

## Formation of microstructure of spray-fused powder coatings

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**Abstract.** The paper describes a NiCrSiB self-fluxing alloys based system, containing particles of tungsten carbide or those of WC-Co hardmetal from the used hardmetal as hard phase, deposited by the spray and fusion processes. Flame and laser spray fusions were used as coating technologies. The study focuses on powder composition, hard phase grain size, processes of deposition, the effect of the composition and process on the formation of the coating structure, and on the evaluation of wear resistance of coatings. We discuss the dependence of the wear resistance of spray-fused coatings on their structure and propose structure criteria for coating selection. The “double cemented” matrix structure of self-fluxing NiCrSiB based coatings, which contain WC-Co particles, was found optimal in impact erosion wear conditions. The paper also discusses the cost-effectiveness of coatings in application areas that are sensitive to the cost and which prefer composite coatings based on low cost recycled materials.

**Key words:** thermal spray, flame spray and fusion, laser spray and fusion, composite coatings, impact erosion, wear.

### 1. INTRODUCTION

Wear-resistant thermal spray coatings intended for sliding wear are hard but brittle. Under the impact loading conditions of wear, sensitive to fatigue sprayed hard coatings are useless. In this case, under extreme conditions of erosive wear (high hardness of abrasive and high velocity of abradant particles), composite spray-fused coatings ensure optimal properties of hardness-toughness. Such

erosive wear occurs in different types of milling and mixing equipment, with predominant cyclic impact loading. It has been shown that under impact loading, for example, by collision milling in disintegrators [1] or by wear in the stream of hard particles [2], materials are exposed to notable strokes. The stresses generated in the particles to be ground or in the materials subjected to impact erosion are approximately an order higher than their strength. As a result, material fractures.

The main properties of coatings under impact erosion conditions are their low porosity and optimal hardness-toughness. With thermal spray methods, only using high-velocity spraying, minimum porosity (in the range 0–3%) is obtainable [3]. By impact erosion with normal impact, such as under extreme conditions of erosive wear, an increase in the coating hardness causes an increase in the wear rate. Direct fracture or low-cyclic fatigue fracture are predominating mechanisms of the fracture and, as a result, abraded material is removed [4]. By oblique-impact erosion, microcutting or fatigue fracture is dominant.

To guarantee high abrasion-erosion wear resistance of coatings, a framed structure with maximum hardness and maximum hard phase content by oblique-impact erosion and a matrix structure with optimal hardness and hard phase content by normal impact conditions should be established as their structural criteria [5].

In the conditions of mixed wear (small, medium, and high impact angles), an optimal structure of sprayed powder coatings of a system of tungsten carbide-cobalt, instead of a simple metal matrix consisting of WC, is a metallic (cobalt) matrix based structure containing particles of WC-Co hardmetal (“double cemented” matrix structure) [3]. In conventional WC-Co hardmetal coatings by abrasion-erosion direct fracture of carbides takes place. With the double cemented matrix structure, fracture toughness of the hard phase (WC-Co particles) is increased due to the ductile binder phase of the cobalt between small WC grains. Small WC particles or other secondary hard phases in the matrix increase resistance to microcutting and cyclic fatigue.

## 2. EXPERIMENTS

*Materials and coating methods.* As a basic component, a metallic matrix of composite coatings, commercial Ni-based self-fluxing alloy powders were used (Table 1). In the hard phase of composite coatings, hardmetal powder from used hardmetal, produced by disintegrator milling, was used. Chemical composition and granulometry of the powder is given in Table 1. A high content of iron in this hardmetal powder is due to the milling process. For comparison, WC-Co agglomerated granules of the HCST Amperit, as a hard phase of coatings, was also used. Selected powder composites for spray-fusion are given in Table 2. Hard phase content varied from 15 to 50%.

**Table 1.** Coating materials, their composition and hardness

No.	Material	Composition, wt %	Granulometry, $\mu\text{m}$	Hardness, HV
Matrix metal – self-fluxing alloys				
1.	12494 <sup>1)</sup>	NiCr11Si2B2	+60–160	430
2.	12495 <sup>1)</sup>	NiCr13Si4B3	+60–160	560
3.	12496 <sup>1)</sup>	NiCr15Si4.5B3.5	+60–160	930
4.	Metco 16C <sup>2)</sup>	NiCr16Si4B4Cu3Mo2.5Fe0.5	+44–127	60HRC
Hard phase hardmetals				
5.	Desirec VK15 <sup>3)</sup>	WCCo14Fe8	+45–125	1400
6.	WC-Co <sup>2)</sup>	WCCo12	+5–45	1300

<sup>1)</sup> Castolin SA

<sup>2)</sup> Sulzer Metco Inc.

<sup>3)</sup> Recycled disintegrator milled hardmetal powder, TTU, Estonia

**Table 2.** Selected composite coatings and their composition, deposition techniques, porosity and hardness

No.	Material	Deposition technique	Porosity, %	Hardness, HV0.2
7.	12494 + 25 wt % VK15 <sup>1)</sup>	FSF <sup>3)</sup>	2–3	675/1410 <sup>6)</sup>
8.	12495 + 15 wt % VK15	FSF	2–3	775/1410
9.	+ 25 wt % VK15	FSFF <sup>4)</sup> , FSF	2–3	685/1545
10.	+ 50 wt % VK15	FSF	2–3	735/1465
11.	12496 + 25 wt % VK15	FSF	2–3	820/1410
12.	Metco 16C + 30 wt % VK15	LSF <sup>5)</sup>	3.2	640/1300
13.	Metco 16C + 30 wt % WC-Co <sup>2)</sup>	LSF	0.5	620/1300

<sup>1)</sup> Desirec VK15 (No. 5, Table 1)

<sup>2)</sup> Sulzer Metco WC-Co (No. 6, Table 1)

<sup>3)</sup> FSF: Flame Spray Fusion

<sup>4)</sup> FSFF: Flame Spray Furnace Fusion

<sup>5)</sup> LSF: Laser Spray Fusion

<sup>6)</sup> Hardness of metal matrix/hard phase

The following technologies and equipment for depositing the coatings were used:

a) flame spray fusion (FSF), using Euro-Set XS-8 gas flame gun and oxy-acetylene flame;

b) laser spray fusion (LSF), using continuous wave-optical fibre coupled HAAS HL 4006D Nd: YAG laser device for industrial material processing [6].

By LSF, deposition velocity was 500–600 mm/min, shield gas – Ar with the flow rate 7.5 l/min, powder feed – 30 g/min. Coatings were deposited on structural steel of 0.45% C content as the substrate material. Thickness of the coatings ranged from 0.5 to 1 mm.

*Determination of the chemical composition of coatings.* Chemical composition of coating constituents was determined by the XRD analysis. Distribution of the elements in coatings was analysed by the mapping of basic elements (W, Co, Ni, Cr). As a result, the influence of the initial composition of composite powders and deposition processes on the chemical composition of coating constituents was determined.

*Porosity and microhardness measurements.* Porosity of spray-fused coatings was determined using image analysis programme Image Pro. Plus [7,8]. Microhardness of the coatings and their constituents was measured by microhardness-meter Micromet 2001. Twelve to fifteen hardness measurements were taken.

*Wear testing of coatings.* Wear testing was conducted by a centrifugal impact erosion tester [9]. Parameters were as follows: abrasive – quartz sand of 0.1–0.3 mm (1100–1200 HV), velocity of abrasive particles 80 m/s. Loss of weight was measured, volumetric wear rate per 1 kg of abradant (mm<sup>3</sup>/kg) and relative wear resistance were calculated. Reference material was normalized steel of 0.45% carbon content (200 HV). Number of specimens for each wear test experiment was three.

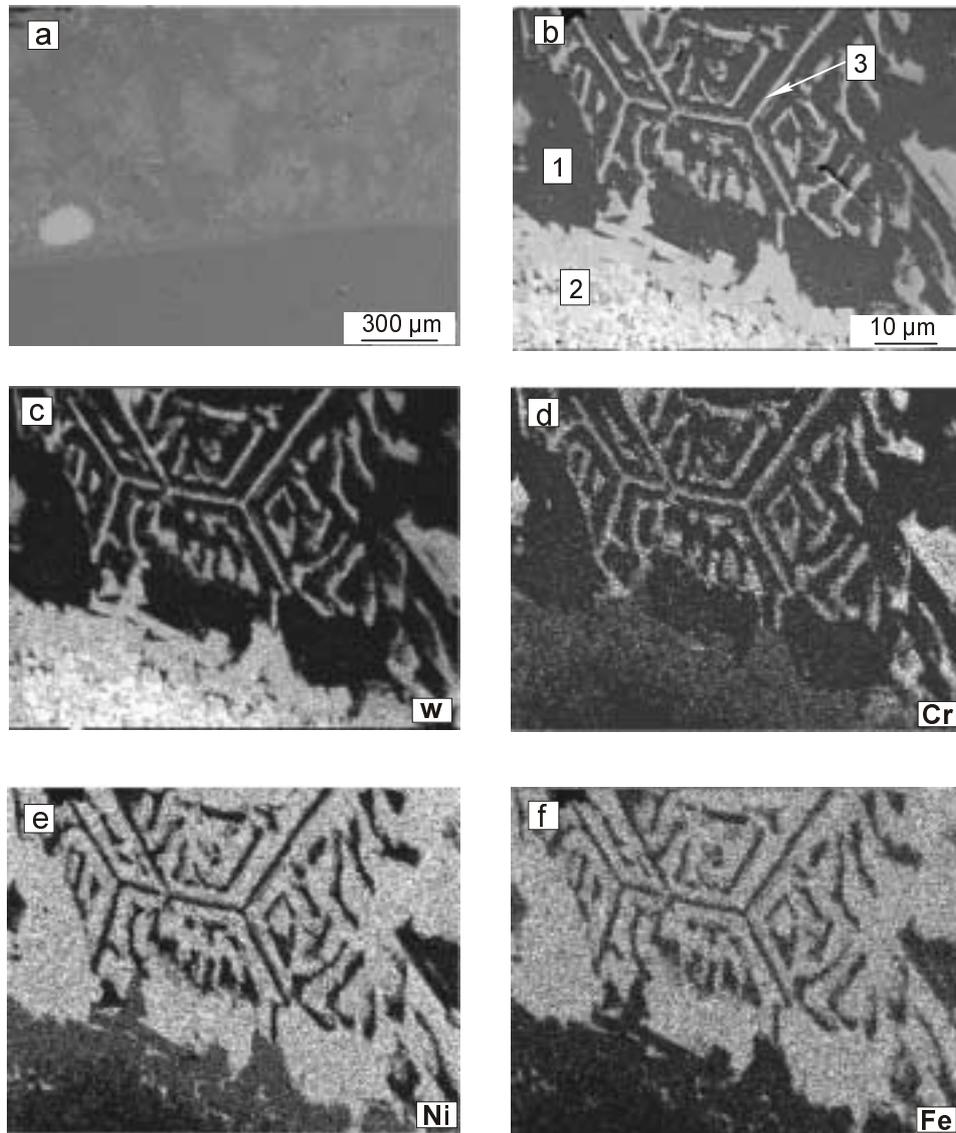
### 3. RESULTS AND DISCUSSION

#### 3.1. Influence of spraying powder composition and coating technology on the coating structure and properties

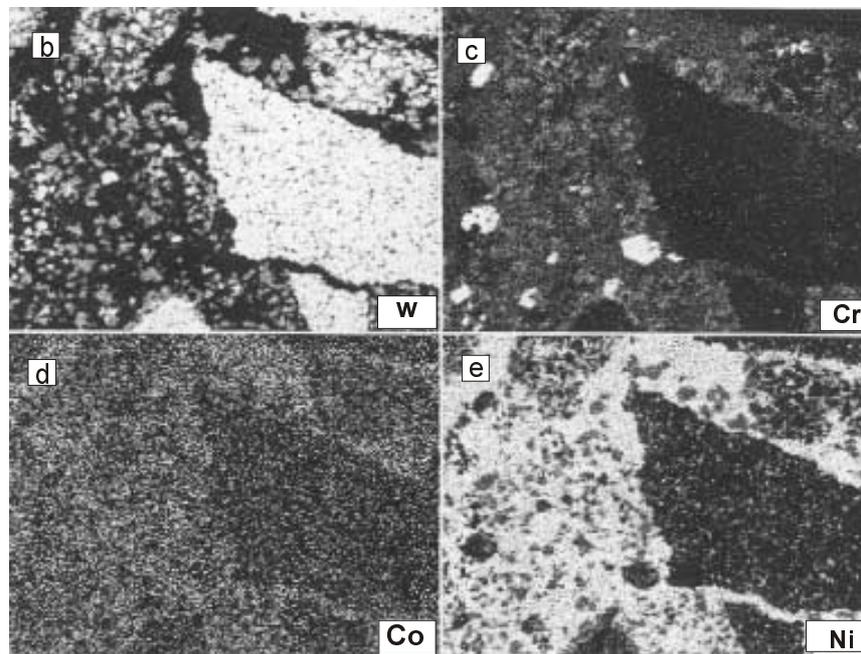
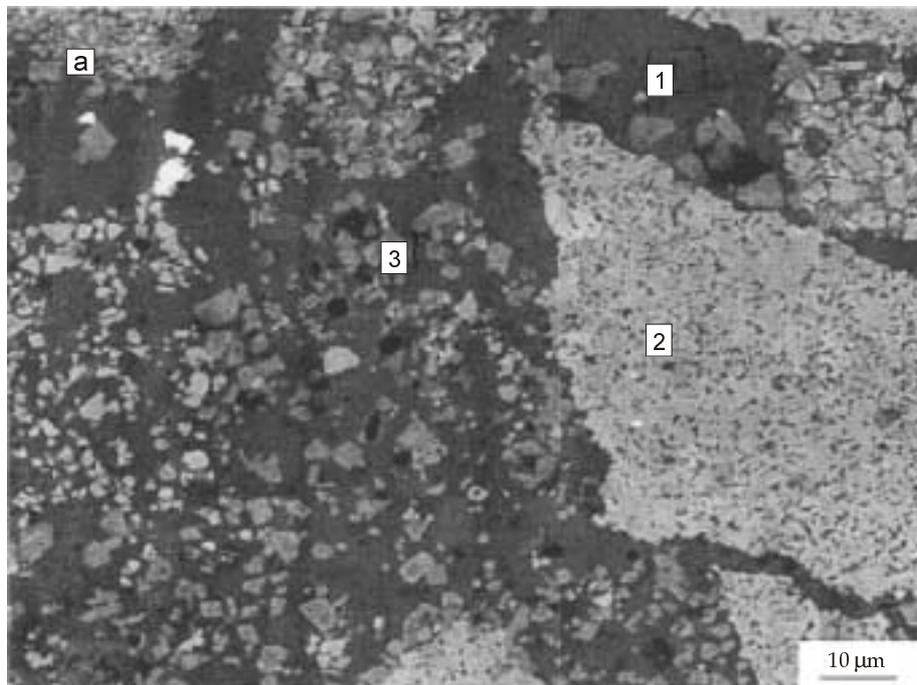
The microstructure of self-fluxing NiCrSiB alloy coatings deposited by FSF consisted of a solid solution on the basis of Ni, eutectic from borides of Ni (NiB, Ni<sub>2</sub>B) and the solid solution. Coatings from powders 12495 and 12496 contained additional borides of Cr (CrB), of Ni (Ni<sub>2</sub>B), carboborides of Cr and Ni, and silicides of Ni (Ni<sub>3</sub>Si). Also additions of slag (oxides of B, Si and Cr) were present in the coatings. It is similar to the results given in [10].

The structure, amount and composition of reinforcements in the metallic Ni-based matrix depends on the fusion method. As it was shown in [11], by fusion of NiCrSiB-(WC-Co) coatings in the furnace (duration of heating about 15–20 min at 1200 °C), a substantial decomposition of hardmetal particles and formation of dendrites in the Ni-based matrix takes place (Figs. 1c–d).

A typical microstructure of FSF NiCrSiB self-fluxing alloy based coating, containing particles of WC-Co hardmetal, differs from the structure of furnace-fused NiCrBSi-(WC-Co) coating. As it follows from the XRD analysis, Ni-based matrix (about 70%) was additionally alloyed with tungsten and cobalt (obtained from partially dissolved hardmetal particles (Figs. 2b and d, Table 3)), and consisted of small particles – reinforcements in the matrix (about 12%). These small particles were mainly grains of WC of dissolved WC-Co hardmetal particles (Figs. 2b and d, Table 3). It was confirmed by the measurement of microhardness of different phases (Table 4).



**Fig. 1.** Micrographs of the cross-section of FS furnace-fused NiCrSiB (12495) + 25 wt % VK15 (WC-Co) coating (a, b) and distribution of elements in it (c, d, e, and f).



**Fig. 2.** Micrograph of the cross-section of FSF NiCrSiB (12495) + 25 wt % VK15 (WC-Co) coating (a) and distribution of elements in it (b, c, d, and e).

**Table 3.** Chemical composition of spray-fused NiCrSiB self-fluxing alloy based coatings

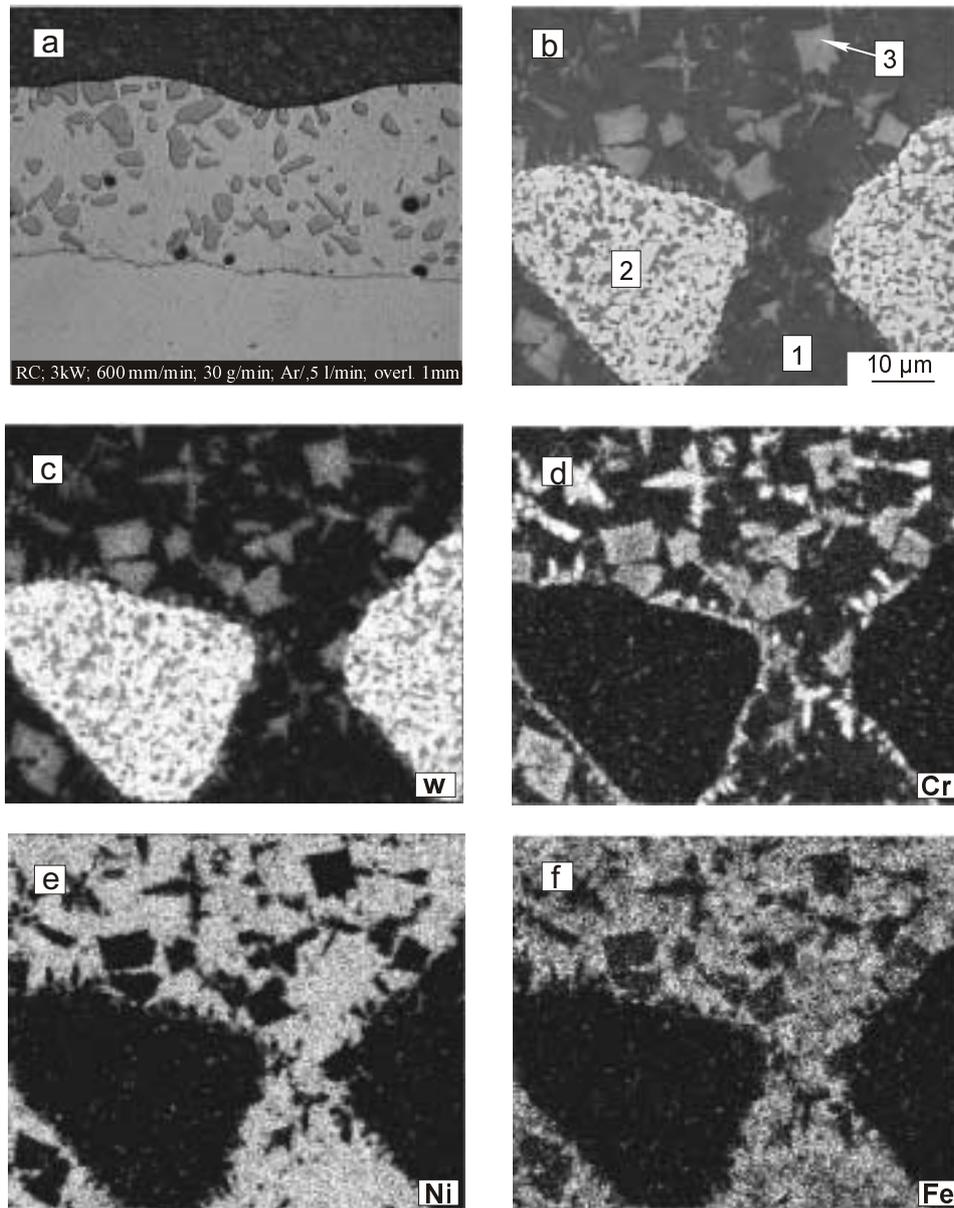
Type and coating material	Chemical composition and vol. %		
	Matrix phase (No. 1, Figs. 1, 2, 3, and 4)	Hard phase (No. 2, Figs. 1, 2, 3, and 4)	Reinforcements in matrix (No. 3, Figs. 1, 2, 3, and 4)
FSFF NiCrSiB (12495) + 25 wt % WC-Co (VK15)	77.0 Ni; 15.1 Cr; 3.6 Si; 3.6 Fe Others: B, etc. 80%	74.0 W; 8.9 Co; 6.4 Ni; 4.0 Ti Others: Fe, C, etc. 8%	Dendrites 54.1 W; 13.1 Ni; 2.4 Co Others: Cr, Fe, C, etc. 12%
FSF NiCrSiB (12495) + 25 wt % WC-Co (VK15)	42.8 Ni; 26.8 Fe; 13.8 Cr; 4.2 Co Others: W, C, etc. 71%	74.0 W; 8.9 Co; 6.4 Ni; 4.0 Ti Others: Fe, C, etc. 17%	WC particles 55.0 W; 20.0 Co; 8.0 Ni; 7.0 Mo Others: Fe, C, etc. 12%
LSF NiCrSiB (Metco 16C) + 30 wt % WC-Co (VK15)	60.0 Ni; 22.0 Fe; 5.5 Cr; 5.0 Co Others: Si, Fe, etc. 73%	74.0 W; 8.9 Co; 6.4 Ni; 4.0 Ti Others: Fe, C, etc. 18%	Dendrites 55.0 W; 20.0 Co; 8.0 Ni; 7.0 Mo Others: Fe, C, etc. 9%
LSF NiCrSiB (Metco 16C) + 30 wt % WC-Co (Metco)	70.1 Ni; 9.0 Cr; 7.0 Co; 4.0 Si; 3.0 Fe; 3.0 Cu Others: W, Mo, etc. 61%	78.0 W; 12.0 Co Other: C 18%	Dendrites 61.0 W; 14.0 Ni; 13.0 Cr; 10.0 Mo Others: Fe, C, etc. 21%

**Table 4.** Hardness of different phases of NiCrSiB (12495) + 25 wt % WC-Co (VK15) (No. 9) spray-fused coatings

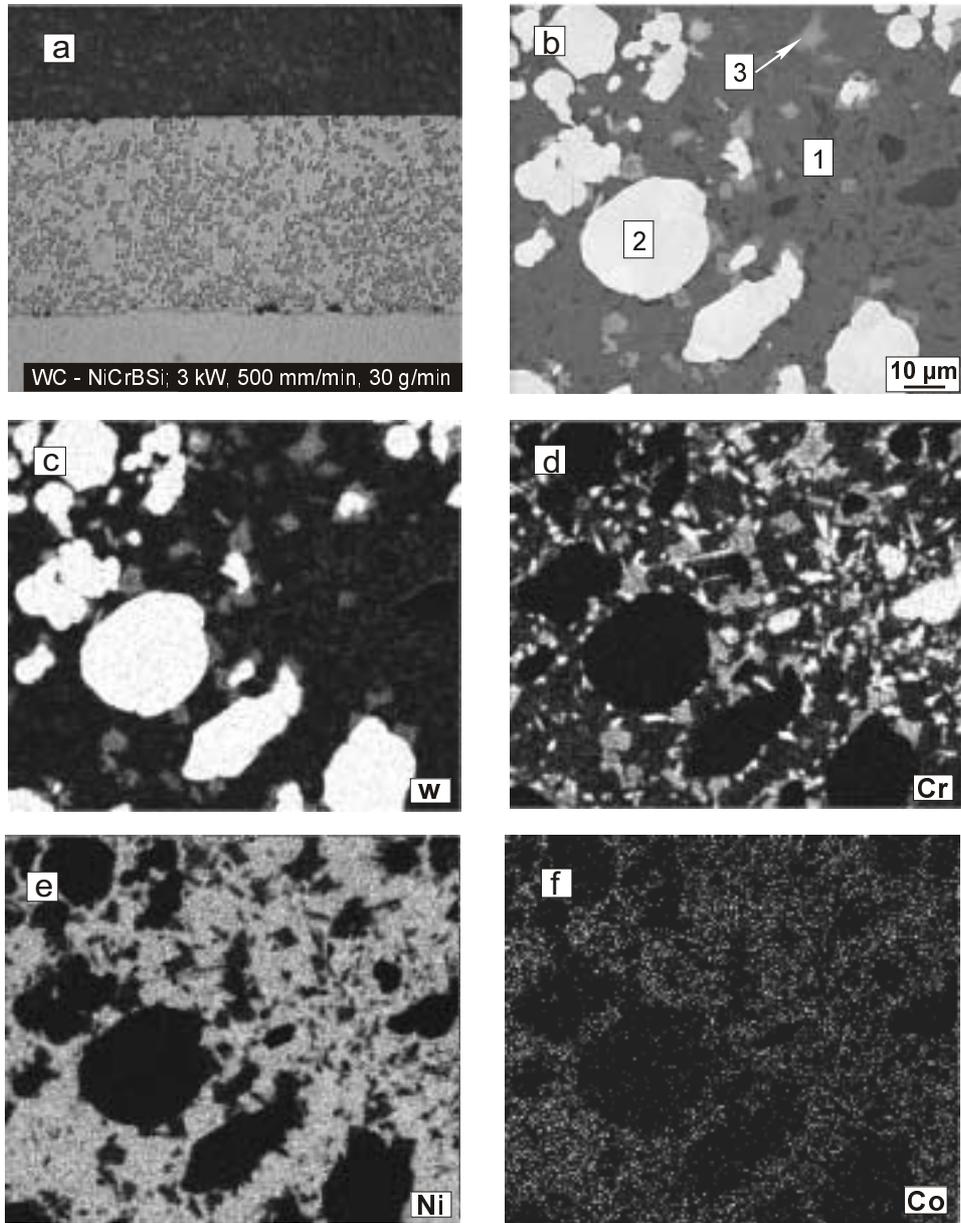
Phase	No. of phase (see Figs. 1–4)	HV 0.05
Ni-based metallic matrix	1 (Figs. 1b, 2a, 3b, 4b)	400–450
WC-Co hardmetal particles	2 (Figs. 1b, 2a, 3b, 4b)	1100–1200
Reinforcements in matrix		
dendrites	3 (Figs. 1b, 3b, 4b)	380–450
carbides	4 (Fig. 2a)	1400–1450

With LSF coatings, the microstructure (Figs. 3a and b) differed slightly from the structure of an analogous FSF coating. The amount of WC-Co particles was about 20% (Table 3) and the amount of reinforcements in the matrix was lower (below 10%). As it follows from the XRD analysis, the metal matrix structure is similar to the composition of FSF coatings, but the amount of micro-sized tungsten carbide (WC) particles within the metal matrix was minimal. Using agglomerated granules of WC-Co as a hard phase of the LSF coating, the small

WC particles were not found within the Ni-based matrix and the amount of reinforcements, dendrites, was bigger (about 20%) and its Cr content higher (Fig. 4, Table 3).



**Fig. 3.** Micrographs of the cross-section of LSF NiCrSiB (Metco 16C) + 30 wt % VK15 (WC-Co) coating (a, b) and distribution of elements in it (c, d, e, and f).



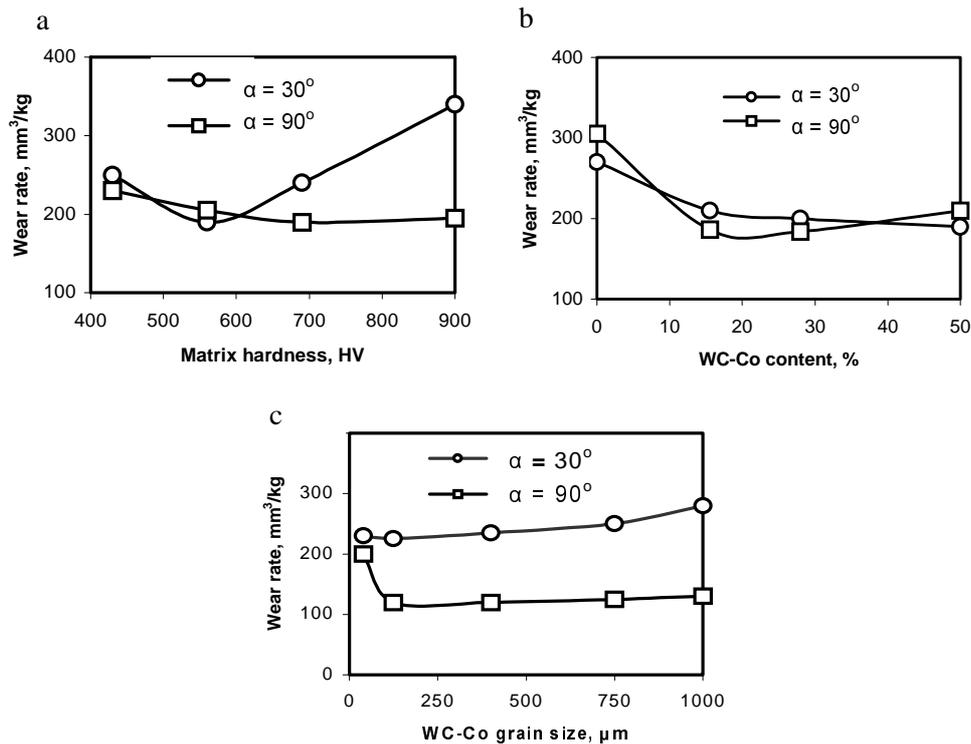
**Fig. 4.** Micrographs of the cross-section of LSF NiCrSiB (Metco 16C) + 30 wt % (WC-Co) coating (a, b) and distribution of elements in it (c, d, e, and f).

The difference in wear resistance of coatings from self-fluxing Ni-base alloys of different hardness was up two times, as shown in Table 5 and in [12]. The effect of the NiCrSiB matrix hardness and WC-Co content on the wear resistance of composite coatings was insignificant (Fig. 5). Depending on the hardness of

the matrix metal (Fig. 5a), NiCrSiB-(WC-15Co) coatings exhibited a higher wear rate which differed by 30 to 40%, because the matrix phase hardness is higher than that of a matrix without hardmetal particles (coating base 12494 and 12495 alloys). Higher hardness is attributed to dissolved hardmetal particles within the Ni-base matrix. In practice, microhardness of different Ni-base matrices with dissolved hardmetal micro-sized particles varied slightly (Table 2).

**Table 5.** Relative abrasion-erosive wear resistance of unreinforced coatings

Coating code	Hardness HV	Relative volume wear resistance $E_v$	
		$\alpha = 30^\circ$	$\alpha = 90^\circ$
12494	430	1.3	0.8
12495	560	1.3	0.6
12496	930	1.6	0.4



**Fig. 5.** Dependence of the wear resistance of FSF NiCrSiB + VK15 (WC-Co) coatings on matrix hardness (a, WC-Co content 25 wt %), on hard phase content (b, matrix 12495), and on hard phase particles size (c, matrix 12495, WC-Co content 25 wt %).

Figures 5b and c show the effect of the amount of hard phase (from 15 to 50 wt %) and hard phase particle size (+60–500  $\mu\text{m}$ ) on the wear resistance of composite coatings. According to the principles of developing abrasion-erosion resistant coatings, an increase in the amount of hardmetal particles of composite coatings led to an increase in the wear resistance under the wear conditions of oblique impact similar to sliding wear. Under operating conditions by normal impact, as a result of an increase in the hard phase content, the wear resistance of the coatings decreased.

The influence of the hard phase particle size on the wear resistance of a coating (Fig. 5c) in the range of the hardmetal particle size studied was insignificant.

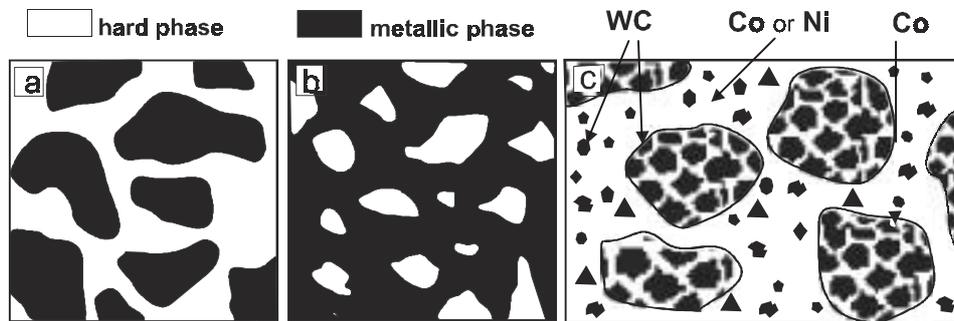
### 3.2. Optimal structure of coatings

Analysis of the results of wear testing revealed a direct dependence between the wear resistance and the microstructure of materials. The wear resistance of coatings, containing WC-Co hardmetal particles, was higher than that of coatings with agglomerated WC-Co granules (Table 6).

In the case of oblique-impact erosion (by small and medium impact angles), when the wear rate decreases with an increase in the hardness and the mechanism of microcutting is dominant, a framed structure is preferred (Fig. 6a). The hard phase content must exceed 50%. By normal impact, the matrix structure with the hard phase content lower than 50% is preferred (Fig. 6b). By mixed impact erosion, such as in different mixing and grinding equipment, the optimal structure of WC-Co and other carbide-metal based spray-fused hardmetal coatings (instead of a simple cobalt matrix, containing particles of WC or other carbides) is cobalt (nickel) matrix based structure which contains particles of WC-Co (or other hardmetals) agglomerated granules or particles of WC-Co (or other carbides based) hardmetal<sup>[4]</sup>. It is the “double cemented hardmetal structure” (Fig. 6c). A similar structure is obtainable with hardmetal powders coated with metal (cobalt or nickel) and by the HVS method. As it was shown by experiments, another way to manufacture such complicated cemented structures is spray and fusion of composite powders based on WC-Co or other carbide

**Table 6.** Impact erosion wear resistance of coatings

No.	Coating material	Method of deposition	Relative wear resistance $E_v$	
			$\alpha = 30^\circ$	$\alpha = 90^\circ$
2.	NiCrSiB (12495)	FSF	1.3	0.6
4.	NiCrSiB (Metco 16C)	LSF	1.4	0.6
9.	NiCrSiB + 25 wt % VK15	FSF	1.9	0.6
12.	NiCrSiB + 30 wt % VK15	LSF	2.1	0.5
13.	NiCrSiB + 30 wt % WC-Co	LSF	1.6	0.3
	NiCrSiB (Tafa 1275)	HVOF	0.6	0.3



**Fig. 6.** Optimal structures of coatings: a – framed structure; b – matrix structure; c – “double cemented” matrix structure of a coating in impact erosion conditions.

based hardmetal powders and, for example, NiCrSiB self-fluxing alloy powder. The resulting structure consists of WC-Co hardmetal particles in the Ni-alloy based matrix with small dissolved tungsten carbide particles (Fig. 2a) [4,5,11].

Similarly to normal impact erosion, direct fracture or low-cyclic fatigue fracture mechanism was predominant; as for residual stresses in coatings, compressive stresses are favourable. Such a compressive stress in coatings is obtained by HVOF-spraying or by thick composite coatings manufactured by spray and fusion [11].

### 3.3. Application areas and cost-effectiveness of coatings

In the application areas that are more sensitive to the cost factor (restoration of worn machine parts, strengthening of large areas of parts, etc.), the proposed composite powders on the basis of NiCrSiB self-fluxing alloy powders and recycled hardmetal powders are most attractive for thermal sprayed coatings. As it follows from Table 7, the relative cost of FSF composite NiCrSiB-(WC-Co) coatings is up to 4 times lower than that of analogous HVOF sprayed WC-Co wear-resistant coatings with similar wear-resistance characteristics.

**Table 7.** Cost-effectiveness of NiCrSiB – VK15 FSF coatings<sup>1)</sup>

Type of materials	Cost of material <sup>2)</sup> , EEK	Total cost, EEK	Relative cost	Relative wear resistance in disintegrator <sup>4)</sup>
NiCrSiB (Castolin, 12495)	1.2	3.0	1.0	1.5
NiCrSiB + 25 wt % VK15	1.2 (0.9 + 0.3)	3.2	1.1	2.5
WC-17Co (Tafa 1343) <sup>3)</sup>	4.4	11–12	4.0	3.0
NiCrSiB (Tafa 1275) <sup>3)</sup>	1.2	4.5	1.5	1.5

<sup>1)</sup> Basis of calculation: coating with area 1 cm<sup>2</sup> of 1 mm thickness

<sup>2)</sup> On the basis of prices of powders of Castolin SA and Tafa Inc.; calculated price of recycled hardmetal powder is in the range of 700–800 EEK/kg

<sup>3)</sup> For comparison: HVOF sprayed (Machine Mounted System Tafa JP5000)

<sup>4)</sup> Results of comparison testing in disintegrator DESI; treated material – quartz sand (1100–1200 HV); reference material – low carbon steel of 0.45%C

#### 4. CONCLUSIONS

1. New composite spraying powders and coatings on the basis of NiCrSiB self-fluxing alloy powder and recycled hardmetal powder intended for deposition by the spray-fusion method have been proposed.

2. The influence of spraying powder composition and spray-fusion methods on the structure and properties of fused composite coatings has been determined. FSF enabled us to produce NiCrSiB alloy based composite coatings containing WC-Co hardmetal particles in dispersion-strengthened with WC and dendrites Ni-based matrix, the “double cemented” matrix structure.

3. In terms of high-impact erosion wear resistance, an optimal coating structure gave high wear resistance under mixed wear (impact angles range from 0 to 90°). Relative wear resistance was up to 2–3 times higher than that of the pure NiCrSiB coating.

4. The cost-effectiveness of the proposed composite powders and coatings based on them has been demonstrated. The potential application areas, more sensitive to the cost, such as restoration of worn parts, strengthening of rapidly wearing large parts of earth-removing machines, etc., are recommended.

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## **Pihustus-sulatuspinnete struktuur ja omadused**

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On vaadeldud iseräbustuvatel nikli-, kroomi-, räni- ja boorisulamitel baseeruvaid ning kõvafaasina volframkarbiidi või kasutatud volframkarbiidsest kõvasulamist pulbriosakesi sisaldavaid pihustamise ja järgneva sulatamise teel valmistatud pindeid. Pindamismoodustena kasutati leek- ja laserpihustussulatust. Põhitähelepanu pühendati komposiitpulbrite koostisele, kõvafaasi osiste suurusele ja pindamismoodustele ning pulbrite koostise ja pindamistehnoloogia mõjule pinde struktuuri formeerimisel ning pinde kulumiskindlusele. Vaadeldi pihustus-sulatuspinnete struktuuri mõju pinnete kulumiskindlusele ning pinnete valiku kriteeriumeid. Optimaalse struktuuriga pinneteks löökerosioonkulumise korral osutusid volframkarbiidkõvasulami osistega armeeritud iseräbustuvate niklisulamite baasil valmistatud nn topeltsementeeritud maatriksstruktuuriga pinded. Parimate pinnete kulumiskindlus löökerosiooni tingimustes kalderosioonil ületab etalonmaterjali – süsinikterase – kulumiskindlust 2–3 korda. On käsitletud ka nikli baasil valmistatud iseräbustuvate sulamite pulbrite ja odava teisese kõvasulampulbri baasil valmistatud pihustus-sulatuspinnete majanduslikku efektiivsust sellises hinnatundlikus valdkonnas nagu kulunud detailide taastamine.