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Effect of large strain cold rolling and subsequent annealing on microstructure and mechanical properties of an austenitic stainless steel

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ABSTRACT

The microstructural evolution of an S304H steel during bar rolling to a strain of 4 and subsequent annealing as well as its effect on the mechanical properties were investigated. The cold working was accompanied by a strain-induced martensitic transformation, leading to the development of lamellar-type microstructure consisting of highly elongated austenite/ferrite subgrains with a mean transverse size of approximately 50 nm; the austenite volume fraction was approximately 0.35. This material exhibited a yield strength above 2000 MPa. The subsequent annealing resulted in grain coarsening following the ferrite → austenite reversion, which led to almost full austenitization at temperatures above 700 °C. The formation of the austenite/ferrite lamellar structure that mixed with separate equiaxed grains occurred after annealing at temperatures of $T \leq 700$ °C. The grain coarsening was accompanied by a degradation in strength, although the yield strength of above 1000 MPa remained after 2 h of annealing at 700 °C. The discontinuous recrystallization of austenite resulted in the development of a relatively coarse-grained microstructure at $T \geq 800$ °C.

1. Introduction

Steels and alloys with nanocrystalline and submicrocrystalline structures are considered advanced engineering materials due to the favorable combination of their mechanical properties [1–5]. Among a number of proposed methods for developing nanograined structures in various metallic materials, severe plastic deformation is the most attractive technique enabling the fabrication of commercially sizeable products [6,7]. Special practical interest is aroused by the methods of severe plastic deformation that are based on conventional metal working techniques and can be used for commercial applications. According to recent studies, the total strain is the most important property for the development of nano/submicrocrystalline structures in metallic materials, whereas processing methods do not play a crucial role in the microstructural evolution [8,9]. Therefore, conventional metal-working techniques, in which one or more dimensions of the work piece is continuously reduced under processing, can be used instead of laborious, redundant, shape-change processes if the kinetics of grain refinement are fast enough for the complete evolution of the nano/submicrocrystalline structure at moderate strains.

Various metals and alloys are characterized by remarkably different kinetics of grain refinement during severe deformation [10–13]. The rapid development of nano/submicrocrystalline structures has been observed for materials allowing pronounced grain subdivision upon plastic working. Typical representatives of such materials are metastable austenitic steels [14,15]. The grain refinement in these materials is accelerated by multiple mechanical twinning and/or strain-induced phase transformation, leading to the fast development of nano/submicrocrystalline structures at relatively small strains, which can be easily attained by ordinary cold rolling. Both the twinning and the martensitic transformation depend on the stacking fault energy (SFE). Twinning has been reported to occur in steels with an SFE above 12–18 mJ m^{-2}, while the formation of martensite requires an SFE below approximately 18 mJ m^{-2} [16–19]. Therefore, austenitic stainless steels with a relatively low SFE can be processed in high-strength products by means of extensive grain refinement. The yield strength can be drastically increased from 250–350 MPa to 1200–1600 MPa by using a thermomechanical treatment consisting of cold rolling with a total reduction ranging from 50 to 90%, which generates the strain-induced martensite, followed by the phase reversion during a final annealing step [14,20–23].

The formation of ultrafine-grained structure in Cr–Ni austenitic stainless steels by the reversion from deformation-induced martensite has been studied in a number of recent studies [14,16,24–32]. The cold rolling brings about a high dislocation density along with dense deformation twins with nanoscale

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spacing. Then, the deformation-induced martensite nucleates at shear bands and deformation twins [16,31,32]. Because the austenitic stainless steels are thermodynamically metastable at room temperature, the austenite easily transforms into deformation-induced martensite with increasing strain [14,24–31,33]. Upon subsequent annealing, the martensite transforms to austenite by shear reversion and/or diffusional (nucleation and growth) reversion mechanisms [14,25,28,33]. The following set of criteria for ultra grain refinement by reversion from deformation-induced martensite was reported by Tomimura et al. [24]: (i) austenite should be almost completely transformed to martensite during cold rolling; (ii) most of the martensite must revert to austenite at a relatively low temperature to avoid grain growth in the reversed austenite; and (iii) the martensite start temperature of the reversed austenite should be below room temperature. The range of C, Ni and Cr compositions of austenitic stainless steels that meet these criteria was considered as well [24].

Recently, an S304H austenitic stainless steel was developed for high-temperature applications [34]. This steel exhibits increased strength and creep resistance with respect to conventional AISI 304 steel, which is associated with dispersion strengthening mainly by Nb(C, N) precipitation. It is expected that this steel, which exhibits nano/submicrocrystalline structure, can exhibit superior strength properties. The value of the SFE in 304-type stainless steels has been reported to be approximately 20 mJ m^{-2} [35,36]. Hence, the mechanical twinning and martensitic transformation are expected to occur concurrently during cold working. The aim of the present work is to explore the feasibility of producing nano/submicrocrystalline structures in an S304H steel by large-strain cold rolling and subsequent annealing. The regularities of microstructural evolution during deformation and annealing were examined in detail to address the relationship between the developed microstructures and the observed mechanical properties.

2. Experimental procedure

An S304H austenitic stainless steel, Fe–0.1C–0.12N–0.1Si–0.95Mn–18.4Cr–7.85Ni–2.24Cu–0.5Nb–0.01P–0.006S (all in mass%), was used as starting material. The steel was hot rolled and then annealed at 1100 °C for 10 min. The mean grain size was 10 ± 1 μm. The cold working was carried out by caliber rolling 9.2 mm × 9.2 mm square bars into 1.25 mm × 1.25 mm square bars at ambient temperature, leading to a total strain of 4. The cold worked rods were cut into specimens of 70-mm in length and annealed in a muffle furnace at temperatures ranging from 400 to 900 °C followed by water quenching.

Strain hardening and annealing softening were studied using Vickers microhardness tests with a load of 1 N. The mechanical properties were evaluated using tensile tests. The specimens with an 8-mm gauge length were prepared by an electric-discharge method. The tensile axis was parallel to the rolling axis. The mechanical tests that were conducted on samples rolled to a strain of 4 and then annealed at different temperatures were performed at ambient temperature using an Instron 5882 testing machine.

Structural investigations were performed on sections parallel to the rolling axis using a JEM-2100 transmission electron microscope (TEM) and a Quanta 600 FEG scanning electron microscope equipped with an electron back-scattering diffraction (EBSD) analyzer incorporating an orientation imaging microscopy system. In addition to EBSD, the phase content, i.e., austenite/ferrite fraction, was evaluated by magnetic force microscopy (MFM) using an NTEGRA Aura scanning probe microscope. The grain/subgrain sizes were measured perpendicular to the rolling axis by a linear-intercept method used to count all boundaries, including dislocation sub-boundaries revealed by TEM micrographs. The dislocation densities were estimated by counting individual dislocations in grain/subgrain interiors in at least six arbitrarily selected TEM images for each data point [37]. Misorientations between nanocrystallites were analyzed by the conventional TEM Kikuchi-line method using the converged-beam technique [38]. Equilibrium volume fractions of the austenite at various temperatures were calculated with the software program ThermoCalc using a TCFE6 database (300 K was used as the ambient temperature).

3. Results

3.1. Strain hardening and structural changes during cold working

The cold rolling of an S304H austenitic stainless steel results in significant hardening (Fig. 1). The most pronounced strain hardening occurs at relatively small strains. The cold rolling to a strain of 0.4 is accompanied by a twofold increase in hardness from 1695 MPa in the initial annealed state to 3275 MPa. Then, the rate of strain hardening decreases to a value of approximately 700 MPa per strain, which remains almost constant during further processing. As a result, the hardness increases almost linearly with an increasing total strain from 0.4 to 4.0. Such deformation behavior is not typical of fcc metallic materials subjected to large-strain cold working. Commonly, the rate of strain hardening gradually decreases during deformation, ultimately approaching nearly zero after sufficiently large strains are sustained [39–42]. The continuous increase in the hardness is indicative of ongoing structural strengthening.

Typical deformation microstructures that developed in an austenitic stainless steel during caliber cold rolling are shown in Fig. 2. Early deformation leads to the elongation of original grains along the direction of plastic flow and results in the development of various strain-induced boundaries, most of which are associated with deformation twinning (Fig. 2a). The number of twins increases with straining (Fig. 2b). Because the austenite in the studied steel is metastable at room temperature, a partial martensitic transformation occurs during cold rolling to strains above 1. The twinning and the martensitic transformation promote grain refinement, leading to the development of dual-phase structures consisting of austenite and ferrite grains. The former can be roughly distinguished as being red and blue (⟨1 0 0⟩ and ⟨1 1 1⟩ parallel to the rolling axis) and the latter as being green (⟨1 1 0⟩ parallel to the rolling axis) in Fig. 2c and d.

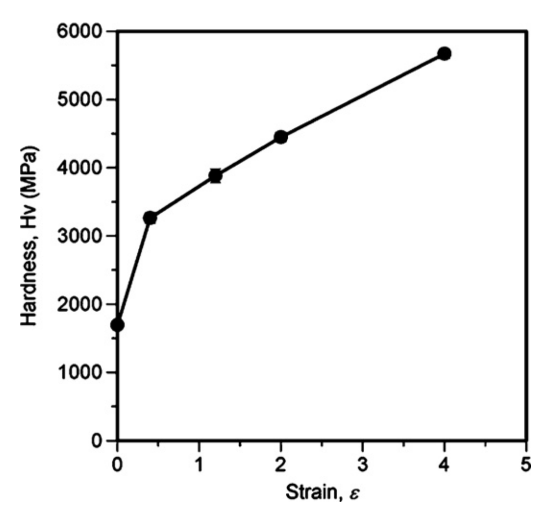

Fig. 1. Strain hardening of an S304H austenitic stainless steel subjected to large-strain cold rolling.