

In Situ Production of B₄C and FeV Enriched Composite Surface on Low Carbon Steel by Cast Sintering Technique

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Abstract: A simple and cost effective technique to obtain locally hardened composite layer on cast alloy was previously developed based on advantages of liquid-sintering theory. This methodology provides an opportunity to produce final product with hardened composite layer with good wear resistance during casting process. The current study investigated the effect of combined B₄C and FeV substrates on the composite layer properties during in situ cast sintering technique for low carbon steel. Microstructure inspection, XRD phase analysis, chemical composition, thickness measurement and the hardness test on the produced composite layer were conducted. The technique produced up to 640 (± 300) μm thick composite layer with maximum hardness of 481 (± 14.6) HV. The hardness of the reinforced surface layer is 3-4 times higher than the base alloy. The increased hardness of composite zone is directly attributed to the presence of the iron boron (FeB), boron vanadium (V₂B₃) and iron carbide (Fe₃C). There were no significant effect of substrate composition on the produced thickness of the composite layer. However, an increase of FeV proportion in the powder mixture leads increased the hardness. The current study is a first attempt to produce locally hardened surface layer on the low carbon steel using simple and cost effective in situ technique with use of relatively inexpensive substrates.

Keywords: Cast steel; hardness; composite; in situ; microstructure; reinforcement.

1. Introduction

Abrasive wear, which is a progressive loss of material from surface of parts and components, is a vital problem in machine parts including shafts, pulley, slide-ways, and other components used for mining, construction and agricultural sectors. It eventually leads failure

of the machine parts and costly repair or replacement required to restore productivity of machinery and function [1]. In order to decrease high cost of repair and replacement of components, wear resistance properties of the material must be kept maximum as possible [2]. The wear resistance materials can be made by bulk and local hardening techniques that purposed to strengthen entire material and target location, respectively [3]. However, from viewpoint of application of the components under wear, the local surface hardening technique including alloying, chilling, cooling, heating and locally reinforced casting methods are more suitable and cost effective [4-7]. Moreover, the surface hardening doesn't cause any alteration in hardness of the core, they also provide toughness by absorbing energy which occurs in materials exposed to static and dynamic loads [8].

A simple and cost effective technique to obtain locally hardened composite layer on casting surface is use of self-propagating high synthesis (SHS) and it is an in situ technology based on advantages of liquid-sintering theory of powder metallurgy [7; 9]. The technique allows forming hard ceramic surface layer with carbide in process of chemical reaction between highly reactive powder substrates placed in mold cavity and molten alloy [3-22]. This methodology provides an opportunity to produce final product shape with locally reinforced surface layer during casting by simplifying the production process with low cost and good wear resistance [3; 15; 16].

A few groups of researchers were focused on the study to produce locally hardened, reinforced composite surface by in situ cast sintering technique. E. Frasz, E. Olejnik and colleagues were concentrated to fabricate in situ composite layer on the low and medium carbon steel with use of TiC particles [4; 5; 9; 17-21]. The microstructure [17; 20], hardness [4; 20], phase analysis [4; 9; 19-21], and wear rate [4; 5] of composite layer were investigated extensively. Y. Wang and the colleagues also studied production of metal matrix surface component layer on the low and medium carbon steel with use of VC [11; 13-15], TiC [12], and SiO₂ [14] particles and the microstructure, phase analysis, hardness, and thickness of hardened surface layer was investigated. For carbon steel, it is evident that the Fe-VC and Fe-TiC composite surface offers great wear resistance under dry slipping condition with heavy loads [11; 13; 15]. A few other studies were focused on boron-enriched local hardening with use of TiB₂, and FeB particles [6; 7]. According to previous studies [2; 23-25], boronizing or boriding is more effective and inexpensive technique to increase wear resistance of surface layer possessed very high hardness. The use of boron in addition to vanadium as an additive particle might be more suitable for in situ cast sintering technique to obtain high wear resistance composite layer on the low carbon steel. Therefore, the current study was designed to investigate the effect of combined boron carbide (B₄C) and ferrovandium (FeV) substrates on the hardened composite surface properties during in situ cast sintering technique for low carbon steel.

2. Experiment

The base alloy selected for the study was low carbon cast steel and its chemical properties were shown in Table 1. The base alloy was selected to ensure to be inexpensive material with low wear resistance and hardness [20; 21].

Table 1. Chemical composition of the base steel (wt. %)

Elements	C	Mn	S	P	Si	Cr
%	0.22	0.5	0.26	0.06	0.04	0.1

Two types of commercially available powders were used for the mixture substrates. The B_4C with about $180\mu m$ of average grain size and FeV with about $200\mu m$ grain size were used as reactant. B_4C was used instead of pure boron due to that is 10 times less expensive [6]. In order to evaluate different proportion of mixture effect, three different composition of mixtures were prepared B_4C -80% and FeV -20%; B_4C -70% and FeV-30%; B_4C -60% and FeV-40% based on the percentage of mixture weight. Powders were mixed using distilled water and uniformly polished on the inner surface of the sand molds with average thickness of 1.5mm [7]. Sodium silicate bonded sand mold (Figure 1) was built with cylindrical cavity of (length of 30mm, diameter of 15mm). CO_2 blowing process was used to accelerate sand mold hardening [18]. The melting procedure was carried out using 10kg capacity of medium-frequency induction furnace. The melt was superheated up to $1620^\circ C$ and held at this temperature for 5 minute before casting process [7; 12; 22].

After solidification and cooling, the cast samples were removed from mold and cut through cross section, polished and deep-etched with 5% volume HNO_3 for further metallographic investigation [6; 20]. The microstructure of each samples were investigated using Opton Axioscope (Carl Zeiss Microscopy GmbH, Germany) light microscope with magnification capacity of 50-1000 [7]. The crystallographic structure of the composite layer was also investigated using X-ray powder diffractometer (Enraf Nonius, Delft) with scan ranges ($2\theta = 13-70^\circ$). The chemical composition of the composite layer was investigated using X-ray microanalysis (EDX) with NeoScope JCM-6000Plus (JEOL Ltd., Japan) benchtop scanning electron microscopy (SEM). The Vickers hardness of composite surface of each samples were measured on 12 clock positions (Figure. 1B) using Qness Model-Q10 micro hardness tester (ATM Qness GmbH, Germany) under a load of 98.07 N. For each specimen, the harnesses were measured also at outer position ($\sim 0.2mm$ from edge), middle position ($\sim 0.5 mm$ from edge) and inner position ($\sim 1mm$ from edge) of the composite layer at the position with maximum thickness. The thickness of the composite layer was measured also at 12 clock positions (Figure. 1B) using IM-5000 inverted metallurgical microscope (PACE Technologies, USA). The paired sample t-test ($\alpha=0.05$) was utilized for data analysis to determine statistically significant difference exist in hardness between groups of different mixture proportions as well as in thickness between different position of the sample.

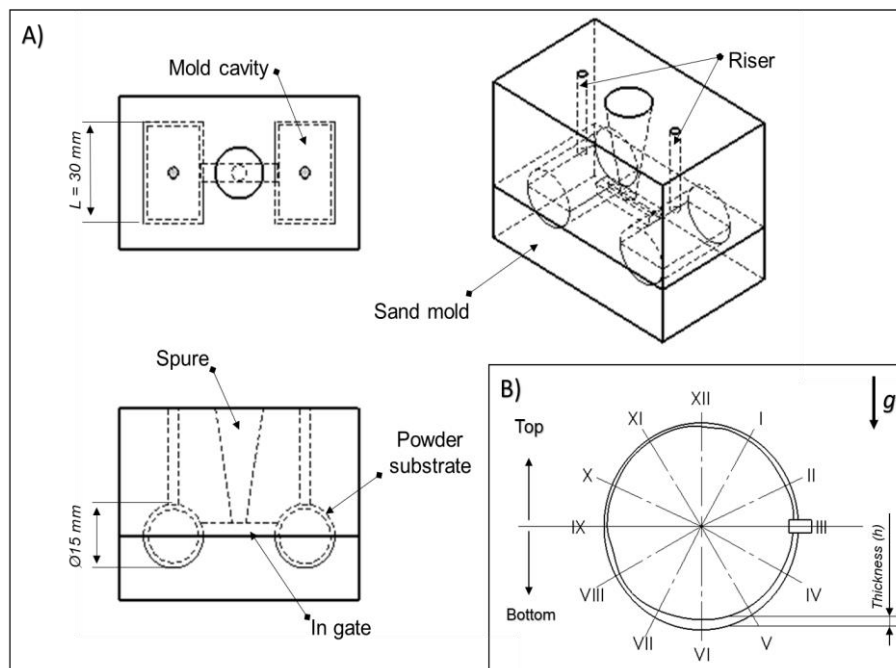


Figure 1. Schematic diagram of sand mold (A) and the schema of 12 clock positions (B) that used for the result measurement.

3. Results

The Figure 2 shows light microscope image of microstructure of base alloy displayed proeutectoid steel with ferrite and pearlite structure (Figure. 2A), reinforced surface layer with uniform thickness (Figure. 2B), bonding interface between base alloy and composite layer (Figure. 2C), microstructure of composite layer for case of B₄C (80%): FeV (20%), B₄C (70%): FeV (30%) and B₄C (60%): FeV (40%) (Figure. 2D-2F). In general, the structures of composite layer were pearlite and eutectic structure (Figure. 2). An increase of FeV composition in powder mixture resulted decrease of pearlite structure and leads an increase of eutectic structure at the composite layer (Figure. 2D-2F). There was a gray layer observed around pearlite structure that was expected to be boron and vanadium carbide formation (Figure. 2F). The result XRD phase analysis (Figure. 3) confirmed existence of iron boron (FeB, 34.6 -54.8% of weight), boron vanadium (V₂B₃, 18.5 - 27.1% of weight) and iron carbide (Fe₃C, 12.3-30.2% of weight). A small proportion of FeB₄₉ (3.0-6.8 % of weight) and vanadium carbide (VC and V₈C₇, less than 2.5% of weight) were detected during phase analysis.

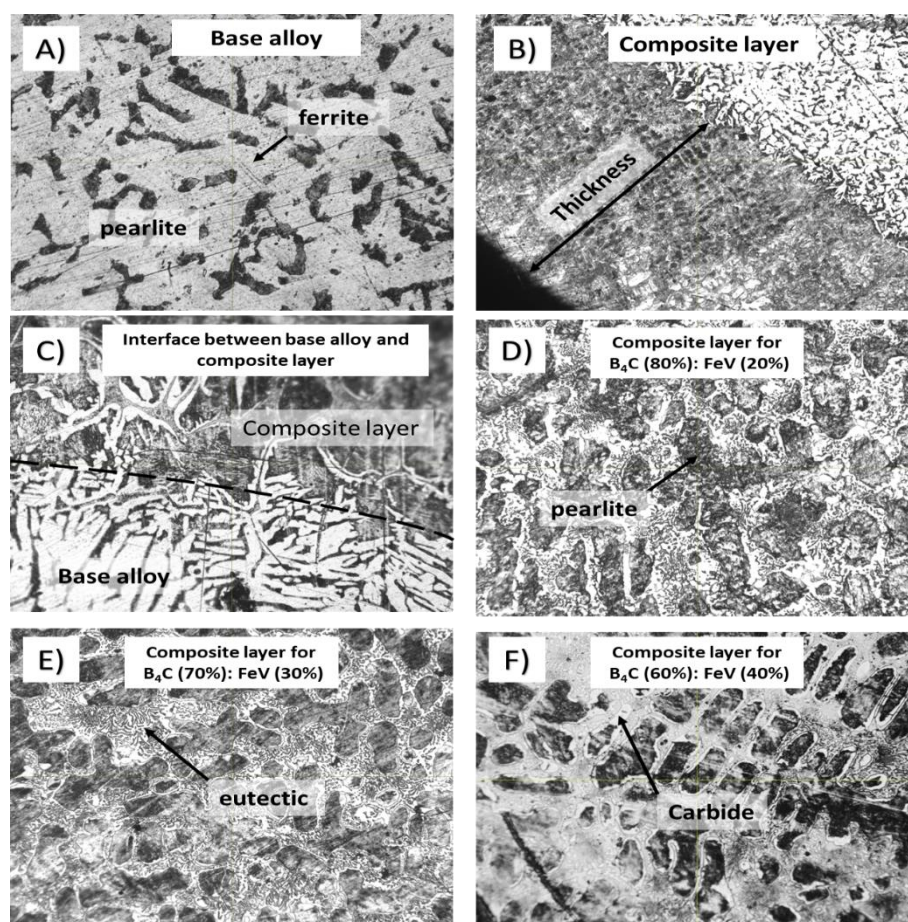


Figure. 2. Metallographic inspection result of base alloy (A), Composite layer formation (B), Interface between base alloy and composite layer (C), Microstructure of composite layer for B₄C (80%): FeV (20%) (D), Microstructure of composite layer for B₄C (70%): FeV (30%) (E) and Microstructure of composite layer for B₄C (60%): FeV (40%) (F).

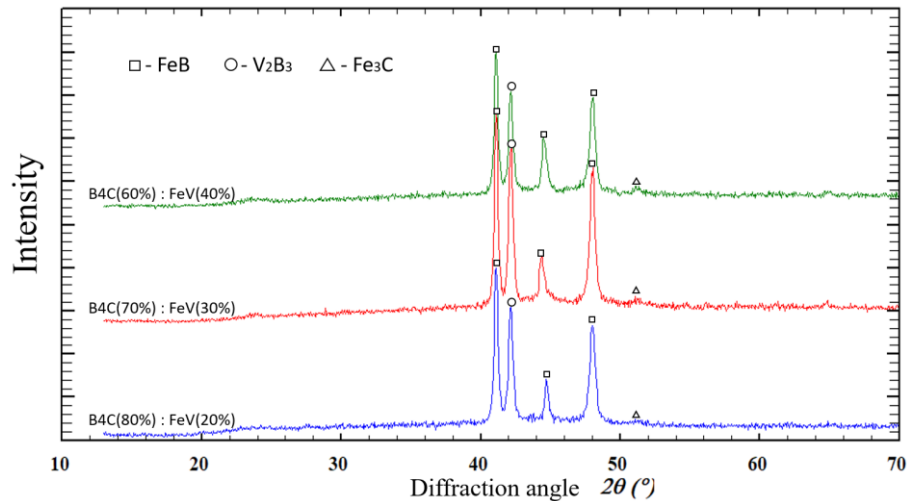


Figure. 3. Phase analysis of composite layer for specimen of B_4C (80%): FeV (20%), B_4C (70%): FeV (30%) and B_4C (60%): FeV (40%).

The result of EDX microanalysis conducted at 6 points marked in Figure 4 are shown in table 2. For all the locations, Fe (ranged from 64.37% to 88.54% of weight) and C (ranged from 10.22% to 27.46% of weight) were mainly present (Table 2). A small amount of Si (about 2%) and V (up to 2.04% of weight) was observed for all 6 points. The B mainly presented P3, P4, and P5 locations with amount less than 5% of weight. While there is no B exist at P6 (Table 2). The results of the EDX microanalysis, the chemical composition, confirmed the result of XRD phase analysis (Figure. 3).

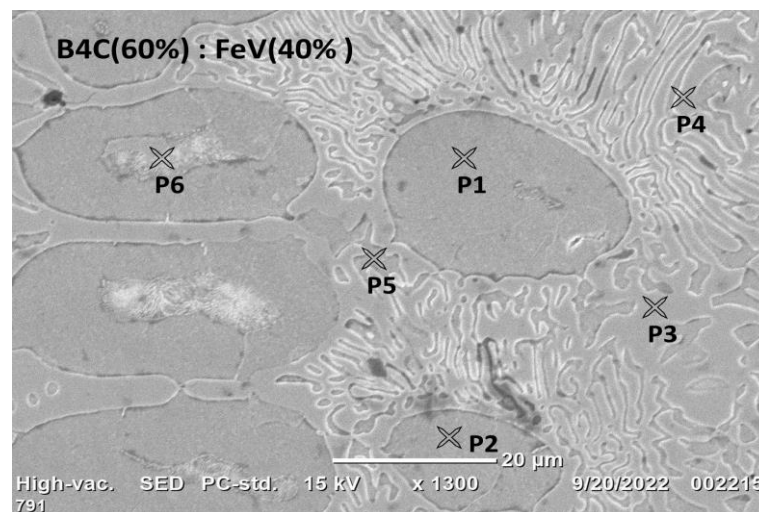


Figure. 4. SEM image of composite layer with marked 6 points of the EDX microanalysis using NeoScope JCM-6000Plus.

The result of Vickers hardness was shown in the Figure 5. The mean of hardness that measured at 12 clock positions (Figure. 1B) were $414 (\pm 68.3)$ HV for mixture of B_4C (80%): FeV (20%), $431.9 (\pm 28.4)$ HV for mixture of B_4C (70%): FeV (30%) and $481 (\pm 14.6)$ HV for mixture of B_4C (60%): FeV (40%), respectively (Figure. 5A). For hardness, a statistically significant difference ($p < 0.05$) observed between B_4C (80%): FeV (20%) and B_4C (60%): FeV (40%) specimens and between B_4C (70%): FeV (30%) and B_4C (60%): FeV (40%) specimens (Figure. 5A). There was no significant difference of hardness resulted between B_4C (80%): FeV (20%) and B_4C (70%): FeV (30%). For each specimen, the harnesses were also measured at outer, middle and inner position of the composite layer at the position of maximum thickness

which generally occurred at bottom of mold (Figure. 5B). These were 424.5 (± 20.1) HV, 460.3 (± 26.3) HV and 405.8 (± 61.9) HV for outer, middle and inner positions, respectively.

Table 2. Chemical composition (Wt. %), the color contrast indicates different mixture types.

	C	Si	V	Fe	B
P1	20.72	2.64	0.33	71.6	-
	15.63	1.78	0.5	82.09	-
	14.93	1.93	0.57	81.52	1.05
P2	22.68	2.91	0.34	74.07	-
	15.72	0.72	-	78.81	4.75
	14.66	1.65	0.56	83.14	-
P3	24.56	2.05	0.76	69.03	3.6
	19.2	0.82	2.04	73.31	4.62
	14.89	0.72	1.75	77.78	4.87
P4	27.9	2.32	1.1	65.05	3.64
	9.92	-	1.57	88.52	-
	17.04	1.36	0.89	77.35	3.35
P5	27.46	2.02	1.31	64.37	4.84
	17.12	1.88	0.35	80.65	-
	16.81	1.59	0.72	77.92	2.96
P6	23.72	2.19	0.27	73.82	-
	10.22	1.25	-	88.54	-
	15.82	2.05	0.35	81.77	-

B ₄ C(80%) : FeV(20%)	B ₄ C(70%) : FeV(30%)	B ₄ C(60%) : FeV(40%)
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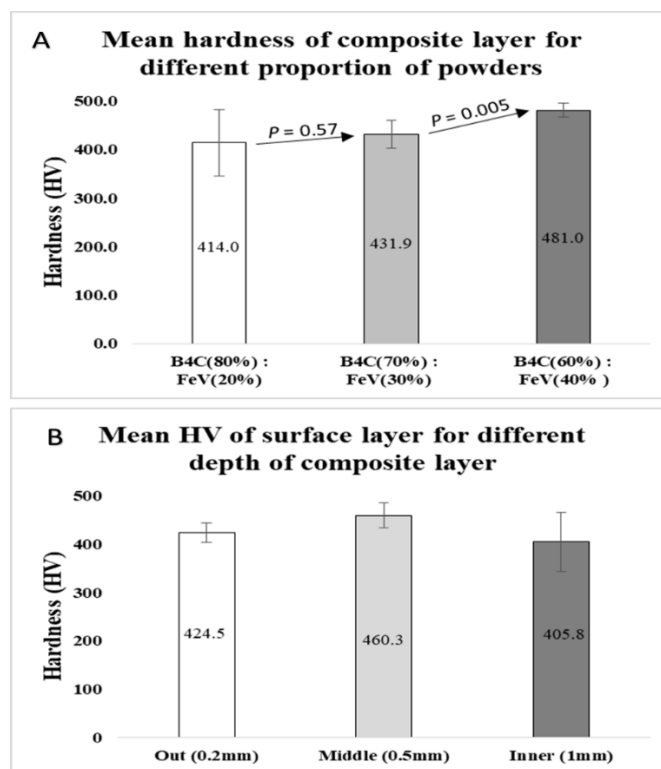


Figure. 5. Mean hardness of the composite layer for different proportion of mixture (A) and for different depth of composite layer (B).

The result of thickness measured at 12 clock positions (Figure. 1B) was depicted in the Figure 6. For top half of specimen, the mean thickness was 0.51 (± 0.30) mm, 0.48 (± 0.28) mm and 0.45 (± 0.21) mm for B₄C (80%): FeV (20%), B₄C (70%): FeV (30%) and B₄C (60%): FeV (40%) mixture proportions, respectively (Figure. 6). For bottom half, these values were 0.76 (± 0.30) mm, 0.84 (± 0.14) mm, and 0.72 (± 0.18) mm, respectively (Figure. 6). An overall, the thicknesses were 0.64 (± 0.30) mm, 0.64 (± 0.29) mm and 0.59 (± 0.26) mm for B₄C (80%): FeV (20%), B₄C (70%): FeV (30%) and B₄C (60%): FeV (40%) mixture proportions, respectively (Figure. 6). For thickness, the result of the paired sample t- test indicated a statistically significant difference ($p < 0.05$) between measurements made at top and bottom half of specimen, except for the case of B₄C (80%): FeV (20%) in which the $p = 0.1$ (Figure. 6).

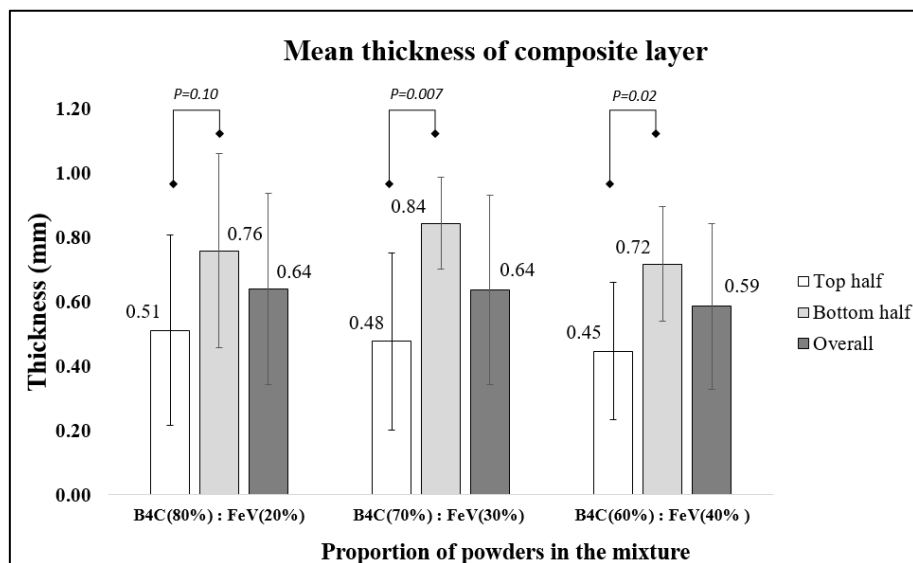


Figure. 6. The mean thickness of composite layer (reinforced surface layer) that measured at 12 positions for the different proportion of mixture and for the different positions of specimen. The “top half” indicates mean of measurements at IX-III and the “bottom half” indicates mean of measurements at III-IX.

4. Discussion

A simple and cost effective technique to obtain locally hardened composite layer on cast alloy was previously developed based on advantages of liquid-sintering theory [7-9]. This methodology provides an opportunity to produce final product with hardened composite layer with good wear resistance during casting process [3; 9; 15; 16]. From previous studies, it was evident that the Fe-VC and Fe-TiC composite surface offers great wear resistance for carbon steel [11; 13; 15; 26]. In other hand, the use of boron or boronizing, which is also more effective and inexpensive technique to increase wear resistance [2; 23-25] as a powder can be more suitable for in situ cast sintering technique to obtain high wear resistance. Therefore, the current study was designed to investigate the effect of combined boron carbide (B₄C) and ferrovandium (FeV) substrates on the hardened composite surface properties during in situ cast sintering technique.

The in situ technique produced hard composite layer containing pearlite and eutectic structure with iron and vanadium carbide formation (Figure. 2). The overall result of Vickers hardness of composite layer (Figure. 5A) were ranged from 414 (± 68.3) to 481 (± 14.6) HV. For in situ technique, previous studies had obtained 406-490 HV for TiB₂-TiC enriched composite layer of medium carbon steel [27], a maximum of 505 (± 87) HV for cast iron using FeB particles [7], and about 700-1134 HV when using TiC as substrates [9; 28]. Therefore, the

result current study indicated a combined use of boron carbide (B₄C) and ferrovanadium (FeV) powder on the low carbon steel can achieve sufficient hardness in the composite layer. Although utilization of TiC as a substrate leads higher micro-hardness on the surface, it is not cost effective choice. It can be seen that the hardness of the reinforced surface layer is significantly higher (3-4 times) than the base alloy with hardness of about 130 HV (Figure. 5A). Previous studies also observed similar hardness increase in the composite zone compare to soft core alloy [4; 20; 27; 28]. The increased hardness in our study is directly attributed to the presence of hard crystals of the iron boron (FeB), boron vanadium (V₂B₃) and iron carbide (Fe₃C) on the composite layer (Figure. 3). Increase of FeV proportion in the powder mixture resulted slightly increased the hardness (Figure. 5A). It is indicating that the increased use of vanadium as an powder can generate higher hardness on the composite layer and eventually leads increased wear resistance [14; 15]. Few study investigated composite zone hardness in relation to the depth of layer. For instance, a study showed hardness in outer measuring zone is higher than the hardness in the inner zone [10]. Our study observed similar trend (Figure. 5B) however the hardness differences between outer and inner zone was not statistically significant.

The produced composite layer thickness by in situ technique is one of the important parameters to resist wear. In our study that tested low carbon steel, an average thickness of composite layer that measured at 12 clock positions were ranged between 590 (± 260) μm to 640 (± 300) μm (Figure. 6). A very few studies demonstrated thickness measurement during cast sintering. For FeB substrate, after solidification of cast iron, a 100-900 μm thick layer of boron- enriched ledeburite was formed on cast surface [7]. For TiC powder enriched cast steel, the composite zone thickness was comprised in a range from 550 μm to 1200 μm [9; 17]. The thickness of composite layer was 380 to 580 μm when using Ni3Al [16]. In our result of composite layer thickness, there were no significant differences between B₄C (80%): FeV (20%), B₄C (70%): FeV (30%) and B₄C (60%): FeV (40%) groups (Figure. 6). The result revealed the powder mixture content or proportion has little influence on the produced layer thickness. The finding was supported by previous study that showed the layer thickness could not be related to the variation in the coating parameters such as powder size and content [7] and more likely correlated to the casting duration and temperature [29]. However in contrast, a previous study demonstrated that the composition of reaction substrates for TiC carbides directly correlated to the produced composite layer thickness [3]. Therefore, further study should be conducted to investigate relationship between substrate composition and formed composite layer thickness for different types of mixture substrates. Our result (Figure. 6) also showed that the composite layer thickness was significantly different ($p < 0.05$) between that measured at the top and bottom half of the cylindrical cast product due to different solidification duration and temperature at these positions. It is the result of high variability of the measured thicknesses at 12 different positions. Therefore, for this in situ cast sintering technique, the shape of casting product and casting position is also an important parameter to obtain locally hardened surface layer on the preferred final product.

There are several limitations in our study associated with lack of equipment. The analysis of composite zone was carried out using light microscopy, XRD test, and EDX with those can only identify general material structure, phases and composition of chemical compound. More detailed and accurate measurement on the composite zone could be obtained using transmission electron microscope (TEM) to determine the correlation between microstructural factors and mechanical properties. The hardness and thickness of composite layer that directly linked to good wear resistant were quantified instead of direct wear testing due to limited resource.

5. Conclusion

The current study is a first attempt to produce locally hardened surface layer on the low carbon steel using in situ cast sintering technique with use of B_4C and FeV substrates. The technique produced up to $640 (\pm 300) \mu m$ thick composite layer with maximum hardness of $481 (\pm 14.6) HV$. The hardness of the composite layer was increased 3-4 times relative to base alloy. The increased hardness is directly attributed to the presence of the iron boron (FeB), boron vanadium (V_2B_3) and iron carbide (Fe_3C). There were no significant effect of substrate composition on the produced thickness of the composite layer. However, an increase of FeV proportion in the powder mixture leads increased the hardness.

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