

# Wear and corrosion behaviour of thermally sprayed cermet coatings

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Received 10 April 2000; received in revised form 10 November 2000; accepted 10 November 2000

## Abstract

In order to protect machining parts against wear and corrosion, they are coated by cermet coatings. The coatings consist of WC or  $\text{Cr}_3\text{C}_2$  particles in a metal binder, which can be a pure metal or a mixture consisting of Ni, Cr, Co. The examined coatings were produced by high velocity oxy-fuel (HVOF) spraying and were investigated with regard to erosion and corrosion resistance. The combined erosion corrosion tests were carried out at ambient temperature in 0.1 M NaOH and 0.1 M  $\text{H}_2\text{SO}_4$  solutions containing sand. Information about the corrosion resistance was gained from electrochemical polarization measurements and salt spray test (NaCl solution). The results of combined wear and corrosion tests showed that the corrosion properties of the sprayed coatings strongly affect the materials loss rate under wear corrosion conditions. Coatings with a less corrosion resistant matrix present enhanced erosion, also. The erosion mechanism of the carbide coatings seems to be controlled by the skeletal network of the carbides. For comparison, flame and plasma sprayed hard  $\text{Cr}_2\text{O}_3$  coatings were examined. Due to the low electrical conductivity the corrosion rate of these coatings was very low. Under erosion conditions the hard  $\text{Cr}_2\text{O}_3$  coatings exhibited a high erosion rate and the erosion mechanism seems to be that of the brittle erosion due to grain-by-grain removal of oxide grains during impact. © 2001 Elsevier Science B.V. All rights reserved.

**Keywords:** High velocity oxy-fuel spraying; Cermet coatings; Erosion; Corrosion

## 1. Introduction

Materials are often exposed to environments that are both corrosive and erosive. In the process industry, in offshore piping and production systems flowing corrosive media often contain solid particles. This leads to a reduction of the lifetime of certain components and high maintenance costs. Protection of the metallic components by ceramic-metallic (cermet) coatings or hard oxide coatings is an effective method to reduce wear and corrosion. Generally the cermet coatings

consist of WC or  $\text{Cr}_3\text{C}_2$  particles embedded in a metal binder, which can be a pure metal or a mixture consisting of Ni, Cr, Co.

Such coatings are applied by thermal spray technologies. The microstructure of a thermally sprayed coating is usually inhomogeneous. Discontinuities, such as pores, oxide lamellas or incompletely molten spray particles are typically present in the sprayed materials. The deposition methods for the wear protective coatings are atmospheric plasma spraying (APS) and high-velocity oxygen-fuel (HVOF) flame spray processes [1–5]. Both of these methods have their own characteristics, e.g. different spray particle velocities and flame temperature. As result the coatings have different microstructure and properties. During the spraying processes and the cooling of deposits complex chemical

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transformations of the materials occur. The main phenomena which occur during spraying are the thermal decomposition of the tungsten monocarbide (WC) or chromium carbide ( $\text{Cr}_3\text{C}_2$ ) and the carbide reactions with the metallic binder [6–9]. The decarburization of WC, followed by the formation of undesirable carbides like  $\text{W}_2\text{C}$ , complex Co-W-C and metallic tungsten occur often during APS because of the high temperature of the plasma flame and the oxidizing spray atmosphere. With a significantly lower flame temperature and higher particle velocity the HVOF spraying leads to less phase transformation and produces denser coatings with lower porosity [10].

The aim of this investigation was to determine the wear and corrosion behaviour of some cermet coatings, which differ not only respective to their chemical composition but also are deposited by using different HVOF spraying devices. The behaviour of cermet coatings was compared with those of chromium oxide sprayed coatings.

## 2. Experimental

Six different thermal spray coatings were investigated (see Table 1). Four coatings consist of carbide particles in different matrices, and two are chromium oxide coatings. The carbide coatings were produced by HVOF and the chromium oxide coatings by flame spraying (FS), respectively, by atmospheric plasma spraying (APS). Two different HVOF systems were used to spray the carbide coatings: the  $\text{Cr}_3\text{C}_2$  containing powders were sprayed using a Top Gun device and the other ones with a CJS torch. No powder was sprayed with both systems. The spraying setting for each device were optimized to achieve a low porosity of the coating and a high coating efficiency.

Due to the lower processing costs, the use of carbide coatings instead of the chromium oxide coatings is being attempted in some application fields and this was the reason to include the hard oxide coatings between

the examined coatings. The high alloyed 1.4571 steel was used as base material for the coatings.

Two types of specimen were used. Some were shaped as rectangular specimens of  $100 \times 100 \times 10$  mm, others as cylinders of height 40 mm and diameter 40 mm coated on the cylinder side (erosion samples).

The test equipment for erosion examinations is shown elsewhere [11]. The specimens were fixed to a rotating disc and exposed to 0.1 M NaOH and 0.1 M  $\text{H}_2\text{SO}_4$  solution. During the tests only the sprayed surface of the specimens was in contact with the test solution. For the erosion test 100 g/l silica sand with a grain size between 100 and 500  $\mu\text{m}$  were added to the solution. The tests were carried out for times up to 48 h and the temperature during the experiments was held at  $20 \pm 2^\circ\text{C}$ . The specimens rotated with a circumferential velocity of  $2.35 \text{ m s}^{-1}$ . The total material loss rates were determined by weight loss measurements. Before weighing, the specimen were ultrasonically rinsed in acetone.

The material loss due to corrosion was determined by electrochemical measurements. Polarization curves were recorded in the positive direction from free corrosion potential at an ambient temperature of  $18^\circ\text{C} \pm 2^\circ\text{C}$ . The scan used for all tests was  $35 \text{ mV min}^{-1}$ . The corrosion current density was converted to corrosion rate through Faraday's law:

$$\omega = i \frac{A}{nF\rho}$$

where:

- $\omega$  = corrosion rate ( $\text{mm a}^{-1}$ )
- $i$  = current density ( $\text{A mm}^{-2}$ )
- $A$  = atomic weight ( $\text{g mol}^{-1}$ )
- $n$  = multiple charge-number of the substance concerned
- $F$  = Faraday's constant;  $F = 96,485 \text{ C mol}^{-1}$
- $\rho$  = specimen density ( $\text{g cm}^{-3}$ ).

Table 1  
Test materials

	Powder (wt. %)	Powder type	Size ( $\mu\text{m}$ )	Hardness ( $\text{N mm}^{-2}$ ) VHN 0.1	Deposition method
Powder 1	93(WC- $\text{Cr}_3\text{C}_2$ )-7Ni	Agglomerated sintered (spheroidized)	–45 + 20	1140	HVOF/Top Gun
Powder 2	75 $\text{Cr}_3\text{C}_2$ -25NiCr	Agglomerated sintered (spheroidized)	–45 + 20	1230	HVOF/Top Gun
Powder 3	83WC-17Co	Agglomerated sintered	–45 + 10	1160	HVOF/CJS torch
Powder 4	86WC-10Co-4Cr	Agglomerated sintered	–45 + 11	1070	HVOF/CJS torch
	80 $\text{Cr}_2\text{O}_3$ -20(97 $\text{Al}_2\text{O}_3$ -3 $\text{TiO}_2$ )	Sintered	–45 + 20	1500	Flame spraying
	$\text{Cr}_2\text{O}_3$	Sintered	–45 + 20	1280	Atmospheric plasma spraying

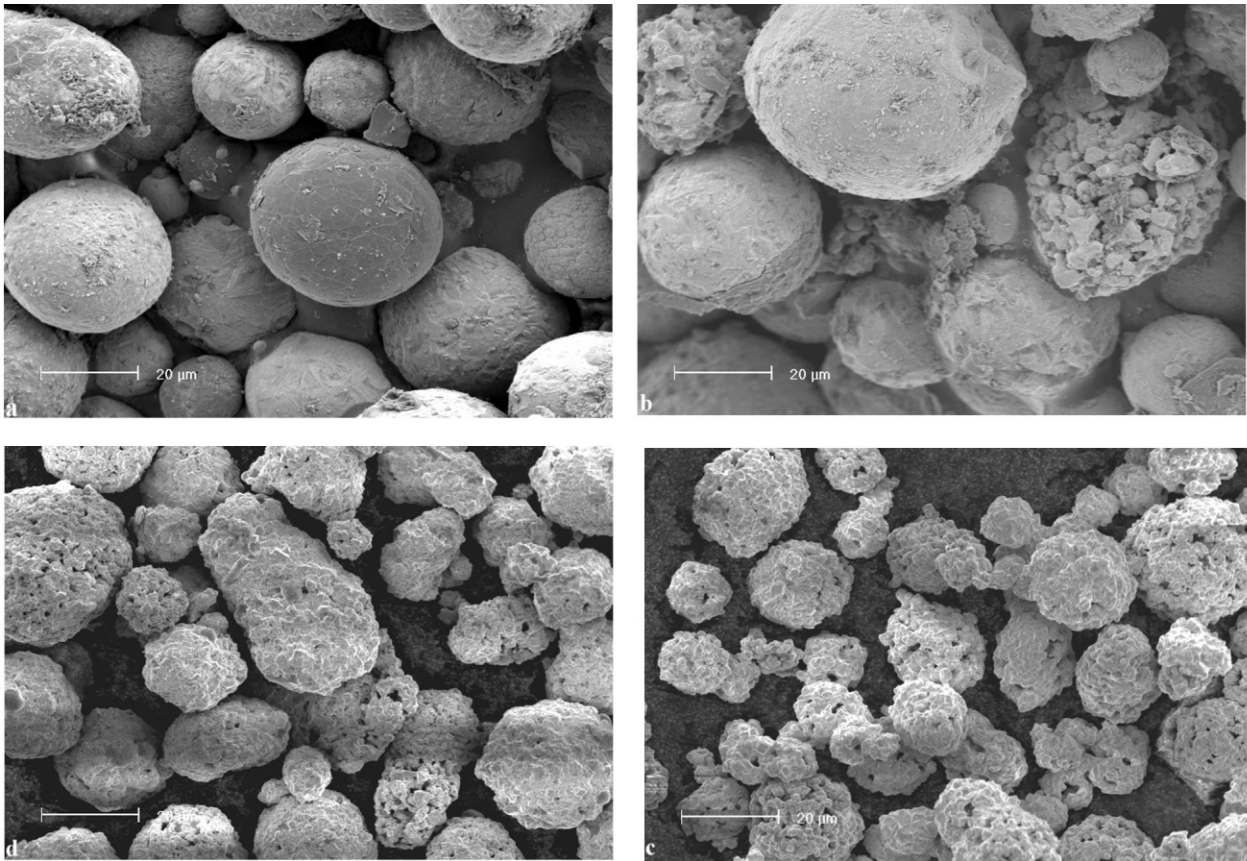


Fig. 1. SEM micrographs of the cermet powders: WC-Cr<sub>3</sub>C<sub>2</sub>-Ni (a), Cr<sub>3</sub>C<sub>2</sub>-NiCr (b), WC-Co (c), WC-Co-Cr (d).

Information about the corrosion resistance of the sprayed coatings was also gained from the salt spray test (NaCl solution), which was performed in accordance to standard DIN 50021.

The powders, as-sprayed coatings and corroded samples were examined by scanning electron microscopy (SEM) and X-ray diffraction (XRD).

### 3. Results

#### 3.1. X-Ray diffraction and SEM examination of the as-sprayed cermet coatings

Fig. 1 shows SEM micrographs of the used powders. Their chemical compositions and characteristics are shown in Table 1.

The XRD patterns were obtained on a Philips 'Xpert diffractometer using CuK $\alpha$  radiation and are shown in Figs. 2–5.

Fig. 2 illustrates the X-ray patterns for Powder 1 and the corresponding HVOF sprayed coating. The powder consists of WC, Cr<sub>3</sub>C<sub>2</sub>, Ni. Additionally, the coating contains NiO, which is formed by the oxidation of Ni during the spraying process. In the sprayed coating the peaks of Ni are very weak and those of NiO more

intensive. This indicates that although the spraying conditions are not very oxidizing, the powder was oxidized during processing. For both spectra the spectral lines between  $2\theta \approx 35^\circ$ – $45^\circ$  are slightly broader indicating a nano-crystallinity state of the materials. Some peaks, for example approximately  $54^\circ$  could not be identified, and it seems that they did not disappear or modify during the spraying. SEM micrograph of the cermet coating shows a very inhomogeneous structure with pure matrix areas (Fig. 6a).

The XRD spectra for the Powder 2 and the corresponding sprayed coating are shown in Fig. 3. The identified phases are Cr<sub>3</sub>C<sub>2</sub>, Ni and Cr. A small amount of NiCr<sub>2</sub>O<sub>4</sub> is present in the powder and the amount seems to increase in the coating. Cr<sub>7</sub>C<sub>3</sub> was found in the sprayed coating. The corresponding peak shows some shifting, indicating dissolution of the carbides. The spectral lines of the sprayed coating are slightly broad and it is supposed that spraying process causes a decrease of the grain size and formation of a nano-crystalline structure. The distribution of the carbide particles in the matrix is more uniform than in the WC-Cr<sub>3</sub>C<sub>2</sub>-Ni, but the porosity of the coating seems to be higher (Fig. 6b).

The X-ray diffraction patterns for Powder 3 and the corresponding sprayed coating are shown in Fig. 4. The

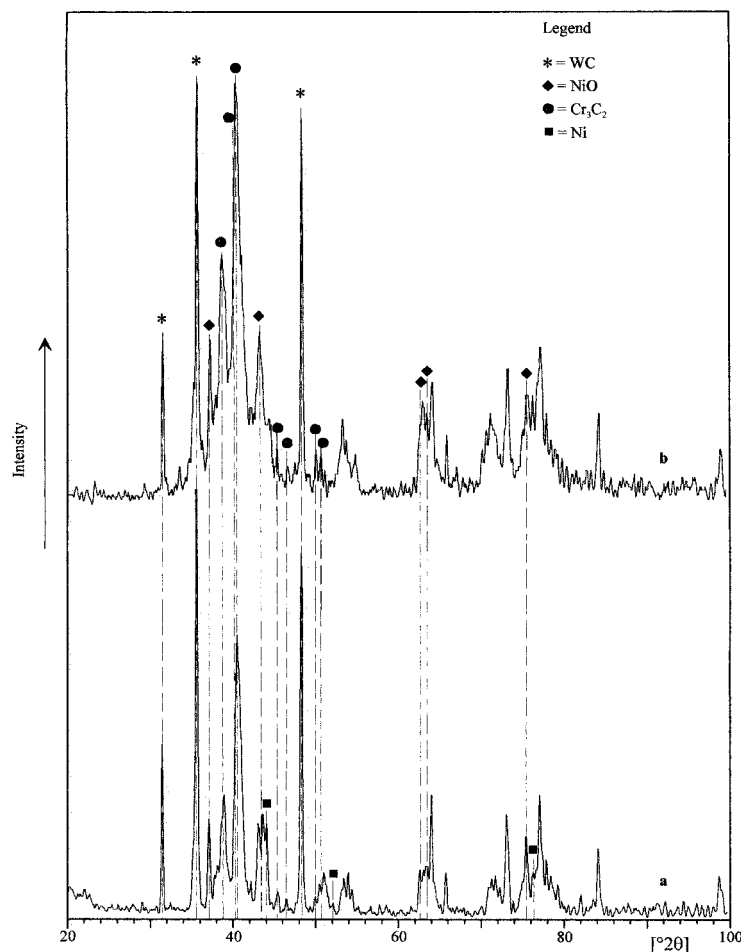


Fig. 2. XRD patterns of the WC-Cr<sub>3</sub>C<sub>2</sub>-Ni powder (a) and the corresponding as-sprayed HVOF coating (b).

powder consists of WC, W<sub>2</sub>C and Co. It should be noted that the XRD patterns of W<sub>2</sub>C are shifted somewhat indicating a dissolution of Co and Cr in the lattice of W<sub>2</sub>C. The sprayed coating contains a considerable amount of Co<sub>3</sub>W<sub>3</sub>C. Its peaks are slightly broad and it seems that the structure is nano-crystalline.

In Fig. 5 the XRD spectra of Powder 4 and HVOF sprayed coating are shown. The powder as well as the sprayed coating consist mainly of WC. In comparison to Powder 3, the mixed Co-W-C compounds are present in both powder and sprayed coatings. It is supposed that the formation of Co<sub>3</sub>W<sub>3</sub>C occurred during the powder processing (sintered) and because of its thermodynamically stability no other changes take place during spraying. Additionally, the W<sub>2</sub>C phase was not identified in the sprayed coating. Neither in the powder nor in the coating were Cr-containing phases identified. This could be explained by the small Cr-content in the powder (4 wt.%).

The structure of both last mentioned coatings is very homogeneous and the porosity is estimated as low (Fig. 6c,d).

### 3.2. Microhardness

The microhardness of the HVOF sprayed coatings was determined by Vickers method. The values are shown in Table 1. With the value of HV<sub>0.1</sub> approximately 1230 N mm<sup>-2</sup> the coating produced with Powder 2 is the hardest one and it can be thought as a substitute for the chromium oxide coating.

### 3.3. Electrochemical measurements

In order to determine the 'pure' corrosion rate electrochemical measurements of the cermet coatings were carried out. According to their application fields the sprayed coatings were tested in different solutions. The coatings produced from Powder 1 and 2 were tested in 0.1 M NaOH and 0.1 M H<sub>2</sub>SO<sub>4</sub> solutions and the coatings produced from Powder 3 and 4 were examined in synthetic sea water solution according to ASTM D1141.

The current densities and the corrosion potentials of

the sprayed cermet coatings tested in alkaline and acid solutions are very different. The WC-Cr<sub>3</sub>C<sub>2</sub>-Ni coating starts to corrode both in alkaline and acid earlier than Cr<sub>3</sub>C<sub>2</sub>-NiCr coating. In NaOH solution the corrosion potentials are approximately –870 mV and –625 mV, respectively, and the corrosion rates calculated from the current densities are 0.38 mm a<sup>–1</sup> and 0.17 mm a<sup>–1</sup>, respectively. In H<sub>2</sub>SO<sub>4</sub> solution the corrosion potentials are approximately –128 mV and –23 mV, respectively, and the corrosion rates are lower than those in alkaline solution: 0.15 mm a<sup>–1</sup> for the WC-Cr<sub>3</sub>C<sub>2</sub>-Ni coating, and 0.077 mm a<sup>–1</sup> for the Cr<sub>3</sub>C<sub>2</sub>-NiCr coating. The differences between the corrosion rates both in the alkaline and in the acid solutions indicate the beneficial effect of the NiCr matrix for the corrosion resistance.

The corrosion rate of the WC-Co coating is approximately two times higher than that of the Cr-containing coating. The values are approximately 0.76 mm a<sup>–1</sup> and 0.32 mm a<sup>–1</sup>, respectively.

As one of the aims of this work was to compare the wear behaviour of the cermet sprayed coatings under corrosion conditions with that of chromium oxide

sprayed coatings two Cr<sub>2</sub>O<sub>3</sub> sprayed coatings were electrochemically tested in alkaline and acid solutions. The chromium oxide has a very low electrical conductivity and it could be possible that the polarization curve is distorted by artefacts due to the steel substrate corrosion. Through additional steps at the sample preparation the appearance of the artefacts can be prevented. As expected the corrosion rates are very low and due to the additional Al<sub>2</sub>O<sub>3</sub> content the flame sprayed coating has the lowest corrosion rate both in alkaline and in acid solutions (Table 2).

### 3.4. Erosion tests

The erosion tests were carried out for the coatings produced from Powder 1 and Powder 2 in the alkaline solution and for the coatings produced from Powder 3 and Powder 4 in synthetic sea water.

The calculated erosion–corrosion rates for the cermet coatings after 48 h testing in the alkaline solution, and 20 h in synthetic sea water are shown in Table 2. It seems that because of the low circumferential velocity of the specimens the impact between the sam-

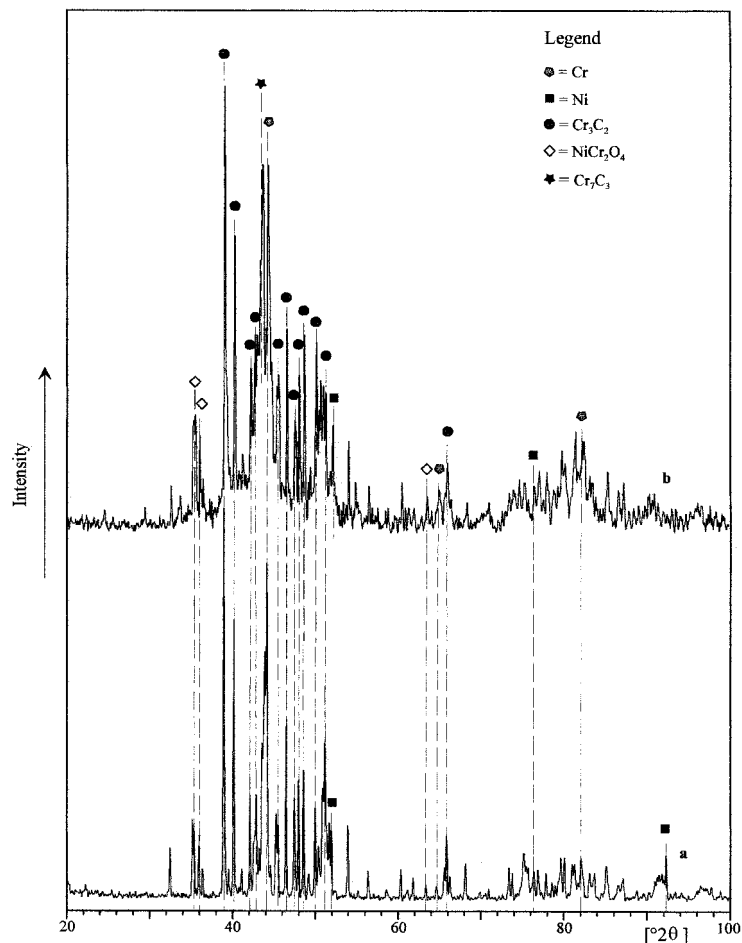


Fig. 3. XRD patterns of the Cr<sub>3</sub>C<sub>2</sub>-NiCr powder (a) and the corresponding as-sprayed HVOF coating (b).

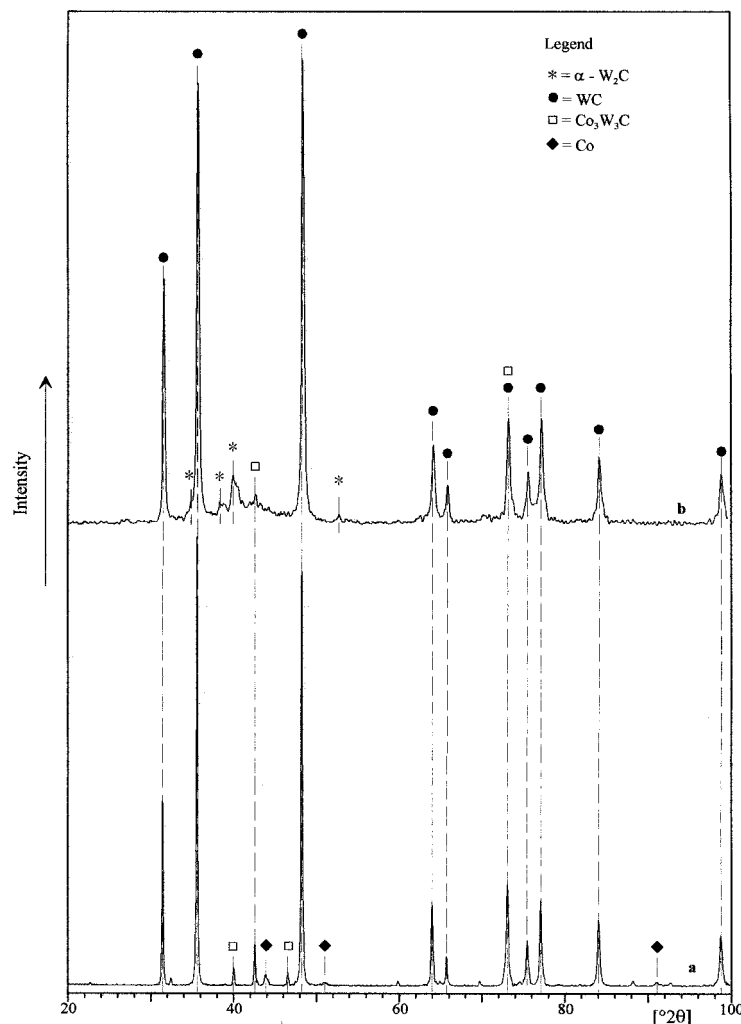


Fig. 4. XRD patterns of the WC-Co powder (a) and the corresponding as-sprayed HVOF coating (b).

ple surface and the sand particles for the cermet coatings containing  $\text{Cr}_3\text{C}_2$  is negligible. The corrosive attack of the solution is the main reason for the weight loss. The WC-Co and WC-Co-Cr coatings lost a significant material amount and their erosion–corrosion rates are approximately two times higher than the pure corrosion rates.

For the chromium oxide sprayed coatings, which were tested under the same conditions, the weight loss due to erosion is very significant and this is an indication for their high brittleness. SEM examination of the eroded surface of both type of sprayed coatings confirmed this observation. The surface of cermet coatings shows only small destroyed areas, which are delimited by carbide particles (Fig. 7a), whereas the eroded surface of the  $\text{Cr}_2\text{O}_3$  coatings shows large areas (cavities), where the material was completely removed (Fig. 7b).

### 3.5. Salt spray corrosion test

The salt spray corrosion test was carried out for

138 h for the coatings produced from Powder 1 and Powder 2, and for 118 h for the coatings produced from Powder 3 and Powder 4. The SEM examination of the coatings shows very different behaviours for the sprayed cermet coatings. For all coatings an irregular corrosion attack took place, but for the coatings without or with a small Cr-content in the matrix (WC- $\text{Cr}_3\text{C}_2$ -Ni, WC-Co and WC-Co-Cr) this attack was more significant (Fig. 8). No indication of internal corrosion was observed. Only on the surface of the WC-Co coating small rust areas were noticed, where iron oxide was identified by energy dispersive spectroscopy.

## 4. Discussion

The tested cermet coatings were produced using two HVOF deposition devices: the Top Gun and the OSU Carbide Jet System (CJS) torch. The main difference between the spraying devices is the pressure in the combustion chamber. The CJS torch is operated at

higher combustion chamber pressure than the Top Gun. Due to the CJS torch design (divergent–convergent nozzle system) this system ensures a higher thermal efficiency. Consequently, the powder particles achieve higher velocities and they are more efficiently heated. High particles velocities produce dense coatings and additionally, the low dwell times and low energy transfer to powder particles significantly reduces oxidation. This also allows production of sprayed coating which are uniform, carbide rich and dense, with exceptional wear resistance [20]. The SEM examination of the as-sprayed coatings shows that the WC-Co and WC-Co-Cr coatings have a more dense structure and a better distribution of the carbide particles than the WC-Cr<sub>3</sub>C<sub>2</sub>-Ni and Cr<sub>3</sub>C<sub>2</sub>-NiCr sprayed coatings. However, the corrosion and erosion properties of the cermet sprayed coatings are not depending only on the microstructure. The chemical compositions of the matrix, the amount and the size of the carbide particles, the phase transformations during the deposition are some of the additional factors, which should be take into

account at the examination of erosion–corrosion behaviour of the cermet sprayed coatings [7,9,12,13].

Among the tested materials WC-Co is clearly the least resistant. Although the sand concentration and the peripheral velocity of the specimens are low the erosion-corrosion rate is very high. Bjordal et al. [14,15] and Berget et al. [16] mentioned that the erosion of WC-Co sprayed coatings is attended by a synergetic effect, which increases the erosion beyond what it would be without corrosion. Cobalt corrodes very fast under the testing conditions and undermines the carbide particles. Additionally, the XRD measurements show that during the spraying of the WC-Co powder a fraction of the WC decomposes into W<sub>2</sub>C and this phase seems to reduce the hardness of the coating [21] and probably also the binding between the carbide particles and the Co-matrix. Karimi et al. [7] reported a higher volume fraction of W<sub>2</sub>C in WC-Co than in WC-CoCr and the substantial loss of carbides during the erosion test of the WC-Co coating is explained through the worse binding between the carbide parti-

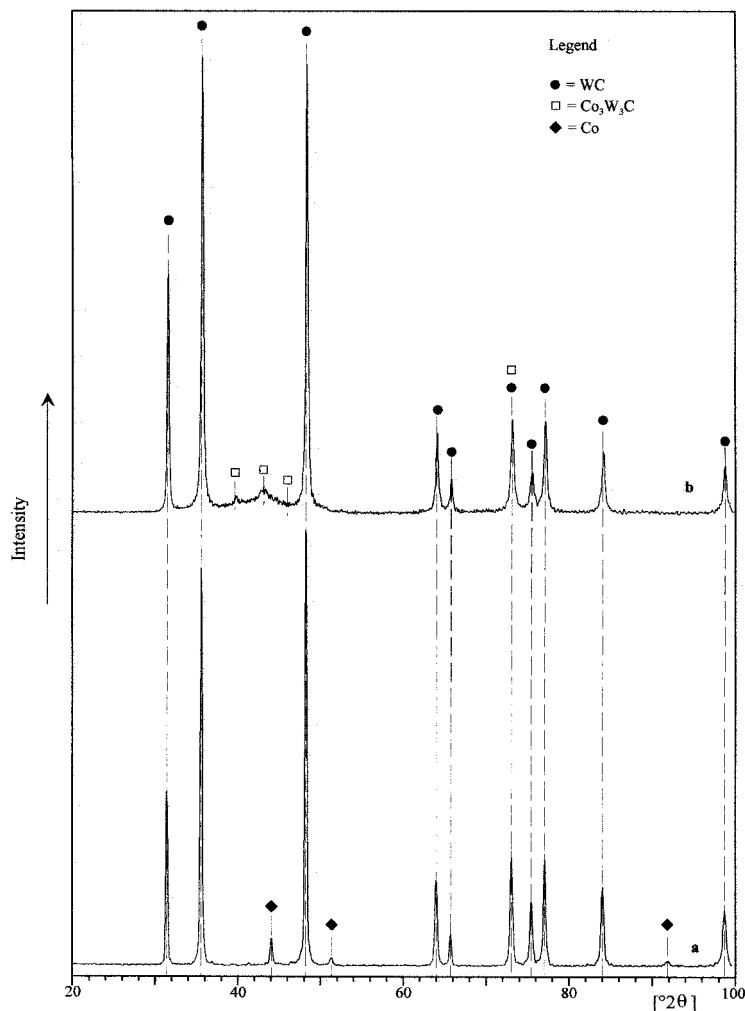


Fig. 5. XRD patterns of the WC-Co-Cr powder (a) and the corresponding as-sprayed HVOF coating (b).

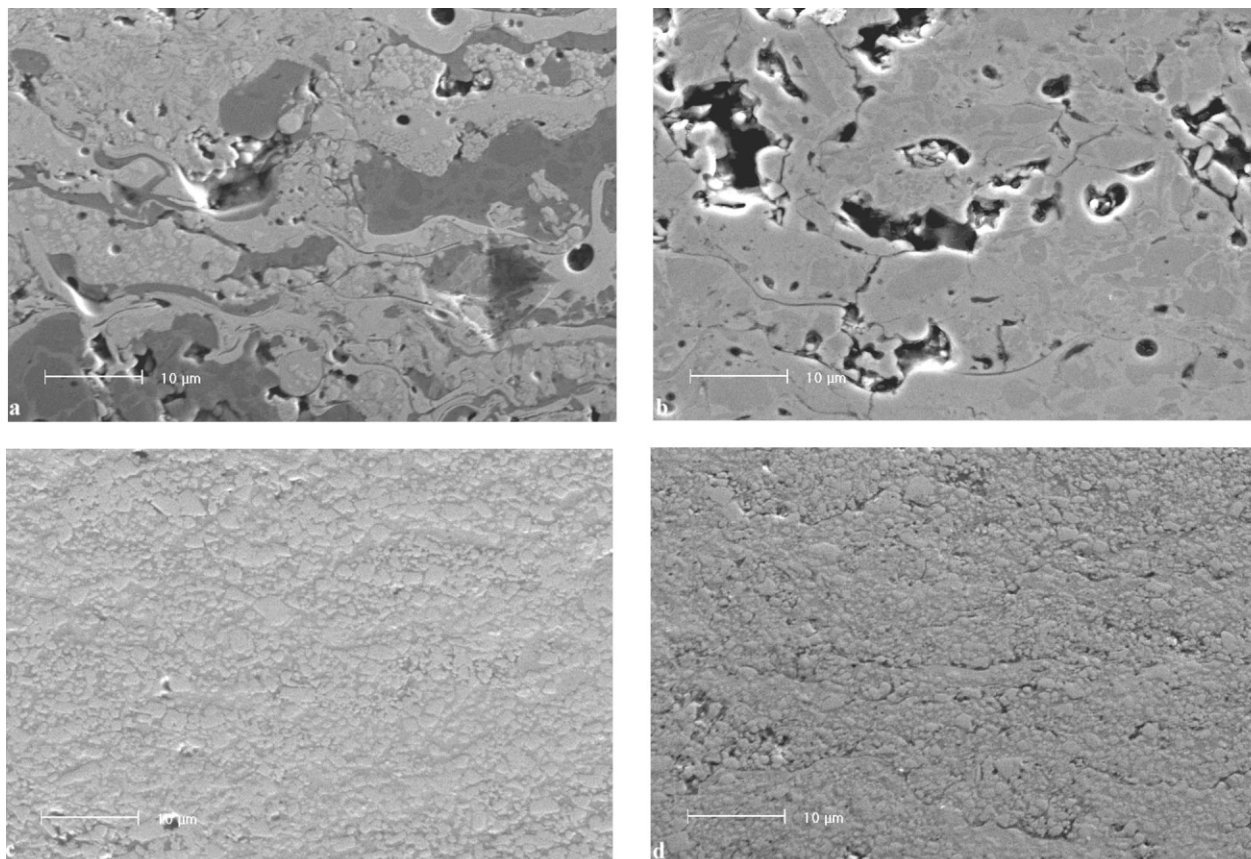


Fig. 6. SEM micrographs of the as-sprayed cermet coatings: WC-Cr<sub>3</sub>C<sub>2</sub>-Ni (a), Cr<sub>3</sub>C<sub>2</sub>-NiCr (b), WC-Co (c), WC-Co-Cr (d).

cles and their surrounding matrix. For coatings with corrosion resistant binder this undermining does not take place. The erosion–corrosion rate of the WC-Co-Cr coating is lower, but under the chosen conditions the corrosion is still important. Additionally, the salt corrosion test shows that at the same thickness and porosity of the sprayed coating the WC-Co coating is less protective. Cobalt corrodes actively, while chromium forms a passive oxide layer, which offers corrosion protection for a defined time.

The Cr<sub>3</sub>C<sub>2</sub>-containing cermet coatings seem to offer a better erosion protection than the WC-based cermet

coatings. This is in agreement with the work of Banerjee et al. [17], which noticed that the addition of Cr<sub>3</sub>C<sub>2</sub> and the partial or complete substitution of binder by Ni greatly improves the corrosion resistance of WC-Co sintered specimens in nitric acid and sulfuric acid. The XRD measurements show that the Powder 2 is more oxidation resistant than Powder 1 and this leads to the more inhomogeneous structure of the corresponding coating. The damage of both coatings through the impact with the erosive particles is low and most of the weight is lost by the corrosion of the binder (Table 2). The HVOF Cr<sub>3</sub>C<sub>2</sub>-containing sprayed coatings exhibit

Table 2  
Results from the electrochemical measurements and erosion corrosion tests

Coating	Electrochemical measurements Corrosion rate, mm a <sup>-1</sup>			Erosion-corrosion test Erosion-corrosion rate, mm a <sup>-1</sup>	
	NaOH	H <sub>2</sub> SO <sub>4</sub>	Sea water	NaOH	Sea water
WC-Cr <sub>3</sub> C <sub>2</sub> -Ni	0.38	0.15	–	0.4	–
Cr <sub>3</sub> C <sub>2</sub> -NiCr	0.17	0.077	–	0.17	–
WC-Co	–	–	0.76	–	1.6
WC-Co-Cr	–	–	0.32	–	0.55
Cr <sub>2</sub> O <sub>3</sub> -Al <sub>2</sub> O <sub>3</sub> -TiO <sub>2</sub>	3.16 × 10 <sup>-5</sup>	3.64 × 10 <sup>-5</sup>	–	0.27	–
Cr <sub>2</sub> O <sub>3</sub>	7.6 × 10 <sup>-4</sup>	1.5 × 10 <sup>-3</sup>	–	0.35	–



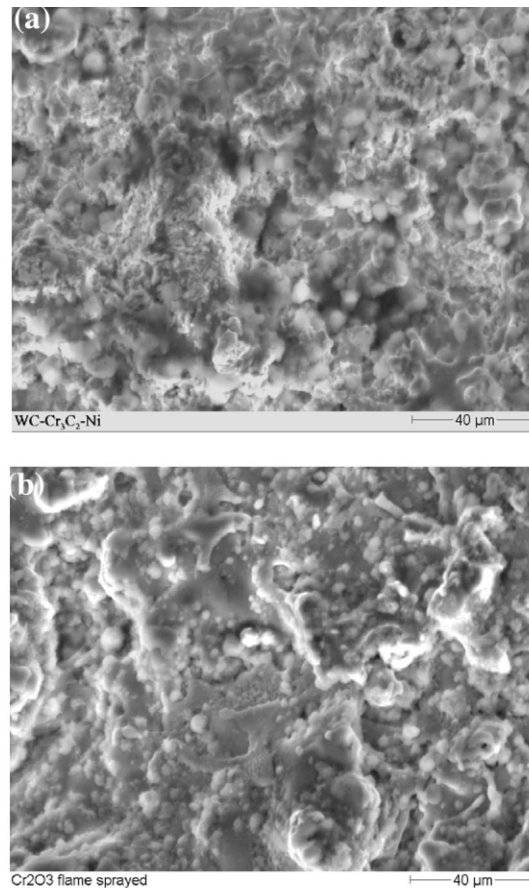


Fig. 7. SEM micrographs of the surfaces of WC-Cr<sub>3</sub>C<sub>2</sub>-Ni and Cr<sub>2</sub>O<sub>3</sub> sprayed coatings after 48h erosion test in 0.1 M NaOH aqueous solution.

typical characteristics of brittle erosion morphology with formation of a cracked and chipped surface [18]. The lower material loss of the Cr<sub>3</sub>C<sub>2</sub>-NiCr is caused not only by the presence of Cr in the matrix, but probably also by the superior quality of this coating compared to the WC-Cr<sub>3</sub>C<sub>2</sub>-Ni (Fig. 6a,b).

The determined erosion-corrosion rates show that the erosion mechanism of the cermet coatings is controlled partly by the carbide particles (WC-Cr<sub>3</sub>C<sub>2</sub>-Ni, Cr<sub>3</sub>C<sub>2</sub>-NiCr) and partly by the metallic binder (WC-Co, WC-Co-Cr). In the first case the erosion occurs by the removal of the carbide grains during the impact and the corrosion of the binder seems to be insignificant, and in the second case the binder erodes inducing a loss of carbide grains after pronounced erosion.

The erosion-corrosion behaviour of the Cr<sub>3</sub>C<sub>2</sub>-NiCr coating can be compared with that of chromium oxide. The cermet coating has a better erosion resistance than both Cr<sub>2</sub>O<sub>3</sub> sprayed coatings and the reason seems to be the presence of the hard carbide particles. SEM examination of the eroded surface of the Cr<sub>2</sub>O<sub>3</sub> sprayed coating shows that the impact between the sand particles and the coating surface leads to grain-by-grain removal of the oxide. Modifications of the chemical composition of the powder could lead to harder cermet

coatings with similar erosion-corrosion properties. The HVOF sprayed Cr<sub>3</sub>C<sub>2</sub>-NiCr coating could be an excellent replacement for the thermal sprayed Cr<sub>2</sub>O<sub>3</sub> coatings as well as for the chromium plating [19].

## 5. Conclusions

Combined erosion and corrosion tests of HVOF sprayed cermet coatings have shown that a coating with a highly corroding matrix will suffer a much greater weight loss than a coating with a less corroding matrix. Among the tested materials the WC-Co coating was the least resistant. The addition of 4 wt.% Cr improved the erosion and corrosion resistance of the WC-based coating. Further modifications of the cermet powder consisting in the partial or complete substitution of WC by Cr<sub>3</sub>C<sub>2</sub> and the use of matrix with higher corrosion resistance as NiCr led to more protective cermet coatings. Due to its low erosion-corrosion rate the HVOF sprayed Cr<sub>3</sub>C<sub>2</sub>-NiCr coating is considered to be an excellent replacement for the thermal sprayed Cr<sub>2</sub>O<sub>3</sub> coatings.

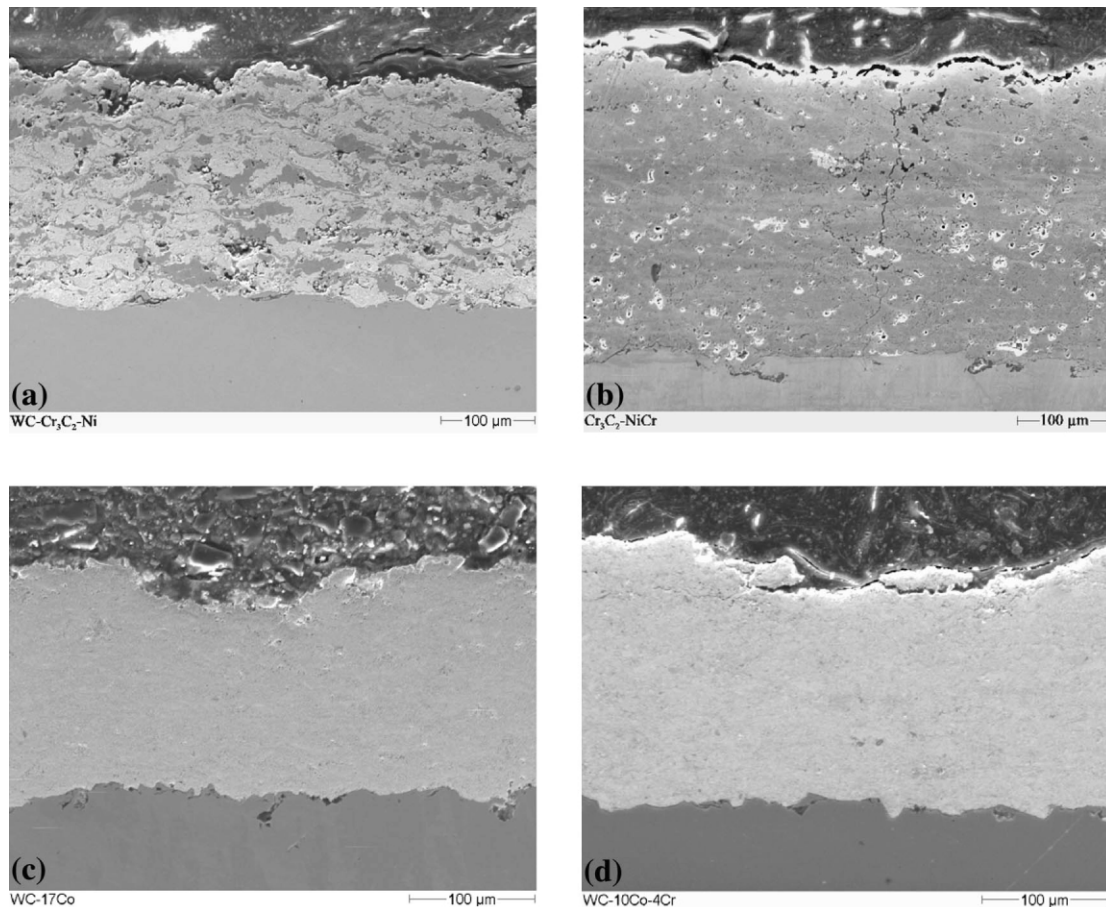


Fig. 8. SEM micrographs of the cermet coatings after the salt spray corrosion test: WC-Cr<sub>3</sub>C<sub>2</sub>-Ni (a), Cr<sub>3</sub>C<sub>2</sub>-NiCr (b), WC-Co (c), WC-Co-Cr (d).

### Acknowledgements

This work is part of the project on thermal sprayed coatings used for wear and corrosion protection of machining parts supported by the Federal Minister of Education and Research, Germany under the contract number 1700198. The authors wish to thank OSU Maschinenbau GmbH and RS Rittel GmbH for the manufacture of the coatings.

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