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Strength and ductility of 316L austenitic stainless steel strengthened by nano-scale twin bundles

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Abstract

By means of dynamic plastic deformation (DPD) with high strain rates, a bulk nanostructured 316L austenitic stainless steel consisting of nano-sized grains embedded with bundles of nanometer-thick deformation twins was synthesized. The average transverse grain size is ~33 nm and the twin/matrix lamellar thickness is ~20 nm. The nano-twin bundles constitute ~24% in volume. The nanostructured samples exhibit a high tensile strength of ~1400 MPa but a limited ductility with a uniform elongation of ~2%. Subsequent thermal annealing of the as-DPD samples in a temperature range of 730–800 °C led to a single-phased austenite structure consisting of static recrystallized (SRX) micro-sized grains embedded with remaining nano-twin bundles and nano-grains. The annealed DPD samples exhibit an enhanced strength–ductility synergy and much more enhanced work-hardening rates than the as-deformed samples. Work-hardening rates of the annealed DPD samples can be even higher than that of the original CG sample. Tensile ductility was found to increase almost linearly with the volume fraction of SRX grains. A combination of 1.0 GPa tensile strength with an elongation-to-failure of ~27% is achieved in the annealed DPD 316L stainless steel samples.

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Keywords: Nanostructures; Nano-twin strengthening; 316L stainless steel; Dynamic plastic deformation; Strength–ductility synergy

1. Introduction

Austenitic stainless steels are widely used engineering materials in industries owing to their excellent corrosion and oxidation resistance, high work-hardening, and good formability [1,2]. One of the major drawbacks of austenitic stainless steels is their low yield strength, usually being 150–300 MPa in the annealed states, which limits their technological applications. Strengthening austenitic stainless steels without compromising other useful properties is challenging. For example, strong austenitic stainless steels containing martensite [3–6] or dispersed precipitates [7,8] are usually less ductile and less corrosion resistant [1,7]. Structural refinement is another option for strengthening, which can be induced by plastic deformation such as cold rolling (CR) [9–12], surface mechanical attribution treatment (SMAT) [13,14], equal channel angular pressing (ECAP) [15], and high pressure torsion (HPT) [16]. Although structural refinement can result in a record-high strength, ductility and work-hardening are decreased considerably. Chen et al. [14] produced a nanostructured 316L steel by using SMAT with a yield strength as high as 1450 MPa, but its work-hardening diminishes and elongation-to-failure drops to only ~3%. In fact, the plastic straining induced ductility loss is more pronounced than the strength gain for the austenitic stainless steels, exhibiting a typical “banana-shaped” strength–ductility trade-off [17]. In addition, martensite phases are frequently induced by plastic deformation in austenitic steels, which is detrimental to ductility and corrosion resistance.

One approach is to strengthen austenitic stainless steels by means of grain refinement together with the phase-transformation-induced plasticity (TRIP) [7,18]. In this
conceived approach, the metastable austenite steels are heavily cold rolled to form a large amount of strain-induced martensite. Then, thermal annealing reverts the deformed martensite back to austenite, forming a nano- or ultrafine-grained austenitic structure [19]. During tensile tests, a strain-induced martensitic transformation occurs, leading to an enhanced strain-hardening ability and ductility [20]. Apparently, the microstructure control during the thermomechanical processes and steel compositions is crucial for the properties as the TRIP effect is sensitive to compositions.

It was recently discovered that nano-scale twins are effective in strengthening metals and alloys with a considerable plasticity [21–23]. The basic principle of nano-scale twin strengthening is that twin boundaries (TBs) are effective in blocking dislocation motion, and at the same time, they can act as slip planes to accommodate dislocations. If a grain contains a large number of TBs spaced in the nanometer regime (referred to as nano-scale twins), it can be very strong as the TBs resist dislocation motion, and meanwhile, it may have a considerable ductility as TBs provide ample room for dislocation slip and storage. Pure ultrafine-grained Cu films containing nano-scale growth twins possess of ~1.0 GPa tensile strength together with a tensile strain of 13% [24]. Bulk Cu and Cu alloys containing nano-scale deformation twins processed by means of dynamic plastic deformation (DPD) at cryogenic temperatures exhibit much higher strength than the ultrafine-grained counterparts without twins [25,26]. For the nano-twin strengthening, a general trend was found that with a decreasing twin thickness beyond a critical value, strength increases together with an increasing ductility and work-hardening ability [27]. The twin thickness in the DPD Cu alloys decreases obviously with a decreasing stacking fault energy (SFE) [26].

The objective of the present work is to strengthen austenitic stainless steels by using nano-scale twins. It has been reported that nano-grained structures embedded with nano-scale twins in the form of bundles can be produced by means of DPD in face-centered cubic (fcc) metals with medium/low SFEs. Subsequent thermal annealing may generate a unique structure with recrystallized grains embedded with nano-twin bundles which are thermally more stable than the nano-sized grains [28]. In this work we will take an AISI 316L austenitic stainless steel as an example to generate such a hierarchical structure, in which the nano-twin bundles with an austenitic structure may be regarded as “hard grains” to strengthen coarse-grained austenite structure. Our preliminary results on an enhanced strength-ductility synergy in the 316L were reported in Ref. [29]. The present paper reports a comprehensive study.

2. Experimental

2.1. Sample

The material studied is a commercial AISI 316L austenitic stainless with chemical compositions listed in Table 1. The as-received steel samples are cylinder bars 50 mm in diameter, hot-forged and solution-heat-treated at 1200 °C for 1 h and water-quenched. The typical microstructure of the as-received sample, as shown in Fig. 1a, shows roughly equiaxed grains with an average size of ~100 μm; in many of them annealing twins are visible. Some inclusions of MnS or FeS are detected in cross-sectional observations after mechanical polishing.

2.2. Dynamic plastic deformation and thermal annealing

The as-received 316L samples were processed by means of DPD. The DPD facility set-up and processing procedures are described in Refs. [30,31]. The basic principle of DPD process is to compress a bulk sample at a very high strain rate (up to 10³ s⁻¹) with a small plastic strain. In the present work, cylinder samples with a diameter of 15 mm and a height of 25 mm were DPD processed at a strain rate of 10²–10³ s⁻¹ at ambient temperature. Multiple impacts (with a strain below 0.1 in each impact) were applied to deform the samples to an eventual strain of ε = 1.6. The deformation strain is defined as \( \varepsilon = \ln(L_{0}/L_f) \), where \( L_0 \) and \( L_f \) are the initial and final thicknesses of the treated sample, respectively. After the DPD treatment, the samples become 5 mm thick discs, in which no crack or pore was observed.

The as-DPD samples were annealed at 730–800 °C for 20 min for modifying their microstructures via partial recrystallization, respectively. Some samples were isothermally annealed at 750 °C for different durations. After thermal annealing the specimens were water-quenched.

2.3. Structure characterization

Microstructures of the as-DPD and the annealed samples were characterized by using optical microscopy (Leica MPS 30), scanning electron microscopy (SEM) with electron channeling contrast (ECC) imaging on a LEO SUPRA 35 microscope, and transmission electron microscopy (TEM) on a JEOL 2010 transmission electron microscope operated at 200 kV, respectively. For optical microscopic observations, the samples were mechanically polished using diamond paste and then electro-etched at

<table>
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<tr>
<th>Material</th>
<th>Composition (wt.%)</th>
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<td>Cr</td>
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<td>316L</td>
<td>16.42</td>
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Table 1: Chemical compositions of 316L stainless steel studied in this work (mass%).
room temperature in a solution of 10 g oxalic acid and 90 ml refined water (10 wt.% oxalic acid solution). The electro-etched current density is 0.6–1 A cm$^{-2}$ with a holding time of 60–90 s. Cross-sectional TEM foils were sliced parallel to the DPD loading direction and thinned to 30 μm thick by grinding. Then the cross-sectional TEM samples were prepared by using a double-jet electrolytic polishing in an electrolyte consisting of 8% perchloric acid and 92% alcohol at $C_2 H_5 Cl$.  

2.4. Mechanical property tests

The Vickers microhardness was measured on a MVK-H 300 hardness testing machine with a load of 100 g and a loading time of 10 s. The hardness value obtained was averaged from at least 20 indents for each sample. Uniaxial tensile tests were performed on an Instron 5848 Micro-Tester system with a strain rate of $5 \times 10^{-3}$ s$^{-1}$ at ambient temperature. A contactless MTS LX 300 Laser extensometer was used to measure strain in the sample gage upon loading. The tensile samples were cut into a dog-bone shape with a gage section of $5 \times 1 \times 0.5$ mm$^3$. More than three tests were performed for each datum point.

3. Results

3.1. Microstructures of the as-DPD samples

3.1.1. Optical microscopy observations of as-DPD samples with different strains

Cross-sectional microstructure evolution in the as-DPD samples with different strains was observed (Fig. 1 b–d). At small strains ($\varepsilon = 0.3$ and 0.6), numerous parallel bands were formed in some grains, which were proved to be deformation twins by TEM observations [31]. These deformation twins seem to initiate from the original grain boundaries (GBs) and terminate inside grains or at the opposite GBs. There are apparently more grains containing deformation twins at $\varepsilon = 0.6$ than that at $\varepsilon = 0.3$. At larger strains ($\varepsilon > 1.0$), the original coarse grains became blurred and could not be clearly imaged due to severe deformation. “Dark-etching” and wavy bands appeared owing to formation of shear bands [31]. At $\varepsilon = 1.6$, the “dark-etching” bands increase considerably and the deformation structures became more homogeneous (Fig. 1d). With a further increase of strains $\varepsilon > 1.6$, microstructure change occurring in the DPD samples becomes unobvious, which is consistent with the hardness variation (Fig. 2) that
shows an increasing hardness at larger strain tending to a saturation value of \(\sim 4.3\) GPa as \(\varepsilon > 1.6\).

X-ray diffraction analysis indicated that the as-DPD samples with different strains consist primarily of a single austenite phase with a very small amount of martensite (\(\sim 5\%\) in volume). This is distinct from that in deformed samples at low strain rates (quasi-static compression) in which a much larger amount of martensite phase is formed at the same strain (\(> 15\%\) in volume). It seems the DPD process with a high strain rate is effective to suppress the martensitic phase transformation, which will be discussed in Section 4.1.

3.1.2. TEM characterization of as-DPD samples with \(\varepsilon = 1.6\)

Microstructure of the as-DPD sample with \(\varepsilon = 1.6\), characterized by using TEM (Fig. 3), consists of two types of substructures: nano-scale twins in the form of bundles embedded in a matrix of nano-sized grains. The nano-twin bundles are submicrons to micrometers in thickness and several micrometers in length. Some bundles are rhomboid in shape, and seem to originate from the cutting of shear bands through the nano-scale twin bundles from different orientations. Statistical TEM measurements showed that the nano-twin bundles constitute \(\sim 24\%\) in volume. As shown in Fig. 4a, there are high-density dislocations locating at TBs, typical of deformation TBs. This is consistent with the observation in the as-DPD Cu samples in which the dislocation density at TBs is as high as \(1.7 \times 10^{16}\) m\(^{-2}\), resulting in an obvious deviation of TB misorientation from 60° [32]. The twin/matrix (T/M) lamellar thickness varies in a range from a few to 90 nm, with an average value of \(\sim 23\) nm (Fig. 4b). The T/M lamellar thickness is slightly changed with an increasing strain, being averagely 20 nm (Fig. 4c).

Most nano-sized grains are derived from fragmentation of the T/M lamellae as well as shear banding in the nano-twin bundles, analogous to that in the DPD Cu [31]. Bright- and dark-field TEM images (Fig. 5a and b) showed that most nano-sized grains are elongated. The average transverse grain size is \(\sim 33\) nm and the longitude size is \(\sim 100\) nm, respectively (Fig. 5c). Selected area electron diffraction (SAED) patterns indicate random orientations in the nano-sized grains. With an increasing strain, both the grain sizes and the aspect ratio do not show a pronounced change (Fig. 5d), supporting the formation mechanism of the nano-grains from T/M lamellae.

3.2. Structure evolution upon annealing of the DPD samples with \(\varepsilon = 1.6\)

The as-DPD samples with \(\varepsilon = 1.6\) were thermally annealed at different temperatures for 20 min. Static recrystallization (SRX) onsets in the deformed structure as annealing temperature exceeds 730 °C, and the volume fraction of SRX grains increases at higher annealing temperatures. When annealed at 730 °C, no obvious SRX grains are seen under SEM-ECC. Under TEM observations with a higher magnification (Fig. 6a and b), clean equiaxed submicron-sized SRX grains have been detected. The SRX grains are randomly oriented with uniform contrasts, a sign of low dislocation densities. Statistical TEM measurements indicated an average size of \(\sim 180\) nm and a volume fraction of \(\sim 11\%\) for SRX grains. It is interesting to note that most SRX grains are found in the nano-grained regions between nano-twin bundles (i.e., the original shear bands of the nano-twin bundles in the as-DPD state), where nucleation of the SRX grains seems to be favorable. No SRX grain was found inside the nano-twin bundles.

When annealed at 750 °C, SRX grains become larger and more obvious under SEM observations, constituting \(\sim 50\%\) in volume (Fig. 7). The SRX grains with a size distribution from 0.6 to 3.5 \(\mu\)m (averagely 1.75 \(\mu\)m) are equiaxed, dislocation-free and with random orientations. The unrecrystallized regions (see Fig. 7) consist of the remaining nano-twin bundles and nano-sized grains. It is clear that SRX proceeds inhomogeneously in the deformed sample as SRX prefers to occur in the regions with high stored energies, such as in shear bands [33] or in the nano-grained regions, see Fig. 8. The SRX grains grow at the expense of the surrounding nano-sized grains. When the nano-grains are consumed, isolated nano-twin bundles are left surrounded by SRX grains (Fig. 9). No obvious change was found in the twin thickness after annealing. The sizes of
the remaining nano-twin bundles are in the micrometer range. A slight increment in size was noticed in the remaining nano-grains after annealing, from 33 nm (as-DPD) to \( \approx 70 \) nm in the annealed samples at 750 °C for 20 min.

Annealed at 770 °C, the SRX grains grow both in volume fraction and in size, to \( \approx 90\% \) and averagely \( 2.38 \) μm, respectively. The amount of SRX grains increases to \( \approx 98\% \) when annealed at 800 °C and the unrecrystallized regions are the only remaining isolated nano-twin bundles, see Fig. 10. Variations of volume fraction and average size of SRX grains with annealing temperature are summarized in Fig. 11a.

X-ray diffraction analysis indicated that the small amount of martensite phase formed in the as-DPD sample transformed into austenite completely when annealed at 730 °C, i.e., the annealed DPD samples contain only a single austenite phase. Careful TEM examination of the nano-twin bundles in the annealed samples showed that the dislocation density in the nano-twin bundles is much lowered by annealing (Fig. 12a). There is a slight increment in the T/M lamellar thickness at higher annealing temperatures, from \( \approx 26 \) nm at 730 °C to \( 31 \) nm at 770 °C (Fig. 12b and c).

Isothermal annealing at 750 °C for different durations was carried out with the as-DPD samples and very similar observations are obtained. As summarized in Fig. 11b, both the volume fraction and average sizes of the SRX grains increase with an increasing annealing duration. With an increasing duration from 45 to 90 min, no obvious increment is seen in SRX volume fraction, indicating the remaining nano-twin bundles are rather stable at 750 °C. During the isothermal annealing, the SRX volume fraction increases with duration in a typical “S” shape, following the classical Johnson–Mehl–Avrami relation [34], while the grain size increases with duration \( (t) \) following a \( \sqrt{t} \) relation. These dependences are consistent with the conventional SRX of deformed structures in CG metals.

3.3. Mechanical properties

3.3.1. Tensile properties of the as-DPD samples with different strains

From tensile engineering stress–strain curves of the DPD samples with different strains, one may see that with an increase of DPD strains yield strength increases substantially, from \( 306 \pm 8 \) MPa (as-received) to \( 1315 \pm 65 \) MPa at \( \varepsilon = 1.6 \), see Fig. 13a. The ultimate tensile strength reaches \( 1377 \pm 79 \) MPa with \( \varepsilon = 1.6 \). Meanwhile, tensile elongation and work-hardening decrease with an increasing DPD strain. The elongation-to-failure drops from 72% to \( \approx 7\% \) at \( \varepsilon = 1.6 \), typical behaviors of deformed metals. Plotting the measured yield strength as a function of DPD strain, one may find a good agreement with the hardness data as depicted in Fig. 2.

3.3.2. Tensile properties of the annealed DPD samples

An obvious decrease in tensile yield strength and an increase in tensile ductility are observed after annealing the as-DPD samples (Fig. 13b). Annealing at 730 °C induced a slight drop in yield strength (from \( 1315 \pm 65 \) to \( 1172 \pm 87 \) MPa), accompanied with an increment in tensile elongation-to-failure by \( \approx 5\% \), but without an obvious improvement in uniform elongation. For the sample annealed at 745 °C for 20 min, yield strength drops to \( 1011 \pm 16 \) MPa, and the elongation-to-failure increases as high as \( 19\% \) with an uniform elongation of \( \approx 6.4\% \). The annealed sample at 750 °C showed a yield strength of \( 864 \pm 81 \) MPa and an elongation-to-failure of \( \approx 27\% \). By comparison of this sample with the as-DPD sample with
$\varepsilon = 0.2$, one may find that these two samples have roughly the identical elongation-to-failure, but the annealed sample has a much higher strength than the as-DPD one. Annealing at 770 °C leads to a further decrease in strength and an elevation in ductility.

Fig. 14 summarizes the tensile properties of the annealed DPD samples with different amounts of SRX grains. It is seen that yield strength and ultimate tensile strength decrease linearly with an increase SRX volume fraction, following roughly the rule-of-mixture. Interestingly, we see that both the elongation-to-failure and the uniform elongation increase linearly with an increasing volume fraction of SRX within a range of 0–98%. Such a linear variation is different from the conventional observations in the recrystallization of deformed samples [28], which will be analyzed in detail in the following section.

4. Discussion

4.1. Formation and stability of nano-twinned austenite grains

Dislocation slip and twinning are usually two major competing mechanisms in plastic deformation of metals and alloys. The dominating deformation mechanism of a metal is determined by both the deformation conditions (such as strain, strain rate, temperature) and the nature of materials such as SFE. In fcc metals twinning is facilitated by lower SFEs and/or favorable deformation conditions such as high strain rates and/or low deformation temperatures [35,36]. For 316L austenite stainless steel with a low SFE, deformation and strain-induced grain refinement during DPD with a high rate is dominated by deformation twinning. TEM observations of a large area of the as-DPD samples verified that the majority of original grains (over 90%) are deformed via twinning while very few grains are deformed via dislocation slip.

For the 316L stainless steel, a strain-induced martensitic phase transformation is frequently observed in cold rolling [9–11] and HPT [16]. After cold-rolling at 77 K with a thickness reduction of 90%, a considerable amount of martensite is formed (45 vol.%) [9]. However, in the present study, martensitic phase is hardly observed under TEM in the as-DPD samples. XRD indicated a very small amount of martensite in the as-DPD samples.

The strain-induced martensitic transformation might be suppressed by the adiabatic heating induced by the high-rate...
deformation [4,37]. The deformation process by DPD at a high rate is considered as an adiabatic process, which may induce a transient temperature rise, as calculated by [38]:

$$\Delta T = \frac{\beta}{\rho c} \int_{\varepsilon_1}^{\varepsilon} \sigma d\varepsilon$$

in which $\beta = 0.9$ (assuming 90% of work of deformation was converted to heat), $\rho$ is density and $c$ is specific heat capacity of the sample. $\varepsilon$ is the plastic strain ($\varepsilon = \ln(L_f/L_0)$; $L_f$ is the final length of the sample and $L_0$ is the initial length).

Fig. 6. Typical bright-field TEM images (a and b) of the DPD sample with $\varepsilon = 1.6$ annealed at 730 °C for 20 min showing the early stage of static recrystallization (SRX) in shear bands between nano-twin bundles.

Fig. 7. An SEM-ECC image showing the inhomogeneous SRX of the DPD sample with $\varepsilon = 1.6$ annealed at 750 °C for 20 min.

Fig. 8. A bright-field TEM image showing growth of SRX grains into the nano-sized grains in the DPD sample with $\varepsilon = 1.6$ annealed at 750 °C for 60 min.

Fig. 9. A bright-field TEM image showing a survived nano-twin bundle embedded in the SRX grains in the DPD sample annealed at 750 °C for 20 min. Inset shows a selected area (circled) diffraction pattern of the nano-twin bundle.

Fig. 10. An SEM-ECC image showing a partially recrystallized structure with embedded nano-twin bundles in the DPD sample with $\varepsilon = 1.6$ annealed at 800 °C for 20 min.
where \( \alpha \) defines the rate of shear band formation which is related to SFE and \( \beta \) represents the probability of an intersection forming \( \varepsilon \)-martensite embryo. Both \( \alpha \) and \( \beta \) are sensitive to temperature in the vicinity of room temperature. Especially, \( \beta \) decreases sharply from 2.0 to 0 when temperature increases from 0 to 50 \( ^\circ \text{C} \). Hence, a small temperature increment around room temperature may significantly suppress formation of \( \varepsilon \)-martensites. In the DPD process, the real deformation temperature is estimated to be \( \sim 40–56 \, ^\circ \text{C} \), at which the probability of an intersection forming a martensite embryo is very small (close to zero). Thereby, formation of \( \varepsilon \)-martensites is effectively suppressed by such a small adiabatic temperature rise induced by high rate deformation. Our observation is consistent with a previous study by Hecker et al. [4] in a 304 stainless steel. The amount of \( \varepsilon \)-martensites formed during uniaxial tensile tests was found to decrease from \( \sim 40\% \) at a low strain rate of \( 10^{-3} \, \text{s}^{-1} \) to \( \sim 10\% \) at \( 10^{3} \, \text{s}^{-1} \) with an average adiabatic temperature rise of \( \sim 40 \, ^\circ \text{C} \).

Formation of nano-sized grains from nano-twin bundles in the 316L sample during DPD may have two paths, analogous to that in Cu and Cu alloys [25,26,31]:

(i) Fragmentation of nano-twin bundles. Within the nano-scale T/M lamellae, interconnecting boundaries (ICBs) are formed by dislocation accumulation and rearrangement, subdividing the two-dimensional T/M lamellae into refined blocks. The orientation of these refined blocks increased with interaction of more dislocation with the ICBs or TBs. Finally, the T/M lamellae were fragmented into nano-sized grains, of which the sizes are nearly equal to the T/M lamellar thickness, which is consistent with the observations.

(ii) Shear banding in nano-twin bundles. With increasing strains, deformation twinning cannot sustain homogeneous plastic deformation and shear banding takes place and penetrates the T/M lamellae owing to stress localization, resulting in twisting and fragmenting of the T/M lamellae. Consequently, nano-sized grains with random orientations are formed within the bands. Formation mechanism of nano-sized grains within shear bands of nano-twin bundles has been intensively investigated in a Cu–Al alloy [41].

Upon annealing, the thermally activated SRX process occurs more preferentially in the nano-grained regions than in the nano-twin bundles in the as-DPD samples, as revealed by plenty of TEM images. The heterogeneity originates from the difference in stored energy in these two...
regions. Due to the much lower excess energy of TBs than the conventional high-angle GBs, the total stored energy in the nano-twin bundles is much smaller than that in the nano-grained regions. This is consistent with the previous observations [33] in which the recrystallized grains are nucleated preferentially in the shear bands. This argument is further verified by the fact that in the late stage of SRX, only nano-twin bundles are survived while nano-grains have been consumed by SRX (Fig. 10), which is in agreement with the observations of Donadille et al. [33] that recrystallization takes place in the nano-twin bundles after complete recrystallization of the nano-grained matrix.

SRX grains were found more frequently in the SBs in the initial stage of SRX. Nano-grained regions derived from shear banding of nano-twin bundles may possess higher stored energy than that from fragmented nano-twins, probably due to the high strain and large strain gradients during shear banding [41]. For the nano-grains derived from fragmentation of nano-twin bundles, a certain amount of boundaries are derived from the low-energy TBs, and hence have a lower excess energy. Therefore, SBs are more preferable for SRX than other regions in the samples.

4.2. Strengthening effect of nano-twinned austenite grains

The microstructure of the annealed DPD 316L is recognized as composites of nano-sized grains, SRX grains and nano-scale twin bundles. In the late stage of SRX in which nano-grains are completely consumed and only nano-twin bundles survive, e.g., in the samples annealed above 770 °C, yield strength can be expressed in terms of the “rule of mixture”:

$$
\sigma_y = f^\text{NT} \sigma_y^\text{NT} + f^\text{SRX} \sigma_y^\text{SRX}
$$

where $f^\text{NT}$ and $f^\text{SRX}$ are the volume fractions of the nano-twin bundles and SRX grains, respectively, and $\sigma_y^\text{NT}$ and $\sigma_y^\text{SRX}$ are their corresponding yield strength. As yield strength of SRX grains can be determined by extrapolating the plot in Fig. 14 to 100% of the SRX grains, yield strength of nano-twin bundles is then calculated by using the measured volume fractions of both components. The calculated yield strength of nano-twin bundles with an average T/M lamellar thickness $\approx 26–30$ nm is $2.03 \pm 0.14$ GPa. It is noted that thermal annealing results in a decreased dislocation density at the TBs compared to that in the as-DPD sample, which lowers the strength of the nano-twinned bundles. Hence, the strength of the nano-twin bundles in the as-DPD samples might be even higher than this value.

The strengthening effect of TBs has been recently addressed with experimental measurements as well as molecular dynamic simulations in fcc metals [21–23,42,43]. It is believed that TBs are effective in blocking dislocation motion, like conventional GBs, and a Hall–Petch-type relationship exists for TB strengthening. A rough estimation of yield strength of the nano-twin bundles with a twin thickness of $\approx 26–30$ nm, following the conventional GB strengthening Hall–Petch relation, results in a strength of $1.90 \pm 0.05$ GPa. A higher strength is expected when a high density of dislocations in the nano-twin bundles is counted. Clearly, the strength estimated from the Hall–Petch relation seems to agree reasonably with that calculated from the rule-of-mixture. Both values of the strength of nano-twin bundles are higher than that of the nano-grained structure ($\approx 1.7$ GPa for a transverse grain size of $d_T = 33$ nm in terms of the Hall–Petch relation) and martensite (in a range of 0.8–1.6 GPa depending on its carbon concentration [44]).

Work-hardening rates ($\Theta = \delta e/\delta c$) of the annealed DPD samples are compared with that of the as-received CG samples, as shown in Fig. 15a and b. It is seen from Fig. 15a...
that all samples exhibit a steep decrease in work-hardening rate when strains are below 2%, corresponding to the elastic–plastic transition. The work-hardening rates of the CG sample decrease slightly with strain after the elastic–plastic transition, from an initial value of \( H = 2400 \) MPa to 900 MPa at a strain of 48%. But the annealed DPD sample at 770°C exhibits a much higher initial work-hardening rate, \( H = 2600 \) MPa. It decreases linearly with increasing strain, to \( H = 1200 \) MPa at a strain of 24% where it crosses over with the CG line. The decreasing rate of \( H \) with strain is larger than that in the CG one. At lower annealing temperatures, initial \( H \) values drop, e.g., in the annealed sample at 730°C, the initial \( H \) is only \( 900 \) MPa, much lower than the CG sample. For the as-DPD sample, the work-hardening rate drops to zero immediately after the elastic–plastic transition, implying the as-DPD structure does not have any work-hardening capability during plastic deformation.

**Fig. 13.** Tensile engineering stress–strain curves of (a) as-DPD samples with different strains and (b) the DPD samples annealed at different temperatures for 20 min. The tensile curve of the as-received coarse-grained sample is included for comparison.

**Fig. 14.** Variations of strength and ductility with volume fraction of SRX grains in the annealed DPD 316L samples.

**Fig. 15.** Work-hardening rate \( H \) vs. true strain \( \varepsilon \) (a) and vs. reduced true stress \( (\sigma - \sigma_y) \) (b) for the DPD samples annealed at 730 °C, 750 °C, 770 °C for 20 min and the as-received CG 316L stainless steel sample. The straight lines in (b) are fitting lines according to the Kocks–Mecking model (Eq. (5)).
According to Kocks [45], work-hardening is a consequence of the change of the dislocation density during plastic deformation, i.e.,

$$d\rho = d\rho_{\text{storage}} - d\rho_{\text{recovery}}$$  \hfill (4)

where $d\rho_{\text{storage}}$ is related to dislocation storage which enhances work-hardening rate due to increasing obstacle density, and $d\rho_{\text{recovery}}$ is related to recovery of dislocations already stored, which decreases work-hardening rate. We use the Kocks–Mecking model [46,47] to analyze the variation of work-hardening rate with stress by:

$$\Theta = \Theta_0 - K\sigma$$  \hfill (5)

where $\Theta_0$ is the initial work-hardening rate and $K$ reflects the rate of dynamic recovery during plastic deformation. The fitting results of the $\Theta$–$\sigma$ curves (Fig. 15b) are listed in Table 2. Clearly, with increasing annealing temperature from 730 to 770 °C, $K$ value decreases, i.e., the dynamic recovery rate decreases during tension.

The high initial $\Theta$ and small $K$ value in the 770 °C-annealed DPD sample originates from its unique micro-structure: micro-sized SRX grains with a very low density of dislocations embedded with nano-twin bundles (10% in volume). The SRX grains are much smaller than those in the as-received CG sample, providing a higher initial work-hardening rate. The nano-twin bundles, which are much stronger than the matrix of SRX grains, may provide additional strain gradient hardening with geometrical necessary dislocations as they may be regarded as hard “inclusions” in plastic deformation [48]. In addition, dislocation density in the nano-twin bundles is reduced by thermal annealing. Thereby, they may regain work-hardening capability as well by accommodating more dislocations at TBs or in T/M lamellae. Interactions of dislocations with TBs may generate glissile Shockley partials on the TBs [42]. The high density of TBs in the nano-twin bundles offers plenty of room for motion of glissile partials, facilitating straining and work-hardening. At lower annealing temperatures, the initial $\Theta$ drops and dynamic recovery rate increases as the volume fraction of SRX grains is reduced and the dislocation density in the unrecrystallized regions (including nano-grains and nano-twin bundles) is higher. Investigations on the detail deformation mechanism of the annealed samples are in progress.

Previous investigations [28] indicated that upon annealing nano-grained and ultrafine-grained Cu samples, no obvious enhancement in elongation is achieved when the volume fraction of SRX grains is below 50%. An obvious increment in the uniform elongation is noticed in both sets of annealed samples only when volume fraction of SRX grains exceeds ~80%. This means that only when the volume fraction of SRX grains is sufficiently large, so that the ductile SRX grains can be networked to effectively transfer plastic strain, can a sustaining uniform elongation be achieved without necking. However, in the present samples, an obvious increment in uniform elongation is achieved when volume fraction of SRX grains is only 22%. In fact, the measured uniform elongation increases linearly with an increasing volume fraction of SRX grains, which is in distinct contrast to that in the annealed nano-grained and ultrafine-grained Cu samples. It implies that the network of ductile grains can be formed when the amount of SRX grains is much smaller than that in the Cu specimens. This can be understood by the fact that the as-DPD 316L sample contains a considerable amount of nano-twin bundles that are ductile and may act as ductile “grains”. Upon annealing, we found that SRX grains

![Table 2](image)

Table 2 Work-hardening parameters ($\Theta$ and $K$) calculated in terms of Kocks–Mecking model (Eq. (5)) for different samples.

<table>
<thead>
<tr>
<th>Sample</th>
<th>$\Theta_0$ (MPa)</th>
<th>$K$</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-received CG</td>
<td>1664.4</td>
<td>1.05</td>
</tr>
<tr>
<td>DPD sample annealed 770 °C</td>
<td>2890.6</td>
<td>3.44</td>
</tr>
<tr>
<td>DPD sample annealed 750 °C</td>
<td>2433.5</td>
<td>5.93</td>
</tr>
<tr>
<td>DPD sample annealed 730 °C</td>
<td>2592.2</td>
<td>27.78</td>
</tr>
</tbody>
</table>

**Fig. 16.** The correlations of yield strength and uniform elongation (a) and ultimate tensile strength and elongation-to-failure (b) for the as-DPD and the annealed DPD 316L samples. Some literature data of 316L stainless steels and of martensitic stainless steels (highlighted) are included for comparison.
were formed preferably in the shear banding regions between these nano-twin bundles; hence the ductile nano-twin bundles are connected by the ductile SRX grains formed in the early stage, as in Fig. 6. Therefore, a network of ductile “grains” (SRX grains and nano-twin bundles) can be readily formed at a small volume fraction of SRX grains.

4.3. Enhanced strength–ductility synergy in nano-twin-strengthened austenite

Analyses above indicated that the nano-twin bundles act as a crucial strengthening component in the as-DPD 316L sample, which is very strong and ductile as well. Due to preferential formation of SRX grains in shear banding regions between the nano-twin bundles in the early stage of SRX, a ductile network may form with a small amount of SRX grains. Hence, tensile ductility increases obviously in the early stage of SRX when strength of the sample is very high. That means an improved strength–ductility synergy is achieved in the annealed DPD 316L sample.

Plotting yield strength vs. uniform elongation and ultimate tensile strength (UTS) vs. elongation-to-failure for the annealed DPD 316L sample, as shown in Fig. 16, one may find a strength–ductility synergy distinct from that of deformed samples by using various techniques. In both plots, the strength–ductility data of the annealed DPD samples are shifted up-rightward relative to that of the deformed samples. For instance, in the annealed DPD samples, a uniform elongation can be as high as 15% at a yield strength of 900 MPa, while for the as-deformed samples, it is only 2% at the same strength level. At a UTS of 1.0 GPa, the annealed DPD 316L sample may have an elongation-to-failure of ~27%, but the as-deformed sample has only ~10%. Evidently, a much better strength–ductility synergy is achieved in the annealed DPD 316L sample than that in the plastically strained samples, which can be attributed to the unique strengthening effect of nano-twin bundles generated during the DPD process. As shown in Fig. 16, the strength–ductility synergy of the 316L austenitic stainless steels strengthened by the nano-twins is also obviously better than the reported data of the martensitic stainless steels [1,49].

2. Thermal annealing induces static recrystallization of the deformed nanostructures in the as-DPD 316L samples. Upon annealing, SRX grains are formed preferably in the shear banding regions between nano-twin bundles, while the nano-twin bundles are thermally more stable. The sizes of SRX grains range from submicrons to several micrometers. The partially recrystallized nanostructured samples are of single austenite and with a mixed structure of micro-sized SRX grains, nano-sized grains, and nano-twin bundles. They exhibit an enhanced strength–ductility synergy compared to the deformed samples as well as martensitic stainless steels. Work-hardening rates of the annealed DPD samples can be even higher than that of the original CG sample. Tensile ductility was found to increase linearly with the volume fraction of SRX grains. A combination of 1.0 GPa UTS with an elongation-to-failure of ~27% is achieved in the annealed DPD 316L stainless steel sample.

5. Conclusions

1. By means of the DPD technique, we synthesized nanostructured AISI 316L stainless steel samples consisting of nano-sized grains (with an average transverse size of ~33 nm) embedded with nano-twin bundles of which the T/M lamellar thickness is ~20 nm. Martensitic phase transformation in the steel samples is suppressed by the high rate deformation. The nano-twin bundles constitute ~24% in volume. The nanostructured steel samples exhibit a high tensile strength of ~1400 MPa but a limited tensile ductility (with a uniform elongation of ~2%).

References
