Comparison of strength–ductility combinations between nanotwinned austenite and martensite–austenite stainless steels

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ABSTRACT

Two types of austenitic stainless steel samples were prepared by means of dynamic plastic deformation followed by annealing: nanotwinned austenitic grains embedded in recrystallized austenite matrix and martensitic/austenitic duplex microstructures. Annealing at 923 K induced martensitic reversion while most nanotwinned grains are stable. An enhanced strength–ductility combination is observed in the annealed nanotwinned samples which exhibit a uniform elongation of ~21% and a yield strength of ~900 MPa, in contrast to a uniform elongation of ~12% with comparable strength in the martensitic/austenitic samples.

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1. Introduction

Martensitic transformation and deformation twinning are two major competing mechanisms in plastic deformation of an AISI 304 austenitic stainless steel (SS). Strain-induced martensitic transformation (SIMT) dominates plastic deformation of cold-worked 304 SS at room temperature or below [1–5]. For example, martensite constitutes beyond 50% in volume for 304 SS under cold rolling at room temperature [1,2], and even as high as 80–100 vol% in the steel deformed by severe plastic deformation (SPD) techniques [3,4]. Introduction of a large amount of martensites can significantly enhance strength to a level of 1.4 GPa [5].

Recently, a novel strategy is proposed for strengthening austenitic steels by introducing nanotwinned austenite (nt-γ) grains which contain multiple twins with twin boundaries (TB) spaced in the nanometer regime [6–9]. These nt-γ grains are strong, ductile and elastically homogenous with the matrix. Moreover, they can co-deform homogeneously in conjunction with the surrounding matrix without generating notable strain localization near their interfaces at some tensile strains of ~5% [10]. Therefore, the nanotwinned austenitic steels exhibited an excellent combination of strength and ductility.

In the present work, a single phase nanotwinned/recrystallized duplex microstructured austenitic 304 samples (hereafter referred to as nanotwinned austenite 304 SS) and a dual phase martensitic/austenitic 304 samples (hereafter referred to as martensite–austenite 304 SS) were synthesized by means of dynamic plastic deformation followed by subsequent thermal annealing, respectively. The objective of this work is to compare the mechanical properties between these two types of samples.

2. Experimental

A commercial AISI 304 stainless steel with a composition of Fe–18.46Cr–8.28Ni–0.012Mo–0.049C–0.42Si–1.64Mn–0.003S–0.021P (wt%) is used in this work. The as-received samples were annealed at 1473 K for 2 h followed by air cooling to obtain fully austenitic coarse grains (averagely ~140 μm). The cylindrical samples were processed by using dynamic plastic deformation (DPD) at 77 K (liquid nitrogen temperature, LNT) and 423 K (Warm) to various strains, respectively. The DPD set up and processing parameters are described elsewhere [11]. Microstructure characterization was performed by using a FEI Nova NanoSEM 430 microscope and a transmission electron microscope (TEM) JEOL 2010 operated at 200 kV, respectively. The DPD samples were cut into a dog-bone shape with a gage section of 5 × 1 × 0.5 mm².

3. Results

3.1. Microstructure of the as-deformed martensite–austenite 304 SS

During the LNT-DPD process, plastic deformation of the sample is dominated by SIMT. Quantitative XRD analyses indicated that...
the LNT-DPD 304 sample (with $\varepsilon=0.3$) is composed of 80 vol% $\alpha$-martensite mixed with 7 vol% $\varepsilon$-martensite and 13 vol% residual austenite. As shown in Fig. 1a, the original austenite grain boundaries are still clearly identified. Numerous parallel strips are observed inside these grains, some of which are intersected with each other. TEM observations revealed that a few parallel strips with sharp boundaries are $\varepsilon$-martensite/austenite lamellae (see the selected area electron diffraction, SAED pattern). The $\varepsilon$-martensites are very fine and only several nanometers of thickness. And some $\alpha'$-martensites nuclei formed inside the $\varepsilon$-martensite lamellae (circled in Fig. 1b). Alternatively, most of the other strips are $\alpha'$-martensites which present regular-shaped blocks and are aligned as long laths, as shown in Fig. 1c. Statistical TEM measurements indicate that the average size of the $\alpha'$-martensites is $\sim 100$ nm. Only a few $\varepsilon$-martensites are survived in the $\alpha'$-martensites region, as arrowed in Fig. 1c.

3.2. Microstructure of the as-deformed nanotwinned austenite 304 SS

Warm-DPD processing results in formation of multiple deformation twins in 304 samples. Specifically, XRD analyses indicated that the Warm-DPD 304 sample (with $\varepsilon=1.0$) is composed of only a single austenite phase without any martensite. The microstructure of this sample also contains a lot of parallel strips, as shown in Fig. 2a. These strips are proved to be high density nano-scale twins by TEM observations (Fig. 2b). Most nanotwins are parallel to each other in the form of bundles which are referred as to nt-$\gamma$ grains. The nt-$\gamma$ grains are quite large with sizes ranging from several micrometers to $\sim 140$ $\mu$m, constituting $\sim 58\%$ in volume by statistical TEM measurements. The twin/matrix (T/M) lamellae thickness is very thin, varying in a range from a few to 95 nm, with an average value of $\sim 10$ nm. High density dislocations exist at the TBs and inside the lamellae. In addition to the nt-$\gamma$ grains, the remained microstructure is massive dislocation structures in the form of tangles, walls and cells (Fig. 2c), as usually observed in the deformed 304 SS [1].

3.3. Microstructure of the annealed martensite–austenite 304 SS

Subsequent isothermal annealing at 923 K resulted in massive martensitic reversion of the martensite–austenite 304 SS. XRD analyses indicated that the volume fraction of the $\alpha'$-martensite dramatically dropped to $\sim 17$ vol% and the $\varepsilon$-martensite could not be detected in the martensite–austenite 304 SS annealed for 1 h. As shown in Fig. 3a, the parallel strips were inconsecutive and their boundaries were blurred. EBSD orientation maps of the austenitic phase (Fig. 3b) indicated that most of these strips were long austenite laths, which originated from the martensitic reversion. Most austenitic grains are very small, with sizes of only hundreds of nanometers.

3.4. Microstructure of the annealed nanotwinned austenite 304 SS

However, for the nanotwinned austenite 304 SS under the same annealing conditions, early recrystallization (SRX) is induced in the deformed structures, forming a hierarchical microstructure consisting of nt-$\gamma$ grains mixed with SRX grains and dislocation

![Fig. 1. Typical cross-sectional microstructures of the martensite–austenite 304 sample: (a) SEM-ECC image; TEM images of the: (b) austenite-$\varepsilon$-martensite lamellae and (c) linearly arranged $\alpha'$-martensites. Insets show the corresponding selected area electron diffraction (SAED) patterns (circle). The arrows in (c) indicate the residual $\varepsilon$-martensites.](image-url)
structures, as shown in Fig. 4a. Most of nt-γ grains are survived upon annealing, still constituting 50 vol%. The average T/M lamellae thickness increases gradually from ~10 nm to ~21 nm, and accompanied by a pronounced decrease in dislocation density at TBs and inside T/M lamellae (Fig. 4b). The SRX grains constitute ~10 vol% with an average size of ~2.2 μm. There is no obvious change in the configuration of dislocation structures.

3.5. Mechanical properties

As shown in Fig. 5, the yield strength of the martensite-austenite sample with ~87 vol% martensites increases significantly to 1250 ± 20 MPa, which is ~100 MPa higher than that of the nanotwinned austenite sample with ~58 vol% nt-γ grains (1135 ± 79 MPa). Subsequent annealing (at 923 K for 1 h) for both samples induces a decrease in strength accompanied by a gain in tensile ductility. Note that the two annealed samples have roughly the identical yield strength (close to ~900 MPa). But interestingly, the annealed nanotwinned austenite 304 sample exhibits an obvious increment in uniform elongation with ~21%, which is ~9% larger than that of the annealed martensite-austenite sample.

![Fig. 2. Typical microstructures of the nanotwinned austenite 304 samples: (a) SEM-ECC image; TEM images of the: (b) nanotwins and (c) dislocation structures. Insets show the corresponding selected area electron diffraction (SAED) patterns (circle).](image)

![Fig. 3. Typical microstructures of the annealed martensite-austenite 304 SS at 923 K for 1 h: (a) SEM-ECC image and (b) EBSD image showing the distribution of austenites with different orientations (black color represents α′-martensite and unresolved area).](image)
In fact, the content of α′ martensites produced by SPD treatments revert to ultrafine austenites with high density of dislocations. For the annealed nanotwinned austenite 304 SS, the hard nano-scale α′-martensites in the present steel. The slightly lower yield strength of the nanotwinned austenite 304 SS compared with that of the martensite–austenite samples is mainly due to the relatively high volume fraction of the undeveloped dislocation structures (40 vol%) of the former in the present work. The dislocation structures are hundreds of nanometers, contributing limitedly to the total strength. Hence, the high strength of the nanotwinned austenite 304 SS may be attributed to the nt-γ grains.

After annealing, the yield strength of the martensite–austenite samples reduced more remarkably than that of the nanotwinned austenite samples. For the annealed martensite–austenite samples, the hard nano-scale α′-martensites with high density of dislocations were reverted to softer submicro-scale austenites with low density dislocations, which resulted in a steep decrease in strength. For the annealed nanotwinned austenite 304 SS, although a few SRX grains are formed and the dislocation density decreased, most nt-γ grains (50 vol%) are still survived. These nt-γ grains impart the high strength of annealed nanotwinned austenite samples.

4.4. Work hardening behavior of the nt-γ grains

The excellent ductility of the annealed nanotwinned austenite sample may be attributed to its specific work hardening behavior. As shown in Fig. 6, in contrast to the classic Kocks-type work hardening behavior for the annealed martensite–austenite samples, the work hardening of the annealed nanotwinned austenite 304 SS is characterized by a multistage behavior. This phenomenon is mainly attributed to the work hardening of nt-γ grains due to the limited work hardening of ultrafine dislocation structures [15]. After the elastic–plastic transition, the annealed nanotwinned austenite 304 SS exhibits a linear decrease of work hardening behavior to the strain of ~5%. The reason is that the nt-γ grains can provide some space to store and accommodate dislocations again after thermal annealing. This is consistent with our previous result that the nt-γ grains can sustain ~5% uniform tensile strains.
in the nanotwinned 316L SS [17]. But beyond 5%, the work hardening rate of the annealed nanotwinned austenite 304 SS maintains a nearly constant value of about 1270 MPa until the strain to 23%. Detailed TEM observations showed [18] that stacking faults (SFs) and SIMT occurred in the nt-γ grains during the tensile deformation of 5–23% strains, which may significantly contribute to the work hardening rate and thus hinder deformation localization and postpone early necking.

### 4.4. Effects of nt-γ grains and martensites on the strength–ductility combinations

The above results indicated a higher thermal stability and a comparable strengthening effect of nt-γ grains in comparison with that of α'-martensites. Accordingly, the strength of the nanotwinned austenite sample decreases more slowly upon annealing at 923 K. Specifically, the annealed nt-γ grains exhibit a pronounced ductility and work hardening capability by means of dislocation accommodation as well as formation of SFs and SIMT during subsequent tensile deformation [18]. Hence, an enhanced strength–ductility combination is achieved in the annealed nanotwinned austenite 304 SS in comparison with that of annealed martensite–austenite samples. As shown in Fig. 7, plotting yield strength versus uniform elongation for the two types of annealed 304 SS, the strength–ductility combination for the annealed nanotwinned samples are shifted up-rightward relative to that of the annealed martensite–austenite samples. Specifically, the annealed nanotwinned samples with strength of ~1 GPa exhibit a sharp increase in the ductility. For the annealed nanotwinned austenite 304 SS with a low dislocation density, SFs and SIMT can be activated in the nt-γ grains before necking to accommodate the further strain. Hence, the annealed nanotwinned austenite 304 SS with a yield strength of ~891 MPa has the uniform elongation of as high as 21%, which is ~10% higher than that of the annealed martensite–austenite 304 SS with a comparable strength.

### 5. Conclusions

We prepared the dual phase martensite–austenite and the single phase nanotwinned austenite duplex microstructured 304 SS and investigated their microstructures and tensile properties, respectively. Thermal annealing induces massive martensitic reversion in the martensite–austenite 304 SS, while nt-γ grains have a better thermal stability and are survived in the nanotwinned austenite 304 SS after annealing. An enhanced strength–ductility combination is observed in the annealed nanotwinned samples which exhibit a uniform elongation of ~21% and a yield strength of ~900 MPa, in contrast to a uniform elongation of ~12% with comparable strength in the martensitic/austenitic samples.

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