Production Processes and Systems, vol. 6. (2013) No. 1. pp. 35-46.

HARDNESS TEST AND MICROSTRUCTURE ANALYSIS OF NiCrBSi SPRAYED, LASER REMELTED COATINGS

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Abstract
Thermal spraying is already used in industry to protect mechanical parts against wear and corrosion, but results are not always satisfactory due to porosity, microstructures and mainly bond strength. In this study, flame- (FS) and high velocity spraying (HVOF) and in situ flame – and laser irradiation by laser processes were combined to modify structural characteristics of metallic NiCrBSi coatings. The microstructure evolution was studied with the chemical composition analysis by XRD and SEM coupled with EDS techniques.

Results show that in situ flame and laser remelting induces the growth of a dendrite structure which strongly decreases the porosity of as-sprayed coatings, without solidification cracking (one of the major defects that can occur during the solidification of metallic or composite alloys) and improves the mechanical properties of the layer.

Keywords*: Flame spraying, deposit, flame remelting, laser remelting, NiCrBSi alloys, microstructure, micro-hardness test*

Introduction

The influence of processing conditions on microstructure and mechanical properties of NiCrBSi coatings deposited on a steel substrate has been studied. Three different NiCrBSi coatings deposited on a steel substrate have been investigated in order to find the relationships between processing conditions and microstructure / mechanical properties.

Flame spraying combined with laser treatment was applied. The microstructure of the coatings was examined by scanning electron microscopy (SEM) and energy dispersive X-ray microanalysis (EDX). Similar phases were observed in the three coatings. Laser cladded coatings exhibit the higher hardness and the smaller wear rate [1].

Ni-based coatings are used in applications where corrosion and wear resistance at moderate and elevated temperatures are required [2-5]. NiCrBSi alloy is one of the alloys with better performances [6-8]. The addition of Si and B increase the self-fluxing capabilities of the Ni alloy, improving its ability to produce coatings by melting process.

Boron addition reduces the melting point due to the presence of a eutectic phase at 3.6%. The broad solidification interval of the Ni alloys with high-boron content makes easier to get coatings by thermal spray process. Although Si is usually added to Ni–B alloys to increase the self-fluxing properties, the effect can be neglected with low contents, as those used in the NiCrBSi alloy studied [9]. The role of Cr is to improve the mechanical and wear properties as a consequence of its combination with others elements to produce hard precipitates [10]. Hardness (*H*) has been directly related with wear behaviour. Some evidences indicate than the elastic modulus (*E*) can also have a strong influence on wear, especially in the case of coatings. Therefore, different relations between these mechanical properties have been developed in order to predict the wear behaviour of materials with acceptable accuracy. It is well known that the *H*/*E* ratio (Hardness/Elastic modulus) can be considered as the material ability to bear elastic deformation [11].

Among the techniques applied to melt and spray material, flame spray and plasma spray processes are widely used. The coatings produced by flame spray are characterized by high porosity and poor adhesion to substrate. For that reason, this technique is commonly combined with a subsequent melting process. Laser cladding is coating technique usually employed to produce coatings for valuable components and products. It is characterized by producing coatings with a very low porosity and, consequently, better wear and corrosion resistance are expected [12 - 15].

Coating discontinuities are induced by the porosity, the presence of unmelted particles, and the interlamellar boundaries within the coating. In the practice it is not possible to achieve defect-free thermal spray coatings, which induce weak wear and corrosion resistance. Lasers present the advantage of building denser coatings, with finer microstructures [16]. Moreover, a laser post-treatment can be conducted on as-sprayed samples to increase their mechanical properties [17].

A laser post-treatment can generate some cracks in an as-sprayed coating, one of the major defects that can occur during the solidification of the metallic alloy, because of a high thermal gradient due to the high temperature of the small irradiated area [18]. In this study, atmospheric plasma spraying (APS) and laser irradiation by diode laser processes were combined in situ to modify structural characteristics of NiCrBSi coatings.

The technique is not considered new because it was applied in the past as a combination of plasma spraying and a continuous wave (CW) CO_2 laser irradiation [19]. However, generally this procedure was applied to ceramic layers to build coatings that exhibited distinctly reduced porosity, uniform microstructure, high hardness, and highly adhesive bonding to the substrate [20].

2. Experimental procedure

Figure 1 shows an experimental procedure of the two-step process: coating deposition using a flame spraying torch and coating remelting with laser. The two influencing preparing phases of substrate surface preparation, that is, surface preparation with particle blasting and preheating of the substrate for the reduction of thermal stresses, were used.

The spray process parameters chosen for experiments were the distance of the torch nozzle from the substrate surface and the mixture ratio of acetylene and oxygen. Specimens were tested for micro-hardness, microstructure and chemical analysis. The aim of the study is to characterize the microstructure of sprayed coating, with different NiCrBSi powders [21].

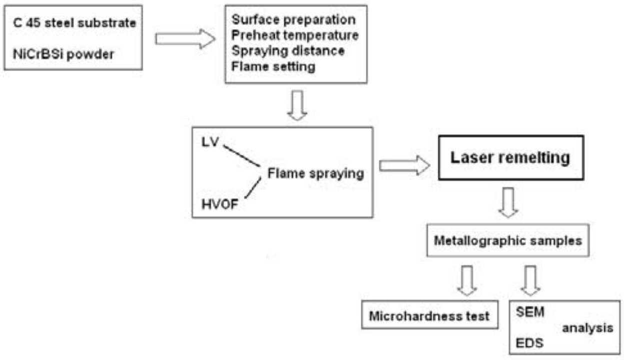

Figure 1. Experimental procedure plan

2. 1 Predeposition with thermal spraying

The application of a laser can improve the properties of thermally sprayed coatings. Improvements recently studied concern biomedical coatings, thermal barrier coatings, wear resistant composite coatings, corrosion resistant alloys, and wear resistant coatings engraved with a laser (anilox rolls). Most of the laser treatments correspond to the process of cladding, and only a few papers concern alloying or hard phase dispersion. Therefore, the following discussion aims to take into account the state of the sprayed coating at the laser processing stage (i.e.: the solid, liquid, and gaseous states) [22].

2.2 Powder materials

Three NiCrBSi super-alloy powders were used as coating material. The powders were composed of spherical particles with an average size of 66 μm. The substrate material was Ck 45 steel in the form of bar, with L = 300 mm and diameter of 50 mm in Table 1.

Table 1. Chemical composition and hardness of substrate steel

Grade of steel	Composition, mass %			Hardness HB	
	C	Mn	Si	Normalized	Hardened
C 45*	0.45	0.60	max 0.45	200 - 235	480 - 515

* MSZEN10083/2-91-A1-2000

NiCrBSi powders from BÖHLER – UTP referenced as ~~grades~~ UB 5-2560, and UB 5-2760 were chosen as deposit materials. The composition of the sprayed powders is listed in Table 2.

Table 2. Chemical composition and particle size of used NiCrBSi spray powders

Trade mark	Composition, mass %						Particle size μm	Hardness of coating HRC	Melting range C°
	Ni	Cr	Si	B	C	Fe			
UB 5-2560*	Balance	16.5	4.2	3.7	0.5	2.9	-125 - +45	55-60	976 - 1063
UB 5-2760*	Balance	15.0	4.4	3.2	0.75	3.5	-125 - +45	60	964 - 1003

*Powders of BÖHLER - UTP GmbH. Bad Krozingen, Germany

2.3. The flame-spray process

The surface of the bars were prepared by corundum blasting using angular Al_2O_3 1% particle with nominal grain size of 0.5 mm before applying the flame-spray deposition process in order to eliminate grease and oxide contamination, and to improve the adherence between the coating and the substrate.

Table 3. Flame spray parameters for the NiCrBSi powders

Thermal spray process	Acetylene flow rate l/min	Propane flow rate l/min	Oxygen flow rate l/min	Powder carrier gas (air) flow rate l/min	Spray distance mm	Spray velocity m/s	Scanning step mm
LV FS	24	-	36	-	160	150	5
HVOF	-	62	240	15	180	450	6

The flame-spray process was applied with a UNY-SPRAY-JET UTP and a UTP TOP GUN AIR HVOF guns at a pressure of 2.5 bar for the oxygen gas and of 1 bar for the acetylene. Powder consumption was 9.0 kg/h and the optimal spraying distance was 150-180 mm.

2.4. The melting process

After spraying, the flame-spray coatings were laser remelted. Incomplete melting along the full thickness of the coating means a lack of metallurgical bonding between substrate and coating, and is a frequent outcome of the process. An alternative technique used for post-treatment coating consists of using a laser beam to melt the flame-sprayed layer; in our experiment we used the two-stage method, (Figure 2).

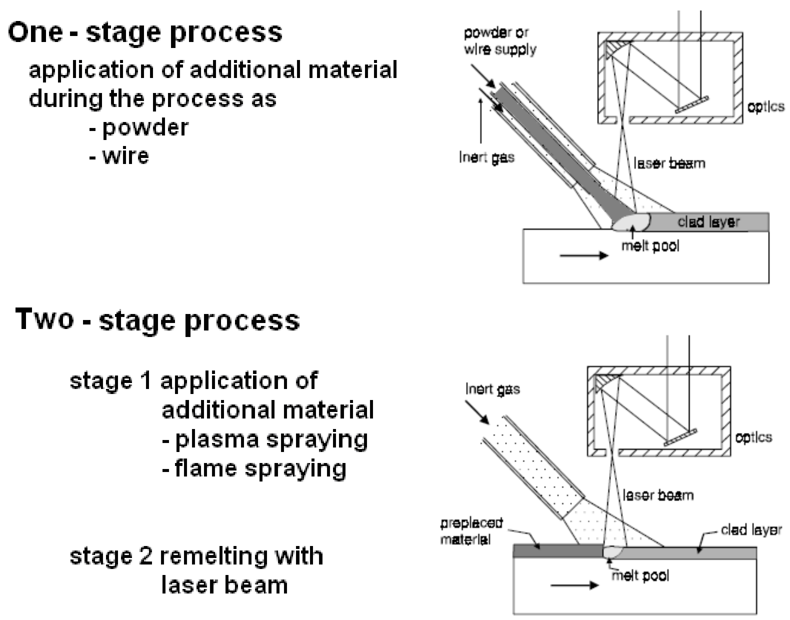

Figure 2. One-stage and two-stage method

The BAYATI TRUMPF TLC 105 5 kW CO_2 laser equipment with a nominal power of 1900 W was used to this end in our experiments and a Kugler LK - 190 laser focusing head at a focal distance of 125 mm was applied for thermal treatment. Equipment optics was protected by a 2 bar pressure cross current of air.

Table 4 CO_2 laser operating parameters

Frequency (f) Hz	Pulse duration (t_p) μs	Scanning speed mm/s	Defocusing (z) mm	Spot diameter (d) mm
1500	20	2	+100	3

The laser equipment was used in continuous mode with argon as protective gas. The different process parameters and their influence on the layers obtained were studied under the above conditions. The most satisfactory results were achieved with the following parameters: power density on the surface of the piece of 38 W/mm^2, beam scanning speed of 150 mm/min and a laser beam diameter of 3 mm. In addition, in order to reduce cracking risk, the test specimens were preheated in a furnace and their cooling rate after the laser treatment was slowed by placing them in perlite.

2.5. Measurement of hardness

Table 5. Vickers micro-hardness test results

Measuring place (measure 0.1 mm) (Loading 1.0 N)	Sample No. 005		Sample No. 007	
	Powder UB 5 - 2560		Powder UB 5 - 2760	
	FS Sprayed	Remelted	HVOF Sprayed	Remelted
	Vickers		Vickers	
1	531		725	
0.9	534		739	
0.8	509	886	721	590
0.7	492	884	715	578
0.6	493	880	705	583
0.5	500	863	695	578
0.4	517	892	690	572
0.3	525	865	697	567
0.2	491	759	693	602
0.1	495	822	689	577
0.0	285	449	284	408
-0.1	282	296	280	289
-0.2	256	289	279	268
-0.3	234	288	253	266
-0.4	227	284	250	255
-0.5	232	267	247	253
-0.6	245	247	254	255

Micro-hardness tests were carried out according to the ASTM E-384 standard with Mitutoyo MVK-H1 micro-hardness tester (Table5).
Scanning electron microscope techniques with energy dispersive spectroscopy ZEISS EVO MA 10 instrument (SEM–EDS) were used for metallographic investigation and to analyse the coatings microstructures (Figure 9 – 11). Specimens for microstructural analysis were firstly ground and polished to a mirror finish and then etched with solution of $1HCl:10HNO_3:10H_2O$.

3. Results and analysis

3.1 Layer micro-hardness results and microstructure

The obtained values of micro-hardness (Table 5) of the as sprayed and the laser remelted coatings, depending on the used powders are shown in Figure 3-6.

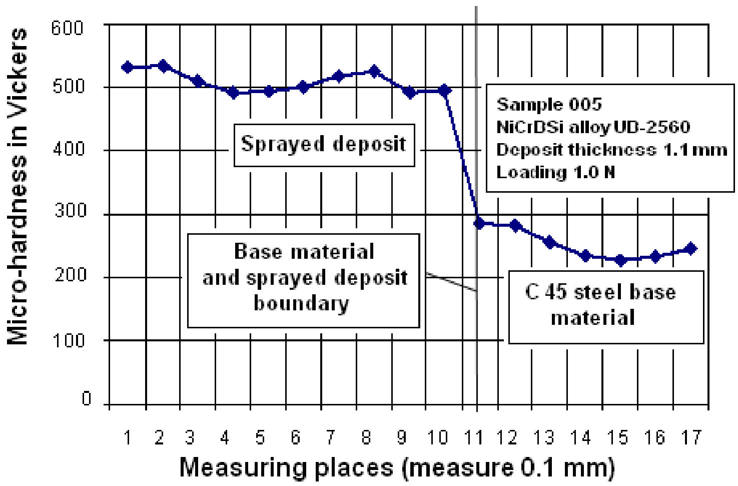

Figure 3. Micro-hardness of UB5-2560 NiCrBSi as-sprayed coating vs. distance from the surface

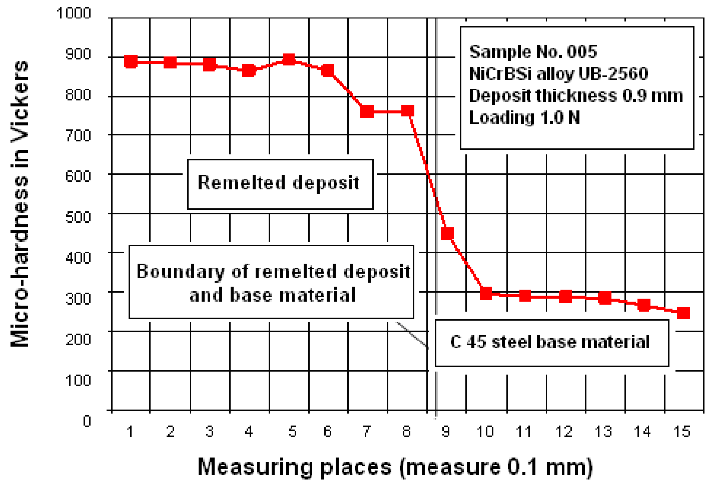

Figure 4. Micro-hardness measuring results of the UB5-2560 NiCrBSi laser remelted coatings as a function of the distance from the surface

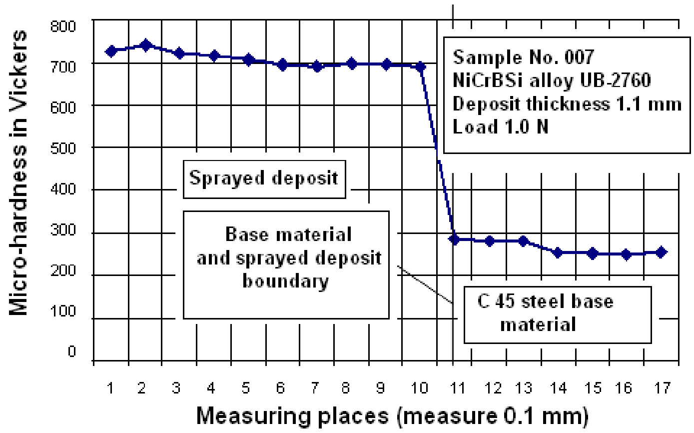

Figure 5. Micro-hardness of UB5-2760 NiCrBSi as-sprayed coating vs. distance from the surface

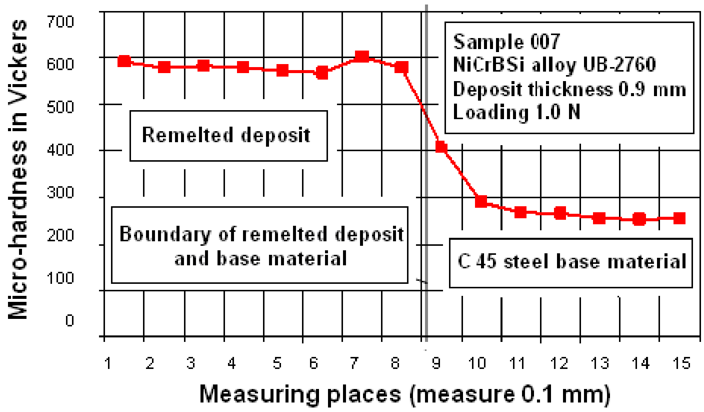

Figure 6. Micro-hardness measuring results of the UB5-2760 NiCrBSi laser remelted coatings as a function of the distance from the surface

3.2. Microstructure

Figure 7. illustrates the microstructure of a LV flame-sprayed NiCrBSi coating (Powder UB – 2560). Figure 8. illustrates the microstructure of a HVOF flame-sprayed NiCrBSi coating (Powder UB – 2760). Layer thickness is approximately 1.1 mm. The high porosity of the layers can be clearly seen (10 vol.%), as can the irregular profile of the interface, a consequence of the shot blasting process applied, with no metallurgical bond between the layer and the substrate.

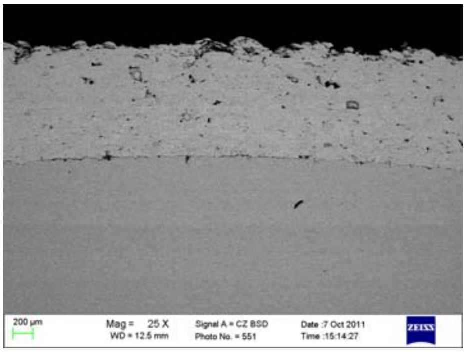

Figure 7.LV flame sprayed NiCrBSi coating (M=25x)

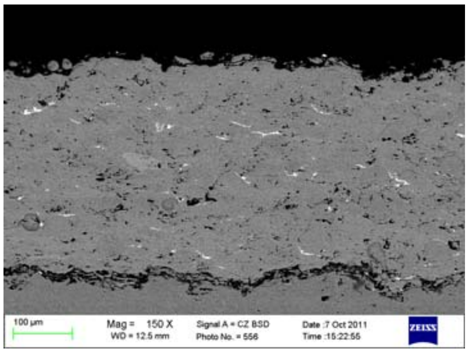

Figure 8. HVOF flame sprayed NiCrBSi coating (M=150x)

Fig. 9 and 10 show the structure obtained after laser remelting. It can be clearly seen that the matrix phase is a solid solution of Ni with some Cr and Fe providing a remelted structure; there is an interdendrite lamellar eutectic phase made up mainly of Ni and small amounts of Si. The surface is dotted with very hard precipitates, mainly of Cr (approximately 80 % by mass). This confirms the tendency of these alloys to form carbides and borides, thus supporting the results for similar alloys obtained by other researchers [23–24], who have identified these precipitates as chromium carbides and borides (mainly Cr_7C_3 and CrB) in a Ni solid solution.

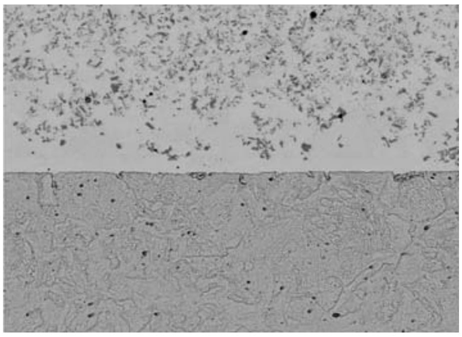

Figure 9. Microstructure of a HVOF flame sprayed NiCrBSi coating fused by laser (M=50x).

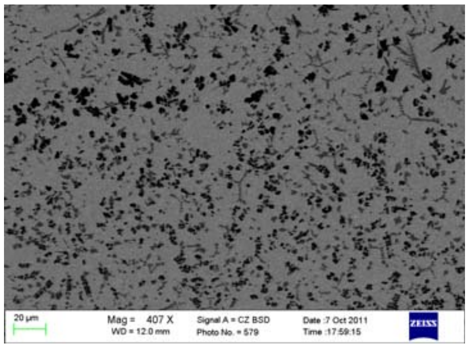

Figure 10. Microstructure of a LV flame sprayed NiCrBSi coating fused by laser (M=407x).

3. 2 SEM - EDS analysis

Composition profiles of NiCrBSi UB5 – 2560 and UB5 – 2760, + laser remelted samples were measured by EDS on the cross section. The EDS analysis of FS-sprayed and in remelted samples shows a similar evolution of the chemical composition (Figure 11-12). (These profiles were not recorded on the complete thickness of the samples, as the chemical composition was uniform across the whole thickness of the coatings). The EDS analysis reveals also a high Cr content of the layer, probably due to the formation of Cr carbides and borides during the remelting.

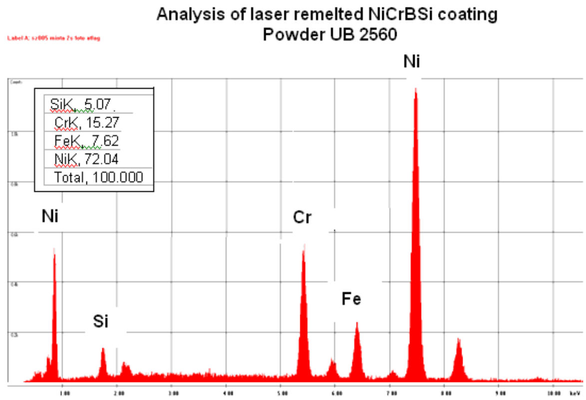

Figure 11.
Details of the coating interfaces: LV FS + laser remelted

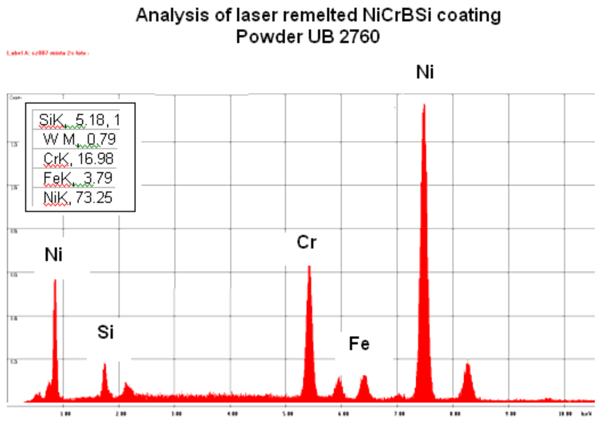

Figure 12.
Details of the coating interfaces: HVOF FS + laser remelted

5. Conclusions

In this work, four different thermal sprayed NiCrBSi coatings have been compared: UB5 – 2560 LV flame sprayed, UB5 – 2760 HVOF flame sprayed with a subsequent remelting treatment of laser (FS + laser). Their micro-hardness and microstructure, properties were studied.

• *Micro-hardness* was measured by micro-Vickers. A fine uniform distribution of equated precipitates increase the hardness, as it can be observed in laser remelted coatings. The non-homogeneous distribution of precipitates decreases the hardness values in FS + laser coatings.
• All coatings show a similar microstructure composed mainly of a Ni solid solution matrix with melted structure and common phases precipitated on it with different distribution and size. Laser remelted coatings show a uniform distribution of small and rounded precipitates, which results in a harder material.
• Results show that in situ laser remelting induces the growth of a dendrite microstructure that strongly decreases the FS-sprayed coating porosity.
• The in situ process allows the construction of denser coatings than FS with finer structures without cracks and porosity. The coating is metallurgical bonded to the substrate, which probably will increase the adhesion.

Acknowledgments

The authors gratefully acknowledge the BÖHLER – UTP Division Ltd. in Hungary for its technical support in this project, TAMOP-4.2.1/B-09/1/KONV-2010-0003: Mobility and Environment: Researches in the fields of motor vehicle industry, energetic and environment in the Middle- and West-Transdanubian Regions of Hungary. The Project is supported by the European Union and co-financed by the European Regional Development Fund.

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