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Short communication

Instrumented indentation study of bainite/martensite duplex microstructure

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ABSTRACT

Local mechanical properties of inverse bainite have been characterized using instrumented indentation. The results show that the hardness and Young's modulus of inverse bainite decreased with the increase in transformation time. The Young's modulus was significantly different from that of the martensitic matrix. Local misorientation analysis confirmed that the dislocation density in the inverse bainite decreases, eventually reducing Young's modulus of inverse bainite as transformation times are increased.

1. Introduction

Inverse bainite is an eutectoid transformation product formed at the bainitic transformation temperatures in hypereutectoid steels. Hillert in 1957 [1] proposed the existence of symmetry among the eutectoid transformation products in the iron-carbon system. According to Hillert, bainite and pearlite are eutectoid transformation products with ferrite nucleation being the primary transformation event from parent austenite, and a cooperative growth between ferrite and cementite from parent austenite respectively. He proposed that there must be a third eutectoid transformation product with cementite nucleation being the primary transformation event from the parent austenite, called “inverse bainite". Inverse bainite has been discussed only a few times since Hillert's proposal [2–5], focusing on understanding the microstructure and presenting evidence for the existence of inverse bainite. Recent investigations on inverse bainite include the one by Kolmskog and Borgenstam [6,7] in which they used the existence of inverse bainite to claim that bainite transformation being a diffusion controlled transformation, Goulas [8] provided evidence for the formation of inverse bainite in hypoeutectoid steel with Cr segregation. In our earlier articles [9,10] on the identification of inverse bainite we studied the microstructure evolution during inverse bainitic transformation. Though microstructure characterization is important in order to understand the evolution of microstructure, the job of a metallurgist is never complete until the structure-property correlation loop is closed. It is well known that the morphology of the bainitic microstructure present in a bainite/martensite duplex microstructure, significantly affects the mechanical properties of the end product [11–14]. The arguments for the effect of the bainite morphology on the strength and ductility are based on the martensite packet size refinement, the carbide size in the bainitic microstructure, and the dislocation density. For example, upper bainite with coarser carbides, growth by filling the prior austenite grains (PAGs), and a lower dislocation density significantly reduces the strength. In the case of lower bainite, which has a finer carbides, growth by partitioning the PAG (thereby refining the martensite packet size), and a higher dislocation density significantly improves the strength. The ductility of bainite/martensite two-phase microstructures is dependent on the carbide size and the extent of prior austenite grain refinement. For example, upper bainite which grows by filling the prior austenite grains and with a coarser carbide size has a poor ductility in comparison with lower bainite which grows by partitioning the prior austenite grains, and with a finer carbide size. Though adequate literature is available on the effect of bainite/martensite duplex microstructure on the mechanical properties, to the author's knowledge, the effect of inverse bainite/martensite duplex microstructure on the mechanical properties has not been characterized.

The instrumented indentation technique is well suited to analyze the mechanical response of individual micro constituents. Many previous studies have utilized this technique to analyze the elastic modulus, hardness, dislocation density in materials with heterogeneous microstructures [15–20]. We believe that microstructure-mechanical property correlation should be made, which will, in turn, facilitate in engineering the microstructure for the required end product application. In this study, we used instrumented indentation technique on a duplex microstructure of inverse bainite in a martensitic matrix, to analyze the mechanical response of the microstructure. Moreover, the strength differences between inverse bainite and martensite are critically analyzed.

2. Experimental procedure

The material investigated here is a hypereutectoid (wt% C in the

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range 0.77–0.85) steel containing 2 wt% Cr and Mn combined. Isothermal holding experiments were conducted in the bainitic transformation regime for the alloy using a RITA L78™ high-speed quench dilatometer. Samples were taken to an austenitization temperature of 1323 K (1050 °C) (at the rate of 10 K/s) and held there for 5 min, followed by cooling the samples (at a cooling rate of 5 K/s which is faster than the critical cooling rate required to form bainite upon continuous cooling) to the isothermal holding temperature of 773 K (500 °C). In order to study the microstructural evolution, samples were held at 773 K (500 °C) for 1 min, 1.5 min, 3 min, 5 min, 7 min, and 10 min and cooled (at the rate of 5 K/s) to room temperature.

For local misorientation characterization using EBSD, the heat-treated samples were mounted, ground and polished by using a Buehler Ecomet™ 250/300 grinder-polisher with a power head. Grit 320, 600, and 1200 SiC sandpapers were used for grinding. 3 µm and 1 µm diamond suspension, 0.5 µm and 0.05 µm alumina suspension and 0.02 µm colloidal silica were used for polishing. EBSD analysis of the heat-treated samples were conducted on a Zeiss Sigma™ FESEM equipped with Oxford AZtec EBSD system. The operating conditions of the SEM were 20 kV accelerate voltage, 60 µm objective aperture, and a step size of 20–80 nm. For indexing the Kikuchi bands, *HKL* and ICSD (Inorganic Crystal Structure Database) databases were used. EBSD data processing (reconstructed maps) was carried out using Channel 5 data processing software.

Indentation tests were carried out using a Fischerscope H100C with a standard diamond pyramid Vicker's indenter. The load resolution and distance resolution of the instrument were < 0.04 mN and < 0.1 nm respectively. The experiments were carried out in the load controlled mode with a peak load of 50 mN applied over a period of 20 s. The instrumented indentation experiments were carried out on the same surface as the one used for EBSD data acquisition. Several indentations were carried out in the bainitic regions and the martensitic matrix of all the samples and the presented load-depth curves are representative. The load-depth curves were analyzed using the Oliver and Pharr analysis [21] for the indentations made and the reported values of elastic modulus and hardness are the average values. To compare the instrumented indentation results, microhardness measurements were also carried out using Tukon™ 2500 Vickers hardness tester. A load of 0.05 Kgf[1] was used for a duration of 10 s to measure the hardness values. The fraction of inverse bainite was measured using thresholding technique [22] and the open source software ImageJ™.

3. Results

3.1. Representative microstructures

Fig. 1a, Fig. 1b, Fig. 1c represents the representative SEM microstructures of the 1 min, 3 min, and the 10 min heat treated samples. It can be seen that in the case of 1-min heat-treated sample, carbide formation is observed. In the case of 3-min heat treated sample, the formation of ferrite surrounding a carbide unit is observed. This microstructure of the 1-min and the 3-min heat-treated samples are similar to the ones which we previously reported for inverse bainite in Ref. [9]. In the case of 10 min heat treated sample, the bainitic microstructure does not appear to have a carbide midrib or the cementite midrib of the inverse bainitic unit has degenerated to typical upper bainitic microstructure. More information on the formation of the degenerated microstructure of upper bainite is explained in Ref. [10]. It is to be brought to the reader's attention that the microstructures of 1.5 min and 7 min isothermal hold were similar to 1 min and 10 min isothermal hold. The microstructure of the 5 min heat-treated sample has been reported in Ref. [9].

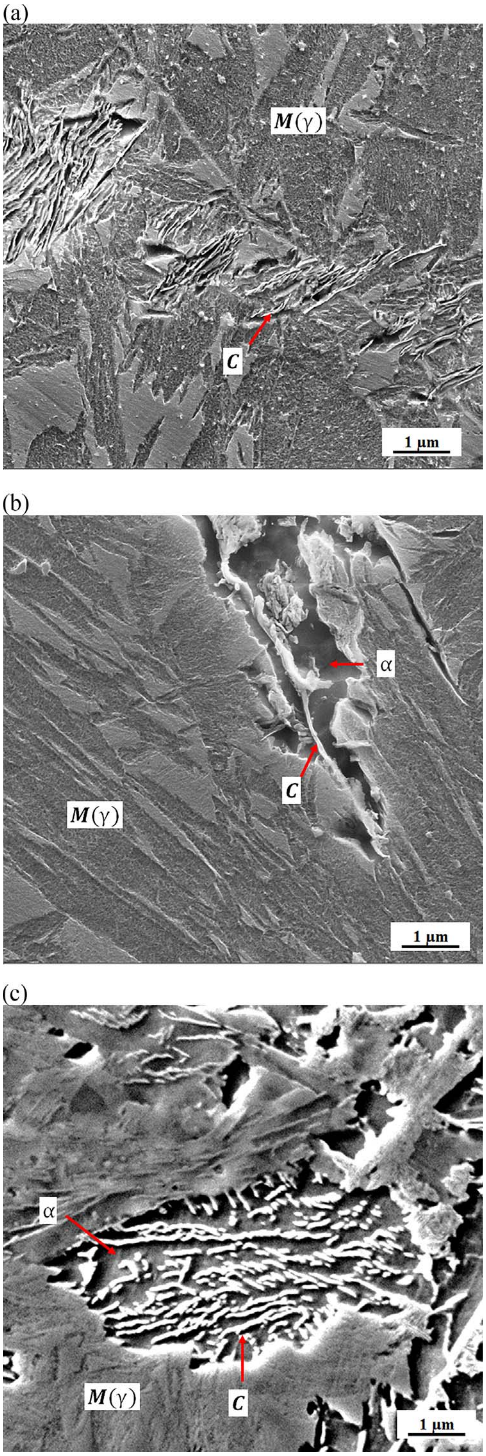

Fig. 1. Representative secondary electron SEM micrographs of (a) 1 min heat treated sample, (b) 3 min heat treated samples, and (c) 10 min heat treated sample. In the figures, C represents the carbide unit of inverse bainite, α represents the inverse bainitic ferrite, and M(γ) represents the martensite/retained austenite matrix.

3.2. Instrumented indentation load-displacement curves

Representative load progression curves during the instrumented indentation experiments are shown in Fig. 2. It can be seen that the martensitic matrix has the least slope of the load-displacement curve. For the bainitic regions, the slope initially increases and appears to be a constant with the increase in the transformation time. The maximum depth of penetration for the as-quenched martensitic matrix, 1 min

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[1] Lower load was used to characterize the hardness of bainitic regions, the fraction of which were low especially at lower isothermal holding time.