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Investigation and Optimization of The Effect of Anhydrous Borax Mineral on The Vickers Hardness and Indentation Modulus Values of Iron Material

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ABSTRACT

In this study, 5% and 10% by weight of anhydrous borax (AHB) was added to the iron (Fe) matrix material by powder metallurgy method and the effects of the additive ratio on the Vickers hardness (HV), Brinell hardness (HB) and Indentation modulus (E_{IT}) values of the composites (Fe/AHB) were investigated. In the productions carried out using Taguchi experimental design method, AHB additive ratio, and sintering temperature parameters were selected as control parameters that were thought to affect the physical and/or mechanical properties of the Fe/AHB composite materials. The productions were carried out according to the Taguchi L4 orthogonal array, which was created depending on the control parameters and levels. Vickers hardness and indentation modulus measurements of pure iron and Fe/AHB composite materials were performed in accordance with BS EN ISO 14577-1 standard and Brinell hardness measurement was performed in accordance with TS EN ISO 6506-1 standard. According to the signal-to-noise ratio (S/N) analysis performed with the experimental data, it was determined that the 10% AHB additive ratio and 950°C sintering temperature optimized all the investigated properties of the Fe/AHB composite material. It was determined that the values for Vickers hardness, Brinell hardness and indentation modulus increased by 142.03%, 69.32% and 144.11%, respectively, in the levels where the properties of the composite material were optimized compared to pure Fe material. As a result of the qualitative examination of all samples after storage in a comfortable environment without daylight, it was also observed that the anhydrous borax additive delayed the corrosion time of pure iron material.

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1. Introduction

Composites are a group of advanced technological materials formed by the combination of at least two different materials, where the components called matrix and reinforcement come together by creating an interface and behave as a single material. As technology has evolved, the properties expected from materials have changed and the increasing demand for lightweight materials with high chemical and mechanical strength in many sectors has increased the interest in composites [1,2].

Metal matrix composites (MMCs) with continuous fibers and whiskers as reinforcement were developed in the 1970s and proposed for applications requiring high performance. [3]. These materials have attracted great interest from the industry due to their unique mechanical and structural properties [3,4].

The improved mechanical strength, wear resistance, hardness, stiffness, damping capacity, thermal stability, ductility of metal matrix composites have attracted attention for a wide range of applications [3,5]. MMCs are ideally suited for use in ground transportation and aviation in the reduction of structural weight and related fuel consumption [5]. When the reinforcement materials preferred in the production of metal matrix composites, which can be produced by different techniques [7] such as powder metallurgy, spray deposition technique, and stir casting, are examined; silicon carbide (SiC) with high hardness, wear resistance, toughness, fatigue resistance properties and boron carbide (B_4C) with high melting point, low density, great resistance to chemical substances are frequently preferred materials [5]. Boron carbide (B_4C), an important reinforcement material, is the hardest known material after diamond and cubic boron nitride (cBN) [6]. Since borides have higher hardness and thermal stability compared to carbides, borides have the potential to be a wear-resistant phase [8]. It is also known that B increases the hardenability of the iron matrix [9].

When the materials used in the production sector do not perform as expected, situations such as production inefficiencies, quality problems and consequently customer dissatisfaction inevitably arise. In this context, there are different methods developed to improve both expected material properties and product quality. The Taguchi method is one of the methods developed to obtain the most information with the least amount of time, cost, and energy by designing experimental studies. It has proven to be an effective tool to create optimal production conditions in a wide range of production environments, especially in achieving more efficient results with reduced trials [10,14].

In this study, a metal matrix composite was produced with iron (Fe) as the matrix material and anhydrous borax (AHB), a type of boron mineral, as an additive.

By evaluating the effects of anhydrous borax and/or anhydrous borax additive ratio and sintering temperature factors on the mechanical properties of the final product (composite material), it is aimed to determine the most effective additive ratio and sintering temperature to increase Brinell hardness, Vickers hardness and indentation modulus values and to develop Fe/AHB composite material. The experimental study was carried out by Taguchi experimental design method and Fe/AHB composites were produced by powder metallurgy method according to the obtained Taguchi orthogonal array.

2. Material And Methods

2.1. Iron (Fe)

Pure iron with a density of 7.87 g/cm^3, a melting temperature of 1535°C and a grain size of 3 µm was used as matrix material for the production of composite materials

2.2. Anhydrous borax

Anhydrous borax mineral with the trade name Etibor-68 purchased from Eti Maden Operations, with a density of 2.37 g/cm^3 and a melting temperature of 742.5°C, was ground and used under a sieve size of 20 µm as an additive material for the production of composite materials.

2.3. Implementation of Taguchi Method

Taguchi experimental design method was used to optimize the Brinell hardness, Vickers hardness and Indentation modulus values of the composites produced and to determine the optimum levels of the factors. In addition to the control factor of sintering temperature, which is thought to affect the quality and mechanical properties of the composite products by powder metallurgy method, the AHB additive ratio was also specified as a control factor in order to investigate the effect of different weight percentages of AHB additive. (see Table 1).

Table 1. *Control factors and their levels*

Factors	Level 1	Level 2
AHB additive ratio (wt. %)	5	10
Sintering temperature (°C)	850	950

Depending on the factors and their levels, Taguchi L4 orthogonal array was selected (see Table 2) and the productions were carried out in accordance with the orthogonal array. At least 3 specimens were produced for each test set-up and Brinell hardness, Vickers hardness and indentation modulus values were determined by taking 5 different measurements on the specimens.

Table 2. *Experimental conditions for L4 orthogonal array*

Trials	AHB additive ratio (wt. %)	Sintering temperature (°C)
1	5	850
2	5	950
3	10	850
4	10	950

Using the experimental data obtained after the measurements of Brinell hardness, Vickers hardness and indentation modulus values, signal-to-noise ratios (*S/N*) were examined by using the Minitab program, in accordance with the "larger is better" method (see Equation 1) to determine the parameters that optimize these values.

$$\mathrm{S/N} = -10 \cdot \log\left(\frac{1}{\mathrm{n}}\sum_{i=1}^{n}\frac{1}{y_i^2}\right) \quad (1)$$

2.4. Composite material production

In the preliminary preparation stage before production, AHB was first subjected to grinding in a RETSCH brand SK100 model device to reduce the grain size and then to sieving in a RETSCH brand AS200 model device to separate the particles according to grain size. In order to obtain a homogeneous mixture, Fe/AHB composites with different weight percentages were prepared in a glass jar with a nickel ball inside using a MSE-TEC brand ball mill machine at a grinding speed of 200 rpm and a grinding time of 30 minutes. Then, Fe/AHB composite materials were obtained (see Figure 1) in a DIEX model induction furnace, in a graphite mold with an inner diameter of 20 mm, at a constant heating rate of 45ºC/min to the sintering temperature, under a constant 15 min holding time at sintering temperature, and at different sintering temperatures (850ºC – 950ºC). Since the sintering temperature was higher than the melting temperature of AHB used as an additive, AHB liquefied in the mold and caused fractures in the mold when pressure was applied. For this reason, the productions were carried out under a constant pressure of 2 MPa to prevent damage to the mold.

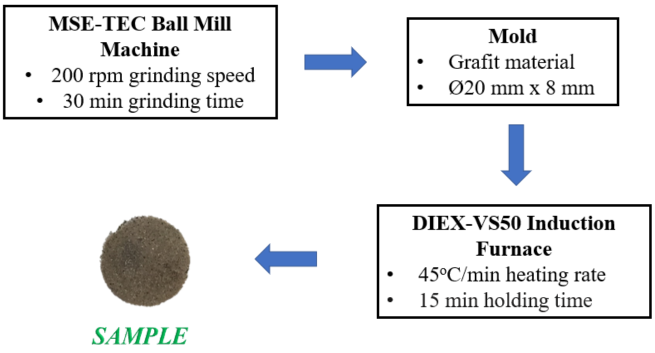

Figure 1. *Sample production steps*

2.5. Brinell hardness, Vickers hardness and Indentation modulus measurement

Vickers hardness and indentation modulus measurements were carried out under 1 kg load according to BS EN ISO 14577-1 standard on a Zwick BZ2.5/TS1S instrumented hardness tester at Tübitak National Metrology Institute. Brinell hardness measurement was carried out under 31.25 kg load according to TS EN ISO 6506-1 standard in a WOLPERT brand hardness tester in Kocaeli University Mechanical Engineering Department.

3. Results and Discussion

It has been determined in previous studies that the strength and stiffness of composite materials can be enhanced by the presence of intermetallic phases and compounds [15]. Due to the structure of MMCs, the interface between the matrix and the additive is important for increasing the mechanical properties. Because in the composite material exposed to load, it is desired to form a structure in such a way that the relevant load is transferred from the matrix to the additive through the interface [11].

3.1. Brinell hardness value

The Brinell hardness values of the materials are comparatively graphed in Figure 2, which was prepared using the experimental data obtained. When Figure 2 is examined, it is determined that the hardness value of the composite material produced at 850ºC sintering temperature with 5 wt% AHB additive increased by 25.40% compared to the reference pure Fe material produced at 850ºC sintering temperature. It was also found that the hardness value of the composite material produced at 950ºC sintering temperature with 10% AHB additive increased by 69.32% compared to the reference pure Fe material.

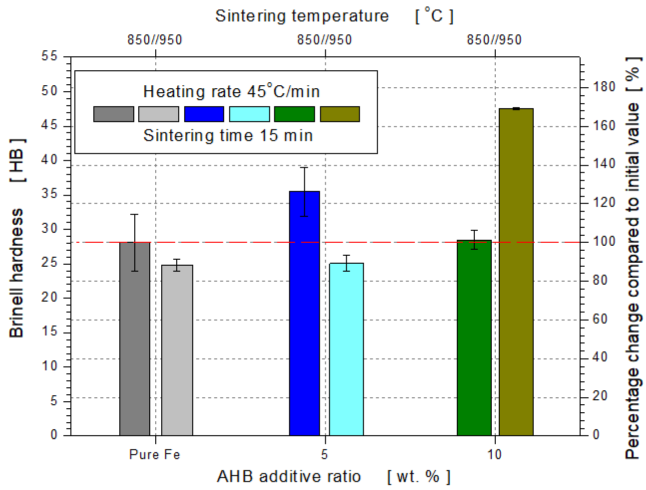

Figure 2. *Graph of the change in Brinell hardness values*

When the *S/N* ratio table (see Table 3) and graph (see Figure 3) are examined, it was determined that the most effective factor on Brinell hardness is the AHB additive ratio, which is the factor with the largest delta value. It was determined that the factor levels that optimized (maximized) the Brinell hardness value were 10% AHB additive ratio and 950ºC sintering temperature.

Table 3. *S/N ratio response table of Brinell hardness values of composites*

Level	AHB additive ratio	Sintering temperature
1	29.27	29.74
2	**31.08**	**30.61**
Delta	1.82	0.88
Rank	1	2

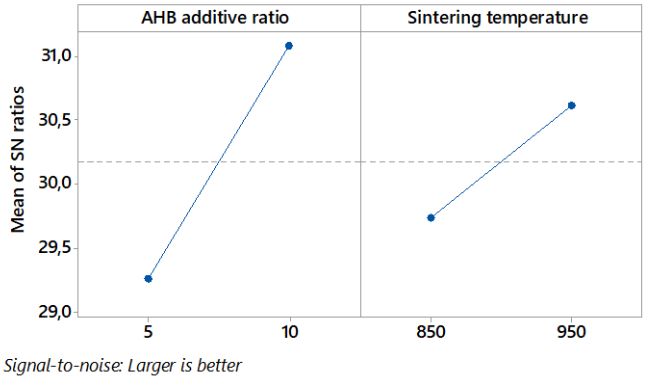

Figure 3. *Main effects plot of S/N ratios of Brinell hardness values of composites*

3.2. Vickers hardness value

The Vickers hardness values of the materials are comparatively graphed in Figure 4, which was prepared using the experimental data obtained. When Figure 4 is examined, it is determined that the hardness value of the composite material produced at 850ºC sintering temperature with 5 wt% AHB additive increased by 36% compared to the reference pure Fe material produced at 850ºC sintering temperature. It was also found that the hardness value of the composite material produced at 950ºC sintering temperature with 10% AHB additive increased by 142.03% compared to the reference pure Fe material.

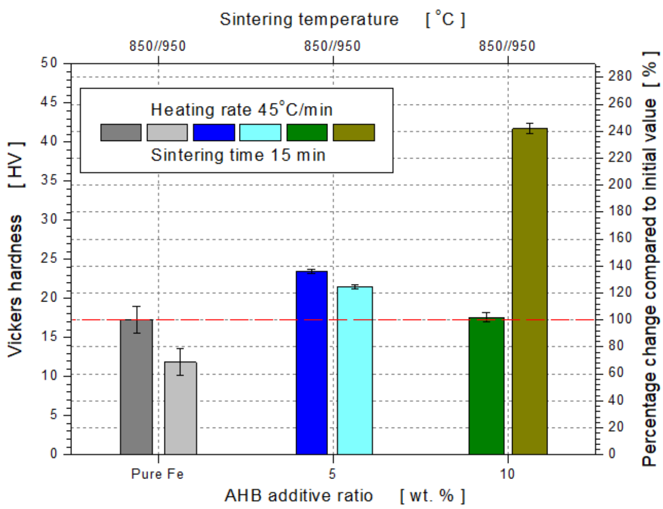

Figure 4. *Graph of the change in Vickers hardness values*

When the *S/N* ratio table (see Table 4) and graph (see Figure 3) are examined, it was determined that the most effective factor on Vickers hardness is the sintering temperature, which is the factor with the largest delta value. It was determined that the factor levels that optimized (maximized) the Vickers hardness value were 10% AHB additive ratio and 950ºC sintering temperature.

Table 4. *S/N ratio response table of Vickers hardness values of composites*

Level	AHB additive ratio	Sintering temperature
1	27.10	26.35
2	**29.07**	**29.81**
Delta	1.97	3.46
Rank	2	1

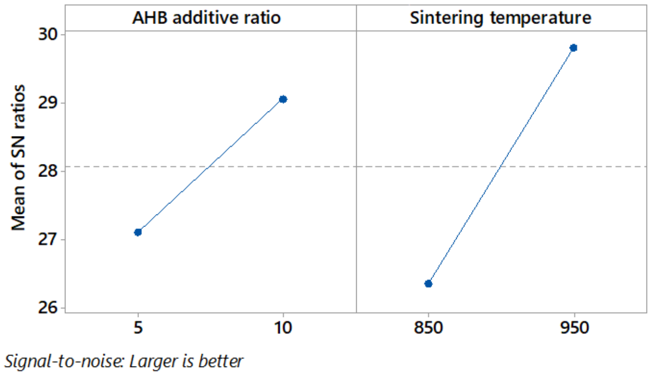

Figure 5. *Main effects plot of S/N ratios of Vickers hardness values of composites*

Because of the boron's low solubility in the iron lattice (approximately 500 parts per million), only minimal quantities are necessary to generate significant volumes of hard phases [12,13]. Additionally, boron enhances the hardenability of the iron matrix [16]. A review of past studies also shows that Fehmi and Mustafa's study [11] has similar results that boride phases formed in the inner structure considerably increase the hardness of iron-based composites.

3.3. Indentation modulus value

The Indentation modulus values of the materials are comparatively graphed in Figure 6, which was prepared using the experimental data obtained. When Figure 6 is examined, it is determined that the indentation modulus value of the composite material produced at 850ºC sintering temperature with 5 wt% AHB additive increased by 43.84% compared to the reference pure Fe material produced at 850ºC sintering temperature. It was also found that the modulus value of the composite material produced at 950ºC sintering temperature with 10% AHB additive increased by 144.11% compared to the reference pure Fe material.

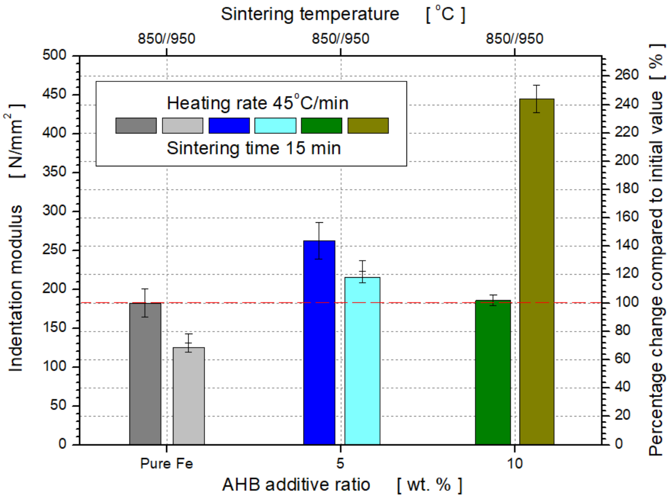

Figure 6. *Graph of the change in Indentation modulus values*

When the *S/N* ratio table (see Table 5) and graph (see Figure 7) are examined, it was determined that the most effective factor on Indentation modulus is the sintering temperature, which is the factor with the largest delta value. It was determined that the factor levels that optimized (maximized) the Indentation modulus value were 10% AHB additive ratio and 950°C sintering temperature.

Table 5. *S/N ratio response table of Indentation modulus values of composites*

Level	AHB additive ratio	Sintering temperature
1	47.47	46.94
2	**49.53**	**50.05**
Delta	2.06	3.11
Rank	2	1

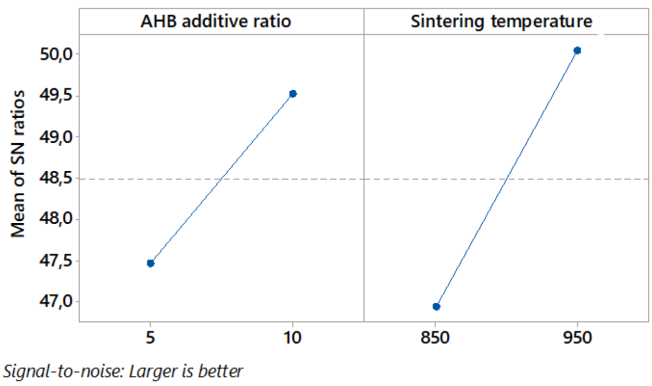

Figure 7. *Main effects plot of S/N ratios of Indentation modulus values of composites*

Calculations indicates that as the boron content increases, there is a heightened hybridization between B 2p and Fe 3d. Consequently, this strengthens the Fe-B bond, elevating both the elastic modulus and thermodynamic stability [12,17]. The rise in covalent bonding corresponds to a concurrent increase in hardness [12]. In their study also Lentz et al [12] also determined that; because of the strengthening of covalent bonding, the indentation hardness and indentation modulus of the Fe_3C phase can be improved significantly by increasing the B content in the $Fe_3(C,B)$ phase.

3.4. Qualitative examination

High wear and corrosion resistance is important to prevent a reduction in the mechanical resistance of equipment and economic loss of mechanical parts. As a result of the qualitative examination of all samples after storage during 12 months in a comfortable environment without daylight, it was observed that the anhydrous borax additive delayed the corrosion time of pure iron material (see Figure 8 and 9) Boron diffusion in the matrix controls the oxidation of Fe_2B boride [13]. Zheng Lva et al [13] also obtained a similar result that increasing boron concentration increases the oxidation resistance of pure iron.

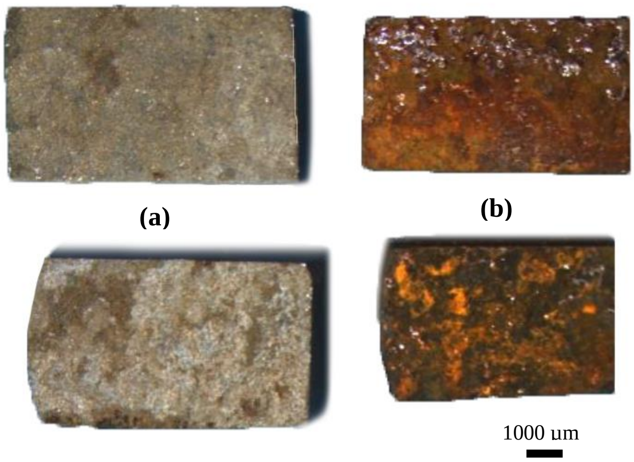

Figure 8. *Macro image of produced at 850°C sintering temperature (a) Fe/AHB composite with 5% AHB additive and (b) pure Fe materials*

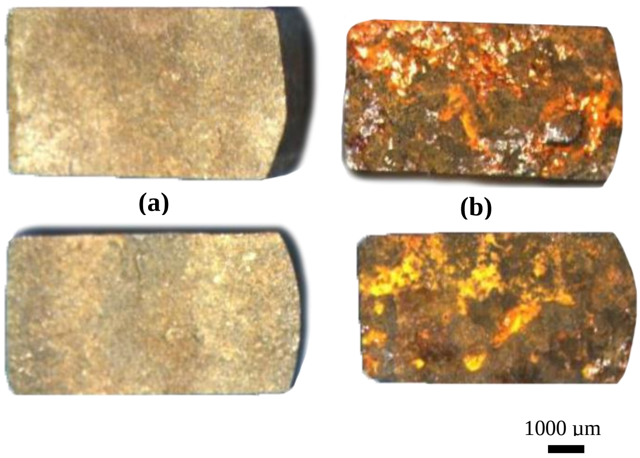

Figure 9. *Macro image of produced at 950ºC sintering temperature (a) Fe/AHB composite with 10% AHB additive and (b) pure Fe materials*

4. Conclusion

As a result of selected and/or preferred 2 control factors and their 2 levels as well as constant heating rate and constant sintering time parameters; Brinell hardness, Vickers hardness and indentation modulus were strongly affected by the AHB additive.

Comparing the pure Fe reference sample (850ºC) with 10% AHB added Fe composite sample prepared at 950ºC sintering temperature, the Fe/AHB composite sample with 10 wt.% additive showed the best results with 69.32%, 142.03% and 144.11% increase in Brinell hardness, Vickers hardness and indentation modulus, respectively.

The factors that optimize the Brinell hardness, Vickers hardness and indentation modulus together are the ratio of 10% SB additive and the sintering temperature of 950ºC.

As a result of the qualitative examination of all samples after storage during 12 months in a comfortable environment without daylight, it was observed that the anhydrous borax additive delayed the corrosion time of pure iron material.

In the future studies on the related subject, it is recommended to examine the effect of different levels of control factors and different constant heating rate, and constant sintering time parameters in addition to decreasing/increasing the particle size and/or additive ratios of anhydrous borax.

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