Influence of transformation temperature on carbide precipitation sequence during lower bainite formation

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HIGHLIGHTS

- Identification of carbides in lower bainite using atom probe tomography.
- Determination of carbon trapping at dislocations in lower bainite.
- Carbide precipitation sequence during lower bainite formation.

ARTICLE INFO

ABSTRACT

The nature of different carbides formed during the lower bainite reaction at different transformation temperatures was determined by atom probe tomography in three steels with different carbon and silicon contents. It is known that steel composition and temperature alter the carbide precipitation sequence during low-temperature bainite formation. However, present results confirm that dislocations in lower bainite that are more prominent at the lower the transformation temperature, trap a substantial amount of carbon and hence, can influence the carbide precipitation sequence and, in particular, can determine where ε-carbide forms before the growth of cementite.

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1. Introduction

The difference in carbide distribution between bainite formed at high and low temperatures, viz., interlath and intralath, respectively, appears to exist in a majority of steels and makes the classical nomenclature of upper and lower bainite useful in describing the appearance of the microstructure. The precipitation of carbides during the bainite reaction is a secondary process, not essential to the mechanism of formation of bainitic ferrite, except where any precipitation influences the reaction rate by removing carbon either from the residual austenite or from the supersaturated ferrite [1,2]. In upper bainite, the carbides precipitate from the carbon-enriched residual austenite between the developing laths. Thus, upper bainitic ferrite itself is generally free from intralath precipitates. In contrast, many observations reveal that lower bainitic cementite nucleates and grows within supersaturated bainitic ferrite in a process identical to the tempering of martensite [3]. The

slower diffusion associated with the reduced transformation temperature provides an opportunity for some of the carbon to precipitate in the supersaturated bainitic ferrite. A fine dispersion of plate-like carbides is then found inside the bainitic ferrite, which also has a plate morphology, with a single crystallographic variant within a given bainitic ferrite plate, although it is possible to observe more than one variant of carbide precipitation in a lower bainite sub-unit [3,4].

Matas and Hehemann [5] first suggested that the initial carbide in lower bainite is ε-carbide, which is subsequently dissolved and replaced by cementite. The rate at which the ε-carbide converts to cementite increases with the temperature, but also depends on the steel composition. In fact, a high silicon concentration retards the reaction, as is commonly observed in the tempering of martensite [6–11]. Owen [8] proposed that silicon rejected from cementite acts as a growth barrier and causes the inhibition of cementite precipitation. Since then, partitioning of silicon between cementite and ferrite has attracted considerable attention [12–15].

The detection of ε-carbide instead of cementite in lower bainite implies the existence of a significant amount of carbon in supersaturation in bainitic ferrite [16]. However, ε-carbide is not always

Table 1
Chemical composition of studied steels, wt-%. The balance is Fe.

Steel	C	Si	Mn	Ni	Cr	Mo	V
MC-LSi (at. %)	0.30 (1.38)	0.25 (0.49)	1.22 (1.22)	0.10 (0.09)	0.14 (0.15)	0.03 (0.02)	–
MC-HSi (at. %)	0.29 (1.32)	1.48 (2.87)	2.06 (2.04)	–	0.43 (0.45)	0.27 (0.15)	–
HC-HSi (at. %)	0.98 (4.34)	1.46 (2.76)	1.89 (1.82)	–	1.26 (1.28)	0.26 (0.14)	0.09 (0.09)

found as a precursor to the precipitation of cementite in lower bainite. Bhadeshia and Edmonds [17] failed to detect ε-carbide in a high silicon medium carbon steel even during the early stages of the lower bainite transformation.

Kozeschnik and Bhadeshia [18] utilised theory of the kinetics of precipitation and provided a quantitative framework for the formation of cementite from supersaturated ferrite. They conducted calculations for the so-called 4340 steel with a chemical composition of Fe-0.4C-0.7Mn-0.28Si-0.8Cr−-.8Ni-0.25Mo (wt.%) and the alloy 300M with essentially the same composition, but with the silicon concentration increased to 1.6 wt.%. The 4340 steel is a high strength steel used in the quenched and tempered condition that exhibits tempered martensite embrittlement at 350 °C [19]. Tempered martensite embrittlement is associated with the formation of coarse cementite, which dramatically reduces toughness in a high strength matrix. By contrast, alloy 300M with a higher silicon concentration, added to retard the para-equilibrium precipitation of cementite, can be tempered at just 315 °C without embrittlement, leading to a much greater toughness.

Calculations by Ghosh and Olson [20] suggested, however, that precipitation is too rapid to have a perceptible effect of silicon. Kozeschnik and Bhadeshia's calculations [18] on the kinetics of cementite precipitation from supersaturated ferrite confirmed that the cementite precipitates rapidly during tempering at 315 °C, with little meaningful difference between the silicon enriched 300M and low silicon 4340 steels. One probable explanation for the discrepancy between calculations [18,20] and experimental observations on the tempering of martensite comes from earlier classic work by Kalish and Cohen [21]. They proposed that in the presence of dislocations, carbon prefers to segregate to dislocations rather than precipitate as cementite or ε-carbide.

Defects can effectively be thought of as a separate trapping site, which is a greater attractor for carbon than cementite. In these circumstances, the carbon available for precipitation as cementite is reduced. The rate of precipitation then depends on the removal of defects during annealing, making carbon available for cementite formation [22]. This process must greatly retard tempering kinetics and, because of the reduced carbon concentration in the perfect lattice, lead to a smaller driving force for precipitation and hence, a larger difference between the silicon-enriched and low-silicon steels.

In the present work, the nature of the iron carbides formed during lower bainite reaction was determined by atom probe tomography (APT) in steels transformed over a wide range of temperatures (200−425 °C). In addition, carbon trapping at dislocations was analysed to elucidate the role of defects altering the carbide precipitation sequence during lower bainite formation.

The carbon concentration of precipitates obtained from APT was used to discriminate amongst ε-carbide ($Fe_{2.4}C$ with ~30 at.% C), cementite (Fe_3C with ~25 at.% C), and other iron transition carbides, such as Fe_4C with ~20 at.% C and Fe_8C with ~11 at.% C. Previous work [23,24] focused on the analysis of iron carbides during tempering of a high-carbon, high-silicon bainitic steel, and the redistribution of substitutional solute across the carbide/ferrite interface, with special attention given to silicon. The goal of this study is to examine the carbide precipitation during lower bainite

reaction at different temperatures in three steels with different carbon and silicon contents.

2. Materials and experimental procedure

Three steels with different carbon and silicon contents were selected to study the precipitation of carbides during bainite transformation at different temperatures. The bainite transformation temperature range of the steel is mainly a consequence of the carbon content, whereas silicon is known to retard cementite precipitation [8] due to its extremely low solubility in the cementite crystal structure. It is expected that the presence of silicon will depress the formation temperature of lower bainite since silicon prolongs the time needed for carbide precipitation. The alloys also contain Mn, Ni, V, Cr, and Mo for hardenability. The chemical composition of the steels is given in Table 1.

The medium-carbon and low-silicon (MC-LSi) steel was supplied as 30 mm hot rolled square bar; the medium-carbon and high-silicon (MC-HSi) steel was supplied as 12 mm hot rolled strips; the high-carbon and high-silicon (HC-HSi) steel was supplied as as-cast ingots after homogenization at 1200 °C for 48 h. Further details about the alloying design and manufacturing processes can be found elsewhere [25−27].

Dilatometric and metallographic analyses were used to determine the transformation temperatures in the selected steels. An Adamel Lhomargy DT1000 high-resolution dilatometer was used [28]. Cylindrical dilatometric test pieces of 3 mm in diameter and 12 mm in length were austenitised at the temperatures listed in Table 2, cooled down at 100 °C s^{-1} to bainite transformation temperatures ranging from 200 to 525 °C and then isothermally holding for different times to ensure complete transformation (see Table 3) before quenching to room temperature at a rate of 100 °C s^{-1}.

The martensite start temperatures (M_S) listed in Table 2 were estimated by dilatometry. Dilatometric specimens were austenised using a heating rate of 5 °C s^{-1} and then rapidly cooled at rate of 100 °C s^{-1}. Each test was performed twice. The formation of martensite during cooling was detected by monitoring the fractional change in dilatation with temperature.

Conventional metallographic examination by Light Optical Microscopy (LOM) and Scanning Electron Microscopy (SEM) was used to determine the bainite start temperatures (B_S) that are shown in Table 2. A JEOL model JSM-6500F Field Emission SEM operated at 7 kV was used for this purpose.

Transmission Electron Microscopy (TEM) specimens were made from disks that were cut from 3-mm-diameter rods and electropolished with a twin-jet electropolisher at room temperature in a mixture of 5% perchloric acid, 15% glycerol, and 80% methanol at

Table 2
Austenitisation conditions and experimental critical transformation temperatures.

Steel	Austenitisation conditions	B_S, °C	LB_S, °C	M_S, °C
MC-LSi	1200 °C for 60 s	525 ± 12	450 ± 12	342 ± 2
MC-HSi	925 °C for 300 s	450 ± 12	400 ± 12	299 ± 8
HC-HSi	1000 °C for 900 s	335 ± 12	335 ± 12	123 ± 4

B_S bainite start, LB_S lower bainite start and M_S martensite start temperatures.