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An experimental and mathematical study on the evolution of ultrafine ferrite structure during isothermal deformation of metastable austenite

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ABSTRACT

Heavy deformation of metastable austenite at intercritical temperature is known to develop 'ultrafine ferrite-grain' (ULFG) structure and provide grain boundary strengthening. Systematic thermomechanical simulation was conducted in Gleeble®3500 by deforming the samples isothermally at two intercritical temperatures: 810 °C (~40 °C below Ae_3 and ~160 °C above Ar_3) and 710 °C (~140 °C below Ae_3 and ~60 °C above Ar_3) to identify the critical conditions for the formation of ULFG structure in a low carbon microalloyed steel. Both single-pass and multi-pass deformations with varying equivalent total strain level were considered in order to provide a solution towards the development of ULFG structure upon industrial rolling. Microstructure evolution suggested that multi-pass intercritical deformation can produce uniform distribution of ultrafine ferrite grains (grain size ≤ 2 μm) as a combined effect of static- and dynamic strain-induced transformations (SSIT and DSIT) and continuous dynamic recrystallization (CDRX). Based on the microstructural evidences and strain analysis following Militzer-Brechet model, a descriptive model has been proposed discussing the mechanism of grain refinement during isothermal intercritical deformation.

1. Introduction

A continuous drive from construction, line-pipe, heavy-engineering and automotive sectors has always existed to increase the strength of low-carbon and low-alloy steels, without sacrificing the other properties, especially toughness and weldability [1–3]. Grain refinement can be an effective solution to the problem. In comparison to the severe plastic deformation techniques such as equal-channel angular pressing, accumulative roll bonding and high-pressure torsion [1,2,4], advanced thermomechanical processing involving heavy deformation of metastable austenite (below A_{e3}) is more feasible for industrial application [1–12]. Extensive research on this processing established that a fine prior-austenite grain size (≤ 20 μm), heavy deformation of metastable austenite (strain, $\varepsilon > 0.5$) below A_{e3}, at a high strain-rate ($\dot{\varepsilon} > 0.1/s$) followed by rapid cooling (cooling rate > 30 °C/s) can develop homogeneous distribution of ultrafine ferrite grain structure (grain size ≤ 2 μm) through dynamic strain-induced austenite to ferrite transformation (DSIT) [1–12]. In an industrial rolling mill, however, the application of aforementioned deformation conditions is difficult due to the requirement of heavy rolling load, precise temperature control and the water cooling capability. Therefore, it is highly desired to explore the possibility of generating the desired 'ultrafine ferrite-grain' (ULFG) structure by dividing the entire deformation into an effective multi-pass schedule where the deformation par pass has to be within the feasible limit.

Previous studies applied different multi-pass deformation schedules to achieve ULFG structure in low-carbon ferritic steels [13–18]. The grain refinement in multi-pass schedule was, however, not as effective as in case of heavy single-pass deformation. To understand and minimize this difference, it is necessary to study the mechanism of grain refinement in multi-pass deformation schedule and compare it with the corresponding single-pass deformation schedule. Previous studies on the mechanism of grain refinement during multi-pass deformation applied the deformation passes at different temperatures [13–18]. Hence, both temperature and applied strain changed with the progress of the deformation schedules. The deformation schedules applied in the present study were isothermal so that the microstructural evolution can be systematically studied as the function of applied strain and interpass time. Two different deformation temperatures (T_{def} of 810 °C or 710 °C) within the intercritical temperature range were selected for this study. Although the temperature drops continuously during application of the industrial rolling passes, isothermal deformation passes were applied to

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understand the evolution of ultrafine grain structure during sequential deformation of the metastable austenite and also during the interpass interval. Based on this understanding an industrial deformation schedule may be designed in future for applying a number of finishing passes with a short interpass interval so that deformation heat can be utilized to maintain a nearly isothermal condition. External heating source can also be tried to maintain an isothermal condition during finishing passes.

Not only the extent of grain refinement but also the evolution of grain size distribution and crystallographic texture needs to be studied for different thermomechanical processing schedules (single-pass and multi-pass) aimed at developing the ULFG structures. Grain size distribution and texture influence the tensile properties and formability of ULFG steels. Recent studies from different research groups reported the effect of grain size distribution and different texture fibers on impact toughness especially considering the delamination phenomenon in ULFG structures during impact testing [8,19–22].

2. Experimental details

2.1. Materials and processing

As-received slab of a low-carbon microalloyed steel was hot-forged and rolled down to 24 mm thick plate. The nominal composition of the steel is given in Table 1. The rolled plate was subjected to a prolonged homogenization treatment for 24 h at 1200 °C to remove segregation as far as possible. Rectangular blocks (10 mm × 15 mm × 20 mm) were machined from the quarter thickness location for plane strain compression testing inside a Gleeble®3500 thermomechanical simulator. Four different deformation schedules were performed in Gleeble® as shown in Fig. 1.

After soaking at the original austenitization temperature of 1200 °C for 3 min, all the samples were cooled down to the respective deformation temperatures (T_{def} = 810 °C or 710 °C) at a cooling-rate of 5 °C/s. The total strain remained the same (ε = 1.2) and it was applied either by a single-pass (Fig. 1**a**) or by multiple passes, specifically two (ε/ pass = 0.6), four (ε/ pass = 0.3) and eight passes (ε/ pass = 0.15), Fig. 1(b-d). In general, the samples were water quenched immediately after applying the complete deformation, i.e. total ε = 1.2, Fig. 1. In one of the multi-pass schedule, the samples were water quenched from different intermediate stages of processing (before and after the deformation passes) as shown by the dotted arrows in Fig. 1**c**. The values of strain-rate ($\dot{\varepsilon}$ = 1.0/s) and interpass time (t_{ip} = 10 s) as typically encountered in the industrial plate rolling, were maintained constant in all the schedules. The processed samples were coded by considering their T_{def} (810 °C or 710 °C), applied total strain up to the point of quenching and finally the number of passes used to apply that strain. The samples that were deformed and isothermally held for 10 s before quenching following the schedule in Fig. 1**c** (from the intermediate processing stages) are coded in terms of T_{def} - applied ε - and the interpass time (10 s). Besides the deformed samples, few samples were soaked at 1200 °C, cooled down to 810 °C / 710 °C (at 5 °C/s) and water quenched without any deformation either immediately or after 10 s of isothermal holding at those temperatures. A couple of thermocouples were attached to the samples during processing for precise monitoring of their temperatures at an accuracy of ± 2 °C. The sample codes and the processing details of all the samples are listed in Table 2.

To determine the actual austenite to ferrite transformation start (A_{r3}) and finish (A_{r1}) temperatures during cooling, a thin rectangular strip (120 mm × 10 mm × 3 mm) was austenitized and cooled at 5 °C/s inside BAHR® Thermoanalyse GmbH dilatometer. The sample dilation curve is shown in Fig. 2**a**, where the transformation temperatures (A_{r3} ~ 650 °C and A_{r1} ~ 530 °C) are indicated by arrows. The equilibrium transformation temperatures (A_{e3} ~ 860 °C and A_{e1} ~ 685 °C) were predicted by the TCFE6 database of the Thermo-Calc® software, Fig. 2b. The prior-austenite and transformed ferrite fractions expected at different temperatures during cooling as obtained from the dilatometric study and predicted from the Thermo-Calc®, respectively, are plotted in Fig. 2b.

Table 1
Chemical compositions of the investigated steel (wt%).

C	Si	Mn	P	S	Al	Nb	Ti	V
0.09	0.33	1.42	0.010	0.003	0.035	0.05	0.019	0.05

2.2. Characterization of microstructure and microtexture

Longitudinal sections facing the mid-plane of the Gleeble® tested samples (Fig. 1**e**) were mounted in bakelite, prepared following the conventional metallographic technique and etched with 2% nital solution. DM2500M optical microscope and Zeiss® Auriga Compact scanning electron microscope (SEM) were used for the microstructural study. The electron backscattered diffraction analysis (EBSD) was carried out over an area (1 mm × 0.7 mm), covered by multiple frames (100 μm × 150 μm) at the central part of the deformed zone of each sample. *HKL* Channel 5 system from Oxford Instruments, UK, fitted in Zeiss® SEM, was used for the EBSD analysis. SEM was operated at 20 kV accelerating voltage, and the EBSD scans were performed at 0.2 μm step size. Based on the angular resolution of EBSD, a minimum misorientation threshold of 2° was chosen. The ferrite and martensite regions of the samples were distinguished by EBSD analysis based on the higher image quality and band contrast of ferrite (in comparison to martensite) following the procedure reported in the recent studies [8,9,23]. A critical band contrast of '60' was selected for this purpose as the ferrite fraction measured by EBSD closely matched with the same measured from SEM imaging (within 2% accuracy) at that critical value. EBSD band contrast maps identified the ferrite regions along with the estimation of ferrite fraction, which is reported. It also distinguished the ultrafine ferrite grains even better than the SEM imaging. Therefore, the EBSD band contrast maps are preferred over the SEM images for the microstructural characterization. The ferrite fraction measured from EBSD was also verified from the high magnification optical and SEM micrographs. Ferrite regions in high magnification optical and SEM images were selected using image analysis software and added to determine the ferrite fraction, considering the total area observed under optical or SEM. A close correlation (within 3%) was found in the fractions obtained from EBSD and measured from optical or SEM images.

Following conventional representation, the low-angle boundaries (i.e. sub-boundaries) (LAB) and the high-angle boundaries (i.e. grain boundaries) (HAB) are considered to have misorientations within 2–15° and greater than 15°, respectively.

EBSD inverse pole-figure maps showing crystal orientations along the normal direction of the sample (ND-IPF) were used to study the microtexture of the ferrite regions, which was presented as the orientation distribution functions (ODF) on the φ_2 = 45° section of the Euler space. Typical orientations that develop in ferritic steel (BCC) can be represented on the φ_2 = 45° ODF section as shown in Fig. 3. The EBSD analysis was also used to estimate the average ferrite grain size. For this study, the grain area and the equivalent circle diameter (ECD) grain size of at least 500 ferrite grains were measured from each sample considering only the high-angle boundaries (i.e. the grain boundaries).

3. Results

3.1. Microstructures of undeformed samples and the selection of deformation temperatures

The deformation temperatures used in the present study (810 °C / 710 °C) are shown by the dotted lines in Fig. 2b, where the equilibrium