The tribological and electrochemical behavior of HVOF-sprayed Cr$_3$C$_2$–NiCr ceramic coating on carbon steel

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Abstract

High Velocity Oxygen Fuel (HVOF) is an excellent approach to prepare a good, wear-resistant lamella of Chromium Carbide-Nickel Chrome (Cr$_3$C$_2$–NiCr) on carbon steel for high temperature application. This research investigates the effect of a thin, deposited layer of Cr$_3$C$_2$–NiCr on carbon steel in terms of wear and corrosion properties. The microstructure of the HVOF-sprayed Cr$_3$C$_2$–NiCr coating was characterized at each step by scanning electron microscopy. Wear testing was performed with a pin-on-disk tester. Wear weight loss was examined by applying different loads over a 9048.96 m sliding distance. Experimental results show that the wear resistance of the coated sample reduced the risk of seizure compared to the uncoated sample. An electrochemical test was also performed and the behavior of the substrate in the coated sample was investigated in 3.5% NaCl for 27 days. Electrochemical Impedance Spectroscopy (EIS) showed that the HVOF coating has high corrosion resistance and protects the substrate from NaCl electrolyte penetration. So deposition this layer of ceramic composite is protected oil piping from synergistic attack of seawater during the transport of crude oil to the refinery.

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1. Introduction

Piping involves many processes including welding, cutting, painting, assembling, and outfitting. Erosion and corrosion are the major issues in oil exploration, particularly during the exploration phase, i.e. drilling in crude oil sand slurries, when wear of materials such as drilling tools and pipes may occur. In such cases, degradation may be caused by a number of factors related to the particles, pipe, drilling materials and the environment [1–3].

Commonly chromium carbide-based materials are used in the production of hard coatings which are resistant against high temperature wear applications, such as abrasion, erosion, fretting, and sliding that may reach a maximum operating temperature of 900 °C [4–6]. Since they have a high melting point, Cr$_3$C$_2$–NiCr coatings also offer superior oxidation and corrosion resistance [7,8]. The carbide ceramic phase mainly causes wear resistance while the corrosion resistance is provided by the NiCr matrix [9]. Experts usually agree that micro structures, such as carbide content, carbide particle size, and carbide distribution within splats, as well as porosity and so on, predominately influence the wear resistance of the cermet coating [10–12].

Beside corrosion protection, the main application field of thermal spray coating is wear protection. Thermal spray processes involves melting the deposited materials by a heat source, which may be an electric arc or fuel gas, located at the nozzle of a spray torch. The process is likely to be extremely
practical in coating metals for a variety of purposes. Once it has melted, the atomized material is accelerated against the substrate. The steel specimens of 10 × 10 × 4 mm were sandblasted prior to deposition. Then the ceramic powder (Cr3C2-20%NiCr) was deposited for an average of seven passes. The particle size of the powder was 45–60 µm and the average thickness of the coating was about 200 µm. The temperature and spray distance were kept constant throughout the deposition process.

2.2. Sample preparation and HVOF coating methods

Surface preparation is regarded as a fundamental step in thermal spraying. Being directly related to surface roughness, coating adhesion is controlled by the model of grit blasting. In this study, thermal spray coatings and especially those applied through high velocity oxygen fuel (HVOF) process are being used to replace traditional heat-treated hard carbon steel to prevent or decrease wear loss of machinery and equipment operating in various industrial processes. Also by thermal spray technique can deposited wide range of materials that can be used in oil and gas application with higher mechanical and chemical properties and cheaper compared with the overlay welding technique that is already used [16]. So it has become a preferred process of depositing cermet coatings such as Cr3C2-NiCr and WC-Co because coating deposited by HVOF exhibits high density, low porosity, as well as excellent adhesive strength and many more carbide particles remain in the matrix compared with the plasma spraying process [17,18]. Particularly, understanding the degradation mechanisms due to corrosion and corrosion-wear in aqueous environments is of utmost importance in order to extend tool service life. Guilemany et al. [19] called attention to the accelerating corrosion from the damage caused by coating surface wear by particles and material removal. Some synergism between corrosion and wear, which increases coating degradation, was also observed. The aim of the present study is deposition of a layer of ceramic composite coating on the surface of carbon steel to compare the wear and corrosion resistance of HVOF thermally sprayed Cr3C2-NiCr with carbon steel in 3.5% NaCl solution, mainly in longer immersion time tests. The microws.

### Table 1
The parameters of HVOF coating.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>Combustion pressure</td>
<td>80 Mpa/sq.cm</td>
</tr>
<tr>
<td>Spray distance</td>
<td>200 mm</td>
</tr>
<tr>
<td>Spray angle</td>
<td>90°</td>
</tr>
<tr>
<td>Powder feed rate</td>
<td>50–60 g/min</td>
</tr>
<tr>
<td>Fuel flow rate</td>
<td>20–30 l/hour</td>
</tr>
<tr>
<td>Oxygen flow rate</td>
<td>60–90 m³/hour</td>
</tr>
<tr>
<td>Carrier gas flow rate</td>
<td>20 l/hour</td>
</tr>
<tr>
<td>Barrel length</td>
<td>198 mm/150 mm</td>
</tr>
</tbody>
</table>

2.3. Microstructure characterization

The surface morphologies of uncoated and coated samples as well as cross sections of coated samples were evaluated using a scanning electron microscope (SEM) at high magnification. The cross sections of the coatings were polished for study using SEM. The coating sample phase composition was examined by X-ray diffraction (XRD) with nickel filtered Cu Kα (λ: 1.54056) radiation on a RIGAGU D/Max 2200 diffractometer with 20, 0–90°. Finally, the roughness (Ra) of the coated sample was measured by optical surface texture analyzer (Alicona 3D Infinity Focus). A total of 100 points of Ra measurement was performed on the coated sample.

2.4. Wear and hardness tests

Coating hardness was measured using a HMV-Shimadzu under a 300 g load for 15 s on the cross section of the coatings. A total of ten indentations were made on a coated sample. Microhardness measurement was performed in air at room temperature.

The wear tests were done using a pin-on-disc tester model TR-20LE. The wear tests for coated as well as uncoated specimens were conducted under four normal loads of 5, 10, 15 and 20 N. The specimens were pushed against WC-6%Co paper in dry condition and disc speed of 200 rpm. A track diameter of D=40 mm and sliding speed of v=1 m/s were kept constant during all the tests. Wear testing was run for 60 min until wear rate for coated samples stabilized so the total sliding distance for each sample was around 9048.96 m. Also for increasing the reliability of results, each load was tested for five samples under the same condition. The mean scores of these tests are reported in Section 3.2. Afterwards, the samples
were ultrasonically cleaned in ethanol and then dried. The weight, free from debris, was measured with a microbalance to an accuracy of 0.0001 g. SEM was used to study the wear tracks produced in the coating and the coating volume loss results were reported.

2.5. Electrochemical corrosion test

Electrochemical techniques such as DC (polarization) and AC (impedance) were done using a potentiostat/galvanostat model Auto Lab PGSTAT30 from Ecochemie (Netherlands). Polarization experiments were done using a three-electrode cell where the samples were the working electrode (WE), while a platinum wire and saturated calomel electrode (SCE) were the counter and reference electrodes respectively. The electrolyte was 3.5% NaCl. In this test, the linear scan voltammetry, the corrosion current \( I_{\text{corr}} \) and corrosion potential \( E_{\text{corr}} \) were calculated from the Tafel plots with scan rate of 0.001 V/s.

A frequency range of \( 10^5 – 10^{-2} \) Hz with amplitude potential of \( V_{\text{rms}} = 5 \) mV was used to conduct Electrochemical impedance spectroscopy (EIS). For obtaining the values for the charge transfer resistance across the electrode-electrolyte interface that could be related with parameters from the polarization output, simulations with analog circuits were performed with the experimental data. Having been installed in a computer interfaced with a potentiostat, general purpose electrochemical software (GPES) and frequency response analyzer (FRA) were used for running the AC and DC techniques respectively.

3. Results, analysis and discussion

3.1. Microstructure analysis

The typical microstructures of the HVOF-sprayed \( \text{Cr}_3\text{C}_2–20\text{NiCr} \) coating are shown in Fig. 1. This chemical composition powder was deposited on the carbon steel surface. The cross section and top surface of the coating exhibited dense microstructure with a high cohesion. However, a few pores appear with black color in the micrograph. Dent et al. reported that these pores are due to the un-melted and semi melted particles in the \( \text{Cr}_3\text{C}_2–\) NiCr coatings as identified by their spherical morphology \([21,22]\). This is considered a low porosity value due to the high impact velocity of the coating particles, which causes high density and high cohesive strength of individual splats \([23]\). Fig. 1(b) shows the micrograph of this coating is uniform, homogeneous and free from surface cracks. A relatively homogenous coating without segregation is critical for improving the wear resistance of \( \text{Cr}_3\text{C}_2–20\text{NiCr} \) coating.

X-ray diffraction patterns for \( \text{Cr}_3\text{C}_2–\text{NiCr} \) coated and uncoated samples are shown in Fig. 2(a) and (b). In the coated sample, \( \text{Cr}_3\text{C}_2 \) and the NiCr binder were the major phases identified in HVOF \( \text{Cr}_3\text{C}_2–\text{NiCr} \) coating. Apart from \( \text{Cr}_3\text{C}_2 \) carbide particles, the carbides \( \text{Cr}_2\text{C}_3 \) and \( \text{Cr}_2\text{C}_6 \) were also detected.
present in the X-ray diffraction analyses. Evidently the decarburization of Cr$_3$C$_2$ results in the formation of Cr$_7$C$_3$ and Cr$_{23}$C$_6$. Nevertheless, Cr$_7$C$_3$ and Cr$_{23}$C$_6$ are formed through diverse routes in reference to the results of the current study, excluding Cr$_3$C$_2$ retained from the starting powder. The carbide Cr$_7$C$_3$ was formed through the decarburization of Cr$_3$C$_2$ [21,24]. Additionally, during coating deposition, the Cr$_{23}$C$_6$ in the HVOF Cr$_3$C$_2$–NiCr coating was observed to precipitate. As Zimmermann and Kreye [25] pointed out, since the main diffraction peaks of Cr$_7$C$_3$ and Cr$_{23}$C$_6$ coincide with the lines referring to the NiCr and Cr$_3$C$_2$, the existence of in the as-sprayed coating could not be confirmed solely through X-ray diffraction approach. Furthermore, the decarburization of Cr$_3$C$_2$ appeared to be related with the heating of spray particles and oxidation of Cr$_3$C$_2$ since HVOF cermet coating is commonly deposited in an ambient condition [25]. As confirmed by previous research findings, the main reason for the carbon loss and change of carbide content is that the Cr$_3$C$_2$ particles rebound during coating formation [24]. Fig. 2(b) shows the chemical composition of the substrate in which two elements were detected by XRD. They are Iron-Coalt (FeCo) and Iron-Nickel (FeNi), with the maximum peak belonging to FeCo.

3.2. Wear behavior

For any selected coating by thermal spray method, substrate roughness has a strong effect on coating wear under given frictional conditions [25]. The carbon steel (substrate) roughness is $R_m = 1.9102 \, \mu m$ for 100 points as detected by the optical surface texture analyzer software. Meanwhile, the coated sample roughness is $6.4166 \, \mu m$. As reported in previous studies, coating roughness plays a critical role in the lifespan of samples. Consequently, the wear resistance of a sample with smooth surface is expected to decrease [25].

A perusal of literature related to wear resistance (erosion, abrasion and sliding wear) of a variety of materials, including coatings, clearly indicates that in the majority of cases, hardness has been utilized as the primary parameter for correlating wear resistance [26]. This has led to further investigation into the hardness of the deposited layer with the specific parameter mentioned in section 2. The critical hardness values of the carbon steel substrate appear to be in the range of 220–260 Hv, while the average microhardness of the coated sample is 890–930 Hv. As reported by Scrivani et al. [27], the hardness of Cr$_3$C$_2$–NiCr coating using HVOF with propane fuel consistently has 950HV hardness. Chatha and colleagues [28] reported that exact high volumes of carbides are well dispersed in the matrix. This factor might be responsible for higher microhardness and lower porosity values found in this type of coating.

In this study, the regions of wear and coarse, worn debris from the uncoated and coated samples at the maximum load (20 N) of wear testing were investigated using SEM. As shown in Fig. 3 the uncoated sample is evidently worn. Wide and deep grooves were created on the surface after wear testing. Fig. 3(a) shows that after applying a load at the edge of the sample, a significant crack that leads to deformation was

**Fig. 3.** SEM photos for uncoated sample at the maximum load (20 N). (a) SEM-800x. (b) SEM-500x. (c) SEM-7000x.
created. Obviously, the wear tracks appeared on the entire substrate surface. These tracks are the effect of maximum load applied by the pin-on-disc during the wear test. After wear testing, wide grooves also appeared beside the cracks on the surface. As Fig. 3(b) and (c) illustrate, although the worn surface of the uncoated sample is smooth and flattened, its wear debris is coarse. This observation shows that the load was extremely high for this substrate, and as a result, the carbon steel surface lost a considerable amount of material. The sample surface turned out to be so rough owing to this wear debris. In contrast, when maximum load was applied in the coated sample illustrated by Fig. 4, no obvious difference could be observed before the wear test. Moreover, no cracks or deformation was created at the edge of the sample, after maximum load had been applied. Applying maximum load resulted in slight abrasion and worn surfaces in some sample regions, which were however smooth, as they looked before testing (Fig. 4(b)). As shown in Fig. 4(c), the coated samples had smaller and rougher wear debris in comparison with the uncoated samples, which indicates that there was considerably more coating wear in the uncoated samples. In addition, within the tracks at maximum load (20 N) a little loose wear debris was found. On the surface of the coated sample which had been exposed to wear testing, no cracks were detected. This was associated with the elevated bonding strength between the splats and component as well as the significant role that bond strength can play in splat propagation and resistance to crack initiation.

Table 2 presents the mean scores of weight losses as well as the wear rate of both coated and uncoated samples under varying loads. Based on the results, it was observed that under different loads the abrasive wear resistance of the coatings changed in significantly due to the effect of the coating density on the volume of wears loss [25]. In contrast, the wear rate in the substrate changed significantly under different loads. Under the 5 N load, the coated and uncoated samples indicated minimum weight losses of 0.00332 and 1.8949 mg respectively (Table 2). Maximum weight losses of 8.2035 and 0.0098 mg were recorded for coated and uncoated samples respectively under the 20 N load (Table 2). As it was found, changing the load from 15 N to 20 N increased the weight loss two-fold for carbon steel, from 4.0926 to 8.2035 mg, while there was insignificant change in the coated sample. Also the

<table>
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<tr>
<th>Loads(N)</th>
<th>Rate of weight loss (mg)</th>
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<tbody>
<tr>
<td></td>
<td>Non-coated sample</td>
</tr>
<tr>
<td>5</td>
<td>1.8949</td>
</tr>
<tr>
<td>10</td>
<td>2.3121</td>
</tr>
<tr>
<td>15</td>
<td>4.0926</td>
</tr>
<tr>
<td>20</td>
<td>8.2035</td>
</tr>
</tbody>
</table>

Fig. 4. SEM photos for coated sample at the maximum load (20 N). (a) SEM-4000x. (b)SEM-500x. (c) SEM-7000x.
rates of wear confirm these trends. It is observed that when different loads were applied on the coated and uncoated samples, the amount of wear for the coated sample changed between 10 and 25 μm, while for the uncoated sample when load was increased the wear rate significantly increased. The rate of wear on the uncoated sample rapidly increased as a result of friction. The maximum values of wear for the uncoated sample were 125, 200, 250 and 657 μm for loads of 5, 10, 15 and 20 N respectively.

Generally for Cr₃C₂–NiCr coating, NiCr alloy is a continuous matrix phase with chromium carbides as hard reinforcement phases. Because the microhardness of chromium carbides is much higher than that of the NiCr matrix, the carbides are more resistant to cutting or gouging than the matrix alloy phase. Accordingly, the carbide phase, having higher wear resistance, would be removed at a lower rate, and the wearing off of NiCr alloy binder would occur more preferentially. Consequently, the abrasive wear of the HVOF Cr₃C₂–NiCr coating in the pin-on-disc test occurred in two steps. The first step was the successive removal of the NiCr binder. Because the metallic Ni–Cr binder phase is deformed by the stress of compression produced by the sliding WC–6%Co pin, it caused the carbide particles to be exposed to the surface. Then the removal of carbide particles occurred by fracturing or loosen- ing followed by subsequent pulling out by abrasive particles. Therefore, an improvement in the matrix alloy hardness and bonding strength between carbides and the matrix may enhance the abrasive wear resistance of the coating. Also, other comparative studies between thermally sprayed coatings and hard chromium electroplating give an idea that the abrasive wear resistance of HVOF coatings could be 2–16 times more resistant than hard chromium coatings [8,26]. Moreover, the wear rate is very sensitive to abrasive characteristics such as chemical composition, shape, size and angularity.

The particle shape can have significant influence on the wear mechanisms and will determine if the particles will be able to roll or to slide during three-body abrasion. Particles with an angular shape can generate wear rates higher than the rounded ones [21,27]. Since finer rounded particles produce less damage on both surfaces, finer carbide size coatings would show a lower wear rate [28]. Furthermore, the lower size of chromium carbide could involve a better distribution and cohesion of the reinforced particles in the binder phase that could decrease the pullout of the carbide particles during the sliding test and consequently produce a lower specific wear rate [29].

3.3. Microstructure analysis of corrosion

The X-ray diffraction (XRD) analysis result in Fig. 5(a) shows that the uncoated sample surface has corroded and

![Fig. 5. XRD and SEM micrograph of uncoated sample. (a) XRD analysis after corrosion. (b) SEM before corrosion. (c) SEM after corrosion.](image-url)
oxidized to Fe₂O₃ because the reaction with NaCl solution and dissolved oxygen has caused the oxidation of the uncoated sample surface. The SEM micrographs of the uncoated sample surface before and after 27 days immersion time in the corrosive electrolyte are shown in Figs. 5(b) and (c) respectively. There is a significant difference between the corroded and un-corroded surfaces. Fig. 5(c) shows two different regions of the corroded sample at high magnification. It can be seen that vigorous corrosion occurred after 27 days, which destroyed the surface. The formation of iron oxide occurred on different areas of the samples and was confirmed by the XRD analysis result.

The X-ray diffraction (XRD) analysis for the coated sample is illustrated in Fig. 6(a). It shows that the deposited layer has oxidized to Cr₃O on the surface of the coating. However, the dense coating protects the substrate from the inward permeation of oxygen. There is no formation of iron oxide from the XRD results of the coated samples. Thus, it can be concluded that the thin coating prevented the corrosive electrolyte from penetrating the substrate, and consequently protected the substrate from the corrosive environment. Furthermore, SEM micrographs of the coated samples before and after 27 days immersion time are shown in Fig. 6(b) and (c) respectively. There is no big difference between the corroded and un-corroded samples. It is proven that the coating protected the substrate from corrosion. Therefore, the chemical composition of the coating has anti-corrosion properties in this condition.

3.4. Corrosion analysis

Polarization curves were made for uncoated and coated samples from day 1 to day 27, and the results are tabulated in Table 3. It can be seen that the corrosion current I_corr for the coated sample is smaller than for the uncoated sample nearly

<table>
<thead>
<tr>
<th>Day</th>
<th>Non-coated sample</th>
<th>Coated sample</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>OCP</td>
<td>I_corr / A</td>
</tr>
<tr>
<td>1</td>
<td>-0.676</td>
<td>7.503 x 10⁻⁵</td>
</tr>
<tr>
<td>3</td>
<td>-0.734</td>
<td>3.976 x 10⁻⁵</td>
</tr>
<tr>
<td>5</td>
<td>-0.680</td>
<td>7.474 x 10⁻⁵</td>
</tr>
<tr>
<td>7</td>
<td>-0.711</td>
<td>4.839 x 10⁻⁵</td>
</tr>
<tr>
<td>9</td>
<td>-0.711</td>
<td>4.749 x 10⁻⁵</td>
</tr>
<tr>
<td>11</td>
<td>-0.719</td>
<td>3.891 x 10⁻⁵</td>
</tr>
<tr>
<td>13</td>
<td>-0.712</td>
<td>2.792 x 10⁻⁵</td>
</tr>
<tr>
<td>15</td>
<td>-0.736</td>
<td>2.949 x 10⁻⁵</td>
</tr>
<tr>
<td>17</td>
<td>-0.735</td>
<td>2.292 x 10⁻⁵</td>
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<td>19</td>
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<td>5.899 x 10⁻⁵</td>
</tr>
<tr>
<td>21</td>
<td>-0.739</td>
<td>2.763 x 10⁻⁵</td>
</tr>
<tr>
<td>23</td>
<td>-0.736</td>
<td>3.109 x 10⁻⁵</td>
</tr>
<tr>
<td>25</td>
<td>-0.713</td>
<td>6.059 x 10⁻⁵</td>
</tr>
<tr>
<td>27</td>
<td>-0.728</td>
<td>5.081 x 10⁻⁵</td>
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</tbody>
</table>

Fig. 6. XRD and SEM micrograph of coated sample. (a) XRD analysis after corrosion. (b) SEM before corrosion (c) SEM after corrosion.
for all measurements (Table 3). The open circuit potential (OCP) of the coated sample moves toward noble potential at the end of day 27, and the OCP values for the coated sample are more noble compared with the uncoated sample (Fig. 7) for nearly all measurements (Table 3). These results suggest that the HVOF coating on carbon steel offers good protection of the carbon steel substrate against corrosion.

Electrochemical Impedance Spectroscopy (EIS) was also performed on the carbon steel substrates. Fig. 8 shows the EIS results for the uncoated sample for day 1 and 27 measurements while the results for the coated sample measurements for day 1 and day 27 are shown in Fig. 9. For all measurements, a circuit diagram of $R_s$ ($Q R_{ct}$) accurately fits the experimental results, and this could be seen in the Bode phase plots with a single maximum in Fig. 8(b) and (d), as well as Fig. 9(b) and (d). The circuit diagram can be explained as follows. The $R_s$ is the solution resistance between the steel surface and reference electrode, and it is in series with the parallel arrangement of the constant phase element (CPE) and the charge transfer resistance $R_{ct}$. The CPE is often approximated to capacitance [30,31], where the parallel arrangement of the capacitance and the charge transfer resistance gives rise to a time constant (CR) which appears as a peak in the Bode phase plot [32]. Therefore, the charge transfer resistance $R_{ct}$ is the parameter, which gives information about the resistance against electron transfer across the uncoated sample/solution interface [33]. Table 3 shows that the $R_{ct}$ value for the coated sample is greater than the uncoated sample for nearly all measurements. These results also confirm the enhanced performance of the HVOF coating compared with bare carbon steel in terms of corrosion protection of the carbon steel surface.

Fig. 7. Example of polarization curves (log I vs E) for uncoated and coated samples. (a) One day experiment. (b) 27 days experiments.

Fig. 8. EIS for uncoated sample. (a) Nyquist plot day 1. (b) Bode plot day 1. (c) Nyquist plot day 27. (d) Bode plot day 27.
4. Conclusions

A thin layer of Cr$_3$C$_2$-20NiCr deposited by HVOF on the surface of carbon steel can significantly change the properties of carbon steel in corrosion and tribological performance. The surface of this layer is very dense, causing the coated sample to have higher corrosion resistance than carbon steel during testing for 27 days. The carbon steel corroded and iron oxides were present on the surface. Both polarization and EIS results confirm the corrosion protection properties of the HVOF coating. In addition, the hardness and tribological properties of the coated sample at different loads applied are more durable and the rate of weight loss in this sample is very limited, especially at loads of 5 N and 10 N.

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