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# Phase identification in boron-containing powder metallurgy steel using EBSD in combination with EPMA



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#### article info abstract

Article history: Received 3 August 2015 Received in revised form 15 January 2016 Accepted 18 January 2016 Available online 19 January 2016

Keywords: EBSD EPMA Boride Powder metallurgy Steel Liquid phase sintering

Boron (B) is extensively used to induce liquid phase sintering (LPS) in powder metallurgy (PM) steels and thereby increase the densification. The alloying elements in B-containing PM steels affect the boride phase, stability of the boride, the temperature of liquid formation, and the progress of LPS. However, the boride phase has not been systematically identified yet. The main objective of this study was to clarify the influences of alloying elements, including C, Cr, and Ni, on the boride phases using electron backscatter diffraction (EBSD) in combination with electron probe microanalysis (EPMA). Network structures consisting of ferrite, Fe<sub>2</sub>B boride, and Fe<sub>3</sub>C carbide were extensively observed in the Fe–0.4B–0.5C steel. The portions of Fe<sub>2</sub>B were sufficiently larger than those of Fe<sub>3</sub>C, and Fe<sub>3</sub>C was mostly distributed at the interfaces between ferrite and Fe<sub>2</sub>B. Adding 1.5 wt.% Cr or 1.8 wt.% Ni to Fe–0.4B–0.5C steel completely changes the Fe<sub>2</sub>B and Fe<sub>3</sub>C phases to a M<sub>3</sub>(B,C) phase, where M represents the metallic elements, including Fe, Cr, Mo, and Ni. Furthermore, Cr, Mo, B, and C atoms tend to concentrate on the  $M_3(B,C)$  phase, but Ni atoms do not.

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### 1. Introduction

PM steels are extensively used as structural parts in the automobile and mechanical fields due to their high productivity and moderate mechanical properties. Unfortunately, the mechanical properties of PM steels are never equal to those of wrought steels, due mainly to their porosity. LPS is a versatile method of densifying PM steels or various porous materials to nearly fully dense without considerably increasing the production cost [1–[7\].](#page--1-0) Among the various alloying elements in PM steels, B is the most effective element for facilitating LPS because B induces a eutectic reaction (Fe + Fe<sub>2</sub>B  $\rightarrow$  L) at ~1175 °C [\[1](#page--1-0)–3]. Therefore, the influences of B additions on the LPS of alloy steels, stainless steels, and other high-alloyed steels have been widely investigated [1–3,8–[16\].](#page--1-0)

Furthermore, the various alloying elements in B-containing PM steels sufficiently affect the densification and progress of LPS. In Fe–B– Mo steels, increasing the Mo concentration from 0 to 1.5 wt.% can improve the sintered density after LPS [\[9,11,16\]](#page--1-0). However, the temperature of the eutectic reaction is increased, which postpones LPS, when the Mo content is higher than 1.5 wt.%, as reported by Sarasola et al. [\[1\].](#page--1-0) In Fe–B–Cr steels, the LPS densification of Fe–0.4B–3Cr steel is slightly superior to that of Fe–0.4B–1.5Cr after sintering at 1250 °C, though

increasing the Cr content from 1.5 wt.% to 3 wt.% raises the temperature of liquid formation from 1190 °C to 1200 °C [\[16\]](#page--1-0). Moreover, Lozada and Castro [\[14\]](#page--1-0) studied the influences of Cr concentrations (11.8, 16.9, and 30 wt.% Cr) on the LPS of B-containing PM stainless steels and indicated that LPS is suppressed by increasing the Cr concentrations from 11.8 wt.% to 30 wt.%. At the high end of that range, the liquid phase cannot be generated even at 1375 °C. Thus, adding Mo or Cr to B-containing PM steels has been found to increase the temperature of liquid formation. In a recent study, thermodynamic simulation was used to predict the influences of Mo and Cr on the liquid formation of Fe–0.4B steel. The results showed that Mo and Cr atoms dissolved in austenite could stabilize the  $M<sub>2</sub>B$  boride to a higher temperature and delay the formation of eutectic liquid [\[16\].](#page--1-0)

Carbon is an indispensable element in PM alloy steels, and thus its role in the LPS of B-containing PM steels has also been studied [\[2,3,12,](#page--1-0) [13,16\].](#page--1-0) Liu et al. [\[12\]](#page--1-0) found that the sintered density of Fe–0.3B–1Ni– 1Mo–xC steel is not apparently affected by carbon contents of up to 0.4 wt.% C. In contrast, Xiu et al. [\[13\]](#page--1-0) found that increasing the carbon content from 0.2 wt.% to 0.44 wt.% facilitates the LPS densification of Fe–B–Mo–C steel. Wu et al. [\[3,16\]](#page--1-0) indicated that adding 0.5 wt.% C to Fe–B–Mo and Fe–B–Cr steels can obviously reduce the temperature of liquid formation and promote the generation of a secondary liquid phase, thereby enhancing the LPS densification. The temperature of liquid formation decreases when carbon is added because the carbon

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Fig. 1. The microstructure of FBC steel sintered at 1250 °C. (a) SE image, (b) BSE image.

atoms dissolved in the austenite lower the temperature of eutectic reaction [16]. In addition, Sercombe [\[17\]](#page--1-0) found that the presence of carbon reduces the LPS temperature of B-containing maraging steel (Fe– 17.6Ni–12.6Co–4Mo–1.8Ti–0.1Al–0.4B) by ~60 °C.

Several studies have indicated that  $Fe<sub>2</sub>B$  or  $M<sub>2</sub>B$ , where M represents the metallic elements, including Fe, Cr, Mo, and Ni, is the typical phase of boride in the various B-containing PM steels [\[1,10,14,15,17\]](#page--1-0). However, little analysis or direct evidence of the boride phases has been reported, not to mention detailed methods for phase identification. Röttger et al. [\[18\]](#page--1-0) used X-ray diffraction (XRD) and energy dispersive X-ray (EDX) analyses to identify the compounds in three ultrahigh-boron (1.73– 3.54 wt.% B) high-carbon (0.56  $\sim$  2.12 wt.% C) steels and found that several phases, including  $M_2B$  boride,  $M_3B_2$  boride,  $M_3(B,C)$  borocarbide,  $M_{23}(B,C)_6$  borocarbide, and  $M_7C_3$  carbide, could be found in these steels. The above findings clearly suggest the complexity of boride structures in PM steels.

In this study, to systematically clarify the phases of borides in PM alloy steels, the borides in Fe–0.4B–0.5C, Fe–1.5Cr–0.2Mo–0.4B–0.5C, and Fe–1.8Ni–0.5Mo–0.4B–0.5C steels were identified using EBSD and EPMA. The first objective of this study was to analyze the effects of alloying elements, including C, Cr, and Ni, on the boride phases of Bcontaining PM steels. Other than the boride phase, the alloying elements could also affect the chemical compositions of the borides, and thus alter the stability of the borides and the temperature of liquid formation, because the segregation tendencies of different alloying elements in the boride could be divergent. The second purpose was thus to study the distributions of the various alloying elements in Bcontaining PM steels and to interpret the segregation behavior of these alloying elements.

#### 2. Material and methods

To investigate the influences of C, Cr, and Ni on the characteristics of borides, commercially available pure Fe (ASC 100.29, Höganäs AB, Höganäs, Sweden), Fe–1.5Cr–0.2Mo (CrL, Höganäs AB, Höganäs, Sweden), and Fe–1.8Ni–0.5Mo (ATOMET 4601, QMP, Suzhou, PRC) powders were used as the base powders in this study. The three base powders were blended with 0.4 wt.% elemental boron (ELECMAT, CA, USA) and 0.6 wt.% graphite (1651, Asbury Carbons, LA, USA) powders in a mixer for one hour to produce Fe–0.4B–0.6C, Fe–1.5Cr–0.2Mo– 0.4B–0.6C, and Fe–1.8Ni–0.5Mo–0.4B–0.6C powder mixtures. The average sizes of the boron and graphite powders were 2 μm and 9 μm, respectively, as examined by SEM (JSM-6360, JEOL, Tokyo, Japan). Furthermore, 0.75 wt.% lubricant (ethylene bis-stearamide) was also added into the previous powder mixtures to improve the compressibility of the powder and reduce wear on the mold.

These three powder mixtures were uniaxially compacted into green disks with a diameter of 13 mm and a thickness of 7 mm at a pressure of 600 MPa. The green densities of Fe–0.4B–0.6C, Fe–1.5Cr–0.2Mo–0.4B– 0.6C, and Fe-1.8Ni-0.5Mo-0.4B-0.6C steels were  $6.92 \text{ g/cm}^3$ , 6.84 g/cm<sup>3</sup>, and 6.84 g/cm<sup>3</sup>, respectively. The prealloyed 1.5Cr or 1.8Ni addition in the steel powder decreased the powder compressibility



Fig. 2. The microstructure of FBC-1.5Cr steel sintered at 1250 °C. (a) SE image, (b) BSE image.

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