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Influence of powder feed rate on corrosion and wear properties of Fe-based HVAF coatings

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Abstract. Large-area applications, such as Yankee cylinders for paper machines, are often exposed to corrosion/wear, and therefore require adequate surface protection. The goal of this study is to develop a cost-efficient coating system that offers comparable protection to the industrially established Fe-based coating systems produced by wire arc spraying (WAS). Cost efficiency is to be achieved by using an economically priced, novel Fe-based feedstock material, the high velocity air-fuel (HVAF) process as well as a coating thickness reduction. In this study, the feedstock material FeCrB/WC-Co with the grain size fraction -32 +11 µm was investigated. Two coating thicknesses $d_{ct} \approx 120$ µm and $d_{ct} \approx 240$ µm and two different powder feed rates p = 40 g/min and p = 200 g/min were considered. An industrially established WAS FeCrBSiMnC coating system was used as reference. To examine the microstructure, cross sections of the coatings were prepared and investigated with a light microscope. Electro chemical polarization and pin-on-disc- (POD) tests were performed to investigate the corrosion and wear properties of these coating systems. Furthermore, XRD measurements were used to investigate the phase composition. The results show that a dense and crack-free coating can be produced with a powder feed rate of p = 200 g/min and that the powder feed rate p has a significant influence on the corrosion behaviour.

1. Introduction

In recent years, economical factors such as raw material and post-processing costs are playing an increasingly important role in the research of novel thermally sprayed wear and corrosion protection coatings. For this reason, cost efficient Fe-based materials have become the focus of research; especially for large area applications. Good corrosion and wear properties of these materials can be achieved by alloying high amounts of Cr and hard phase forming elements such as B or C [1, 2]. A further research trend in the field of thermal spraying technology is the HVAF process. This process is a modification of the high velocity oxygen-fuel (HVOF) process [3] with which higher deposition rates [4, 5] and lower particle in-flight temperatures, compared to standard HVOF processes, can be achieved [4]. The lower process temperature is achieved by using air instead of oxygen as oxidant [6]. Wang et al. showed that dense coatings with a low content of oxides can be produced with the HVAF process and explains this with the lower particle temperatures and the higher particle velocities compared to the HVOF process [4]. These results are confirmed by a number of studies [5 – 10]. However, the variety of different HVOF torch designs must not be neglected, which means that no general statement can be made about the oxygen content in the coatings applied by means of HVOF and HVAF spraying [13].

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A typical example for a large area application, which requires high wear and corrosion resistance, is cylinders of paper machines, like Yankee dryers. Usually, these cylinders are coated using WAS with a Fe-based feedstock material, e.g. a FeCrBSiMnC cored wire. Typically, as-sprayed coating thicknesses range from d_{ct} = 700 to 1,200 µm in this application [3, 11]. Experience has shown that the sufficient corrosion resistance of porous coatings can only be achieved with a high coating thickness. The corrosion resistance of the coating can be further increased by using sealers. However, this requires an additional post-production step. Due to the high roughness of WAS coatings, a time-consuming grinding process is necessary to achieve low surface roughness values required for application. Although suitable coatings can be produced by WAS, the process-inherent economic advantages such as low investment costs and high deposition rates are usually countered by the high post production costs. Furthermore, thick and porous coatings exhibit low thermal conductivities, which leads to higher energy expenditure during paper manufacture [11, 12]. Thicker coatings usually result in higher thermal insulation. Coatings exhibiting sufficient corrosion and wear resistance can also be realised in smaller thicknesses using HVOF and HVAF spraying with the utilisation of cemented carbide based feedstock materials. Due to the higher production costs, however, these coating systems are only used for cylinders which are exposed to very high loading conditions [13, 14]. In order to produce good and cost-effective wear and corrosion protection coatings for this application, one approach can be the use of the HVAF process in combination with a highly alloyed Fe-based feedstock material. With the HVAF process, higher powder feed rates can be achieved compared to the HVOF process. This can significantly shorten the process time. Compared to the WAS process, dense, near-net-shape and thinner coatings can be produced, which can have a positive effect on post-processing costs and on the energy consumption during paper production.

In a previous study, the authors have already shown that a novel FeCrB/WC-Co coating exhibits corrosion and wear properties, which are comparable to a HVOF sprayed Cr_3C_2/NiCr 75/25 coating system [15], although the grain size fraction G of the used feedstock material was not optimized for the HVAF process. However, the coating system was prone to cracking in case of high powder feed rates (p >> 40 g/min). Cracking behaviour of the coating system was attributed to a non-uniform heating of the particles and a high local heat input in the coating due to the high powder feed rate and a low surface velocity of the gas and particle jet. Both effects result in high residual stresses in the coating. The study presented in this paper is a continuation of [15] with the goal of producing dense and crack-free FeCrB/WC-Co coatings with powder feed rates upto p = 200 g/min. This goal is to be achieved among others by using a grain size fraction G = -32 +11 µm. This finer grain size fraction can result in more uniformly molten particles, which can decrease the residual stress and avoid cracks. Moreover, near-net-shape and thinner coatings can be realised with a fine powder fraction. Furthermore, the influence of a coating thickness reduction below d_{ct} = 150 µm on the corrosion and wear behaviour is also investigated. Thinner coatings can save feedstock material, decrease the production time and lead to energy savings due to the reduction of the thermal insulation effect. The focus of this study is, therefore, set to the development of a novel FeCrB/WC-Co coating system with a powder fraction of G = -32 +11 µm and a powder feed rate of p = 200 g/min by means of HVAF spraying. The influence of such a high powder feed rate on the wear and corrosion properties is investigated and compared to properties of a WAS sprayed FeCrBSiMnC reference coating system.

2. Experimental setup

All HVAF and WAS coatings were applied on 1.0038 (S235JR) steel substrates with the dimensions of 40 x 50 x 8 mm³. 1.0038 is a widely used construction steel, which has poor wear and corrosion performance in paper manufacturing. However, construction steel and cast iron are widely used as structural material in Yankee dryers. Sufficient wear and corrosion resistance of such parts is achieved through the use of coatings. Prior to the coating process, the substrates were roughened using an injector blasting system with corundum.

As already mentioned, cost-efficient Fe-based coating systems with high depositions rates can be realised by means of WAS. Therefore, a FeCrBSiMnC coating applied by WAS (G30/4SF-Push-LD/U2, Oerlikon Metco AG, Winterthur, Switzerland) was chosen as reference coating system. The commercially available cored wire SP112 (Corodur Fülldraht GmbH, Willich, Germany) was used as feedstock material. The chemical composition of the wire is given in Table 1.

Table 1: Chemical composition of the FeCrB/WC-Co feedstock material and the FeCrBSiMnC (SP112) wire, as given by the distributors [wt.-%]

Alloy	Fe	Cr	B	C	W	Co	Si	Mn
FeCrBSiMnC (SP112)	Rest	27.5 – 29	3.8	0.1	-	-	1.5	1.5
FeCrB/WC-Co	Rest	20 – 23	3.5 – 4	1 – 2	10 – 12	1 – 1.5		

Industrially established process parameters were used to produce the reference coating, see Table 2. Subsequent to the coating process, the coatings were sealed in the "as-sprayed" state with an epoxy resin, to close open pores and to increase corrosion resistance. The industrially established sealer dichtol HM #2407 (DIAMANT Metallplastic GmbH, Mönchengladbach, Germany) was used for this purpose. The sealer was processed as specified by the manufacturer.

Table 2: WAS process parameters

Parameter	Value
Current I [A]	150
Voltage U [V]	30
Pressure [bar]	3.5
Stand-off distance d [mm]	150
Surface speed [mm/s]	600
Wire feed rate w [g/min]	115

For the application of novel HVAF coatings, the feedstock material FeCrB/WC-Co (Above Material Technology Co., Ltd., Beijing, China), which is composed of separate powder particles made of FeCrB and WC-Co, has been used. The chemical composition is given in Table 1. The powder includes a high content of Cr and B to achieve a good corrosion resistance and to promote the formation of hard phases, respectively. WC particles, embedded in a Co matrix, were added to the material composition to achieve a high wear resistance. Coating parameters for the HVAF coatings are given in Table 3. These parameters are based on the parameters developed in [15] and modified to achieve crack-free coatings with higher powder feed rates. The HVAF samples were produced using an AK-07 system (Kermetico Inc., Benicia, USA). An additional sealing of the HVAF coatings was omitted. In [15], the used FeCrB/WC-Co feedstock material had a grain size fraction of G = -45 +11 µm. In preliminary studies, a significant influence of the grain size fraction on crack formation was identified. For this reason, prior to the coating process, the feedstock material FeCrB/WC-Co was sieved to a grain size fraction of G = -32 +11 µm which is typical for the HVAF process. Due to the lower particle temperature compared to the HVOF process, it is assumed that the coarser grain fraction can cause non-uniform heating of the larger particles, which can lead to high residual stresses induced by the hard impact of non-uniform molten particles. The high compressive stress can result in cracks in the coating, especially for high powder feed rates.

Table 3: HVAF process parameters

a)

Parameter	Value
Compressed air [bar]	6.3
Propane [bar]	5.6
Nitrogen [SLPM]	18
Stand-off distance d [mm]	380
Surface speed [mm/s]	900, 4,500
Powder feed rate p [g/min]	40, 200
Cooling air	1.5 bar

The experimental setup for the HVAF samples investigated in this study is illustrated in Figure 1 a). The substrates were fixed into the sample holder. The sample holder was mounted onto an electro motor, which allows a rotatory movement of the specimen holder. The HVAF gun only moves into z-direction to coat the samples. The samples were cooled from two sides. This experimental setup was used because preliminary studies showed that the layer thickness, which mainly depends on the powder feed rate and the surface speed of the particle-laden free jet, has a great influence on crack formation in FeCrB/WC-Co coatings applied by HVAF. The surface speed was increased depending on the powder feed rate. This results in the same layer thickness as in [15], with which crack-free coatings were already produced. Thicker layer thicknesses can lead to a higher local heat load, which in turn can lead to higher residual stress and thus to cracks in the coating. The heat propagation on the samples was additionally controlled with a cooling system to reduce the accumulating residual stress. Compared to study [15], the stand-off distance d was increased to d = 380 mm. At a higher spray distance, the particles dwell longer in the free jet and exhibit a more uniform melting, which in turn can reduce the induced stress due to non-uniform molten particles and related crack initiation. Subsequently, the wear and corrosion properties of the coatings are determined and compared to WAS sprayed reference coating.

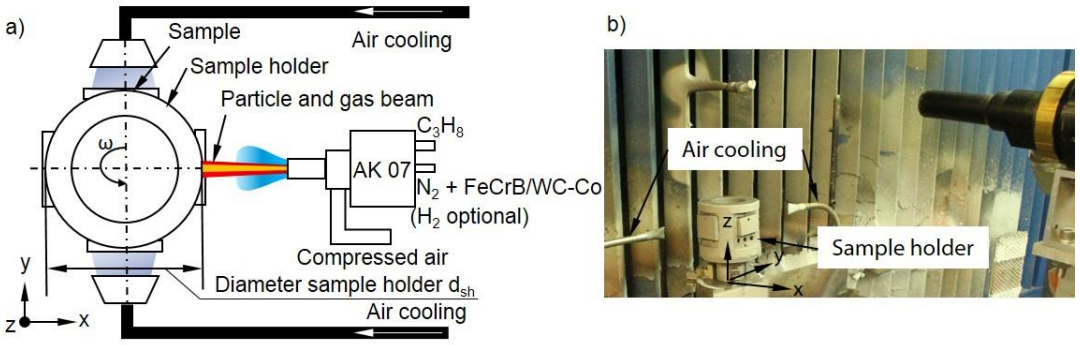

Figure 1: a) Sketch of the experimental setup
b) Experimental Setup

An overview of the sample names and the varied process parameters is given in Table 4. The different coating thicknesses of the HVAF samples were achieved by adjusting the number of transitions. Images of the cross sections were taken with a Zeiss Axiophot light microscope (Carl Zeiss AG, Oberkochen, Germany) to investigate and compare the microstructure of the HVAF and WAS samples. Surface roughness of the samples in the "as-sprayed" state was measured with the confocal laser scanning microscope (CLSM) VKX 200 (Keyence Corporation, Osaka, Japan). The phase composition of the FeCrB/WC-Co feedstock material and the HVAF coating systems was analysed with X-Ray Diffractometry (XRD) with a Seifert XRD 3000 from GE Sensing & Inspection Technologies GmbH (Hürth, Germany) using a Cu anode. The angle of incidence ω = 10°, step width

$\Delta 2\Theta = 0.05°$, holding time $t_h = 10$ s and measurement interval $2\Theta = 20°$ to $80°$ were kept constant for all measurements.

Table 4: Overview sample names and varied parameters

Sample	Powder feed rate p [g/min]	Surface speed [mm/s]	Transitions	Note
WAS unsealed	115	600	26	
WAS sealed	115	600	26	Sealed with dichtol HM #2407
HVAF 01	40	900	20	
HVAF 02	40	900	10	
HVAF 03	200	4,500	20	
HVAF 04	200	4,500	10	

For the investigation of the wear properties, a POD tribometer (CSM Instruments SA, Freiburg, Germany) was used and the wear coefficient K was determined. The wear coefficient K describes the amount of energy that must be added to a system in order to obtain a certain amount of wear. To keep the initial conditions constant, the samples were ground and polished to a surface roughness of $Ra > 0.5$ µm. An Al_2O_3 ball with a diameter $ø_{cb} = 6$ mm was used as counter body to characterise the wear properties of the coatings. Al_2O_3 was chosen to conduct abrasive wear tests and to reduce adhesive wear. The used test parameters are shown in Table 5. All tests were performed without lubricant. Subsequent to the wear tests, the wear tracks were investigated using CLSM to determine the volume and diameter of track. With these values, the wear coefficient K was calculated according to equation (1) [2]. For every wear track, three measurement positions along the track were used to determine the mentioned parameters.

Table 5: POD test parameters

Parameter	Value
Wear track diameter d	5 mm
Wear track length s	1,000 m
Normal load w	10 N
Rotation speed	100 mm/s
Test temperature	RT

$$K\left[\frac{mm^3}{Nm}\right] = V \cdot \left[w(s \cdot \frac{d}{d_t})\right]^{-1} \quad (1)$$

In order to analyse the corrosion resistance, polarisation tests and subsequent cross-section analyses of the corroded samples were performed to determine whether corrosion occurs only on the surface or also in the interface between substrate and coating. The polarization tests were performed in a 5 % NaCl solution with a pH value of 7.00 ± 0.05 at room temperature (Reference 600+, Gamry Instruments, Warminster, England). Prior to the polarization test, the samples were prepared in the same manner as for the POD test. A calomel electrode was used as reference electrode and the potential scan rate was set to 0.5 mV/s. The rest potentials and the corrosion current densities were investigated using the Tafel slope analysis.

3. Results and discussion

Microstructure

In Figure 2, the cross sections of the WAS reference coating and the HVAF samples are shown. The WAS reference sample shows the typical lamellar structure of a WAS coating. Splat boundaries, pores, particles solidified in flight and few oxidized particles can be observed in the cross section. These splat boundaries and porosities can be a weak point regarding corrosion loading. The WAS sample has a coating thickness $d_{ct} \approx 720$ µm and is thus significantly higher than that of the HVAF samples. For the HVAF samples, the darker phases can be identified as WC-Co and the brighter phases as FeCrB matrix. No cracks were detected, even for a powder feed rate of p = 200 g/min and a coating thickness of $d_{ct} = 277$ µm. The microstructure of the samples HVAF 01 – HVAF 04 looks comparable, is very dense and exhibits a low oxide content. In the FeCrB phase, no grain boundaries between the individual particles of the feedstock material can be detected. Furthermore, only a very small number of unmolten particles were observed. The roughness of all HVAF samples in the as-sprayed state is in a similar range and significantly lower than the roughness of the WAS reference. The smoother surface of the HVAF samples is beneficial with respect to lower post-production costs.

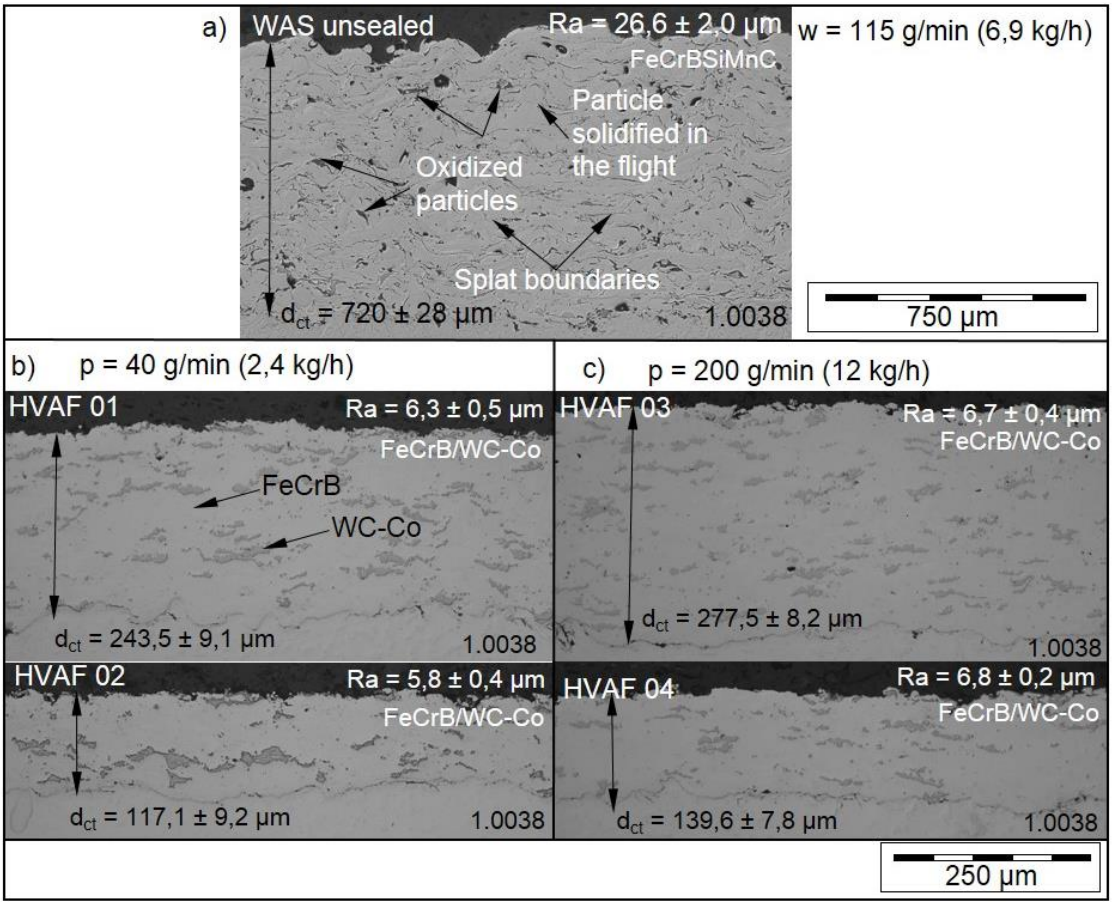

Figure 2: a) Cross section of the WAS reference coating
b) Cross section of the HVAF samples coated with 40 g/min
c) Cross section of the HVAF samples coated with 200 g/min

Phase composition

The XRD measurements of the HVAF samples and the FeCrB/WC-Co feedstock material are shown in Figure 3. For the feedstock material FeCrB/WC-Co, peaks corresponding to FeCrB and WC were identified. The measured spectra of the HVAF sample and the feedstock material look comparable indicating that the spraying process did not significantly alter the phase composition.

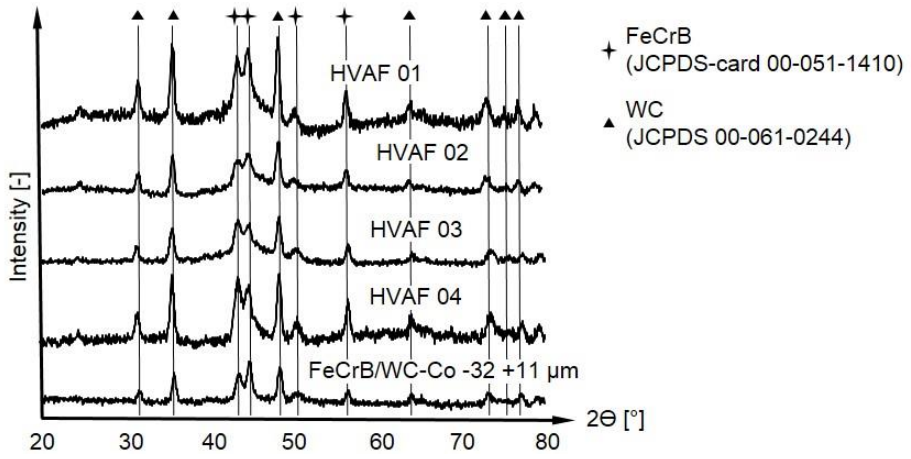

Figure 3: XRD analyses of the HVAF samples and the feedstock material FeCrB/WC-Co

Wear test

The analysis of the POD results is shown in Figure 4. In Figure 4 a), the wear tracks of the samples WAS sealed, HVAF 01 and HVAF 03 are shown. For all samples, non-homogenous wear tracks can be observed. It can be seen that the width of the wear track of the WAS sealed sample is wider than that of HVAF 01 and HVAF 03 while the width of HVAF 01 and HVAF 03 is comparable. The microscope images of the wear marks suggest that HVAF samples are less prone to wear then the investigated WAS samples. The wear tracks of the samples WAS unsealed, HVAF 02 and HVAF 04 did not exhibit any relevant differences to the corresponding wear track shown in Figure 4. The measured wear coefficient for all samples is in a similar range as the wear coefficient measured for the coating system developed in [15] by the authors, $K = 5.0\ 10^{-6}$ mm³/Nm.

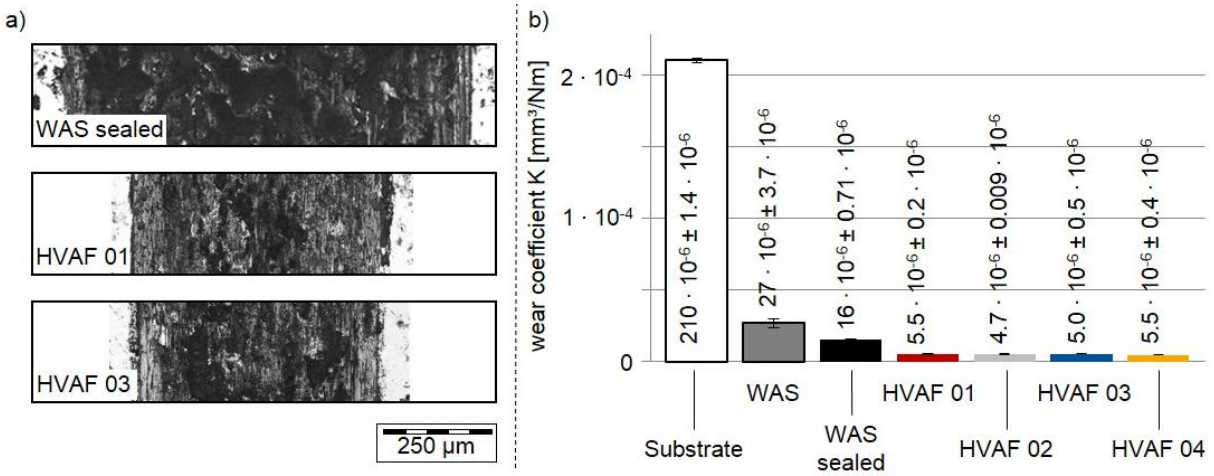

Figure 4: a) Wear tracks of the samples WAS sealed, HVAF 01 and HVAF 03
b) Wear coefficient K of all HVAF samples compared to WAS samples

In Figure 4 b), the wear coefficients K for all investigated samples are shown. WAS sealed has a lower wear coefficient K than the unsealed WAS sample. It is assumed that the lower wear coefficient can be traced back to the sealer, which prevents the coating from breaking out. The wear coefficient of all HVAF samples is significantly lower than the wear coefficient of the WAS samples. A slightly lower wear coefficient K among HVAF samples was measured in case of sample HVAF 02. This sample exhibits a wear coefficient of $K = 4.7 \cdot 10^{-6}$ mm³/Nm. The wear coefficients for the other HVAF

samples are slightly higher. It can be stated that the lower wear coefficients of HVAF coatings can be attributed to the high density of the coatings. In contrast, the WAS coatings exhibit a more porous microstructure, which can negatively influence the wear resistance. It can be also stated that the coating thickness and the powder feed rate have no significant influence on the wear behaviour of the HVAF samples in the considered tribological test.

Corrosion test

The corrosion tests showed significant differences between substrate and considered coating systems. The rest potential U_R and corrosion current density i_c is given in Table 6. The corresponding curves are designated in Figure 5. HVAF 01 exhibits the highest rest potential of U_R = -251 mV_{SCE} and lowest corrosion current density i_c. The thicker coating HVAF 02 has s comparable rest potential. The rest potentials of the samples produced with a powder feed rate of p = 200 g/min, HVAF 03 and HVAF 04, are significantly lower than the rest potentials of HVAF 01 and HVAF 02. The measured rest potential for the sealed WAS reference is in the same range as HVAF 03. An interesting aspect was observed in the samples HVAF 01 and HVAF 02 for higher overpotentials of $\eta \approx 530$ mV, the corrosion current density stays below 3.3 mA/cm². It is assumed that the powder feed rate influences the phase composition of HVAF samples and thus influences their corrosive properties. Although the exact reason is subject to further investigation, it can be stated that the powder feed rate has a significant influence on the corrosion resistance of the HVAF samples.

Table 6: Rest potential U_R and corrosion current density i_c

Sample	Rest potential U_R [mV_{SCE}]	Corrosion current density i_c [µA/cm²]
1.0038	-654	3.98
WAS unsealed	-605	4.28
WAS sealed	-419	5.44
HVAF 01	-251	0.50
HVAF 02	-293	0.39
HVAF 03	-435	4.52
HVAF 04	-478	4.98

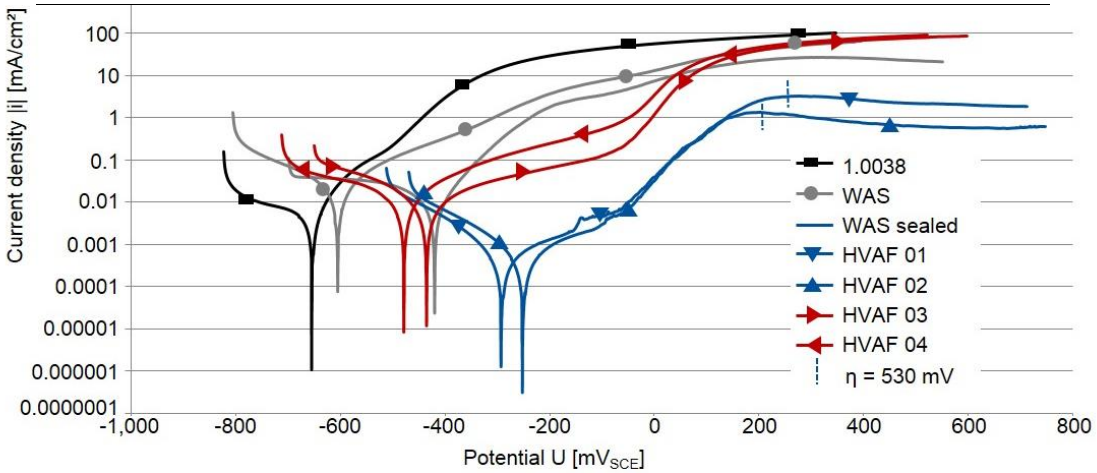

Figure 5: Corrosion current potential curves of the reference and the HVAF samples in 5 % NaCl-solution at room temperature

The corroded cross sections after the destructive polarization test are shown in Figure 6. The maximum corrosion depth was measured. For this purpose, the polished surface in the non-corroded area of the sample was selected as the reference plane. Figure 6 a) shows the cross section of the sealed WAS coating. No under-corrosion could be identified for this sample. Maximum corrosion

depth measured in this test correspond to a value of d_{cd} = 82 µm. Here it must be stated that in deeper layers, some spots with beginning of corrosion have been observed. Therefore, the given value for the corrosion depth shall be considered carefully.

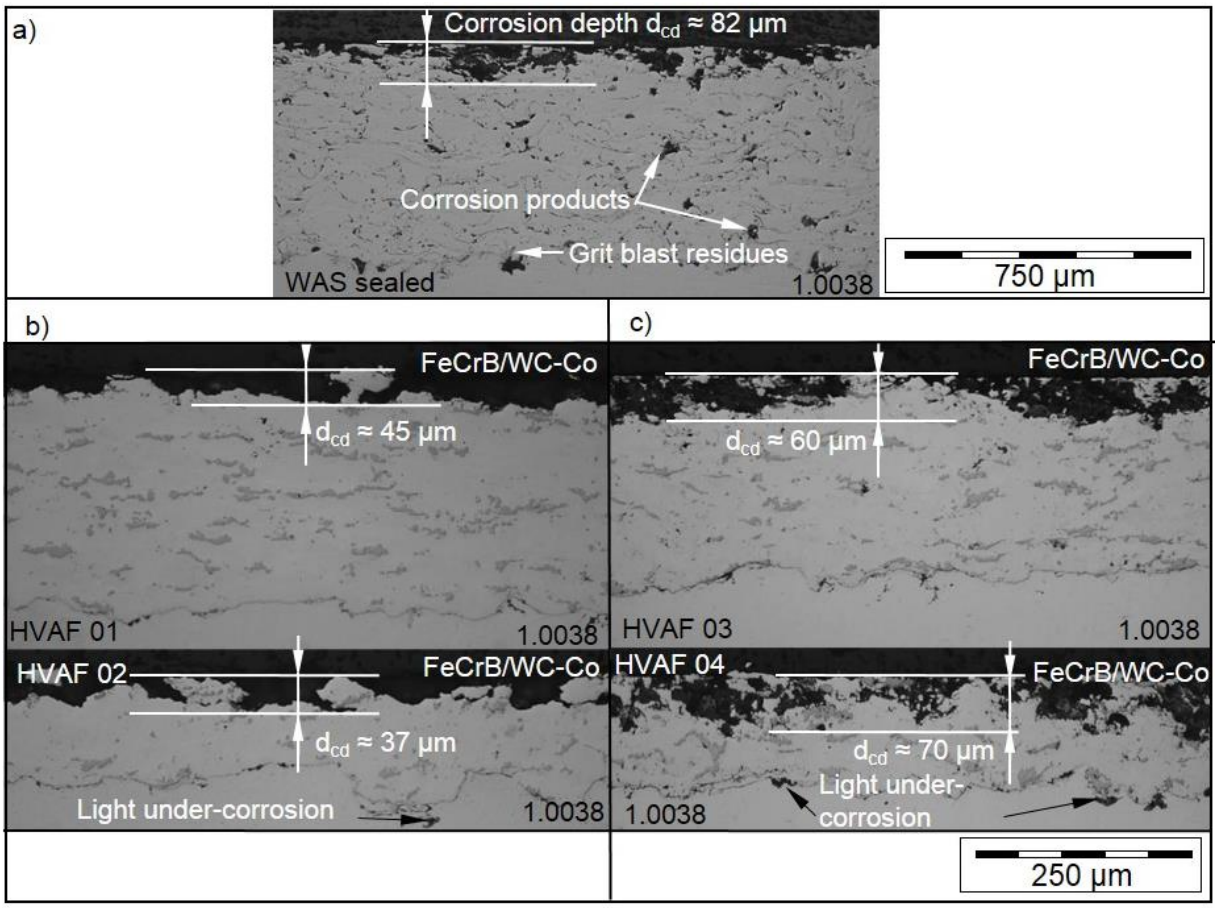

Figure 6: Cross sections after the corrosion test of the a) sealed WAS reference
b) HVAF 01 and HVAF 02
c) HVAF 03 and HVAF 04

The maximum corrosion depth d_{cd} of the HVAF sprayed coatings are significantly lower. For the samples HVAF 01 and HVAF 02, a maximum corrosion depth d_{cd} = 45 µm was measured. In case of the samples HVAF 03 and HVAF 04, the maximum corrosion depth increased to d_{cd} = 60 – 70 µm, see Figure 6 b) and c). This is in accordance with the higher corrosion current densities of these samples measured using electrochemical polarisation test, see Figure 5. These results confirm once again that the powder feed rate influences the corrosion properties significantly. A reason for the difference in corrosion properties observed in case of low and high powder feed rates might be the reduced available heat energy per particle for high powder feed rates, which can decrease the melting degree of the particles, whereby nano-pores can occur at the splat boundaries. These pores cannot be detected with the used light microscope, therefore scanning electron microscope / energy dispersive X-ray spectroscopy (SEM/EDS) analyses are necessary. Another reason assumed for the changed corrosion properties at high powder feed rates is the altered phase composition of the coating due to the different degree of melting of the particles. The different powder feed rates might lead to local Cr depletion and, therefore, no protective passive layer can be formed, which protects the coating against corrosion. These local differences can be in nano dimension, whereby these differences cannot be detected in the XRD analysis used in this study. Further analyses such as particle diagnostic have to verify this

hypothesis. In-depth analyses using the above-mentioned methods are planned in order to investigate, which mechanisms are mainly responsible for the altered corrosion properties at high powder feed rates. For the thick HVAF coatings, HVAF 01 and HVAF 03, no under-corrosion was detected. For the thin HVAF coatings, HVAF 02 and HVAF 04, light under-corrosion was detected in the interface. Normally, it is assumed at high overpotentials that the polarization curve of HVAF 02 has to approach the substrate's curve due to the observed under-corrosion, see Figure 5. One reason for not approaching the substrate's curve might be, that under-corrosion was only observed in very few places, which means the influence of the substrate cannot be observed in the polarization curve. Thus, the coating thickness and density of these samples is not sufficient to protect the substrate from corrosion at such high overpotentials, but it has to be stated that such high overpotentials are not realistic for most applications. Further investigation should check if under-corrosion would also occur if the overpotential is kept smaller. To sum up the results of the corrosion tests, it can be stated that the powder feed rate has a significant influence on the corrosion behaviour of the HVAF samples. For the samples WAS sealed and HVAF 03, almost the same rest potentials were measured. However, the corrosion depth d_{cd} of all HVAF samples is lower than of the WAS sealed sample.

4. Conclusion and outlook

The goal of this study was to investigate the influence of high powder feed rates on coating properties of a cost-efficient wear and corrosion protection coating system, using the HVAF process and the novel FeCrB/WC-Co feedstock material. Therefore, coatings with a thickness of $d_{ct} \approx 130$ µm and $d_{ct} \approx 260$ µm and with a powder feed rate of $p = 40$ g/min and $p = 200$ g/min were produced. Subsequently, the microstructure, corrosion and wear properties of the new coating system were investigated and compared to a FeCrBMnSiC WAS sprayed reference protection coating system. The results can be summarized as follows:

- Dense, crack-free and low-oxide coatings can be produced with powder feed rates upto $p = 200$ g/min.
- The surface roughness Ra of the HVAF samples is significantly lower compared to the WAS sample. The powder feed rate and coating thickness of the HVAF samples have a negligible influence on the surface roughness.
- The XRD analyses show that the HVAF process did not alter the phase composition of the processed feedstock material significantly.
- With high powder feed rates of $p = 200$ g/min and a coating thickness of $d_{ct} \approx 270$ µm, the HVAF coatings achieve a rest potential comparable to that of the WAS sealed reference coatings.
- The powder feed rate has a significant influence on the corrosion behaviour of the HVAF samples. Corrosion resistance decreases with increased powder feed rate. Nonetheless, all HVAF samples exhibited a higher corrosion resistance than the sealed WAS reference.
- HVAF coatings show a superior wear behaviour under considered testing conditions in comparison to WAS coating independent of the powder feed rate.

Investigations under the considered testing conditions show that novel Fe-based coatings, exhibiting superior wear and corrosion resistance in comparison to conventional coating, can be realised by means of HVAF spraying. Here it must be noted that, depending on the real loading conditions, a sufficient coating thickness should be ensured to avoid under corrosion. In the near future the following investigations are planned to investigate the developed coating systems more detailed:

- Further analysis, e.g. SEM/EDS, are necessary to explain why the rest potential U_R decreases with increasing the powder feed rate.
- Investigation of the corrosion behaviour of the HVAF samples for lower overpotentials, to compare the corrosion at the beginning of the anodic saturation.

- Processing of the same feedstock material with a grain size fraction G = -20 +3 µm, to investigate if an even smaller grain size fraction has a positive effect on the wear and corrosion properties.

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