Effect of boro-sintering process on mechanical properties and wear behaviour of low alloy steel produced by powder metallurgy

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Abstract
Boro-sintering is subjecting P/M metal components to boronizing process simultaneously with the sintering process. In this study, powder particles with AISI 1010 steel composition were compressed by cold pressing method under 1000 Psi pressure and then, were subjected to sintering in silicon (Si) and EKabor 2 medium at 1000 °C for 2, 4 and 6 h, respectively. Following the sintering process, the specimens produced by P/M were characterized by optical microscope, Scanning electron microscope, XRD analyses, 2D profilometer, micro hardness and wear test. The porosity values of the samples decreased and the specific gravity and surface roughness increased depend on the increase in boro-sintering temperature and time. A coating layer, similar to the boride layer formed in the steels, was observed in the specimens sintered with boron. On the other hand, no coating layer was observed in the specimens sintered in Si medium but there was some increase in their surface hardness in a limited area. The boride layer formed on the surface as a result of boro-sintering process has a thickness of 120–150 μm and hardness values of 1300–1500 HV. In this study, it has been proven that sintering and thermal treatment with boron carried out separately in the existing literature have a positive effect on mechanical and wear properties when they are carried out in a single step.

1. Introduction
Powder Metallurgy (P/M) is an essential technique for producing components with complex and complicated properties that resemble the clear shape the most among other metallurgical methodologies. The basic features of P/M include dimensional accuracy, high surface quality, repeatability, cost-effectiveness and material savings due to the lack of secondary processes [1, 2]. Unlike conventional production methods, this method does not require melting to form complex components and allows high alloy components to be used due to the rapid solidification of powder production [3]. PM is a versatile method of producing almost clearly shaped metal components in mass production. PM steels are widely used in the manufacture of structural components in automobile and mechanical fields [4]. This processing technique allows the modification of the microstructure of materials to achieve a good combination of mechanical properties, wear and corrosion resistance [5]. However, the mechanical properties of PM steels are lower than those of forged steels of the same composition usually due to their porosity ranging from 5 to 15% [6].

Production by powder metallurgy generally consists of mixing powders, pelleting the mixed powders and sintering at a temperature below the melting temperature. High pressures can be achieved in the pressing phase to produce denser components and therefore, products with superior physical and mechanical properties. Wear and corrosion properties of the material can be improved by the phases formed from the sintering phase. The sintering process first increases the contact area of the powders in the pressed component. The voids between the powder particles become spherical. Increased dwell time causes the particles to aggregate, resulting in the condensation of the fragment by allowing the voids to migrate [7].

Boronizing is a thermo-chemical surface treatment process involving the diffusion of boron atoms to the metal/alloy surface in order to form a boride layer [8–10]. Boronizing can be applied to a wide variety of steel
alloys such as carbon steel, low alloy steel, tool steel and stainless steel. In addition, materials such as nickel-based alloys, cobalt-based alloys, molybdenum and titanium can be borided to obtain very high hardness and wear resistance on their surfaces [11, 12]. During the boronizing of iron alloys, the boron potential of the boriding medium, the chemical composition of the substrate, the process temperature and time control the phase structure of the boride layer. Depending on the concentration of the diffused boron, the phase structure of the boride layer may consist of tetragonal Fe₃B (8 wt% B) and orthorhombic FeB (16 wt% B) [13–15].

Borided steel components show excellent performance in a large number of tribological applications in the mechanical engineering and automotive industries. For practical applications, the formation of a single phase (Fe₂B) is more desirable than a layer of FeB and Fe₂B phases [16, 17]. This results from the fact that boron-rich FeB phase is more brittle than the Fe₂B phase although it is harder. Furthermore, crack formation is often observed as FeB and Fe₂B phases exhibit significantly different thermal expansion coefficients at the FeB/Fe₂B interface of a biphasic layer [18, 19].

This study investigates concomitant boronizing of a component during its production with powder metallurgy without any need for an additional process (boro-sintering process). Review of the literature reveals that any study on boro-sintering process has not been performed. However, it is known that both post-production boronizing and boron additive affect the mechanical properties of alloys and composites positively. By applying the boronizing process during sintering, it can be ensured that the substrate material is free from problems such as grain coarseness and loss of mechanical properties during subsequent boronizing. In addition, additional boronizing for iron-based alloys with a residence time of 800 °C and above can also save costs. For this purpose, in this study, the effect of performing sintering and boronizing that are carried out in close temperatures in a single process step in the form of boron-sintering on microstructure, hardness and phase formation of AISI 1010 steel was investigated.

2. Materials and method

Commercially available powders in the range of 50–150 μm were weighed to meet AISI 1010 steel and prepared by mechanical alloying for a homogeneous mixture. Then, the powder blend was poured into the prepared mould at amounts to allow producing specimens of 40 mm in diameter and 5 mm in thickness and cold pressed by applying a pressure of 1000 Psi. Cold pressed specimens were subjected to boro-sintering and sintering processes at 1000 °C for 2, 4 and 6 h, respectively in two different sintering media, i.e. commercial EKabor 2 boriding powder and Si powder alone.

The density measurements of the materials, from which raw specimens were produced, were determined by placing the weights measured in air and pure water in the equation (1) based on the Archimedes’ principle.

$$\rho = \frac{\text{(dry weight/dry weight} - \text{weight in the liquid}) \times \text{liquid density}}{\text{mass of the specimen}}$$

The boro-sintered specimens for metallographic examination were cross-sectioned and hot mounted. Then they progressively ground using grit abrasive papers (from 320 to 1500) and polished with 3 μm, 1 μm and 0.5 μm diamond paste. To reveal fine microstructure, after polishing processes, the specimens were etched with a solution of 4 wt% nital and 96 wt% water. Optical investigation was carried out using a Nikon MA-200 metal microscope equipped with Clemex image analysis software. X-ray diffraction (XRD) analyses were carried out using a computer-controlled Rigaku-SmartLab with Cu Kα radiation (40 kV, 30 mA) and 2θ angles ranging from 5 to 90.

Micro hardness measurements were recorded utilising QNESS Q10 with a Vickers pyramid indenter at 10, 20, 40, 80, 120, 160, 240, 360, 480, 600 μm along the cross-sections from the surface to the interior by using 100 gf load and 15-s dwell time.

The surface roughness (Ra) and (Rz) were measured on all produced specimens by using a MITUTOYO–SJ-210. The hardness values were reported as the average values from five measurements.

Sliding friction and wear experiments were performed using a ball-on-disk tribometer (TURQUOISE 2.0 Ttribometer T10/20) with linear motion at room temperature and under dry conditions against 6 mm diameter WC (1917 HV hardness) ball. Prior to the wear experiments, all specimens were slightly ground with abrasive paper with a grit size of 2500 for 5 min. Then abrasive balls and surface of the specimens were ultrasonically cleaned in acetone for 15 min and dried in air conditions. Considering the application areas of P/M specimens, they were exposed to linear wear test experiment at 250 m sliding distance under 5 N, 10 N and 20 N, respectively. When the wear tests were performed in short distance (50 and 100 m), the wear marks were not clearly seen. Therefore, we have not evaluated them. Three wear tests were conducted for each sample. After wear experiments, worn surfaces of each sample were analysed by using Evo LEQ SEM with EDS. The wear marks were determined as the average values of three measurements which were taken from the widths and
depths of the wear marks formed on the surface of the sample as a result of wear experiments. Wear volume losses were calculated as described in detail by the previous study [4].

3. Research findings and discussion

3.1. Microstructure

SEM microstructure images of AISI 1010 steel T/M specimens sintered for 2, 4 and 6 h, respectively in Si medium are shown below.

As shown in figure 1, specimens sintered in Si medium have three different structures. These are matrix structure (Spot 3, Spot 6 and Spot 9), porous structure (Spot 1, Spot 4 and Spot 7) and grain-resembling
structure (Spot 2, Spot 5 and Spot 8). The matrix structure contains Fe in the range of 89.10–93.63 wt% and it draws attention that the Fe content is <60 by weight in the approx. 80% regions referred to as 'porosity', which have a structure resembling to grain boundaries. This shows that discontinuities in the microstructure are closed and bond formation between powder metal particles becomes stronger as sintering time increases. Figure 2 shows SEM images and EDS analyses of boro-sintered specimens for 2 h at 1000 °C in the EKabor 2 powder medium.

When cross-sections of specimens that were subject to sintering process in Ekabor 2 medium are examined, it can be observed that a boride layer with a toothed structure similar to that of low alloy steels is formed on their surfaces in the range of 100–125 μm, a diffusion zone underneath it in the range of 250–350 μm and a P/M matrix with a high porosity content below it. When the resulting boride layer is examined, it can be observed that the boride layer increased with the increasing boronizing time; however, the amount of oxide in the diffusion zone also increased. When examined in terms of morphology, a saw tooth boride layer was formed similarly with
low alloy steels. However, it is noteworthy that the boride layer formed has higher porosity compared to the steels. This can be attributed to the penetration of O₂ into the P/M material at high temperatures as well as the boron in the medium during the boro-sintering process, since the cold-pressed powders are only mechanically bonded (no chemical bond) prior to the boro-sintering process because O₂ ratios detected in boro-sintered specimens are higher compared to the specimens sintered in Si medium. When the boride layer formed on the surface of P/M materials is examined, it is found to contain approx. 4% of B by weight. This is a low result based on the Fe-B balance diagram (8.93% Fe₂B and 16.23% FeB). However, XRD analyses show that both Fe₂B and FeB phases are formed in the boro-sintered specimens (figure 3).

When figure 3 is examined, it is ascertained in the specimens sintered in the Si medium (figures 3(a)–(c)) that the dominant phase was Fe, but a very small amount of FeMn phase and Si and SiC was formed due to the Si content in the sintering medium. In the specimens subjected to boro-sintering process (figures 3(d)–(f)), Fe phase as well as Fe₂B and FeB phases determined in the boronizing of low alloy steels were observed. With the increasing time in the boro-sintering process, Fe₂B phase replaced the dominant Fe. This supports the SEM and EDS results since thicker boride layers with higher boron contents were obtained with the increased boro-sintering process (figure 2).

In order to ascertain some post-sintering properties of the specimens, surface roughness measurements were taken from the specimen surfaces, and surface thickness and hardness measurements were taken from the sections of the specimens (table 1).

### 3.2. Hardness behaviour

When figure 4 is examined, it is observed that surface roughness values and thickness of heat-affected regions increased in both Si medium and EKabor 2 sintered specimens. The temperature affected area of the sintered specimen in Si medium is 100 μm thick and has a hardness value of approximately 2 times of the internal specimen. This can be attributed to the rapid post-sintering cooling. In boronized specimens, the boride layer was in the range of 120–150 μm and hardness values in this region changed in the range of 1300–1500 HV. The resulting hardness values increased by 13–15 times of the hardness values of P/M AISI 1010 specimens (108 HV). It is observed that the hardness values gradually decrease from surface to matrix in a similar manner with steels and correspond to the hardness of untreated P/M material after 500 μm (figure 4). However, it was observed that the hardness values obtained when boride layer hardness values were compared with steels were lower. This is associated with the amount of porosity and oxide within the P/M materials, which are used to characterize such materials. This results from high differences between the hardness values of some regions and the hardness values of 10 taken from the boro-sintering layer, as can be seen from the standard deviations.

### 3.3. Dry-sliding wear tests

During dry sliding wear, cold boil occurs between materials due to the sudden increase in temperature and pressure that occurs at the actual contact areas in the local regions between the interactive surfaces. And as the wear progresses, these bonds break again. Depending on the characteristics of the tribological system, these broken particles are either discarded from the surface or rejoined by holding onto one or both surface of the wear pairs. A number of parameters play a role in the dry sliding wear system. Archard reports that the volume of wear
Table 1. The thickness, surface hardness and roughness of the P/M specimens subjected to sintering and boro-sintering heat treatment.

| Sample | Heat treatment       | Treatment temperature (°C) | Treatment duration (h) | Coating thickness (μm) | Diffusion Region | Surface hardness (HV0.1) | Surface roughness (Ra, μm) | Density (gr/cm³) |
|--------|----------------------|-----------------------------|------------------------|------------------------|------------------|--------------------------|-----------------------------|-----------------|
| S1     | Sintering (Si)       | 1000                        | 2                      | —                      | 22 ± 2.8         | 195 ± 15                 | 3.08                        | 5.8015          |
| S2     |                      |                             | 4                      | —                      | 29 ± 4.7         | 201 ± 15                 | 3.54                        | 5.9920          |
| S3     |                      |                             | 6                      | —                      | 38 ± 4.5         | 210 ± 15                 | 4.06                        | 6.1830          |
| S4     | Boro-sintering (EKaboron2) | 1000                     | 2                      | 105 ± 10.5            | 275 ± 5.1        | 1452 ± 150              | 4.60                        | 5.6845          |
| S5     |                      |                             | 4                      | 123 ± 12.3            | 283 ± 8.4        | 1490 ± 161              | 5.70                        | 5.7652          |
| S6     |                      |                             | 6                      | 135 ± 17.1            | 275 ± 6.5        | 1525 ± 175              | 6.70                        | 5.8571          |
occurs as a function of sliding rate, normal load and material hardness (Archard, 1953; Archard and Hirst, 1956). According to Rabinowicz, the hardness of the roughness is more crucial than the volume hardness of the material as the interaction in the actual field occurs through roughness.

Volume loss values obtained as a result of wear tests are shown in figure 5. Firstly, it was observed that the volume losses in the specimens increased with the increasing normal load. Any increase in the normal force on the ball as the load increases will cause the abrasive to sink further into the specimen. Due to the pressure and shear forces that occur, the loss of volume will increase as the abrasive will cause more plastic deformation of the material. When boro-sintering process and specimens sintered with Si were compared, boro-sintered specimens showed better wear performance. In addition, the volume loss values of the specimens subjected to boro-sintering at 5 and 15 N loads were found to be closer to each other. This is primarily due to the higher surface hardness of the specimens subjected to boro-sintering. Furthermore, the increase in the boride layer thickness of the specimens due to the increased boronizing time played an effective role in this process.

Figures 6(a), (b) show the wear surface images of specimens subjected to dry sliding wear under loads at 5 and 15 N and sintered in the Si medium for 6 h. It was concluded that the specimen treated with a load of 15 N had a higher volume loss but there was no direct correlation between the increase in load and the increase in volume loss. This can be better seen from SEM images of the specimen because it is understood that a stronger oxide layer is formed on the surface of the material at higher loads.

When the high temperature dry-sliding behaviours of the materials are tested, the wear process starts and as it continues, the wear particles breaking off the surface are oxidized with the effect of excessive plastic deformation and temperature on the material surface and these particles form a tribological layer on the material surface. This formation occurs as a result of a sintering reaction under the repetitive load and nominal stresses of the wear
particles that break off at the micron level and accumulate at the surface. This layer creates a solid lubricant effect between the material surfaces. The solid lubricant effect plays a vital role in reducing the shear forces that have a significant effect on the wear of the specimen. Similarly, the formation of the oxide layer on the surface of the material is also applicable for studies carried out at room temperature since the interaction that emerges in a narrow area during the wear process causes a sudden increase in pressure and temperature in this region. This leads to a layer formation similar to the formation at high temperatures. A similar case was also observed in the study as in some specimens, the volume losses decreased at high loads. Wear results of the specimen abraded with lower load in SEM wear scar images shown in figure 7 a-b were found to be similar to the wear values obtained in the test conducted at 15 N.

4. Conclusion

In this study, powder particles, equivalent to AISI 1010 steel that is commonly used in industry, were produced in 40 mm diameter and 10 mm thickness with cold pressing method at 1000 Psi. Then, specimens produced with T/M were sintered in two different media (Si and EKabor 2) at 1000 °C for 2, 4 and 6 h, respectively and the effects of sintering in different medium and at different temperatures on metallographic properties and mechanical properties were investigated. The results are outlined below:

1. The amount of porosity decreased in the specimens sintered with Si and Ekabor 2 in terms of microstructure as the duration of sintering process increased. The amount of porosity in boro-sintered specimens is higher. This enabled the specimens to have lower specific gravity values.
2. A boride layer obtained during the boronizing of steels in specimens subjected to boro-sintering process and a coating layer similar to diffusion and matrix regions were obtained.

3. Depending on any increase in the boro-sintering time of the obtained coating layer, layer thickness and hardness values increased. Depending on post-sintering Fe₂B and FeB phases formed on the surface, a layer having a hardness value of 13–15 times higher than the untreated specimen having a hardness value of 1300–1500 HV up to a depth of 150 μm was obtained.

4. The surface roughness values and specific gravity values increased depending on the increase in the boro-sintering process time.

5. As a result of XRD analyses, Fe was determined as the dominant phase and Si and SiC phases were determined at small amounts in the specimen sintered in Si medium. On the other hand, following the boro-sintering process, Fe, Fe₂B, and FeB phases were found to be the dominant phase and small amounts of SiC phase was detected.

6. The wear resistance of the specimens subjected to boro-sintering showed better wear resistance compared to other specimens. With the increased load, the glaze layer formed on the surfaces of the specimens increased the stability and wear volume losses.

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