Surface Hardening of St41 Low Carbon Steel by Using the Boronizing Powder and Pressured-heating Technique

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Abstract

The powder-pack boriding process by using pressured-heating technique was applied on the surface of St41 steel to produce the boride layer. The pressured-heating technique can reduce the operational cost of the boriding process if compared by the conventional technique. The concept of pressured-heating technique was verified in a laboratory scale. Well defined pressured-heating technique was achieved by using the stainless steel 304 as the container and sealed with a 10 kN pressure. This container was filled by boronizing powder consisting of $5\%B_4C$, $90\%SiC$, and $5\%KBF_4$ to close the St41 steel specimen inside the container. The container was treated at the temperature of 900^0C with the treated time of 2, 4, 6, and, 8 hours. A single phase occurs on the substrate was found as FeB layer with the hardness about 1750 HV. This value was more than ten times if compared with the untreated specimen that only was about 123 HV. Depend on heat treatment temperature, heat treatment time, and powder-pack boriding pressure, the depth of boride layer range from 126 to 186 µm.

Keywords: Boride layer, microhardness, pressured-heating technique, low operational cos.

Introduction

To obtain the hard iron that has a higher hardness if compared to the original material can be done by the boronizing process on low carbon steel. In the process of increasing the hardness of material, there is change in phase on the surface of low carbon steel, while the structure of the material is essentially not change[1]. In the technique of the surface development, there are many methods that can be used to improve the performance of the metal material surface. In general, these methods can be classified in two ways. First way, a thermal heat treatment that did not influence the chemical composition of the base material, such as flame hardening and induction hardening method. Second way, influence chemical composition of the base material, such as carburization, nitriding, karbonitridation, and boronizing methods[1]. The second way is frequent done in industry at a particular temperature and became known as thermo chemical treatment[2].

Among the four methods above, boronizing has the low operational cost and can produce the high surface hardness between 1450 – 5000 HV[2]. In the surface hardening method, boronizing is a thermo chemical process that can be treated on the various metal materials[3]. Boronizing on the metal surface is generally performed at the temperatures of 700^0C to 1000^0C for 1 to 12 hours and can be done in a medium of solids, liquids, and gas[4].

The result of boriding process on low carbon steel can form a single phase of boride layer FeB, Fe_2B, or a combination of FeB and Fe_2B phase[5,6]. The achievement of either single phase Fe_2B or FeB expected more because it will result in mechanical properties are better than in the combined phase. Other alloying elements in low alloy carbon steels also have the possibility to form a boride phase that will appear phases other than phase Fe2B or FeB. To reduce the reactivity between iron and boron and avoid the formation of oxide compounds that can inhibit the diffusion process, boriding method conducted with techniques to minimize oxygen[7]. The technique is usually done by creating an inert gas conditions with argon gas flow, or it can also be done by creating a vacuum during the heat treatment[5]. Besides that, it can also be done under pressure conditions during heat treatment.

The boronizing treatment has done on low carbon steel AISI 1018 with B_4C and KBF_4 powder mixture. Heating process was carried out at a temperature of 850^oC for 4 hours under argon gas and produce two-phases of borided layer Fe_2B and FeB with a layer thickness 75-80 µm and micro hardness HK 2250[8]. The same study has also been conducted on St37 and S45C steel producing two-phase borided layer Fe_2B and FeB, and the microhardness of these layers reached 1400 HV[9].

Boriding method under vacuum conditions was performed on the 99.9% pure iron with the boronizing powder mixture that consist of B4C, SiC, and KBF4. Sample heating at a temperature of 850^oC for 15 hours and a borided layer was formed as the Fe_2B and FeB phases and used three different types of composition for B4C powder was 10%, 100%, and 90%

by weight[10]. Then by the same technique, the boriding also conducted on 25% Cr alloy steel produce two-phase borided layer Fe_2B and Feb, using powder mixture of B4C and KBF4. The microhardness of resulted borided layer was 18 Gpa[11].

Both inert gas and vacuum system require the high cost and difficult to do in the world of industry-oriented business doe to less practical use. To face these conditions need to find a solution so that the pressured-heating technique can be done in a simple, fast, and more practical implementation without reducing the quality of the expected results.

Material and Method

The specimen used for this study is St41 low carbon steel whose chemical composition are: 0.20wt%C, 0.80-1.40wt%Mn, 0.015wt% S ,0.012wt%N, 0.020wt%Nb, 0.03wt%Ti, 0.40wt% Si, 0.025wt%P, 0.020wt% Al, 0.30wt% Cr, 0.08wt% Mo, 0.30wt% Ni, and 0.02wt% Vi, respectively[12,13]. The cylindrical samples dimensions are 3 cm in diameter and 1 cm in length. To get a fine surface, the samples were polished before heated[14].
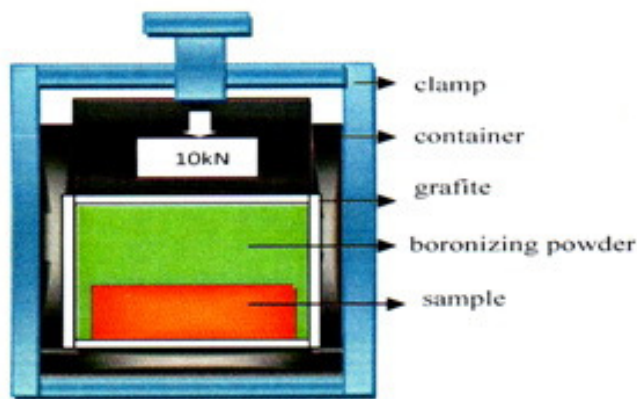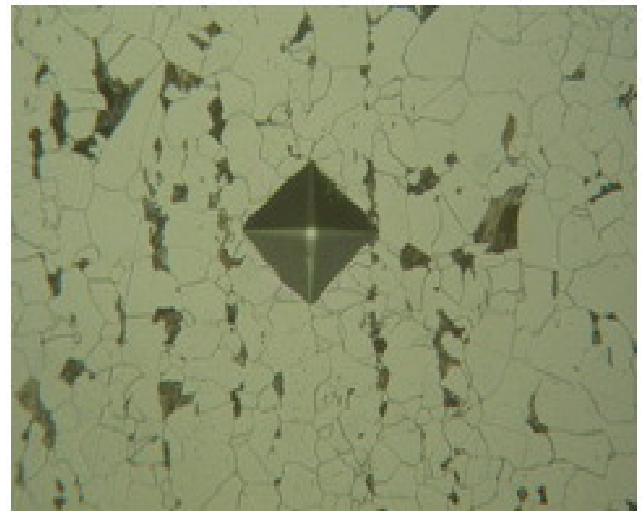

Figure-1
The clamped pressured-heating container

The heating of boronizing samples were done by using the boronizing powder that consist of 5% B4C, 5% KBF4 and 90% SiC. During the heating process, the samples were embedded by boronizing powder and pressed by 10 kN in the container made from stainless steel 304 as shown in figure 1. The pressured-heating container was used to create the boronizing samples and clamped container to keep the constant pressure and ready to treat in the furnace[15].

The heat treatment was conducted in the furnace under pressure condition at the temperature of 900^0C with the treatmen time 2, 4, 6, and 8 hours and cooled in the room temperature. The borided layers that formed on the St41 surface was analysed by X-ray diffraction (XRD) machine[16,17]. The microhardness of borided layers was identified using a Vickers identation. To calculate the microhardness used the following formula

$$HV = \frac{2F\sin\frac{136}{2}}{d^2},$$

HV is microhardness, F is weight in kgf, and d is the diagonals as shown in figure 2. The depth of boride layers from the surface can be determined by the image of the optical microscope.
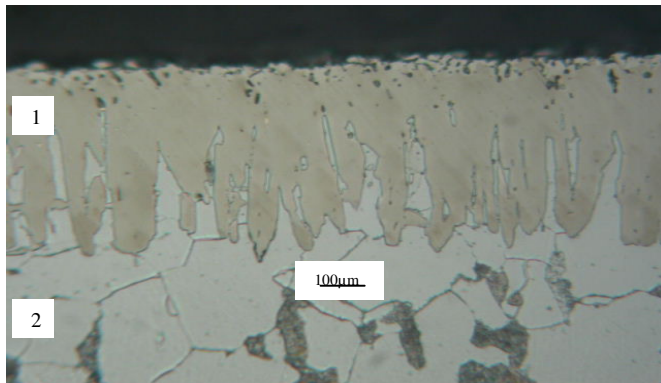

Figure-2
**The micro hardness trace on the borided material**

Results and Discussions

The optical images in figure 3 describe the boronized layer "1" and the subtrates "2". The borided layer has the saw-tooth morphology formed on the substrate. The occurring of borided iron is described by boron diffusion on the substrate. Morphology of borided iron on the surface of St41 like a shape of column[18]. The depth of the borided layer depended on the pressure, treatment time, and temperature, has ranges from 126 to 186 µm. The relation between treatment time and borided layer thickness formed the parabolic curve (figure 4).
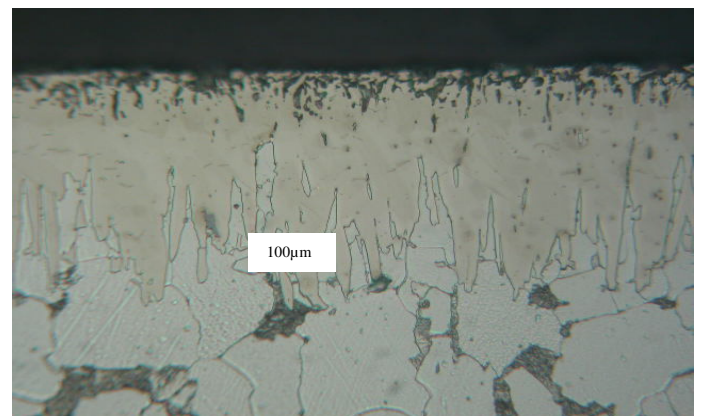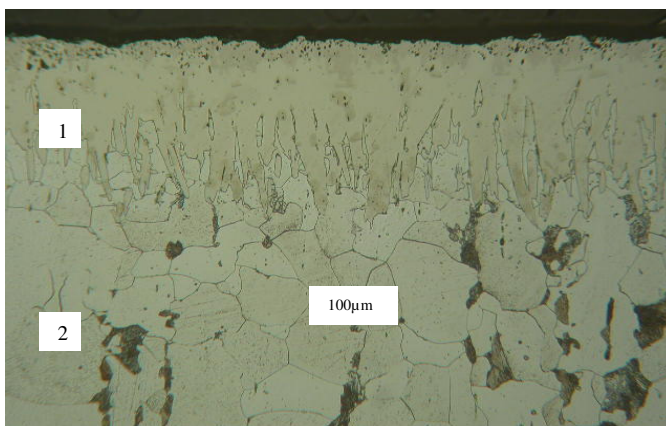

(a)

(b)
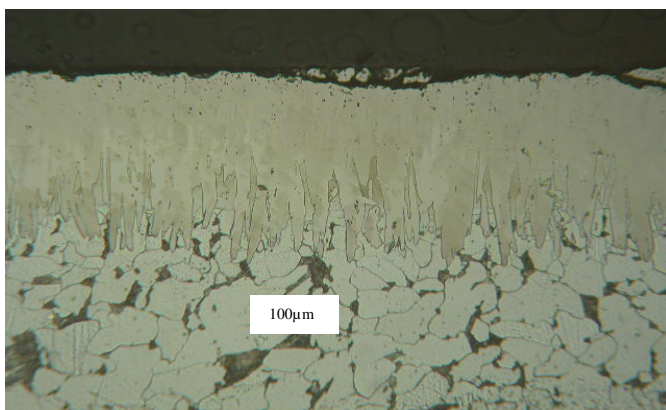
(c)

(d)

Figure-3
The optical images of boronizing treatment on the St41 steel at the temperature of 900°C for 2,4,6, and 8 h

Generally, the single phase of Fe$_2$B or FeB can be created on the surface of St41 by boronizing treatment. By monitoring the activity of boron, it is possible to obtain a microstructure consisting of FeB or Fe$_2$B phase only. Figure 5 described the x-ray diffraction pattern of FeB single phase that form the hard borided layer on the substrate.
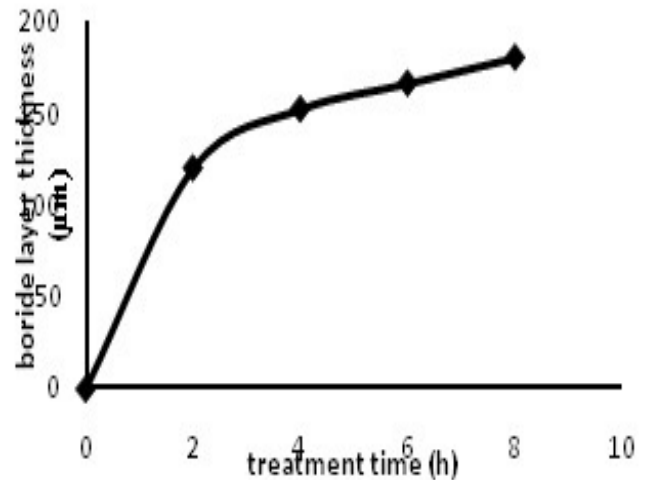

figure-4
The thickness variation of borided layer on the St41 steel was depend on the treatment and formed the parabolic curve

Acording to the x-ray diffraction data (figure 5), there was change in crystal system from the body center cubic (St41 steel) to orthorhombic (FeB phase). The detailed numerical change in crystal system parameter from the GSAS analysis data: phase was FeB, space group Pbnm , crystal sistem orthorhombic, lattice parameter a=4.058Å, b=5.503Å, c=2.947Å, lattice angle α=β=γ=90°, atom position x=0.125, y=0.180, z=0.250 for Fe, and x=0.610, y=0.040, z=0.250 for boron atom. The volume of FeB phase V=65.810Å^3 and chi-squared χ^2 = 1.18
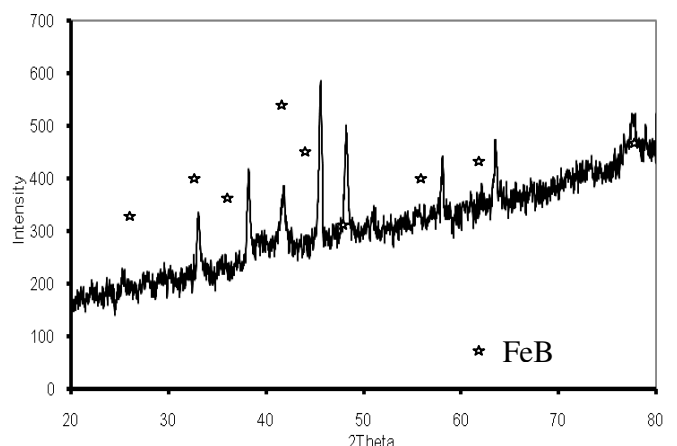

Figure-5
X-RD data of FeB single phase boride layer on the St41 low carbon steel.

The hardening of boronizing sample was identified from the surface to certain depth to observe a variation hardness of borided layer, transition zone, and matrix, respectively. An

optical micrograph of borided layer on the surface of St41 steel including Vikers indentation marks is shown in figure 5. In this figure, the hardness of borided layer is higher than the matrix. The hardness of borided layer and substrate on the cross section were about 1750 HV and 123 HV respectively can be seen on the figure 6.
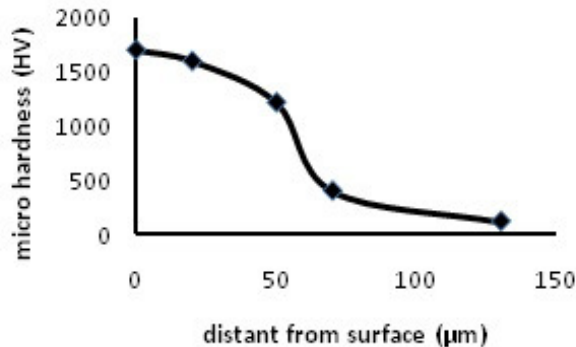

Figure-6
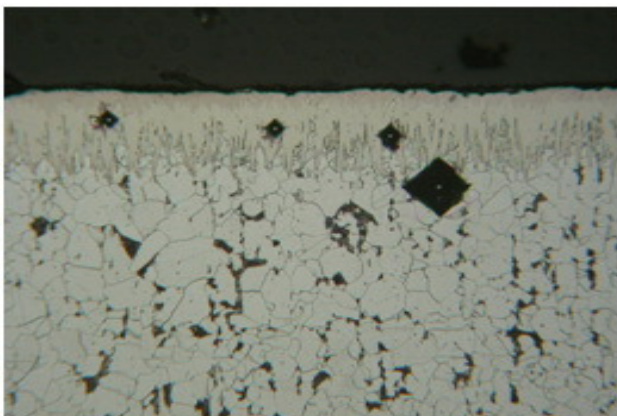**The relationship between distant from surface and micro hardness**

Figure-7
The variation of borided layer hardness from surface until the certain depth of the substrate on the St41 steel at 900°C for 8 hours

Refering to the phase diagram of Fe-B relationship, there is two phases boride layers FeB and Fe$_2$B with the shape of morphology like column. According to the Fe and B atom ratio between Fe$_2$B and FeB, there is different of boron concentration in those solid solution. The Fe$_2$B boride layer has the lower boron concentration than the FeB. Because of that, the single-phase structure of Fe$_2$B is better doe to a brittle of FeB. It is possible to obtain the Fe$_2$B phase only by selecting the chemical composition of specimen and boron activity in the boriding powder. On the cross-sections of borided St41 steel there is two areas can be clearly identified (figure 3). The first area is FeB

borided surface layer "1", and second area is substrate "2" there is no boron content to form the boride layer.

The microhardness of borided sample St41 steel is measured along the cross section. Microhardness decreased from borided layer area to substrate that can be seen at the figure 7. The hardening of borides layer on the St41 steel is higher than the matrix due to the hardness boride layer FeB. The transition area has microhardness more than untreated sample but it is softer than the borided layer on the surface of specimen[19]. This was can be shown by microhardness test at the figure 7. According to the hardness of transition area less than the boride layer due to solid solution hardening between iron and boron[20].

Thickness measurement of harder layer was done by optical microscope for treatment time variation from 2 hours to 8 hours (figure 3). It was showed that the thickness of boride layers on the St41 steel is depended on treatment time. The longer boronizing treatment time will be resulted the thicker boride layer[21]. The plotting result between treatment time and boride layer thickness formed the parabolic curve as shown in figure 4.

The growth of reaction kinetics was influenced by gradient and barrier of chemistry that leads the diffusion in the interface reaction and moves the reactant up to the interface. The kinetic growth was influenced by reaction; the layer would grow according to the treatment time. If the kinetic growth was influenced by diffusion process, the layer would grow according to the square root of the treatment time[22]. In this study, the boride layer growth was controlled by diffusion mechanism which formed the FeB layer on the surface of St41 steel.

Conclusion

Have successfully prepared a layer of boride iron with the higher hardness by using the pressured-heating technique. The borided material consist of three parts, borided layer, transition area where occur solid solution, and the substrate which is not affected by boron. It was found that the hardness of surface borided layer about 1750 HV and on the cross section the hardness of borided layer about 1200 HV. From the optical image analysis indicate that the boride layers on St41 low carbon steel has saw teeth morphology.

Acknowledgments

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