Novel Fe-based and HVAF-sprayed coating systems for large area applications

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Abstract. In paper-machines, components such as dryer cylinders are often exposed to a combined load of wear and corrosion and, therefore, require an adequate surface protection. A high thermal conductivity of these coatings is desired, to increase the overall thermal efficiency. In this study, a novel high velocity air-fuel- (HVAF-) sprayed FeCrB/WC-Co coating is compared to an industrially established sealed FeCrBMn coating applied by wire-arc-spraying (WAS) in terms of wear behaviour of aged samples and thermal conductivity. The chosen feedstock material had powder fractions of -32 +11 µm and -20 +3 and were applied with a powder feed rate of \( m = 200 \) g/min by means of HVAF. A high powder feed rate and fine powder fractions were considered to reduce the required time to coat large areas and to reduce the post-processing effort. The samples were aged in saturated NaCl-solution steam at a temperature of \( T = 105 \) °C for \( t = 100 \) h. For the aged HVAF samples, a low influence of the aging on the wear rate was observed for both powder fractions. In contrast, for the aged WAS reference coating, a noticeable higher wear rate was measured. The thermal conductivity of the HVAF-sprayed coatings is significantly influenced by the employed powder fraction.

1. Introduction
In order to dry raw material during paper production, dryer cylinders are used. These cylinders are heated by hot steam, which flows through the cylinders [1] and are typically made of grey cast iron or mild steel such as 1.0038. In many applications, both materials have a poor wear and corrosion resistance and need to be protected against corresponding loading conditions, e.g. with thermally sprayed coatings. In case of dryer cylinders, it is state of the art to apply Fe-based coatings by WAS or cemented carbides by high velocity oxygen-fuel (HVOF) spraying [2]. Both coating systems have several advantages and disadvantages.

Fe-based coatings applied by WAS exhibit a good cost-efficiency, due to the inexpensive raw materials and the high achievable wire feed rates of \( m = 100 \) g/min [3]. A high corrosion and wear resistance of Fe-based materials can be achieved by including a high amount of Cr and B in the alloy respectively [4, 5]. The microstructure of Fe-based WAS coatings is porous and the coating surface is rough in the as-sprayed condition [6]. Therefore, a time consuming grinding process is necessary to achieve the required surface roughness. Experience shows that high coating thicknesses of \( s_c = 700 – 1.200 \) µm [7, 8] are necessary to achieve the required corrosion protection. The corrosion resistance can be further increased by sealing the pores using an epoxy resin, which requires an additional process step. When using epoxy resin-based sealers, the long-term temperature resistance of the sealer must be considered as well. The high coating thicknesses and a low thermal conductivity of these coatings result in a decreased heat flow from the inner part of the cylinder to the coated surface. For
this reason, the coating insulates the cylinder thermally and the thermal efficiency of the whole system can decrease.

Thinner, more near net shape and denser coatings can be produced by applying cemented carbides with HVOF-spraying [1]. For these coatings, a lower powder feed rate, higher hardness and higher material prices have to be considered. Fe-based feedstock materials can be a cost efficient alternative to cemented carbide coating systems. It has already been shown that novel Fe-based and HVOF-sprayed coating systems can protect mild steel substrates as good as industrially established cemented carbides, depending on the load collective [9, 10]. A further trend in thermal spraying is the use of finer feedstock materials [11], due to the achievable increased powder deposition efficiency, decreased porosity and surface roughness. However, experience shows that processing of finer powder feedstock materials by means of HVOF spraying can result in nozzle clogging.

The HVAF process is a modification of the HVOF process [12, 13]. It uses air instead of O2 as oxidant [12, 13]. Due to the N2 in the air, high gas flow rates and a lower flame temperature, compared to HVOF, are achievable. The high gas flow rates enable high powder feed rates [14] and high particle velocities [13]. The lower flame temperature can result in a lower particle temperature, which might be helpful to process finer feedstock materials without nozzle clogging [12]. According to a study of Verstak et al., the lower particle temperature and the high particle velocities can result in dense and smooth coatings with a low oxide content [14]. These results were also confirmed in [12, 13, 15–17].

In the studies [18–20], a novel HVAF sprayed FeCrB/WC-Co coating system with high wear and corrosion resistance was developed. FeCrB/WC-Co was chosen as a cost efficient feedstock material in the powder fractions of -32 +11 µm and -20 +3 µm [19, 20]. Using the HVAF-process, it was possible to produce dense, near net shape coatings with high powder feed rates and to process the finer feedstock materials. These investigations showed that the new coating system provides comparable or even better corrosion and wear resistance than the sealed WAS FeCrBMn reference coating system, depending on the powder feed rate and powder fraction. In the current study, the novel HVAF-sprayed FeCrB/WC-Co coating system is analysed with regard to the wear behaviour in aged condition. Moreover, the thermal conductivity and the thermal resistance of the final coating system have been analysed. The aging of the samples was performed in a corrosive environment at increased temperature. These conditions are similar to those in paper machines.

2. Experimental setup and materials

The mild steel 1.0038 (S235JR) is widely used for dryer cylinders and was, therefore, chosen as substrate material. The substrates with the dimensions of 40 x 50 x 8 mm³ were cleaned and roughened using grit blasting. The coatings were applied onto the prepared surfaces via HVAF and WAS. The chemical compositions of the used feedstock materials are given in Table 1.

| Table 1: Chemical compositions of FeCrB/WC-Co and FeCrBMn (SP112) feedstock material, as given by the producer companies [wt.%]. |
|-----------------|-----|-----|-----|-----|-----|-----|
| Alloy           | Fe  | Cr  | B   | C   | W   | Co  | Si  | Mn  |
| FeCrBMn (Sp112)| Bal.| 27.5 - 29 | 3.8 | 0.1 | -   | -   | 1.5 | 1.5 |
| FeCrB/WC-Co     | Bal.| 20 - 23  | 3.5 - 4 | 1 - 2 | 10 - 12 | 1 - 1-5 |

The wire FeCrBMn (Corodur Fülldraht GmbH, Willich, Germany) is often used for protecting components against corrosion at increased temperature and, chosen therefore as reference. For the HVAF process, the powder FeCrB/WC-Co (Above Material Technology Co., Ltd., Beijing, China) was chosen as feedstock material. Both materials FeCrB/WC-Co and FeCrBMn have high amounts of Cr and B to achieve a high corrosion and wear resistance. WC-Co was added to the FeCrB-powder to further increase the wear resistance.
The process parameters used in order to apply HVAF and WAS coatings are given in [18–20]. An overview of all samples and the varied process parameters in this study are given in Table 2. The FeCrB Mn coating applied by means of WAS was used as reference and is designated hereafter as WAS-Ref. The WAS-system G30/4SF-Push-LD/U2 of Oerlikon Metco AG (Winterthur, Switzerland) was used to apply the reference coatings. To increase the corrosion resistance of the sample WAS-Ref, it was sealed with the epoxy based industrially established sealer #HM2407 (Diamant Metallplastic GmbH, Mönchengladbach, Germany). The HVAF-spraying system AK-07 (Kermetico Inc., Benicia, USA) was used to apply the FeCrB/WC-Co coatings.

| Sample name | Process | Powder fraction [µm] | Powder feed rate ṁ [g/min] |
|-------------|---------|----------------------|--------------------------|
| WAS-Ref*    | WAS     | Cored wire           | 110                      |
| Fe/W D-32  | HVAF    | -32 +11              | 200                      |
| Fe/W D-20  | HVAF    | -20 +3               | 200                      |

*sealed

The experimental setup developed in [19] was used in this study to apply HVAF coatings, see Figure 1. With this experimental setup a thin layer thickness of approximately 13 µm can be achieved, even for high powder feed rates of ṁ = 200 g/min. Using this setup, crack-free coating could be produced with high powder feed rates [19, 20].

2.1 Aging test and wear behaviour of aged samples

Dryer cylinders are exposed to a combined load of wear and corrosion. Corrosion reactions can change the chemical bonds, increase the volume by including additional atoms or lead to material loss by dissolution. Any of these mechanisms can reduce the cohesion between two splats which in turn, can reduce the wear resistance. To investigate the influence of corrosion on the wear behaviour, coated samples were aged in application-oriented corrosive conditions and then investigated regarding their wear behaviour using the Pin-on-Disc (PoD) -test to simulate the combined load. Before the aging, all samples were ground and polished to Ra ≈ 0.5 µm using a 10 µm diamond pad to ensure all sample have comparable initial conditions. The polished samples were then aged using the experimental setup illustrated in Figure 1 b). The coatings were exposed to saturated NaCl steam at a temperature of T = 105 – 110 °C for a time period of t = 100 h. Subsequently, the wear resistance of the aged samples was investigated using a PoD tribometer from CSM instruments (Freiburg, Germany). Al₂O₃-balls with a diameter of Øcb = 6 mm were used as counterbody, in order to force an abrasive type of wear.
and to avoid adhesion. The further PoD-test parameters are listed in Table 3. The wear tracks were analysed using the confocal laser scanning microscope (CLSM) VKX-210 (Keyence Corp, Osaka, Japan). Furthermore, the cross-section of the wear tracks was determined using CLSM and the wear rate was calculated using equation (1). The wear rates of the aged samples were compared to the wear rates of not-aged samples, determined in studies [19, 20]. In a last step, cross-sections of the aged and worn samples were prepared and investigated using the light microscope Zeiss Axiophot (Carl Zeiss AG, Oberkochen, Germany).

### Table 3: PoD test parameters.

| Parameter       | Value     |
|-----------------|-----------|
| Wear track diameter $d$ | 10 mm     |
| Wear track length $s$  | 1,000 m   |
| Normal load $F$     | 10 N      |
| Rotation speed $v$   | 100 mm/s  |
| Test temperature   | Room temperature (RT) |

$K \left[ \frac{\text{mm}^3}{\text{Nm}} \right] = V \cdot F \left( s \cdot \frac{d}{dt} \right)^{-1}$

(1)

#### 2.2 Determination of the thermal conductivity and thermal resistance

The thermal properties of the wear and protection coatings influence the energy efficiency of the dryer cylinder and therefore the overall operating costs. A higher thermal conductivity and a thinner coating, compared to the reference, can decrease the thermal resistance of the system consisting of coating and cylinder. The thermal conductivity $\lambda$ depends on the thermal diffusivity $\alpha$, the density $\rho$ and the thermal capacity $c_p$ of the coating. All values were measured using freestanding coatings. These coatings were produced on a special substrate, which enables peeling of the coatings from the substrate. In contrast to the samples used for aging and wear investigation, the samples used for determining the thermal properties of coatings were produced with a powder feed rate of $\dot{m} = 100$ g/min, due to the speed limit of the robot. The further process parameters were kept constant. The thermal conductivity $\lambda$ of coatings was calculated using equation (2) [21]. The density $\rho$ was measured using the gas pycnometer AccuPyc 1330 (Micromeritics GmbH, Aachen, Germany) at $T = \text{RT}$. It was assumed that the density stays constant in the considered temperature range. The thermal diffusivity $\alpha$ was determined using the laserflash system Flashline 4010 (Anter Corp., Pittsburgh, USA) and the heat capacity $c_p$ was determined using the thermo analysis system Setsys Evolution (Setaram Instrumentation, Caluire, France). Both parameters were determined at a temperature of $T = 200$ °C. In the literature, steam temperatures between $100 \degree C < T < 300 \degree C$ are mentioned for dryer cylinders, depending on the type of paper [1, 22].

$\lambda \left[ \frac{W}{\text{mK}} \right] = \alpha \cdot \rho \cdot c_p$

(2)

The thermal resistance of the different coatings was calculated according to equation (3) [21]. The following assumptions and approximations were made:

- Length of rotary dryer ($L$) = 3 m
- Inner radius of rotary dryer ($r_{i1}$) = 0.9 m
- Outer radius of rotary dryer ($r_{i2}$) = 0.91 m
- Thermal conductivity of rotary dryer ($\lambda_c$) = 48 W/mK
- Coating thickness WAS-Ref ($s_{ct}$) = 500 $\mu$m
- Coating thickness of HVAF-sprayed coatings ($s_{ct}$) = 250 $\mu$m
3. Results and discussion

3.1 Aging test and wear behaviour of aged samples

The corrosive atmosphere of dryer cylinders was simulated by a saturated NaCl-steam at a temperature of \(T = 105\) - \(110\) °C. In initial conditions, the surface of all coatings and the substrate were comparable and shined metallic bright, see Figure 2. After the aging, the substrate surface was heavily corroded. This result show that the mild steel 1.0038 has no protection against corrosion under investigated conditions. For WAS-Ref, Fe/W_D-32 and Fe/W_D-20, selective corrosion and crevice corrosion were observed at the surface. Crevice corrosion could only be observed at the edge where the sample is in contact with the sealing.

![Figure 2: Images of the aged coating surfaces at initial conditions and after \(t = 100\) h in saturated steam of 0.5 \% NaCl-solution at \(T = 105\) °C](image)

Selective corrosion can be observed distributed evenly over the entire surface of WAS-Ref. In contrast the samples Fe/W_D-32 and Fe/W_D-20 exhibit selective corrosion only in a few areas. Qualitatively, Fe/W_D-20 shows less selective corrosion than Fe/W_D-32. This result confirms the good corrosion resistance of the coatings presented in [20]. It is assumed that selective corrosion is a result of a local Cr segregation. In study [23], the local reduction of Cr content, due to the formation of Borides (Fe, Cr), was observed and proven for HVOF-sprayed FeCrNiBC and FeCrNiMoBC coatings. Selective corrosion can be traced back to the local differences in the amount of free Cr in the coatings, as a result of boride formation and related Cr segregation.

The microstructure of the aged samples is presented in Figure 3 a). No under corrosion was observed in the interface between the coating and the substrate, for all coatings. WAS-Ref exhibits oxidized particles in deeper layers, see Figure 3 a). These particles could have formed during the spraying process or during the aging. In contrast to WAS-Ref, oxidized areas could not be observed in deeper layers for the HVAF-sprayed samples Fe/W_D-32 and Fe/W_D-20. Both samples show a very dense microstructure after the aging, which is comparable to that in as-sprayed condition [19, 20].
In Figure 3 b), cross sections of the wear tracks are presented. Pits in the surface of the wear tracks can be observed for all samples. Compared to the HVAF-sprayed coatings, significantly bigger pits in the wear track can be observed for WAS-Ref. Furthermore, bigger pores can be observed in the vicinity of the wear track for WAS-Ref. It is assumed that these pores are a result of the metallographic preparation and caused by a weakened cohesion in the coating after the aging and wear tests. Using light microscopy and a magnification of 500x, no cracks or a delamination could be observed, for all coatings.

The cross sections and surface images of the wear tracks captured by CLSM are presented in Figure 4. The CLSM-images were taken with the integrated light microscope and coloured in a post processing step to visualize the surface topography of the wear track. The widths of the cross-sections of the wear tracks in not-aged and aged conditions of the HVAF-sprayed conditions looks significantly smaller compared to those of WAS-Ref. A further interesting aspect, which can be observed in the cross-section areas is that the width and height of the wear tracks are comparable for the samples Fe/W_D-32 and Fe/W_D-20 in aged and not-aged conditions. In contrast, the height and width of the wear track of WAS-Ref increases in the aged condition. This aspect is a first indication that the
cohesion in the coating WAS-Ref has been reduced after the aging process due to the formed corrosion products between the splats. Furthermore, non-homogeneous wear tracks, pits and debris were observed for all samples, see Figure 4. Compared to the HVAF-sprayed coatings, the pits and debris of WAS-Ref are significantly more pronounced. It is assumed, that the significantly smaller pits and debris observed in case of the HVAF-sprayed coatings in the aged condition can be attributed to the dense microstructure, the high cohesion of these coatings and the smaller particle size during spraying.

Figure 4: Wear tracks of the aged and not-aged samples and CLSM surface images of the wear tracks of the aged samples for sample a) WAS-Ref, b) Fe/W_D-32 and Fe/W_D-20.

The wear tracks were analysed quantitatively regarding the wear rates of aged and not-aged samples, see Figure 5. Since the substrate severely corroded, the aged samples is not measured for substrate. The wear rates of Fe/W_D-32 and Fe/W_D-30 for aged and not-aged samples are in a similar range between $K = 5.0 \cdot 10^{-6}$ and $K = 6.1 \cdot 10^{-6}$ mm$^3$/Nm. In contrast, the wear rate of the sample WAS-Ref increases significantly after aging. Another interesting aspect can be observed for the deviations in measured wear rates. In case of aged WAS-Ref, the deviation from mean value of wear rate increases significantly after PoD-test. The deviations in case of HVAF-sprayed samples are barely affected by the aging. The higher wear rate and increase in the deviation of the measured values in case of the aged WAS-Ref sample are can be ascribed to a decreased cohesion of the coating, which was caused by corrosion. Although Figure 2 shows selective corrosion of the HVAF-sprayed coatings in a few areas, it seems that the cohesion of Fe/W_D-32 and Fe/W_D-20 remains at a sufficient level under the used test conditions.
Figure 5: Wear rates of the substrate, the WAS-Ref, Fe/W_D-20 and Fe/W_D-32 samples in aged and not-aged conditions; not-aged values taken from [19, 20]. The error bars represent the standard deviation.

3.2 Determination of the thermal conductivity and thermal resistance

Thermal properties are important to describe the thermal insulation of thermally sprayed coatings and thus, are measured in the following. In Table 4 the measured values for the density, specific heat capacity and thermal diffusivity of the coatings are given. The deviations from measured values of thermal diffusivity are not presented, since this results in zero after rounding to third decimal places.

Table 4: Determined density, specific heat capacity and thermal diffusivity of the coatings.

| Density coating [g/cm³] | Specific heat capacity [J/gK] | Thermal diffusivity [cm²/s] |
|-------------------------|-------------------------------|-----------------------------|
| WAS-Ref                 | 6.936 ± 0.049                 | 0.519 ± 0.003               |
| Fe/W_D-32               | 7.332 ± 0.095                 | 0.772 ± 0.004               |
| Fe/W_D-20               | 7.186 ± 0.029                 | 0.676 ± 0.002               |

The calculated thermal conductivities and thermal resistances are shown in Figure 6. The highest thermal conductivity was determined for Fe/W_D-32 with a value of $\lambda = 10.20$ W/mK. Compared to WAS-Ref, the thermal conductivity of Fe/W_D-32 is higher, which reduces the thermal insulation of the dryer cylinder. The lowest thermal conductivity was measured for Fe/W_D-20 with a value of $\lambda = 4.94$ W/mK. Although the samples Fe/W_D-32 and Fe/W_D-20 were produced with a feedstock material of the same chemical composition and both coatings show a very dense microstructure [19, 20], the thermal conductivity of both coatings is significantly different. Two reasons are assumed to be responsible for this phenomenon. A finer powder fraction can result in a higher amount of splat interfaces. These splat interfaces are working as a barrier, which decreases the thermal conductivity. A partly amorphous microstructure was assumed for the fine powder coatings in [20]. Amorphous phases might influence the thermal conductivity, as well. In general, it is assumed that metallic amorphous coatings exhibit a lower thermal conductivity compared to crystalline coatings [24–26]. The thermal conductivity depends among others on the mean free path of electrons and phonons [27]. To decrease the thermal conductivity the mean free path can be decreased by scattering defects in the coating, to
hinder the mobility of electrons and/or phonons. In [26] it was reported that in case of coatings with a mount of amorphous phases, the phonon contribution to thermal conductivity is suppressed and therefore the thermal conductivity decreases. Both theories could explain a lower thermal conductivity of the fine powder coating. Which of these assumptions applies here, must be evaluated in more detail in further investigations, e.g. using transmission electron microscope (TEM).

![Figure 6: Determination of the a) thermal conductivity and the b) thermal resistance at a temperature of \( T = 200 \, ^{\circ}C \)](image)

Besides the thermal conductivity, the coating thickness influences the thermal efficiency of dryer cylinders considerably. The thermal resistance of a dryer cylinder with different coatings was calculated. The bar chart presented in Figure 6 b) shows that Fe/W_D-32 has the lowest thermal resistance, which is the result of the low coating thickness and high thermal conductivity. Although the lowest thermal conductivity was measured for Fe/W_D-20, a theoretical cylinder coated with this coating would still exhibit a lower thermal resistance compared to the same cylinder with the reference coating. This is a result of the higher thickness of WAS-Ref, which need to be assured in order to ensure sufficient corrosion protection. A higher thermal resistance can insulate the dryer cylinder and therefore results in a higher energy consumption. For this reason, the new developed HVAF-sprayed coating system shows a high potential with regard to increasing the energy efficiency in paper machines.

### 4. Conclusions

The goal of this study was to investigate the thermal properties and the wear behaviour of aged samples of the novel HVAF-sprayed FeCrB/WC-Co coating system developed in [18–20]. Therefore, the HVAF-sprayed samples were produced with a powder feed rate of \( \dot{m} = 200 \, g/min \) and a coating thickness of \( s_{ct} = 250 \, \mu m \). Subsequently, the samples were aged for \( t = 100 \, h \) at \( T = 100 - 105 \, ^{\circ}C \) in a saturated steam atmosphere of a 0.5 % NaCl-solution. Afterwards, PoD-tests were performed on the aged samples. The wear tracks were analysed and compared to not-aged samples. Furthermore, the thermal conductivity and thermal resistance of the coatings were determined. The novel coating
system developed by means of HVAF-spraying were compared to an industrial established sealed FeCrBMn coating system, which was produced with a wire feed rate of \( \bar{m} = 110 \text{ g/min} \) and a coating thickness of \( s_{ct} = 750 \mu\text{m} \) by means of WAS. The results can be summarized as follows:

- WAS-Ref showed selective corrosion evenly distributed over the whole surface after aging. In contrast, only slight selective corrosion was observed for the HVAF sprayed FeCrB/WC-Co coatings.
- No under corrosion was observed for any samples.
- The wear rate of the HVAF-sprayed samples was nearly constant for aged and not-aged samples.
- The wear rate of WAS-Ref increased significantly after aging.
- The thermal conductivity of HVAF-sprayed coatings depend strongly on used powder fraction.
- A finer powder fraction leads to a reduced thermal conductivity.
- Since HVAF coatings allow a thinner deposition, the thermal resistance of the HVAF-sprayed coatings was lower compared to the WAS-Ref sample independent of used powder fraction.

In the used laboratory tests, the HVAF-sprayed coatings had a superior wear behaviour in aged conditions compared to the sealed WAS FeCrBMn reference coating. The superior corrosion and wear resistance in not-aged condition was already proven in [19, 20]. The reduced thermal insulation promises a higher efficiency of the paper dryers. In conclusion, the novel HVAF-sprayed FeCrB/WC-Co coatings system has a high potential for large area applications such as for dryer cylinders. Nevertheless, more application oriented investigations and field tests are needed to verify the promising results shown in this study.

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