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Crystallographic analysis of nanobainitic steels

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Electron back-scattered diffraction in conjunction with transmission electron microscopy was employed to investigate the crystallographic nature of bainitic laths formed at relatively low transformation temperatures where a nanostructured bainite forms. It was revealed that the bainitic ferrite laths are close to the Nishiyama–Wasserman orientation relationship with the parent austenite. Furthermore, the temperature showed a significant effect on the retained austenite characteristics and the variant selection of the bainitic ferrite laths. A decrease in temperature generally refined the bainitic structure and weakened the variant selection.

Keywords: Steels; Nanostructured bainite; Crystallographic analysis; Variant selection; Temperature

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The development of high-strength steels generally involves a trade-off between strength and toughness. The only current mechanism to achieve both strength and toughness is through refinement of the microstructure, as all other strengthening mechanisms reduce the toughness. A new generation of nanostructured bainitic steels appears to offer notable strength (2.3 GPa), toughness (30 MPa $m^{1/2}$) and ductility (30%) in both quasi-static and dynamic loading conditions [1–3]. They have incredible potential in the transport, construction and offshore industries, as well as defence applications. The excellent combination of mechanical properties appears to be partly due to the formation of nanostructured bainite consisting of very fine bainitic ferrite laths with an average thickness of ~50 nm with retained austenite films between these laths.

The mechanical properties of bainitic steels such as toughness [4–6] and ductility [7–8] are controlled by the microstructural characteristics. For example, the cleavage fracture resistance of bainitie is closely related to both the prior austenite grain and packet size [9–10]. In the bainite transformation, the prior austenite grain is divided into a three-level hierarchy similar to the lath martensite structure in terms of morphology: packet, block and lath. The packet consists of one, or several sets of blocks that are individually further subdivided into a group of laths with the same habit plane with respect to the parent austenite and similar orientation. The packet characteristics (i.e. size, variant selection) strongly depend on the characteristics of the austenite to bainite phase transformation (i.e. temperature) [4,11] and steel composition (i.e. carbon content) [11,12].

While there have been some studies performed on the morphology and crystallography of martensite [4,12–14] and upper bainite [4,11] in Fe alloys, there has been no attempt to examine the bainitic lath structure formed at relatively low transformation temperatures (i.e. 200–350 °C), where nanostructured bainite is formed providing excellent mechanical properties combination. In this study electron back-scattered diffraction (EBSD) in conjunction with transmission electron microscopy (TEM) examination was employed to characterize the morphology and crystallographic characteristics of nanostructured bainite.

The composition of the steel used in this study was 0.79C–1.5Si–1.98Mn–0.98Cr–0.24Mo–1.06Al–1.58Co (wt.%) based on previous work [15]. The B_s and M_s temperatures of the alloy were estimated to be 385 and 155 °C, respectively [15]. The steel was reheated to 1100 °C and held for 30 min, followed by isothermal heat treatment at either 200 or 350 °C. The average austenite grain size was 60 μm. The microstructural characteristics and crystallographic analysis at different conditions were examined using EBSD and TEM crystallographic analysis.

Samples for EBSD were prepared by standard mechanical polishing finished with a colloidal silica slurry polish. EBSD was carried out using a FESEM JSM7000F scanning electron microscope operated at 25 kV. The instrument was equipped with a fully automatic EBSD device attachment. TexSEM Laboratories

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software (TSL) was used to perform data acquisition and post-processing. EBSD maps were acquired either using a step size of 40 nm for the characterization of the retained austenite and bainitic ferrite interface or 0.2 μm to study the variant selection mechanism. The map area was approximately $300 \times 110\ \mu m^2$ covering about 10 prior austenite grains for both transformation temperature conditions. All grains showed similar features for a given transformation temperature. Pattern-solving efficiencies generally varied between 65% and 75%, depending upon the step size of mapping and transformation temperature. A confidence index (CI) of less than 0.2 was employed to clean up the data points through the CI correlation functions and grain dilation using the TSL software.

TEM foils, discs 3 mm in diameter, were mechanically ground to a thickness of about 0.07 mm and then twin-jet electropolished using a solution containing 5% perchloric acid and 95% methanol at a temperature of −25 °C and a voltage of 50 V. TEM examination of thin foils was performed using a JEM 2000FX microscope operated at 200 kV. The phase and interface characterization were performed using Kikuchi pattern analysis by TOCA software (TSL Inc.) within a TEM image. A conventional X-ray diffraction technique was performed to measure the volume fraction of retained austenite at different heat-treatment conditions using a Philips PW 1130 (40 kV, 25 mA) diffractometer at Sumitomo Metal Industries Ltd. Hardness was measured by the standard Vickers technique using a 20 kg load.

A bainitic structure was formed at temperatures of 200 and 350 °C. However, the time for completion of the bainitic transformation increased significantly with a decrease in the isothermal transformation temperature from 1 day at 350 °C to 10 days at 200 °C. The microstructure after isothermal treatment at 200 °C was notably refined compared with 350 °C (Fig. 1) and resulted in an increase in the hardness from 420 ± 6 to $648 \pm 7\ HV_{20kgf}$.

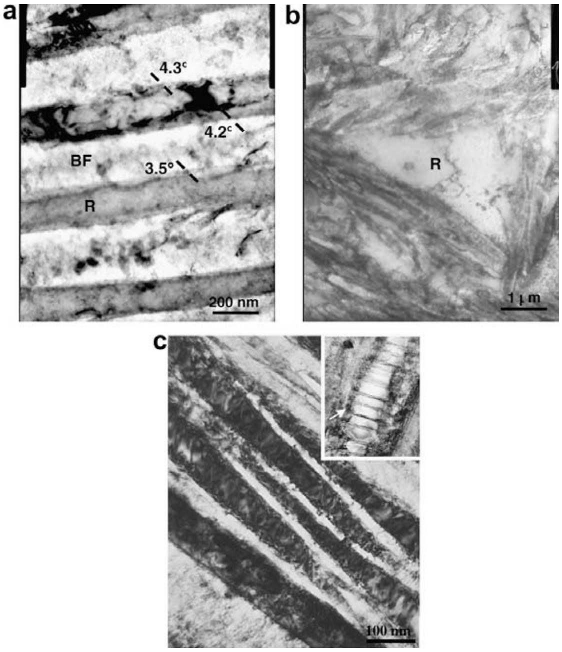

Figure 1. TEM images of bainitic structure at different transformation temperatures: (a and b) 350 °C, (c) 200 °C. R and BF correspond to the retained austenite and bainitic ferrite, respectively. Numbers illustrate the deviation angle from the ideal K–S orientation relationship.

The isothermal bainite transformation temperature had a significant effect on the retained austenite characteristics such as volume fraction, morphology and size. At 350 °C the retained austenite appeared in both blocky and film morphologies (Fig. 1a and b). The bainitic ferrite laths and retained austenite films had an average thickness of 0.3 ± 0.1 and 0.07 ± 0.03 μm, respectively. However, the microstructure after the 200 °C isothermal treatment consisted of nanosized retained austenite films (i.e. 30 ± 10 nm) embedded with the highly dislocated bainitic ferrite laths with an average thickness of 60 ± 10 nm (Fig. 1c). Extensive twining was also observed in the retained austenite films after isothermal treatment at 200 °C (shown by the arrow in Fig. 1c). Interestingly, the volume fraction of retained austenite reduced with a decrease in the isothermal temperature from 53 ± 1% at 350 °C to 21 ± 2% at 200 °C.

In bainitic transformation, it is generally assumed that a close-packed {1 1 1} plane of the austenite [face-centered cubic (fcc)] is exactly parallel to a {1 1 0} plane of the bainitic ferrite [body-centered cubic (bcc)] similar to the martensitic transformation. The transformed products, however, possess the lattice invariant line rather the plane and direction parallelism between bainite and parent austenite. Therefore, the relative orientation can never be precisely the Nishiyama–Wasserman (N–W) relation or the Kurdjumov–Sachs (K–S) relation, though it may alter between K–S and N–W depending on the ratio of lattice parameters between the parent austenite and bainite [16]. In this study, the closest ideal orientation relationship to the nanostructured bainite and parent austenite was assumed and the crystallographic characteristics were further examined accordingly. The difference between the K–S and N–W orientation relationship is about 5.26° [17]. TEM crystallographic analysis of the bainitic ferrite lath Kikuchi patterns and their adjacent retained austenite films were performed to find their interface characteristics (i.e. orientation relationship). The result revealed that the orientation relationship of the interface generally deviated from K–S (Fig. 1b).

In addition to the TEM technique, which can only examine a limited area, EBSD was also employed to characterize the bainitic ferrite lath/retained austenite interface over a relatively large area (Fig. 2). Possible

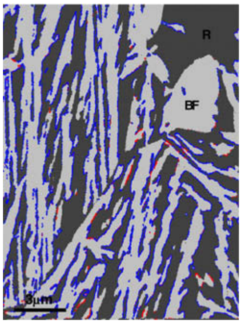

Figure 2. EBSD image of bainitic structure formed at 350 °C. Blue and green lines represent N–W and K–S orientation relationship, respectively. R is the retained austenite and BF corresponds to the bainitic ferrite.