

Determination of microstructure and microhardness properties of Boronized Vanadis 4 steel

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Abstract: Vanadis 4 (V4) is a cold work tool steel that usually used manufacturing of die cutting punches. V-4 is a high vanadium steel and is made by powder metallurgy. It has high wear resistance, high hardness, and dimensional stability more than AISI D2 tool steel. This steel is especially preferred in the manufacture of stainless steels in cutting molds. During the shaping and cutting of the stainless, a high amount of wear occurs. Different surface coatings can be used to increase the wear resistance of this steel such as TiC, TiN, Si₃N₄, and Al₂O₃ etc.

In this study, boronizing process which is different from common and conventional coating methods, were used as surface coating technique for V4 steel. Specimens were kept in EKabor II powder at 900 °C and 950 °C for 6 and 8 hours by using solid boronizing method. The microstructure and microhardness analyses were done for characterizing of obtained boron coating surface.

Keywords: Boronizing, Boriding, Vanadis 4, Cold work tool steel, Microstructure, Microhardness

1 INTRODUCTION

Traditional manufacturing processes can be mainly classified as casting, machining, plastic deformation techniques, and welding. Powder metallurgy can also be added to these basic methods. In addition, the methods which are not included in this classification but have increased their application fields, have taken their place among today's manufacturing methods, such as rapid prototyping, additive manufacturing, micro fabrication, nanofabrication, electrochemical machining processes, and chemical machining [1]. In all these manufacturing processes, the element used to shape the material are called as tool. Tool forms a material using dies, removing chip (turning, milling, die cutting, drilling, etc), or chipless methods (rolling, die forging, extrusion, etc). It is expected that the tools have hardness, high strength, high wear resistance and high corrosion resistance properties.

Steels and hard metals are the most commonly used tool materials. Tool steels are a special class of steels produced for use as industrial cutting tools, dies, and molds [1,2]. They are classified as cold work, hot work, and high speed tool steels (HSS). Generally, although they are high alloyed to provide the desired high performance, unalloyed carbon steels are also available. In AISI grades, a prefix letter is used to identify the tool steel: T for tungsten HSS, M for molybdenum HSS, H for hot-working tool steels, D for cold-work tool steels, W for water-hardening tool steels, S for shock-resistant tool steels, P for mold steels, L for low-alloy tool steels [1].

Powder metallurgy (P/M) are used to obtain special alloys which can not be produced using traditional manufacturing methods. Tool steel which produced via P/M especially are used for blades, knives, punches, dies, cutting tools, forming rollers, etc. requiring high-performance steels [3]. These steels are produced in different contents and sizes by different commercial companies. The statements of the steels vary according to the company they are produced: DuraTech Pyrovan HC, CPM, Vanadis, and so on [4,5,6].

The most known P/M tool steels are Vanadis tool steels which are a trademark of Uddeholm Company. In addition to their high vanadium content, these steels have high chromium and molybdenum in their chemical composition. They are used as hot work and cold work tool steels in different fields according to their chemical compositions and mechanical properties. Compared to traditionally produced tool steels, they exhibit very high hardness values as well as good machinability, dimensional stability, and high toughness. And, V4, V6, V8, V23, V30, and V60 varieties are available. Vanadis 4 steel has combination of high wear resistance with good ductility. Vanadis 4 cold work steel especially is preferred for forming applications of austenitic stainless steel and Advanced High Strength Steel (AHSS), which are needed mixed wear resistance required [6].

Despite the use of high quality steels, additional measures may be required, in particular, to prevent tools against to mixed wear mechanism, which may occur in the continuing forming process [7]. Heat treatment processes such as hardening and tempering are used to obtain desired properties from tool steels. Thermochemical surface treatment of hardening, nitriding, carbonitriding, boriding, etc can be used. Baykara and Bedir reported that the microstructural features of Vanadis 4 and Vanadis 10 steels are directly depended upon the distribution of the carbide grains which affected by quenching conditions [8]. Sobotová et al investigated that the effect of heat treatments including austenitizing, quenching in sub-zero conditions, and tempering, on microstructure of Vanadis 6 cold-work steel [9]. It was determined that while the amount of austenite decreased, the amount of martensite increased with sub-zero operations.

Boronizing, as a thermochemical diffusion process, is one of the methods used to increase the wear performance of tool steels [10,11,12,13]. The diffusion boriding process was performed at 900 °C for 5 h in the EKabor® powder mixture for Vanadis 6 tool steel by Bartkowska et al [14,15]. The boronized layers had a dual-phase microstructure composed of two types of iron borides, FeB and Fe₂B, and their microhardness ranged from 1800 to 1400 HV. After boriding, they applied

laser surface modification, so the transition zone was enlarged and the hardness gradient decreased.

TiN, TiB₂, VN, TaC, Cr, WC, Al₂O₃ coatings can be obtained using commercial PVD coatings [7,16], also. Podgornik et al. [7] stated that carbon based coatings on Vanadis 4 tool steel provided the best protection against the work material transfer, while forming soft materials. Zeghni and Hashmi [16] investigated that the influence of pretreatment on the wear behaviour of Vanadis 4 tool steels coated with TiC, TiN and Al₂O₃. It was determined that the nitrided tool steels prior to PVD coatings improved the wear resistance of the tool steels.

Laser deposition provides a good metallurgical bonding with the substrate between coating material and can be used to modify worn mold parts [17]. The hardness of the laser deposited coatings can be highly improved without negatively affecting the base material. Surzhenkov et al. used laser surface modification on Vanadis 6 tool steel [18]. The applied laser hardening was expected to increase the hardness and abrasion resistance, but on the contrary, the abrasion resistance was decreased close to 2 times, because of formation of the large amount of retained austenite.

In this study, Vanadis 4 cold work tool steel which finds a lot of use in mold parts, was borided in a solid EKabor® II medium. The microstructure and microhardness were investigated using SEM analysis, and microvickers indenter. To understand the effect of time and temperature on the element distribution, EDX analysis also applied. The relationship between determined elemental distribution and hardness distribution was investigated.

2 MATERIALS and METHODS

Vanadis 4 Extra Superclean steel is a cold work tool steel produced by powder P/M by Uddeholm Tooling AB, and used for cutting, shaping, and punching parts of dies. In this study, Vanadis 4 steel with dimensions of 10x10x20 mm is used for examining its boronizing properties. The chemical composition are given in Table 1.

Table 1. The chemical composition of Vanadis 4 steel [6]

Element	C	Si	Mn	Cr	Mo	V
wt. %	1,4	0,4	0,4	4,7	3,5	3,7

For boronizing process, containers made of stainless steel were used (Fig.1) for each specimens separately. EKabor® II were selected as boronizing medium. Specimens to be coated were placed into the stainless steel containers to be covered with 15 mm powder. The boronizing process parameters were selected as for 6 h and 8 h at 900 °C and 950 °C, can be seen in Table 2. After the coating process was finished, all of the materials were cooled in air and then the cleaning process was completed with warm water and alcohol. After cleaning, the specimens' surfaces were grinded with abrasive papers (200,360,600,800,1000

mesh), and polished with 1µm diamond paste. The etching process were applied for 10 minutes with Vilella's Reagent (1 gr picric acid + 5 ml hydrochloric acid + 100 ml ethanol) [19,20], for microstructural analyses.

Microhardness measurements were made via digital micro vickers hardness tester (Hardway Hardness Testing Equipments DV-1AT-4.3), under 50 g load at certain distances from the center to the surface of the specimens. The microstructure was observed by SEM (Zeiss Supra 40 VP) in high vacuum mode. By using EDX module, pointal chemical analysis was performed, and elemental distribution was investigated in the obtained layers.

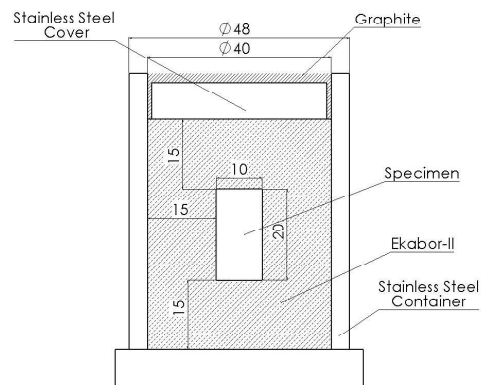


Fig.1. Schematic view of boronizing container

Table 2. Boronizing Parameters

Number of Specimens	Process Temperature (°C)	Time (h)
1	900	6
2	900	8
3	950	6
4	950	8

3 EXPERIMENTAL RESULTS

During the boronizing process, boron diffusion occurs from the surface into the inner area of the substrate. In this process, boron interacts with the elements of the main material. Some elements move from the surface to the inner region of substrate during the boronizing process, and the others move in the opposite direction, and affect on the thickness and properties of the boron layer. Figures 2-5 show that obtained microstructures using SEM. Boride layer could not be obtained at 900 °C and 950 °C for 6 hr, in Fig. 2 and 3. A very thin boride layer was obtained at 950 °C for 6 hrs: approx 4 µm. It is obtained that a visible boride layer, 30 µm, at 950 °C for 8 hrs. It can be concluded that the process time effective on boride layer thickness than the temperature. While the temperature was rised parallel with the process duration time, the boride layer thickness was increased extremely, also.

Tables 3-6 indicate that the point analysis values taken from the closest points on the coating's surface of

all samples. So, changes in Fe, B, Cr, C, and V ratios can be seen in Tables.

It can be seen that in Table 3 and Table 5, at 900 °C and 950 °C for 6 hs, samples 1 and 2, the C content were identified very high in orderly 37,79% and 36,89%, in wt. The Fe content in the same samples were founded 47,66% and 33,22%. The B content was determined as approx. 10%. As a result of increasing time from 6 hr to 8 hr, a decreasing in the C content were identified in orderly 4,22% and 4,26% in wt.in samples 2 and 4.

In all samples, while boronizing time increased, the Fe content of the surface increased at same temperatures. The Fe rate of 47.85% after 6 hours of operation at 900 °C was increased to 77.66% after 8 hours of operation. Similarly, the rate of 33.22% for 6 hours at 950 °C was increased to 72.54% after 8 hours. However, as the boronizing temperature was increased, a decrease in Fe rate was observed. The Fe ratio of 47.85% in the sample 1 was decreased to 33.22% in the sample 3. Similarly, the value of 77.66% in the sample 2 is decreased to 72.54% in the sample 4 (Table 7). The B content was increased via process time and temperature were increased, also.

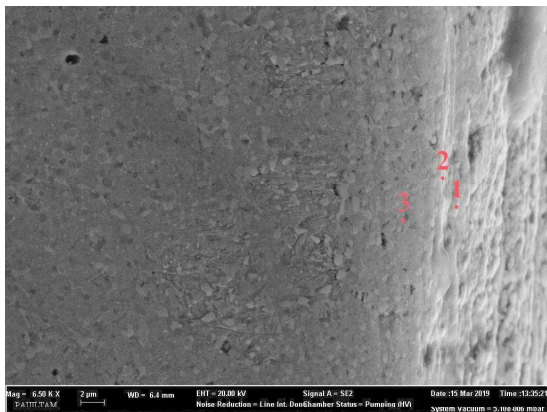


Fig. 2. SEM image of of borided specimen at 900 °C for 6 hs (1)

Table.3. EDX analysis results of borided specimen at 900 °C for 6 hs (1)

Point	Elements, wt.(%)				
	Fe	B	Cr	C	V
1	47,66	10,19	2,88	37,79	1,16
2	47,85	8,29	2,17	39,04	0,60
3	46,31	-	2,66	48,05	1,11

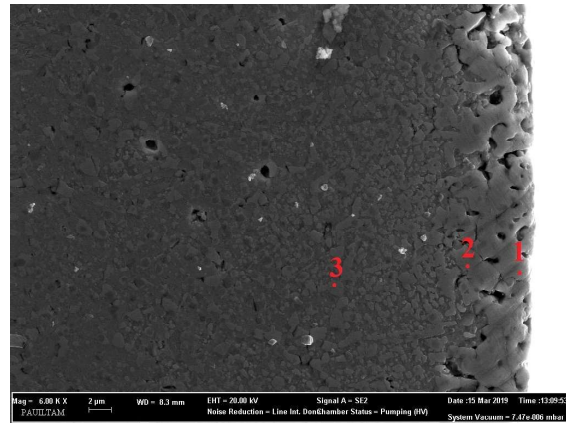


Fig. 3. SEM image of of borided specimen at 900 °C for 8 hs (2)

Table 4. EDX analysis results of borided specimen at 900 °C for 8 hs (2)

Point	Elements, wt.(%)				
	Fe	B	Cr	C	V
1	77,66	11,41	4,49	4,22	2,23
2	77,84	10,07	3,59	4,80	2,14
3	80,69	8,81	3,46	4,17	2,03

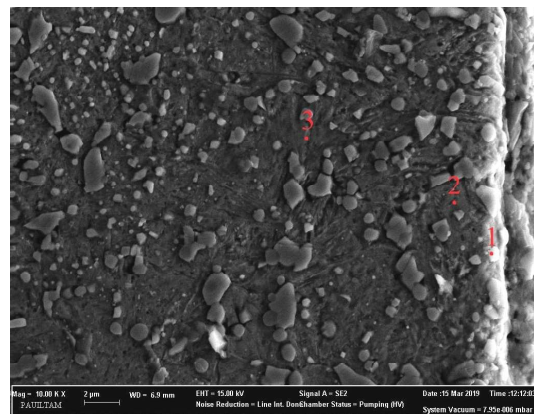


Fig.4. SEM image of of borided specimen at 950 °C for 6 hs (3)

Table 5. EDX analysis results of borided specimen at 950 °C for 6 hs (3)

Point	Elements, wt.(%)				
	Fe	B	Cr	C	V
1	33,22	10,34	1,98	36,89	1,75
2	50,14	11,87	3,46	31,97	2,11
3	78,13	5,37	3,39	5,92	4,11

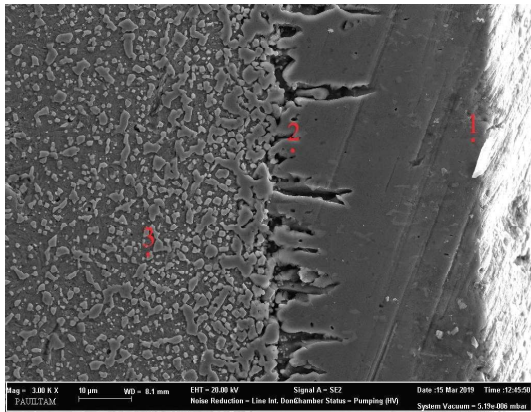


Fig.5. SEM image of of borided specimen at 950 °C for 8 h (4)

Table 6. EDX analysis results of borided specimen at 950 °C for 8 hs (4)

Point	Elements, wt.(%)				
	Fe	B	Cr	C	V
1	72,54	17,66	3,82	4,26	1,70
2	71,57	16,51	4,03	5,12	2,77
3	62,35	16,00	5,75	-	15,90

Table 7. EDX analysis results of top points of borided specimens

Specimen	Fe	B	Cr	C	V
1	47,85	8,29	2,17	39,04	0,60
2	77,66	11,41	4,49	4,22	2,23
3	33,22	10,34	1,98	36,89	1,75
4	72,54	17,66	3,82	4,26	1,70

3.3 Microhardness analysis

The central hardness of the borided material was determined as 930 HV_{0,05}. The maksimum hardness value was obtained at 950 °C for 8 hr as 1967 HV_{0,05}. The obtained hardness values can be seen in Fig. 6. It is concluded that the obtained hardness values were increased while increasing boronizing time and temperature.

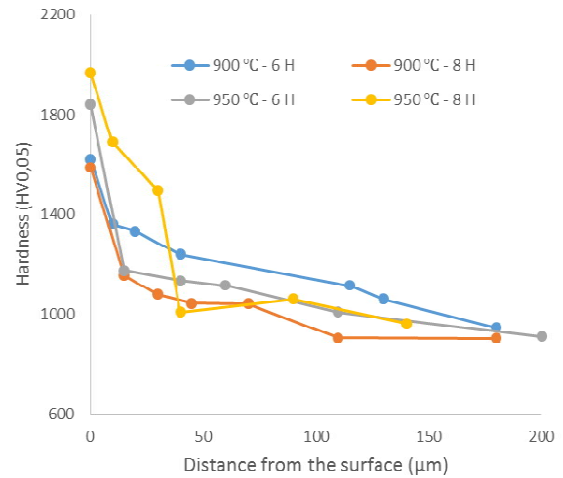


Fig.6. Microhardness distribution of borided specimens in 200 µm

4 CONCLUSIONS

- This study's results can be summarized as below:
- Boride layer could not be obtained in the boronizing experiments which applied for 6 hrs. Boride layer formation was started for 8 hr process time. The maksimum boride layer thickness was observed as to be 30 µm at 950 °C for 8 hrs.
 - The hardness values were increased while process time and temperature were increased. The maksimum hardness value was obtained at 950 °C for 8 hr as 1967 HV_{0,05}.
 - As the boronizing temperature was increased, a decrease in Fe rate was observed.
 - The B content was increased via increasing process time and temperature.

REFERENCES

- [1] Groover, M. P., "Fundamentals of Modern Manufacturing: Materials, Processes, and Systems", John Wiley & Sons Inc., 2010.
- [2] Kalpakjian, S., Schmid, S.R., "Manufacturing Engineering and Technology", Pearson, 2013.
- [3] <https://www.thyssenkrupp-materials.fr/en/products/tool-steels/high-speed-steels-produced-by-powder-metallurgy/> Erişim Tarihi: 22.03.2019.
- [4] <https://www.alphaknifesupply.com/Pictures/Info/Steel/Pyrovan-DS.pdf> Erişim Tarihi: 22.03.2019.
- [5] <http://arascelik.com.tr/en/categories/powder-metal-steels/6> Erişim Tarihi: 22.03.2019.
- [6] https://www.uddeholm.com/files/PB_Uddeholm_vanadis_4_extra_english.pdf Erişim Tarihi: 22.03.2019.
- [7] Podgornik, B., Hogmark, S., Sandberg, O., "Proper coating selection for improved galling performance of forming tool steel", *Wear*, 261, 15-21, 2006.
- [8] Baykara, T., Bedir, H. F., "Effects of Heat Treatment on the Mechanical Properties of the Vanadis 4 Extra and Vanadis 10 Tool Steels", *J Material Sci Eng*, 6:2, 2017.
- [9] Sobotova, J., Jurči, P., Adamek, J., Salabova, P., Prikner, O., Šuštaršič, B., & Jenko, D. Diagnostics of the Microstructural Changes in Sub-zero Processed Vanadis 6 P/M Ledeburitic Tool Steel. *Materials and technology*, 47, 93-98, 2013.
- [10] Armağan, E., Uluköy, A., Kaplan, Y., Onar, V., "An Investigation of the Effect of Boronizing on Cutting Performance of HSS Twist Drill", 7 th International Symposium On Machining, 2016.
- [11] Genel, K., "Boriding Kinetics of H13 steel, Vacuum", 80(5),451-457, 2006.
- [12] Armağan, E., Kaplan, Y., Onar, V., Uluköy, A., "Wear Behavior of Boronized AISI D2 Tool Steel", 16th International Materials Symposium IMSP'2016.
- [13] Uluköy, A., Kaplan, Y., Onar, V., Yıldız, M., Armağan, E., Çarık, O., Can, A. Ç., "The Microstructure and Hardness Analysis of Decarburization Followed by Boronizing AISI D3 Tool Steel", ICENS International Conference on Engineering and Natural Science, 15-19 May 2015, Skopje, Macedonia.
- [14] Bartkowska, A., Swadźba, R., Popławski, M., Bartkowski, D., "Microstructure, microhardness, phase analysis and chemical composition of laser remelted FeB-Fe2B surface layers produced on Vanadis-6 steel", *Optics & Laser Technology*, 86, 115-125, 2016.
- [15] Bartkowska, A., Bartkowski, D., Swadźba, R., Przystacki, D., Miklaszewski, A., "Microstructure, chemical composition, wear, and corrosion resistance of FeB-Fe2B-Fe3B surface layers produced on Vanadis-6 steel using CO2 laser", *Int J Adv Manuf Technol*, 95, 1763-1776, 2018.
- [16] Zeghni, A. E., Hashmi, M.S.J., "The effect of coating and nitriding on the wear behaviour of tool steels", *Journal of Materials Processing Technology*, 155-156, 1918-1922, 2004.
- [17] Leunda, J., Navas, V. G., Soriano, C., Sanz, C., "Improvement of Laser Deposited High Alloyed Powder Metallurgical Tool Steel by a Post-tempering Treatment" *Physics Procedia*, 39, 392 - 400, 2012.
- [18] Surzhenkov, A., Kulu, P., Viljus, M., Vallikivi, A., & Latokartano, J. Microstructure and wear resistance of the laser hardened PM tool steel vanadis 6. *J Mat Sci*, 148(2), 22-8, 2010.
- [19] Arslan, F.K., Altinsoy, I., Hatman, A., Ipek, M., Zeytin, S., Bindal, C., "Characterization of cryogenic heat treated Vanadis 4 PM cold work tool steel", *Vacuum*, 86(4), 2011.
- [20] Gecü, R., Kısasöz, A., Karaaslan, A., "Soğuk İş Takım Çeliklerinde Cr ve V İçeriğinin Malzeme Özellikleri Üzerindeki Etkisinin İncelenmesi", 5. Isıl İşlem Sempozyumu, 2016.

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