1. Introduction

The exclusive chemical and physical properties like high hardness, excellent sliding wear, abrasive resistance, excellent corrosion, and oxidation resistance, as well as high-temperature stability, made transition metal nitride coatings as the refractory compounds have uses in molds, dies, automotive, aerospace, forming and cutting tools, electric and magnetic, superconducting devices and many other applications [1–4]. Coatings based on zirconium, titanium, and chromium nitrides have been used as protective coatings against corrosion and wear because they have lower friction coefficient, high chemical stability, and high melting point [5–9]. The CrN coatings were found that could show superior wear, corrosion properties, better adhesion on steel, and even better oxidation resistance compared with TiN coatings. Also, ZrN coating has comparable excellent corrosion, wear resistance, and mechanical properties in comparison to TiN coatings [10].

Some studies have shown that the multilayer coatings have excellent mechanical, tribological, and corrosion properties such as very high hardness, corrosion stability and wear resistance [6,11] compared to most commonly used binary (like CrN, TiN, HbN, and ZrN) [5,12] and ternary single layer coatings (like TiCN, TiAlN, and VCN). The enhancement of these properties is ascribed to the nanoscale structure of the multilayers coatings that was gained by deposited alternate layer of two different materials, thickness in nanometric size, and many interfaces [9,10,13,14]. In some protective coating applications, the ZrN/CrN multilayer has been used because they have the best adhesion and mechanical properties, radiation stability, hardness, excellent tribological efficiency, oxidation resistance, and thermal stability [5,9,11,13]. Transition metal nitride coatings that were deposited by PVD, generally have a columnar microstructure and high defect density. Some researchers have reported that across the coating/electrolyte interface, concentration ions decrease by the straight columnar structure. But, the fine-equiaxed structure by zig-zag grain boundaries could result in a remarkable decrease in concentration of ions through the electrolyte/coating interface [15,16]. The defects of the coating surface and depth are non-uniformly distributed, while their size, form, and density depend on coating conditions. They are created either coating deposition process incorporation of microdroplets and small particles or substrate irregularities (pits, asperities), foreign particles (dust, debris, polishing residues) or by the [17]. Some parameters such as frequency, duty cycle, and bias voltage characteristics affect the formation of defects. For example, an increase in the amplitude or duty cycle resulted in a decrease of the roughness and defect density of the surface area covered by the defects.
These phenomena were explained by increasing the energy level of impinging ions and increasing the surface diffusion and mobility of adatom. An additional mechanism could be the higher electric field density on the conical features resulting in a higher resputtering rate [17]. The micropores-pinholes in columnar structure in coating thickness permits which corrosive agents may attack the substrate–coating interface. The micropores-pinholes present in these coatings affect the corrosion behavior; however, transition metal nitride coatings are chemically inert apart from other properties [18]. In order to improve corrosion resistance of the PVD coatings, it is necessary the porosity, and columnar growth was prevented. To modify the corrosion resistance, the multilayer deposition has been used for transition of metal nitride coatings. It is expected a layered deposition that minimizes the columnar structure of the PVD coatings. Renucleation in the multilayer coatings with sequential deposition of sublayers decreases the grain size and inhibits the growth of defects and pores during the coating [18].

One of the most essential progress in the PVD process is the CAE to deposit multilayered coatings [19–23]. The advantages of this method are high density and hardness, excellent adhesion, and homogeneity [24]. The characterization of the CrN/ZrN multilayer coatings, especially corrosion behavior, has not been enough studied yet [5–7,10,25,26]. The aim of the present work is to make a desirable corrosion-resistant coating on the AISI 304 substrate. Hence, the nano-multilayered CrN/ZrN coating was deposited on 304 substrates using the CAE-PVD method. After that, the quality and the present phases of the coating were evaluated by FE-SEM and XRD, respectively. Then, the electrochemical behavior of CrN/ZrN multilayer coating was assessed and was compared to the substrate using EIS and potentiodynamic polarization (PDP) measurements in 3.5% wt NaCl solution.

2. Experimental details

Nano-multilayer coating of CrN/ZrN was made by the CAE-PVD method. We used a high purity (99.96%) N₂ atmosphere pressure in the vacuum chamber and a vacuum-arc set including low-alloyed chromium and zirconium cathodes were used. The multilayer coating was deposited on the substrate of AISI 304. Sample size was about 50 × 50 × 2 mm. Then, the specimen was mechanically polished and degreased in an ethanol ultrasonic rinse before coating. Then, the sample was inserted in set and in order to increase the adhesion strength of coating, ionic bombardment was conducted by Ar⁺ ions at 800 V in 10 min. Table 1 shows the details of deposition conditions. The microstructural composition and coating chemical were detected by SEM JEOL 840 in 20 keV and FE-SEM. The cross and planar parts of the coating were analyzed. Grounding up to #1200 SiC for the embedded specimen in phenolic resin and by abrasive grinding papers was done to prepare the cross-section. The cross-sections were then polished. After cleaning samples by distilled water, they were dried in flow of warm air. In this study, the crystal structure and phase identification were used for XRD in a Panalytical X’Pert Pro MPD. The patterns collecting from 10° to 80° in θ–2θ Bragg–Brentano mode, Cu Kα radiation was done. The phase identification and quantification were specified by X’Pert High Score software. For all electrochemical measurements a three-electrode flat cell was used including prepared samples as a working electrode, auxiliary electrode (Pt), and a reference electrode (Ag/AgCl). A potentiostat/galvanostat autolab was used in Impedance tests in the frequency range of 100 kHz to 10 mHz in 3.5 wt. % NaCl solution with a wavelength of 10 mV at the intended potential [27]. Simulation of EIS experimental results for attaining the best equivalent electrical circuit (EEC) by Nova software was done. For evaluation adhesion, the VDI 3198 standard was carried on the ZrN/CrN nano-multilayers coating out by means of a Rockwell C indenter and using a force of 150 N for a loading time of 30 s [28].

3. Result and discussion

3.1. XRD

The XRD of nano-multilayered ZrN/CrN coatings has been presented in Figure 1. The deposited coating phases form a face-centered cubic (FCC) lattice of CrN, face-centered cubic (FCC) lattice of ZrN, and hexagonal lattice system of Cr₂N. The CrN/ZrN multilayer coatings tend to form randomly at different directions including (111), (200) planes of CrN phase and with (110), (111) plane of Cr₂N phase and (200), (311) planes of ZrN phase [12,25,29,30]. These planes of phases were detected in 29 equal 37°, 43° for CrN, 39°, 67° for ZrN and 37°, 42°, 74° for Cr₂N. Since there were different nitrogen pressures in the chamber, two-phase of CrN and Cr₂N formed at deposited coatings. Maksakova et al. reported that the Cr₂N was formed in the low pressure of nitrogen and CrN in higher pressure [29].

3.2. Microstructure

Figure 2(a,b) illustrates the FE-SEM images of cross-section of ZrN/CrN nano-multilayers. These figures display architecture of layers ZrN/CrN nano-multilayers in 75,000
and 135,000 magnifications, respectively. The FE-SEM result shows the coatings have a compact and dense morphology multilayer structure. Three regions of the coating were distinguishable; the first and closest layer to the substrate is Cr interlayer, while the second region is CrN or Cr$_2$N, and the third region is nano-multilayers, including ZrN/CrN. This configuration, except interlayer repeated many times until the deposition process was completed. The CrN layers are darker contrast and light ZrN layers in a layered architecture, respectively. Also, it seems ZrN layers are bigger than CrN layers. Some researchers attributed that to current of targets [25]. Growth shape of interlayer is related to the roughness of the substrate. In this figure, it seems that there is a sharp interface between layers. The composition of layers was revealed by EDS analysis that is shown in Figure 3. This figure disclosed the chemical composition of Zr, Cr, and N has fluctuation in multilayers coating. This fluctuation is due to the order of layers in a multilayer structure. Also, in the analysis of coating, Cr is higher than Zr and N elements. The Fe and Ni elements appear near substrate in spectra that is related to substrate composition.

Figure 4 shows EDX-elemental map of ZrN/CrN nano-multilayer on AISI 304, including Fe, Zr, Cr, and N elements. The Zr, Cr, and N are in the coating and Fe is at the substrate. This map confirms the distribution of elements in nano-multilayers image analysis in coating thickness. In this figure, it is clear that distribution of Nitrogen is almost homogenous throughout coating, but zirconium and chromium elements have non-homogenous distribution. As a result, it could be found that in some regions of coating, distribution of Zr and Cr is layer and layer. This unique nanostructure of ZrN/CrN was not reported in other literature.

3.3. Adhesion of ZrN/CrN nano-multilayers coating

In Figure 5, image of Rockwell C indentation is shown by using optical microscopy (according to the adhesion
standard VDI 3198 [28,31,32]). In this figure, delamination was not observed. This shows there was good adhesion of the ZrN/CrN nano-multilayers coating on the AISI304 substrate, and it is like to the HF1 class adhesion.

### 3.4. EIS measurements

EIS approach as a rapid, convenient, surface-sensitive, efficient, powerful, and nondestructive technique was used to compare the corrosion...
behavior of the nano-multilayered CrN/ZrN coating and the substrate in 3.5 wt. % NaCl solution as a function of immersion time [33–35]. The EIS spectra of the samples were recorded at OCP condition, and the results are illustrated in Figure 6. Paying attention to the Nyquist formats of the EIS spectra (Figure 6(a,c)), the typical capacitive behavior of both samples is observable. The effect of immersion time on the Nyquist curves of the samples is observed in the capacitive arcs or semi-circles, where they become more incomplete and larger. The augmentation of the diameters in the Nyquist format of the EIS spectra is ascribed to the better corrosion behavior of the samples [36]. It is worth mentioning that increasing immersion time from 24 to 168 h had a greater effect on the enhancement of the corrosion behavior of the substrate than that of the nano-multilayered CrN/ZrN coating. This is

Figure 5. Optical microscopy of rockwell C indentation of ZrN/CrN nano-multilayers coating.

Figure 6. (a, b) Nyquist and bode plot of AISI 304 substrate, and (c, d) Nyquist and bode plot of ZrN/CrN multilayer coating.
comprehensible by the comparison of the semi-circles in the Nyquist plots corresponding to the immersion time of 24 and 168 h.

Considering the Bode plots of both specimens (nano-multilayered coating and substrate) in Figure 6(b,d), the total impedance and phase angle values at high-frequency region (≥10,000 Hz) are an independent frequency, which discloses a perfect resistance behavior (R) [34,35]. Also, the presence of two constant phase elements (CPEs) at the middle-low frequencies of the Bode plots of the samples is distinguishable.

Figure 6(b) shows the Bode-modulus and Bode-phase diagrams for substrate 304 at 3.5% wt. % sodium chloride medium. In this figure, at frequencies above 10,000 Hz, the phase angle and the total impedance are independent of frequency, which indicates pure resistance behavior. Also, Figure 6(b) is in accordance with Figure 6(a), which increased by increasing impedance immersion time. In Figure 6(d), the diagrams of Bode modulus and bod phase diagrams for the nano-multilayered CrN/ZrN coating on the substrate 304 are shown, pure resistance behavior is observed at frequencies above 10,000 Hz. According to this figure, it can be said that the total impedance and phase angle did not change significantly at different immersion times, indicating that the 3.5 wt% NaCl corrosion environment did not cause much change in the total impedance and phase angle. Also, Figure 6(d) is in very good agreement with Figure 6(c) where it did not change as increasing impedance immersion time.

From Figure 6(b,d) it can be seen that at low and medium frequencies two constant phase elements (CPEs) are distinguishable.

The reliability of the acquired EIS data (Figure 6) can be proved by Kramers–Kronig (K-K) transforms which is performed by transforming the imaginary and real axes into the real and imaginary axes, respectively [37,38]. More details about K-K transforms including general and integral equations have been described elsewhere [39,40]. The comparison between the K-K transforms and the experimental EIS data for the substrate and the coating after being immersed in 3.5 wt. % NaCl solution are shown in Figure 7. As can be seen, consistency between the experimental data and the corresponding K-K transforms verifies the conformity of the studied system to the linear system theory [39,40].

Based on the EIS spectra (Figure 6), the appropriate EECs for the substrate and the CrN/ZrN coating were selected and depicted in Figure 8(a,b), respectively. According to Figure 8(a) and in the ECC of the substrate, $R_s$, $R_{pf}$, CPE$_{pf}$, $R_{ct}$, and CPE$_{dl}$ are assigned, respectively, to the solution resistance, passive film resistance, CPE of the passive film, charge transfer resistance, and double layer CPE. Also, $R_s$, $R_{por}$, CPE$_{por}$, $R_{ct}$, and CPE$_{dl}$ are related, respectively, to the solution resistance, pore resistance of the coating, CPE of the coating, charge transfer resistance, and double-layer CPE in the EEC of the nano-multilayered CrN/ZrN coating. The appearance of CPE in experimental EIS is due

![Figure 7](image-url) **Figure 7.** K-K transformations of the EIS plots: (a, b) AISI 304 without coating after 24 and 168 h, and (c, d) AISI 304 coated ZrN/CrN multilayers after 24 h and 168 h.
to surface heterogeneity, electrode porosity, distribution of surface reactivity, fractal geometry or roughness, and potential and current distributions is related to the geometry of the working electrodes (WEs) [41]. The impedance of CPE $(Z_{\text{CPE}})$ is expressed using Equation (1) [33–35]:

$$Z_{\text{CPE}} = \frac{1}{Q(j\omega)^n}$$

where $Q$ and $n$ as frequency-independent parameters are assigned, respectively, to the distribution of the dielectric relaxation times (in the frequency space for $0.5 < n < 1$) and the deviation from pure capacitance (C), $j$ is for the imaginary unit and $\omega$ indexes the angular frequency (rad s$^{-1}$).

The variation of the EEC elements of the substrate and the CrN/ZrN coating in 3.5 wt. % NaCl solution was summarized in Tables 2 and 3, respectively. As illustrated in Tables 2 and 3 and parallel to increasing the exposure time from 24 to 168 h, the polarization resistance value ($R_p = R_1 + R_2$) of both studied samples increases. Comparing the polarization resistance values of the nano-multilayered CrN/ZrN coating and the substrate, it is clear that the $R_p$ related to the coating is higher than that of the substrate which marked better corrosion behavior of the deposited coating at a certain immersion time [42]. Also, polarization resistance of the coating did not show remarkable variations by increasing immersion time. Chipatecua et al. [43] reported similar results, in the case of the multilayered CrN/ZrN coating deposited by unbalanced magnetron sputtering (UBM) procedure. These results can be attributed to the formation of a compact and dense structure of the multilayered coating resulting from the CAE procedure. In the other words, the multilayered interfaces of CrN/ZrN coating could decrease permeable defects (such as pores, pinholes, and

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### Table 2. Values of EEC elements corresponding to AISI 304 substrate as a function of immersion time in 3.5\% NaCl solution.

| Immersion time (h) | 24 | 48 | 72 | 96 | 120 | 144 | 168 |
|--------------------|----|----|----|----|-----|-----|-----|
| $R_s$ (\(\Omega\cdot\text{cm}^2\)) | 15.48 | 15.09 | 14.62 | 15.12 | 14.82 | 14.71 | 15.13 |
| $Q_1(x\times10^{-4}$ S$^n$/\(\Omega\cdot\text{cm}^2$) | 62.8 | 65.7 | 61.30 | 61.60 | 39.90 | 35.60 | 32.05 |
| $n_1$ | 0.89 | 0.92 | 0.92 | 0.92 | 0.929 | 0.93 | 0.94 |
| $R_1$ (k\(\Omega\cdot\text{cm}^2$) | 5.57 | 6.67 | 15.12 | 20.16 | 23.18 | 23.94 | 25.20 |
| $Q_2(x\times10^{-4}$ S$^n$/\(\Omega\cdot\text{cm}^2$) | 1.46 | 1.83 | 2.91 | 2.04 | 70.38 | 52.8 | 44.9 |
| $n_2$ | 0.69 | 0.62 | 0.72 | 0.66 | 0.63 | 0.69 | 0.72 |
| $R_2$ (M\(\Omega\cdot\text{cm}^2$) | 0.028 | 0.037 | 0.048 | 0.062 | 0.345 | 0.345 | 0.345 |

### Table 3. Values of EEC elements corresponding to ZrN/CrN multilayer nanostructured coating as a function of immersion time in 3.5\% NaCl solution.

| Immersion time (h) | 24 | 48 | 72 | 96 | 120 | 144 | 168 |
|--------------------|----|----|----|----|-----|-----|-----|
| $R_s$ (\(\Omega\cdot\text{cm}^2\)) | 16.08 | 15.60 | 15.35 | 15.36 | 15.35 | 15.19 | 15.48 |
| $Q_1(x\times10^{-4}$ S$^n$/\(\Omega\cdot\text{cm}^2$) | 0.50 | 0.59 | 0.59 | 0.58 | 0.39 | 0.39 | 0.60 |
| $n_1$ | 0.88 | 0.86 | 0.86 | 0.86 | 0.86 | 0.86 | 0.85 |
| $R_1$ (k\(\Omega\cdot\text{cm}^2$) | 11.99 | 31.8 | 37.8 | 40.03 | 50.04 | 63.04 | 101.50 |
| $Q_2(x\times10^{-4}$ S$^n$/\(\Omega\cdot\text{cm}^2$) | 0.18 | 0.10 | 0.12 | 0.03 | 0.10 | 0.10 | 0.10 |
| $n_2$ | 0.71 | 0.57 | 0.59 | 0.59 | 0.56 | 0.59 | 0.63 |
| $R_2$ (M\(\Omega\cdot\text{cm}^2$) | 1.31 | 3.23 | 5.55 | 7.56 | 20.1 | 50.4 | 75.6 |

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**Figure 8.** EECs for the simulation of the EIS plots: (a) AISI 304 and (b) ZrN/CrN multilayers coating samples.
cracks) by repeating deposition of CrN and ZrN layers, showing the diffusion of the corrosive electrolyte to reach the coating/substrate interface to enhance the corrosion behavior [43].

3.5. PDP assays

The PDP curves of the studied samples after being immersed for 168 h in 3.5 wt. % NaCl solution are displayed in Figure 9. The corrosion current densities ($i_{corr}$) and corrosion potentials ($E_{corr}$) of the samples extracted from PDP plots are summarized in Table 4. Compared to the uncoated sample (substrate), the multilayered CrN/ZrN coating displays lower corrosion current density and nobler corrosion potential, reflecting the better corrosion behavior of the coating. Similar results were reported by other researchers in the case of the corrosion behavior of the multilayered coatings deposited by PVD-based methods [18,44,45].

3.6. SEM observations

The SEM observations of the samples after PDP measurements showed different forms of defects for the substrate and the multilayered CrN/ZrN coating. The surface topography of the substrate is shown in Figure 10(a). As can be seen, the formation of pits as the vestiges of surface corrosion attack is clear. Surface topography of the coating after the PDP test indicated the characterization of uniform corrosion and small surface pinholes as represented in Figure 10(b). According to the obtained results, it can be concluded most of the defects are weakened as a result of repeated deposition of CrN and ZrN layers, which is responsible for the enhanced corrosion behavior of the coating.

4. Conclusion

Zirconium nitride/chromium nitride nano-multilayer coating with high adhesion and density was successfully created on AISI 304 stainless steel specimen by cathodic arc evaporation (CAE)-PVD technique. The results are summarized as following:

(a) The corrosion resistance of the nano-multilayer coating was better than AISI 304 substrate in 3.5 wt. % NaCl media. In addition, it is evident from PDP curves that $E_{corr}$ and $i_{corr}$ in zirconium nitride/chromium nitride nano-multilayer coating are lower than 304 stainless steel. This corrosion behavior was attributed to the distinct layer presence resulting in a barrier against penetration of the corrosive media.

(b) FE-SEM micrograph was indicated a unique nanostructure, including alternate ZrN and CrN

Table 4. $E_{corr}$ and $i_{corr}$ corresponding to AISI 304 and ZrN/CrN multilayer nanostructured coating specimens in 3.5% NaCl media.

| Material            | $E_{corr}$ | $i_{corr}$     |
|---------------------|------------|----------------|
| AISI 304            | -0.071     | 20.06 x 10^-7  |
| ZrN/CrN nano-multilayer | +0.111     | 1.63 x 10^-8   |

Figure 9. PDP curves of (a) AISI 304, and (b) ZrN/CrN nano-multilayers coating after 168 h immersion in 3.5% NaCl media.
that was in architecture of coating. Also, EDS revealed this composition in the coating.
(c) This unique nanostructure in ZrN/CrN nanomultilayer coating that has a lot of interfaces makes corrosion agent not to be able to penetrate in the substrate and high corrosion resistances in the type of coating are attributed to inertness of nitrides and interfaces.
(d) Considering SEM images, there were pits of corrosion in the AISI 304 stainless steel but there were not any severe damages or pits in the surface of the ZrN/CrN nano-multilayer coating.

Disclosure statement
No potential conflict of interest was reported by the authors.

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Figure 10. Planar SEM micrograph after corrosion: (a) AISI 304 substrate, (b) AISI 304 ZrN/CrN nano-multilayer coated sample.
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