Technology of Iron Hardening by using the Pressure and Heating Technique

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Keywords: technology; surface hardening; iron

Abstract: Iron surface hardening technology is done with a particular technique to minimize oxygen in order to avoid the formation of oxide compounds that can inhibit the diffusion process and reduce the reactivity between the iron and the reinforcement material. Conventionally, this technique is done by creating an inert gas conditions using argon gas flow, or it can also be done by creating a system under vacuum conditions during heating. This technique is operationally costly because it must provide argon gas or vacuum machines are expensive. This study is intended to reduce the magnitude of these costs using the new methods with less operational costs. In this study, iron surface hardening process conducted by using mechanical pressure and heating without vacuum or argon gas flow. Samples iron S45C to be hardened is placed in a container shaped cylindrical tube made of 304 stainless steel and covered with a powder mixture consisting of 5% boron carbide (B_4C), 90% of silicon carbide (SiC), and 5% potassium boron fluoride (KBF_4) under certain pressure. Samples were prepared is heated at a temperature of 700, 800, and 900ºC with heating time for 8 hours. Layer formed on the metal surface can be identified as a layer of iron borides I (FeB) on the surface and a layer of iron borides II (Fe_2B) at certain depths. From the micro hardness test results using Vikers identor expected to obtain micro hardness value is greater than the base sample so it can be used as a raw material component machining.

1 INTRODUCTION

The results of research published by the German Federal Ministry of Research and Technology reported that the loss was economically due to abrasion and material wear of more than 10 billion DM and this figure is close to 1% of the state's budget (Walter, 1981). Every year large economic losses are also experienced by some industries caused by corrosion and wear damage to machinery and its components. The condition triggers the demand for improved surface performance of metallic materials whose impact has brought about advancements in the field of surface development technologies (Suwattananon, 2005).

The role of the surface of metal material is very important because it is the part that directly in contact with the environment. To maintain this role, efforts should be made to improve the surface resistance to environmental influences has a longer lifetime. The damaging effects of the environment on the surface of metallic materials include wear, corrosion, oxidation, and collision (Sugondo, 2010).

In addition to counteracting destructive environmental influences, the role of metal surfaces can also be increased so as to have added value and their usefulness can be extended to other areas.

Carbon steel metal material has been widely used in various components in the fields of industry, agriculture, machinery, and automotive (Bintang, 2005). The metal has advantages and disadvantages. Based on carbon composition, carbon steel can be grouped into several parts, including low carbon steel with carbon composition of 0.08 to 0.35%, medium carbon steel with carbon composition 0.35 to 0.50%, and high carbon steel with composition carbon 09.55 to 1.70% (Smallman, 1999).

Low carbon steel has several advantages, including strong and ductile, easy to do with machining, easy to shape, easy to weld, cheap price, and many available in the market. In addition, low carbon steels also have disadvantages such as relatively low hardness, low wear resistance, no resistance to corrosion attacks, and easilyoxidized so that pearlite phase decomposition occurs at high temperatures (Bintang, 2005).

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The surface hardening can be performed on low carbon steels so that the material obtained has a higher hardness when compared to the hardness of the original material but remains strong and ductile. In the process of increasing the hardness occurs phase changes on the surface of low carbon steel, whereas in the basic material structure does not occur changing the phase (Bintang, 2005) .

There are many methods that can be used to improve the surface performance of metallic materials as a result of surface development technologies. In general the method can be done in two ways. First, the method does not alter the chemical composition of a base material called thermal heat treatment, such as the flame hardening and induction hardening methods. Second, the way is done by changing the chemical composition of the base material called thermochemical heat treatment, for example carburization method, nitriding, carbonitriding, and boronizing (Bintang, 2005). This second way is mostly done in the industrial world at a certain temperature so known as thermochemical treatment (Roumiana, 2008).

Among the four methods mentioned above,boronization is the most superior method because it can provide better results on the surface performance of metallic materials (Anil Kumar Sinha, 1990). Boronization is a thermochemical process in surface hardening that can be applied to a variety of metal materials, both ferrous and non-ferrous metals. Boronization methods on the surface of metallic materials are generally carried out at temperatures of 700ºC up to 1000ºC for 1 to 12 hours and can be carried out in solid, liquid, and gas media (Anil Kumar Sinha, 1990).

As a result of the process of boronization on low carbon steel will form a layer of boride iron with the possibility of a single phase FeB, Fe_2B, or FeB and Fe_2B combined phases (Gopalakrisnan, 2001). The formation of single phase both FeB and Fe_2B is more desirable because it will produce better mechanical properties than the combined phases. In addition, other constituent elements in low carbon steel alloys also have the possibility to form a boride phase so the other phases (Setiawan, 2010) will occur.

The method of boronization is done by minimizing oxygen technique to avoid the formation of oxide compounds that can inhibit diffusion process and reduce the reactivity between iron and boron (Martini, 2004). The technique is usually performed by creating an inert gas condition with an argon gas flow, or it can also be done by making a vacuum during heat treatment (Martini, 2004). In addition it can also be carried out under atmospheric pressure conditions during heating.

Roumiana et al in 2008 has carried out boronization of powder on low carbon steel AISI 1018 with a mixture of B_4C and KBF_4 powders. The heating process was carried out at 850° C for 4 hours under argon gas conditions and resulted FeB and Fe_2B borate layers with 75 to 80 μmthickness and 2250 HK hardness. The same study has also been done by Sugondo in 2007 on St37 steel resulted FeB and Fe_2B borate layers with hardness reaching 1400 HV (Sugondo, 2010).

Boronization methods under vacuum have been performed by Martini et al in 2004 at 99.9% pure iron with different powders B_4C, SiC, and KBF_4 compositions. The sample heating was carried out at 850ºC for 15 hoursformedFeB and Fe_2B using 3 different composition types for B_4C powder 10%, 100%, and 90% weight (Martini, 2004). Then in 2006 with the same technique Dybkov et al do boronization on iron alloy 25% Cr resulted FeB and Fe2B borate layers using mixed powders B_4C and KBF_4. The micro hardness that occurs in the boride layer is 18 Gpa (Dyvkov, 2006). Both inert gas and vacuum engineering need the high cost and difficult to do in a business-oriented industry because of its less practical use.

To overcome these conditions need to find a solution so that the heating technique can be done simply and the implementation is more practical without reducing the quality of the expected results.In this research will be applied the pressure and heating technique with a certain pressure on boronization powder during the heating process without reducing the quality of expected results. As the basic material selected S45C low carbon steel which is cheap and easily available in the market.

Neither the first method involves altering the chemical composition of the base material nor the second way by changing the chemical composition of the base material, all by a vacuum or by an inert gas stream. If the equipment is not good then leakage will often occur so that the hardening process on the sample that is scientifically manifested in the form / phenomenon of diffusion can not take place. Both inert gas engineering and vacuum engineering both cost considerable and difficult to do in a business-oriented industry because of its less practical use.

2 RESEARCH METHOD

The basic materials used as the basic samples are iron S45C. Iron S45C consists of elements 0.42 -

0.48 wt% C, 0.15 - 0.45 wt% Si, 0.60 to 0.90 Mn, 0.03 P, and 0.035 S (Setiawan, 2010). To know the mechanicalproperties of the sample before boronizing, sample characterized by XRF, XRD, and micro hardness testing. The surface of the sample to be diboronized first cleaned from the impurity layer and smoothed in order to obtain maximum results.

Figure 1. Basic sample of soft iron type S45C

The composition of the powder mixture used includes 50wt% B_4C, 5wt% KBF_4, and 45wt% SiC. The mixed powder is inserted into the iron base sample S45C, and pressed with a pressure of 15 tons and then heated in a furnace with a temperature of 700, 800, and 900ºC for 8 hour heating time.A container containing a working sample that has been sealed with a boron powder is fed into the furnace to be heated. Figure 2 shows the container that has contained the working sample put in the furnace and is ready to be heated.

a

b

c

d

Figure 2. The sample into cylinder and lead (a), boriding powder covered the sample (b), cylindrical container claimed by pressure (c) container in the furnace is heated.

By applying pressure to the boronization powder, the heating of the container in the furnace is carried out directly without using a vacuum system or draining the argon gas into the furnace.The heating setting begins by raising the heating temperature from room temperature to 700°C within 30 minutes. After reaching the temperature 700°C held for 8 hours. This process is carried out also for temperature of 800 and 900°C during 8 hours . When finished the heater is stopped and the cooling is done naturally at room temperature.

If it has been shown room temperature, the boronized treated work sample is removed from the heater.To find out the changes, the already boronized sample is characterized and tested. Characterization was carried out to find out the change of micro structure, whereas the test was performed to see micro hardness at the cross section of the boride layer. The diffraction pattern of the obtained XRD data was analyzed using GSAS to determine the phase composition of the boride layer.

In a sample that has been boronized observations of microstructure to see the morphology of the boride layer formed. The observations use a Nikon optical microscope with 200 times magnification. The depth of the boronized borate layer is obtained from the average depth of sawtooth (Setiawan, 2010). All of the boronized samples were tested for hardness using a LeitzMiniload test kit with a 5 kg load. The indenter used is Vikers type. The vertical pyramid indicator angle is 136 °. Giving style is done slowly without collision. When touching the indenter is held for 10 to 15 seconds. Styles are given according to the load mounted on the test equipment. After the force is removed, the track of the indenter on the sample is measured. The material hardness is calculated by equation (Setiawan, 2010):

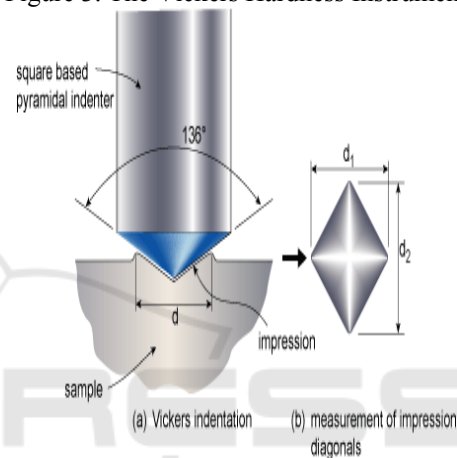Figure 3. The Vickers Hardness Instrument

Figure 4. The Vikers identor Leitz Miniload

$$HV = \frac{2000\ P\sin(a/2)}{d^2} = \frac{1854.4P}{d^2}$$

HV = vikers micro hardness;

d = average identor spell;

P = given force

Micro hardness testing is performed on two different points to see the gradationhardness of the boride layer. The assumption in this test is at a certain depth of valuethe violence is the same.To determine the diffraction pattern and the atomic position in the molecule the samplecharacterized by XRD. From the obtained diffraction pattern can be calculated size of crystallites (particle size). The crystallite size can be calculated using the Debye equation Scherrer as follows (Setiawan, 2010):

$$\beta_{hkl}\ \frac{K\lambda}{L_{hkl}Cos\alpha}$$

Where:

K : form factor, was 0.9 for ball shape.

λ : X-ray wavelength (Å).

β_{hkl}: FWHM (rad)

L_{hkl}: crystal size (Å)

θ : diffraction angle.

3 RESULT AND DISCUSSION

The basic material samples performed by initial characterization are S45C which is a soft iron or low carbon alloy steel. Both samples characterized using XRF (X-ray flourence) to determine the composition elements in the material and XRD to know the phases and crystal structures. The boronization powder used in this study is B4C, SiC, and KBF$_4$. After that the three powders are mixed with each other so that obtained boronizingpowder ready to use. The steps of execution of sample characterization and boronization powder are as follows.

Table 1. The composition in Steel S45C

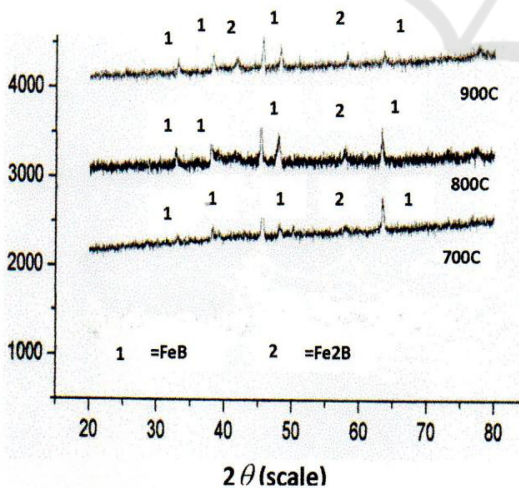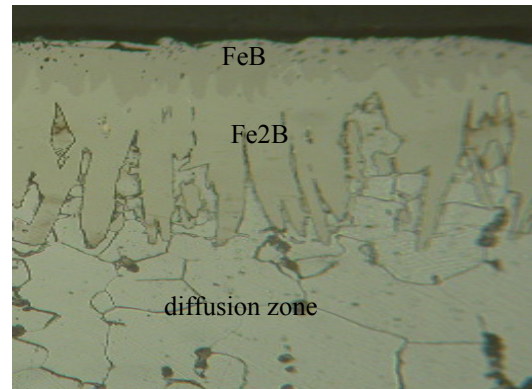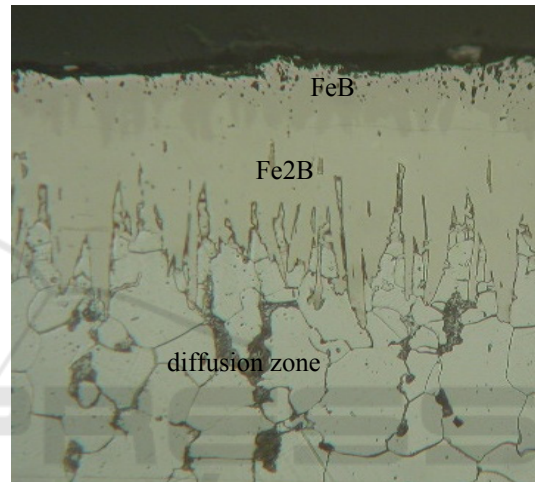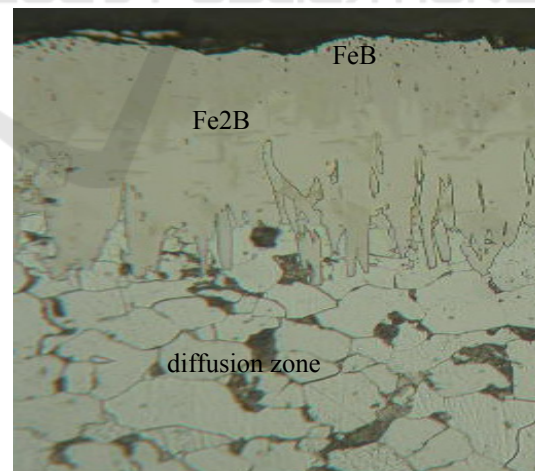| Element	C	Si	Mn	P
S45C	0.45%	0.30%	0.6-0.9%	0.03%

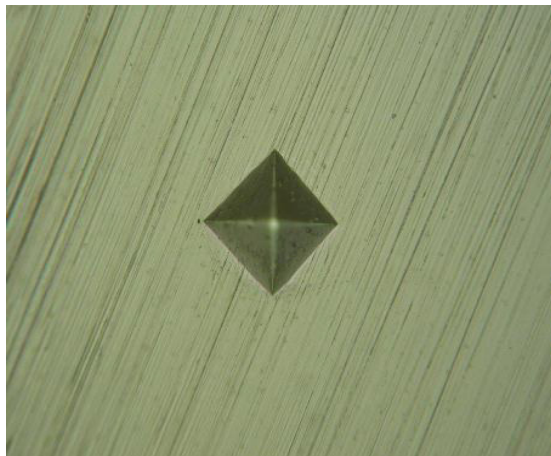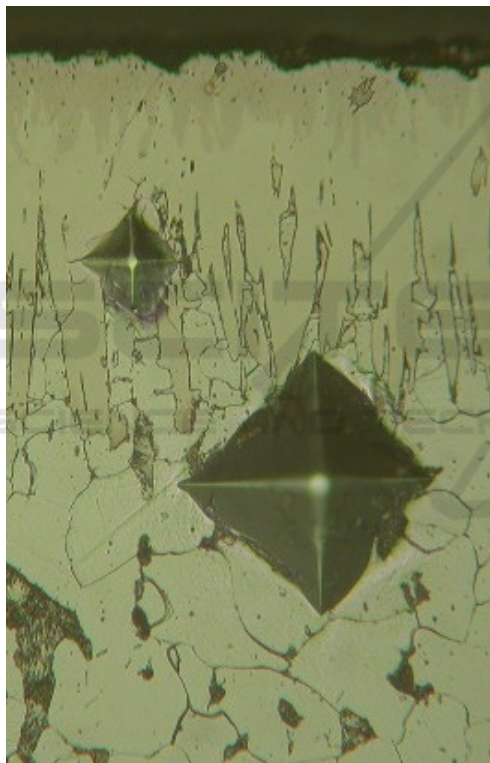Figure 5. XRD data of iron sample on the temperature of 700, 800, and 900°C.

Figure 6. View of boridedcross-sections treated at (a)700°C, (b) 800°C, and (c) 900oC for 8 hours with powder pack boriding thickness.

a

b

Figure 7. The hardness of iron S45C (a) before treatment (b) after treatment

The results of characterization with XRD for the S45C iron samples indicate Fe iron phases at certain angles (figures 4). While the results of characterization with XRF written in table 1 also indicated the composition of carbon elements with low concentration. This suggests that the S45C iron samples are low carbon steels which are often used

as samples on the engineering of metal surface hardening.

In order to obtain better results in the surface hardening of the S45C iron samples, it is necessary to minimize the particle size of the boronized powder particles which will serve as the raw material for metal hardening. The size of the particle diameter can be obtained by milling (piercing) the boronization powder within 10 hours. Boronized powder is inserted into tubes containing iron balls of different diameter sizes. Tubes that already contain iron balls and boronisasi powder then vibrated or rotated with a certain speed and time. From the results of the milling obtained a decrease in particle diameter size from 8.374 micron to 0.6885 micron boronized powder.

The comparison of hardness S45C iron samples between before and after boronization has increased. The hardness on the iron surface S45C was 125 HV before treatment and after boronization the hardness increased to 887 HV.

4   CONCLUSION

From the sample preparation and characterization results temporarily, it can be concluded:

1. With heating and pressure technologies applied to powder boronization hardening methods can increase the surface hardness of iron samples S45C from 125 HV to 887 HV.
2. In terms of research operational costs, heating and pressure techniques can save up to 60% when compared to vacuum or argon gas drainage techniques

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