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On the crystallographic characteristics of nanobainitic steel

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ABSTRACT

This study aims to elucidate the crystallographic characteristics of bainite transformed in a temperature range of 200–350 °C, where a nanobainitic structure is formed. The microstructure, consisting of bainitic ferrite laths and retained austenite, became significantly refined and its crystallographic arrangement changed with a decrease in the phase transformation temperature. At 200–250 °C, the bainite packets mostly consisted of one or more blocks (i.e. bainitic ferrite laths and retained austenite lamellae) with different orientations, having a common habit plane. Some of the bainitic laths formed in this temperature range were composed of small segments with similar orientations, while others displayed a ragged morphology with small protrusions, suggesting face-to-face and face-to-edge sympathetic nucleation, respectively. At 300–350 °C, the latter nucleation mechanism appeared to be dominant, as bainite packets mostly consisted of two sets of bainitic ferrite laths with similar orientations and inclined to each other (i.e. having different habit planes). In general, all rational orientation relationships (ORs) ranging from Kurdjumov-Sachs (K-S) through to Nishiyama-Wassermann (N-W) were observed within the transformation temperature range. The N-W OR was dominant at 350 °C and progressively changed towards the K-S OR, which was prevalent at 200 °C. The five-parameter crystallographic approach was used to statistically measure the habit plane distributions for both bainitic ferrite and retained austenite, which were generally found to be irrational and exhibited a significant anisotropy. The bainitic ferrite interface plane distribution displayed a wide peak spreading from (101) to (535). The retained austenite revealed a maximum at the (111) orientation, extending towards the (554).

1. Introduction

Bainite transformation taking place in the iron-based alloys in the intermediate temperature regime between polygonal ferrite and martensite is of a major technological significance, as the bainite phase represents an important microstructure constituent in a range of modern steels. Bainite consists of ferrite crystals (e.g., laths), containing medium to high density of dislocations, arranged in complex packets [1,2]. The crystallography of bainite is generally described through different orientation relationships between parent austenite and bainitic ferrite, ranging from Nishiyama-Wassermann (N-W) through Kurdjumov-Sachs (K-S). This theoretically results in the formation of 12 or 24 distinct crystallographic equivalent variants/orientations from a given parent austenite crystal depending on the orientation relationship. The inter-variant boundaries, resulted from the impingement of these variants, play a significant role in controlling the crack propagation path and consequently the fracture toughness of the steel [3,4].

New advances in bainitic steels led to the development of nanostructured bainite consisting of very fine bainitic ferrite laths (~50 nm) and retained austenite films between them, which offers a unique combination of mechanical properties. The composition of nanobainitic steels was designed based on the thermodynamic approach to obtain a very fine bainitic microstructure at relatively low isothermal transformation temperature regime (i.e., as low as 150 °C) [5–7]. These steels usually contain high amount of carbon content (i.e., >0.7 wt%) and other alloying elements (e.g., Si, Mn, Co, Al, …), which significantly slow down the bainite formation kinetics and result in the transformation taking up to 14 days to complete. The slow phase transformation kinetics, as well as the

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presence of a relatively high volume fraction of retained austenite (20–50%) [8], offers an opportunity to undertake a direct examination of various aspects of the bainitic phase transformation. The nanobainitic microstructure characteristics markedly differ from those of the conventional lower bainite microstructure typically formed at a temperature regime of 200–350 °C in steels having a carbon content of >0.8 wt% with relatively lower alloying elements compared with nanobainitic steels. The lower bainite microstructure is characterized by a plate-like bainitic ferrite morphology frequently containing very fine transition carbide precipitates largely having a common crystallographic orientation [9]. By contrast, bainitic ferrite in nanobainitic steel has a lath morphology containing few transition carbides formed through an auto-tempering phenomenon during the prolonged isothermal treatment. The extent of carbide formation is significantly lower than that in lower bainitic ferrite due to the presence of Si in the nanobainitic steel composition [10].

Some aspects of nanobainitic steels have been rather thoroughly studied, e.g., the variant selection mechanism [8,11,12] and the carbon partitioning using an *in situ* neutron diffraction [11,13,14]. This unique microstructure also potentially enables the characterization of the orientation relationship and the interface character (i.e. habit plane) between bainitic ferrite and retained austenite because these crystallographic characteristics, to a large extent, control the plasticity of steel. For example, slip transfer across the interface appears to be easier when the adjacent phases possess an orientation relationship close to K-S [15,16]. Furthermore, it is reasonable to expect that the ease of dislocation transfer across the interface might be controlled by the character of the habit plane.

Recent reports have also revealed that the phase transformation temperature might have a strong effect on the variant selection, size and microstructural arrangement of bainite laths [2,8,11,12]. Lowering the transformation temperature enhances the phase transformation driving force, which leads to more frequent nucleation of ferrite variants (i.e. weakening the variant selection). Furthermore, it promotes self-accommodation between ferritic laths because of increasing strength in the austenite matrix. Both these aspects ultimately limit the bainitic lath growth resulting in refinement of the microstructure [1,2,8,17]. Nevertheless, the effect of transformation temperature on the orientation relationship and the interface character (e.g., habit plane) has to date received only limited attention. The current study aims to further elucidate these issues in the relatively low temperature regime based on observations in an advanced nanobainitic steel.

As for grain boundaries, five independent crystallographic parameters are required to characterize the phase transformation interfaces (i.e., habit plane). They consist of three parameters specifying the lattice misorientation across adjacent retained austenite and bainitic ferrite, and two parameters defining the habit plane orientation/s [18]. The former can be potentially measured using conventional electron backscatter diffraction (EBSD) technique, while the latter requires three-dimensional (3-D) measurements using transmission electron microscopy (TEM) or serial sectioning in conjunction with EBSD. TEM was used extensively in both martensite and bainite microstructures to examine the austenite habit plane [19,20], though the results were not consistent, probably because of the limited number of interfaces examined in TEM.

3-D techniques have mostly been employed to measure the grain boundary distribution in single-phase polycrystalline materials with a grain size of more than 2 μm [21–23]. There were also some attempts to measure the orientation and morphology of coarse martensite using 3-D EBSD, though this was restricted to only a few crystals [24–26]. The presence of nano-scale constituents (i.e. 50–200 nm) in the nanobainitic steel microstructure restricts the use of 3D-EBSD due to its limited spatial resolution of ~40 nm. New advances in interface/boundary characterization led to the development of a novel approach to statistically measure all five independent interface parameters using the conventional EBSD orientation mapping. The five-parameter approach, which is described in detail elsewhere [18], was employed for a wide range of single-phase materials such as ceramics [27–29] and metals [30–32].

One of the objectives of the current work is to further develop the five-parameter analysis approach to characterize the habit plane/interface distribution in a two-phase microstructure. To overcome the spatial resolution of the conventional EBSD technique, the relatively new orientation mapping technique, viz. precession electron diffraction (PED) in the TEM, was employed to carry out orientation and phase mapping with high resolution. This technique has a spatial resolution of ~2 nm, which makes it possible to map all constituents present in the nanobainitic microstructure accurately.

2. Experimental procedure

Steel with a composition of 0.79C–1.5Si-1.98Mn-0.98Cr-0.24Mo-1.06Al-1.58Co (wt.%) was used in the current study. The presence of high fractions of alloying elements in the steel resulted in high hardenability and a relatively low bainitic transformation temperature regime. The starting bainite (Bs) and martensite (Ms) phase transformation temperatures were 385 and 155 °C, respectively [33]. The specimens were initially reheated to 1100 °C in a muffle furnace and held for 30 min, obtaining an average austenite grain size of ~60 μm. The samples were then placed in a salt bath furnace at different bainitic transformation temperatures ranging from 200 °C to 350 °C at 50 °C intervals. The sample was held isothermally at each temperature so that the bainitic phase transformation went to completion. The isothermal holding times were 1 day, 2 days, 5 days and 10 days for isothermal holding temperatures of 350 °C, 300 °C, 250 °C and 200 °C, respectively.

Thin foil samples for transmission electron microscopy (TEM) were initially prepared using 3 mm diameter discs mechanically ground to ~0.07 mm thickness. They were 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. The TEM examination was conducted using JEOL JEM 2100F and Philips CM20 microscopes operated at 200 kV. The dislocation density of the bainitic ferrite was calculated as $\Lambda = 2N/Lt$, where N is the number of intersections with dislocations made by random lines with length L and t is the foil thickness [34]. Four to five of the bright and dark field images at different tilts and magnification of 100,000 times were used. The foil thickness, t, was determined from intensity oscillations in the two-beam convergent beam electron diffraction patterns [35].

Samples for electron backscatter diffraction (EBSD) were prepared by standard mechanical polishing, finished with a colloidal silica slurry polish. EBSD measurements were carried out using a FEGSEM Quanta 3D FEI scanning electron microscope operated at 20 kV. The instrument was equipped with a fully automated EBSD device attachment. Data acquisition and post processing were performed using the TexSEM Laboratories, Inc. software (TSL). The maps were acquired using a spatial step size of 0.15 μm on a hexagonal grid. The total mapped area covered approximately $245 \times 255\ \mu m^2$, containing more than 20 prior austenite grains. The average confidence index generally varied between 0.45 and 0.55, depending on the isothermal holding temperature. All EBSD maps were first subjected to a grain dilation clean up function to eliminate ambiguous data. A single orientation was then assigned to a given grain by averaging all orientation data belonging to that