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Three-dimensional characterization of bainitic microstructures in low-carbon high-strength low-alloy steel studied by electron backscatter diffraction

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ABSTRACT

We investigated the microstructural evolution of high strength low alloy steel, Fe–2.0Mn–0.15Si–0.05C (wt.%), by varying the continuous cooling rates from 1 K/s to 50 K/s using three-dimensional electron backscatter diffraction and transmission electron microscopy. Granular bainitic microstructure was prevalent under a slow cooling rate of 1–10 K/s, while lath-type bainite was dominant at a high cooling rate of 50 K/s. The acicular ferrite that was the major microstructure under the intermediate ranges of cooling rates between 10 K/s and 30 K/s was tangled with each other, leading to a three-dimensional interwoven structure with highly misoriented grains. Because of the formation of three-dimensional structures, we propose that the terms “acicular ferrite” and “bainitic ferrite,” which are currently used in steel, be replaced by the terms “interwoven acicular bainite” and “lath bainite,” respectively. Moreover, we also confirmed that the cooling rate is an important factor in determining whether bainitic microstructures occur in the form of granular bainite, interwoven bainite, or lath bainite.

1. Introduction

High-strength low-alloy (HSLA) steel has received a great deal of attention for constructing buildings, bridges, and gas transmission pipelines because of their high impact toughness and good weldability [1–3]. By controlling both the microstructure and the alloying elements, the manufacture of advanced HSLA steels with superior mechanical properties can be more feasible. Especially, bainite-based HSLA steel is a promising material for saving production cost by giving an opportunity to increase line pressure, for reducing both wall thickness and transportation cost of pipes [2]. Such bainitic X80–X100 line pipe steel grades showed an ultimate tensile strength of 800–1000 (MPa), a yield strength of 500–600 (MPa), and an elongation of 20%. These HSLA steels are classified by their nominal carbon content, since their microstructure depends significantly on the amount of carbon in the steel. For example, the microstructure of steel with carbon content between 0.2 wt.% and 0.7 wt.% consists of “upper bainite” and “lower bainite”. These kinds of bainite can be determined by the location of the cementite precipitates, namely, whether cementite particles are distributed between laths or within laths [4–7]. In contrast, the bainitic microstructure of HSLA steel containing less than 0.2 wt.% carbon (the so-called “low-carbon HSLA steel”) is complex. This complexity is due to the fact that during a continuous cooling process, the

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austenite phase in low-carbon HSLA steels can be transformed into various bainitic microstructures at elevated temperatures [2,8-10]. Moreover, the kinetics of cementite precipitation in such steel is retarded during continuous cooling [2,9], which leads to significant confusion in characterizing several bainitic phases. Hence, microstructural characterization is essential for the optimal design of low-carbon HSLA steel consisting of bainitic grains. Several studies have been conducted to reveal the bainitic microstructure in low-carbon steel using scanning electron microscopy (SEM) and transmission electron microscopy (TEM) [11,12]. In addition, more detailed characteristics of low-carbon bainitic microstructure have been investigated using focused ion beam (FIB) milling [13,14]. According to Yakubtsov et al. [2], the final microstructure of the low-carbon HSLA steel was controlled mainly by continuous cooling rates, and hence consisted of either granular bainite and acicular ferrite microstructure, or their mixture. However, additional details regarding the misorientation angles between phase boundaries within several bainitic grains have not been fully identified so far. To reveal these issues, we used two-dimensional (2D) and three-dimensional (3D) electron backscatter diffraction (EBSD). Indeed, the 3D EBSD is a well-suited tool for evaluating microtexture and for estimating misorientation angles between the phase boundaries of austenite and bainite [15]. As a result, it gives information on the crystallographic orientation gradients in the vicinity of ferrite/austenite interface and thus on the topology and morphology of grains on a 3D microscopic scale [16–19]. For instance, previous 3D EBSD studies revealed a hard Laves particle of a warm-rolled Fe_3Al based alloy [15]. West and Thomson [17] studied interdiffusion in a Ni-based superalloy using combined EBSD/EDS tomography. Also, Xu et al. [18] reported crystallographic information for microstructures in N–Si alloys. Recently, Seol et al. [19] addressed the influence of 3D bainitic microstructures on the combination of strength and elongation in high-carbon containing transformation induced plasticity (TRIP) steel.

The present study aims at investigating the influence of continuous cooling rates on the microstructural evolution of low-carbon bearing bainite-based HSLA steel using 2D and 3D EBSD. The present steel has three types of bainitic microstructures: granular bainite, acicular ferrite, and bainitic ferrite [13]. The bainitic grains are transformed from austenite during a continuous cooling process. Even though the morphology of the bainitic grains has been investigated using joint FIB and SEM imaging [13], some ambiguities with the nomenclature still remain, especially the terms "acicular ferrite" and "bainitic ferrite". Therefore, another aim is to clarify the exact terminology of bainitic microstructures by giving the actual 3D microstructure of bainitic grains displayed in the current steel.

2. Experimental

The HSLA steel used in this study contained 2.0 wt.% Mn, 0.15 wt.% Si, and 0.05 wt.% carbon and $<\sim 0.10$ wt.% Ti and Nb as well as trace amounts of Mo, Ni, Cr and B. The nominal composition of the steel material was determined by inductively coupled plasma mass spectroscopy. The steel was melted and continuously cast. The cast ingot was subsequently machined into cylindrical samples each with a diameter of 7 mm and a length of 12 mm in order to simulate a thermo-mechanical controlled process (TMCP). The TMCP simulations were performed using a Gleeble 3500 system (Dynamic Systems, Inc.) [3]. The cylindrical samples were heated to 1150 °C for 10 min to dissolve Ti and Nb. Next, rough milling and finish milling were carried out as part of the hot-rolling process. The steel thickness reduced by 20% at 1000 °C, followed by a further reduction of 60% reduction at 850 °C. Finally, the hot-rolled samples were quenched in water. The phase-transformation initiating temperatures of bainite (B_s) and martensite (M_s) as determined by dilatometry were 550 °C and 440 °C, respectively. All specimens for EBSD measurements were prepared using electro-polishing and chemical etching (LectroPol-5, Struers™) with a solution of acetic acid and perchloric acid in order to stop further transformation of austenite to α'-martensite during mechanical polishing [6]. Hikari EBSD camera capable of indexing 450 Kikuchi patterns per second was combined with a dual-beam FIB machine (FEI, Helios NanoLab™ 600). Joint FIB milling and EBSD observation for a specific area was automatically performed using 3D orientation imaging microscopy (3D OIM). We used standard markers on the surfaces of the samples in order to inhibit electron drift, which can be caused by the rotation of the specimen holder during FIB milling and EBSD detection. Consequently, the exact position could be etched by Ga^+ ions and detected by EBSD, as shown in Fig. 1. Because of the beam-channeling effect during Ga^+ milling at 30 kV, a curtain-like structure was formed at a certain depth (the so-called "curtaining effect") [20]. Recent work has even reported that the relative degrees of the surface penetration of Ga^+ ions during FIB milling affect the coordination number of either fcc or bcc metals during EBSD detection, leading to a significant influence on the quality of an EBSD pattern generated from a Ga^+ ion-milled surface [21]. To avoid these problems, the EBSD scanned data were taken from the curtain-free area of the samples, as shown in Fig. 1. Moreover, the accelerating voltage for the Ga^+ ions was selected as 15 kV and the milling current used in this study was 9.3 nA, with a step size of 70 nm. The working distance was 10 mm for all EBSD phase maps, which were post-processed using the OIM software. A confidence index (CI) value of >0.15 was chosen for phase identification [22]. Three 3D EBSD analyses were carried out for each sample.

3. Results and Discussion

The continuous cooling rates lead to the microstructural evolution of the bainitic matrix in the present steel, as shown in Fig. 2. From the SEM micrographs, we confirmed the presence of three types of bainitic microstructures depending on the employed cooling rates: granular bainite (Fig. 2(a)), acicular ferrite (Fig. 2(b)), and bainitic ferrite (Fig. 2(c)). As the cooling rate increased, the microstructure of the steels showed granular bainite, mixture of granular and file-type bainite, and bainitic ferrite. Especially, the arrows shown in