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# Hard iron boride (Fe<sub>2</sub>B) on 99.97 wt% pure iron

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#### Abstract

Some properties such as hardness and fracture toughness of boride formed on the 99.97 wt% pure iron were investigated. Boronizing was carried out in a solid medium, consisting of Ekabor powders of 5% B<sub>4</sub>C as donor, 5% KBF<sub>4</sub> as an activator and 90% SiC as diluent at 800 °C for 2, 4 and 8 h. The dominant phase formed on the substrate was found to be Fe<sub>2</sub>B that had a finger-like shape morphology. The hardness of boride on the 99.97% pure iron was over 1700HVN, while the hardness of pure iron was about 130HVN. It was found that the fracture toughness of boride formed on surfaces of 99.97% pure iron, depending on the process time, ranged from 3.59 to 3.83 MPa m<sup>1/2</sup>. Depending on process time and temperature, the depth of the boride layer ranges from 22 to 43 µm, leading to a diffusion-controlled process.

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Keywords: Boronizing; Borides; Pure iron; Hardness; Fracture toughness

### 1. Introduction

Thermochemical boriding of iron alloys allows both single Fe<sub>2</sub>B and FeB-based polyphases coatings to be obtained and then used mainly to improve surface hardness and wear resistance of the components for tribological applications [1]. Boronizing being a thermochemical diffusion process has been applied to a wide range of materials including ferrous materials, non-ferrous materials and some super alloys. Boronizing of a steel surface allows to reduce essentially a velocity of corrosion, wear and shapes of fatigue cracks occurring in an outcome of its operation. Thermal diffusion treatments of boron compounds used to form iron borides typically require process temperatures between 700 and 1000 °C. The process can be carried out in solid, liquid or gaseous medium. The most frequently used method is a pack boriding which is a process similar to pack carburizing process [2,3]. The diffusion of boron into the surface of selected metal alloys creates a fully dense reaction zone of metal borides. This effectively generates superior surface properties of materi-

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als [4]. The diffusion of B into the steel results in formation of iron borides (FeB and Fe<sub>2</sub>B), and the thickness of the boride layer is determined by the temperature and time of the treatment. Usually, depending on process temperature, chemical composition of substrate materials, boron potential of medium and boriding time, single-phase Fe<sub>2</sub>B or two intermetallic phases of FeB and Fe<sub>2</sub>B are obtained by diffusing boron atoms into the surface of metallic materials [5]. Generally, the formation of a monophase ( $Fe_2B$ ) with saw-tooth morphology is more desirable than a doublephase layer with FeB and Fe<sub>2</sub>B for industrial applications. The boron-rich FeB phase containing approximately 16.23 wt% B is not desirable because FeB is more brittle than the iron subboride and Fe<sub>2</sub>B phase which is containing 8.8 wt% B. Furthermore, since FeB and Fe<sub>2</sub>B phases exhibit substantially different coefficients of thermal expansions, CTE,  $(\alpha_{\text{FeB}} = 23 \times 10^{-6} / ^{\circ}\text{C}, \alpha_{\text{Fe}_2\text{B}} = 7.85 \times 10^{-6} / ^{\circ}\text{C}),$ crack formation is often observed at the interface between FeB and Fe<sub>2</sub>B. These cracks often lead to flaking of the coated layer when mechanical load is applied. Through the control of boronizing process parameters, i.e. boronizing powder composition, temperature time and laser heat treatment after boriding, Fe<sub>2</sub>B phase can be consistently achieved during pack boriding [6]. A single Fe<sub>2</sub>B layer

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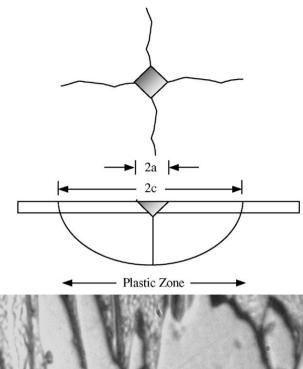
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produces superior wear resistance and mechanical properties. The main interest has been focused on two peculiar characteristics of the boride coatings. They are as follows: (i) high hardness that is expected to give a high wear resistance; and (ii) columnar morphology that is required for a good adhesion between coating and substrate [7]. Borides are non-oxide ceramics and are often brittle [8]. Boronized steel consistently outperforms nitrided and carburized steels essentially, because the iron boride formed on the steel exhibits substantially higher hardness (HVN = 1600-2000) than the carburized or nitrided steels (HVN = 650-900). In particular, boronized steel exhibits excellent resistance to a variety of tribological wear mechanisms. In addition, the resistance of boronized steel to attack by non-oxidizing dilute acids, alkalis and molten metals is also outstanding. In general, the commercial boronizing mixture contains B<sub>4</sub>C as donor, KBF<sub>4</sub> as an activator and SiC as diluent which control the boronizing potential of the medium [9]. Some pure refractory materials such as Nb, W have been borided by using that mixture [10-12].

At the present study, we attempt to elucidate some mechanical properties of boronized 99.97 wt% pure iron, e.g., morphology of boride layer, fracture toughness, etc. A Vickers indenter and an optical microscope were utilized to determine hardness and microstructures of boronized 99.97 wt% pure iron.

# 2. Experimental procedure

The substrate used for this study was 99.97 wt% pure iron. The purity of the iron was obtained by Perkin Elmer Analyst 300 atomic adsorption spectrophotometer. The rectangular test pieces dimensions are 10 mm in length and 5 mm in width. Before boronizing heat treatment, all the samples were polished using fine polishing paper to obtain a good surface finish. The Vickers hardness of the untreated iron is 130HvN. Boronizing heat treatment was carried out by using pack boriding method that is similar to pack carburizing process. Boronizing mixture contains of 5% B<sub>4</sub>C, 5% KBF<sub>4</sub> and 90% SiC which control the boronizing potential of the medium. All samples to be boronized were packed in the powder mix and sealed in a stainless steel container. Boronizing experiment was performed in an electrical resistance furnace under atmospheric environment at 800 °C for 2, 4 and 8 h and the boronized steel samples were cooled in air. The presence of borides formed on the surface of 99.97% iron was confirmed by X-ray diffraction (XRD) analysis. Rigaku X-ray diffractometer (DMAX 2200) with a Cu Ka radiation source of a wavelength of 1.541 Å over a  $2\theta$ range from  $40^{\circ}$  to  $90^{\circ}$  was employed for the phase characterization of borided layer of the samples. The microhardness and fracture toughness of borides were measured using a Vickers microhardness tester. The equation used for calculating fracture toughness was  $K_{\rm c} = XP/c^{3/2}$ , where X is the residual-indentation coeffi-



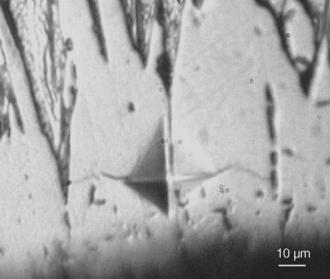


Fig. 1. Schematic representation of a Vickers indent in a coated material.

cient [8,13] which depends on hardness-to-modulus ratio (E/H) of the boride layer. The constant X is  $0.028(E/H)^{1/2}$  where E and H are the Young's modulus and hardness of boride layer, respectively. P is the applied load and the value of E is approximately 34300 kg/mm<sup>2</sup> [13] for fracture toughness calculations and c is the indentation half crack length as shown in Fig. 1. The thickness of boride layer was determined by a digital measurement instrument (Olympus OSM-DC) attached to an Olympus BHM-313 optical microscope.

## 3. Results

#### 3.1. Microstructure

In general, surface boronizing treatment can form single  $Fe_2B$  phase layer on the surface of plain carbon substrate.

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