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Production of ferroboron powders by solid boronizing method

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ABSTRACT

Ferroboron is an iron-boron alloy containing 10-20% of boron by weight. Commercial ferroboron production is made by two main processes: carbothermic reaction and aluminothermic reaction. Ferroboron also occurs in steel surfaces due to boronizing, which is applied to increase surface hardness in steel. Boronizing is a thermo-chemical surface hardening treatment. The ferroboron phases like Fe₂B, FeB form by diffusing of boron element into iron. These phases are very hard, wear strengths are high, and friction coefficients are low.

In this study, ferroboron powder was obtained by boronizing ASC 100.29 iron powder that was used widely in powder metallurgy area. Solid boronizing method was preferred due to its advantages in applications and Ekabor-HM powder was used as the boronizing agent. The 80% ASC 100.29 and 20% Ekabor HM were mixed homogeneously and subjected to boronizing at 850–950 °C for 1–6 h. Formation and development of ferroboron phase on the samples was determined by metallographic studies depending on various treatment conditions. The X-ray diffraction analysis revealed that the Fe₂B phase did form but FeB phase did not. Micro hardness distributions were measured on the powder grains. Eighteen GPa hardness was measured at Fe₂B phase obtained by boronizing while hardness of non-boronized iron powders was 1.06 GPa. The thickness of ferroboron layer formed by boronizing changed with boronizing conditions. The thickness of ferroboron layer increased with boronizing temperature or boronizing time. Depending upon processing parameters, ferroboron layers was formed partially or throughout ferrous powder structure. Since boronizing can be applied to iron powders having any size or shape, ferroboron production with required shape and size is possible.

Finally, a new method, namely solid boronizing method, was developed in ferroboron powder production.

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1. Introduction

Ferroboron is a binary alloy of iron with boron content between 10% and 20% and is the lowest cost boron additive for steel and other ferrous metals. Limited amount of elemental boron is widely used to increase hardness in steel. Boron is also utilized in aluminum castings to refine the grain; in copper-base alloys and high-conductance copper as a degasifier; in the nonferrous metals industry as a deoxidizer. It has been also used as a hot spray coating powder [1,2]. After realizing superior magnetic properties of ferroboron alloys and with the rapid development of electric industry, the demand for ferroboron in permanent magnetic materials has significantly increased [2,3].

Ferroboron is commercially produced by one of the two main processes, namely carbothermic reaction and aluminothermic reaction. Carbothermic production is carried out in electrical arc furnaces using carbon, boric acid and iron powder as raw materials. Aluminothermic production is conducted in lades. The raw materials fed into the ladle are boric acid, iron ore, aluminum powder, but magnesium powder can also be used [2–4].

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Impurities at different concentrations occur in all ferroboron production methods which restricts the usage of the alloy in high – technology materials [5,6]. Ferroboron powders are produced by milling. Milled powders have irregular shapes and poor flowability [7].

Ferroboron also occurs in steel surfaces due to boronizing, which is applied to increase surface hardness in steel. Boronizing, a thermochemical process, is widely used for boride-type ceramic coatings to achieve high hardness and wear resistance on various ferrous alloys [8,9]. It is a diffusion process whereby a single phase Fe₂B or a double intermetallic phase (Fe₂B, FeB) is obtained by diffusing boron atoms into the surfaces of metallic materials. The lower boron containing Fe₂B phase is especially desirable for industrial applications [10].

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There are a number of methods for carrying out boronizing. The boron source may be in solid, liquid or gaseous state. However, boronizing in the solid state has technical advantages, including ease of treatment, ability to achieve a smooth surface, simplicity of the required equipment and the possibility of changing the composition of powder [11,12]. Boronizing is carried out generally under 800 °C–1000 °C and for 2–8 h [12].

Boronized materials are characterized by their high surface hardness and wear resistances [13]. The hardness of the boride layers are in the range of 1400–2100 HV[14], and their thickness may be up to 380 μ m, depending on the process time and temperature and on alloying elements in the substrate [14,15]. FeB phase is harder and more brittle than the Fe₂B phase [16]. Boronized layers have good tribological properties. Very low coefficient of friction and high hardness are obtained on boronized surfaces. Since boron is reactive to oxygen, borides have thin oxide films on their surfaces, which lower the coefficient of friction [17]. Although atmospheric corrosion resistance of the ferroboron is not very good, ferroboron have high corrosion resistance to acids such as HCl, H₂SO₄, H₃PO₄ and to liquid metals such as Al, Pb, Zn [18].

Studies on boronizing rather concentrated on characterization of boride layers on machine parts and improving their properties. In this study, production of ferroboron powders having properties of the boride layers was aimed. Since boronizing treatment can be applied to iron powders having different dimensions and shapes, the flowability problems observed in powders made by milling will not be occurred. Thus, ferroboron powders having low impurity can be achieved.

2. Materials and method

In this study, ASC100.29 iron powder produced by Hoganas (Sweden) was used for boronizing. The ASC100.29 is an atomized iron powder with high purity (carbon $\leq 0.01\%$ and hydrogen loss $\leq 0.15\%$) and compressibility [19]. Its powder particle size distribution was measured by using a laser particle sizer (Malvern MasterSizer) and is given in Fig. 1. Mean size (d_{50}) was 77.45 µm and all powders passed through 65 mesh sieve (208 µm). Fig. 2 shows powder particle size distribution of Ekabor-HM (Bortec, Germany) boronizing agent. The Ekabor-HM commercial powder smaller than 150 µm was used as boronizing agent. The Ekabor-HM contained B₄C as boron source and SiC as deoxidant. The Boronizing mixture was prepared by mixing 80% iron powder with

20% Ekabor-HM powder by weight. Then the mixture was filled into a steel box whose lid was tightly sealed. The steel box was placed in a muffle furnace and kept at temperatures of 850, 900 and 950 °C for 1–6 h. After the boronizing treatment, the mixture was removed from the box and ferroboron powders were removed from the mixture by magnetic separation.

Metallographic samples were prepared from the boronized iron powders by cold mounting them in polyester resin. Polished or polished and etched samples were examined under light microscope (Nikon Eclipse LV100D, Japan). Micro hardness measurements were made across the polished particles by using Vickers diamond indenter with 0.1 N load (Future-Tech FM700, Japan). Phase characterization of the structures formed on boronized layers were identified by X-ray diffraction (XRD) with a Cu-K α (1.5406 Å) radiation source using Rigaku RTP 300 Model diffract meter.

3. Results and discussion

By boronizing, ferroboron phases having different thicknesses were obtained. Boride layer thickness was measured from the microstructure and average of three measurements was recorded. Fig. 3 gives the dependence of boride layer thickness on the treatment time and temperature successively. As it is seen from the figure, the boride thickness increases parabolically with the treatment time and almost linearly with the treatment temperature. The 6 h treatment produced the following boride thickness: 29 μ m, 45 μ m and 59 μ m at 850 °C, 900 °C and 950 °C successively.

The boronizing process obeys a parabolic reaction. Therefore, Fick's second law is reduced to a simple form as given below for the case where a specific concentration of the diffusing element is desired in the diffused metal or alloy.

$$\frac{x}{2\sqrt{Dt}} = c_1$$

where c_1 is a constant. Thus, $x = 2c_1\sqrt{Dt}$ which shows that diffusion distance is proportional to the square root of time which is a parabolic dependence.

Parabolic dependence of borided layer to boronizing time is in agreement with the other studies of surface hardening [9,11,14,18]. However, boride layer thickness values obtained in this study are less than the literature values at the same conditions. Maximum layer thickness obtained in this study was 59 μ m for boronizing 6 h at 950 °C. The mean layer thickness was about



Fig. 1. Powder particle size distribution of ASC100.29 iron powder.

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