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Investigation of The Effect of Different Activators on Borosintering Process

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***Abstract*— In parts produced by the P/M method, boriding is generally applied as a separate process after sintering. If the package boriding process is carried out during the sintering of the powder compact, sintering and boriding can be integrated; This not only prevents parts from deforming due to secondary heating, but can also reduce energy consumption. Carrying out sintering and boriding processes simultaneously is called Borosintering (B/S). Studies in the literature have stated that various material properties are improved thanks to the boride layers formed as a result of the borosintering process. It has been observed that the effect of different boronizing environments on the borosintering process has not been examined in any of the studies. In this study, media containing different weight ratios of silicon carbide (75%, 85%, 90%), boron carbide (5%, 10%, 20%) and the remainder one of various activators (Na_2CO_3, NaCl, $Na_2B_4O_7$ (anhydrous borax) and NaF) in the boronizing medium were tested. Wear test (0.6 m/s sliding speed, 52 N load, 500 m distance, counter: 16MnCr5) was applied to some selected samples, the boride layer was examined by SEM, and elemental analysis was performed by EDX. Phases in the boride layer were detected by XRD. The hardness of the layer was measured with a microvickers test machine. The study showed that many different activators can be used in the borosintering process and that activators have a significant effect on the specifications of the boride layer. It was concluded that the NaF activator accelerates the formation of the boride layer more than other activators and creates a boride layer with better properties.**

***Index Terms*—Boriding, Borosintering, Powder Metallurgy, Sintering**

I. INTRODUCTION

INCREASING hardness is a common way to achieve better wear resistance but leads to loss of impact strength and can be very costly. Since wear is a surface phenomenon, the use of hard coatings can be an effective solution that allows the substrate to maintain impact resistance, and the coating can be manufactured to withstand wear, corrosion, scratching and thermal loads [1,2]. It also reduces costs by using less amount of hard material [3].

Carburizing, nitriding and boriding are often used to create hard coatings on ferrous materials by a thermo-reactive process (TRD). In this method, carbon, nitrogen, boron or their combinations are dispersed into the substrate material, forming a surface layer of carbides, nitrides or borides along with alloying elements in the material. These layers are metallurgically bonded to the substrate material and are very dense [4].

Boriding is a surface treatment that provides high surface hardness (up to 2000 HV) and low coefficient of friction [2]. The boriding process is generally carried out between 800 and 1050 °C for periods ranging from 0.5 to 10 hours. Among boriding methods, package boriding is the most widely used in the industry due to its simplicity and cost-effectiveness [5]. In parts produced by the Powder Metallurgy (P/M) method, boriding is generally applied as a separate process after sintering [6-11]. If the pack boriding process is carried out during the sintering of the powder compact, sintering and boriding can be integrated; This not only prevents parts from deforming due to secondary heating, but can also reduce energy consumption. The process of boriding the parts produced by the P/M method at the sintering stage is called Borosintering (B/S) [12,13].

In the study conducted by Dong et al [14], borosintering process was applied to 2% Cu and 0.4% C iron-based samples by weight. The processes were carried out in an atmosphere of 97% N_2 and 3% H_2 at a temperature range of 1050 - 1150 °C for a holding period of 4 hours. The thickness of the borided layers varied between 63 and 208 µm depending on the processing temperature. The presence of Fe_2B and FeB phases in the structures of borided layers was confirmed by X-ray diffraction analysis. It has been stated that the microhardness values of borided layers vary between 1360 and 2066 $HV_{0.3}$ and are much higher than the hardness of the substrate material (approximately 186 $HV_{0.3}$). According to the wear test results, it was determined that the wear resistance of borided and sintered samples improved significantly with increasing surface hardness.

The sintering and borosintering characteristics of AISI 1010 (Fe–0.6Mn–0.4Si–0.1C) steel were investigated by Erdoğan et al [13]. The sintering procedure was carried out for two hours at 1000°C in an Al_2O_3 powder environment, and the borosintering procedure was carried out for two, four, and six hours at 1000 °C in an Ekabor 2 powder environment. In the borosintered samples, a 120–400 µm thick FeB+Fe_2B layer was found, depending on the temperature and sintering period. Hardness values for sintered samples ranged from 205–215 HV, while borosintered samples had values between 1252–1705 HV.

It has been shown that wear resistance diminishes but hardness increases with increasing borosintering time. Iron-based material was borosintered by Fang et al. [12] for three, five, and ten hours at 850°C, 950°C, and 1050 °C. It has been shown that the boride layer is mostly composed of a single phase that contains Fe_2B. It is uniformly thick and has a strong bond with the substrate.

It has been reported that the layer's hardness and thickness progressively increase with temperature and time. It has been noted that an excessively thin or thick layer will result in a decrease in the bonding force between

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the substrate material and the boride layer. It was reported that boriding at 850°C and 950°C produced a single-phase Fe_2B layer, and at 1050°C, a double-layer structure with FeB and Fe_2B phases was created. Friction and wear testing have indicated that the boride layer has good wear resistance. It has been noted that corrosion is brought on by microcracks in boronized samples due to plastic deformation brought on by fatigue, and by significant oxidation and plastic deformation in non-borided materials.

Koç [15] investigated the AISI 1010 (Fe–0.6Mn–0.4Si–0.1C) steel's sintering and borosintering characteristics. He claimed to have used silicon powder as the sintering medium and EKabor 2 powder as the boronizing medium when pressing metal powders using the cold pressing method at 1000 Psi pressure. The coating thickness and microhardness of the borosintering samples were tested at 120-150 µm and 1300-1500 HV, respectively. The research currently in publication has noted that when boriding and sintering are done in one step, the mechanical and wear properties benefit from their separate execution.

The borosintering of Fe-1.75Ni-1.5Cu-0.5Mo steel, which is widely used in automotive parts, was studied by Turgut and Günen [16]. The study contrasted the processes of sintering and borosintering. An environment including 90% B_4C + 10% $NaBF_4$ powder mixture was used for the boriding process, whereas pure argon was used for the sintering phase. In the borosintered samples, a 160–325 µm-thick FeB+Fe_2B layer was found. In the samples sintered under argon atmosphere, the hardness value was measured as 1405-1688 HV, the elasticity modulus as 122.21-162.42 GPa, and the hardness value as 215-250 HV with the elasticity modulus as 63.28-94.86 GPa. Furthermore, it has been reported that when compared to traditional sintering, borosintered samples exhibit better corrosion resistance.

It has been observed that none of the studies in the literature have examined the effect of different boronizing environments on the borosintering process. In this study, environments containing different weight ratios of silicon carbide, boron carbide and the remainder one of various activators in the boronizing medium were tested.

II. METHOD

The iron-based powder mixture prepared by mechanical alloying (0.5% graphite, 0.2% silicon, 0.6% manganese, 0.6% zinc stearate and the remainder iron powder) was pressed at 750 MPa pressure. The pressed samples were subjected to borosintering process in a chamber type furnace at 1050 °C for 2 hours. The boronizing medium contains silicon carbide (75%, 85%, 90%), boron carbide (5%, 10%, 20%) in different proportions by weight and the rest contains one of various activators (Na_2CO_3, NaCl, $Na_2B_4O_7$ (anhydrous borax) and NaF) have been tested. For metallographic examinations, ultrasonic cleaning with acetone, cutting, bakelite mounting, sanding and polishing were applied to the samples. In order to better distinguish the boride layers in the polished samples, the surfaces were etched with 2% nital solution for 10 seconds. The etched surfaces were examined with an optical microscope. Wear test (0.6 m/s sliding speed, 52 N load, 500 m distance, counter: 16MnCr5) was applied to some selected samples, the boride layer was examined by SEM, and elemental analysis was performed by EDX. Phases in the boride layer were detected by XRD. The hardness of the layer was measured with a microvickers test machine.

III. RESULT AND DISCUSSION

Optical microscope images of the samples subjected to the borosintering process are given in Fig. 1-7. When the microstructures of borosintered materials were examined, it was seen that when NaF was used as an activator, a more uniform and thicker boride layer was formed compared to when other activators were used.

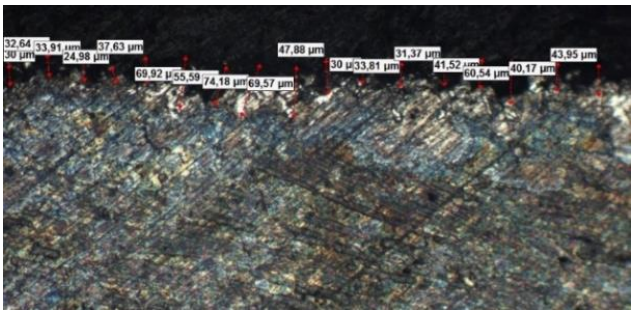


Fig. 1. Optical Image (boriding media: %5 Na_2CO_3 + %20 B_4C + %75 SiC).

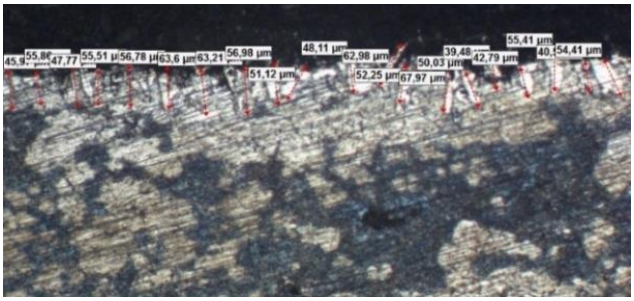


Fig. 2. Optical Image (boriding media: %5 $Na_2B_4O_7$ + %20 B_4C + %75 SiC).

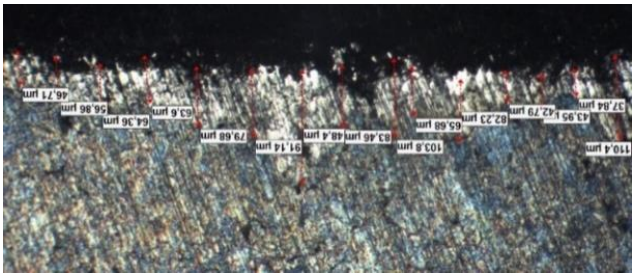


Fig. 3. Optical Image (boriding media: %5 NaCl + %20 B_4C + %75 SiC).

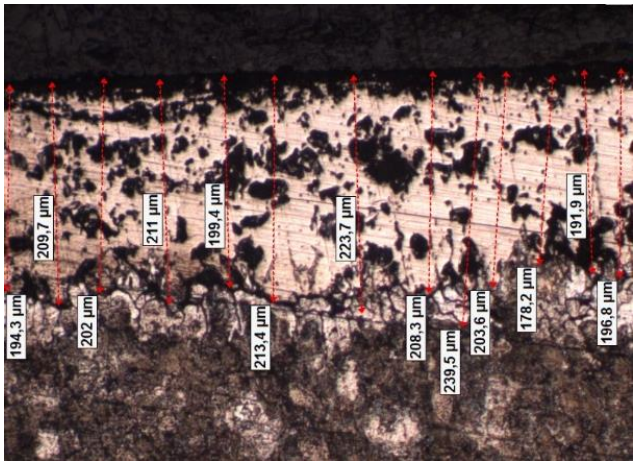


Fig. 4. Optical Image (boriding media: %5 NaF + %20 B_4C + %75 SiC).

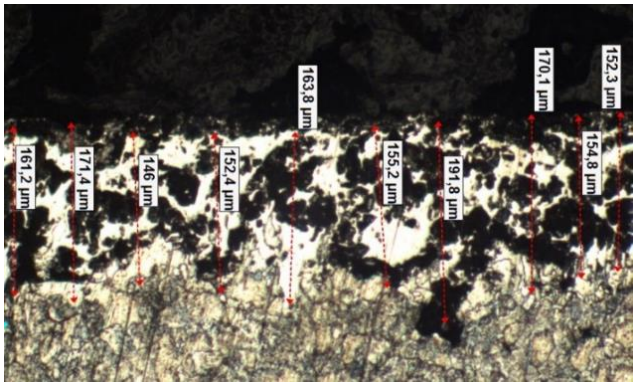


Fig. 5. Optical Image (boriding media: %5 NaF + %10 B_4C + %85 SiC).

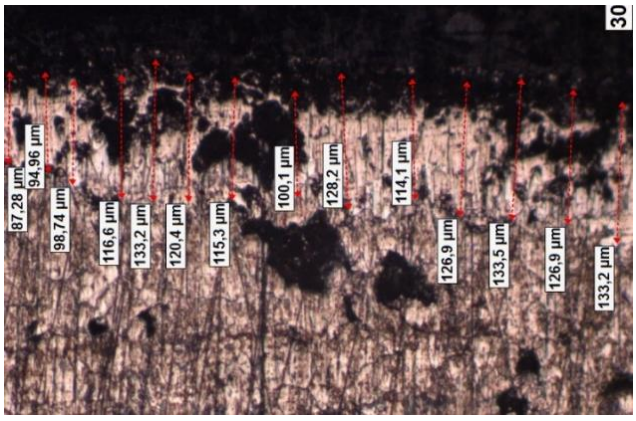


Fig. 6. Optical Image (boriding media: %5 NaF + %5 B_4C + %90).

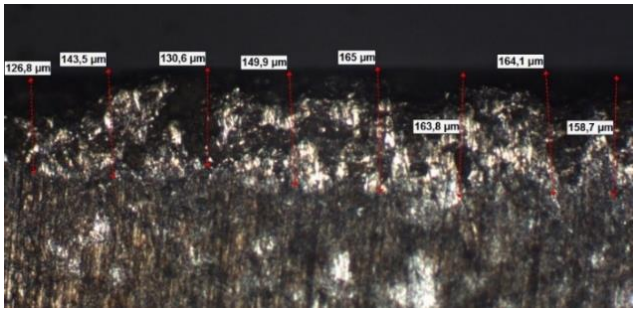


Fig. 7. Optical Image (boriding media: %10 NaF + %10 B_4C + %90 SiC).

Mean of thickness of boride layer are given in Table I and Fig. 8. When the environments with the same B_4C and SiC ratio (20% B_4C + 75% SiC) are examined, it is seen that the NaF activator accelerates the formation of the boride layer more than other activators and creates a thicker layer. When media with the same activator amount (containing 5% NaF) are examined, it is concluded that the boride layer thickness increases with the increase in the amount of B_4C. It was determined that the lowest boride layer thickness was 45 µm in an environment containing 5% Na_2CO_3 + 20% B_4C + 75% SiC by weight, and the highest boride layer thickness was 206 µm in an environment containing 5% NaF + 20% B_4C + 75% SiC.

TABLE I. MEAN TICKNESS OF BORIDE LAYER RESULTING FROM BORIDING IN DIFFERENT BORONIZING ENVIRONMENTS

Boriding media (by weight)	Average boride layer (µm)
%5 Na_2CO_3 + %20 B_4C + %75 SiC	45
%5 $Na_2B_4O_7$ + %20 B_4C + %75 SiC	54
%5 NaCl + %20 B_4C + %75 SiC	68
%5 NaF + %20 B_4C + %75 SiC	206
%5 NaF + %10 B_4C + %85 SiC	162
%5 NaF + %5 B_4C + %90 SiC	116
%10 NaF + %10 B_4C + %80 SiC	150

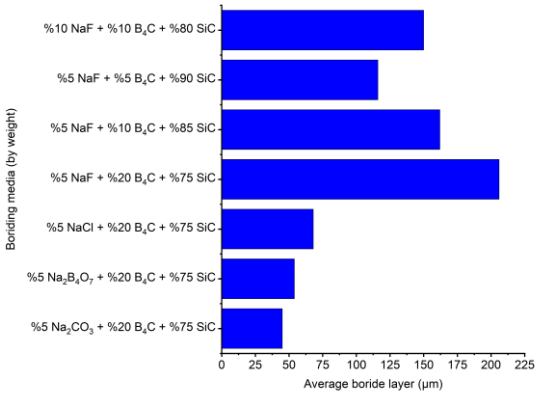
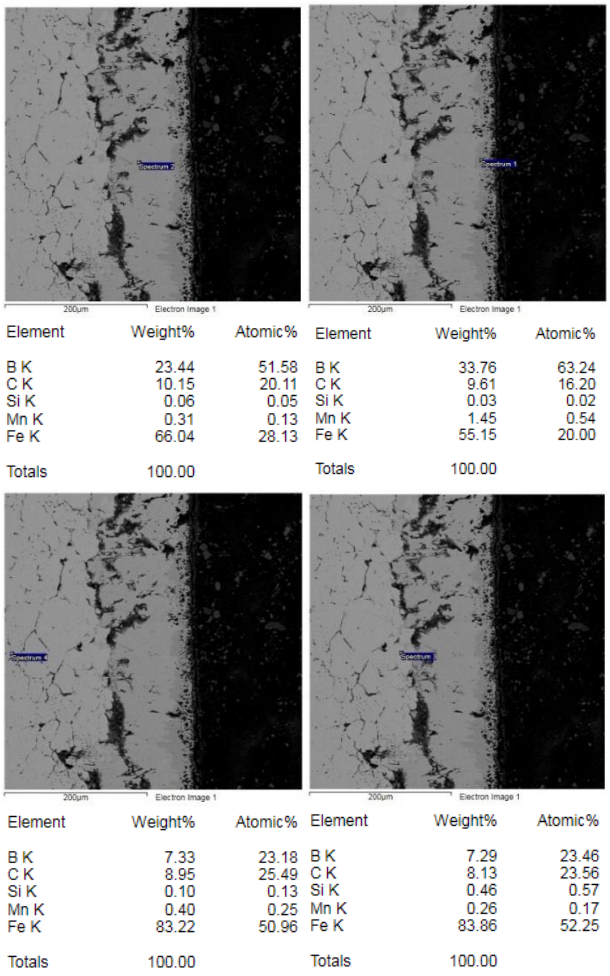


Fig. 8. Mean tickness of boride layer resulting from boriding in different boronizing environments.

The amount of wear was measured 19.8 mg in the sample borosintering in 5% Na_2CO_3 as activator, 19.2 mg in 5% NaCl, 18.8 mg in 5% $Na_2B_4O_7$, and 1.1 mg in 5% NaF (%20 B_4C + %75 SiC).

SEM images and EDX analyzes of the sample borosintered in 5% NaF + 20% B4C + 75% SiC environment are given in Fig.9. It has been observed that the amount of boron decreases atomically as the distance from the coating, while the amount of iron increases. Looking at the color differences, it can be said that there is a FeB phase on the outer part of the boride layer, but the structure is largely composed of the Fe_2B phase. It is thought that the black spots in the coating are voids that appear during production or as a result of metallographic processes. Due to the use of SiC-based sandpaper, silicon and carbon were observed as a result of the analysis. In addition, because the atomic numbers of C and B are close, some of B may appear as C. Silicon and manganese are seen in very small amounts because they are used as sintering aids. The average microhardness values of the sample were measured as 1518 $HV_{0.1}$ in the boride layer and 106.55 $HV_{0.1}$ in the

substrate material region. The XRD spectrum of the sample is given in Fig.10. It has been observed that the boride layer consists largely of the Fe_2B phase.



Element	Weight%	Atomic%
B K	23.44	51.58
C K	10.15	20.11
Si K	0.06	0.05
Mn K	0.31	0.13
Fe K	66.04	28.13
Totals	100.00	

Element	Weight%	Atomic%
B K	33.76	63.24
C K	9.61	16.20
Si K	0.03	0.02
Mn K	1.45	0.54
Fe K	55.15	20.00
Totals	100.00	

Element	Weight%	Atomic%
B K	7.33	23.18
C K	8.95	25.49
Si K	0.10	0.13
Mn K	0.40	0.25
Fe K	83.22	50.96
Totals	100.00	

Element	Weight%	Atomic%
B K	7.29	23.46
C K	8.13	23.56
Si K	0.46	0.57
Mn K	0.26	0.17
Fe K	83.86	52.25
Totals	100.00	

Fig. 9. SEM images ve EDX analyses.

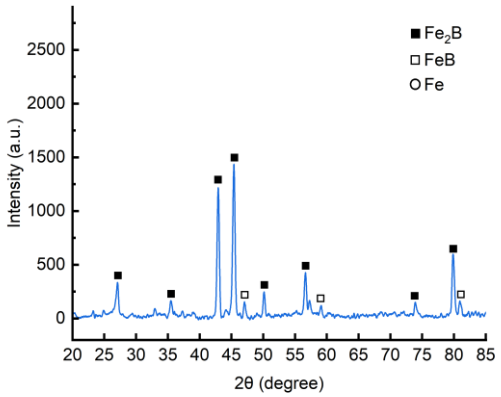


Fig. 10. XRD spectrum.

IV. CONCLUSION

The study showed that many different activators can be used in the borosintering process and that activators have a significant effect on the boride layer thickness. It has been observed that hard, wear-resistant structures are formed as a result of borosintering. It was concluded that the NaF activator accelerated the formation of the boride layer more than other activators and formed a boride layer with better properties. It was observed that as the amount of B_4C increased, the boride layer thickness increased. In wear tests, it is obvious that the sample borosintered in NaF medium is more resistant to wear. Considering the properties of the formed boride layers, it can easily be said that the boriding media mentioned in this study can be used instead of commercial boriding powders.

When we examine the literature, the borosintering process and the traditional boronizing process (sintering and then boriding) are similar in many features. In both cases, boride layers are formed and thus many properties of the material are improved. The fact that there are fewer process steps in borosintering and that there is no need for an additional protective atmosphere such as Argon gas in iron-based materials reduces the cost considerably. For this reason, choosing the borosintering process in materials to be produced with powder metallurgy will provide a serious advantage.

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