Impact resistance of WC-Co reinforced HVAF-sprayed FeCrB-coatings

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Impact resistance of WC-Co reinforced HVAF-sprayed FeCrB-coatings

K Bobzin, W Wietheger, H Heinemann, J Sommer and M Schulz*
Surface Engineering Institute, RWTH Aachen University
Kackertstrasse 15, 52072 Aachen, Germany
*e-mail: schulz@iot.rwth-aachen.de

Abstract. This study describes the development of cost-effective FeCrB-coatings and the evaluation of these novel coatings against impact loads. The cost reduction is achieved by using a new and economical iron-based feedstock material with a small particle size distribution, which can be processed at high feeding rates with the HVAF process. The reduced particle size distribution enhances the application of near-net-shape coatings, minimizing the need for expensive, time-consuming grinding postprocesses. Thicker coatings usually result in higher thermal insulation, whereas thin near-net-shape coatings can reduce the thermal insulation. Dense FeCrB and FeCrB/WC-Co coatings were applied by HVAF with feeding rates of $\dot{m} = 200 \, \text{g/min}$ and a particle size distribution of $-20 + 3 \, \mu\text{m}$. Through curvature measurements during the application it is shown that by integrating WC-Co into FeCrB the tensile residual stress state of the coating was minimized, which in turn increased the fracture toughness of the coating. The evaluation of the impact resistance was investigated by an impact test. The results show that the crater volume was halved by adding WC-Co in the FeCrB-coating. However, it is shown that the plastic deformability of the coating is minimized and a stronger cracking behavior can be observed.

1. Introduction
Iron-based coatings are in the current focus of research, since there is an increasing demand for economical and ecological coatings for the protection against corrosion and wear. In order to provide an adequate protection, established coatings such as Cr$_3$C$_2$/NiCr are often applied using high velocity oxygen fuel (HVOF) or high velocity air fuel spraying (HVAF). Due to the higher costs, such coatings are only an economic solution for applications where very high loads are present. A much more cost-effective process is wire arc spraying (WAS). The high cost efficiency results from the high application rates of $\dot{m} \leq 500 \, \text{g/min}$ [1] and the usage of cost-effective feedstock materials. A typical iron-based feedstock material, that is applied by means of WAS are FeCrBSiMnC cored wires. However, due to the high roughness in as-sprayed condition, expensive and time-consuming grinding is often necessary. Furthermore, the high porosities of $\eta = 3-20 \%$ [2] of WAS coatings, require high coating thicknesses of 800-1,200 $\mu\text{m}$ [3, 4] to ensure sufficient corrosion protection in some applications. Sealers can improve the corrosion properties, yet they need again an additional post-production step. Sufficient corrosion protection could also be achieved with coatings with lower coating thicknesses and lower porosity using HVOF or HVAF, where the porosity is $\eta \leq 2\%$ [2]. If hard materials, e.g. WC, are sufficiently attached in a surrounding matrix, the wear properties of these composite coatings can be improved. For example, Terajimi et al. showed that the wear can be significantly reduced by increasing the WC/Co content in a FeCrMoCB-WC/Co coating against an Al$_2$O$_3$ ball in a reciprocating wear
Similar results were observed by Bolelli et al. with FeCrAl-WC coatings \[6\]. They compared the wear behavior of the iron-based coatings with established HVOF-sprayed coatings made of WC/CoCr. While the wear coefficient was comparable, the iron-based coatings showed a significantly more brittle behavior. They explain this with a lower cohesion and higher oxide content of the HVOF-sprayed coating. In \[7\] FeCrNiSiBC+WC coatings were applied by HVAF. This process is a modification of the established HVOF system, where instead of oxygen, air is used as oxidizing agent. In this study from Bolelli et al. it is shown that by the integration of WC particles in the FeCrNiSiBC coating the wear coefficient can be reduced by an order of magnitude. However, they also show that an exact process control is necessary, since otherwise un- or partially molten particles can weaken the cohesion of the coating and micro cracks along the splat boundaries occur during the wear test.

In own previous studies the basic wear and corrosion properties of FeCrB/WC-Co-coatings were investigated \[8–10\]. In \[8\] crack-free FeCrB/WC-Co coatings were produced with a powder mass flow rate of $\dot{m} = 40$ g/min and a particle size distribution of -45 +11 µm. By reducing the particle size distribution to -32 +11 µm homogeneously molten particles were realized \[9\]. Through the finer distribution and the higher possible gas flow rates compared to HVOF, crack-free coatings with an increased feed rate of $\dot{m} = 200$ g/min could be achieved. The higher mass flow rates of the iron-based coatings are beneficial for a further reduction of the production costs compared to the state of the art. In \[10\] a FeCrB/WC-Co-powder with a particle size distribution of -20 +3 µm and the same chemical composition as described in \[8, 9\] was used to achieve a near-net-shape coating. The wear in the pin-on-disk test was comparable to the results obtained in \[9\], but the corrosion resistance was significantly improved.

Periodic impacts, such as those caused by vibrations in machines, are a special type of load for coatings. Therefore, the influence of WC-Co in the FeCrB/WC-Co coating on the impact behavior is investigated in this study. Knotek et al. have introduced an impact machine to evaluate the impact behavior of thin CVD and PVD coatings \[11\]. Based on this, Bouzakis et al. \[12\] made the first basic investigations with this new investigation method. However, thermal sprayed coatings have not been investigated very much so far. First investigations on thermally sprayed Cr$_3$C$_2$/NiCr coatings were made in 2017 \[13\]. Results on FeCrB/WC-Co are not yet available and are discussed in this study.

### 2. Experimental and materials

As substrate flat samples of 1.0038 (EN 10027, S235JR) were used. Prior to the coating process, all samples were grit blasted with corundum (F16) at a pressure of $p = 0.4$ MPa to roughen the surface. For the application of the coatings, commercially available powders from Above Material Technology Co, Ltd, Beijing, China, were used. These powders are distributed in Germany by the company GTV Luckenbach under the trade names 80.44.0S-0 and 80.44.0S-3. The WC/Co content is 15 percent by weight in the WC/Co-containing coating. The chemical composition of the powders is given in Table 1. While the high Cr content shall provide adequate corrosion protection, the contents of hard phase-forming elements C and B shall ensure adequate wear protection \[3, 4\].

| Coating | Particle size distribution | Fe | Cr | B | C | W | Co |
|---------|---------------------------|----|----|---|---|---|----|
| F20     | -20 +3 µm                 | Bal| 22-26 | 4.3-5 | 0.4-0.7 | - | - |
| W20     | -20 +3 µm                 | Bal| 20-23 | 3.5-4 | 1-1.2 | 10-12 | 1-1.5 |

Subsequently, the samples were coated with the parameters given in Table 2. All coatings of this study were applied with the HVAF system AK-07 (Kermetico Inc., Benicia, USA). The parameter set for F20 was developed based on experience with other Fe-based powders. However, no dense coating could be achieved with this parameter set and the WC-Co-containing powder. Therefore, the spray distance was
increased and the pressure of the compressed air was reduced to increase the heat input to the particles. Through stronger flattening of the hotter particles a dense coating could be applied in case of W20, too.

### Table 2. Spraying parameters of the investigated samples.

| Sample name | Propane [MPa] | Compressed air [MPa] | Surface speed [mm/s] | Powder feed rate [g/min] | Stand-off-distance [mm] | Cooling air [MPa] |
|-------------|---------------|---------------------|---------------------|--------------------------|------------------------|-----------------|
| F20         | 0.56          | 0.72                | 4,500               | 200                      | 180                    | 0.15            |
| W20         | 0.56          | 0.63                | 4,500               | 200                      | 350                    | 0.15            |

In a preliminary investigation it was shown that the coating thickness per pass has a considerable influence on the crack formation in the coating [8]. It is assumed that the thermal load per pass is higher and thus the thermally induced residual stresses are increased. In order to reduce these residual stresses, the samples were fixed on a cylindrical sample holder. The sample holder rotates, while the HVAF gun movement is restricted to a linear movement parallel to the rotation axis of the sample holder. Due to the high surface velocity of 4,500 mm/s the thermal load per pass is reduced. The exact design can be taken from [9].

The evaluation of the microstructure was performed with the light microscope Axiophot (Zeiss, Oberkochen, Germany). The phase composition was determined by X-ray diffraction, XRD. The Seifert XRD 3000 X-ray diffractometer (GE Sensing Inspection Technologies GmbH, Hürth, Germany) was used for this purpose. For the measurements a Cu-anode was utilized. The angle of incidence was \( \omega = 10^\circ \) and the step size was set to \( \Delta 2\theta = 0.05^\circ \). The determination of the Vickers micro hardness HV was performed according to DIN EN ISO 6507 with the hardness tester Micromet 1 (Bühler Ltd, Illinois, USA). To determine the hardness, a load of \( F = 0.98 \) N and a holding time of \( t_{\text{mh}} = 15 \) s were used at 15 measuring points in each coating. The fracture toughness was calculated by use of indention method [14]. For the determination, cracks were created in the cross sections of the coatings using the hardness measurement system Wilson Hardness 452 SVD device (Bühler Ltd, Illinois, USA) with a Vickers indenter. A high load of load \( F_P = 98 \) N was used to produce sufficient cracks. The maximum crack length \( c_{\text{max}} \) was determined with the light microscope. The Young's modulus \( E \) was determined with the Fischerscope HM2000 (Helmut Fischer GmbH, Sindelfingen-Maichingen, Germany). The fracture toughness was calculated according to equation 1 [14].

\[
K_{IC} = 0.015 \left( \frac{E}{HV} \right)^{1/2} \left( \frac{F_P}{c_{\text{max}}} \right)
\]  

As shown in [6], the residual stress state of the coating has a significant influence on its wear resistance, which is why the residual stresses of the coatings were measured in this study. During the coating process, residual stresses are generated primarily by impact and shrinkage of the particles. The impact can induce compressive residual stresses, whereas the hindered shrinkage of the particles induces tensile residual stresses. In addition, thermal residual stresses occur after the coating process due to the different thermal expansion coefficients of the coating and the substrate. The residual stresses lead to a curvature of the coating. This curvature is determined by means of ICP8 sensor (ReliaCoat Technologies, LLC, Setauket, USA) and three precision lasers. Through the measured curvature the residual stresses in the coating are calculated. From the literature it is known that tensile residual stresses can enhance crack propagation, therefore the results of this investigation are compared with the fracture toughness [15].

The evaluation of the impact resistance was performed using the impact tester Apollo NXG (impact-bz Ltd, London, UK). In this test, a WC-Co ball with a diameter \( \varnothing = 5 \) mm impacts cyclically a sample with a defined force \( F_N = 1,000 \) N. The frequency was set to \( f = 50 \) Hz and \( n = 500,000 \) impacts were
performed. The crater volume was determined by means of a confocal laser microscope VKX 210 (Keyence Corporation, Osaka, Japan). The wear mechanisms were investigated by scanning electron (SEM) and light microscopy. The SEM investigations were performed with the scanning electron microscope Phenom XL (Thermo Fisher Scientific, Waltham, USA). For an evaluation of the crater volume, both samples were ground identical. In analogy to the previous investigations, a 10 µm diamond pad was used as the last step [8–10]. To influence the residual stresses in both coatings equally, a fully automatic grinding routine with the grinding machine Saphier 550 (ATM, Golling, Austria) was used, see Table 3.

Table 3. Grinding routine used for the investigated samples.

| Step 1 | Step 2 | Step 3 | Step 4 | Step 5 |
|--------|--------|--------|--------|--------|
| Grind Pad | 180 SiC* | 400 SiC* | 800 SiC* | 1,200 SiC* | 10 µm Diamond |
| Velocity [rpm] | 100 | 100 | 100 | 100 | 100 |
| Force [N] | 30 | 30 | 30 | 30 | 30 |
| Time [min] | 6 | 2 | 2 | 2 | 6 |

In order to prove how far the grinding process influences the impact resistance, coatings in the as sprayed condition were also examined. All experiments were repeated three times on each samples, to ensure that the results are statistically reliable.

3. Results and discussion

Figure 1 shows the morphology of the investigated coatings. For both coatings a coating thickness of about 150 µm was obtained. Figure 1a shows the cross section of the coating F20 without WC-Co inclusions. No splat boundaries can be identified, which suggests a low oxidation of the particles in flight. Only in the vicinity of the surface small pores can be identified, due to the missing peening-effect of subsequent impacting particles. Figure 1b shows the cross section of the WC-Co containing FeCrB coating W20. Also in this coating, it is hardly possible to identify the splat boundaries. In contrast to the WC-Co free coating, no pores can be identified even in the vicinity of the surface. This can be explained by an additional peening-effect due to the WC particles. Both coatings suggest a good bonding to the substrate, since little to no pores at the interface to the substrate are visible.

![Figure 1](image1.jpg)

Figure 1. Cross section of the examined coatings: a) F20, b) W20.
Figure 2 shows the diffractogram of the coatings. Unwanted phases such as oxides could not be detected by XRD. As expected, the detected main peaks can be assigned to Fe and WC in the case of WC-Co containing coating. Furthermore, the presence of borides in the coating is evident. The broadening of the peaks and the reduction of the peak intensity also suggests nanocrystalline phases [16].

**Figure 2.** X-Ray diffractogram of the examined coatings. Only the dominant peaks of the phases are shown.

Figure 3 shows the results of the micro hardness measurements. Both coatings have a comparable micro hardness of around 900 HV0.1. The quartiles shown in this diagram indicate the limit where 75 % of all hardness measurements are below the 3rd quartile or above the 1st quartile. The medians are in the middle of these quartiles, showing that this measured micro hardness values have a normal distribution. The small width between the quartiles and the position of the medians show a homogeneous hardness distribution in the coating. However, a significant influence on the hardness by the integration of WC-Co in FeCrB could not be observed. At the chosen load, the hardness seems to be more influenced by the FeCr matrix than by the WC-Co inclusions. Only at individual points in the vicinity of WC-Co-particles a significantly higher hardness value can be measured in the W20 coating, which in turn is shown in the higher maximum value of the coating.
Figure 4 shows the residual stresses which are introduced by the coating process. Since the used sample holder could not be used with the ICP8 sensor, the velocity at which the spray gun passes over the sample was reduced to $v_{\text{rel}} = 2250 \text{ mm/s}$ and the mass rate was set to $\dot{m} = 100 \text{ g/min}$, to achieve a comparable coating thickness per pass of 12.5 $\mu$m/pass. The remaining parameters are shown in Table 2. The coatings were also applied with a coating thickness of 150 $\mu$m. Both coatings exhibit tensile residual stresses. With regard to the small particle size and the low melting temperature of the FeCrB particles [8], a completely or partially molten state can be assumed. Due to the rapid cooling, the flattened particles remain in a strong tensile residual stress state. A comparison between the coating W20 and F20 shows that in the WC-Co-containing coating W20 significantly lower tensile residual stresses exist. The WC-Co particles impact on the sample in a solid state. The impact of the heavy WC-particles introduces compressive residual stresses that counteract the tensile residual stresses. Since the influence of the quenching stresses is greater than the compressive residual stresses induced by the peening effect, the resulting deposition stresses are on tension.

The thermally induced residual stresses resulting from the shrinkage of the substrate and the coating after the deposition process are slightly higher in the case of W20. Due to the hotter process parameters, originating from the lower volume flow of compressed air, the particles were presumably heated to higher temperatures. Consequently, the coating and its substrate reached higher temperature as well, Figure 4. However, the thermally induced residual stresses are an order of magnitude lower than the deposition residual stresses, which is why the resulting residual stresses are predominantly determined by the deposition residual stresses. It can be assumed that the crack propagation behavior and consequently the impact behavior is influenced by the residual stress state of the coating. In order to show the influence of the residual stresses on the crack propagation behavior, fracture toughness tests were performed.
Figure 4. Residual stress states and substrate temperatures of the examined coatings systems measured by ICP8 Sensor.

Figure 5 shows the results of the fracture toughness tests. The coating W20 shows a slightly higher fracture toughness. The higher variation of the WC-Co-containing coating can be explained by the more inhomogeneous microstructure of the coating. In accordance with the results of the hardness measurement, a higher maximum value is also shown for the fracture toughness of the coating W20. A reason for this is that the indenter penetrates less in the vicinity of WC-Co inclusions, resulting in shorter crack lengths. Another reason that has to be considered is that the higher tensile residual stresses in the F20 coating promote the crack propagation in the coating and therefore slightly lower values were measured in the coating F20.

Figure 5. Fracture toughness of the examined coatings.
To analyse the influence of the previously determined coating properties on the impact behaviour, the crater volume of the coatings was determined as a quantifiable variable of the impact behaviour. Figure 6 shows the crater volumes of the F20 and W20 coatings after the impact tests. It is shown that the crater volume was significantly reduced by integrating WC-Co particles in FeCrB. It can be assumed that the plastic deformability was strongly reduced by the incorporated WC-Co particles. However, it should be noted that the real volume can be a little higher due to spallings at the interface. The crater volume of the as sprayed coatings cannot be determined exactly due to the roughness peaks, but the ratio between the crater volume in polished and as sprayed conditions is comparable between F20 and W20. HVOF sprayed Cr$_3$C$_2$/NiCr coatings were already used as a reference for wear and corrosion resistance in a previous study [8]. The impact behavior of a Cr$_3$C$_2$/NiCr coating was investigated in a previous study [13]. The results show that with the same test parameters and device a comparable crater volume could be achieved with the coating W20.

Figure 6. Crater volume of the examined coatings, the error bar indicates the standard deviation.

Figure 7 shows detailed images of the impact craters of the polished samples. In sample F20, circular cracks can be detected in the peripheral zone. These cracks are caused by high stresses at the edge of the impact and indicate a cohesive failure. While these cracks are relatively small in case of the coating F20, they are very large in case of W20. In W20 large cracks can be identified, which also indicate an adhesive failure, what is also seen in the cross section of this coating after the impact test, Figure 7e. In the impact ground cracks can only be detected for W20. The corresponding cross sections show that the cracks are in the vicinity of the surface and have little depth. The coating F20 shows no cracks at the impact ground. It can be assumed that the energy on impact at F20 was reduced by plastic deformation, whereas in the case of W20 the energy led to cracking due to the lower deformability. The hypothesis is supported by the cross sections of the coatings. The stronger deformation at F20 can be recognized by the distinct curvature of the coating. Cracks can only be identified at the edges. The curvature is much less pronounced for W20 and a much stronger crack network can be identified.

Both coatings show a radial crack pattern. In case of F20 many smaller radial cracks with a maximum length of $l \approx 300 \mu m$ can be identified. The coating W20 shows fewer but significantly longer radial cracks with a maximum length of $l \approx 950 \mu m$. A more detailed analysis of the cracks, Figure 7c and 7f, shows that such cracks propagate mainly along weak points like partially molten particles, pores and splat boundaries. This observation is consistent with the observations of Bolelli et al. of the crack propagation of an HVAF-sprayed FeCrNiSiBC coating in the ball-on-disk test [7]. The coating W20 shows an adhesive failure at the bottom of the impact crater. In addition, a significantly more pronounced crack network can be identified. The detail images show that cracks propagate between the WC-Co-inclusions. Here, especially the cohesion between the Co-matrix and the iron-based coating seems to be insufficient.
Figure 7. Wear marks of F20 (a-c) and W20 (d-f): a,d: overview of the impact crater; b,e: cross section of the impact crater; detail view of the introduced cracks.

The results of the fracture toughness test show that the indentation depth was reduced in W20 and a stronger crack propagation was most probably prevented by a reduced tensile residual stress state. However, with higher indentation forces of the impact tester, a significantly more pronounced crack network could be detected in case of W20. This is due to the fact that cracks are concentrated between the WC/Co particles. Due to the cyclical impacts, such cracks are ripped off more strongly, which results in longer cracks compared to F20. Therefore, the main influence on crack formation in the coating seems to be the distribution of the WC-Co particles and the indentation force. Reduced tensile residual stresses appear to be advantageous, which is shown by an increased fracture toughness. The results show that
the integration of WC-Co can significantly improve the dimensional stability under impact stresses, but that the WC-Co content must be chosen with regard to the load, to avoid cracks and delaminations.

4. Conclusions
The aim of this study was to investigate the influence of WC-Co-inclusions in FeCrB coatings. For this purpose, HVAF-sprayed FeCrB and FeCrB/WC-Co coatings were applied with a powder feed rate of \( \dot{m} = 200 \text{ g/min} \) and a coating thickness of \( s = 150 \mu\text{m} \). Impact tests with a load of \( F = 1,000 \text{ N} \) and a number of cycles of \( n = 500,000 \) were performed. The residual stresses, fracture toughness, microhardness, phase composition and microstructure of the coating were investigated to identify the influences on the impact behavior. The results can be summarized as follows:

- The crater volume can be reduced significantly by integrating WC-Co into the FeCrB coatings. However, the WC-Co inclusions cause the cracks to agglomerate between the WC-Co particles. The splat boundaries between the WC-Co particles and the FeCr matrix are an additional weak point in the coating where cracks can develop and propagate.

- The low tensile residual stress state of the WC-Co-containing coating W20 are the reason for the higher fracture toughness. However, the high impact loads in the impact test outweigh the fact that the coating can be deformed less plastically due to the integration of the WC-Co particles and tends to crack more easily.

- Cracks propagate primarily at weak points in the coatings, such as pores and splat boundaries of partially molten particles.

In previous studies it could be shown that the integration of WC-Co can be useful for certain wear loads [8–10]. In the case of impact loads, dimensional stability can be significantly improved, but crack propagation is also increased. To minimize crack propagation, the amount of WC-Co-particles must be chosen with regard to the load.

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