Ferrite Formation Behaviors from B1 Compounds in Steels

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The effect of chemical composition of B1 compounds on the formation of intragranular ferrite during isothermal austenite-to-ferrite transformation was investigated by embedding single crystalline grains of chemically pure B1 compounds in low-alloy steel. Two kinds of B1 compounds having similar misfit against ferrite, TiO and TiN, were adopted in this study. Isothermal transformation experiments were carried out both above and below the bainitic transformation temperature. The orientation relationships between compound and ferrite and between ferrite and prior austenite were characterized by scanning electron microscope equipped with electron backscatter diffraction analyzer.

It was found that the Baker-Nutting (B-N) orientation relationship played a dominant role in ferrite formation from B1 compounds at higher transformation temperature, but some low-index orientation relationships other than the Baker-Nutting orientation relationship were also observed as the transformation temperature decreased, which led to an increase in ferrite formation having the Kurdjumov-Sachs (K-S) orientation relationship with prior austenite. Ferrites from TiO had larger misorientation angles from the exact B-N orientation relationship and thus tended to have the K-S orientation relationship with prior austenite at lower transformation temperatures.

KEY WORDS: intragranular ferrite; acicular ferrite; B1 compounds; Baker-Nutting orientation relationship; Kurdjumov-Sachs orientation relationship.

1. Introduction

As a key concept to improve properties of weld metals and coarse-grained heat affected zone (CG-HAZ) of low alloy steel, intragranular ferrite nucleated from inclusions has attracted much attention, owing to its capability to improve toughness by forming high angle boundaries inside prior austenite grains.^{1,2)} Thus, a number of experiments have been conducted to clarify the chemical nature of inclusions and mechanism that are responsible to the formation of the intragranular ferrite.^{3–6)} Among others, B1(NaCl)-type oxides and nitrides have attracted much attention because they have a crystal orientation relationship with low lattice mismatch with ferrite, the so-called Baker-Nutting (B-N) orientation relationship, *i.e.*, $((001)_\alpha // (001)_{B1}, [110]_\alpha // [100]_{B1})$.^{7–10)} The inclusions found in those studies, however, are usually composed of many phases, which prevents us from identifying particular phases and mechanisms effective to the formation of the intragranular ferrite. In order to overcome the difficulty, a new experimental setup, in which chemically pure compounds are pressure-bonded to steel, has been utilized to create interfaces as nucleation sites for ferrite.

Using the technique, several studies have clarified the noticeable effect of chemistry of B1 compounds on the ferrite formation behavior for different transformation temperatures. For instance, after isothermal experiments at 510°C, Gregg and Bhadeshia reported that ferrites were nucleated only on TiO but not on TiN,⁷⁾ while at higher temperatures, 670–730°C, Zhang *et al.* reported that TiO had inferior fer-

rite nucleation ability to TiN.⁸⁾ In addition to the temperature dependency of potential of B1 compound for promoting ferrite formation, Kasai *et al.* recently clarified from the continuous cooling experiments conducted above the bainite formation temperature the chemistry effect on the orientation relationship between B1 compounds and ferrite nucleated there. It was reported that most of ferrites nucleated on TiN particles hold more strictly B-N orientation relationship with the substrate compound than those nucleated on TiO particles, and the result was explained by electron bonding behaviors at the interface between B1 compounds and ferrite through the application of the first-principle calculation.¹¹⁾

In this study, interest was focused on orientation relationship between B1 compounds and ferrite to understand the effect of chemistry of B1 compounds on the temperature dependence of ferrite transformation behavior from B1 compounds.

2. Experimental Procedure

The composition of the steel used for this study is shown in **Table 1**. The steel was shaped into rods of 8 mm diameter and 5 mm height. Among B1 compounds, TiO and TiN, which have similar lattice parameters, were used to exclude the effect of lattice matching. Lattice parameters of ferrite and B1 compounds used in this study and misfits between the B1 compounds and ferrite were listed in **Table 2**. In fact, TiO has either of B1 (NaCl) structure ($a=4.18\text{\AA}$) or monoclinic structure ($a=5.86\text{\AA}$, $b=9.34\text{\AA}$, $c=4.14\text{\AA}$, $\beta=107.3^\circ$) at the

Table 1. Composition of the steel used in this study.

C	Si	Mn	P	S	B
0.20	0.25	1.41	0.014	0.004	0.009

Table 2. Lattice parameters of ferrite and B1 compounds used in this study and misfits between the B1 compounds and ferrite.

	Lattice constant (Å)	Misfit with ferrite
TiO	4.18	3.0%
TiN	4.24	4.5%
bcc Fe	2.87	–

temperatures below 990°C. However the monoclinic structure has a pseudo-cubic unit cell ($a'=4.20\text{Å}$, $b'=4.14\text{Å}$, $c'=4.14\text{Å}$, $\gamma=89.9^\circ$) which is very similar to B1 structure, having 2.8% misfit with ferrite.¹²⁾ Thus, considering the similarity of these two structures, TiO was assumed to have B1 structure in this study. To gain easy access to the investigation of crystal orientation relationships with ferrite, the B1 compounds used were in the form of single crystalline grains with size distribution in the range of 5–40 μm and 1–15 μm for TiO and TiN, respectively.

The single crystalline grains of TiO and TiN were inserted between two steel rods and the rods were then hot-pressed to each other at 1050°C. The specimens were reheated at 1050°C for austenitization of the steel and held for 15000 s so that the austenite grains grew and the single crystalline grains of B1 compounds were embedded inside the austenite grains. Subsequently, the specimen was cooled down to 500°C, 600°C or 650°C at the cooling rate of 50°C/s and held at those temperatures for 10–200 s. Lastly, the specimen was gas quenched to room temperature at the cooling rate of 50°C/s. Additionally, an experiment to quench the sample just after holding at 1050°C was executed.

The heat-treated specimens were sectioned along the direction normal to the interface between two steel rods. They were etched in 2% nital after mechanically polished using SiC papers and 1 μm , 0.06 μm alumina powder. Scanning electron microscopy (SEM), Energy dispersive X-ray spectroscopy (EDS) and electron backscattering diffraction (EBSD) were employed to analyze the specimens. Ferrites which nucleated from TiO and TiN with size larger than 1 μm were investigated by EBSD and the numbers of ferrites from TiO which were investigated by EBSD were 21, 80 and 66 at 650°C, 600°C and 500°C, respectively, while 14, 50 and 50 ferrites from TiN were investigated at 650°C, 600°C and 500°C, respectively.

3. Results and Discussion

3.1. Ferrite Formation from B1 Compounds

From EDS analysis, any formation of oxide, nitride or carbide was not observed around the B1 compounds. Mn depletion was not identified either. In addition, any apparent change of chemical composition or crystal structure of B1 compounds during heat treatments was evidenced either through EBSD analysis.

Optical micrographs of specimens quenched from 1050°C to room temperature were shown in Fig. 1, which

Fig. 1. Optical microscopic images of specimens quenched after austenitization at 1050°C for 15000 s. No ferrites formed during quenching process were observed. **Fig. 2.** Optical microscopic images of specimens isothermally held (a) at 650°C with TiO (b) at 650°C with TiN (c) at 600°C with TiO (d) at 600°C with TiN (e) at 500°C with TiO (f) at 500°C with TiN.

clearly indicate that ferrite was not formed from B1 compounds during the quenching process.

Both TiO and TiN showed very good ferrite formation capability at temperatures of interest, while the shape of the ferrite from B1 compounds was changed as the transformation temperature decreased. As the result of isothermal holding at 650°C for 200 s, polygonal ferrites were observed to nucleate from the entire surface of TiO and TiN as shown in Figs. 2(a) and 2(b), respectively. It is seen that the formation of ferrites from B1 compounds was faster than that from prior austenite grain boundaries. After isothermal holding at 600°C for 200 s Figs. 2(c) and 2(d), more plate shaped ferrites were formed as compared to the sample prepared at 650°C. After isothermal holding at 500°C for 20 s, it was observed that ferrites were nucleated from almost the entire surface of TiO and TiN as shown in Figs. 2(e) and 2(f), respectively and more acicular ferrites were observed around TiO than TiN.

3.2. Orientation Relationship between B1 Compound and Ferrite

Crystal orientations of ferrites nucleated from B1 compounds were investigated by EBSD to clarify orientation

relationship between ferrite and B1 compounds. In this study, all the pole figures are plotted using the crystal axes of B1 compounds, $\langle 001 \rangle_{B1}$, as the reference frame, RD, TD, and ND, instead of the sample axes, so that we can easily identify the orientation relationship between ferrite and B1 compounds. For instance, if a ferrite and a B1 compound have the B-N orientation relationship with a misorientation angle less than 15 degrees, all of the three $\{001\}_\alpha$ poles of the ferrite must fall into the circles shown in Fig. 3(a). Likewise, low index orientation relationships other than the B-N

orientation relationship can be identified and the typical pole figures of ferrites with low-index orientation relationships, other than the B-N orientation relationship, are shown in Figs. 3(b)–3(d).

Figure 4(a) shows the proportion of B-N orientation relationship described by a number fraction of ferrites having B-N orientation relationship with a misorientation angle less than 15 degrees out of those supposed to nucleate from B1 compounds at 650°C. It is clear that more than 70% of ferrites had the B-N orientation relationship both with TiO and

Fig. 3. (a) EBSD phase map and pole Figure of Fe (001). Circle area inside the pole figure stands for crystal orientation of ferrites having the Baker-Nutting orientation relationship ($\{100\}_{B1} // \{100\}_\alpha$, $\langle 110 \rangle_{B1} // \langle 100 \rangle_\alpha$) with B1 compounds under the condition that each 001 direction of B1 compounds are parallel to RD, TD and ND of this pole figure. (b) EBSD phase map and pole figure of a ferrite having orientation relationship $\{110\}_{B1} // \{110\}_\alpha$, $\langle 100 \rangle_{B1} // \langle 110 \rangle_\alpha$ (c) EBSD phase map and pole figure of a ferrite having orientation relationship $\{110\}_{B1} // \{110\}_\alpha$, $\langle 100 \rangle_{B1} // \langle 100 \rangle_\alpha$ (d) EBSD phase map and pole figure of a ferrite having orientation relationship $\{110\}_{B1} // \{112\}_\alpha$, $\langle 100 \rangle_{B1} // \langle 110 \rangle_\alpha$.

Fig. 4. Proportion of ferrites having some low index orientation relationships (OR) with B1 compounds (a) at 650°C (b) at 600°C (c) at 500°C. The numbers of ferrites investigated by EBSD are indicated inside the Parentheses.

with TiN, which indicates that the B-N orientation relationship played a dominant role in the ferrite formation on those B1 compounds, and apparent difference between TiO and TiN was not observed at this temperature. As the transformation temperature decreased, the proportion of ferrites having the B-N orientation relationship decreased. As shown in Fig. 4(b), 45% and 52% of ferrites had the B-N orientation relationship with TiO and TiN, respectively, and most of the ferrites without the B-N relationship had low-index orientation relationships other than the B-N relationship. As the transformation temperature decreased further, the tendency became more prominent. That is, at 500°C, only 38% and 42% of ferrites had the B-N orientation relationship with TiO and TiN, respectively, and the proportion of other low-index orientation relationships increased as shown in Fig. 4(c). From Figs. 4(b) and 4(c), it is suggested that ferrite from TiN has a little more likely to have the B-N orientation relationship than that from TiO at lower transformation temperatures.

The difference of ferrite formation behavior from TiO and TiN was confirmed clearly by quantitative analysis of misorientation angles from the exact B-N orientation relationship. In Fig. 5, ferrites having the B-N orientation relationship with the B1 compound of interest were classified by the misorientation angle from the exact B-N relationship. At the transformation temperature of 600°C, 72% of ferrites from TiN had misorientation angles less than 5 degrees, while only 26% from TiO. At 500°C, 48% of ferrite from TiN had misorientation angles less than 5 degrees, while

only 15% from TiO. It was demonstrated that ferrite can be nucleated with larger misorientation angle from TiO than from TiN, which is consistent with the result of the continuous cooling experiment conducted by Kasai *et al.*¹¹⁾ It is also noted that there is a tendency that ferrites having larger misorientation angles increases as the transformation temperature decreases.

3.3. Orientation Relationship between Prior Austenite and Ferrite

For the nucleation of ferrite on a B1 compound, increase in interfacial energy caused not only by the ferrite/compound interface but also by the ferrite/austenite should be overcome. Thus, in this study, orientation relationship between ferrite and prior austenite was also investigated. Since Nagano and Enomoto reported that interface between ferrite and austenite has low interfacial energy when it has the Kurdjumov-Sachs (K-S) orientation relationship,¹³⁾ it was checked on the ferrites nucleating from B1 compounds.

It is well known that martensite and bainite have the K-S orientation relationship with the prior austenite, and thus, it is possible to estimate the crystal orientation of the prior austenite grain by EBSD analysis based on the specific pattern of the K-S orientation relationship of martensite and bainite. Figures 6(a) and 6(b) are typical EBSD results showing (100) pole of the ferrite growing from TiO and TiN at 600°C, respectively, as well as the K-S relationship obtained from the surrounding microstructure. Figure 6(a) shows a ferrite that has a low-index orientation relationship with TiO, other than the B-N relationship, and at the same time, has the K-S relationship with the prior austenite. Figure 6(b) shows a ferrite having the B-N relationship with TiN but not having the K-S relationship with the prior austenite. Figures 6(c) and 6(d) are the examples of ferrite formed at 500°C. Figure 6(c) shows a ferrite which has the B-N relationship with TiO and the K-S relationship with the prior austenite, and Fig. 6(d) a ferrite having the B-N relationship with TiN but without the K-S relationship with the prior austenite.

The number of ferrites having the K-S orientation relationship was almost negligible at the transformation temperature of 650°C, but increased as the transformation temperature decreased, the tendency of which was different between TiO and TiN. The proportion of ferrites having the K-S orientation relationship with prior austenite was 55% and 73% at 600°C and 500°C, respectively, in the case of TiO, while it was 50% and 52% respectively at 600°C and 500°C in the case of TiN. Through the investigation in more detail, it was indicated that ferrites nucleated from B1 compounds without the B-N relationship tended to have the K-S relationship with prior austenite. At the transformation temperature of 600°C, among the ferrites having the B-N relationship with TiO, only 28% had the K-S relationship with prior austenite, while 77% had the K-S relationship when the ferrite did not hold the B-N relationship with TiO, as seen in Fig. 7(a). In case of TiN, 27% of ferrites having the B-N relationship had the K-S relationship with prior austenite while 75% had the K-S relationship with prior austenite when the ferrite did not hold the B-N relationship. This result suggests that a low-index orientation relationship other than the B-N was selected to achieve lower total interfa-

Fig. 5. Distribution of misorientation from the exact B-N orientation relationship between ferrite and (a) TiO (b) TiN. Bigger misorientation is allowed between ferrite and TiO than TiN and the misorientation increases as the transformation temperature decreases.

Fig. 6. Typical examples of ferrites according to B1 compounds and the transformation temperatures. (a) a ferrite having a low index orientation other than the B-N orientation relationship with TiO and the K-S orientation relationship with the prior austenite at 600°C (b) a ferrite having the B-N orientation relationship with TiN but not having the K-S orientation relationship with the prior austenite at 600°C (c) a ferrite having the B-N orientation relationship with TiO and the K-S orientation relationship with the prior austenite at the same time at 500°C (d) a ferrite having the B-N orientation relationship with TiN and not having the K-S orientation relationship with the prior austenite at 500°C.

Fig. 7. Proportion of ferrites having the K-S orientation relationship (OR) with the prior austenite in the case that they have the B-N orientation relationship with B1 compounds and in the case that they do not (a), (b) At 600°C, ferrites not having the B-N orientation relationship with B1 compounds tend to have the K-S orientation relationship with the prior austenite at this temperature. (c), (d) At 500°C, differently from at 600°C, ferrites of increased number had the B-N orientation relationship with TiO and the K-S orientation relationship with the prior austenite at the same time. On the other hand, no big difference was observed for TiN compared to at 600°C.

cial energy. At the lower transformation temperature, 500°C, the fraction of ferrite having the B-N orientation relationship with TiO and the K-S relationship with prior austenite is increased, while the change is insignificant for TiN, as seen in Figures 7(c) and 7(d). This is probably because of the fact that larger misorientation angles are

allowed for ferrite to nucleate from TiO at lower transformation temperatures. In fact, as in **Fig. 8** showing the proportion of ferrites having the K-S orientation relationship with the prior austenite among ferrites having the B-N orientation relationship with B1 compounds according to their misorientation from the exact B-N orientation relationship,

Fig. 8. Proportion of ferrites having the K-S orientation relationship (OR) with the prior austenite among ferrites having the B-N orientation relationship with B1 compounds according to their misorientation from the exact B-N orientation relationship. Ferrites of relatively big misorientation from the exact B-N orientation relationship tended to have the K-S orientation relationship with the prior austenite.

ferrites nucleating from B1 compounds with larger misorientation from the exact B-N orientation relationship achieved the K-S relationship with prior austenite.

4. Conclusions

From the isothermal austenite-to-ferrite transformation

experiment conducted by embedding single crystalline grains of chemically pure B1 compounds in low-alloy steel, the following conclusions can be drawn:

(1) The Baker-Nutting orientation relationship plays a dominant role in ferrite formation from B1 compounds at 650°C. The dependence of the Baker-Nutting orientation relationship decreases as the transformation temperature decreases. Instead, the Kurdjumov-Sachs orientation relationship is more dominant than the Baker-Nutting orientation relationship.

(2) Ferrite nucleation from B1 compounds with low-index orientation relationships other than the Baker-Nutting orientation relationship is also increased with decreasing transformation temperature. This leads to an increase in ferrite formation having the Kurdjumov-Sachs orientation relationship with prior austenite.

(3) Ferrites from TiO can have larger misorientation angles from the exact Baker-Nutting orientation relationship and thus tend to have the Kurdjumov-Sachs orientation relationship with prior austenite at lower transformation temperatures.

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