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Effect of cryorolling on the microstructure and tensile properties of bulk nano-austenitic stainless steel

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ABSTRACT

We report the synthesis of nanostructured austenitic AISI 304L stainless steel (SS) through cryorolling (CR) and reversion annealing in the temperature range of 700–800 °C. Severe CR at sub-zero temperature promotes twinning in γ-austenite, which transform into α'-martensite with lath thickness of 50–100 nm. Whereas, 50–300 nm size γ-grains recrystallize in nano-twinned α' through reversion annealing as confirmed by transmission electron microscopy (TEM) and electron back scattered diffraction (EBSD) imaging. The evolution of highly processable bulk nano-austenitic SS with bimodal grain size distribution on achieving high strength (~1295 MPa), large tensile ductility (~0.47), and true necking strain of 0.59, have been discussed.

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1. Introduction

Austenitic SS is one of most attractive engineering alloys due to their good corrosion resistance and malleability [1]. However, the major drawback of austenitic SS is low yield strength about ~100 MPa due to the presence of soft face centered cubic (fcc) γ-austenite phase [2,3]. The strength of the austenitic SS can be improved by grain refinement, solid solution strengthening and/or work-hardening [1]. Among all these mechanisms, the grain refinement has been achieved by adopting thermo-mechanically controlled processing (TMCP) treatment, which improves both strength up to 1200–1600 MPa and toughness of austenitic SS, simultaneously [4]. On the other hand, severe plastic deformation techniques introduce large structural defects into the bulk specimen, and produce much fine grain structure (~300 nm) than that obtained by adopting TMCP (2–5 μm) [5]. As example, nano-/ultrafine-grained austenitic SS has been synthesized by multiple compression [6,7], hydrostatic extrusion [8], equal channel angular pressing (ECAP) [9–11], and high-pressure torsion (HPT) [12–15].

Grain refinement in austenitic SS is accelerated during severe plastic deformation (SPD) due to the formation of deformation twins in metastable γ-grains, which undergo stress-induced phase transition causing rapid microstructural refinement [16–20]. On the other hand, deformation twinning and stress induced martensitic transformation in γ can be influenced by the stacking fault energy (SFE) of SS [21]. The plastic deformation of γ occurs through dislocation glide in SS for SFE ≈ 45 mJ m^{-2} [21–24]. It has been reported that direct γ→α' transformation is promoted upon straining of SS with SFE below 18 mJ m^{-2} [17,21,25]. Whereas, deformation twinning has occurred in austenitic SS with SFE in the range of 18–45 mJ m^{-2} [22–24]. Olson et al. [22] and Sato et al. [26] have proposed two major phase transformation pathways in 304SS on the basis of SFE: (i) γ-austenite→ε-martensite →α'-martensite (SFE < 18 mJ m^{-2}), and (ii) γ-austenite→twinned austenite →α'-martensite (SFE > 18 mJ m^{-2}).

The evolution of microstructure and its effect on the mechanical properties of austenitic SS upon cold rolling and subsequent annealing, have been studied by several researchers [27–29]. Ma et al. have reported that severe cold rolling in the range of 75–90% introduces 200–300 nm α'-lath in 304L SS, which finally recrystallized into 300 nm size γ-grain upon reversion annealing at 640 °C for 10 min, resulting an improvement in the yield strength from 120 MPa to 708 MPa without loss of macroscopic plasticity [27]. Kumar et al. have performed cyclic annealing at 900 °C for 45 s and 850 °C for 45 s of a 90% cold-rolled 304L SS, and have produced 300–2000 nm size γ-grains, which has shown 710 MPa yield strength and 36% tensile ductility [28]. Similarly, high yield strength of 2050 MPa with negligible plasticity (2.2%) have been achieved in a 98% cold rolled 304H SS with 50 nm size α'-subgrains. Whereas, annealing of the as-rolled specimen at 800 °C for 30 min produced 450 nm size γ-grain, which improved the ductility upto 21% with low yield strength of 670 MPa only [29].

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Recently, cryorolling (CR) has been employed to synthesize nanocrystalline alloys with low SFE [30–32]. Severe rolling at subzero temperature helps in accumulating high defect density, restricts dynamic recovery, which in turn forms enhanced nucleation sites during controlled annealing treatment resulting a finer grain structure [30–36]. It has been reported that the SFE of 304L SS is 18 mJ m^{-2} [37]. Therefore, both deformation twinning and stress induced martensitic transformation are expected to occur in 304L SS upon CR, which may promote extensive grain refinement. In this work, the effect of severe rolling at subzero temperature on the evolution of microstructure in 304L SS has been studied. A processing window has been developed on obtaining nano-austenitic SS through CR and reversion annealing. The effect of processing parameters on the evolution of bulk nanostructure and its influence on the tensile properties, have been discussed.

2. Experimental procedure

Long bars ($100 \times 75 \times 12$ mm^3) of AISI 304L austenitic SS (Fe–0.04C–20Cr–8Ni (wt%)), were solution treated (ST) at 1100 °C for 1 h followed by water quenching. The as-quenched samples were rolled at −153 °C, and specimens were collected after achieving true strain in the range of 0.2, and 1.8 at interval of 0.2, which were identified as CR0.2, CR0.4, CR0.6, CR0.8, CR1.0, CR1.2, CR1.4, CR1.6 and CR1.8, respectively. The method of CR and the processing parameters were discussed earlier [30–32]. In the present study, CR1.8 was annealed at 700 °C, 750 °C and 800 °C for 5 min followed by water-quenching, which were identified as CR1.8-700, CR1.8-750, and CR1.8-800, respectively. Structural investigation was carried out using X-ray diffraction (XRD, X′pert PRO, PANanalytical, Netherlands) with Cu-Kα radiation. The crystallite size and the lattice strain have been analyzed using Rietveld whole X-ray profile fitting technique by using Material Analysis Using Diffraction (MAUD) software [38]. It simulates a diffraction pattern by fitting a series of structural, microstructural, peak shape, width, and background parameters. The simulated XRD pattern is compared with the experimentally observed pattern, and all the variable parameters were refined by adopting an iterative least-squares procedure through minimization of the residual parameters [39]. The detail description of the analysis techniques is reported elsewhere [39]. The bulk hardness of all the CR specimens have been measured at 20 different locations using a Vickers hardness tester (Leco, LV-700, USA) at 30 kgf load for 15 s dwell time. A scanning electron microscope (SEM, ZEISS EVO 60, Germany) was used to collect the crystallographic information of the CR and annealed specimens at micrometer scale. In addition, a high-resolution transmission electron microscope (HRTEM, JEM-2100, JEOL, Japan) operated at 200 kV, was used for microstructural characterization. Dog-bone shaped specimens with a gauge section of $4 \times 2 \times 2$ mm^3 were prepared by electro-discharge machining (EDM). Tensile tests were carried out at room temperature using a universal testing machine (Tinius Olsen HSKOS, Germany) at initial strain rate of 2×10^{-3} s^{-1}. The specimens were loaded along rolling direction. Factographic studies were performed using the same SEM as mentioned earlier.

3. Results

3.1. X-ray diffraction

XRD pattern of ST consists of only fcc γ phase revealing (111)γ, (200)γ, (220)γ, (311)γ and (222)γ peaks, as shown in Fig. 1. Analysis of XRD pattern of CR0.2 revealed the coexistence of γ-austenite and α′-martensite phases pointing γ→α′ phase transformation upon CR. Whereas, peaks of α′ have been observed in XRD patterns of CR1.0 and CR1.8 without any trace of γ. On the other hand, differently annealed specimens (CR1.8-700, CR1.8-750, and CR1.8-800) show the presence of γ pointing that recrystallization of γ has been occurred upon CR and subsequent martensitic reversion annealing treatment. It should be noted that the preferred orientation of γ-peaks has been shifted from (111)γ towards (220)γ plane upon annealing, as depicted in Fig. 1.

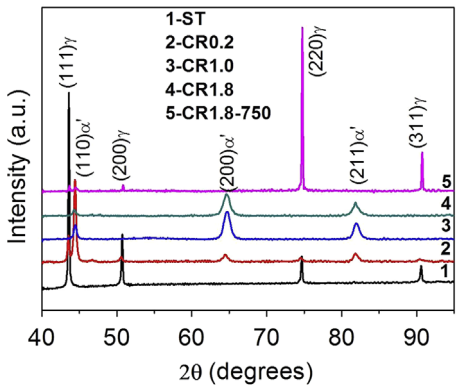

Fig. 1. XRD patterns of solution treated (ST), cryorolled up to plastic strain of 0.2 (CR0.2), 1.0 (CR1.0), 1.8 (CR1.8), and subsequently annealed at 750 °C for 5 min (CR1.8-700).

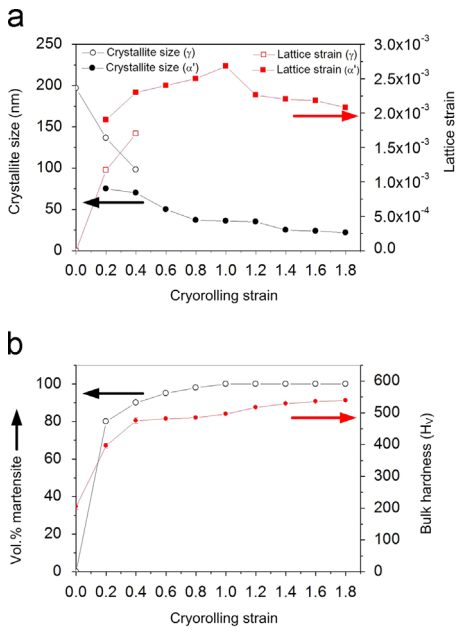

Fig. 2. (a) Variation of crystallite size and lattice strain of austenite and martensite with CR strain. (b) The variations of the vol% martensite and bulk hardness with CR strain.

The evolution of crystallite size (d), lattice strain ($\langle\varepsilon^2\rangle^{1/2}$), and vol% of martensite upon CR have been estimated qualitatively from the XRD peak broadening using Rietveld method. The estimated values of d and $\langle\varepsilon^2\rangle^{1/2}$ are plotted against CR strain, as shown in Fig. 2(a). Rietveld refinement confirmed CR0.2 to contain 80 vol.% α′. The d of γ in the ST specimen has been estimated to be 200 nm, which has been refined down to 98 nm in CR0.4. On the other hand, the $\langle\varepsilon^2\rangle^{1/2}$ of γ increases from 3.5×10^{-4} (ST) to 1.7×10^{-3} (CR0.4) during CR. XRD analysis has been revealed that the microstructure of CR0.6, CR0.8, and CR1.0 contain 95 vol%, 98 vol%, 100 vol% martensite, as shown in Fig. 2(b). No trace of any peaks from γ-austenite has been detected in the XRD patterns of CR1.0. Therefore, the d and $\langle\varepsilon^2\rangle^{1/2}$ of γ cannot be estimated using Rietveld method at CR strain ≥ 0.6. The d of α′ in CR0.2 has been measured to be 75 nm, which has been

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