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Incomplete bainite transformation in Fe-Si-C alloys



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ABSTRACT

Bainite isothermal transformation kinetics for Fe-(1.5% and 3%)Si-0.4%C alloys (mass%) was investigated at 400–500 °C and incomplete transformation phenomenon (ICT) of bainite transformation was observed at 450 °C for the 3Si alloy and at 400 °C for the two alloys. Unlike to the ordinary ICT reported in other alloy systems, cementite precipitation with Si partitioning took place from the beginning of ICT. Carbon enrichment in austenite during ICT was measured by three-dimensional atom probe and was found to be higher than T_0 or T_0' prediction while significantly deviates from NPLE limits and PE predictions to lower carbon content. Theories for bainite transformation, such as T_0 limit, solute drag and WB_s limit, were examined based on the experimentally measured carbon content in austenite during ICT. T_0' limit theory is difficult to rationalize the much larger measured carbon content than T_0' prediction. In addition to solute drag effect and spike development in the NPLE mode, solute drag theory should incorporate dissipations caused by other sources in order to account for the estimated 1250–1700 J/mol deviation from PE predictions. In addition, WB_s limit theory gives good descriptions on the carbon enrichment in austenite during ICT stage.

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1. Introduction

Bainitic steels whose production involves bainite transformation have been widely applied in industries such as steel sheet for automobile body parts or thick steel plate for vessels or pipelines [1,2]. As bainite transformation proceeds, carbon (C) would be enriched in austenite resulted from the difference of chemical potential of C in austenite and ferrite. Amount and stability of retained austenite which are directly related to its C content are critical for transformation induced plasticity (TRIP) steels to ensure their formability.

During bainite transformation in alloyed steels, transformation may stop prematurely before the equilibrium amount is attained and this phenomenon is called incomplete transformation (ICT) phenomenon [3]. ICT determines the amount of untransformed austenite before cooling as well as its C content which determines its stability during cooling. The cause of ICT phenomenon is interpreted differently in relation to growth mechanism of bainitic ferrite. Two different growth mechanisms, i.e. diffusionless

mechanism [4,5] and C diffusion controlled mechanism [3,6–8], have been proposed. According to diffusionless school, bainitic ferrite inherits all the C of the parent austenite during its growth while C atoms are rejected from supersaturated ferrite into austenite or precipitate as carbide in bainitic ferrite is a latter process. T_0 temperature where austenite and ferrite shares the same Gibbs free energy at the same composition or T_0' temperature which further account the strain energy accumulated during transformation (~400 J/mol [5]) is the upper limit for bainite transformation. Without the interference from other reactions, e.g. carbide precipitation or pearlite formation, ICT will be naturally expected in bainite transformation. Another view insists that bainitic ferrite growth process is controlled by C diffusion in austenite and there is no essential difference between Widmanstätten ferrite and bainitic ferrite. ICT phenomenon was caused by additional energy dissipations resulted from coupled solute drag effect [3,6], Gibbs energy balance [7], thermodynamic barrier for acicular ferrite growth [8,9].

Different theories explaining ICT phenomenon which are further related to the BF growth mechanisms usually give different predictions on C enrichment in untransformed austenite during ICT stage. Fe-Mn-C and Fe-Mn-Si-C alloys are two most widely studied systems and the agreements with diffusionless theory or diffusion control growth theory have been reported [4,9–13]. Si is frequently

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added to Mn [4,9–12] or Ni [5] bearing steels to prevent cementite precipitation [14] for ICT phenomenon. Although C content in austenite during bainite transformation in Fe-1.5%Si-(1–2.5)%Mn-C (mass%, hereafter) is close to T_0 or T_0' predictions, its dependency with Mn content seems to differ from T_0 or T_0' model [9]. Therefore, investigation of C enrichment in austenite during bainite transformation in Mn-free steels, i.e. Fe-Si-C alloy, should shed some light on the mechanism of bainite transformation.

However, there are only few researches studying bainite transformation in Fe-Si-C ternary system [15–17]. The conditions investigated are Fe-0.11%C-1.83%Si and Fe-0.38%C-1.73%Si alloys reacted at temperatures ranging between 395 and 560 °C [15], Fe-0.6%C-2%Si alloy reacted at 450 °C [16] and Fe-0.9%C-3.85%Si alloy at temperature ranging between 250 and 420 °C [17]. However, none of them has found ICT so far. These investigations on the kinetic information were either insufficient or worthy further examination since some of them are based on optical microscope observations [15]. A more systematic investigation on bainite transformation behavior especially on the ICT phenomenon in Fe-Si-C ternary system is needed.

Most frequently applied method to measure C content in austenite is to measure lattice constant of retained austenite using X-ray diffraction (XRD) measurement while compressive stress was suggested to affect lattice constant of retained austenite and may deteriorate accuracy of C content measurement [18]. Recently, direct measurements such as three-dimensional atom probe (3DAP) [13,18] and field emission electron probe microanalysis (FE-EPMA) [19–21] were used to quantitatively measure the C enrichment during phase transformation and to compare various phase boundaries. In present study, ICT phenomenon was systematically investigated with Fe-Si-C alloys to clarify the effect of Si addition as well as bainite transformation mechanism. In particular, the C content of untransformed austenite was measured by 3DAP since the size of untransformed austenite between bainitic ferrite is too fine (less than 0.5 μm in thickness) and FE-EPMA is no longer applicable.

2. Experimental

Two high purity Fe-0.4%C-1.5%Si and Fe-0.4%C-3%Si alloys were used in this study. Hereafter they are referred to as 1.5Si and 3Si alloys. Table 1 lists their exact composition with their para-Ae₃, T_0 and M_s temperatures. The M_s temperatures were calculated based on the empirical equation proposed by Andrew [22]. The other temperatures as well as other thermodynamic calculations in the present study were all performed by Thermo-Calc [23] with TCFe7 database. The alloy ingots were prepared by vacuum melting and casting, followed by hot rolling to ~15 mm. The two alloys were encapsulated into Ar-filled silica tubes and homogenized at 1150 °C for 24 h, after which no banded structure could be observed. C contents after homogenization are measured and shown in Table 1 since slight decarburization occurs during homogenization.

Plate-shaped specimens of ~10mm^l × 5mm^w × 3mm^t were cut from the homogenized alloys and austenitized in vacuum furnace at 1150 °C for 10 min which produces nominal austenite grain size of about 190 μm for both alloys. After austenitization, the

specimens were immediately quenched into salt bath and isothermally held at 500, 450 and 400 °C, respectively for 10 s–1800 s and followed by quenching into iced water.

Transformed specimens were cut in half parallel to the face. Thus, the sections close to the center of the specimens were observed to avoid the effect of the possible loss of C or oxidization during heat treatment. Before etching, standard metallurgical polishing method was performed. 3% nital was used for observation with OM while 1% nital was used for observation with scanning electron microscopy (SEM; JEOL JSM-7001F, operated at 5 kV). Due to the fine microstructure, point counting method with SEM images by two steps was utilized to quantify the fraction of the transformed regions (f_{trans}). In the first step, 10 images with magnification of 1000 (containing ~2 prior austenite grains) was used to count the fraction of transformed area, f_{TA} . Transformed area consists of bainitic ferrite lathes and thin martensite/austenite constituent (MA) between them. In the second step, Another 10 images obtained in transformed areas with magnification of 10,000 are used to count the fraction of MA in those transformed areas, f_{MA} . As a result, under a given condition, f_{trans} could be expressed as:

$$f_{\text{trans}} = (1 - f_{\text{MA}}) \times f_{\text{TA}} \quad (1)$$

XRD measurement was applied to measure the fraction of retained austenite (f_γ) during bainite transformation. The mechanically polished specimens were electrolytically polished using 6% perchloric acid solution with ethanol as solvent for 1 min under 25 V to remove possible damaged layer on the surface. XRD measurement were carried out with a diffractometer (Bruker AXS D8) equipped with a Co X-ray tube (K_α radiation, $\lambda = 1.7889 \text{ \AA}$), Goebel mirror optics and a LynxEye Linear Position Sensitive Detector. A current of 40 mA and a voltage of 35 kV were employed as tube settings. 2.5 × 2.5 slit and 1.0 mm collimator were equipped to align the X ray routine. The XRD data were collected over a 2θ range of 57.5° to 111.5° with a step size of 0.05°/step. In order to minimize a texture effect, specimens were tilted and rotated during measurement. Intensities of (002) γ , (022) γ , (113) γ , (002) α and (112) α peaks are used to calculate fraction of retained austenite based on the method described by Cullity [24]. Amount of retained austenite measured by XRD should be smaller than that of untransformed austenite quantified by point counting. The gap corresponds to the fraction of fresh martensite formed during final quenching.

In order to observe the carbide formed during bainite transformation, a combination of TEM (Philips CM300, operated at 300 kV) observation and 3DAP (CAMECA, LEAP 4000HR) measurement were carried out. Thin-foil specimens for TEM observation were prepared by twin-jet electropolishing at 22 V in 6% perchloric acid solution at –15 °C. The specimens for 3DAP measurement were prepared using a lift-out method [25] with focused ion beam (FIB; FEI Quanta 3D). The temperature for measurement, detection rate, pulse fraction and pulse rate were 80 K, 1%, 20% and 200 kHz, respectively. 3DAP results were analyzed with IVAS Software (CAMECA). The C concentrations in untransformed austenite were also obtained from 3DAP measurement. The apparent C and Si contents were corrected based on a method considering the loss in detection of Fe ions [26]. In this condition, measurement carried out on the as-quenched 3Si specimens showed a typical error of 0.07 mass% for C content.

3. Results

Fig. 1(a) shows the variation in volume fractions of austenite transformed with time for the 1.5Si alloy held at 500 °C. The para-equilibrium (PE), non-partition local equilibrium (NPLE), T_0 predictions in this graph were calculated based on lever rule according

Table 1
Chemical compositions and characteristic temperatures for the two alloys.

Alloy	Compositions (mass%)			Temperatures (°C)		
	C	Si	Fe	Para-Ae ₃	T_0	M_s
1.5Si	0.375	1.48	Bal.	835	674	380
3Si	0.379	3.09	Bal.	902	688	379

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