A study of the electrochemical and tribological properties of TiN/CrN nanolayer coating deposited on carburized-H13 hot-work steel by Arc-PVD technique

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ABSTRACT
The purpose of this study is to investigate the tribological and corrosion behavior of TiN/CrN nanostructured multilayer coating on H13 carburized hot-work tool steel. Pack cementation and cathodic arc physical vapor deposition (Arc-PVD) techniques were employed to apply diffused layer and hard coating, respectively. FE-SEM and XRD were used for coating characterization. A pin-on-disc wear test and Rockwell Cindenter were employed to study wear behavior and adhesion of the coating. To evaluate the corrosion behavior of the specimens, the specimens were tested by potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) after immersion in 3.5% sodium chloride solution for 48 hours. The results of potentiodynamic polarization and EIS curves showed that the TiN/CrN nano-layer coating with pre-carburized treatment demonstrated a better corrosion behavior than H13 and carburized samples. According to the electrochemical test, the current density of the substrate, carburized, and pre-carburized TiN/CrN coated samples were 0.107, 0.875, 0.487 μA/cm², respectively. This improvement in corrosion resistance is due to the high density of the TiN/CrN nano-layers that coating prevents the diffusion of the corrosion solution into the substrate.

1. Introduction
Heat treatment is the process of changing mechanical and metallurgical properties of metallic alloys through controlled heating and cooling steps. Hardness of a heat-treated ferrous article is strongly influenced by the amount of carbon imposed on the surface during the rapid cooling of it [1]. In general, carbon is the most important element in commercial alloyed steels, which increases hardness and strength of steels. It also stabilizes austenite. Addition of carbon to steel affects its mechanical properties. Meanwhile, it may increase the risk of brittleness and reduce weldability [2].

Cathodic arc evaporation method is considered as a popular coating technique due to the unique structural features it gives to the coating [3–6]. Applying a coating by this technique results in a uniform and dense structure, excellent adhesion, proper control of the morphology of the coating during coating deposition, ability to use materials with high melting points, and the possibility of layer deposition at various rates from one nanometers per minute to several micrometers per hour [7].

Physical vapor deposition (PVD) of TiN/CrN multilayer coatings has been emerging as an attractive surface protection and modification technology. Research works have shown that multilayer coatings demonstrate high toughness, good adhesion to the substrate, high abrasion resistance, low friction coefficient, and good resistance to oxidation and corrosion compared to single-layer coatings. These multilayer coatings are deposited on the surfaces of cutting and forming tools due to their superior characteristics [2,7–11]. Nitride-based hard coatings are created through various processes of PVD and high hardness and excellent wear and corrosion resistance of these coatings enable them to greatly improve the service life of the tools [12–14].

Recent advances in thin-layer deposition technologies have led to a new generation of self-lubricant coatings [15]. In addition to high hardness, these new multilayer coatings can withstand high oxidizing environments (TiN/CrN coatings), improve toughness (TiN/NbN coatings), provide low friction coefficients (TiAlN/VN coatings), enhance corrosion protection (CrN/ZrN coatings), and improve thermal stability (TiN/NbN and TiN/AlN coatings), which all are helpful in wear-resistant applications [16–21]. Hardness has a great influence on tribological performance of the coatings and increasing hardness leads to a decrease in their wear rate [22].

Combining PVD coatings with a pre- or post-treatment can further boost the properties. A duplex treatment composed of plasma nitriding and subsequent TiN coating deposited by PVD is effective in increasing the abrasive wear resistance of uncoated...
AISI H13 steel substrates [23]. The CAPVD TiAlN/oxynitriding duplex treated on AISI H13 alloy steel can improve corrosion resistance, erosion resistance and hardness [24]. TiN/TiC multilayer coatings fabricated by plasma-assisted chemical vapor deposition on AISI H13 hot-work tool steel can increase hardness, toughness, wear resistance and decrease friction coefficient [25]. Most studies on duplex treatment have focused on combining nitride coating with nitriding process and the information about other type of duplex treatment is limited. The focus of the current work is on using carburization as the pre-treatment for PVD nitride coating. The aim of this paper is to study the corrosion and tribological behavior of carburized hot-worked H13 tool steel coated with nanoscale multilayer TiN/CrN coating.

2. Experimental details

2.1. Substrate preparation

The results of optical emission spectroscopy elemental analysis of the substrate are summarized in Table 1. The results of the analysis show that the substrate steel complies with the German standard DIN: X40CrMoV5-1 (1.2344) and with the English standard BH13. The samples were cut into discs with a diameter of 33 mm.

| Table 1. Results of the optical emission spectroscopy for the substrate. |
|---------------------------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|
| Element | C | Si | S | P | Mn | Ni | Cr | Mo | V | Ti | Cu | W | Fe |
| Weight percent | 0.380 | 1.20 | 0.015 | 0.020 | 0.388 | 0.276 | 4.97 | 1.17 | 1.17 | 0.877 | 0.002 | 0.015 | rem |

2.2. Carburization process

To achieve an appropriate surface condition before doing the carburization process, the samples were ground with 60 to 800 sandpapers. To perform the carburization, the pack cementation method was used. The parts were placed in a cast-iron box with a weight ratio of 70% charcoal and 30% cast iron chips, and then the box was completely sealed with clay. Then, the cementing box was heated for 3 hours at 1050°C and after that the samples were quenched in water and heated again at 550°C for 1 hour for tempering. To achieve a proper surface quality, after the carburization process, the specimens were ground by 600 to 2000 sandpapers and polished by 5-microns diameter alumina powders.

2.3. PVD-coating process

The coating was applied on the carburized samples using a cathodic arc evaporation PVD machine (DS & CA601, Yar-Nikan, Iran). The applied nano-coating was composed of titanium nitride and chromium nitride (TiN/CrN) layers. Table 2 shows the operating parameters of the PVD machine used during the coating process.

| Table 2. Operating parameters of the cathodic arc evaporation process. |
|-----------------------------|-------------|
| Parameter | Value |
| Vacuum pressure before coating | $5 \times 10^{-3}$ torr |
| Vacuum pressure during coating | $5 \times 10^{-3}$ torr |
| Distance between target and substrate | 15 cm |
| Cathode arc current | 120 A |
| Substrate temperature | 200°C |
| Substrate bias voltage | $-150$ V |
| Coating time | 90 min |

2.4. Characterization of specimens

The thicknesses of both PVD layer and carburized layer were measured using a JEOL JSM-840A scanning electron microscope (SEM). The sequential layers of TiN and CrN were observed with the help of a MIRA3 TESCAN field emission scanning electron microscope (FESEM). A Rockwell-C hardness machine was used to evaluate adhesion of the coating to the substrate at a load of 150 Kgf for a duration of 25 seconds and then the surface of the samples was judged according to the VDI 3198 standard with the help of an optical microscope [26]. A Unisantis XMD300 XRD system with the Cu-K$_\alpha$ radiation source and 0.02 step was also used to examine the phases formed in the layers.

2.5. Evaluation of surface properties

To measure the hardness of the specimens, micro-hardness Vickers test was performed with a force of 25 g. To eliminate the influence from of the substrate hardness on the results, Jonsson-Hogmark method [27] was applied. Reciprocating pin-on-disk machine was used to test wear resistance of the samples. The testing parameters were normal load of 2 N and speed of 0.1 m/s for a total distance of 500 m. The pin was 52,100 bearing tool steel and test temperature and humidity were 33°C and 50%. An optical microscope was used to examine the worn paths.

Electrochemical measurements were performed using a galvanostat/potentiostat (Autolab Type III/FRA2) device. Prior to the electrochemical tests, all samples were immersed for 48 hours in 3.5 wt% sodium chloride solution under open circuit potential. An ordinary three-electrode flat cell was used to test the specimens, in which the studied specimens were used as working electrodes, silver/silver chloride as reference electrodes and platinum electrodes as auxiliary electrodes. The experimental spectra of EIS were
modeled using Nova software. Potentiodynamic polarization tests were performed at a rate of 1 mm/s and EIS tests were performed in the frequency range of 100 kHz to 10 mHz with a wavelength range of 10 mV (in open circuit potential).

3. Results and Discussion

3.1. Microstructure and composition of the coating

The first feature to be evaluated was adhesion quality of the coating, which was examined based on the VDI 3198 standard [26]. According to Figure 1, the adhesion of the coating to the carburized substrate is excellent (HF1 level). The adhesion quality is important since corrosion and wear behavior of the coating strongly depends on its adhesion to the substrate.

![Figure 1](image1.png)

**Figure 1.** Optical microscope image of the effect of Rockwell-C on the coated sample for evaluation of adhesion.

As shown in Figure 2, the peaks obtained from the X-ray diffraction (XRD) patterns are on planes (111), (200), and (311) which represent TiN (PDF# 96-101-0975) and CrN (PDF# 96-101-0975) structures. Also, Cr$_3$C$_2$ (PDF# 96-900-9907) and Fe$_2$C (PDF# 96-901-2188) phases are detected in the XRD pattern. The peaks for the substrate are also visible, which is due to the very thin nature (less than 5 μm) of the coating layer.

![Figure 2](image2.png)

**Figure 2.** XRD pattern of the TiN/CrN coated sample.
### 3.2. Tribological behavior of the coating

#### 3.2.1. Hardness test

The hardness values of the specimens are given in Table 3. For the hardness evaluation, the micro-hardness Vickers test was performed at a load of 25 g, which makes it possible to subtract the effect of the substrate on the coating by means of the Jonsson–Hogmark method [27,32]. In this method, the value of hardness of the thin-hard coating is calculated as the following relation (Equation (1)):

\[
H_F = H_S + \frac{H_C - H_S}{2C(\frac{t}{d}) + C^2(\frac{t}{d})^2}
\]

where \(H_S\), \(H_F\), and \(H_C\) are the hardness of the substrate, the hard-coating, and the composite of coating-substrate obtained from micro-hardness Vickers, respectively. Also, \(t\) has indexed the thickness of the coating, and \(d\) value is the average diameter of the Vickers indent effect. In the equation, \(C\) is the indenter constant which is considered as 0.14 [27]. Using this equation, the hardness of the pre-carburized coated sample was calculated as 2136 HV.

![Figure 3. SEM images of the cross-sectional area of the pre-carburized coated sample in two different magnifications.](image)

![Figure 4. FE-SEM image of the cross-sectional area of the TiN/CrN nanoscale multi-layer coating.](image)
Figure 6 shows the hardness profile measured from the surface of the pre-carburized coated sample to the depth of 110 μm from the surface. As shown in Figure 6, there is no sharp decrease in hardness due to the use of a pre-carburization treatment. The pre-carburization treatment creates a gradient of hardness from higher hardness values down to the lower hardness of the substrate. The value of the hardness gradually decreases from the surface to the inside. The depth of penetration of carbon in the H13 steel is measured as about 75 μm. This distance is consistent with the measured thickness of the carburized layer which leads us to conclude that the increase in the hardness is the result of the carburization process. According to the results of XRD (Figure 2), the carburization forms Fe₃C and Cr₃C₂ phases on the surface of the sample which in turn, increases the hardness of the surface.

### 3.2.2. Wear test

The friction coefficient graphs for the substrate, the carburized sample, and the pre-carburized TiN/CrN coated sample are presented in Figure 7. As shown, the substrate has the highest mean value of the friction coefficient.
coefficient and the TiN/CrN coated sample demonstrates the lowest mean value of friction coefficient. The wear rate (W) of the specimens were calculated using the following equation [13]:

\[ W = \frac{V}{F \times S} \]  

(2)

where \( V \) is the wear volume loss (mm\(^3\)), \( F \) is the normal load applied (N), and \( S \) is the sliding distance (m). The result of the tests is presented in Figure 8. According to the results, the wear rate of the H13 and the carburized samples are \( 5.8 \times 10^{-4} \text{ mm}^3/\text{N.m} \) and \( 4.1 \times 10^{-4} \text{ mm}^3/\text{N.m} \), respectively. The wear rate of the pre-carburized TiN/CrN coated sample is \( 1.2 \times 10^{-4} \text{ mm}^3/\text{N.m} \) which is more than four times less than that of the substrate.

The wear rate and coefficient of friction results are consistent with the hardness measurements from the samples. The TiN/CrN coating has a very high hardness due to the presence of hard TiN and CrN layers which make it very resistant to steel pins and causes considerable abrasion on them [33]. Optical microscopy images and micrographs of the sliding wear tracks on the surfaces of the samples are presented in Figure 9. For the substrate steel, signs of both adhesive and abrasive wear are observed in the worn area. The substrate is softer than the sliding pin. In addition, the pin and the substrate, both are iron-based alloys and compatible with each other. Therefore, it is expected to detect adhesion and abrasion behavior between the

Figure 7. Friction coefficient as a function of the sliding distance for the specimens.

Figure 8. Comparison of wear rate of the specimens after 500 meters of sliding.
surfaces of the pin and the substrate sample. The adhesion between the surfaces could explain the higher coefficient of the friction measured for the H13 substrate steel.

In the case of the carburized sample, it is harder than the substrate due to the existence of a large amount of Fe₃C and Cr₃C₂ on the surface of the carburized sample. This makes the sample more resistant against both adhesive and abrasive sliding wear. In Figure 9(b), no significant signs of plowing are observed at the worn area and the material loss could be due to the adhesion between both surfaces. This is consistent with the coefficient measurement results. According to Figure 7, the average value of the coefficient of the friction for the carburized sample is smaller than the one for the substrate which could be attributed to the lower adhesion tendency between the surface of the pin and the carburized sample and also to the harder nature of the carburized surface.

The wear rate for the sample coated with TiN/CrN coating is the least between all three samples (Figure 8) and the worn surface of the sample (Figure 9(c)) is not clear well. In fact, the width of the track is very small, and from both optical and micrograph images, we can deduce that the track is very shallow. The average friction coefficient for the coated sample is the smallest between the samples. All these confirm the superiority of the TiN/CrN coated sample in resistance against wear. The coating creates a very hard layer on the sample, about 7 times harder than the substrate steel and about 2.5 times harder than the carburized surface. In addition, as discussed above, the coating layer makes a perfect bonding with the substrate underneath. Moreover, the nature of the TiN and CrN is different from the steel pin which makes the role of adhesion wear minor. These reasons provide the coated sample with an excellent resistance against sliding motion wear. Other researchers have attributed the improvement in the properties of CrN/TiN coatings

![Figure 9](image_url). Optical and scanning electron microscope images from the wear tracks (a) H13 sample (b) carburized sample (c) pre-carburized coating.
3.3. Investigating the corrosion behavior of the coating

3.3.1. Potentiodynamic polarization test

The potentiodynamic polarization curve of the samples after 48 hours of immersion in 3.5 wt% solution of sodium chloride under the open circuit potential, is presented in Figure 10. The linear parts of the plots in both the anodic and cathodic branches near the corrosion potential, express the behavior of the anode and cathode Tafel [34–36]. Corrosion current density (based on the linear Tafel extrapolation method using cathode branch [35,37–40]) and corrosion potential of the samples have been reported in Table 3. According to Table 3, the carburized sample and the pre-carburized TiN/CrN coated samples have lower corrosion current densities compared to the substrate. According to the results of the XRD pattern reported in Figure 2, the low corrosion current density of the carburized sample compared to the substrate can be attributed to the presence of Fe3C which increases the density of the surface atoms and increases the compressive stress on the surface. In addition, according to the corrosion current density values reported in Table 4, the sample coated with TiN/CrN nano-layers showed the lowest corrosion current density and the highest corrosion potential. The lower corrosion current density of the pre-carburized TiN/CrN coating indicates a better corrosion resistance [41].

The excellent corrosion resistance of TiN/CrN coating applied to stainless steel 304 L [8,42] and 316 L [43] in 3.5 wt% of sodium chloride solution and mixture of sulfuric acid and hydrochloric acid solution has been reported by other researchers. The nobler corrosion potential and lower corrosion current density of the pre-carburized TiN/CrN coating sample compared to the carburized sample can be attributed to the presence of titanium and chromium elements in the nano-layer coating. These elements form a layer with a high protective role and, as a result, better electrochemical behavior [41].

3.3.2. EIS test

Figure 11 shows the EIS curves of the samples studied in a 3.5 wt% solution of sodium chloride (under open circuit potential after 48 hours). From Nyquist (Figure 11(a)) and Bode-phase plots (Figure 11(b)), it can be seen that there are two time constant (or constant phase element (CPE)) for TiN/CrN coating, while one time constant for H13 and carburized samples. For the experimental EIS data to be reliable, the electrochemical system should conform to three requirements (stability, linearity and causality) formulated for the constraints of the linear system theory (LST). Thus, the criterion to accredit to the experimental EIS data is the Kramers-Kronig (K–K) transformation. K–K transforms have been applied to EIS results by transforming the imaginary axis into the real axis and the real axis into the imaginary axis [44–48]. Figure 12 indicates good similarity between corresponding K–K transforms and the experimental points, representing compliance with LST.

According to Figure 11, the equivalent electric circuits shown in Figure 13 were used to simulate experimental data. In the electrical circuit shown in Figure 13 (a), Rs is the solution resistance, R1 load transfer resistance and the fixed phase element CPE1 is related to

![Figure 10. Potentiodynamic polarization curves for the substrate, carburized sample, and TiN/CrN coating in the 3.5 wt% of chloride sodium solution.](image-url)
the double electric layer. Also, in Figure 13(b), Rs is the solution strength, $R_2$ TiN/CrN coating resistance, $R_1$ load transfer resistance, the fixed phase element CPE$_2$ to TiN/CrN coating and the fixed phase element CPE$_1$ is related to the double electric layer [49–52].

Modeling the results of the EIS test related to the substrate, the carburized sample, and the sample with TiN/CrN coating shown in Figure 13 was performed. It is clear that the polarization resistance ($R_p$) regarding to the TiN/CrN coating sample is obtained from the total coating resistance and load transfer resistance, and the polarization resistance for the substrate and the carburized sample is equivalent to the load transfer

| Sample          | $E_{corr}$ (mV) | $i_{corr}$ ($\mu$A cm$^{-2}$) |
|-----------------|-----------------|--------------------------------|
| Substrate       | −638            | 1.070                          |
| Carbonized      | −656            | 0.875                          |
| TiN/CrN         | −571            | 0.487                          |

Table 4. Amount of the corrosion current density and the corrosion potentials of the substrate sample, carburized sample, and TiN/CrN coating sample.

Figure 11. (a) Nyquist and (b) Bode/phase curves for the substrate, carburized sample, and TiN/CrN coated sample after 48 hours of immersion in the 3.5 wt% solution of chloride sodium.
resistance associated with them. The impedance of the fixed phase element \((Z_{CPE})\) defined as follows (Equation (3)) [53]:

\[
Z_{CPE} = \frac{1}{Q \, (j\omega)^n}
\]  

(3)

The constant \(Q\) is related to the constant phase element with the unit \(\Omega^{-1}.S^{-n}.\text{cm}^{-2}\), \(\omega\) is the angular frequency with the unit rad.s\(^{-1}\), \(n\) is the uniformity coefficient of the surface and \(j^2 = -1\) is the imaginary number. The parameters \(Q\) and \(n\) are independent of frequency. In general, \(1 \geq n \geq 0\). Depending on the value of \(n\), the constant phase element can show resistance behavior \((n = 0)\), capacitive \((n = 1)\) and/or impedance Warburg \((n = 0.5)\) [54].

Results from the simulation of impedance tests (Figure 11) are summarized in Table 5. According to the values of the obtained elements, in 3.5 wt% solution of sodium chloride, the polarization resistance of
the sample with composite TiN/CrN coating is more than the substrate and the carburized sample. In addition, the reviewed substrate showed the lowest amount of polarization resistance. The information reported in Table 5 is in complete compliance with the results of potentiodynamic polarization tests.

4. Conclusion

(1) The results obtained from the friction coefficient and the wear rate test showed that the friction coefficient of the substrate, carburized and pre-carburized TiN/CrN coated samples are 0.6, 0.5, and 0.3, respectively. Also, their wear rates are \(5.8 \times 10^{-4}\), \(4.1 \times 10^{-4}\), and \(1.238 \times 10^{-4}\) mm²/Nm, respectively.

(2) Smaller values of friction coefficient and wear rate indicate superior wear resistance of TiN/CrN coated sample. The lower coefficient of friction can be attributed to the high hardness and lack of metal bonding between the abrasive pin and the sample. Also, according to the wear track images, the width and depth of wear path for the TiN/CrN coated sample is very small.

(3) The results of the potentiodynamic polarization and EIS curves showed that the current density of the substrate, carburized, and pre-carburized coated samples are 1.070, 0.875, and 0.487 microamperes per square centimeter (µA/cm²), respectively, that less current density indicates better corrosion resistance. The polarization resistances of the above samples are obtained as 1.677, 1.954, and 3.875 kΩ.cm², and the pre-carburized coated sample has the highest corrosion resistance.

(4) Also, the corrosion potentials of these samples are –638, –656, and –571 mV, respectively, and the nobler potential for less corrosion and lower corrosion current density can be attributed to the formation of passive layers containing chromium and titanium on the surface of the sample with coating. Improved corrosion resistance can be caused by the high density of the layers in the TiN/CrN nano-layer coating, which will prevent the corrosive solution from penetrating toward the substrate.

Disclosure statement

No potential conflict of interest was reported by the authors.

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