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A repetitive thermomechanical process to produce nano-crystalline in a metastable austenitic steel

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Abstract

Nano-crystalline grains of about 100 nm were obtained in a metastable austenitic steel by a repetitive thermomechanical process consisting of conventional cold rolling and annealing. The nano-grained austenite was transformed on annealing from the strain-induced martensite, which had formed during cold rolling. The nano-structured austenitic steel exhibits not only high strength (above 1 GPa) but also good elongation (above 30%).

Keywords: Nano-crystalline; Reverse transformation; Metastable austenite; Grain refinement

1. Introduction

In recent years severe plastic deformation (SPD) processes, such as high pressure torsion (HPT), equal channel angular pressing (ECAP), and accumulative roll-bonding [1–4], have been developed to produce bulk nano-crystalline materials. The SPD processes have successfully refined the coarse grains of pure metals and alloys to the grain size of a few tens to a few hundreds of nanometers. The nano-grained materials revealed very high strengths as expected. However, their elongation, especially uniform elongation, was dramatically decreased to only a few per cent because of the low strain hardening in nano-sized grains [5,6]. In addition, the present SPD processes still need some improvements in terms of materials dimensions, mass production, sample preparation, etc.

Some researchers have tried to fabricate nano-crystallines of 200–500 nm size by using a reverse transformation from strain-induced α'-martensite to γ-austenite in metastable austenitic stainless steels [7,8]. The process is characterized by heavy cold rolling to induce the γ to α'-martensitic transformation, followed by annealing for the reverse transformation of strain-induced α' to γ. The nano-crystalline exhibits both high strength and good uniform elongation, resulting from enhanced work hardening ability through the strain-induced martensitic transformation during tensile tests [7,8].

As the process is carried out only by conventional cold rolling and annealing, it seems more appropriate for large-sized sheets, and has more potential for actual applications, as compared with other SPD techniques. It is wondered, however, whether a repetitive application of the thermomechanical process, as schematically shown in Fig. 1, can reduce the grain size below 200 nm, and whether the chemical composition can be extended beyond that of stainless steel, which is normally defined as a steel containing Cr at more than 12 wt%. The mechanical properties of the resultant nano-crystalline are also of great interest. The present study has been performed to address these issues.

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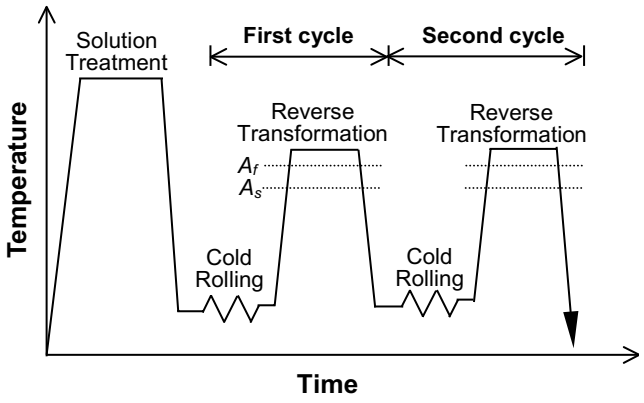

Fig. 1. A schematic illustration of a repetitive thermomechanical process to obtain the nano-crystalline in a metastable austenitic steel. A_s and A_f are the reverse transformation start and finish temperatures, respectively.

2. Experimental

An Fe-0.1%C-10%Cr-5%Ni-8%Mn alloy in nominal composition was prepared using a high frequency vacuum induction furnace. The actual chemical composition of the alloy is listed in Table 1. The Cr content of the alloy is less than 12%, which is usually regarded as a minimum Cr concentration for stainless steels. The Ni content is half of the previous one [7,8] and replaced by Mn to reduce the material cost.

The ingot was homogenized at 1200 °C for 12 h under a protective atmosphere, hot-rolled to the plates of 10 mm thick, and followed by solution treatment at 1200 °C for 30 min. Using the plates, the repetitive thermomechanical processes were performed, as shown in Fig. 1. The reverse transformation start and finish temperatures (A_s and A_f) were determined from dilatational curves measured at the rate of 10 °C/s during the heat-up of the cold-rolled sheets. The annealing for the reverse transformation was carried out at a temperature of A_f+10 °C for 10 min using a salt bath. The reduction in thickness of the first cold rolling was about 75% and the annealing temperature was 640 °C. These two parameters were 50% and 630 °C, respectively, for the second cycle.

The microstructures were observed using a transmission electron microscope (TEM). Thin foils for TEM investigation were jet-polished in a solution of 10% perchloric acid +90% ethanol at −40 °C, and observed in a JEM 2000 EX operating at 160 KV. The phase components were identified by using X-ray diffraction with Cu Kα radiation.

Table 1
Chemical composition of the steel used (wt%)

C	Ni	Cr	Mn	Fe
0.078	4.92	9.94	7.72	Balance

Tensile tests were performed at ambient temperature using an Instron 1127 machine at a crosshead speed of 2 mm/min. The tensile direction was parallel to the rolling direction. The size of the gauge part of the tensile specimen after the repetitive thermomechanical treatment was 6 mm wide, 1.0 mm thick, and 15 mm long according to the relationship of $L_0 = 5.65 \times A^{1/2}$ [9], where A is the cross-sectional area and L_0 is the length of the gauge part. The gauge length of the first reverse transformed specimen was 20 mm because the specimen thickness was about 2.2 mm then.

3. Results and discussion

3.1. Microstructure

The microstructural changes investigated by X-ray diffraction at each stage of the repetitive thermomechanical process are shown in Fig. 2. The microstructure of the solution-treated specimen consists of a mixture of γ-austenite (face-centered cubic), α′-martensite (body-centered cubic), and a little ε-martensite (hexagonal close-packed), respectively. This means that the martensitic transformation start temperature (M_s) of the solution-treated specimen is higher than the room temperature. After the first cold rolling, as shown by the X-ray pattern (B) in Fig. 2, the microstructure changed to fully α′-martensite due to the heavy cold rolling. After the first annealing, the α′-martensite reversely transformed to γ-austenite, and no peaks of martensite could be monitored from the pattern (C) of Fig. 2. This indicates that M_s temperature has dropped to below room

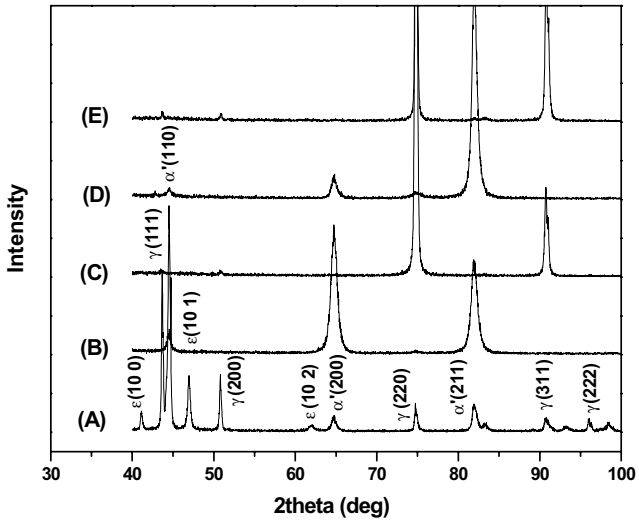

Fig. 2. Microstructural changes during the repetitive thermomechanical process: (A) after solution treatment; (B) after the first cold rolling; (C) after the first annealing; (D) after the second cold rolling; and (E) after the second annealing.