^{16}\ \mathrm{m}^{-2}$ in the as-DPD Cu [19]). The ability of nanoscale deformation twins to accommodate dislocations and work hardening is exhausted in subsequent plastic deformation. However, when deformation-induced nanotwins are recovered by thermal annealing so that the dislocation density within the