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A nanograined/ultrafine-grained low-carbon microalloyed steel processed by warm rolling

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ABSTRACT

We have obtained grain refinement in the nanoscale regime (~300–400 nm) by combining dynamic transformation, intragranular nucleation ferrite, and dynamic recrystallization. We elucidate a novel thermomechanical process and microalloy design to obtain ferrite–martensite microstructure with nanoscale features in low-carbon chromium-containing steel with yield strength, tensile strength, and elongation of 745 MPa, 935 MPa, and 19.5%, respectively. The mechanism associated with increased work hardening and superior formability is discussed.

1. Introduction

Among the various strengthening mechanisms, grain refinement is considered as the appropriate and convenient method to simultaneously improve both strength and toughness [1]. Nanograined/ultrafine-grained steels exhibit outstanding properties including high strength/weight ratio, wear resistance, and are also favorable for cellular activity [2–5]. Thus, there exists a strong potential for using ultrafine-grained steels in lieu of the conventional coarse-grained counterpart. In recent years, a number of methods have been developed to produce ultrafine-grained steels. Severe plastic deformation methods such as equal channel angular processing (ECAP) and hot torsion is limited to laboratory-scale curiosity. The advanced thermomechanical processing routes are important and viable methods for improving conventional processing routes of high strength steels. The advanced thermomechanical processing methods include deformation-induced ferrite transformation, large-strain warm deformation (deformation after transformation), intercritical hot rolling, multi-directional rolling, and cold-rolling plus annealing of martensitic steel [6]. Recently, strategies to control transformation have been considered to obtain nanograined/ultrafine-grained steels through low temperature bainitic transformation and intragranular nucleation ferrite [2,7].

The strain-induced transformation, dynamic recrystallization, and intragranular nucleation of ferrite are effective methods to refine the grain size, but the grain size is restricted to micrometer regime [7–11]. We describe here the potential to obtain significant grain refinement to nanoscale regime by combining the above outlined approaches that have not been explored to the best of our understanding. Second, low carbon V-microalloyed steels containing chromium as austenite stabilizers and subjected to warm-rolling have not been explored. The process of cold rolling combined with annealing of martensitic steel is also a good method to obtain bulk nanostructured low carbon steel. But to obtain a steel strip of ~5 mm thickness in cold rolled hardened martensite microstructure requires high rolling force [12]. Thus, a novel alloy and process design is required to process nanograined/ultrafine-grained steel.

Here, we explore the possibility of processing Cr-containing microalloyed steel through a novel warm rolling process involving dynamic transformation, intragranular ferrite nucleation, and dynamic recrystallization to obtain nano-grained ferritic steel strip of ~5 mm thickness. In comparison to the cold rolling plus annealing of martensitic steel, thick plates can be obtained at lower rolling force condition encountered during dynamic transformation and dynamic recrystallization, reducing the deformation resistance.

2. Experimental

The experimental steel was melted in a vacuum induction furnace and cast as a 150 kg ingot. The chemical composition of

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Table 1
Chemical composition of the experimental steel.

Elements	C	Si	Mn	Al	P	S	Cr	V	N	Fe
wt%	0.08–0.12	0.16	1.8	0.02	0.003	0.002	0.46	0.2–0.4	0.015–0.02	Bal.

the steel in wt% is shown in Table 1. A small amount of Cr (0.46 wt %) was added to stabilize the under-cooled austenite and thereby decreased the warm-rolling temperature to ensure that the dynamically transformed nanograined ferrite is thermally stable. The V–N microalloying concept [13,14] was adopted to exploit VN precipitates in austenite for the intragranular nucleation of ferrite, given the small lattice mismatch between VN (lattice parameters = 0.4139 nm) and ferrite (lattice parameters = 0.2865 nm) for $(100)_{VN}//(100)_{\alpha}$ planes, facilitating ferrite nucleation. The two-step cycle quenching was conducted on the 50 mm thick slab for refining the prior austenite grain size [15] and involved heating of slab to 900 °C and water-quenching to room temperature.

The continuous cooling transformation diagram of austenite was measured using the Formaster-FII dilatometer. The specimens were cut from the two-step cycle water-quenched slab and machined to the dimensions of 10 mm in length and 3 mm in diameter. The samples were heated to 900 °C at 5 °C/s and held for 300 s, and then continuously cooled to 20 °C at cooling rates in the range of 2–80 °C/s. This range of cooling rates can distinguish the critical cooling rate for inhibiting the ferrite nucleation, and the measured cooling rate followed the targeted cooling rate.

The 50 mm thick slab was heated to 900 °C for 300 s for complete austenization followed by water-cooling to 550 °C at a cooling rate of 35 °C/s. The slab was warm-rolled to plate thickness of 5.5 mm in 10 passes using a ⌀450 mm rolling mill, and finally air-cooled to room temperature. During finish rolling the temperature rises from 550 °C to 580 °C. Another plate was water-quenched to room temperature after warm-rolling.

The materials were mounted and polished to mirror finish using the standard metallographic procedure and etched with 4 vol% nital solution. Microstructures were observed using a Zeiss Ultra 55 scanning electron microscope (SEM). The chemistry of the precipitates was determined using energy-dispersive X-ray spectroscopy (EDX) within the SEM. Transmission electron microscopy (TEM) was conducted using FEI Tecnai G^2 F20 TEM at an accelerating voltage of 200 kV using 3 mm diameter thin foils, electro-polished using a solution of 8% perchloric acid and alcohol. The tensile tests were conducted at room temperature on a Shimadzu AG-X universal testing machine according to the ISO standard 6892-1: 2009 standard using the dog-bone-shaped specimens with the gauge of 25 mm. The crosshead speed was 3 mm min^{-1}.

3. Results and discussion

The transformation temperature for under-cooled austenite to transform to bainite is ~500 °C at a cooling rate of 35 °C/s (Fig. 1). The addition of Cr to the experimental steel stabilizes austenite and avoids decomposition of austenite prior to warm-rolling. The coarse-grained polygonal ferrite is first formed along the prior austenite grain boundaries at cooling rates of 2–5 °C/s (Fig. 2a and b). The ferrite nucleation is inhibited when the cooling rate is increased to 10 °C/s (Fig. 2c), and bainite and martensite microstructure is obtained at cooling rate of 40 °C/s (Fig. 2d).

The microstructure of warm-rolled and air-cooled steel consists of polygonal ferrite and uniformly dispersed (V, Cr, Fe)(C, N) precipitates (Fig. 3). The polygonal ferrite grain size is in the range of ~300–400 nm and the precipitates are in the range of ~10–50 nm. The TEM micrographs indicate that the grain boundaries of polygonal

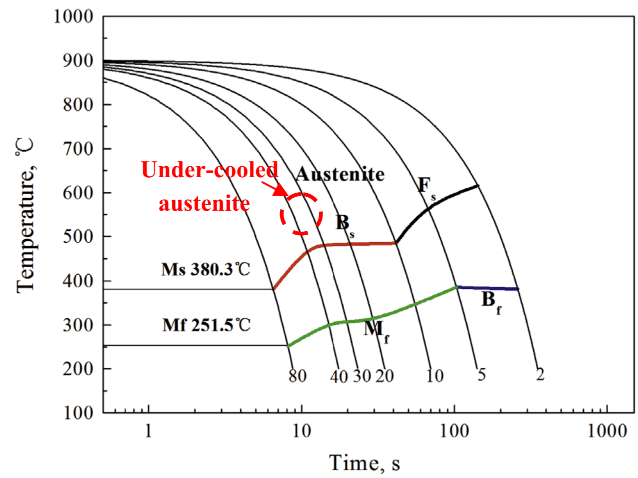

Fig. 1. CCT curve for the experimental steel subjected to two-step cycle quenching.

ferrite grains of grain size ~300–400 nm are well defined and there is low density of dislocations inside the grains, implying complete recrystallization (Fig. 4a). There are, however, some ferrite grain boundaries that are not well-developed and extensive extinction contours exist in the vicinity of grain boundaries. Furthermore, the high density of dislocations implies non-equilibrium state with high internal stress and partial recrystallization [16–18] (Fig. 4b). The (V, Cr, Fe)(C, N) precipitates are present both within the polygonal ferrite grain and at the grain boundaries.

In a manner similar to warm-rolled and air-cooled steel, the polygonal ferrite grains of warm-rolled and water-quenched steel are ~300–400 nm (Figs. 5a and 6a), and the grain boundaries are well developed. This suggests that the dynamic transformation and dynamic recrystallization occurred during warm-rolling. However, few ferrite grains of ~200 nm thick are elongated and martensite is present (Figs. 5b and 6b). Compared to the warm-rolled and air-cooled experimental steel, the warm-rolled and water-quenched experimental steel consists of higher fraction of non-equilibrium grain boundaries and higher density of dislocations (Fig. 6). Thus, the few ferrite grains of warm-rolled and air-cooled experimental steel are nucleated after deformation and involved static recrystallization of elongated grains [19]. Compared to warm-rolled and air-cooled experimental steel, there is lower fraction of coarse precipitates in the warm-rolled and water-quenched experimental steel (Figs. 3a and 5a). Thus, fine precipitates are generated during warm-rolling, and the coarsening of the precipitates is hindered due to high cooling rate after warm-rolling.

The dynamic strain-induced austenite-to-ferrite transformation is thermodynamically feasible. The nanoscale microstructure is thermally stable during holding for short durations at 580 °C and no significant grain coarsening occurs. It is proposed that the addition of a small amount of V is effective in enhancing the microstructural stability of ultrafine-grained low carbon steel against thermal exposure because of the combined effect of V, retarded recrystallization and suppressed grain growth induced by nanometer-sized carbides precipitates, even if recrystallization were to occur [20].