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MORPHOLOGICAL BEHAVIOR OF FEB AND FE_2B IN BORIDE LAYER OF 304 STAINLESS STEEL UNDER DIFFERENT MEDIUM

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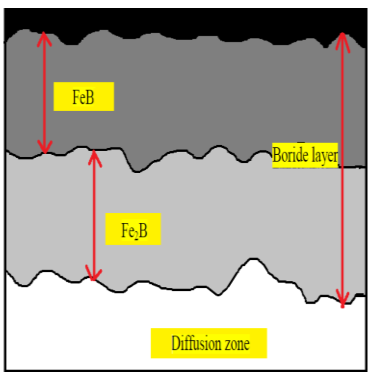
Graphical abstract



Abstract

This study investigates the morphological behavior of FeB and Fe_2B phase in boronized 304 stainless steel under two different mediums which is powder and paste. Surface attrition was implemented in order to initiate better boron diffusivity. The comparison of microstructure in term of grain size and boride layer thickness were also analyzed. Scanning electron microscopy, X-Ray diffraction analysis and energy dispersive X-Ray Analysis were conducted to prove the existence of FeB and Fe_2B phases. The relationship between the phase and Vickers microhardness was also investigated. The results show formation of flat-toothed boride layer in both sample with different FeB and Fe_2B layer uniformity. Paste boronized sample with120 µm boride layer thickness had outperformed the powder boronized sample that with 43 µm boride layer thickness, thus providing significant improvement in microhardness values from 1500 Hv to 1800 Hv. In conclusion, paste medium provide outstanding boride layer thickness with 300% enhancement and excellent microhardness with 20% improvement. The results of this findings could offer a new insight in pack boronizing of stainless steel that can be complicated to boronized due to its high alloying element content.

Keywords: FeB, Fe_2B, 304 stainless steel, surface attrition, morphological behavior

Abstrak

Kajian ini menyiasat kelakuan morfologi bagi fasa FeB dan Fe_2B dalam keluli tahan karat 304 boron di bawah dua medium yang berbeza iaitu serbuk dan adunan. Pergeseran permukaan dilaksanakan untuk memulakan kemeresapan boron yang lebih baik. Perbandingan mikrostruktur dari segi saiz bijian dan ketebalan lapisan borida juga dianalisa. Imbasan mikroskop elektron, analisa belauan sinar-X dan analisa serakan tenaga sinar-X dilakukan untuk membuktikan kewujudan fasa FeB dan Fe_2B. Hubungan antara fasa dan mikrokekerasan Vickers juga disiasat. Hasil kajian menunjukkan pembentukan lapisan borida bergigi rata pada kedua-dua sample dengan keseragaman lapisan FeB dan Fe_2B yang berbeza. Sampel adunan boron dengan 120 µm ketebalan lapisan borida telah mengungguli sampel serbuk boron dengan 43 µm ketebalan lapisan borida, sehingga memberikan peningkatan yang ketara pada nilai mikrokekerasan dari 1500 Hv hingga 1800 Hv. Kesimpulannya, medium adunan memberikan ketebalan lapisan borida dengan peningkatan 300% dan mikrokekerasan yang sangat baik dengan peningkatan 20%. Hasil penemuan ini dapat memberikan gambaran baru mengenai proses boronisasi keluli tahan karat yang agak rumit kerana kandungan unsur mengaloi yang tinggi.

Kata kunci: FeB, Fe_2B, Keluli tahan karat 304, pergeseran permukaan, kelakuan morfologi

1.0 INTRODUCTION

Formation of FeB and Fe_2B phases in boride layer of boronized steels play an important role in determining the constituent properties of the material. Boride layer which acts as a protective layer on the surface of heat-treated steel often characterized by the layer thickness and the formation of whether single/mono of Fe_2B or dual/double layer of FeB and Fe_2B phases. Thicker boride layer will offer better surface protection especially in term of oxidation and wear resistances, as well as hardness of the boronized surface.

Past studies indicated that formation of boride layer with thickness in between 50 – 100 µm provide the most prominent improvement of more than twice the hardness and wear properties of unalloyed or low alloy steel [1-2]. With proper selection of temperature, time and also boronizing medium, this improvement can be achieved successfully in low alloy steels.

On the other hand, the presence of chromium and other transition elements such as molybdenum and tungsten in low steels offer extensive improvement in microhardness and hardness of stainless steel. This is due to the fact that the addition of these alloying elements helps stabilized the formation of FeB and Fe_2B phases as well as avoiding de-boronizing which is a condition that reduce or remove boron from the surface during boronizing process [3].

In contrast with low alloy steel, the formation of boride layer in stainless steel or high alloy steel is relatively thinner which is only up to 50 µm thickness. This is generally because the presence of alloying elements such as chromium, nickel and molybdenum than hinder the boron diffusion on the surface of stainless steel and high alloy steel. These alloying elements will rise to the top of the surface when reacted to heat specifically above the eutectoid temperature, thus impeding the boron to be diffuse into the surface. The formation of coarse chromium carbide ($Cr_{23}C_6$) in stainless or cast alloy steel will induce more formation of free chromium that will diffused in the matrix of the stainless or cast alloy steel. This will be resulted in decreasing of the wear and corrosion resistances of these steels [4]. Due to this reason, surface modification was often implemented before boronizing to initiate the boron diffusion during boronizing [5].

The morphological shape of the boride layer produced is also one of the factors that affected the resultant properties of the hard surface. The boride layer formed could be either in a flat-toothed or saw-toothed morphology. Saw-toothed boride layer was defined by its high peak and narrow tips while flat-toothed layer has smoother and flat layers with more blunt tips. Saw tooth layer is said to have better boride layer adhesion for extensive surface protection due to the depth of the tips, while flat tooth boride layer exhibited lower adhesion due to slower diffusion rate. Past researcher also concluded that the formation of saw-toothed boride layers displays outstanding adherence between FeB and Fe_2B layers and also between the Fe_2B layer and the substrate/matrix which was associated with the thermal process nature [1]. On the contrary, the flat-toothed boride layer which was often found in alloyed and stainless steel has lower adherent due to higher number of alloying elements which slows the diffusivity of the boron. Increasing the amount of chromium and other alloying elements also resulted in thinner boride layer as the interface between the matrix and the boride layer will gradually flatter [6].

The depth of boride layer which can characterized the enhancement properties of boronized steels can also be determined through the length of the tooth column and the number of teeth produced on the boride layer.

Past researches conferred about the effects of treatment time on the thickness of boride layer and found that longer boronizing time resulted in favourable effect of boride layer thickness enhancement. Sufficient treatment times which are in between 2 hours to 10 hours were often applied in order to initiate the boride layer formation, in which depending on other factors such as applied temperature, boronizing medium, carrier gas and vacuum/ non- vacuum environment [8-9]. Most studies found that the effective boronizing time to produce sufficient boride layer thickness provide adequate surface protection are above 6 hours treatment times [10]. Generally, the formation of boride layer can be formed through proper treatment time which include boride intubation time and effective growth time of Fe_2B layer, as shown in Figure 1.

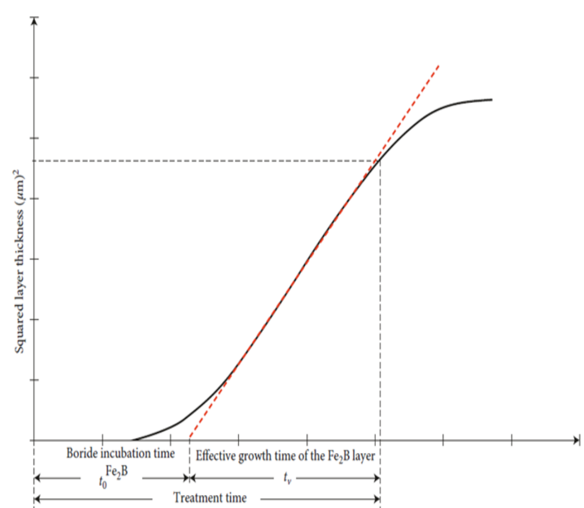


Figure 1 Boronizing treatment time [7]

Pack boronizing was known as one of the simplest and cost-effective boronizing treatment which provide significant effect on the hardness and wear resistance of plain and alloy steels [11-12]. Powder pack boronizing was conducted in a dry-dry conditions and thus has lower adhesion and diffusivity as compared to paste boronizing that was conducted in dry-wet condition. Paste medium also offer more benefits as the particle sizes are smaller, thus increasing the diffusivity of the boron into the surface [13]. Most of past studies only concentrate on the effect of powder and paste boronizing in low alloy steels as it provides prominent affect in term of boride layer thickness. Implementing these types of boronizing on high alloy steel was considered as ineffective as only thin boride layer was produced [14].

As the samples used in this study was grade 304 stainless steel which exhibit higher amount of chromium and nickel, surface attrition was applied before conducting boronizing process. As there are two types of mediums in pack boronizing, the effect of powder and paste boronizing on the morphological behaviour of FeB and Fe_2B phases shall be investigate after surface attrition process.

2.0 METHODOLOGY

The sample used in this study is grade 304 stainless steel rods with diameter of 1cm and samples were cut into the dimension of 3cm for morphology observation and analysis preparation.

Surface attrition treatment was conducted according to BS EN ISO 11124- 4:1997 standard in order to initiate surface deformation and increase the diffusivity rate using Finimac shot blasting machine. Silicon Carbide (SiC) was used as the shot with diameter of 2 mm and velocity in between 150 to 200 m/s-1, as shown in Figure 2.

Figure 2 Silicon Carbide shots

The samples were boronized at 850°C using 2 types of pack boronizing mediums which were powder and paste mediums for 8 hours holding times. Powder boronizing was conducted using EKabor powder with 100-150 µm particle sizes and paste boronizing was conducted using commercial boron paste containing the donor (5 % B_4C), diluents (90 % SiC) and activator (5% KBF_4) which was coated up to 2 mm thickness and paste size of 50 µm.

The samples were then cut, ground and polish, in accordance ASTM E3 standard. The etching process was conducted using Kalling No. 2 etchant with respect to ASTM E407 standard.

Olympus B X 41 M microscopes were used to measure the grain size of the sample using MPS Digital image analyser software in order to validate the effect of surface attrition treatment on the grain size of the treated surface. 20 readings were taken per 1 mm² area and compared to ASTM E1382-97(2010) standard.

The grain size was calculated using to the following equation:

$$n = 2^{G-1}$$

Where:
n = number of grain size per square inch at 100x magnification
G = ASTM grain size number [24]

Scanning electron microscopy (SEM) was conducted using Hitachi S-3400 Scanning electron microscope with voltage of 15kV and the energy dispersive X-Ray (EDX) spectrometry also conducted to determine the percentage of boron in each phase of the boride layer.

X-Ray diffraction (XRD) analysis was performed using Rikagu brand X-Ray diffractometer implementing CuKa radiation with 2θ angle between 30 to 120° with 0.5 step sizes. The graph of intensity (c.p.s) versus 2θ angle was then plotted and the existences of the FeB and Fe_2B phases were then validated.

The microhardness measurements on the phases produced was performed using Mitutoyo MVX-H1 Vickers microhardness tester according to ASTM E-384 standard applying 10N loads. Indentations were taken with the interval of 10 µm, from the outer boride layer until the substrate.

Figure 3 below shows the relationship between length of column and the numbers of tooth in which the calculation of boride later thickness or depth is equal to the sum of column tooth length over the numbers of tooth at predetermined length/area [7].

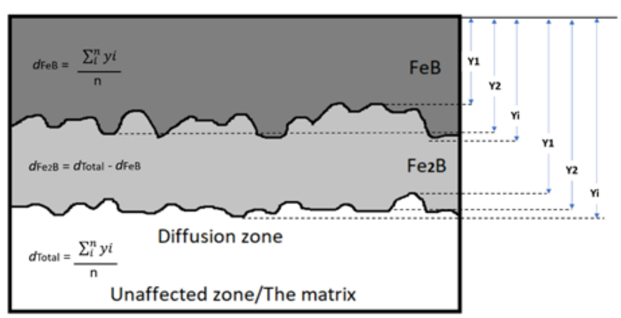


Figure 3 Calculation of boride layer thickness

3.0 RESULTS AND DISCUSSION

Figure 4 (a) shows the SEM micrograph of the cross section of 304 stainless steel after surface attrition process and Figure 4 (b) shows the deformed surface on the top area of the sample after surface attrition process. The SEM reveals that the surface attrition process successfully conducted on the surface of 304 stainless steel as a layer of plastically deformed surface with thickness of approximately 30µm was produced with numerous dented areas. It was necessary to performed surface attrition process in order to refine the grain sizes of the impacted sub-surfaces and initiating crystalline defects such as vacancies and dislocation, thus alloying better boron diffusion during boronizing [15 - 16].

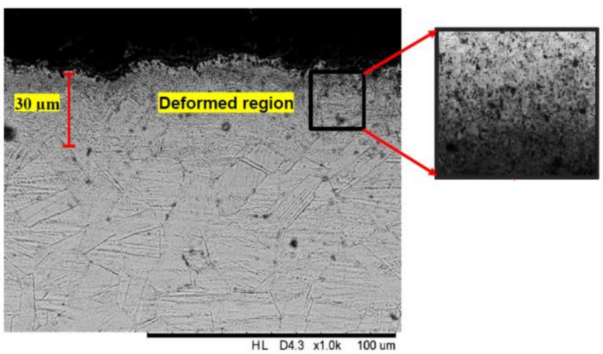


Figure 4 (a) SEM micrograph of 304 stainless steel after surface attrition

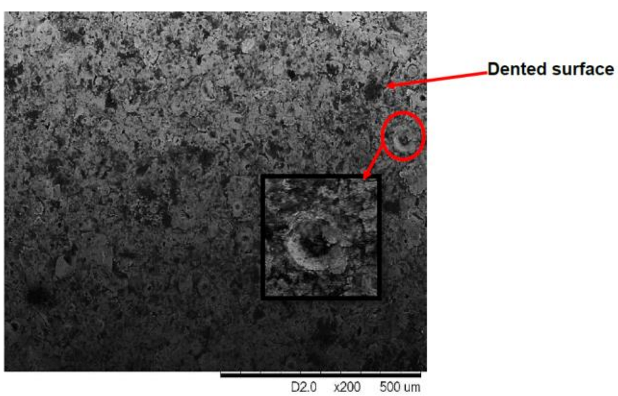


Figure 4 (b) SEM micrograph of 304 stainless steel after surface attrition

The comparison of grain sizes of 304 stainless steel before and after surface attrition was shown in Figure 5 and Figure 6 respectively and the results show that there was significant difference in the grain sizes of the sample. The results revealed that the substrate grain sizes were in accordance to ASTM Grade 3.5 (5.6 grains per inch square measurement) and with the average grain size of 95.1 µm). On the contrarily, application of surface attrition had effectively reduced the grain sizes to ASTM grade 7.5 standards (90.5 grains count per inch square measurement) with the average grain size about 23.8 µm, which is 75% smaller than non-surface treated samples. It was expected that the refinement of grain sizes on surface attrited samples would increase the boron diffusivity rate, thus alloying boron to diffuse deeper on 304 stainless steel surface which before was hindered by uprising of chromium and other alloying elements creating a hard layer on the surface of non-treated 304 stainless steel.

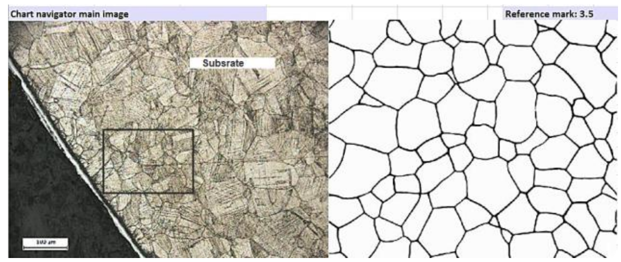


Figure 5 Grain size measurement for samples before surface attrition

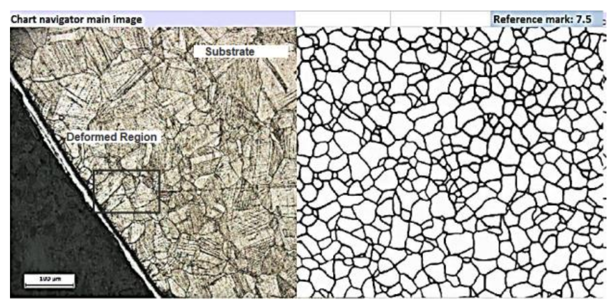


Figure 6 Grain size measurement for samples after surface attrition

Morphological behaviour of boronized stainless steel was characterized through the phase presences and thickness of FeB and Fe_2B in the boride layer produced. Figure 7 shows the morphology of Po-B850 samples. As powder pack boronizing was conducted in dry-dry condition, less than 50 µm of boride layer thickness were produced (approximately with average of 43 µm) with low thickness layer of Fe_2B compared to FeB phase, although surface attrition had been applied before boronizing process. The result shows the existence of boride layer containing FeB on the upper surface represented by darker grey shade and Fe_2B on the second layer. It could be seen that a thicker layer of FeB phase with thickness of 30µm were produced as compared to Fe_2B phase with thickness of 13 µm. Tt could be seen that the thickness of FeB layer was more than twice the thickness of Fe_2B phase. The formation of Fe_2B phase is more desirable as it acts as the diffusion barrier it accumulates the active within the layer. Consequently, the thicker the barrier, the better the surface protection of the material. The Fe_2B growth will occurs from the substrate interior which is within the interface. In industries, Fe_2B layers are more preferable compared to double phase layer of FeB and Fe_2B because the Fe_2B layer is less brittle than FeB [3].

The thickness of FeB and Fe_2B were unproportionate as boron flux has to pass through the previously formed diffusion barrier of Fe_2B. Thus, the FeB phase were thicker as it was the outmost surface (230% enhancement) that was exposed to longer time and higher treatment temperature [17].

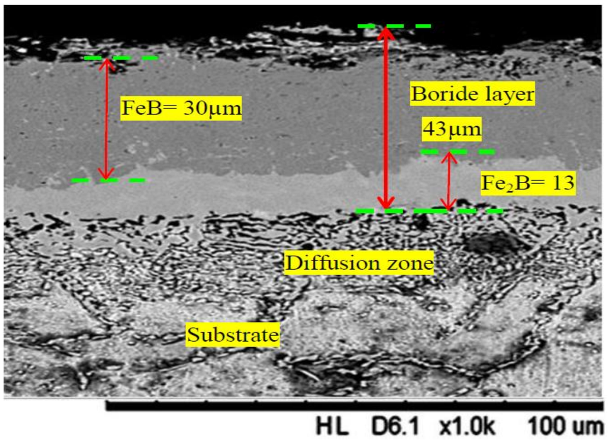


Figure 7 Morphology of Powder Boronized sample showing presences of FeB and Fe_2B

Figure 8 shows the morphology of paste boronized sample after surface attrition process. As compared to powder boronized samples, paste boronized samples provide significant improvement of almost 3 times (+/-280%) boride layer thickness with average value of 120 µm as compared to powder boronized samples with average value thickness of 43 µm. The improvement of boride layer thickness because the paste sample had better boron diffusivity because it has smaller powder particle as well as smaller interface due to wet-dry condition that enable better boride layer adhesion. During the treatment, active boron ion, B+1 will be diffused and reacted with Fe from the substrate to form the boride layer [18]. The thickness of the boride layer will be influenced by the amount of active boron, in which in this study come from the boron paste with smaller particle size that promote better diffusion.

In contrast to powder boronized samples, the formation of Fe_2B was thicker than FeB phase. Fe_2B exhibited the average boride layer thickness of 70 µm while FeB exhibited the average boride layer thickness of 50 µm with 40% boride layer thickness improvement. Generally, this is because faster intubation times could be achieved due to smaller boron paste particles that reacted with the iron substrate. As the Fe_2B phase was generated through nucleation and growth mechanism, the boron element in Fe_2B will be saturated below the FeB phase after the intubation time, creating deeper boride layer thickness. After exceeding the incubation period, the Fe_2B crystals start to energetically nucleate on the sites, thus diffusing more boron to the surface [19]. Similarly, the growth of first Fe_2B crystals will grow as needle but once it is in contact with the chromium and other alloying elements that act as diffusion barrier, flatter boride layer as produced in stainless steel and alloy steel as compared to acicular needle shape boride tips produced in plain alloy and low alloy steel [20].

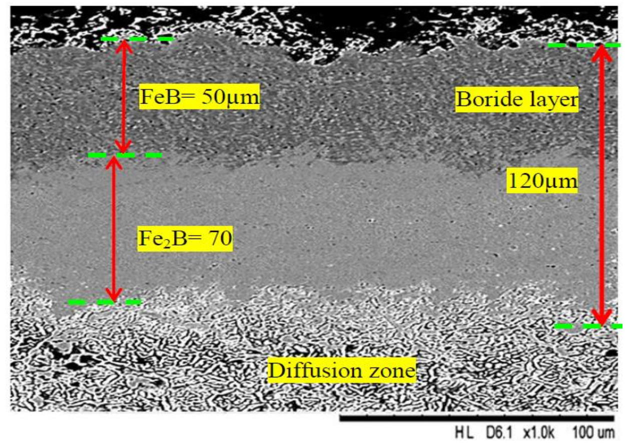


Figure 8 Morphology of Paste Boronized sample showing thicker presences of FeB and Fe_2B

The existence of FeB and Fe_2B phases were shown through the EDX Spectrum which provide the prominent atomic percentage(at) of the prominent elements in the material compositions, shown in Table 1. The EDX spectrum for powder boronized samples shows that the atomic percentage of Boron (B) was at 50.42 at. %, iron (Fe) at 30.04 at.% and chromium (Cr) and 12.04 at.% when it was spark spotted at FeB phase. In addition, the EDX spectrum also found that the atomic percentages of boron, chromium and iron were at atomic percentage of 33.1at. %, 16.94 at. % and 49.89 at % respectively for Fe_2B phase. Higher atomic percentage of boron was found in the FeB layer due to thicker boride layer produced at outer surface. An example of EDX spectrum for FeB phase of powder boronized sample was shown in Figure 9.

As for paste boronized sample, the atomic percentage of boron and other prime elements in FeB phase were 54.56 at. %, iron (Fe) and chromium (Cr) containing 33.91 at. % and 11.54 at. % respectively while the atomic percentage of boron and other prime elements in Fe_2B were 35.12 at. %, 21.02 at. % and 43.86 at. %, correspondingly. The decrement of boron atomic percentages in the Fe_2B phase was mainly because of the location of this phase was nearer to the substrate as compared to FeB phase [21].

Table 1 Atomic percentage (at%) of phases in boronized samples

Sample	Phase	B (at%)	Fe (at%)	Cr (at%)
Powder boronized sample	FeB	50.42	30.04	12.04
	Fe_2B	33.1	49.89	16.94
Paste boronized sample	FeB	54.56	33.91	11.54
	Fe_2B	35.12	43.86	21.02

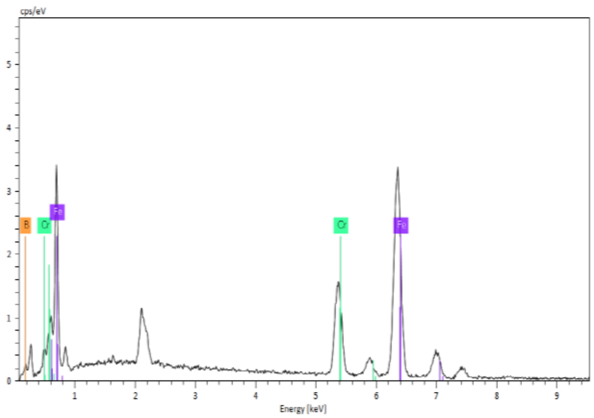


Figure 9 EDX powder boronized sample

Figure 10 provide the evidence on the presence of distinctive boride layer containing FeB and Fe_2B phase which was performed using XRD analyser. FeB phase was spotted at angles of 37° (1 1 9), 62° (4 3 4) and 80° (6 1 1) the validation of Fe_2B phase are at the angle of 35° (2 0 8), 42° (0 1 1) and 82° (7 1 6).

The presence of both FeB and Fe_2B phase was further validated on Figure 11 showing the 2 Theta angles of 37° (1 3 4), 42° (0 3 9) and 80° (5 2 3) for FeB phase while for Fe_2B phase at angles of 35° (3 2 1), 58° (0 3 9) and 80° (2 5 2) respectively.

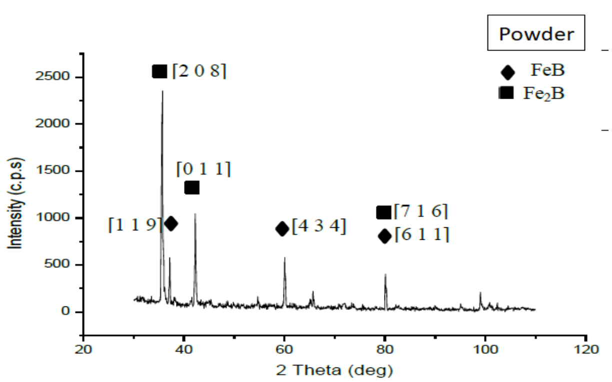


Figure 10 XRD pattern for Po-B850 FeB

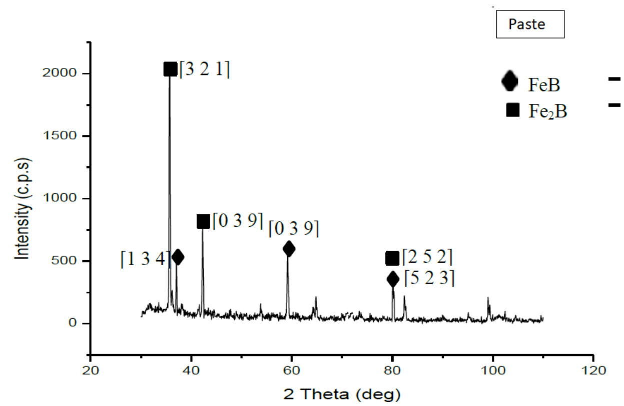


Figure 11 XRD pattern for paste boronized sample

Figure 12 shows the relationship between the layer produced and the microhardness of paste boronized stainless steel. The highest hardness value was attained by the outer FeB phase with the hardness value of 1800 Hv. As the distance move toward the substrate, the hardness reduced drastically to approximately to 900 Hv as Fe_2B exhibited more ductile nature compared to hard FeB phase. Further reduction to 257 Hv was noted on the substrate of 304 stainless steel, 7 times lower than the hardness of FeB phase.

Powder boronized stainless steel also exhibited similar trend as paste boronized stainless steel as the highest value of hardness obtained was 1500Hv at FeB phase and the value reduced to 850Hv at Fe_2B phase. Due to better boron diffusion on paste boronized sample, higher hardness value with improvement of 20% was achieved. In comparison, paste researchers also found that the implementation of paste boronizing induce better activation energy that allow boron to be intensively diffused [22-23]. The XRD results were in accordance to PDF card no 00 – 033 – 0397 for Alloys and Metal from Rikagu brand X-Ray diffractometer.

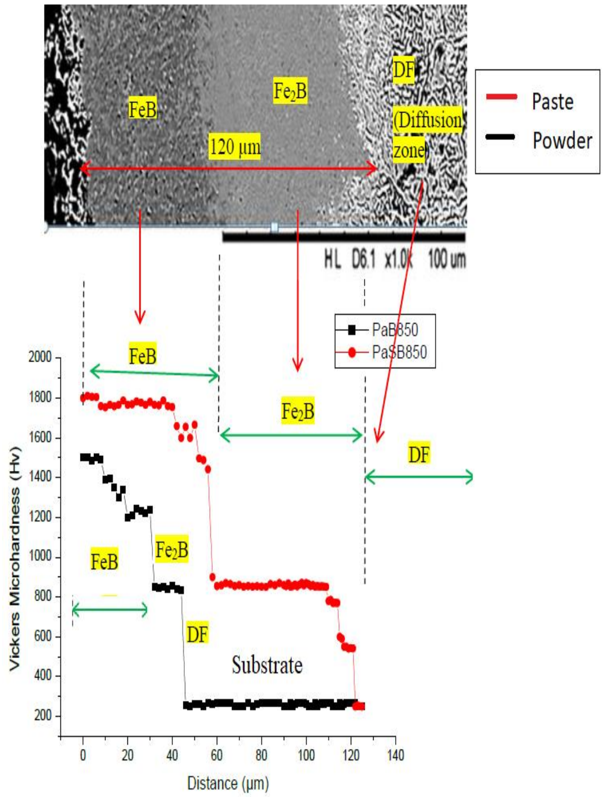


Figure 12 Microhardness comparison between Powder and Paste Boronized samples

4.0 CONCLUSION

The effect of powder and paste medium on the morphological behavior of 304 stainless steel after surface attrition at the same temperature of 850°C had been successfully evaluated. Both mediums had successfully produced FeB and Fe_2B phase in the boride layer with different thickness layer. Paste boronized samples had successfully outperformed the powder boronized samples in term of the thickness of boride layer produced (43 µm to 120 µm) due to better boron diffusivity into the treated surface. Consequently, the improvement of boride layer thickness also resulted in microhardness enhancement of more than 20% compared to powder boronized samples (1500Hv to 1800Hv).

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