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PII: S0272-8842(14)00889-X
DOI: http://dx.doi.org/10.1016/j.ceramint.2014.06.017
Reference: CERI8702

To appear in: Ceramics International

Received date: 28 April 2014
Revised date: 25 May 2014
Accepted date: 2 June 2014

Cite this article as: Mitra Akhtari Zavareh, Ahmed Aly Diaa Mohammed Sarhan, Bushroa Binti Abd Razak, Wan Jeffrey Basirun, Plasma thermal spray of ceramic oxide coating on carbon steel with enhanced wear and corrosion resistance for oil and gas applications, Ceramics International, http://dx.doi.org/10.1016/j.ceramint.2014.06.017

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Plasma thermal spray of ceramic oxide coating on carbon steel with enhanced wear and corrosion resistance for oil and gas applications

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Abstract

In this work, carbon steel surface was coated with Al₂O₃-40wt% TiO₂ composite using thermal spraying method. The tribological properties of the plasma-sprayed Al₂O₃-40 wt% TiO₂ coating were investigated with a tribometer (pin-on-disc) to evaluate and compare the wear properties of coated and uncoated samples under different loads. Additionally, cumulative weight analysis was done to compare wear loss. The results indicate significant anti-wear improvement with an increase in TiO₂ from 13 to 40 wt%. The corrosion on coated and uncoated samples was analyzed using AC and DC methods, namely open circuit potential from potentiodynamic polarization and electrochemical impedance spectroscopy in 3% NaCl solution for 20 days.

Keywords: Al₂O₃-40 wt% TiO₂; Plasma deposition; Wear; Corrosion
1. Introduction

Offshore oil and gas production environments represent aggressive conditions in terms of erosion and corrosion. Consequently, material selection requires meticulous attention at every stage of design, construction as well as system operation and equipment including piping systems and their accessories, such as bends, elbows, tees, and valves [1, 2]. Also included are devices that impart movement to fluid, such as pumps, impellers, propellers, and blowers, which are exposed to, or transport particle-laden fluids such as seawater [3].

Chemical damage is additionally caused by the surface conditions and inherent nature of the bare metal and corrosive fluid. Thus, the specification and use of materials that combine erosion and corrosion resistance with high mechanical strength is a fundamental requirement in the petroleum industry and is the focus in numerous research works [3, 4].

In the petroleum industry, carbon steel is the most commonly used material in piping, both upstream and downstream. As such, carbon steel is an important material due its good mechanical performance and relatively low cost compared to higher alloy materials [4]. But pure carbon steel suffers from poor corrosion resistance, so it is imperative to protect its surface in such aggressive environments.

Oxide ceramics such as alumina, zirconia, titania, chromia, silica and yttria are used extensively as surface coating materials to improve wear, erosion, cavitation, fretting and corrosion resistance [5, 6]. They are especially useful in applications where wear and corrosion resistance are required simultaneously [6]. Ceramics made from Al₂O₃–TiO₂ composite powders are prospectively attractive. Aluminum titanate ceramic composite has
excellent abrasion, heat and corrosion resistance properties. It also reportedly has high
fracture toughness and a low frictional coefficient [7, 8]. It can sustain up to 540°C
(1000°F) and is suitable for heat resistant materials. Combinations of wear and corrosion
resistance with high mechanical strength contribute to minimizing failure factors that would
eventually prevent total shutdown, economic loss and even more importantly, ensure
offshore safety. Besides, hazardous effects are also minimized. Thus, corrosion prevention,
monitoring and testing can save offshore industries billions of dollars [9-11].

For these reasons, applying protection methods to material surfaces is very important. The
methods’ effectiveness depends on the careful selection and regular service monitoring of
corrosion and wear. Metallic and non-metallic coatings along with cladding or surface
deposition are some of the most common corrosion and wear prevention techniques [12,
13]. Thermal spraying is the more widespread and appealing method of depositing different
types of materials and is widely applied in the aerospace, agriculture, automotive, oil and
gas as well as chemical industries. Flame, electric arc, plasma, detonation gun and high
velocity oxygen fuel are the main thermal spraying techniques for coating purposes [13,
14].

The plasma spray process essentially entails spraying a molten or heat-softened material on
a surface to provide a coating. Material in the form of powder is injected into a very high
temperature plasma flame, where it is rapidly heated and accelerated to a high velocity. The
hot material impacts the substrate surface and rapidly cools to form a coating. The plasma
spray process, if carried out correctly, is called a "cold process" (relative to the substrate
material being coated) as the substrate temperature can be kept low during processing to
avoid damage, metallurgical changes and distortion of the substrate material. Inert gases can be used very accurately as working media to reduce the oxidation of sprayed particles besides ensure high bond strength and low porosity of the coatings by controlling the process parameters [15-17]. High quality plasma-sprayed coating can be achieved due to the high melting point of the ceramic oxide material and also the advantage of plasma-sprayed coating compared to the other coating methods. For this reason, the deposition of Al₂O₃-40 wt% TiO₂ on mechanical part surfaces by plasma spray technique is an effective way of functionally improving the surface [17, 18]. In the present work, the wear and corrosion of Al₂O₃–40 wt% TiO₂ on coated and uncoated carbon steel surfaces in aggressive environments are analyzed.

2. Materials and method

In this study, carbon steel (Fe-Ni-Co) was used as a coating substrate. Prior to deposition, steel specimens of 10 x 10 x 4 mm were sandblasted (next section). Then ceramic oxide powder (Al₂O₃-40 wt% TiO₂) was deposited for an average of 7 passes. The particle size of the powder was between 45-60 µm and the average thickness of the coating was roughly 128 µm. The temperature and spray distance were kept constant throughout the deposition process.
2.2. Surface preparation and plasma coating

Al$_2$O$_3$-40 wt% TiO$_2$ composite powder coating was deposited on carbon steel coupons with a Metco 9MB plasma gun. The coupons were sandblasted to remove rust and their surface was cleaned prior to thermal spray deposition. Surface preparation is a very important step in thermal spraying because coating adhesion is directly related to surface roughness and is controlled by the type of grit blasting machine, blasting pressure, angle distance, time and grit blasting nozzle [19]. Grit blasting was carried out with a highly efficient sand blaster with Alumina grit size of 10-20 mesh, a sand blaster with a 10 mm nozzle, operating at a blasting pressure of >0.5 MPa. The distance between the substrate and nozzle was 150 mm at a 30° angle. The duration of the grit blasting process depends on the required surface roughness. Upon completing grit blasting, deposition was performed using a Metco 9MB plasma gun. The parameters for plasma spraying the ceramic coatings were adjusted to control the material chemical reactions, and also to control phase stability at high temperatures. The parameters adopted in the spraying process are shown in Table 1.

2.3 Microstructural analysis

The coated surfaces and cross section morphologies were evaluated using a scanning electron microscope (SEM) at high magnifications. The coating cross sections were polished prior to SEM analysis. Atomic force microscopy (AFM) experiments were carried out using an Autoprobe CP atomic force microscope. The measurements were performed on dry membrane samples under ambient atmospheric conditions. Silicon cantilevers with
integrated pyramidal tips (Model No: MPP-11100, VEECO Instruments Inc., Fremont, CA) were employed for membrane surface topography imaging. Also, the coating sample phase composition was examined by X-ray diffraction (XRD) with nickel-filtered Cu Kα (λ: 1.54056) radiation on a RIGAKU D/Max 2200 diffractometer with 2θ, 0-90°.

Finally, the coated samples’ surface roughness (Ra) was measured with an optical surface texture analyzer (Alicona 3D Infinity Focus). A total Ra measurement of 100 points was performed on the coated samples.

2.4 Wear and hardness tests

The coatings’ hardness was measured with HMV-Shimadzu under a 300 g load exerted for 15 s on the coating cross section. A total of 10 indentations were made on a coated sample and microhardness was measured in air at room temperature. Wear tests were performed in a pin-on-disc tester model TR-20LE for coated and uncoated specimens under four normal loads of 5 N, 10 N, 15 N and 20 N. The specimens were ground at 200 rpm disc speed with 120 grit SiC sandpaper in dry condition. The track diameter of D=40 mm and sliding speed of v=1 m/s were kept constant throughout the measurements. Wear testing was done for a total sliding distance of 2010.88 mm (20 min). Weight loss for the pins was measured after each load application to determine the amount of wear loss. After wear testing, the samples were ultrasonically cleaned in ethanol and dried; then their weight was measured with a microbalance to an accuracy of 0.0001 gm. The wear rate and coefficient of friction were
determined from the friction force and normal loads in all cases. The wear tracks produced in the coating were studied by SEM. The results of coating volume loss were reported.

### 2.5 Electrochemical corrosion test

Electrochemical techniques such as DC (polarization) and AC (impedance) were performed using an AutoLab PGSTAT30 potentiostat/galvanostat from Ecochemie, the Netherlands. Polarization experiments were done using a three-electrode cell: the steel samples were the working electrodes (WE), while a platinum wire and saturated calomel electrode (SCE) were the counter and reference electrodes, respectively. With 3% NaCl electrolyte and linear scan voltammetry, the corrosion current ($I_{corr}$) and corrosion potential ($E_{corr}$) were calculated from the Tafel plots. Electrochemical impedance spectroscopy (EIS) was performed at a frequency of $10^2 - 10^2$ Hz and amplitude potential of $V_{rms} = 5$ mV around the open circuit potential (OCP). Simulations with analog circuits were performed on the experimental data to obtain the charge transfer resistance across the electrode-electrolyte interface, and these were correlated with parameters from polarization experiments. A frequency response analyzer (FRA) and general purpose electrochemical software (GPES) were installed in a computer interfaced with a potentiostat, and the DC and AC were run, respectively.
3. Results and discussion

3.1 Microstructure analysis

As seen in Fig. 1, the polished and un-etched cross section of the coating contains plate-like lamella and is parallel to the substrate. Lamella formation indicates that the molten droplets hit the substrate at high velocity to create splat morphology [20]. The SEM micrographs confirm that Al₂O₃-40 wt% TiO₂ coating powder was successfully deposited onto the steel substrate by plasma spraying. In these images, the grey areas are attributed to Al₂O₃, while the light stringers between the lamellae are attributed to Titania (TiO₂). Pores appear black in the micrograph. Clearly, porosity in this coating is less than one percent (<1%); therefore the dense coating protects the substrate from inward permeating oxygen and the corrosive environment [21].

Fig. 2a shows the micrograph of a uniform, homogeneous coating that is free from surface cracks. Applying homogenous coating with no segregation is critical for improving the wear resistance of Al₂O₃-TiO₂ coatings. As per Fig. 2b, some regions partially melted because the powder temperature is not sufficiently high during spraying [27, 28], but other regions melted completely and the grain boundary increased. The melted regions are attributed to the arrest and deflection of crack propagation through the partially melted regions [22, 23].

An XRD of Al₂O₃-40 wt% TiO₂ and uncoated samples is presented in Fig. 3, while Fig. 3a indicates that the major phase is γ-Al₂O₃ and the minor phases are α-Al₂O₃ and rutile-TiO₂. The subsequent, complete melting resulted in a smaller number of pores, a reduced volume fraction of partially melted regions formed inside the coatings, as well as less α-Al₂O₃ phases and more γ-Al₂O₃ phases [22, 24].
As reported by McPherson, $\gamma$-$\text{Al}_2\text{O}_3$ experienced homogeneous nucleation because the critical free energy of nucleation from liquid was less than that of $\alpha$-$\text{Al}_2\text{O}_3$. Also, the temperature during plasma coating was higher than 610$^\circ$C, so the small peak in the XRD is attributed to rutile-$\text{TiO}_2$ [25]. In Fig. 3b two main peaks are detected, with the major phase being FeCo and the minor phase FeNi.

### 3.2 Wear Behavior

Uncoated sample roughness strongly influences coating wear under given frictional conditions [26]. Figs. 4a and b demonstrate that the roughness of uncoated and coated carbon steel surfaces is 7.4166 µm and 1.5668 µm respectively, for 100 points in the sample. Yussef et al. reported a roughness value of 3.70 when coating with 13% TiO$_2$ microparticles and no bonding layer with plasma spraying technique [20]. Additionally, increasing TiO$_2$ from 13 wt% to 40 wt% resulted in a smaller friction coefficient, while coating lifetime can be extended with smaller roughness values [26]. In addition, a lower as-sprayed surface roughness is very important for technological applications because it reduces the number of post-deposition mechanical treatments necessary [27, 28]. The amount of wear increases due to the increased friction rate; therefore soft coating has a lower friction coefficient [26].

The pure Al$_2$O$_3$ coating obtained with plasma spraying has a microhardness value of around 1000-1100 HV [29]. Meanwhile, commercial Metco 130 powder, which consists of 13 wt% TiO$_2$ in Al$_2$O$_3$, has lower microhardness (880 HV) [30]. The lower microhardness could be attributed to a combined effect of greater porosity, microstructural differences and the
presence of a softer TiO₂ phase upon the disappearance of the harder α-Al₂O₃ phase. Therefore, when TiO₂ is increased to 40 wt%, hardness decreases to 790 HV. Such reduction produces better coating properties with less porosity, because this amount of TiO₂ significantly reduces the melting temperature of the oxide rather than pure Al₂O₃ [28]. Microhardness generally tends to decrease with increasing TiO₂ percentage owing to the softer nature of the TiO₂ material (TiO₂: 50~55 HRC versus Al₂O₃: 60~70 HRC) [32]. This result is consistent with other similar studies [29, 31].

Finally, the wear mechanism should be investigated to evaluate the wear properties of Al₂O₃-TiO₂ coatings. Wang et al. indicated that the coating’s wear resistance is not only related to the hardness level, but also toughness, microstructure, and defects [33]. They suggested that the primary cause of wear in coatings sprayed with conventional powder is grain dislodgment, which is due to grain boundary fracture combined with lateral crack chipping. Ann et al. stated that serious delamination under wear condition occurs along TiO₂-rich zones in conventional coating [22]. Song et al. compared the wear mechanism of fully melted regions with partially melted areas. It was concluded that the wear mechanism involves crack initiation and propagation, which lead to eventual delamination, spallation and coarse, worn debris in coatings with fully melted regions. Delamination is not serious for partially melted regions where the wear debris is slighter [34].

In this study, the regions of wear and coarse, worn debris in uncoated and coated samples at maximum wear testing load (20N) were investigated by SEM and AFM, as shown in Figs. 5 and 6 respectively. Fig. 5a indicates that the uncoated sample is evidently worn. Obviously, a wear track is present from the top left to the bottom right of the image. This
track is caused by the maximum load on the equipment pin exerted against the sample’s surface. Several wide grooves on the surface are attributed to the wear test. For a more detailed investigation, AFM imaging of the sample’s surface after the sliding wear test can be done. A wear groove from Fig 5a appears in the corner of Fig. 5b. The AFM image (scale of 10 µm) shows a deep, wide groove in regions where this load is applied, which causes the removal of huge amounts of material from the sample surface. As shown in Fig. 5c, upon applying maximum load (20 N) wear debris spreads over the sample surface. This wear debris is responsible for the surface having changed from smooth to rough.

Fig. 6a illustrates the presence of semi bright particles on the coated samples’ worn surface after applying maximum load. Additionally, it appears that abrasion is minimal. Fig. 6b illustrates the AFM image of a coated sample after the wear test. Horizontal micro tracks were created on the surface and abrasion was very slight. Jun Wang et al. reported that a very thin coating surface layer was plastically deformed and had a weak bond to the base coating, and this layer was prone to removal by friction. The formation and removal of the thin plastic layer behaves as a wear strain fatigue mechanism. In Fig. 6c, slight loose wear debris is found within the tracks at maximum load (20 N), and the wear surfaces are relatively smooth.

In Fig. 5d, the uncoated sample’s wear debris is coarse and its surface is smooth and flat. Fig. 6d of the coated sample at the same magnification indicates smaller and rougher wear debris than the uncoated sample. This signifies that compared with the uncoated sample, the amount of wear on the coated sample is considerably less.
Fig. 7 illustrates the cumulative weight loss for coated and uncoated samples. The weight loss rate for each sample after a 20 min cycle with loads of 5, 10, 15 and 20 N are compared. From the figure it is clear that the weight lost from the coated samples is negligible compared to the uncoated samples. Furthermore, the software wear result presented in Fig. 8 confirms this trend. Fig. 9 shows the amount of wear illustrated in Fig. 8. When different loads were applied on both sample types, the coated samples had between 25-28 µm wear; however, the wear rate significantly increased for the uncoated samples as load increased. The greater rate of wear on the uncoated samples is due to the rapid increase in friction. The maximum wear for the uncoated samples was 125, 200, 250 and 657 µm for loads of 5, 10, 15 and 20 N respectively. Fig. 8 indicates that the wear rates settled for all loads except 20 N, and the uncoated sample wear rate continuously increased even at the end of 20 min. Also, the wear rate for 40 wt% TiO₂ at 5 N was significantly less than 0.1, or around 0.0040 (Cm³/N.m). Yussof et al. reported that the wear rate for 13 wt% TiO₂ in the same experimental conditions was around 0.0055 (Cm³/N.m). Thus, the wear rate for 40 wt% TiO₂ at 5 N load was significantly less. In this research, however, when the load was increased to 10, 15 and 20 N, and time and sliding distance were increased as well, wear was still less than for 13 wt% TiO₂ as found by Yussof et al. [20, 35].

### 3.3 Corrosion measurements

X-ray diffraction (XRD) results (Fig. 10) demonstrate that oxidation occurred on the coating surface to produce AlTiO₅. The uncoated sample surface also corroded to Fe₂O₃.
due to the presence of oxygen in the NaCl solution. However, the XRD results show the absence of iron oxide in the coated samples, thus the dense coating protected the plasma-sprayed steel substrate from the inward oxygen permeation.

The SEM micrographs of an uncoated sample before and after 20 days of immersion in NaCl are shown in Fig. 11. Fig. 11b indicates a significant difference between the immersed (corroded) samples’ surface and those before immersion (un-corroded). Sample corrosion obviously occurred during immersion in electrolyte after 20 days, and the surface was destroyed. According to XRD analysis, the corrosion products contained iron oxide.

The SEM micrographs for samples before and after 20 days of immersion (Fig. 12) show that both surfaces are similar. It can be concluded that the plasma-coated samples are resistant to corrosion. However, the electrolyte penetration through the coating defects can apparently strongly affect the electrochemical behavior of thermal spray coatings. In this case, the electrolyte did not penetrate into the substrate nor did it produce iron oxide according to the XRD results [11, 23 and 24].

The open circuit potential (OCP) of both coated and uncoated samples shows that after an initial drop to negative potentials, the OCP moved towards noble regions for the final 8 days, and the OCP of the coated sample was more positive compared to uncoated sample. While \( I_{\text{corr}} \) showed fluctuations for both samples, the charge transfer resistances \( R_{\text{ct}} \) were consistent with the OCP values from polarization measurements. The higher \( R_{\text{ct}} \) (EIS data) is due to the greater resistance to charge transfer across the electrode-electrolyte interface, which is consistent with the positive OCP result values. From the computer simulations, the equivalent circuit \( R_s(Q_1R_1)(Q_2R_2) \) accurately fits the experimental data of plasma-treated
steel with Al₂O₃-40 wt% TiO₂ as the top layer. The resistance between WE and RE is the solution resistance Rₛ, which is in series with two time constants (parallel arrangement of R and Q) that are also in series. The two QR in series correspond to two different surfaces with Al₂O₃-40 wt% TiO₂ as the top layer on the steel substrate. In these simulations, it is assumed that the Al₂O₃-40 wt% TiO₂ coating has higher charge transfer resistances Rᶜₜ compared to the uncoated sample owing to the higher resistance of the Al₂O₃-40 wt% TiO₂ material against corrosion. For uncoated sample, the Rₛ(QR) circuit diagram accurately matches the experimental data. With the absence of coating on pure carbon steel surface, only one semicircle was observed in the Nyquist plot (Fig. 13a). The Bode phase diagram of an uncoated sample (Fig. 13c) shows one-phase maxima, consistent with the presence of one semicircle in the Nyquist plot from Fig. 13a with a Rₛ(Q₁R₁) circuit. The Bode phase diagram of plasma steel shows two-phase maxima in lower and higher frequency regions (Fig. 13d), which is due to the presence of two semicircles in the Nyquist plot (Fig. 13b). The smaller semicircle in the higher frequency region of the Nyquist plot from Fig. 13b is caused by the corrosion of uncoated sample. The charge transfer resistance Rᶜₜ (R and R₂ for carbon and plasma, respectively) values are tabulated in Table 2.

4. Conclusions

The Al₂O₃-40 wt% TiO₂ chemical composite resulting from the plasma spray technique produced a coating with superior mechanical and chemical properties. According to the XRD results, the 40 wt% TiO₂ contained fully and partially melted regions of γ-Al₂O₃. The
hardness and wear test results signify that splat delamination and wear rate were negligible for the coated samples. Finally, the ceramic oxide had higher charge transfer resistance $R_{ct}$ compared to the uncoated sample, due to the higher resistance of $\text{Al}_2\text{O}_3$-40 wt% $\text{TiO}_2$ to corrosion compared to pure carbon steel.

Acknowledgement

"The authors would like to acknowledge the University of Malaya for providing the necessary facilities and resources for this research. This research was funded by the high impact research (HIR) grant number “HIR-MOHE-16001-00-D000001” from the Ministry of Higher Education, Malaysia and UMRG research grant number “RP005B-13AET” from University of Malaya, Malaysia.

References


Table 1. The parameters of Plasma coating

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>primary Ar gas</td>
<td>690 kPa</td>
</tr>
<tr>
<td>secondary H₂ gas</td>
<td>380 kPa</td>
</tr>
<tr>
<td>Powder feed rate</td>
<td>50-60 g/min</td>
</tr>
<tr>
<td>Argon flow rate</td>
<td>110 m³/hour</td>
</tr>
<tr>
<td>Carrier gas flow rate</td>
<td>40-60 m³/hour</td>
</tr>
<tr>
<td>Barrel length</td>
<td>160mm</td>
</tr>
<tr>
<td>Current</td>
<td>550A</td>
</tr>
<tr>
<td>Voltage</td>
<td>60V</td>
</tr>
<tr>
<td>Spray distance</td>
<td>150mm</td>
</tr>
</tbody>
</table>

Table 2. OCP, $I_{corr}$ and $R_{CT}$/kOhm of coated and uncoated samples in 3%NaCl solution for 20 days

<table>
<thead>
<tr>
<th>Day</th>
<th>Carbon Steel</th>
<th>Plasma (Al₂O₃-40TiO₂)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>OCP</td>
<td>$I_{corr}$</td>
</tr>
<tr>
<td>-----</td>
<td>-----</td>
<td>---------</td>
</tr>
<tr>
<td>2</td>
<td>-0.74</td>
<td>$5.167 \times 10^{-12}$</td>
</tr>
<tr>
<td>4</td>
<td>-0.477</td>
<td>$6.211 \times 10^{-16}$</td>
</tr>
<tr>
<td>6</td>
<td>-0.851</td>
<td>$1.115 \times 10^{-8}$</td>
</tr>
<tr>
<td>8</td>
<td>-0.773</td>
<td>$5.804 \times 10^{-27}$</td>
</tr>
<tr>
<td>10</td>
<td>-0.762</td>
<td>$1.047 \times 10^{-22}$</td>
</tr>
<tr>
<td>12</td>
<td>-0.776</td>
<td>$9.962 \times 10^{-73}$</td>
</tr>
<tr>
<td>14</td>
<td>-0.887</td>
<td>$4.081 \times 10^{-39}$</td>
</tr>
<tr>
<td>16</td>
<td>-0.770</td>
<td>$5.068 \times 10^{-24}$</td>
</tr>
<tr>
<td>18</td>
<td>-0.768</td>
<td>$2.809 \times 10^{-26}$</td>
</tr>
<tr>
<td>20</td>
<td>-0.762</td>
<td>$5.323 \times 10^{-21}$</td>
</tr>
</tbody>
</table>
Fig. 1. Cross section of Plasma sprayed Al₂O₃-40wt%TiO₂

Fig. 2. SEM of coating samples in different magnification (a) 300X, (b) 1200X
Fig. 3. X-ray diffraction patterns of the (a) coated and (b) uncoated samples
Fig. 4. represent the schematic roughness of (a) uncoated and (b) coated samples.
Fig. 5. SEM and AFM micrograph of wear debris of the uncoated samples at the maximum load (20 N)
Fig. 6. SEM and AFM micrograph of wear debris of the coated samples at the maximum load (20 N)
Fig. 7. Dry wear rate of uncoated and coated samples for different loads.
Fig. 8. The amount of micrometers wear at different loads: (a) 5N, (b) 10N, (c) 15N and (d) 20 N for coated and uncoated samples.
Fig. 9. The amount of micrometers wear at different loads: (a) 5N, (b) 10N, (c) 15N and (d) 20 N for coated and uncoated samples.
Fig. 10. XRD result of a) coated and b) uncoated samples in NaCl solution after 20 days
Fig. 11. SEM micrographs of the uncoated sample (a) before and (b) after corrosion testing.

Fig. 12. SEM micrograph from surface of coated sample (a) before and (b) after corrosion testing.
Fig. 13. Nyquist plots for (a) uncoated and (b) coated samples; Bode plots for (c) uncoated and (d) coated samples