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Ultrahigh strength nano/ultrafine-grained 304 stainless steel through three-stage cold rolling and annealing treatment

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ABSTRACT

An approach involving grain refinement through three-stages of cold rolling and annealing was explored with 304 austenitic stainless steel to obtain ultrahigh strength. Equiaxed nano/ultrafine austenite grains of average grain size of ~150 nm (the smallest grain size was ~80 nm) was obtained with desirable combination of strength and moderate ductility (yield strength — 1120 MPa, tensile strength — 1440 MPa, and elongation — 12%). The mechanism of transformation of martensite to austenite was diffusional reversion, when the specimens were annealed in the temperature range of 550–650 °C.

1. Introduction

304 austenitic stainless steel is one of the most frequently used stainless steel because of its strong corrosion resistance, high thermal stability, excellent weldability, and superior impact toughness [1–3]. However, the yield strength of hot rolled and annealed steel plates is low (~250–350 MPa), which restricts application.

Among different strengthening mechanisms applied to austenitic stainless steels, grain refinement is considered as the appropriate and convenient method which increases strength and retains ductility. Hence, there is increased focus on developing nano/ultrafine grained austenitic stainless steel [4–15]. One important method is severe plastic deformation (SPD) such as high pressure torsion, equal-channel angular pressing and accumulative rolling bonding [16–18]. An effective approach is advanced thermo-mechanical processing involving severe cold rolling and subsequent reversion annealing of martensite phase [1–2,4–9, 19], which can be conveniently extended to large-scale processing. This method is considered to be the most promising process to produce nano/ultrafine grained microstructure in industrial application. Actually, some austenitic stainless steels, such as 304, 301, and 201 stainless steel consist of thermodynamically metastable austenite at room temperature that is readily transformed to strain-induced martensite on deformation below M_d temperature (at which martensite is formed by 30% true strain in tension) [13,20]. The face-centered-cubic austenite phase (γ) transforms to the body-centered-cubic martensite phase (α′-martensite) during heavy cold rolling, and finally, α′-martensite reverts to γ during subsequent annealing, leading to a noticeable grain refinement. [4–9,21].

Generally, to obtain nano/ultrafine grained microstructure in austenitic stainless steels through the advanced thermo-mechanical processing, metastable γ should be completely transform to martensite during heavy cold rolling, followed by reversion annealing in the temperature range of 600 °C–900 °C for time period governed by the thickness of sheet [1,11,21–22]. Using this method, Eskandari et al. [13,20,23], Rajasekhara et al. [24], Somani et al. [19,21], and Forouzan et al. [1] have obtained nano/ultrafine grained microstructure in 301, 201, and 304 stainless steel. However, it is Misra's group that have pointed the phase reversion process and extensively studied in recent years [4–9]. They have proved that it is possible to obtain nano-grained/ultrafine-grained structure without comprising ductility at high strength.

Motivated by the studies conducted by Misra's group [4–9], we describe here a modified approach of repetitive cold working and annealing. The primary motivation of the study is to fabricate bulk nano/ultrafine grained 304 austenitic stainless steels through three-stage cold rolling and annealing treatments taking advantage of repeated phase transformation mechanism. In the premise of not entirely

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Table 1
Chemical composition of experimental 304 austenitic stainless steel in weight %.

C	Si	Mn	P	Al	Nb	V	Ni	Cr	Mo	Fe
0.055	0.40	1.63	0.030	0.015	0.04	0.08	8.45	17.30	0.12	Bal.

metastable austenite transforming into strain-induced martensite, the effects of the last stage annealing treatment on the microstructure and mechanical properties were studied. The nano/ultrafine grained 304 austenitic stainless steel sheets, 100 mm wide and 0.7 mm thick with no edge cracking, were obtained.

2. Experimental

2.1. Materials and processing

304 austenitic stainless steel of chemical composition listed in Table 1 was studied. The steel sheet of dimensions 50 mm × 60 mm × 120 mm was homogenized at 1200 °C for 3 h before hot rolling via 7 passes using Φ450 mm trial rolling mill and thickness was reduced from 50 to 4.5 mm. The start and finish rolling temperatures were 1180 °C and 1080 °C, respectively. The hot-rolled sheet was water-quenched to ambient temperature. The three-stage cold rolling and annealing treatments to obtain nano/ultrafine grains is schematically presented in Fig. 1. Multistage unidirectional cold rolling with uniform reduction in thickness was carried out at room temperature using a four-calendar rolling mill with oil lubrication, and the final thickness of 0.7 mm and total reduction of ~85%. Annealing treatments were performed in an electric furnace.

2.2. Materials characterization

Standard metallographic techniques were utilized for the preparation of specimens. Microstructure along the thickness direction was observed by LECIA DMIRM optical microscope (OM) and Zeiss Ultra 55 scanning electron microscope (SEM) after electroetching in 65% nitric acid solution. Image J software was used to estimate the reverted austenite grain size of annealed samples. Transmission electron microscopy (TEM) studies were conducted with 3 mm diameter thin foils, electropolished using a solution of 8% perchloric acid and alcohol at a potential of 32 V and −20 °C, and examined by FEI Tecnai G^2 F20 TEM at an accelerated voltage of 200 kV. Phase identification was determined by X-ray diffraction (XRD) technique using X'pert PRO diffractometer with Co K$_\alpha$ radiation at 40 kV and 40 mA. The quantitative estimate of phase volume fraction was determined using the following equation [25]:

$$V_i = \frac{1/n\sum_{j=1}^{n} I_i^j/R_i^j}{1/n\sum_{j=1}^{n} I_M^j/R_M^j + 1/n\sum_{j=1}^{n} I_A^j/R_A^j}$$

where n is the number of peaks examined, R_M and R_A are the relative intensities for three hkl selected reflections of γ and α′ phases, while I_M and I_A are the integrated intensities of the corresponding phases. Tensile test specimens were cut along the rolling direction into “dog-bone” shape with a gauge length of 10 mm, width of 4.9 mm. The uniaxial tensile test was performed at a crosshead speed of 1 mm/min using a SANS micro-force testing system.

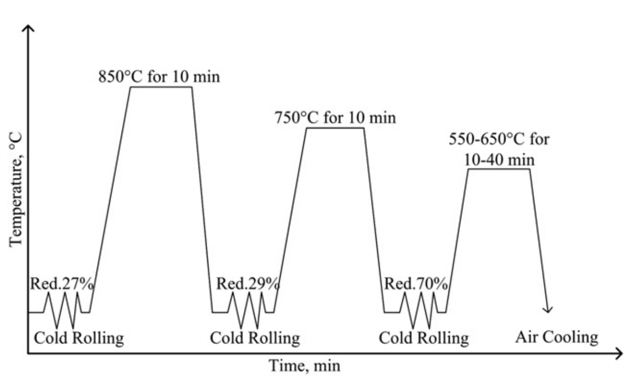

Fig. 1. Schematic diagram of three-stage cold rolling and annealing treatment.

3. Results and discussion

The optical microstructure of hot-rolled experimental steel is presented in Fig. 2. The microstructure consisted of austenite grains with an average grain size of ~18 μm. Optical micrographs of specimens subjected to first-two stages of cold rolling and annealing are presented in Fig. 3. Microstructure after the first stage of cold rolling indicated a combination of work-hardened austenite and a small percentage of strain-induced martensite (Fig. 3a). The martensite was presented as deformation bands because of high stress concentration. Microstructure of specimens annealed at 850 °C for 10 min is presented in Fig. 3b. The grain structure exhibited a bimodal distribution containing coarse grains greater than 10 μm and small grains below 5 μm. Compared with the hot-rolled austenite (Fig. 2), the grain size was significantly refined. The recrystallization temperature of 304 stainless steel was in the range of 850–1100 °C [21], hence, grain refinement can be facilitated in this annealing temperature range. After the second stage of cold rolling and annealing, the smallest austenite grain was obtained on annealing at 750 °C for 10 min (Fig. 3d). The deformation bands disappeared and the microstructure was composed of uniform, fine, and equiaxed austenite with an average grain size of ~1.8 μm. Thus, the third stage of cold rolling was initiated to obtain smaller austenite grain size. Reverted austenite was obtained after the annealing treatment.

Fig. 4 shows microstructure and XRD spectra of specimens after the third stage of cold rolling. Typical fibrous type structure normally obtained in heavily cold-worked material was observed (Fig. 4a). The microstructure consisted of lath-type martensite and dislocation-cell-type

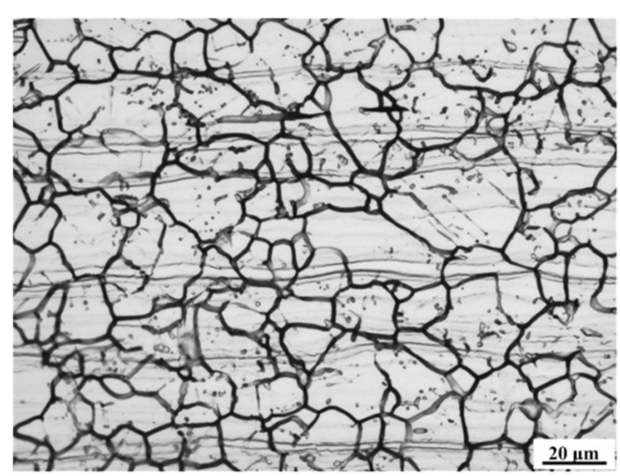

Fig. 2. The optical micrograph of hot-rolled experimental steel.