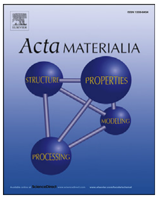
Acta Materialia 133 (2017) 41–54

Contents lists available at ScienceDirect

Acta Materialia

journal homepage: www.elsevier.com/locate/actamat



Full length article

Atomic-scale study on segregation behavior at austenite grain boundaries in boron- and molybdenum-added steels

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ARTICLE INFO

Article history:
Received 6 January 2017
Received in revised form
15 April 2017
Accepted 10 May 2017
Available online 11 May 2017

Keywords:
Segregation
Atom probe
Steel
Hardenability
Cooling
Martensite
Boron
Prior austenite grain boundary

ABSTRACT

The addition of small amount of boron (B) to steels has been found to significantly increase the hardenability of steels during cooling. This is further increased by the addition of molybdenum (Mo) and the combined addition of both. To clarify the mechanism of the Mo-B combined addition effect, quantitative analysis of segregating atoms at prior austenite grain boundaries in B- and Mo-added low-carbon (C) steels was performed using atom probe tomography. The dependences of Mo content and cooling rate on the amounts of B and Mo segregations were systematically investigated in the steels with austenizating temperature of 950 °C. The amount of B segregation at the prior austenite grain boundary was mostly independent of Mo content at a high cooling rate of 30 °C/s. In contrast, at a low cooling rate of 5 °C/s, the amount of B segregation increased in the B-added steel with Mo, and decreased without Mo, whereas the amount of Mo segregation increased with increasing Mo content and with lowering cooling rate.

Such segregation behaviors of B and Mo were explained by the diffusion limited equilibrium segregation and precipitation of $Fe_{23}(CB)_6$ at the austenite grain boundary. We discussed the influence of B and Mo segregations on hardenability from the relation between hardenability index and actual segregation amount in the steels.

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1. Introduction

Hardenability is originally defined as the ability of a steel to transform martensite on quenching. It is an important property particularly in thick plate production to obtain sufficient hardness on water-quenching since the cooling rate in inner region of a plate is sluggish due to the mass-effect. It is known that the addition of small amount of boron (B) into steels significantly increases their hardenability in continuous cooling from austenite [1,2]. It is mainly understood that segregation of B atoms at the austenite grain boundary lowers the grain boundary energy, resulting in the inhibition of ferrite and bainite nucleation [1,3]. The effect can be obtained by an only very small amount of B, of the order of ppm, providing significant impacts on the production of steels. It is however not easy to control the effect of B in an industrial production process because the state of B atoms in the steel significantly influence the its effect on hardenability [1,2].

Furthermore, co-addition of molybdenum (Mo) into the B-added steels increased the hardenability [4–10]. Ueno et al. reported that Mo effectively exhibited a combined addition effect with B in increasing the hardenability in B-added steels, whereas other elements such as silicon, manganese (Mn), and chromium (Cr) did not show such a combined effect [4]. In addition, the Mo-B combined addition effect was also sensitive to the B content and heat treatment. The mechanism of the Mo-B combined addition effect has been investigated by many researchers. Asahi reported that the combined effect of Mo was caused by the retardation of B precipitation $M_{23}(CB)_6$ [5]. Hara et al. reported that the retardation of B precipitation arose from the decrease in C diffusion in the grain because of the formation of Mo–C clusters [6]. Han et al. reported through thermo-dynamic calculations that the suppression of B precipitation was caused by the destabilization of $M_{23}(CB)_6$ phase [7,9]. In addition, we have to consider a possibility that the amount of B segregation at austenite grain boundaries may be increased by Mo addition owing to attractive interaction between the two atoms at the grain boundary.

To clarify the state and distribution of B, the direct observation of B segregation at prior austenite grain boundaries has been

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http://dx.doi.org/10.1016/j.actamat.2017.05.021
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performed using alpha-ray track etching method (ATE) and secondary ion mass spectroscopy (SIMS) [4–11]. The two analysis methods are very sensitive to B segregation; however, quantitative analysis of the amount was difficult. Karlsson et al. reported the cooling rate dependence of B segregation at austenite grain boundary in austenite stainless steels using atom probe field ion microscopy (AP-FIM) in addition to ATE and SIMS [12,13]. It is, however, difficult to obtain a correct profile of segregation at the grain boundary because AP-FIM could not produce a three-dimensional (3D) distribution of atoms segregating at the grain boundary. Shigesato et al. recently reported through aberration-corrected scanning transmission electron microscopy (Cs-STEM) with electron energy loss spectroscopy (EELS) analysis that the profile of B segregation at the prior austenite grain boundary was changed with the cooling rate from austenite [14], but the segregation state of Mo and other elements have not been reported.

Atom probe tomography (APT) has great advantages in conducting a quantitative analysis of each segregating element in high spatial resolution [15]; however, systematic researches have not been performed because it was very difficult to produce a needle tip containing the prior austenite grain boundary at the tip apex. Recently, Li et al. successfully reported the APT analysis of prior austenite grain boundaries in B-added steels with and without Mo [16,17]. They reported that the B atoms segregated at the prior austenite grain boundary but did not segregate at other martensite boundaries (lath, block, and packet) and that the amount of B segregation was not changed by Mo addition. However, the reports did not deal with the influences of Mo content and cooling rate although they are very important factors for hardenability.

In this study, systematic researches were performed to clarify the behavior of B and Mo segregations at austenite grain boundaries in B-added low C steels with different Mo contents. For the research, we developed a needle tip fabrication technique to observe the aimed prior austenite grain boundary and realized the APT analysis of the prior austenite grain boundary with high success rate. The dependence of Mo content on the amounts of B and Mo segregations in the steels at different cooling rates was investigated. On the basis of the results, we quantitatively discussed the behavior of B and Mo segregations at the boundary, and the influence of B and Mo segregations on hardenability.

2. Experimental

2.1. Materials

Low C steels with various B and Mo contents were used in the study [10]. The chemical compositions of the sample steels are shown in Table 1. Nitrogen (N) content was set to be sufficiently low (<10 massppm) and was scavenged by titanium (Ti) of 0.02 mass%. Thus, solute B is not consumed by the precipitation of boron nitride (BN) [2]. Base is a reference steel without B and Mo. 10B and 05Mo are steels with 10 massppm B and 0.5 mass% Mo, respectively. 05M10B is a steel with 10 massppm B and 0.5 mass% Mo, and 10M10B is a steel with 10 massppm B and 1.0 mass% Mo. The temperatures of M_s in the steels were estimated to be approximately 440 °C.

The steels were vacuum-melted and cast into 50 kg ingots, which were heated at 1250 °C for 1 h in an argon (Ar) atmosphere, hot-rolled into 35 mm-thickness plates, and then air-cooled to room temperature. Cylindrical specimens for the hot-deformation simulator (Thermecmater-Z, Fuji Electronic Industrial Co., Ltd.), 8 mm in diameter and 12 mm in height, were machined from the hot-rolled plates. Fig. 1a shows two heat patterns of the specimens used in the study. Homogenizing treatment of 1200 °C × 600 s was first conducted in the specimens. Subsequently, after the austenization of 950 °C × 20 s, the specimens were cooled down to 650 °C at two cooling rates of 30 °C/s and 5 °C/s, and then quenched by a helium (He)-gas blow. Fig. 1b shows the actual profile of the surface temperature of the specimen (10B) at the cooling rate of 30 °C/s. The cooling rate during the He-gas quenching was approximately 200 °C/s in the region between 650 °C and 400 °C, but the cooling rate went rusty from 400 °C because of the thermal heat release by martensite transformation.

In addition, water-quenched specimens from 950 °C, which is indicated by the dashed arrow in Fig. 1a, were prepared in only the 05M10B steel for investigating the cooling-rate dependence on the amounts of B and Mo segregations at the austenite grain boundary. The actual cooling rate was estimated to be approximately 640 °C/s from the measurement using a thermocouple.

The automatic Jominy end quench test apparatus was used to measure the hardenability of the steels [5]. The Jominy specimen of the bar with a diameter of 25 mm was heated to the austenizating temperature at 950 °C and then cooled automatically following the programs of heat pattern. Hardness profile from the quenched end was measured on two flat grounds, 1 mm deep and parallel to the bar axis, using a Rockwell hardness tester with 1470 N load (HRC). The critical cooling rate V_{c-90}, at which the hardness corresponding to 90% martensite is achieved, was employed for the hardenability index. Ueno et al. reported that the logarithm of V_{c-90} has a linear relation with the solute B content in B-added steels [18,19]. In this study, the values of V_{c-90} were 239 °C/s for the reference steel (Base), 41 °C/s for the steel with 10 massppm B (10B), 107 °C/s for the steel with 0.5 mass % Mo (05B) and 4.2 °C/s for the steel with both B and Mo (05M10B).

Fig. 2 shows the values of V_{c-90} as a function of Mo content in the sample steels with and without 10 massppm B. The values of V_{c-90} decreased with increasing Mo content. As reported by Ueno and Asahi, Mo addition effect and B addition effect were individually observed [4,5]. Furthermore, Mo–B combined addition effect was observed, where the effect was almost the same for Mo contents of 0.5 and 1.0 mass%. The degradation of hardenability indicates that sufficient amounts of ferrite and bainite are generated during the cooling before martensite transformation, since the hardness of ferrite and bainite is smaller than that of martensite.

2.2. Needle tip fabrication

There have been very few studies on the APT analysis of prior austenite grain boundaries because of following two reasons: First

Table 1
Chemical compositions of the sample steels used in the study [10].

(mass%)						(massppm)	
Steel	C	Si	Mn	Ti	Mo	B	N
Base	0.15	0.27	1.31	0.020	–	–	7
10B	0.15	0.27	1.32	0.018	–	10	7
05M	0.14	0.27	1.29	0.020	0.50	–	8
05M10B	0.15	0.28	1.28	0.020	0.50	9	7
10M10B	0.14	0.28	1.28	0.019	1.00	10	7

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