Investigation of the metatectic reaction in iron-boron binary alloys

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ABSTRACT

The use of advanced high strength steels, such as boron containing steels, in the automotive industry is enabling lighter weight vehicles leading to increased fuel economies. However, boron containing steels are difficult to commercially produce. Thermodynamic studies of the Fe-B system predict a metatectic reaction, $\delta \rightarrow L + \gamma$, to occur on the iron-rich side of the diagram. This would result in solid material remelting as the temperature is decreased, which could be the source of the casting defects. To experimentally investigate the predicted metatectic reaction, levitation zone melting is used to control the boron segregation in two simple Fe-B binary alloys, and confocal scanning laser microscopy allows for in-situ observation of local microstructural changes as temperature is varied. Both experimental methods show evidence of a metatectic reaction providing a good comparison to predicted phase diagrams. Based on the experimental results, the metatectic reaction occurs over a broader range of compositions than predicted with $C_S = 0.0025$ wt% B, $C_L = 2.0$ wt% B, and C_R ranging from approximately 0.03 to 0.06 wt% B.

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1. Introduction

There is a current push in the automotive industry to increase the fuel economy of passenger vehicles. One way to achieve this increase is through the use of advanced high strength steels (AHSS). AHSS with yield strengths greater than 550 MPa allows for thinner gauge components to be implemented in vehicles without sacrificing safety, thereby lowering the weight of the vehicle and improving fuel economy [1]. Included under the umbrella of AHSS are boron-based hot formed (HF) steels. Typically HF steels contain between 0.002 and 0.005 wt% B [2]. Even in these small amounts, the addition of boron makes the steel difficult to commercially produce, specifically via continuous casting. Defects in the continuously cast boron containing steels include surface cracks, internal half way cracks, folds, pockets, ridges, and, in more severe cases, breakouts [3–5].

While the motivation behind the use of AHSS is clear, the causes and thus solutions to the casting defects caused by boron are presently unclear. In some thermodynamic studies of the iron-boron system, a $\delta + L \rightarrow \gamma$ peritectic reaction, such as that shown in Fig. 1a, is predicted [6]. However, in other investigations, a $\delta \rightarrow L + \gamma$ metatectic reaction is predicted [3,7]. This metatectic reaction results in a completely solid material partially remelting with a decrease in temperature, Fig. 1b. Four predicted Fe-B phase diagrams found in literature are detailed in Table 1 including the reaction temperature, T_R, the reaction composition, C_R, the solid composition at T_R, C_S, and the liquid composition at T_R, C_L. The compositions and temperatures match to the locations labeled in Fig. 1. Three of the sources predict a metatectic reaction while one predicts a peritectic reaction [6–10]. The three predicted metatectic reactions differ on the compositions that undergo the reaction, specifically the minimum amount of boron that will cause remelting through the metatectic reaction. A recent diagram produced from the TCFE8 thermodynamic database (Thermo-Calc: Steels/Fe alloys database) predicts that a boron composition as low as 0.0025 wt% will remelt through the metatectic reaction [8,10]. Two older diagrams from Kubaschewski and the TCFE5 thermodynamic database predict the minimum amount of boron that will remelt at an order of magnitude larger than TCFE8, 0.039 wt% and 0.022 wt% respectively [7–9]. Also, the diagram from the TCFE5 database predicts post-metatectic remelting, $\gamma \rightarrow L + \gamma$, due to a retrograde solidus between compositions of 0.015 and 0.022 wt% B, unlike any of the other predicted diagrams [8,9]. It is evident that metatectic reaction and the corresponding solidification behavior is not well characterized in the Fe-B system.

This study utilizes levitation zone melting to experimentally investigate the low temperature remelting through the controlled segregation of boron. An early description of this process is given by B. F. Oliver [11]. The large range of compositions and microstructures contained in the zone melted ingot are characterized with
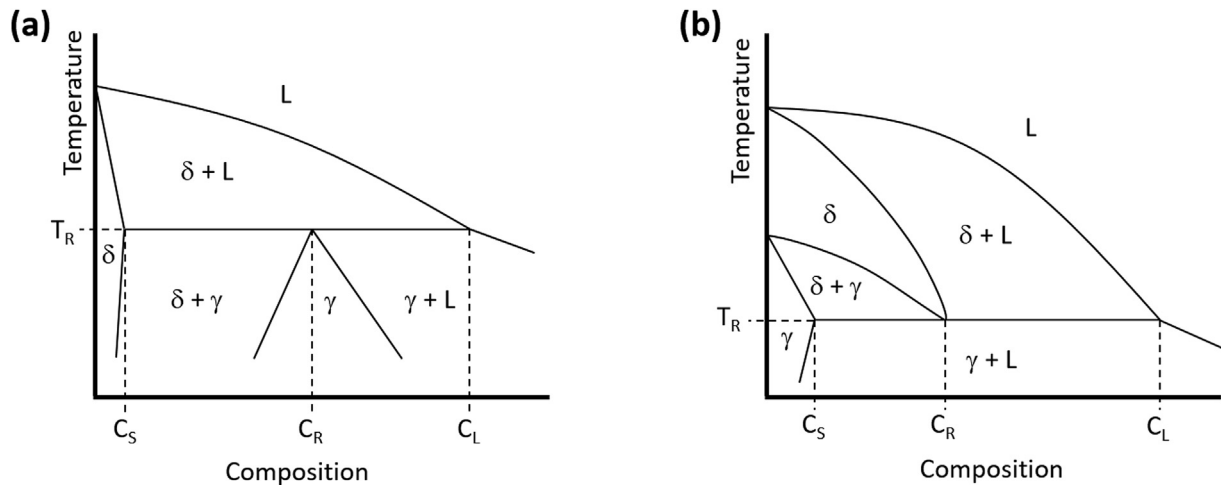
Fig. 1. Partial binary phase diagrams showing a typical (a) peritectic and (b) metatectic reaction occurring at composition C_R and temperature T_R.

Table 1

Comparison of the reaction temperature, T_R, the solid composition, C_S, the reaction composition, C_R, and the liquid composition, C_L, of four predicted Fe-B phase diagrams.

Reference	T_R (°C)	C_S (wt% B)	C_R (wt% B)	C_L (wt% B)	Reaction type
Rompaey et al. [6]	1396	0.00086	0.0020	1.7	Peritectic
Kubaschewski [7]	1381	0.039	0.097	1.5	Metatectic
Thermo-Calc TCFE5 database [9]	1360	0.022	0.045	2.1	Metatectic
Thermo-Calc TCFE8 database [10]	1384	0.0025	0.0097	2.1	Metatectic

optical microscopy and confocal scanning laser microscopy (CSLM). These experimental results are then compared to the predicted phase diagrams to better understand the remelting reactions.

2. Experimental technique

Two iron-boron alloys were analyzed for this study. A low boron alloy containing nominally 0.0168 wt% B was a 45 kg ingot cast in a vacuum induction furnace at the East Chicago (USA) Center of ArcelorMittal Global R&D. A high boron alloy containing nominally 0.045 wt% B was a 1 kg ingot cast in a small tilt-pour vacuum induction melter at Purdue University. After achieving vacuum, the furnace was backfilled with argon. The sample was melted in a zircona crucible and cast into a chilled permeant Cu mold to produce 25 mm diameter samples for zone melting. In both alloys, electrolytic iron and vacuum induction melting was utilized to limit any impurities in the cast material.

Each alloy was levitation zone melted to create a large range of compositions and microstructures within each ingot. Levitation zone melting is a containerless processing method as illustrated in Fig. 2a. This process is similar to vertical float zone melting, however, at a ~25 mm diameter, surface tension is insufficient to shape and constrain the liquid zone between the two solid bars. The liquid zone would tend to roll over onto the lower solid bar resulting in a possible spill if only surface tension was relied upon [12]. In this process, an induction coil and eddy current plate assembly is used to melt, levitate, and shape the liquid zone to a height similar to that of the solid bar diameter, ~20 mm in this study. A melting solid/liquid interface is present at the top of the hot zone while a freezing solid/liquid interface is located at the bottom. The shape of the liquid zone and dimensions of the freezing solid/liquid interface are controlled by moving the top solid portion relative to the bottom. To maintain a smooth solid/liquid interface, the bottom solid bar is rotated [13]. This ingot rotation, along with induction stirring, contributes to a well-mixed liquid zone via turbulent flow [14]. In

this study, the zone melted ingot is moved through the assembly at a rate of ~12 mm/h.

The levitation zone melting process allows for the controlled segregation of solute elements with a large portion of the solute being swept towards the end of the bar. Depending on the partition coefficient, k, of the particular solute element, a single pass in the levitation zone melter will result in a composition profile such as those shown in Fig. 2b [15]. This composition profile can be divided into two distinct zones, the directionally solidified (DS) zone and the last zone to solidify. As the partition coefficient approaches unity, such as $k = 0.8$ in Fig. 2b, the DS zone can be further broken down into a transient zone, where initial solidification occurs, followed by a steady state zone where steady state planar solidification occurs at the original alloy composition, C_0. When the partition coefficient is smaller, such as $k = 0.4$ and $k = 0.1$ in Fig. 2b, the composition of the whole DS zone is less than the original alloy composition, C_0, with the solute balance residing in the last zone.

Predicted iron-boron phase diagrams show $k < 0.1$, so an expected composition profile from a single pass through the levitation zone melter is expected to look similar to the curve for $k = 0.1$ in Fig. 2b. The compositional gradient and microstructure from the zone melted ingot can also be used to investigate the differences in the predicted phase diagrams, Table 1. For example, an iron-boron alloy can be zone melted for a certain length to reach a specific target liquid composition for the last zone. The induction power can then be reduced, and, with an increased cooling rate, the solidification growth front will transition from planar to dendritic growth. Analysis of the microstructure, composition, morphology, and degree of microsegregation provides experimental evidence that can be compared to predicted phase diagrams.

While the last zone contains higher solute concentration compared to the DS zone it also solidifies faster, as mentioned previously. The cooling rate of the last zone for both alloys, as measured by a high temperature infrared pyrometer, was found to be ~54 °C/min. Similarly, the cooling rate of the DS zone was