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Fabrication of bimodal-grained Al-free medium Mn steel by double intercritical annealing and its tensile properties

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ABSTRACT

The objectives of the present study were to develop the bimodal-grained microstructure in a medium Mn steel without the addition of Al only through a thermo-mechanical process, and to systematically investigate the relationship between the bimodal-grained microstructure and yielding behavior. The cold-rolled Fe–7Mn–0.05C (wt.%) steel with deformed α' martensite was annealed twice with different temperatures and holding times. When the cold-rolled specimens were first-annealed at 660 °C for 4 h –22 h, the specimens showed a dual-phase microstructure consisting of globular-shaped ferrite (α_G) and thermally induced α' martensite at room temperature. Whereas the sizes of both α_G grains and α' martensite constituents increased with increasing the first annealing time, both the volume fraction and the chemical concentration of each phase were insignificantly changed. When the first-annealed specimens were annealed again at 640 °C for 5 min, the specimens exhibited bimodal-grained structures with coarse α_G and ultrafine lath-shaped ferrite (α_L) and retained austenite (γ_L). The coarse α_G came from α_G pre-existing in the first-annealed specimen; the fine α_L and γ_L formed by the α' martensite to γ diffusive reverse transformation. The second-annealed specimens exhibited a transition of yielding behavior from discontinuous yielding with yield-point elongation (YEL) to continuous yielding without YEL due to the coarsening of α_G grains with increasing the first annealing time up to 22 h.

1. Introduction

In recent years, medium Mn steels with less than 10 wt% Mn and 0.3 wt% C have received significant attention due to an excellent combination of reasonable materials cost and high mechanical properties [1–16]. The medium Mn steels normally exhibit an α' martensite microstructure after hot rolling and a dual-phase microstructure of globular-shaped α ferrite and retained γ austenite (γ_R) after cold rolling and intercritical annealing [1–5]. The grain sizes of both α ferrite and γ_R range from approximately 300 nm to 1 µm, which correspond to the so-called ultrafine grain size; the volume fraction of γ_R ($V\gamma_R$) is between approximately 0.2 and 0.4 [1–5]. The medium Mn steels with an ultrafine-grained microstructure show a remarkable combination of high strength (over 800 MPa) and high ductility (maximum 40%) due to active transformation-induced plasticity (TRIP) occurring in a large amount of γ_R during plastic deformation [1–5].

Until now, most of studies on medium Mn steels have focused on achieving a large $V\gamma_R$ by controlling intercritical annealing conditions, such as temperature [4,9], time [6,10], heating rate [11], and cooling rate [12]. Both $V\gamma_R$ and total elongation become greater with increasing annealing temperature and time, reach the maximum values, and then decrease again with annealing further [4,6,9,10]. This change of $V\gamma_R$ with annealing temperature and time is closely related to a phase stability of reverted γ, which forms during intercritical annealing. For example, when the annealing temperature is too high, the phase stability of reverted γ becomes low because the high annealing temperature results in the large fraction of coarse-grained reverted γ with the low concentrations of γ stabilizers, such as Mn and C atoms. The reverted γ with a low phase stability transforms to martensite during cooling from annealing temperature, leading to a low $V\gamma_R$ at room temperature.

When Fe–(5–9)Mn–0.05C (wt.%) steels are slowly heated up to annealing temperatures at a rate less than 15 °Cs^{-1}, a diffusive reverse transformation from α' martensite to γ occurs; the reverted γ possesses a high phase stability due to the high enrichment of Mn and C atoms [11]. However, when the steels are rapidly heated at a

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rate higher than 15 °Cs^{-1}, a diffusionless reverse transformation from α′ martensite to γ occurs, resulting in the reverted γ with a low phase stability [11]. When the Fe–5Mn–0.3C (wt.%) steel is rapidly cooled after intercritical annealing, the $V\gamma_R$ increases due to the suppression of cementite precipitation; both the total elongation and the tensile strength are improved [12].

An alternative method to improve the mechanical properties of medium Mn steels is to fabricate a bimodal-grained microstructure [13–15,17,18]. According to Suh et al. [13,14], the hot-rolled Fe–5Mn–0.12C–0.5Si (wt.%) steel with over 3 wt% Al exhibited a dual-phase microstructure of α ferrite and α′ martensite. After cold rolling and intercritical annealing, the α grains were recrystallized to be relatively coarse grains (approximately 3 μm); the α′ martensite reverted to ultrafine-grained (approximately 300 nm) α and γ phases. The Al-bearing medium Mn steel with this bimodal-grained microstructure exhibited a higher elongation and a shorter Lüders strain compared to an Al-free medium Mn steel with an ultrafine-grained α and γ microstructure.

Lee et al. [15] have also observed such a bimodal-grained microstructure in Fe–6Mn–2Al–1.5Si–0.08V–0.08C (wt.%) steel after cold rolling and intercritical annealing, and reported that the steel revealed continuous yielding without the Lüders strain and a higher elongation than the Fe–6Mn–0.08C (wt.%) ternary steel with an ultrafine-grained α and γ microstructure.

Although the addition of Al to medium Mn steels results in a bimodal-grained microstructure to improve tensile properties, it causes several problems related to steelmaking and continuous casting processes besides an increase in materials cost, such as nozzle clogging [19] and changes in properties of the mold flux [20]. Therefore, the objectives of the present study were to develop the bimodal-grained microstructure using a medium Mn steel only through a thermo-mechanical process without the addition of Al, and to elucidate the reasons why the bimodal-grained medium Mn steels exhibit the short Lüders strain or continuous yielding without Lüders strain.

2. Experimental procedure

A 30 kg ingot of Fe–7Mn–0.05C (wt.%) steel was fabricated using a vacuum induction furnace; the ingot size was approximately 100 × 170 × 220 mm^3 and an actual chemical composition of the ingot was measured to be Fe–6.7Mn–0.05C–0.3Si–0.08 Al–0.0097P–0.0058S in weight percent after homogenization at 1200 °C for 12 h. The ingot was solution-treated at 1200 °C for 2 h, hot-rolled to a 3-mm-thick plate, maintained at 600 °C for 1 h, and then furnace-cooled to room temperature for coiling simulation. The hot-rolled plates were cold-rolled to 1.2 mm with a 60% thickness reduction after surface descaling. The cold-rolled sheets showed full deformed α′ martensite with no other phases present [11].

Cold-rolled specimens were annealed twice using a tube furnace; at first, the specimens were heated to 660 °C at a rate of 10 °C s^{-1}, held for 4 h, 8 h, 14 h, and 22 h, and then air-cooled to room temperature. The relatively long annealing time of over 4 h was determined to cause the grain growth of both α and reverted γ, resulting in a dual-phase microstructure of coarse α and α′ martensite after cooling due to the low γ stability. The second annealing was performed at 640 °C for 5 min to transform coarse α′ martensite to fine α and γ phases, and then air-cooled to room temperature. The heating rate was approximately 10 °C s^{-1}. The first and second annealing temperatures were determined based on equilibrium eutectoid (A_{e1}) and ferrite start (A_{e3}) temperatures, which were calculated using Thermo-Calc software with TCFE7 database [21].

The microstructures of both single- and double-annealed specimens were examined using a field-emission scanning electron microscope (FE-SEM; JEOL, JSM-7001F) equipped with an electron backscattered diffractometer (EBSD; EDAX-TSL, Digiview) and an energy dispersive X-ray spectroscope (EDXS; EDAX-TSL, TEAM™) and a field-emission transmission electron microscope (FE-TEM; JEOL, JEM-2100F) equipped with an EDXS (Oxford, INCA energy). Specimens for SEM and EBSD observation were polished using a suspension including 0.04 μm colloidal silica particles, and then electro-polished in a mixed solution of 90% glacial acetic acid (CH_3COOH) and 10% perchloric acid ($HClO_4$) at 15 V for 30 s to remove the mechanically damaged layer. The accelerating voltage, probe current, working distance, and step size were 20 kV, 12 nA, 15 mm, and 30 nm, respectively. Thin foils for TEM observation were jet-polished in the same mixed solution used for the EBSD specimens.

The volume fraction and the lattice parameter of γ_R were examined using an X-ray diffractometer (XRD; RIGAKU, D/MAX-2500H) with a Cu–Kα radiation (λ = 1.5405). The XRD specimens were prepared by the same method used for the EBSD specimens. The scanning range, rate, and step size were 40–100°, 2° min^{-1}, and 0.02°, respectively. The $V\gamma_R$ was calculated using the integrated intensities of all diffracted peaks and the lattice parameter of γ_R was determined by the Nelson-Riley method [22].

The dog-bone-shaped tensile specimens were machined along the rolling direction of the cold-rolled sheets. The size of the gauge portion was 1.2 mm in thickness, 4.0 mm in width and 15 mm in length. The tensile specimens were mechanically polished, and then electro-polished at 20 V for 2 min in the same mixed solution used for the EBSD specimens. Tensile tests were conducted at an initial strain rate of 1 × 10^{-3} s^{-1} at room temperature using an Instron 3382 machine. The propagation of Lüders bands during the tensile tests was observed at the gauge portion using a low-magnification optical microscope (OM; Olympus Bx51).

3. Results and discussion

Fig. 1a shows the XRD patterns of the specimens first-annealed at 660 °C for 4 h, 8 h, 14 h, and 22 h. All specimens exhibited the diffraction peaks of only a *bcc* phase, regardless of annealing time; this means that the specimens did not possess γ_R after the first intercritical annealing because reverted γ with the low phase stability transformed to α′ martensite during cooling. To clarify the microstructures of the first-annealed specimens, simultaneous EBSD and EDXS analyses were conducted (Fig. 1b–e). In the IQ-phase maps, the red is γ_R; the gray is α or α′ martensite; blue lines are low-angle boundaries with misorientation angles between 3° and 15°; black lines are high-angle boundaries with misorientation angles of over 15°. The kernel average misorientation (KAM) maps were drawn using an average misorientation angle around a measurement point with respect to a defined set of the nearest neighbor points to evaluate the amount of lattice distortion of each grain. The KAM values of over 2° were excluded for mapping because such high values are obtained when the measurement points are located at the boundaries of sub-grains or grains.

The IQ-phase maps exhibited that all specimens had a bright gray region with the high confidence index (CI) values of over 0.8 and a dark gray region with the low CI values of below 0.5. The bright and dark gray regions are considered to be globular-shaped α (α_G) and α′ martensite, respectively, based on the facts that the dark gray region shows low CI and high KAM values due to significant lattice distortion, and it has the higher Mn concentration than the bright gray region (refer to EDXS maps). Meanwhile, the γ_R was rarely observed in the IQ-phase maps, which agrees well with the XRD patterns (Fig. 1a).

The volume fractions of α_G and α′ martensite were measured