

FORMATION OF BIMODAL MICROSTRUCTURE IN A 316L-TYPE AUSTENITIC STAINLESS STEEL

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Abstract

The formation of bimodal microstructure in a 316L austenitic steel subjected to large strain cold rolling and subsequent annealing and its effect on the mechanical properties were studied. The cold rolling was accompanied by the strain-induced martensitic transformation and grain refinement. The strain-induced martensite comprised 25 % after rolling to a total strain of 3. The deformation microstructures consisted of flattened austenite / martensite grains with the transverse grain sizes of about 130 nm. The steel with nanocrystalline structure exhibited high yield strength ($\sigma_{0.2} = 1680$ MPa), but low total elongation ($\delta = 5$ %). The subsequent annealing was accompanied by austenite reversal, static recrystallization and grain growth. The annealing at a temperature of 700 °C (2 hours) led to a fully austenitic structure with bimodal grain size distribution. This bimodal microstructure consisted of individual micrometer-sized grains surrounded by the nanocrystalline matrix. The development of bimodal microstructure resulted in an increase of the total elongation above 15 % while maintaining high strength ($\sigma_{0.2} = 960$ MPa).

Keywords: Austenitic stainless steel, cold rolling, grain refinement, annealing, recrystallization

1. INTRODUCTION

Steel and alloys with a nanocrystalline structure are important materials, because they have the high strength at room temperature [1]. It has been shown that the nanocrystalline structures could be obtained in almost all metallic materials after sufficiently large strains at relatively low temperatures [2, 3]. Austenitic stainless steels are characterized by rather fast kinetic of grain refinement during cold working owing to their susceptibility to mechanical twinning and martensitic transformation [4-6]. On the other hand, austenitic stainless steels with nanocrystalline structure exhibit low ductility at room temperature [7]. Development of a bimodal microstructure, in which separate coarse grains are arranged in a nanocrystalline matrix, allows obtaining a unique combination of high strength and plasticity [8]. The aim of present paper is to introduce our current studies on the development of bimodal microstructure in 316L austenitic stainless steel by cold rolling and subsequent annealing.

2. EXPERIMENTAL

A 316L austenitic steel (Fe-0.04C-17.3Cr-10.7Ni-1.7Mn-0.4Si-0.04P-0.05S-2Mo, all in wt. %) was investigated. The steel was hot forged at 1100 °C followed by air cooling to produce an initial annealed austenite microstructure. The plate cold rolling (CR) was carried out at room temperature to total true strains of $\epsilon = 3$ (thickness reduction from 30 mm to 1.5 mm). The rolled samples were annealed at various temperatures in the range from 600 °C to 800 °C for 2 hours followed by water quenching. The microstructure characterization was performed using a Nova Nanosem 450 scanning electron microscope equipped with electron back-scatter diffraction (EBSD) analyzer on the sample sections normal to the transverse direction (TD). The transverse grain size was measured using linear intercept method on the orientation imaging microscopy (OIM) images as a distance between the high-angle boundaries (HAB), including twin-related $\Sigma 3$ CSL boundaries. The dislocation density was estimated by counting individual dislocations in the grain / subgrain interiors revealed by transmission electron microscope, JEM-2100. The martensite fractions were

averaged through X-ray analysis, magnetic induction method and EBSD technique. The mechanical properties of processed samples were evaluated by means of tensile tests using flat specimens with a gauge length of 12 mm and cross section of $3.0 \times 1.5 \text{ mm}^2$. The tensile axis was parallel to the rolling direction (RD).

3. RESULTS AND DISCUSSION

3.1. Deformation and Annealing Microstructures

The initial and deformation microstructures evolved in a 316L-type austenitic steel during cold rolling to a total strain of 3 are shown in **Figure 1**. The initial uniform microstructure consists of equiaxed austenite grains with the mean grain size of about $21 \mu\text{m}$ and a large fraction of annealing twins of ~ 0.5 . The initial microstructure is characterized by a low dislocation density of about $2 \times 10^{12} \text{ m}^{-2}$. Cold rolling results in a flattening of the original grains and the development of micro-shear bands.

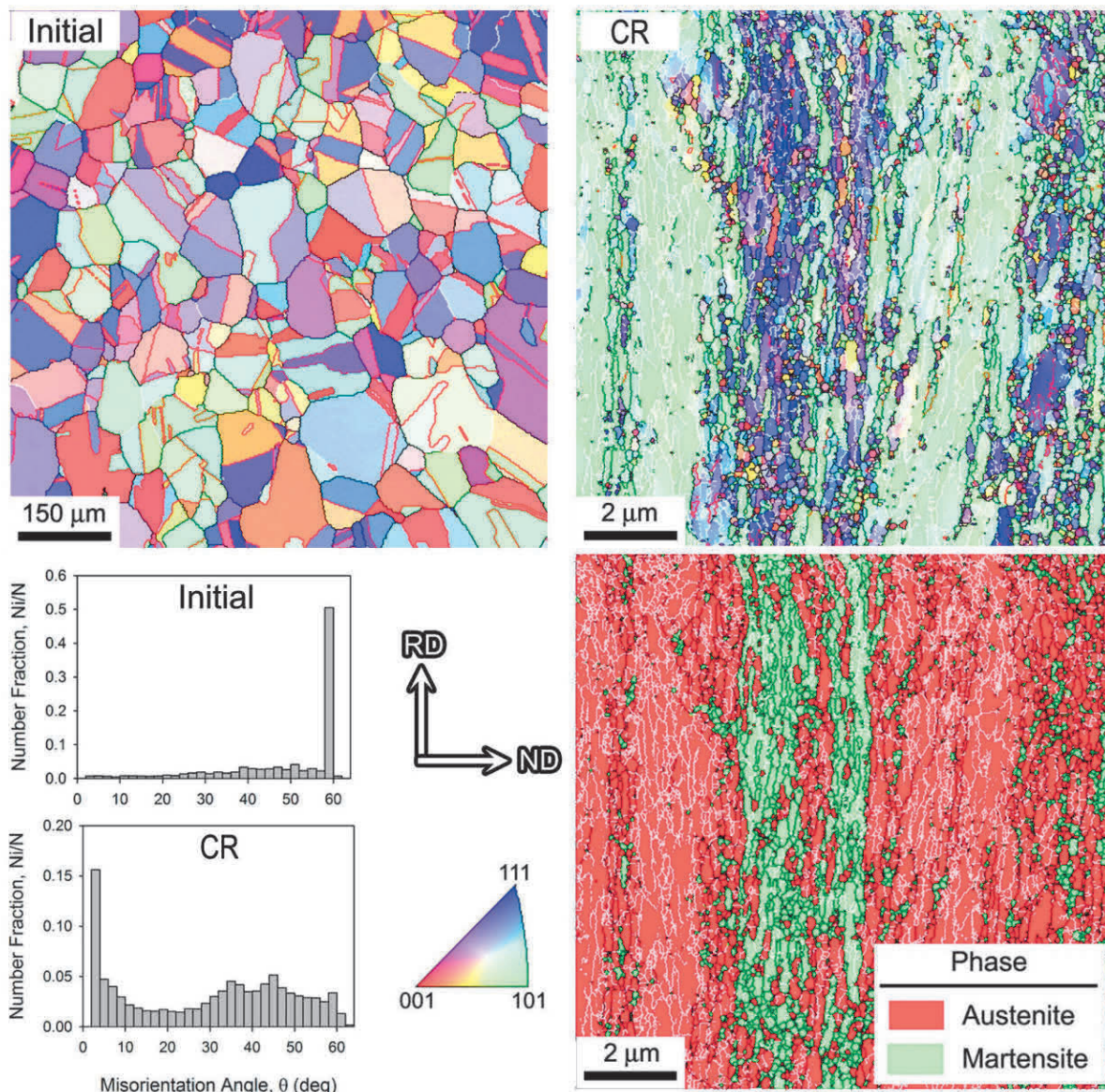


Figure 1 Initial microstructure, deformation microstructure and grain boundary misorientation distributions evolved in a 316L-type austenitic steel. The black, white and red lines indicate the high-angle, low-angle and $\Sigma 3$ CSL boundaries, respectively. The inverse pole figures are shown for the normal direction (ND)

Besides, cold rolling was accompanied by strain-induced martensitic transformation. The fraction of strain-induced martensite (bcc-martensite) comprises 0.25 in the investigated steel after cold rolling to a total strain of 3. Therefore, the cold rolled microstructure consists of flattened wavy austenitic / martensitic grains with the mean grain size of about 130 nm, which are highly elongated along the rolling direction. Cold rolling leads to an increase in the dislocation density to $5.4 \times 10^{15} \text{ m}^{-2}$ and $4.8 \times 10^{15} \text{ m}^{-2}$ in austenite and martensite nanocrystallites, respectively. The grain boundary misorientation distribution evolved at cold rolling looks like random distribution, which is superimposed with two peaks against small angles below 10° and large angles around 45° . The first peak is associated with a number of low-angle deformation sub-boundaries that are commonly brought out by plastic deformation [9]. The second peak around 45° can be attributed to martensitic transformation. The orientation relationships between austenite and martensite in stainless steels are close to those predicted by Kurdjumov-Sachs and Nishiyama-Wasserman, which result in misorientations of 42.9° and 46° , respectively [4, 10].

Typical annealing microstructures evolved in the cold rolled samples are shown in **Figure 2** and **Figure 3**. Annealing at a temperature of 600°C does not result in remarkable changes of the morphology of cold rolled microstructure (**Figure 2**). The strain-induced martensite does not completely transform to austenite at 600°C . Therefore, two-phase microstructure with about 85 % of austenite and 15 % of martensite evolves during annealing at 600°C . The presence of martensitic grains stabilizes the duplex (austenite-martensite) microstructure against grain coarsening during annealing at 600°C . The mean austenitic / martensitic grain size was about 220 nm and the dislocation density was about $3 \times 10^{15} \text{ m}^{-2}$ after annealing at a temperature of 600°C . An increase in the annealing temperatures above 600°C results in the complete transformation from strain-induced martensite to austenite followed by recrystallization of austenite.

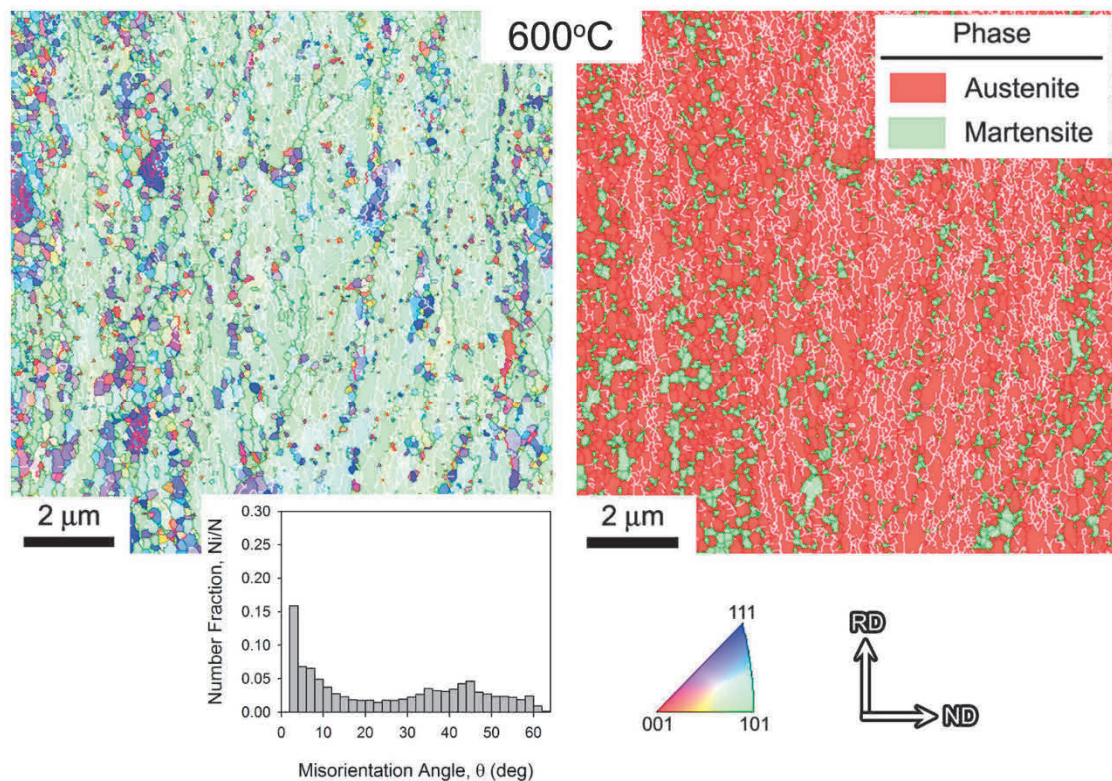


Figure 2 Typical microstructure and grain boundary misorientation distribution in a 316L-type austenitic steel subjected to cold rolling to a total strain of 3 and then annealed at a temperature of 600°C . The black, white and red lines indicate the high-angle, low-angle and $\Sigma 3$ CSL boundaries, respectively. The inverse pole figure is shown for the normal direction (ND)

Continuous recrystallization followed by normal grain growth leads to the formation of a bimodal microstructure at a temperature of 700 °C (**Figure 3**). This bimodal microstructure is composed of individual equiaxed large grains with the mean size of 1-1.5 μm surrounded by the nanocrystalline matrix with the mean grain size of about 300 nm. It is worth noting that the grain boundary misorientation distribution is also characterized by a peak against small angles below 10°, and the dislocation density did not change significantly ($\rho = 2 \times 10^{15} \text{ m}^{-2}$) after annealing at a temperature of 700 °C. An increase in the annealing temperature to 800 °C accelerates significantly the recrystallization kinetics. The uniform microstructure with a grain size of 1.2 μm develops in investigated steel after annealing at 800 °C (**Figure 3**). The rapid development of static recrystallization at 800 °C leads to an increase in the fraction of annealing twins to ~0.27 and a sharp decrease in the dislocation density ($\rho = 0.03 \times 10^{15} \text{ m}^{-2}$).

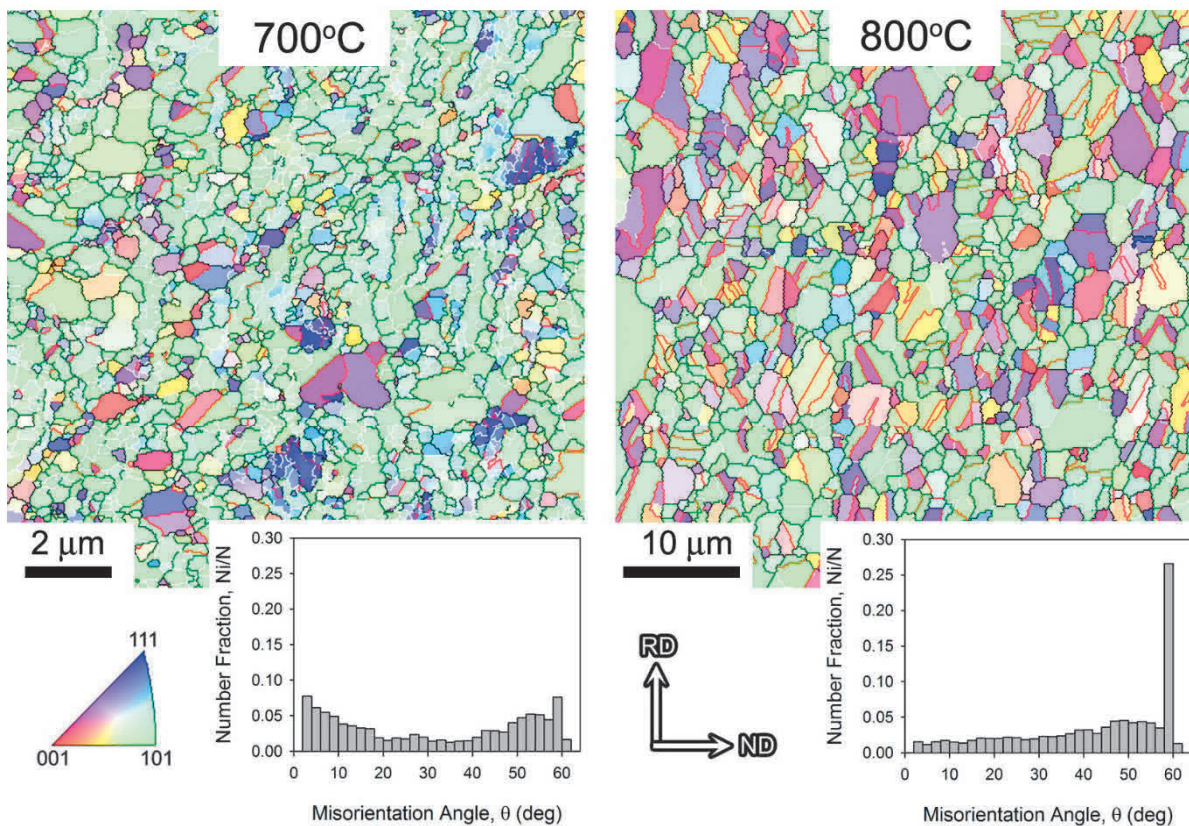


Figure 3 Typical microstructures and grain boundary misorientation distributions in a 316L-type austenitic steel subjected to cold rolling to a total strain of 3 and then annealed at temperatures of 700 °C and 800 °C. The black, white and red lines indicate the high-angle, low-angle and $\Sigma 3$ CSL boundaries, respectively. The inverse pole figures are shown for the normal direction (ND)

3.2. Mechanical properties

The engineering stress-engineering strain curves obtained by tensile tests of 316L steel subjected to cold rolling to a strain of 3 and annealing are shown in **Figure 4**. The large strain cold rolling results in a high ultimate tensile strength of above 1800 MPa and a quite low plasticity (total elongation $\delta \approx 5 \%$) in the studied steel. Subsequent heat treatment improves plasticity of severely deformed steel. The optimum combination of strength and plasticity is observed in the sample with a bimodal microstructure (after annealing at 700 °C). Namely, the yield strength was about 960 MPa, the ultimate tensile strength was about 1055 MPa and the total elongation $\delta \approx 17 \%$. Annealing at 800 °C leads to a recrystallized microstructure with a large grain size, thus, this sample has low yield strength about 530 MPa.

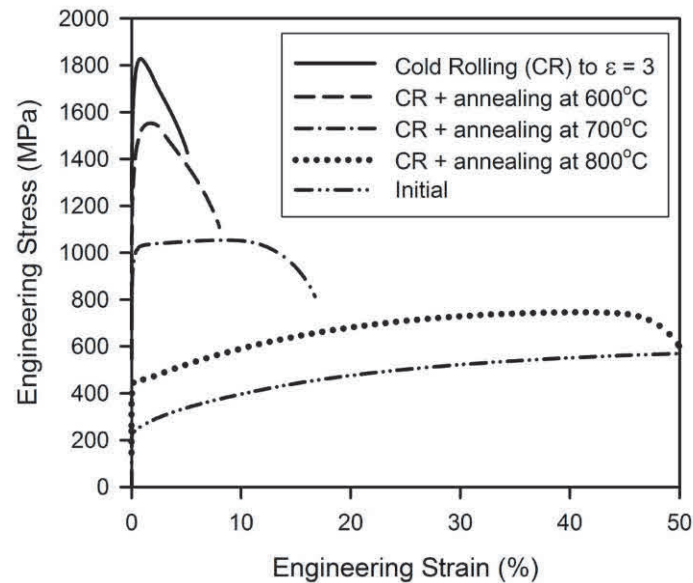


Figure 4 Tensile stress-strain curves for a 316L steel subjected to cold rolling to a strain of 3 and then annealed at the indicated temperatures

4. CONCLUSION

The microstructures and mechanical properties of a 316L austenitic stainless steel subjected to cold plate rolling and subsequent annealing were studied. The cold rolling leads to the duplex austenite-martensite microstructure with the transverse grain sizes of about 130 nm. The development of the nanocrystalline structure results in significant strengthening during cold rolling (the ultimate tensile strength of above 1800 MPa). On the other hand, the sample after cold rolling has a low plasticity (the total elongation $\delta = 5\%$). The subsequent annealing at 700 °C leads to the formation of a bimodal microstructure as a result of the development of static continuous recrystallization followed by normal grain growth. The formation of a bimodal microstructure provides an optimum combination of strength (the yield strength of 960 MPa) and plasticity (the total elongation $\delta \approx 17\%$).

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