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Deformation mechanisms in an austenitic single-phase duplex microstructured steel with nanotwinned grains

F.K. Yan,[a] N.R. Tao,[a] F. Archie,[b] I. Gutierrez-Urrutia,[b] D. Raabe[b] and K. Lu[a,c,*]

[a] *Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences, Shenyang 110016, People's Republic of China*

[b] *Max-Planck-Institut für Eisenforschung, Max-Planck Str. 1, D-40237 Düsseldorf, Germany*

[c] *Herbert Gleiter Institute of Nanoscience, Nanjing University of Science & Technology, Nanjing 210094, People's Republic of China*

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Abstract—A novel type of duplex microstructure is generated in a single-phase austenitic steel (AISI 316L; X2CrNiMo19-12), consisting of plastically compliant recrystallized austenitic grains as the matrix containing coarse non-recrystallized grains with a nanotwinned austenitic (*nt*-γ) structure as strengthening inclusions. This novel type of single-phase yet duplex microstructured steel exhibits an excellent combination of strength and ductility. We study the plastic co-deformation mechanisms between the nanotwinned and the recrystallized grains under tension using electron backscatter diffraction (EBSD) and transmission electron microscopy (TEM). At tensile strains below 5%, the *nt*-γ grains nearly deform homogeneously in conjunction with the surrounding statically recrystallized (SRX) grains without generating notable strain localization near their interfaces. The anisotropic plastic deformation of the *nt*-γ grains with predominant shear parallel to the twin boundaries results in a higher dislocation density in the neighboring SRX grains. As the strain exceeds 12%, localized deformation occurs within the *nt*-γ grains in the form of shear banding. A strain gradient is developed in the surrounding SRX grains as a function of distance from the *nt*-γ/SRX interface. Deformation twinning is observed in the SRX grains near the *nt*-γ grains, while away from *nt*-γ grains dislocation slip dominates the deformation. The strengthening effect of the strong and ductile *nt*-γ grains may offer a novel approach to strengthen austenitic steels and related alloys by generating a nanotwinned/recrystallized duplex microstructure.

Keywords: Nano-twinned structures; Austenitic steels; Plastic deformation mechanism; TEM and EBSD characterization

1. Introduction

The development of high-strength steels suffers from a trade-off between strength and ductility [1–3]. For example, in dual-phase (DP) steels, with an increasing fraction of hard martensite phase the strength rises at the expense of ductility [4,5]. Studies on maraging steels showed a similar phenomenon that strength rises significantly due to the formation of nanosized intermetallic precipitates, but their ductility drops markedly [6,7]. The strength–ductility relation, like that in many other metallic alloys, exhibits a typical "banana shaped" inverse trend, i.e., ductility drops more significantly than additional strength is gained [4,7,8]. This phenomenon originates in many multiphase alloys from the incompatibility in plastic deformation between the reinforcing (hard) phase and the (soft) matrix, as well as their interfaces where geometrically necessary dislocations (GNDs) and strain gradients are generated [9–12]. Also, insufficient strain-hardening reserves of the matrix material at higher loads promote such an inverse strength–ductility effect.

Recently, a novel approach was proposed for strengthening metals by means of nanoscale twins [13–16]. Nanotwinned metals and alloys have attracted considerable attention over past years due to their excellent mechanical properties and high thermal stability. The nanotwin strengthening is based on the fact that twin boundaries (TBs) not only are effective in blocking dislocations motion, but also enable dislocation slip and accumulation. Thereby, metals can be strengthened significantly while keeping ductility. For instance, ultrafine-grained Cu films containing nanoscale growth twins exhibit a strength of ~1 GPa with a tensile strain of 13% [17].

With this strengthening mechanism, austenitic steels can be strengthened by means of nanotwinned austenitic grains which are very strong (yield strength comparable to or even higher than martensite) and ductile, with high work-hardening capability [8,18–22]. Recent work in our group has demonstrated the feasibility of producing austenitic duplex-type microstructures in an AISI 316L (X2CrNiMo19–12) stainless steel by means of dynamic plastic deformation (DPD) followed by thermal annealing [18].

* Corresponding author at: Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences, Shenyang 110016, People's Republic of China.; e-mail: lu@imr.ac.cn

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The developed duplex-type microstructures consist of nanotwinned austenite (*nt*-γ) grains that are embedded in a matrix of softer recrystallized coarse-grained austenite. The novelty lies in the elastic compatibility of the two types of coexisting microstructures and the idea of using nanotwinned grains as hard inclusions for strengthening. These single-phase duplex microstructured austenite steels exhibit an excellent combination of strength and ductility. For instance, the yield strength can be as high as ~900 MPa while keeping a high uniform ductility (~15%) [18].

In contrast to conventional hard second phases or structures (e.g. carbide, martensite or bainite) to strengthen steels, nanotwinned austenitic grains possess the same elastic modulus as the austenite host matrix and do not create any phase boundary. This means that the single-phase duplex microstructure is more attractive than conventional multi-phase design concepts due to the better elastic and plastic compatibility between the two types of interacting austenitic grains (hard *nt*-γ and soft statically recrystallized (SRX)). Studying the plastic deformation mechanisms associated with such a unique type of single-phase duplex microstructure is crucial for understanding the obtained superior mechanical properties. The present work is thus aimed to reveal the plastic co-deformation mechanisms of the 316L stainless steel strengthened by nanotwinned austenitic grains by using systematic electron backscattered diffraction (EBSD) and transmission electron microscopy (TEM) characterization.

2. Experimental

2.1. Sample preparation

The material studied is a commercial AISI 316L austenitic stainless steel 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 cylinder bars 50 mm in diameter, hot forged and solution-heat-treated at 1200 °C for 1 h. The microstructure is fully austenite with an average size of ~100 μm.

Plastic deformation of the 316L samples was performed on a DPD facility at a strain rate of $10^2 - 10^3\ s^{-1}$ at room temperature. The set-up and processing procedures of the DPD facility are described in Ref. [23]. In the present work, cylinder samples with a diameter of 12 mm and a height of 9 mm were processed to an accumulative strain of $\varepsilon = 1.6$ after multiple impacts with a strain of ~0.1–0.2 in each impact. The deformation strain 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. The as-processed samples were subsequently annealed at 750°C for 45 min prior to water-quenching. Microstructure evolutions and mechanical properties of the as-DPD and the annealed-DPD 316L samples were reported previously [18].

2.2. Tensile tests

Uniaxial tensile tests were performed in an Instron 5848 Micro-Tester system with a strain rate of $5 \times 10^{-3}\ s^{-1}$ at room temperature. A contactless MTS LX 300 laser extensometer was used to measure the sample strain upon loading. Tensile specimens were cut into a dog-bone shape from the annealed disk samples with a gauge section of $5 \times 1.5 \times 1.5\ mm^3$. The annealed samples exhibit a yield strength of 642 MPa and ultimate tensile strength (UTS) of 869 MPa. Their uniform elongation and elongation-to-failure are 24.6% and 46.1%, respectively. Tensile tests were interrupted at various strains ($\varepsilon = 0.5\%, 1.8\%, 5\%, 12\%, 22\%$) for structural analysis (see Fig. 1c).

2.3. Microstructure characterization

Microstructures of the tested samples were examined by using a field emission gun scanning electron microscope (FEG-SEM) FEI Nova NanoSEM 430 and a transmission electron microscope JEOL 2010 operated at 200 kV. The cross-sectional (parallel to the tensile axis) TEM foils were sliced from the tensile samples with different strains using a fine diamond saw and thinned to a thickness of ~30 μm by grinding. The foils were thereafter fixed to Mo rings 3 mm in diameter with a hole of 0.6 mm after punching followed by thinning using double-jet electrolytic polishing in an electrolyte of 8% perchloric acid and 92% alcohol at −15°C.

The texture evolution and development of deformation (orientation) gradients were investigated by means of the EBSD technique. EBSD maps were taken in a 6500F JEOL FEG-SEM equipped with a TSL OIM EBSD system at 15 kV acceleration voltage and working distance of 15 mm. Orientation gradients through multiple grains were analyzed using grain reference orientation deviation (GROD) maps. GROD was calculated as the angular deviation from a reference point having the lowest KAM within a given grain [24,25].

3. Results

3.1. Microstructure of the annealed DPD samples

The microstructure of the annealed DPD samples (750°C for 45 min) is composed of two types of unrecrystallized regions, namely, remaining nanoscale twins in the form of bundles (referred as *nt*-γ grains) and some blocks of dislocation structures (DSs), which are embedded in the matrix of SRX grains (Fig. 1a). The SRX grains constitute ~77.0% of the total volume fraction with an average size of ~2.2 μm. Most of the SRX grains are equiaxed with a weak crystallographic texture (see Fig. 3a). The volume fraction of the DS is ~9.4%. Sizes of most *nt*-γ grains range from several to several tens of micrometers, much larger than that of SRX grains. Statistical TEM measurements indicate that the volume fraction of *nt*-γ grains is $\sim 13.6 \pm 4.1\%$. The average twin/matrix (T/M) lamellar thickness is ~23 nm. High density dislocations exist around deformation TBs, although the dislocation density is reduced significantly in comparison with that in the as-DPD state.

Bright field TEM images (Fig. 1b) showed that most SRX grains are clean and uniform in contrast with very low dislocation densities. Some annealing twins are observed in SRX grains. The interfaces between *nt*-γ grains and SRX grains are clear without detectable dislocations inside the SRX grains near the interfaces.