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Investigation of Substrate Microstructure after Flame Spraying and Fusing

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In this paper, influence of flame spraying and fusing on microstructure of substrates made from heat treated carbon steel C45, a heat treated low alloyed 42CrMo4 steel and stainless austenitic steel X6CrNiMo18-10-2, has been investigated. Flame spraying and fusing is a deposition technique commonly applied to nickel-base self-fluxing alloys (NiCrBSi alloys). Because of high temperature of fusing (approximately 1000 °C), a diffusion bond between the coating and the substrate is created and a homogeneous coating with decreased porosity is obtained. It has been proved that there is a significant influence of fusing temperature on the microstructure of substrate materials, especially on the coating/substrate interface.

Istraživanje mikrostrukture podloge nakon toplog postupka plinskog naštrcavanja

Izvornoznanstveni članak

U ovome je radu prikazan utjecaj toplog postupka plinskog naštrcavanja na mikrostrukturu podloge od toplinski obrađenog ugljičnog čelika C45, toplinski obrađenog niskolegiranog čelika 42CrMo4, te nehrđajućeg austenitnog čelika X6CrNiMo18-10-2. Ovaj postupak se vrlo često koristi za naštrcavanje legura na osnovi nikla. Zbog visoke temperature taljenja ovih legura (približno 1000 °C), između sloja i podloge stvara se metalurška veza, a dobiveni homogeni sloj je smanjene poroznosti. Dokazano je da postoji značajan utjecaj temperature taljenja sloja, na mikrostrukturu materijala podloge, osobito na granicu sustava podloga/sloj.

1. Introduction

Flame spraying is a thermal spraying technique which uses thermal energy produced by combustion of fuel gas (mostly acetylene, propane, propylene or hydrogen) in oxygen, to melt the coating material particles. It is mostly used for the protection of new parts against wear, corrosion and high temperatures and also for repairing damaged and worn out parts.

Flame spraying and fusing is a new type of flame spraying, where metallurgical or diffusion bond between coating and substrate is formed. It is also called flame spraying with fusion, flame spraying with remelting, flame spraying with post heat treatment or simply hot flame spraying**.** This procedure is commonly applied to self-fluxing nickel based alloys. Self-fluxing alloys represent a complex Ni-Cr-B-Si-Fe-C system. These alloys have high corrosion resistance (presence of chromium) and good wear resistance due to hard phases (carbides, borides, silicides).

Flame spraying and fusing can be performed according to the following procedure. After the flame spraying process, fusing process must be applied, where sprayed deposit is heated and melted to form homogenous and dense coating. Subsequent fusion can be carried out by oxy-acetylene flame, electric arc, induction heating as well as laser or heat treatment in vacuum furnace.

Flame spraying and simultaneous fusing is a technique where spraying and fusing are simultaneously performed.

If we compare it with conventional flame spraying, higher bond strength and lower porosity are achieved. Fusing temperature amounts to approximately 1000°C. It depends on the used powder. High fusing temperature affects the structure of the substrate material.

Wear and corrosion resistance, residual stress and cracking resistance investigations have been mostly applied by many authors. Microstructure of coatings has been investigated by the application of the X-ray

Symbols/Oznake

ϑ_a	- temperature of austenitisation, °C - temperatura austenitizacije	t_t	- time of tempering, min - vrijeme popuštanja
t_a	- time of austenitisation, min - vrijeme austenitizacije	HV0,1	- Vickers microhardness, load ≈1 N - Vickers mikrotvrdoća, opeterećenje 1 N
ϑ_t	- temperature of tempering, °C - temperatura popuštanja	HV30	- Vickers hardness, load ≈300 N - Vickers tvrdoća, opeterećenje 300 N

diffraction method, optical microscope as well as scanning or transmission electron microscope. Hardness and microhardness tests have been very often performed. The powder type [6, 7, 18], the method of fusing [1, 3], fuel gas/oxygen ratio [18, 2], powder particle shape and dimensions [6, 7], presence of hard phases in powder [11, 14, 15, 17, 19] are the most influential factors on the microstructure of coating. The majority of authors have investigated coatings behaviour on steel substrates [5, 7, 10, 12, 18]. The authors [12] investigated a microstructure of nickel base self fluxing alloys as well as the interface reaction between the coating and a steel substrate. Microstructure of the coating consists of the crystals of M_6C carbide with chromium, molybdenum and nickel, M_3B_2 boride with chromium and molybdenum and M_7C_3 carbide with chromium. Also Ni-Ni_3B eutectic phase was found. The author [8] reported that nickel based self fluxing alloy coatings contain the following hard phases: $Cr_{23}C_6$, $(NiFe)_{23}C_6$, Cr_7C_3, W_2C carbides, Ni_2B, Ni_3B, Cr_2B borides and Ni_5Si_2 silicides, which are precipitated like primary/secondary carbides or like binary/ternary eutectics.

Coating/substrate interface has also been investigated, by the following authors [1, 12]. The authors [1] reported that coating/substrate interface contains more Fe than the coating. It was concluded that this is due to Fe diffusion from the substrate. The precipitates of Fe_2B boride were observed at the interface [12].

This paper examines the influence of the fusing temperature on the structure of heat treated steel substrates and stainless steel substrate, by means of light optical microscopy and microhardness testing.

2. Experimental

Three types of NiCrBSi alloy powders have been flame sprayed on steel substrates and simultaneously fused, Figure 1.

Figure 1. Flame spraying and simultaneous fusing

Slika 1. Topli postupak plinskog naštrcavanja

The chemical composition of the three types of steels is shown in Table 1. Substrate materials (steels) are signed according to their chemical composition, by EN 10027 standard.

The chemical composition of the three types of NiCrBSi powders is shown in Table 2.

The powder NiCrBSi+WC is a composite powder consisting of 60 % of WC carbide mechanically mixed with the NiCrBSi matrix powder. The powder NiCrWBSi also contains WC carbides, but in the form of fine phases uniformly dispersed in the NiCrBSi powder particle.

Table 1. The chemical composition of steels, wt(%)

Tablica 1. Kemijski sastav čelika (maseni udio, %)

	C45	42CrMo4	X6CrNiMo18-10-2
C	0,45	0,43	0,04
Si	0,26	0,25	0,54
Mn	0,74	0,53	1,86
Cr	-	1,18	17,8
Ni	-	-	10,72
Mo	-	0,3	2,48
Ti	-	-	0,34
S	0,032	0,016	0,01
P	0,027	0,015	-
Fe	balanced / ostalo	balanced / ostalo	balanced / ostalo

Table 2. The chemical composition of powders, wt(%)

Tablica 2. Kemijski sastav prahova (maseni udio, %)

	NiCrBSi + WC	NiCrWBSi	NiCrBSi
Cr	7	15	15
B	3	3	3,2
Si	4,5	4	4,4
Fe	5,8	3,5	0,7
C	0,1	0,8	0,7
W	-	17,3	-
Ni	balanced / ostalo	balanced / ostalo	balanced / ostalo

The following flame spraying parameters are used: acetylene pressure 50000 Pa, oxygen pressure 200000 Pa, coating thickness 0,3 mm, sample thickness 12 mm, temperature of fusing 1000 ^{o}C and spraying distance 120 mm.

The samples made from steels C45 and 42CrMo4 are previously heat treated (hardened and tempered), according to parameters presented in Table 3. The third type of steel X6CrNiMo18-10-2 is not heat treated before the flame spraying and fusing.

Table 3. Substrate materials and heat treatment parameters

Tablica 3. Materijali podloge i parametri toplinske obrade

Substrate Material / Materijal podloge	Heat treatment parameters / Parametri toplinske obrade
C45	Austenitisation/Austenitizacija (ϑ_a = 840 ^{o}C, t_a = 15 min) Water quenching/Gašenje u vodi Tempering/Popuštanje (ϑ_t = 600 ^{o}C, t_t = 30 min)
42CrMo4	Austenitisation/Austenitizacija (ϑ_a = 840 ^{o}C, t_a = 15 min) Oil quenching/Gašenje u ulju Tempering/Popuštanje (ϑ_t = 600 ^{o}C, t_t = 30 min)
X6CrNiMo18-10-2	-

Figures 2, 3 and 4 show microstructures of steel substrates taken at a light optical microscope.

After the hardening and tempering, the structure of C45 steel is tempered martensite (Figure 2), with the 320 HV30 average hardness.

After the hardening and tempering, the structure of 42CrMo4 steel is tempered martensite (Figure 3), with the 400 HV30 average hardness.

The structure of stainless X6CrNiMo18-10-2 steel is austenitic (Figure 4), with the 202 HV30 average hardness.

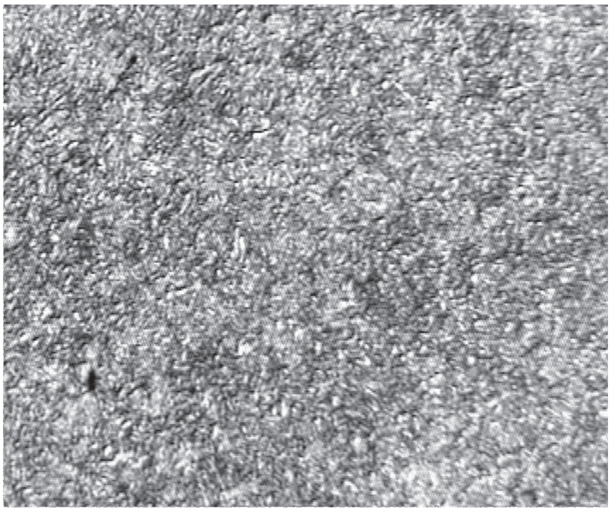

Figure 2. Microstructure of C45 steel after the hardening and tempering (cross section of the sample, 250x magnification, etching: nital solution)

Slika 2. Mikrostruktura čelika C45 nakon kaljenja i popuštanja (poprečni presjek uzorka, povećanje 250x, nagrizanje: nital)

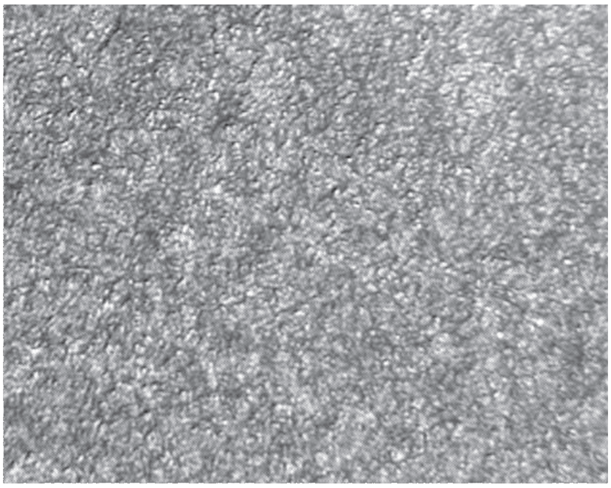

Figure 3. Microstructure of 42CrMo4 steel after the hardening and tempering (cross section of the sample, 250x magnification, etching: nital solution)

Slika 3. Mikrostruktura čelika 42CrMo4 nakon kaljenja i popuštanja (poprečni presjek uzorka, povećanje 250x, nagrizanje: nital)

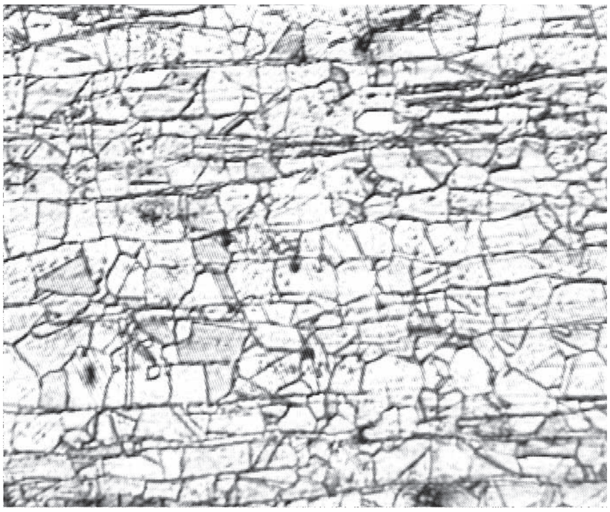

Figure 4. Microstructure of stainless steel X6CrNiMo18-10-2 (cross section of the sample, 250x magnification, etching: glyceregia)

Slika 4. Mikrostruktura čelika X6CrNiMo18-10-2 (porečni presjek uzorka, povećanje 250x, nagrizanje: carska voda u glicerinu)

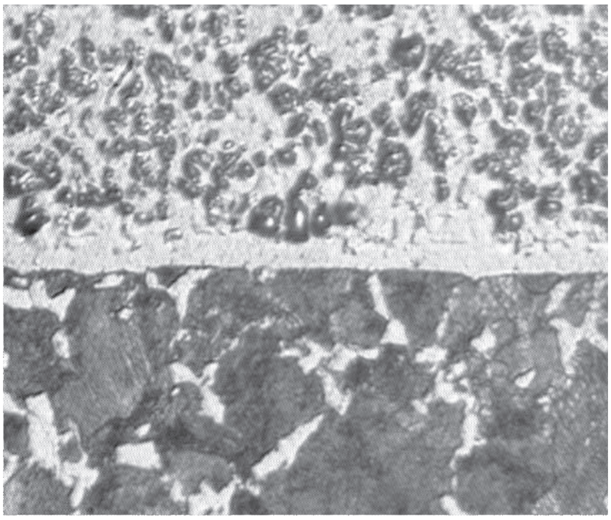

Figure 5. Microstructure of the cross sectioned sample: NiCrBSi coating-C45 steel substrate, 450x magnification, etching: nital solution

Slika 5. Mikrostruktura poprečno prerezanog uzorka: sloj NiCrBSi - podloga čelik C45, povećanje 450x, nagrizanje: nital

3. Results and Discussion

After the spraying and fusing, light optical microscopy and microhardness measurements of some of the combinations of coatings and substrates have been performed.

Figure 5 shows the microstructure of the cross sectioned sample of NiCrBSi coating on the hardened and tempered C45 steel substrate. A coating/substrate interface is shown. In Figure 6, a microstructure of the substrate under the coating is shown. In Figure 7 a microstructure of the substrate remote from the coating is shown.

A microphotograph in Figure 5 shows white diffusion zone between the coating and the substrate. The thickness of this zone is from 0,01 to 0,025 mm [16] and microhardness amounts to 550 HV0,1 (Figure 8). The microhardness of coating amounts to 900 HV0,1 and microhardness of substrate is 210 HV0,1. Under the coating (Figure 5 and Figure 6), there is a heat affected zone (0,2 to 0,3 mm) with coarse pearlite and ferrite grains. Remote from the coating (Figure 7) there are finer pearlite and ferrite grains.

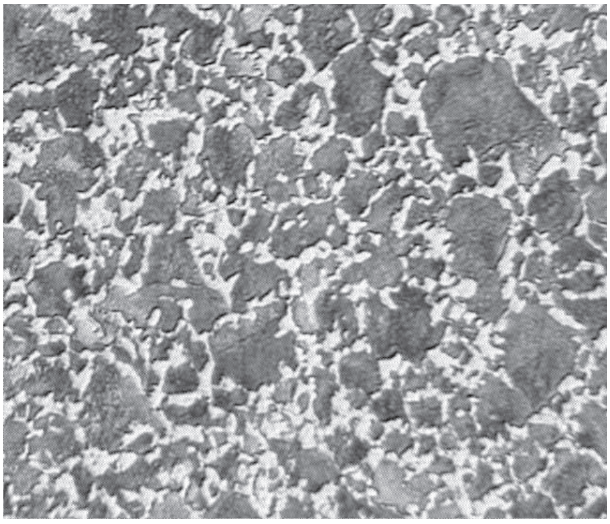

Figure 6. Microstructure of the substrate under the coating, 250x magnification, etching: nital solution

Slika 6. Mikrostruktura podloge ispod sloja, povećanje 250x, nagrizanje: nital

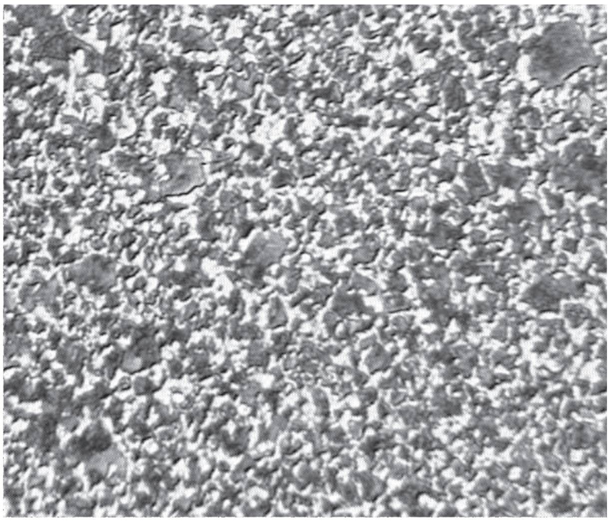

Figure 7. Microstructure of the substrate remote from the coating, 250x magnification, etching: nital solution

Slika 7. Mikrostruktura podloge dalje od sloja, povećanje 250x, nagrizanje: nital

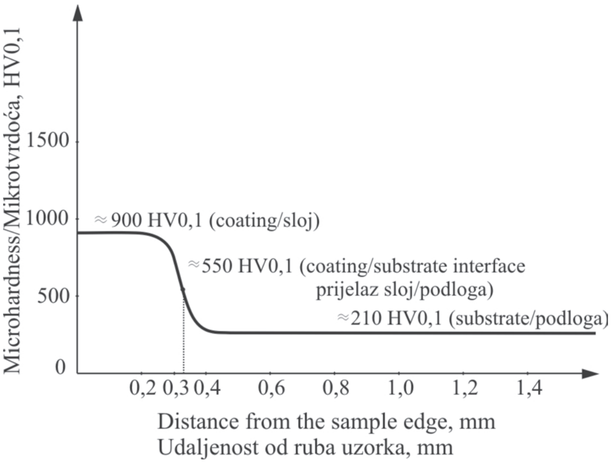

Figure 8. The results of microhardness measurements for the sample NiCrBSi coating-C45 steel

Slika 8. Rezultati mjerenja mikrotvrdoće za uzorak: sloj NiCrBSi - podloga čelik C45

Figure 9 shows the microstructure of the cross sectioned sample of NiCrBSi+WC coating on the hardened and tempered 42CrMo4 steel substrate. A coating/substrate interface is shown. A microstructure of the substrate under the coating is shown in Figure 10. A microstructure of the substrate remote from the coating is shown in Figure 11.

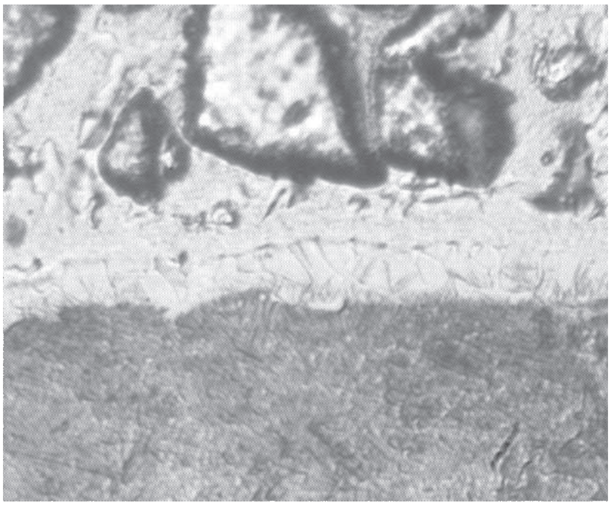

Figure 9. Microstructure of the cross sectioned sample: NiCrBSi+WC coating-42CrMo4 steel substrate, 450x magnification, etching: nital solution

Slika 9. Mikrostruktura poprečno prerezanog uzorka: sloj NiCrBSi+WC - podloga čelik 42CrMo4, povećanje 450x, nagrizanje: nital

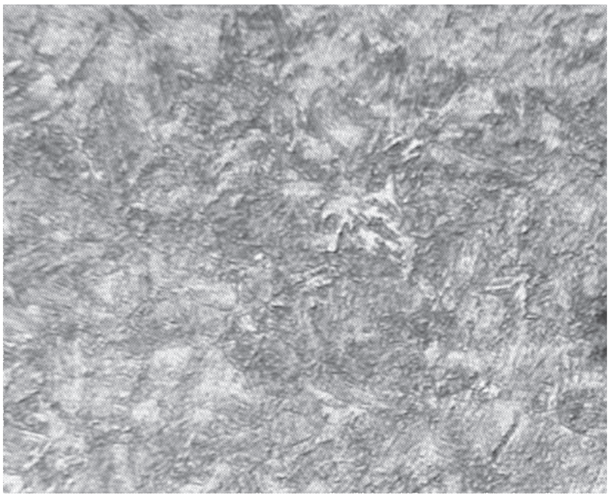

Figure 10. Microstructure of the substrate under the coating, 250x magnification, etching: nital solution

Slika 10. Mikrostruktura podloge ispod sloja, povećanje 250x, nagrizanje: nital

A microphotograph in Figure 9 shows two zones. Under the coating, there is a white diffusion zone (thickness about 0,013 mm) [16] with microhardness of 550 HV0,1. Under this diffusion zone, there is the zone (thickness 0,016 mm) with dendritic grains (microhardness 400 HV0,1). Under this dendritic zone (Figure 9 and Figure 10), there is a zone of 0,05 to 0,1 mm thickness, with the 700 HV0,1 microhardness. Higher hardness is the result of carbon diffusion. The microhardness of coating matrix amounts to 820 HV0,1 and microhardness of WC carbides is 2200 HV0,1 (Figure 12). Remote from the coating (Figure 12) microhardness amounts 460 HV0,1.

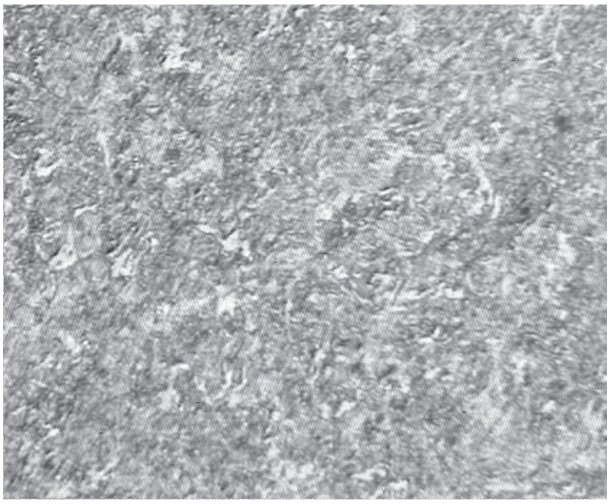

Figure 11. Microstructure of the substrate remote from the coating, 250x magnification, etching: nital solution

Slika 11. Mikrostruktura podloge dalje od sloja, povećanje 250x, nagrizanje: nital

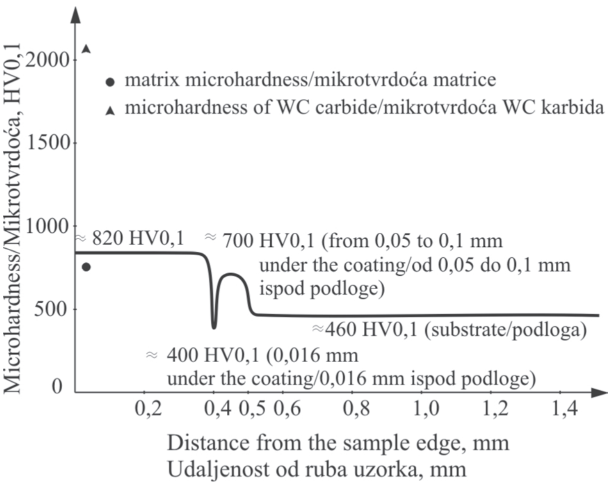

Figure 12. The results of microhardness measurements for the sample NiCrBSi+WC coating-42CrMo4 steel

Slika 12. Rezultati mjerenja mikrotvrdoće za uzorak: sloj NiCrBSi+WC - podloga čelik 42CrMo4

Figure 13 shows the microstructure of the cross sectioned sample of NiCrWBSi coating on the stainless X6CrNiMo18-10-2 steel substrate. A coating/substrate interface is shown. A microstructure of the substrate under the coating is shown in Figure 14 and a microstructure of the substrate remote from the coating is shown in Figure 15.

A microphotograph in Figure 13 shows a very thin white diffusion zone between the coating and the substrate. The microhardness of this zone amounts to 550 HV0,1. The microhardness of coating amounts to 1100 HV0,1 and the microhardness of the substrate is 190 HV0,1 (Figure 16).

Figure 13. Microstructure of the cross sectioned sample: NiCrWBSi coating-X6CrNiMo18-10-2 steel substrate, 450x magnification, etching: glyceregia

Slika 13. Mikrostruktura poprečno prerezanog uzorka: sloj NiCrWBSi - podloga čelik X6CrNiMo18-10-2, povećanje 450x, nagrizanje: carska voda u glicerinu

Under the coating (Figure 13), there is a heat affected zone with fine austenitic grains (microhardness 290 HV0,1). Fusing temperature (1000 ^{o}C) is the temperature of the heat treatment of stainless austenitic steels (Krumes 2000), so the finer austenitic grain is the result of the heating at the temperature of 1000 ^{o}C. Remote from the coating (Figure 15), there are coarser austenitic grains.

It can be seen that the coating/substrate interface is not straight, which is due to mechanical deformation (impact of melted powder particles to the soft substrate material).

4. Conclusion

Martensitic structure (Figure 2) of the hardened and tempered C45 steel after the spraying, fusing (1000 ^{o}C) and slow cooling, transforms to the pearlite ferrite structure (Figures 5, 6 and 7). In the heat affected zone under the coating, there are coarse pearlite ferrite grains, but remote from the coating they are finer.

When compared to the previously described steel, the high fusing temperature does not influence so significantly the microstructure of hardened and tempered 42CrMo4 steel. But there is a significant influence on the coating/substrate interface. There are two interface zones: the diffusion zone of 550 HV0,1 microhardness and dendritic zone with the smaller microhardness (400 HV0,1). This is due to carbon diffusion out of this zone down to the substrate (it results with the higher microhardness of 700 HV0,1).

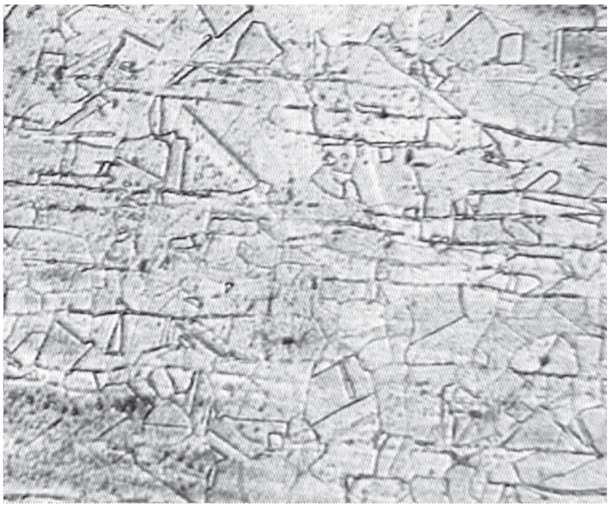

Figure 14. Microstructure of the substrate under the coating, 250x magnification, etching: glyceregia

Slika 14. Mikrostruktura podloge ispod sloja, povećanje 250x, nagrizanje: carska voda u glicerinu

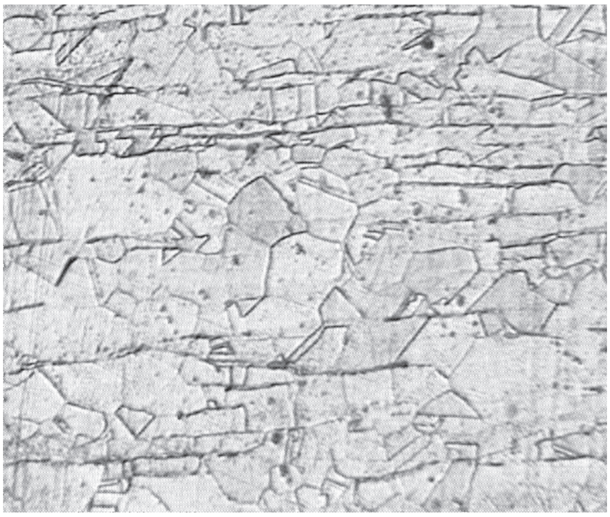

Figure 15. Microstructure of the substrate remote from the coating, 250x magnification, etching: glyceregia

Slika 15. Mikrostruktura podloge dalje od sloja sloja, povećanje 250x, nagrizanje: carska voda u glicerinu

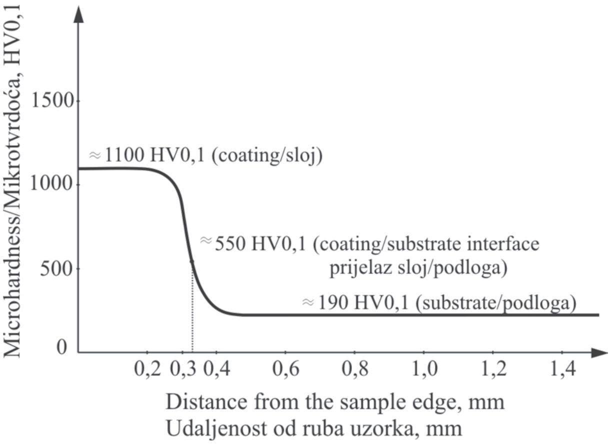

Figure 16. The results of microhardness measurements for the sample NiCrWBSi coating- X6CrNiMo18-10-2 steel

Slika 16. Rezultati mjerenja mikrotvrdoće za uzorak: sloj NiCrWBSi - podloga čelik X6CrNiMo18-10-2

Opposite to carbon C45 steel, the microstructure of stainless austenitic X6CrNiMo18-10-2 steel contains finer grains under the coating. The reason is that the fusing temperature (1000 °C) is the temperature of the heat treatment of stainless austenitic steels. Austenitic grains are coarser remote from the coating. Because of the high content of alloying elements, and consequently the lower thermal conductivity coefficient, the diffusion zone and heat affected zone are thinner than the zones of carbon C45 and low alloyed 42CrMo4 steels.

Finally, it can be concluded that the high fusing temperature significantly affects the microstructure of steel substrates, thus affecting the mechanical properties of the coating/substrate system.

Also, there is a diffusion zone of 550 HV0,1 microhardness, between the coating and the substrate. Diffusion zone is decreasing with the increasing of the content of alloying elements in the steel.

REFERENCES

[1] GOMEZ-DEL RIO, T.; GARRIDO, M.A.; FERNANDEZ, J.E.; CADENAS, M.; RODRIGUEZ, J. : *Influence of the deposition techniques on the mechanical properties and microstructure of NiCrBSi coatings*, Journal of Materials Processing Technology, 204 (1-3), 304–312, 2008.

[2] GIL, L.; STAIA, M.H.: *Microstructure and properties of HVOF thermal sprayed NiWCrBSi coatings*, Surface and Coatings Technology, 120-121, 423-429, 1999.

[3] GONZALES, R.; CADENAS, M.; FERNANDEZ, R.; CORTIZO, J.L.; RODRIGUEZ, E.: *Wear behaviour of flame sprayed NiCrBSi coating remelted by flame or by laser,* Wear, 262 (3-4), 301-307, 2007.

[4] GONZALEZ, R.; GARCIA, M.A.; PENUELAS, I.; CADENAS, M.; DEL ROCIO FERNANDEZ, MA.; HERNANDEZ BATTEZ A.; FELGUEROSO, D.: *Microstructural study of NiCrBSi coatings obtained by different processes*, Wear, 263 (1-6), 619-624, 2007.

[5] HEJWOWSKI, T.; SZEWEZYK, A.; WERONSKI, A.: *An investigation of the abrasive and erosive wear of flame-sprayed coatings,* Journal of Materials Processing Technology, 106 (1-3) 54-57, 2000.

[6] IORDANOVA, I.; FORCEY, K.S.; GERGOV, B.; BOJINOV, V.: *Characterisation of flame-sprayed pure metallic and alloyed coatings*, Surface and Coatings Technology, 72 (1-2), 23-29, 1995.

[7] IORDANOVA, I.; SURTCHEV, M.; FORCEY, K.S.: *Metallographic and SEM investigation of the microstructure of thermally sprayed coatings on steel substrates,* Surface and Coatings Technology, 139 (2-3), 118-126, 2001.

[8] KNOTEK, O.: *Thermal spraying and detonation gun processes*, Handbook of Hard Coatings, (Rointan F. Bunshah, ed.), Noyes Publications/William Andrew Publishing LLC, Norwich, New York, p. 77-107, 2001.

[9] KRUMES, D.: *Toplinska obrada*, Strojarski fakultet, Slavonski Brod, 2000.

[10] MIGUEL, J.M.; GUILEMANY, J.M.; VIZCAINO, S.: *Tribological study of NiCrBSi coating obtained by different processes,* Tribology International, 36 (3), 181-187, 2003.

[11] MIRANDA, J.C.; RAMALHO, A.: *Abrasion resistance of thermal sprayed composite coatings with a nickel alloy matrix and a WC hard phase. Effect of deposition technique and re-melting,* Tribology Letters, 11 (1), 37-48, 2001.

[12] OTSUBO, F.; ERA, H.; KISHITAKE, K.: *Interface reaction between nickel-base self-fluxing alloy coating and steel substrate*, Journal of Thermal Spray Technology, 9 (2), 259-263, 2000.

[13] OTSUBO, F.; ERA, H.; KISHITAKE, K.: *Structure and phases in nickel-base self-fluxing alloy containing high chromium and boron*, Journal of Thermal Spray Technology, 9 (1), 107-113, 2000.

[14] RODRIGUEZ, J.; MARTIN, A.; FERNANDEZ, R.; FERNANDEZ, J.E.: *An experimental study of the wear performance of NiCrBSi thermal spray coatings*, Wear, 255 (7-12), 950-955, 2003.

[15] SHIEH, Y.; WANG, J.; SHIH, H.; WU, S.: *Alloying and post-heat treatment of thermal sprayed coatings of self-fluxing alloys,* Surface and Coating Technology, 58 (1), 73-77, 1993.

[16] ŠIMUNOVIĆ, K.: *Optimizacija tribomehaničkih svojstava plinski naštrcanih slojeva*, PhD Thesis, Fakultet strojarstva i brodogradnje, Zagreb, 2004.

[17] TU, J.P.; LIU, M.S.; MAO, Z.Y.: *Erosion resistance of Ni-WC self-fluxing alloy coating at high temperature,* Wear, 209 (1-2), 43-48, 1997.

[18] VAMSI KRISHNA, B.; MISRA, V.N.; MUKHERJEE, P.S.; SHARMA, P.: *Microstructure and properties of flame sprayed tungsten carbide coating*, International Journal of Refractory Metals and Hard Materials, 20 (5-6), 355-374., 2002.

[19] WANG, H.; XIA, W.; YIN, Y.: *A study on abrasive resistance of Ni-based coatings with a WC hard phase,* Wear, 195 (1-2), 47-52, 1996.