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Atomic scale observations of bainite transformation in a high carbon high silicon steel

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Abstract

A fine-scale bainitic microstructure with high strength and high toughness has been achieved by transforming austenite at 200 °C. X-ray diffraction analysis showed the carbon concentration of these bainitic ferrite plates to be higher than expected from para-equilibrium. Atom-probe tomography revealed that a substantial quantity of carbon was trapped at dislocations in the vicinity of the ferrite–austenite interface. These results suggest that the carbon trapping at dislocations prevents the decarburization of super-saturated bainitic ferrite and therefore alters the carbide precipitation sequence during low-temperature bainite formation.

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1. Introduction

Previous research has shown that a steel with \sim 2 wt.% of silicon can, in appropriate circumstances, lead to a carbide-free bainitic microstructure. In such steels, the bainitic microstructure consists of a mixture of bainitic ferrite and carbon-enriched residual austenite. The presence of silicon retards the precipitation of cementite from austenite because of its low-solid solubility in the cementite crystal structure. Cementite is a cleavage and void-initiating phase and therefore elimination of the same is expected to yield a high strength and high toughness bainitic microstructure. In agreement with this hypothesis, with careful alloy design and using phase transformation theories steels with impressive combinations of strength and toughness can be developed [1–11]. In particular, the experimental validations based on this design have led to a new carbide-free bainitic steel with a very high toughness (30 MPa m^{1/2}) and a strength of 2500 MPa [8-11]. In this steel, the bainitic

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microstructure was obtained by transforming at temperatures as low as 200 °C. At this temperature, within the given time-scales, the diffusion of iron or substitutional solutes is very sluggish. The low B_s (bainitic start) temperature is a consequence of the high carbon content and, to a lesser extent, the addition of solutes such as manganese and chromium, which in the present context increase the stability of austenite relative to ferrite. As a result of the low transformation temperature, bainite plate widths of 20–40 nm thickness were achieved, which in turn leads to high strength. Therefore, this alloy can be considered as a bulk nanocrystalline material that can be obtained by a cheap manufacturing route without expensive processing.

Extensive transmission electron microscopy (TEM) of this novel microstructure has failed to reveal carbide particles inside the bainitic ferrite. This is indeed an interesting observation, since at these temperatures a steel with such high carbon levels would transform to a lower bainitic microstructure. After extensive aging at 200 °C for 2 weeks, just a few 20 nm wide and 175 nm long cementite particles were observed in a thicker bainitic ferrite plate [11]. The difference between upper and lower bainite comes from

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competition between the rate at which carbides can precipitate from ferrite and the rate with which carbon is partitioned from super-saturated ferrite into austenite [12]. The precipitation of cementite from lower bainite can occur at temperatures below 125 °C in time periods too short to allow any substitutional diffusion of iron atoms. The long-range diffusion of carbon atoms is of course necessary, but, because carbon resides in interstitial solution, it can be very mobile at temperatures as low as -60 °C [13]. The formation of cementite or other transition carbides of iron, such as \(\epsilon\)-carbide, in these circumstances of incredibly low atomic mobility must differ from diffusional decomposition reactions. It has been suggested that [14] the cementite lattice is generated by a displacive mechanism with para-equilibrium, i.e. a homogeneous deformation of super-saturated ferrite combined with the necessary diffusion of carbon.

X-ray diffraction analysis indicated that the carbon concentration in the bainitic ferrite was much higher than that expected from the para-equilibrium thermodynamics between austenite and ferrite [11]. This super-saturation was attributed to the trapping of carbon at the dislocations in the bainitic ferrite. Smith [15] estimated a mean dislocation density of $4\times10^{14}/m^2$ using TEM in a Fe–0.07C–0.23Ti wt.% alloy when isothermally transformed to bainite at 650 °C. This relatively high dislocation density was attributed to the fact that shape deformation accompanying displacive transformations is accommodated partially by plastic relaxation [16]. However, no direct observation has been yet reported of the interstitial carbon Cottrell atmosphere in bainitic ferrite.

Therefore, the high dislocation density in bainitic microstructures may play an important role on transformation processes such as the decarburization of super-saturated ferrite. The goals of this work were to analyze the solute redistribution between ferrite and austenite as well as evaluating the hypothesis of carbon trapping at dislocations within bainitic ferrite by atom-probe tomography (APT).

2. Experimental procedure

The chemical composition of the steel studied is given in Table 1. The alloy was supplied as a cast ingot, samples of which were then homogenized at $1200 \,^{\circ}\text{C}$ for 48 h in partially evacuated sealed quartz capsules that were flushed with argon. The sealed samples were cooled in air following the homogenization heat treatment. The martensite start (M_s) temperature of the steel was measured by dilatometry monitoring the related change in dilatation with temperature. A value of $M_s = 120 \,^{\circ}\text{C}$ was recorded on dilatometric

Table 1 Chemical composition

	C	Si	Mn	Mo	Cr	V
wt.%	0.98	1.46	1.89	0.26	1.26	0.09
at.%	4.34	2.76	1.82	0.14	1.28	0.09

curves. Homogenized specimens were austenitized for 15 min at 1000 °C and then isothermally transformed at temperature in the range 200–325 °C for different times before quenching into water.

Quantitative X-ray analysis was used for determining the volume fraction of retained austenite. After grinding and final polishing with a 1 μ m diamond paste, the samples were etched to obtain a non-deformed surface. Moreover, TEM specimens were machined to 3 mm diameter rods and electro-polished with a twin-jet electro-polisher at room temperature in a mixture of 5% perchloric acid, 15% glycerol and 80% methanol at 40 V until perforation occurred.

APT specimens (see Table 2 for details) were cut from the bulk material and electro-polished with the standard double-layer and micro-polishing methods [17]. Atomprobe analyses were performed using the Oak Ridge National Laboratory local electrode atom-probe system. The large field of view and rapid analysis capability of this instrument facilitated the analysis of dislocations in these materials. The local electrode atom-probe was operated with a specimen temperature of 60 K, a pulse repetition rate of 200 kHz and a pulse fraction of 20%.

3. Results and discussion

3.1. Kinetics of bainite transformation

The micrographs obtained after isothermal transformation of austenite to bainitic ferrite at 200 and 300 °C at different time intervals are illustrated in Fig. 1. After 24 h of holding time at 200 °C, bainite transformation had not started and a mixture of martensite and retained austenite was obtained by quenching (Fig. 1a). A longer annealing time (48 h) at this temperature was required in order to obtain a significant amount of bainitic transformation, as shown in Fig. 1b. Transformation was completed after 144 h of holding time when a fully bainitic microstructure was obtained (Fig. 1c). The rate of transformation increased at 300 °C. For example, a visible amount of bainitic microstructure (~10%) was observed at 300 °C after 4 h of transformation. A fully bainitic microstructure was achieved at this temperature after 12 h (see Fig. 1d and f).

Table 2 Samples analyzed by APT

Transformation temperature (°C)	Time (h)	
200	24	
200	48	
200	96	
200	144	
200	240	
300	1	
300	4	
300	8	
300	12	
300	19	

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