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SHORT COMMUNICATION

The Hardness of Boride Layer on the S45C Iron (A preliminary study on surface hardening of ferrous material)

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Abstract - The property such as microhardness of boride layer formed on S45C iron was investigated. Boronizing was carried out in a solid medium consisting of nano size powders of 50% B_4C as a donor, 45% SiC as a diluent, and 5% KBF_4 as an activator treated at the temperature of 1000^0C for 8 hours. The phases that were formed on the substrate was found as Fe_2B and FeB layer that had smooth and flate shape morphology. The hardness of boride layer on S45C was over 2000 HV, while the hardness of untreated S45C iron was about 196,39 HV. Depending on process time and temperature, the depth of boride layer ranges from 25 to 55 μm, leading to a diffusion controlled process.

Keywords : Boronization, hard layer, heat treatment, $B_4C.KBF_4$.SiC powder, low carbon steel.

Thermochemical boriding of iron alloys produces both single Fe_2B and FeB-based multyphases to be obtained and then used mainly to improve surface hardness and wear resistance of the components for tribological applications. 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 (Chalik *et al*., 2009). Boronizing of a steel surface can also to reduce the velocity of corrosion and to improve the surface hardness. Thermal diffusion treatments of boron compounds used to form iron borides typically require process temperatures between 700^0 and 1000^0C. The process can be carried out in solid, liquid or gaseous medium. The most frequently used method is a pack boriding which is similar to pack carburizing process (Bindal and Ucisik, 2008).

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 materials. The diffusion of boron into the steel results in formation of iron borides (FeB and Fe_2B), 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_2B or two intermetallic phases of FeB and Fe_2B are obtained by diffusing boron atoms into the surface of metallic materials (Martini and Palombarin, 2004; Ozdemira *et al*., 2006).

Generally, the formation of a singlephase (Fe_2B) with saw-tooth morphology is more desirable than a doublephase layer with FeB and Fe_2B for industrial applications. Through the control of boronizing process parameters, i.e. boronizing powder composition, temperature time and laser heat treatment after boriding, Fe_2B phase can be consistently achieved during pack boriding. A single Fe_2B layer produces superior wear resistance and mechanical properties (Matsuda *et al*., 1984).

The main interest has been focused on two characteristics of the boride layer. 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. Borides are non-oxide ceramics and are often brittle. Boronized steel consistently exhibits substantially higher hardness (HVN 1600–2000) than the carburized or nitrided steels (HVN 650–900)[5].

In particular, boronized steel exhibits excellent resistance to a variety of tribological wear mechanisms. In general, the commercial boronizing mixture contains B_4C as donor, KBF_4 as an activator and SiC as diluent which control the boronizing potential of the. In this research found two phases FeB and Fe_2B that differ with the research done by Ozkan Ozdemir that found the single phase Fe_2B (Ozdemira *et al*., 2006). This phase is desirable because Fe_2B is more elastic than the FeB layer.

The substrate used for this study was S45C iron from PT Krakatau Steel product whose chemical composition are: 0,42 - 0,48 wt % C, 0,15 – 0,45 wt % Si, 0,60 – 0,90 Mn, 0,03 P, dan 0,035 S, respectively The cylindrical test pieces dimensions are 11 mm in diameter and 12 mm in length. Before boronizing heat treatment, all the samples were polished using fine polishing paper to obtain a good surface finish. The Vickers hardness test of this amples before boronizing is 196,38 HV (Setiawan, 2010).

Boronizing heat treatment was carried out by using pack boriding method. In this method, the cilindrical sample closed by boronizing mixed powder and treated in the kwarsa tube under vacuum condition. The size of boronizing mixed powder less than 1 μm in diameter. This powder contains of 50% B4C, 5% KBF_4 and 45% SiC which control the boronizing potential of the medium. All samples to be boronized were packed in the mixed powder and pressed by 5 ton force. Boronizing experiment was performed in an electrical resistance furnace under vacuum environment at 1000^{0}C for 8 hours and the boronized steel samples were cooled in the air.

The presence of borides formed on the surface of St37 iron was confirmed by X-ray diffraction (XRD) analysis. Shimadzu X-ray diffractometer (XRD 7000) with a Cu Ka radiation source of a wavelength of 1.541A ˚ over a 2θ range from 40^0 to 90^0 was employed for the phase characterization of borided layer of the samples. The microhardness of borides were measured using a Vickers microhardness tester. The thickness of boride layer was determined by a digital measurement instrument optical microscope.

In general, surface boronizing treatment can form single Fe_2B phase layer on the surface of plain carbon substrate. Figure 1 shows optical images of the boride layer on the S45C iron boronized by a solid boronizing method. According to the XRD data (Figure 2) and hardness test, "a" represents the boronized layer and "b" represents the substrate. The layer growth is dominated by the boron diffusion across the phase FeB substrate. Depending on process time and temperature, the depth of the boride layer ranges from 25 to 55 µm. Table 1 is a result nof GSAS analysis based on the XRD data. There is two kind of borided layers, FeB and Fe2B with the composisin of 85.42 % FeB layer and 14..58 % Fe2B.

Figure 1. Optical images of borided S45C iron at 1000^{0}C heat treatment for 8 h showing two distinct regions (a) refers to a layer having borides, and (b) refers to a substrate.

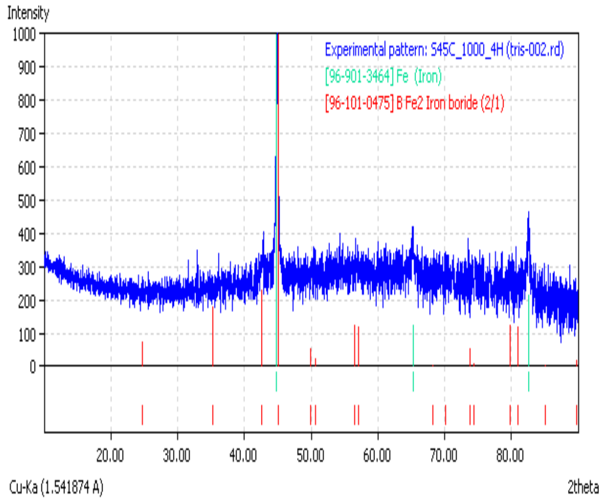

Figure 2. XRD data of borided layer on the S45C iron

Table 1. Mass fraction of borided layer on the S45C iron from the XRD data by using GSAS analisys

No.	Name of coumpound	Phase	Reference	Mass fraction (%)
1.	Iron Boride	FeB	ICDD-96-901-3464	85.42
2.	Iron Boride	Fe_2B	ICDD-96-101-0475	14.58

Microhardness measurements were carried out on borides layer both on the surface and cross section layer and substrate to compare the hardness of boride layer and transition zone respectively. An optical micrograph of borided iron including Vickers indentation marks is shown in Figure 2. According to the Vickers hardness test, the hardness of boride surface layer is much higher than the cross section layer, and the hardness of cross section layer is much higher than the matrix (Setiawan, 2010). According to Fe–B phase diagram, the obtained layer consists of orthorhombic boride FeB and tetragonal boride Fe_2B, which form columnar morphology that grow in a preferential direction [002], due to the maximum density of boron atoms in that direction. In this study, the borides formed on the surface of S45C iron substrate has flate morphology due to the tendency of Fe_2B crystals to grow along a direction of minimum resistance perpendicular to the external surface. A single-phase structure is desirable and Fe_2B is preferred to FeB, since FeB is very hard and brittle (Ozdemira *et al.,* 2006).

By controlling the boron activity of the boriding medium and chemical composition of substrate to be treated, it is possible to obtain a microstructure consisting of only Fe_2B phase without the FeB phase. Two regions were identified on cross-sections of borided S45C iron which are clearly indicated (Figure 1). These are: (a) a surface layer, FeB and Fe_2B phase; and (b) the substrate, which is not affected by boron to form borides. Hardness measurements were carried out from the surface to the substrate. It was shown (Figure 3) that the hardness decreased from region "a" (boride layer) to region "b" (substrate). It is important to show that the boride layer was harder than the substrate.

Microhardness of borides formed on the surface of S45C iron is much higher than the matrix due to hard boride Fe_2B. In addition, it was possible to observe a transition region formed between region "a" (boride layer) and region "b" (substrate). Moreover, that region is harder than pure iron, but it is softer than the boride layer. This was proved by microhardness test (Figure 3). The reason for the higher hardness of the transition region just below the boride layer is probably a result of a solid solution hardening between iron and boron.

In the Figure 5 of the test results using the XRD spectrum shows the phases formed Fe2B and FeB. With the formation of these phases have been changes in the lattice constants on the basis of the sample. Changes in the lattice constant of the system resulted in the formation of new crystals of the FCC (face central cubic) for steel St37 become ortorombic for FeB and tetragonal phase to phase Fe2B. The composition of borided layer can be seen from the EDX data analysis (Figure 4). The composition of borided was dominared was dominated by Fe about 74,3 mass %. Table 2 is a result of EDX data analysis. This table shows the composition of borided layer in mass %.

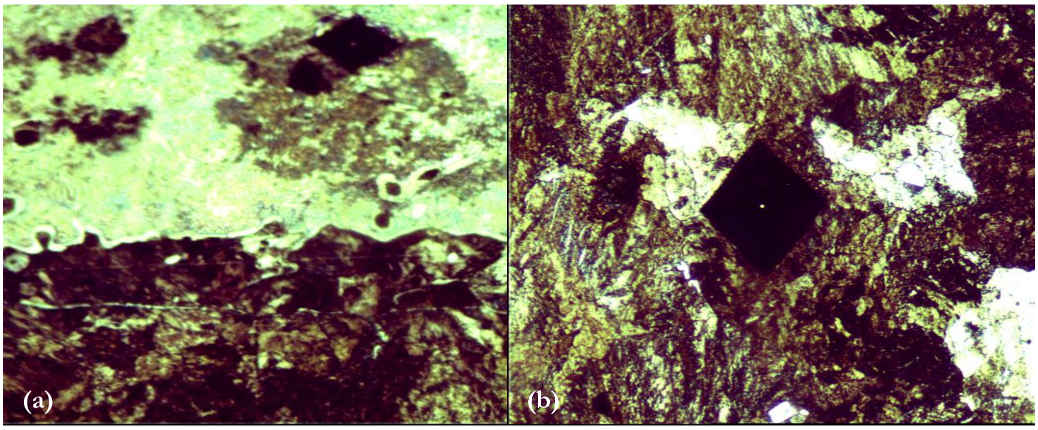

Figure 3. The variation of hardness of borides layer on the surface of S45C iron, a) on the surface, b) in the substrate.

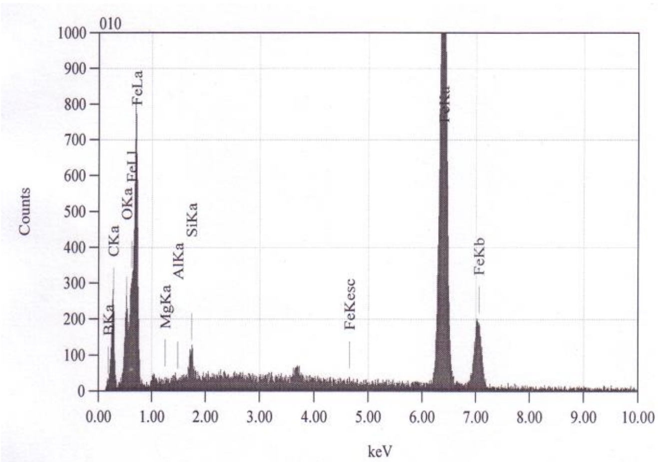

Figure 4. EDX data of borided layer on the S45C iron

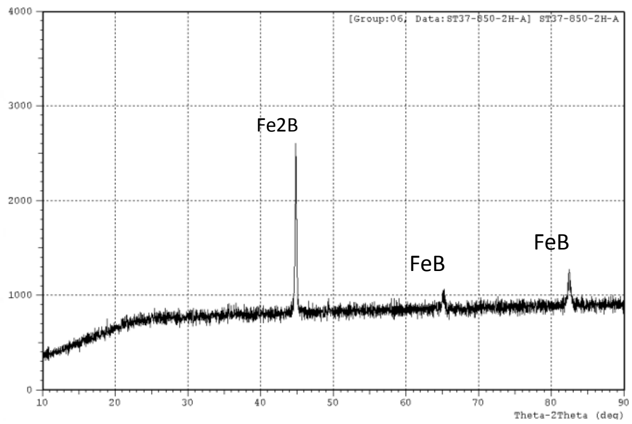

Figure 5. Spectrum of X-RD data on the borided layer of ST37 steel.

Table 2. Composite content of borided layer on the S45C iron from the EDX data.

Elemen	B	C	O	Mg	Si	Fe
Mass %	2.0	17.3	6.9	0.1	1.4	74.3

Conclusions

The borided materials are divided into three parts: (i) layer having boride; (ii) transition zone where boron makes solid solution; (iii) the matrix which is not affected by boron. X-ray diffraction studies revealed that S45C iron causes nucleation growth of Fe_2B and FeB borides. It was found from the XRD data and GSAS analysis that mass fraction of FeB and Fe_2B layer was 85.42 and 14.58 respectively. From the EDX data there is 2 mass percent boron as the hardener factor in the borided layer. Optical microscope analysis showed that the boride formation on the surface of S45C iron has a smooth and flate morphology.

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