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The effects of multi-cyclic thermo-mechanical treatment on the grain refinement and tensile properties of a metastable austenitic steel

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ABSTRACT

The effects of the multi-cyclic cold rolling and annealing process on the grain refinement and tensile properties of a metastable austenitic steel were investigated. The multi-cyclic thermo-mechanical process yielded the bimodal ultrafine-grained structure through both the reverse transformation and recrystallization during annealing. The kinetics and temperatures of the reverse transformation were related to the average size of coarse grains. Not only yield and tensile strengths but also ductility of the bimodal-grained structure increased with the number of thermo-mechanical cycles.

1. Introduction

For the past a couple of decades, vigorous researches have been performed to improve the mechanical properties of metals and alloys by reducing their grain size to nanometers [1–15]. Severe plastic deformation (SPD) processes, such as equal-channel angular pressing (ECAP) [1,2], high-pressure torsion (HPT) [3], accumulative roll-bonding (ARB) [4,5], etc., have been explored for bulk nano- or ultrafine-grained (UFG) materials. The main principle of SPD processes is to break a grain into many smaller grains by generating a lot of dislocations under large shear strains. Even though SPD processes endowed various materials with superior strengths, the limited uniform elongation caused by a lack of strain hardening in heavily pre-hardened materials became a critical issue to be overcome [7].

Therefore, many studies have been conducted to improve the uniform elongation without a great loss of strength by enhancing strain hardening in UFG materials [8–10]. Koyama et al. [11] reported that deformation twins increased both the ductility and strength of the UFG Fe–17Mn-0.6C (wt.%) steel, which was fabricated by recrystallization annealing after cold rolling. Koch and Zimmerman et al. [12,13] reported that the ductility of a nanocrystalline Ni alloy was improved due to the strain hardening rate enhanced by SiC particles. Wang and Ma et al. [7,14] used a bimodal microstructure with relatively coarse recrystallized grains in the nanostructured matrix for the better ductility of UFG Al alloys and Cu alloys. In the bimodal microstructure, fine grains contributed to the high strength and coarse grains bore the high ductility.

Lee and coworkers have been studying both the thermo-mechanical process and chemical composition of alloys for phase transformation-aided grain refinement, which is called the Strain-Induced Martensitic transformation and its Reverse Transformation (SIMRT) process [15–17]. The SIMRT process fabricates ultrafine grains by annealing for the reverse transformation from strain-induced martensite to the stable high-temperature parent phase. Because ultrafine grains are diffusively nucleated and grow in the martensite matrix in most cases, they are not pre-hardened and have a low density of dislocations unlike those made by the SPD processes. The UFG alloys produced by the SIMRT process exhibited not only high strength but also good ductility because of extra-strain hardening caused by the strain-induced martensitic transformation occurring in ultrafine grains during tensile tests.

The effects of the grain size, nano-sized precipitates, strain rate, and process conditions on the martensitic transformation kinetics and tensile properties of SIMRT-processed UFG alloys were investigated [15–26]. Of them, to investigate the effect of the SIMRT process condition, the two-cyclic SIMRT process has been tried to produce the UFG structure in metastable stainless steels [27,28]. The repetitive SIMRT process improved the tensile strength without a great loss of ductility. However, a systematic examination on the multi-cyclic SIMRT process has yet to be performed, although other SPD processes adopt cyclic severe plastic deformation. Therefore, in the present study we investigated the variations

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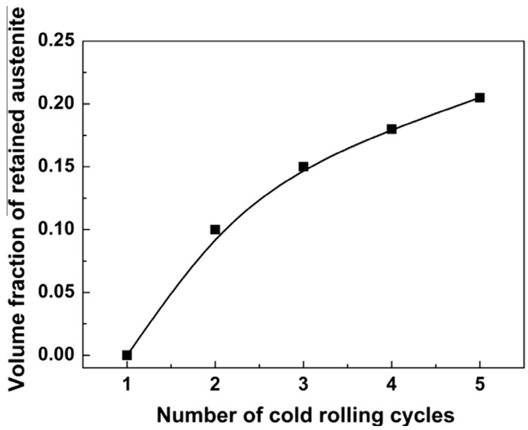

Fig. 1. The change in the volume fraction of retained austenite with the number of cold rolling cycles in a metastable austenitic steel.

in the microstructure, grain size, and tensile properties of a metastable austenitic alloy with increasing the number of SIMRT cycles to the maximum five cycles, and found different results from the previous results obtained by the two-cyclic SIMRT process.

2. Experimental procedure

An ingot of Fe–0.023C–10.9Cr–9.0Ni–7.0Mn (wt.%) alloy was prepared using a high-frequency vacuum induction furnace. This material is not a commercialized alloy and its chemical composition was determined to obtain metastable austenite at room temperature based on our previous experience. The ingot was homogenized at 1100 °C for 12 h in a protective atmosphere, hot-rolled to the 40-mm-thick plates. Based on the previous result about the relationship between the reduction in thickness of cold rolling and the volume fraction of strain-induced martensite [15,27,29], the thickness reduction of cold rolling was determined to be 50% for each SIMRT cycle. The SIMRT process was repeated five times using the hot-rolled plate with an initial thickness of 40 mm.

Cold-rolled sheets were annealed for the reverse transformation from α' martensite to γ austenite at the temperature of $A_f + 20$ °C for 10 min using a salt bath. The A_f temperature is the reverse transformation finish temperature, which was determined from a dilatometric curve measured during continuous heating to 1000 °C at 10 °C/s using a dilatometer. Dilatometric specimens were taken from cold-rolled sheets after each cycle of the SIMRT process.

Thin foil specimens for transmission–electron microscopy (TEM) were prepared from the annealed sheets after each SIMRT cycle to observe the microstructure of reverted austenite. Thin foils were jet-polished in a solution of 90% glacial acetic acid + 10% perchloric acid at 15 °C under 15–20 V and 38–40 mA. Thin foils were observed using a TEM (JEM 2000EX) operated at 200 kV. The phase change was confirmed by X-ray diffraction (XRD) tests at room temperature. The scanning range was 40–100° and a Cu target was used.

Room-temperature tensile tests were performed with a constant crosshead speed of 2 mm/min to evaluate the change in tensile properties of UFG alloys with the number of SIMRT cycles. The gauge portion of the tensile specimen was 6.25 mm wide, 1.2 mm thick, and 25 mm long according to the following equation:

$$L_0 = 5.65 \cdot A^{0.5} \quad (1)$$

where L_0 and A are the length and area of the gauge portion, respectively.

3. Results and discussion

Fig. 1 shows the change in the volume fraction of retained austenite (γ_R) with the number of cold rolling cycles. Retained austenite means untransformed austenite after cold rolling. The volume fraction of γ_R increased rapidly until three cycles, and then gradually increased to approximately 20% after five cycles. This result indicates that austenite stability against the strain-induced martensitic transformation during cold rolling was improved due to the grain refinement by the multi-cyclic SIMRT process.

Figs. 2(a and b) exhibit the TEM images of fully reverted γ after the first and the fifth SIMRT cycles, respectively. Although the first-cycled specimen had the relatively uniform grain size (Figs. 2(a)), the homogeneity of the grain size was undermined with increasing the number of SIMRT cycles so that the difference in size between fine and coarse grains increased. The average sizes of fine, coarse, and total grains were separately measured and are plotted in Fig. 2(c). The average size of total grains decreased drastically from 40 μm after hot rolling to 410 nm after the first SIMRT cycle, and then was nearly saturated to be approximately 300 nm after three cycles. Whereas the average size of fine grains reduced steadily until the fifth cycle, that of coarse grains decreased greatly until the second cycle, and then was almost unchanged with further cycles, resulting in the bimodal-grained structure after the fifth SIMRT cycle.

The bimodal-grained structure is thought to form by two different phase transformations occurring during annealing; the reverse transformation from α' martensite to γ and the recrystallization of γ_R. The recrystallization of γ_R means that the retained austenite (γ_R), which was heavily deformed by cold rolling, is recrystallized during annealing. These two different kinds of γ formations could cause different grain sizes even at the same annealing condition because of differences in driving force and kinetics between the reverse transformation and the recrystallization. However, it is difficult to determine which mechanism caused coarse grains because grain coarsening can occur in both the recrystallized γ_R and reverted γ grains during annealing.

Fig. 3 shows the variations in the kinetics and critical temperatures for the reverse transformation from α' martensite to γ as a function of the number of SIMRT cycles. Both the kinetics and critical temperature data were obtained from dilatometric curves measured during continuous heating to 1000 °C at 10 °C/s. The kinetics of the reverse transformation was converted by applying the lever rule to dilatometric curves, based on the thermal expansion lines of both γ and α' and their phase fractions before heating.

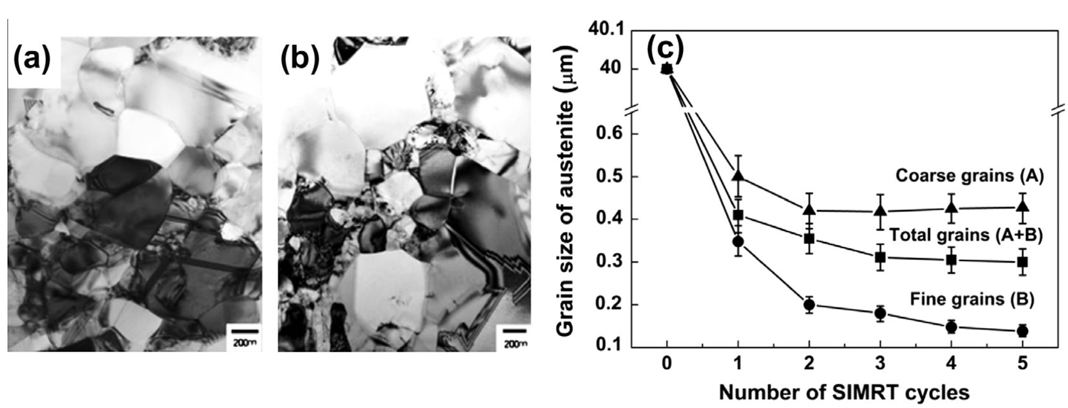

Fig. 2. TEM images showing ultrafine austenite grains fabricated by the multi-cyclic SIMRT process: (a) the first-cycled, (b) the fifth-cycled specimens and (c) the variations in the average sizes of coarse, fine, and total austenite grains with the number of SIMRT cycles.