

Effect of Sputtering Voltage on the Microstructure, Hardness, and Corrosion Resistance of Ni-Cr Coatings on Zirconium for Accident-Tolerant Fuel Applications

Agus Jamaludin^{1,2}, Wulan Tri Wahyuni², Syaiful Bakhri¹, Yusuf Gigih Wicaksono¹, Maman Kartaman Ajiriyanto¹, Usman Sudjadi¹, Suminar Setiati Achmadi^{2*}

¹Research Center for Nuclear Material and Radioactive Waste Technology, National Research and Innovation Agency, Tangerang Selatan, Banten, 15314, Indonesia

²Department of Chemistry, Bogor Agricultural University, Bogor, West Java, 16680, Indonesia

*Corresponding author: ssachmadi@apps.ipb.ac.id

Abstract

Zirconium alloys are commonly employed as fuel cladding materials in pressurized water reactors; nevertheless, their low corrosion and oxidation resistance under harsh operating circumstances remain a significant barrier for accident-tolerant fuel applications. This work investigates how sputtering voltage affects the microstructural evolution, as well as the mechanical and corrosion performance, of DC-sputtered Ni-20Cr coatings deposited on Zr-4 substrates. The coatings were formed at sputtering voltages of 4, 5, and 6 kV and evaluated by X-ray diffraction, scanning electron microscopy-energy dispersive spectroscopy, microhardness and nanoindentation tests, and potentiodynamic polarization in a simulated PWR primary coolant. The findings show that sputtering voltage has a significant impact on crystallite refinement, coating compactness, and the integrity of the coating-substrate interface, all of which influence the functional performance of coatings. Among the investigated deposition conditions, the coating deposited at 5 kV had the best combination of microstructural, mechanical, and corrosion properties, whereas increasing the sputtering voltage to 6 kV resulted in microstructural degradation, which reduced protective performance despite increasing thickness. These results show a clear relationship between sputtering voltage, microstructural evolution, and the mechanical and corrosion performance of Ni-Cr coatings. The study offers important guidance for optimizing sputtering parameters and illustrates the efficacy of DC-sputtered Ni-Cr coatings for accident-tolerant nuclear fuel cladding.

Keywords

Zirconium Alloy, Ni-Cr Coating; DC Sputtering, Hardness; Corrosion Resistance; Accident-Tolerant Fuel

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1. INTRODUCTION

Zirconium (Zr) alloys have been widely used as fuel cladding materials in pressurized water reactors (PWRs) due to their excellent corrosion resistance, mechanical strength, and low thermal neutron absorption cross-section (Kashkarov et al., 2021; Kautz et al., 2023). Nonetheless, their performance is considerably tested during a loss-of-coolant accident (LOCA), since high-temperature steam settings can promote rapid oxidation and cause severe cladding deterioration cladding (Brachet et al., 2025; Ma et al., 2021). In such cases, the exothermic reaction between Zr and steam (H₂O) accelerates the oxidation process and generates significant volumes of hydrogen, which poses a serious safety issue due to the possibility of hydrogen explosions (Amaya, 2024; Sidelev et al., 2022).

In response to these problems, significant efforts have been directed toward enhancing the accident tolerance of Zr-based fuel claddings. Several strategies have been investigated to

mitigate the effects of LOCA conditions, such as improving uranium fuel performance to improve fission product retention, investigating alternative cladding materials with superior high-temperature stability, and optimizing the performance of conventional Zr-based claddings to withstand severe accident environments (Zinkle et al., 2014). Among the many options, enhancing cladding performance through Zr surface modification has emerged as a very promising one. Surface modification using thin protective coatings is an efficient way to improve oxidation resistance while retaining the intrinsic features of the Zr substrate (Kashkarov et al., 2021).

Nickel-chromium (Ni-Cr) alloys have received a lot of interest as coating materials because of their outstanding high-temperature oxidation resistance, mechanical stability, and strong adherence to metallic substrates (Sidelev et al., 2019; Smola et al., 2019). Recent research has shown that magnetron-sputtered Cr, NiCr, and multilayer coatings are among the

most attractive alternatives for accident-tolerant fuel (ATF) claddings due to their superior oxidation resistance, hardness, and corrosion performance. However, these coating systems may still have limitations due to residual stress, coating brittleness, or interfacial degradation under extreme operating conditions, prompting the search for alternative coating materials with a more balanced combination of mechanical and corrosion properties (Han et al., 2024). Nickel is commonly utilized as a protective coating material due to its superior corrosion resistance, mechanical strength, and hardness (Gill et al., 2021; Rosyidan et al., 2024). Meanwhile, chromium (Cr) stimulates the creation of a dense and stable protective oxide layer, effectively limiting further oxidation and lowering hydrogen generation during accidents (Li et al., 2024a; Ma et al., 2021). Furthermore, Ni-Cr coatings have strong compatibility with zirconium-based substrates and can preserve structural integrity under combined thermal and mechanical loads (Sidelev et al., 2019). Ni-20Cr was chosen for this study because it provides the best balance of oxidation resistance and mechanical performance. The 20 wt.% Cr concentration promotes the creation of a continuous and protective Cr_2O_3 layer, effectively suppressing oxygen diffusion and increasing high-temperature oxidation resistance, while the Ni-rich matrix maintains acceptable ductility and toughness (Gill et al., 2021). Furthermore, Cr-induced solid-solution strengthening increases the hardness and wear resistance of the coating (Kumar et al., 2013). These properties are especially beneficial for ATF applications, where minimizing oxidation, lowering hydrogen generation, and maintaining cladding integrity under LOCA conditions are crucial requirements (Kashkarov et al., 2021). As a result, Ni-20Cr represents a promising alternative coating system that combines corrosion protection, oxidation resistance, and mechanical stability, while also allowing for further optimization of coating performance through proper deposition parameter controls.

Among many coating processes, DC magnetron sputtering provides fine control over layer thickness, uniform deposition, and strong interfacial adhesion (Cubillos et al., 2021; Ul-Hamid, 2021). It facilitates the creation of dense, homogeneous microstructures, which are essential for increasing oxidation resistance and reducing defect formation (Li et al., 2020). Furthermore, this approach provides for precise control over coating composition and microstructural evolution, which improves high-temperature stability and overall coating performance on zirconium-based fuel cladding (Han et al., 2024). Although prior research has shown that Cr^- and Ni-Cr-based coatings offer potential for ATF applications, most studies have focused on coating composition, deposition processes, and the consequent mechanical and corrosion properties (Sidelev et al., 2019). Fewer studies have looked systematically at how sputtering voltage influences the coupled evolution of coating microstructure, such as crystallite refinement, coating compactness, and interfacial diffusion, and how these microstructural features collectively govern the mechanical and electrochemical performance of Ni-Cr coatings on zirconium substrates

(Kashkarov et al., 2021). As a result, the effect of sputtering voltage on coating development mechanisms and the optimal deposition conditions within the examined voltage range is still insufficiently understood.

The purpose of this work was to investigate how different sputtering voltages (4, 5, and 6 kV) affected the mechanical characteristics and corrosion resistance of Ni-Cr coatings produced on Zr-4 substrates using DC sputtering. In a simulated reactor with H_3BO_3 and LiOH solutions, the impact of sputtering voltage on film hardness was assessed. Potentiodynamic polarization (PDP) was also used to analyze corrosion behavior. Furthermore, the correlations between deposition parameters, microstructural features, and coating performance were investigated to determine the optimal deposition condition. Although prior research has shown the potential of Cr^- and Ni-Cr-based coatings for ATF applications, it has primarily focused on coating composition, deposition techniques, or individual mechanical and corrosion properties (Han et al., 2024; Sidelev et al., 2019). The impact of sputtering voltage on the combined evolution of crystallite size, coating compactness, and interdiffusion layer formation, as well as their link to the mechanical and electrochemical performance of Ni-Cr coatings, has received very little study. To address this gap, the current study used integrated SEM, XRD, EDS, hardness, and electrochemical investigations to link sputtering voltage to the ensuing microstructural evolution, hardness, and corrosion behavior. The findings would advance our understanding of voltage-dependent coating development and offer practical advice for improving Ni-Cr coatings for ATF cladding applications.

2. EXPERIMENTAL SECTION

2.1 Materials

DC sputtering was performed on a Zircaloy-4 alloy (Zr-4) substrate with a chemical composition of 0.22 ± 0.06 wt.% Fe, 0.10 ± 0.05 wt.% Cr, and 1.35 ± 0.15 wt.% Sn, with the balance being Zr. The coating was applied using a Ni-Cr target containing 80 wt.% Ni and 20 wt.% Cr.

2.2 Methods

2.2.1 Ni-Cr Coating Deposition

Prior to deposition, Zr-4 substrates (11 mm in diameter and 3 mm in thickness) were mechanically ground with silicon carbide (SiC) abrasive sheets ranging in grit size from P500 to P2400, then mechanically polished to achieve a mirror-like surface finish. The samples were then washed with distilled water and ethanol to eliminate any surface impurities and ensure optimal coating adherence.

NiCr coatings were deposited on Zr-4 substrates via DC sputtering with a Ni-Cr target (Figure 1). Before deposition, the chamber was emptied using a rotary pump to a pressure of 2.7×10^{-2} mbar, followed by a diffusion pump to reach a base pressure of 2.7×10^{-3} mbar. The evacuation operation was continued for 1.5 hours to assure vacuum stability. The target-to-substrate distance was kept constant at 2.5 cm during

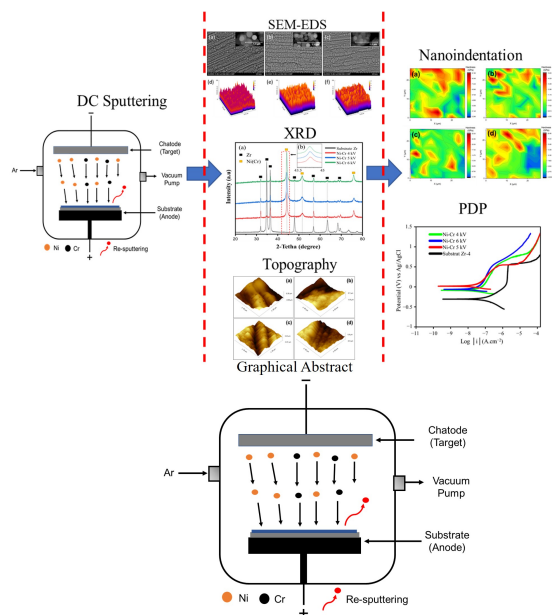


Figure 1. Schematic Illustration of DC Sputtering

the deposition process. The deposition was performed under a working pressure of 2.67×10^{-3} mbar. The sputtering medium consisted of 173 mL/min of ultra-high purity (UHP) argon (Ar) gas. The sputtering voltage was changed between 4, 5, and 6 kV to study its effect on coating qualities, with a constant current of 10 mA applied. Each deposition was carried out for 4 hours to ensure enough film thickness and equal coverage. Following deposition, the samples were chilled in the chamber for 30 minutes to reduce thermal shock and maintain coating integrity.

2.2.2 Sample Characterization

The surface morphology and cross-sectional properties of the Ni-Cr coatings were investigated using a JEOL scanning electron microscope (SEM) with energy-dispersive spectroscopy (EDS). X-ray diffraction (XRD) using Cu K α radiation was used to study phase composition, crystallographic structure, and lattice microstrain. Microhardness was determined using the Vickers indentation method. The measurement error was 0.44%, falling within the allowable tolerance level (<2%) set by ASTM E92-17 and E384-14. The hardness values were obtained using Equation 1 (Metzger et al., 2017). The corrosion behavior of the Zr-2 alloy was assessed at room temperature using potentiodynamic polarization (PDP) with a GAMRY Reference 600 system in an electrolyte comprising 4000 ppm H₃BO₃ and 2.5 ppm LiOH. Equation 2 was used to compute the corrosion rate, after determining the corrosion potential (E_{corr}) and corrosion current density (I_{corr}). All trials were carried out at the National Research and Innovation Agency (BRIN) in Indonesia.

$$HV = 1.854 \times 10^3 \times \frac{F}{d^2} \quad (1)$$

$$Corrosion\ rate = \frac{K \times (a \times I_{corr})}{n \times D} \quad (2)$$

The formulae used to assess the mechanical and corrosion properties of the coatings are provided below. Vickers hardness (HV) was calculated using Equation 2, where F (kgf) represents the applied stress and d (mm) is the average diagonal length of the indentation measured on the cross-sectional surface. The corrosion rate was calculated using Equation 2 (Basori et al., 2025), where K is the conversion constant ($3250 \text{ mm} \cdot \text{g} \cdot \text{A}^{-1} \cdot \text{cm}^{-1} \cdot \text{year}^{-1}$), EW (g/equivalent) is the equivalent weight of the material ($EW = a/n$), a (g/mol) is the atomic mass, n is the number of electrons involved in the electrochemical reaction, I_{corr} (A/cm²) is the corrosion current density obtained from potentiodynamic polarization measurements, and ρ (g/cm³) is the material density. The corrosion rate estimations for Zr-4 were based on its equivalent weight of 22.81 g/equivalent and density of 6.56 g/cm³.

3. Results and Discussion

3.1 X-ray Fluorescence (XRF)

The elemental compositions of the Zr-4 substrate and Ni-Cr coatings deposited at 4, 5, and 6 kV were determined using XRF, with the results reported in Figure 2 and Table 1. The presence of Ni and Cr in all coated specimens indicates the successful deposition of the Ni-Cr layer and shows a distinct change in surface composition when compared to the untreated substrate. The coating covered at 4 kV had $44.57 \pm 0.50\%$ Ni and $16.25 \pm 0.36\%$ Cr. At 5 kV, Ni and Cr concentrations decreased somewhat, to $40.94 \pm 0.50\%$ and $15.78 \pm 0.36\%$, respectively. Increasing the sputtering voltage to 6 kV resulted in increased concentrations of $46.54 \pm 0.50\%$ Ni and $16.46 \pm 0.38\%$ Cr. Zr levels in coated specimens ranged from $36.34 \pm 0.48\%$ to $42.58 \pm 0.50\%$. This finding suggests that the coating thickness was insufficient to completely obscure the contribution of the underlying substrate during XRF studies (Revenko et al., 2022).

The simultaneous detection of Ni, Cr, Zr, Fe, and Sn suggests that the detected XRF signal came from both the deposited Ni-Cr layer and the underlying Zr-4 substrate. This trend is explained by the penetration depth of X-rays during XRF analysis, which allows excitation of both the coating and the substrate when the coating thickness is relatively thin, resulting in a noticeable substrate contribution to the recorded spectra (Revenko et al., 2022).

3.2 Scanning Electron Microscope-Energy Dispersive Spectroscopy (SEM-EDS)

SEM micrographs (Figures 3a-3c) show the surface shape changes significantly as the sputtering voltage increases, and these findings are verified by the quantitative ImageJ analysis. At 4 kV (Figure 3a), the coating has a non-uniform shape and unevenly scattered granules. According to Borowski and Myśliwiec (2025), the less developed and diverse morphology could

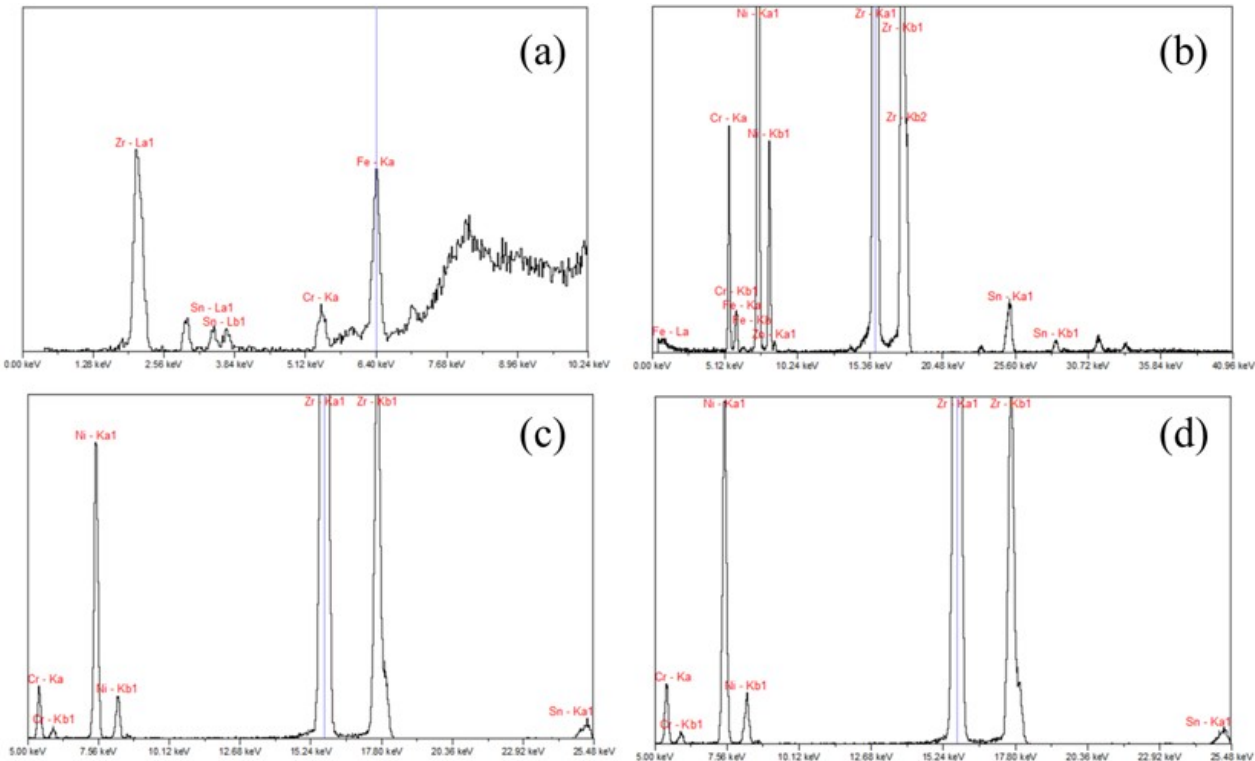


Figure 2. XRF Spectra of Ni–Cr Coatings Deposited on Zr Substrates at Different Sputtering Voltages: (A) Bare Zr Substrate, (B) 4 kV, (C) 5 kV, and (D) 6 kV

Table 1. XRF Elemental Concentration of the Zr-4 Substrate and Ni–Cr Coatings Deposited at Different Voltages

Elemental Composition	Elemental Concentration (wt.%)			
	Substrate Zr-4	Ni–Cr 4 kV	Ni–Cr 5 kV	Ni–Cr 6 kV
Fe	0.22 ± 0.06	0.15 ± 0.05	-	-
Cr	0.10 ± 0.05	16.25 ± 0.36	15.78 ± 0.36	16.46 ± 0.38
Ni	-	44.57 ± 0.50	40.94 ± 0.50	46.54 ± 0.50
Zr	98.33 ± 0.17	38.48 ± 0.48	42.58 ± 0.50	36.34 ± 0.48
Sn	1.35 ± 0.15	0.51 ± 0.07	0.53 ± 0.07	0.50 ± 0.06

be due to insufficient kinetic energy of deposited species and limited adatom mobility during film formation. The restricted adatom mobility limits surface diffusion, resulting in intergranular gaps and the production of a less dense, non-homogeneous coating, as is characteristic for sputtered coatings formed under low-energy circumstances (Greene, 2017). Increasing the sputtering voltage to 5 kV (Figure 3b) improves adatom mobility and promotes the development of a more uniform coating (Borowski and Myśliwiec, 2025). The quantitative ImageJ analysis reveals that the average grain size drops from 0.27 μm at 4 kV to 0.21 μm at 5 kV, demonstrating improved nucleation and grain refinement. Increasing the sputtering voltage to 6 kV (Figure 3c) results in a somewhat smaller grain size of 0.20 μm. Despite the continuous grain refinement, the coating deposited

at 6 kV has more visible surface protrusions and isolated flaws. This morphological evolution may be linked to enhanced ion bombardment, which can cause re-sputtering and defect formation under high-energy sputtering circumstances. Although re-sputtering was not directly investigated in the current study, earlier studies have linked similar surface properties to the rivalry between deposition and atom removal (Abadias et al., 2018; Greene, 2017). Sputtered coatings exhibit similar behavior, with deposition and atom removal competing for layer compactness, defect formation, and residual stress generation (Borowski and Myśliwiec, 2025). The three-dimensional surface topography obtained from ImageJ analysis (Figures 3d-3f) provides additional evidence for the morphological change shown in the SEM pictures. The

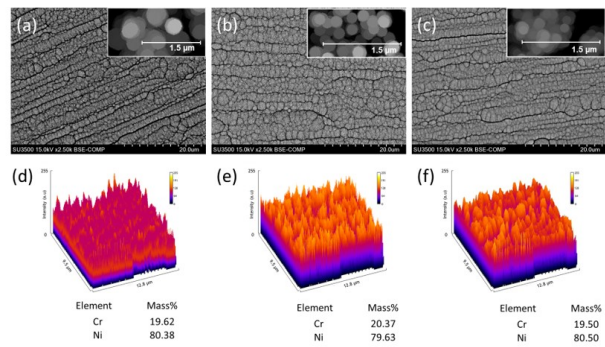


Figure 3. SEM Images Showing the Surface Morphology of Ni–Cr Coatings Deposited at Varying Sputtering Voltages: (A, D) 4 kV, (B, E) 5 kV, and (C, F) 6 kV

4 kV coating has relatively tiny height changes, which indicate insufficient coating growth and limited surface evolution. At 5 kV, the surface becomes more evenly distributed with fine and densely packed asperities, indicating the establishment of a rather compact and homogeneous coating morphology. Such morphology may be linked to higher kinetic energy of sputtered species and improved surface diffusion, facilitating atomic rearrangement and coating densification during film formation (Hnilica et al., 2024). In comparison, the 6 kV coating has prominent peaks and valleys, indicating increased relative surface roughness and larger surface asperities. This trend is consistent with earlier research indicating that excessive bombardment under high-energy sputtering conditions may increase re-sputtering and surface instability, however the current study does not directly test this process. Increasing sputtering voltage promotes grain refinement, but excessive deposition energy at 6 kV may deteriorate surface morphology due to re-sputtering-induced defects, despite continued reduction in grain size (Abadias et al., 2018; Borowski and Mysliwiec, 2025).

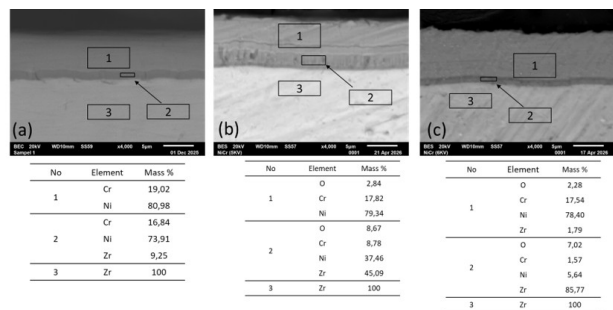


Figure 4. Representative EDS Spot Spectra Obtained From the Cross-Sections of Ni–Cr Coatings Deposited Under Varying Sputtering Voltages: (A) 4 kV, (B) 5 kV, and (C) 6 kV

EDS investigations of cross-sectional Ni-Cr coatings (Figures 4 and 5) show a well-defined interdiffusion zone at the coating-substrate interface, which is characterized by the pres-

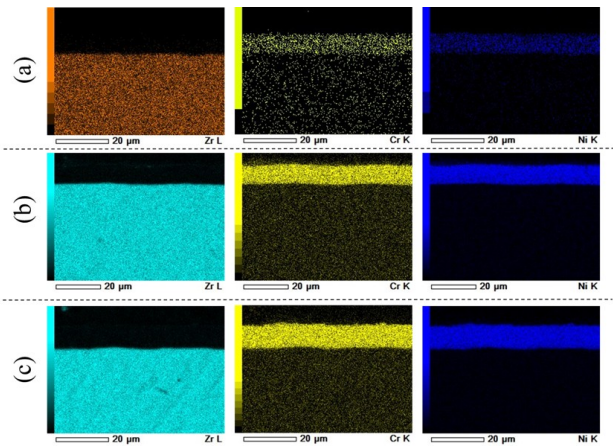


Figure 5. Cross-Sectional EDS Elemental Mapping of Zr, Cr, and Ni for Ni–Cr Coatings Deposited at Sputtering Voltages of (A) 4 kV, (B) 5 kV, and (C) 6 kV

ence of Ni, Cr, and Zr. This discovery suggests the presence of interfacial elemental mixing, which is most likely caused by diffusion-assisted processes during sputtering. The creation of this interdiffusion layer is due to increased atomic mobility caused by intense particle bombardment, which facilitates interfacial mixing (Fan et al., 2024; Hnilica et al., 2024). The EDS elemental mapping data supplements the cross-sectional EDS investigation by validating the elemental distribution inside the coating-substrate system. Ni and Cr are predominantly confined in the coating layer, while Zr is concentrated in the substrate. Although the elemental maps show a rather distinct coating-substrate border, the presence of Ni, Cr, and Zr near the interface supports the establishment of an interdiffusion area indicated by the EDS study. Similar elemental distributions and interfacial mixing have been observed for sputtered protective coatings on zirconium alloys, implying better interfacial bonding between the coating and substrate (Li et al., 2024a; Sidelev et al., 2019). Furthermore, the equal distribution of Ni and Cr throughout the coating indicates good chemical homogeneity of the deposited layer. Furthermore, the detection of oxygen in samples deposited at higher voltages (5 to 6 kV) suggests the possibility of surface oxidation, which might be caused by residual oxygen in the chamber or post-deposition exposure. This behavior is compatible with previously observed oxidation events in sprayed metallic coatings under non-ideal vacuum circumstances (Abadias et al., 2018).

Table 2. Deposition Rate of Ni–Cr Coatings at Different Sputtering Voltages

Sputtering Voltage	Thickness (μm)	Deposition Rate (μm/min)	Interdiffusion Zone (μm)
4 kV	7.9 ± 0.1	0.0329	1.38 ± 0.11
5 kV	8.3 ± 0.1	0.0346	2.48 ± 0.16
6 kV	9.1 ± 0.15	0.0379	1.16 ± 0.1

Cross-sectional SEM measurements (Figure 6 and Table 2) show that coating thickness gradually increases with increasing

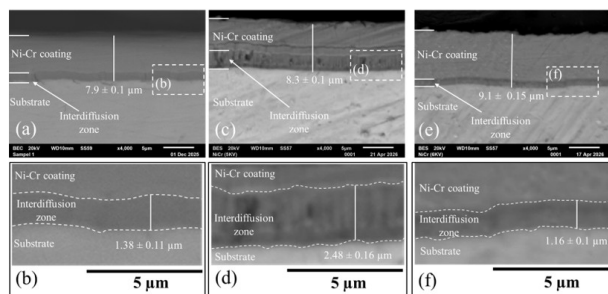


Figure 6. Cross-sectional SEM images of Ni-Cr coatings prepared at varying sputtering voltages: (a, b) 4 kV, (c, d) 5 kV, and (e, f) 6 kV

sputtering voltage, reaching $7.9 \pm 0.1 \mu\text{m}$, $8.3 \pm 0.1 \mu\text{m}$, and $9.1 \pm 0.15 \mu\text{m}$ at 4, 5, and 6 kV, respectively. Higher ion energy leads to increased sputtering yield and deposited species flux toward the substrate (Borowski and Mysliwiec, 2025). These thickness fluctuations show that sputtering voltage has a considerable influence on coating growth kinetics by altering sputtered species flow, surface diffusion, and film growth behavior.

In contrast, the interdiffusion zone exhibits a non-monotonic evolution, reaching its maximum thickness at 5 kV ($2.48 \pm 0.16 \mu\text{m}$) before decreasing at 6 kV ($1.16 \pm 0.1 \mu\text{m}$), indicating a transition in the dominant interfacial growth mechanism. At 5 kV, ion-enhanced atomic mobility promotes effective interfacial mixing, resulting in a graded interface that improves coating integrity and contributes to the lowest corrosion rate observed in this study (Gill et al., 2021). However, despite the continued increase in coating thickness at 6 kV, the reduction in interdiffusion thickness, together with the deterioration in hardness and corrosion resistance, suggests that excessive ion bombardment may alter the dominant growth mechanism. Re-sputtering can limit atomic incorporation and boost defect development during high-energy sputtering conditions (Borowski and Mysliwiec, 2025; Petrović et al., 2007). Although this mechanism was not directly tested in the current study, the observed microstructural evolution is consistent with recent research on the competition between ion-enhanced diffusion and ion-induced atom removal during sputter deposition (Anders, 2017; Hnilica et al., 2024). Overall, the SEM-EDS data show that the sputtering voltage controls the balance of deposition, interfacial diffusion, and probable re-sputtering processes. Among the examined circumstances, the 5 kV coating had the best microstructural properties, although having a somewhat lower coating thickness than the 6 kV condition. These findings indicate that the optimal coating performance was influenced not only by coating thickness, but also by the balance of coating densification, interfacial mixing, and defect development.

3.3 X-ray Diffraction (XRD)

Figure 7a depicts the X-ray diffraction (XRD) patterns of the Zr-4 substrate and Ni-Cr coatings deposited at sputtering volt-

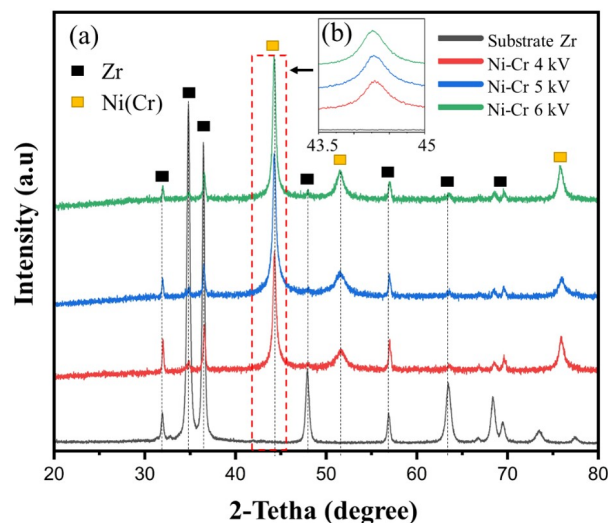


Figure 7. XRD Patterns of Zr Substrate and Ni-Cr Coatings Deposited at Different Voltages: (A) Overall Diffraction Profiles Showing α -Zr and Ni(Cr) Phases, and (B) Magnified Ni(Cr) Peak Region ($2\theta = 43.5\text{--}45^\circ$)

ages of 4, 5, and 6 kV, while Table 3 summarizes the peak indexing and structural characteristics. The diffraction peaks at $2\theta = 31.939^\circ$, 34.816° , 36.468° , and 47.931° correspond to the (100), (002), (101), and (102) planes of α -Zr with a hexagonal close-packed (HCP) crystal structure, suggesting that the substrate kept its original crystal structure after sputtering. The coated samples show diffraction peaks at 44.3° , 51.6° , and 75.9° , which correspond to the (111), (200), and (220) planes of the γ -Ni(Cr) face-centered cubic (FCC) solid-solution phase, confirming successful coating formation. No distinct diffraction peaks corresponding to body-centered cubic (BCC) Cr were found, showing that chromium did not precipitate as a discrete crystalline phase (Liu et al., 2024). The absence of Cr reflections and strong mutual solubility in the Ni-Cr system indicates the possibility of forming a γ -Ni(Cr) solid solution. This observation supports the potential of Cr integration into the γ -Ni lattice rather than the creation of a separate Cr-rich crystalline phase (Li et al., 2024b). However, the current XRD results are insufficient to unambiguously prove substitutional solid-solution formation; additional characterization using supplementary techniques such as XPS, TEM, or Rietveld refinement is necessary to confirm the phase constitution.

Table 4 summarizes the results of calculating the average lattice parameter of the FCC γ -Ni(Cr) phase from reflections (111), (200), and (220). The average lattice parameter increased significantly from $3.5394 \pm 0.002 \text{ \AA}$ for the 4 kV coating to $3.5400 \pm 0.002 \text{ \AA}$ and $3.5424 \pm 0.002 \text{ \AA}$ for the 5 and 6 kV coatings. The average lattice parameter increased slightly with increasing sputtering voltage, showing that the FCC γ -Ni(Cr) crystal structure remained stable and consistent with

Table 3. XRD Peak Indexing and Structural Parameters of the Zr-4 Substrate and Ni–Cr Coatings Deposited at Different Sputtering Voltages

Sample	Phase	hkl	2θ (°)	d-spancing (Å)	FWHM (°)
Substrate Zr-4	α -Zr	(100)	31.939	2.799	0.326
		(002)	34.816	2.575	0.447
		(101)	36.468	2.462	0.358
		(102)	47.931	1.898	0.460
		(111)	44.317	2.042	0.544
Ni–Cr 4 kV	γ Ni(Cr)	(200)	51.599	1.770	1.171
		(220)	75.947	1.252	0.745
		(111)	44.305	2.043	0.631
Ni–Cr 5 kV	γ Ni(Cr)	(200)	51.569	1.771	1.276
		(220)	75.972	1.252	0.814
		(111)	44.278	2.044	0.516
Ni–Cr 6 kV	γ Ni(Cr)	(200)	51.548	1.772	1.011
		(220)	75.875	1.253	0.750

Table 4. Structural Parameters of the Zr-4 Substrate and Ni–Cr Coatings Calculated From Williamson–Hall Analysis and XRD Lattice Parameter Determination

Sample	Crystal structure	Average lattice Parameter (Å)	Crystallite size (W-H) (nm)	Microstrain ($\times 10^{-4}$)
Substrate Zr-4	HCP	-	43.05	+6.44
Ni–Cr 4 kV	FCC	3.5394 ± 0.002	9.01	-3.83
Ni–Cr 5 kV	FCC	3.5400 ± 0.002	7.75	-5.35
Ni–Cr 6 kV	FCC	3.5424 ± 0.002	11.54	-0.65

chromium incorporation lattice (Li et al., 2024a). Figure 7b exhibits a small trend toward lower diffraction angles as sputtering voltage increases. According to Bragg’s law, this shift results in a modest increase in the interplanar distance and lattice parameter. Although the change is relatively small, the combination of the slight peak shift, the increase in lattice parameter, and the absence of detectable Cr reflections is consistent with the formation of a γ -Ni(Cr) solid solution accompanied by lattice distortion, which may be associated with Cr incorporation into the Ni lattice rather than the precipitation of a separate Cr-rich crystalline phase (Li et al., 2024a; Zhao et al., 2023).

Table 4 also summarizes the results of the Williamson–Hall (W-H) approach, which was used to separate the contributions of crystallite size and lattice strain to the XRD peak broadening. Compared to the untreated Zr-4 substrate, all coated samples had much lower crystallite sizes, showing that DC sputtering effectively corrected the coating microstructure. The crystallite size of the substrate reduced from 43.05 nm to 9.01 nm, 7.75 nm, and 11.54 nm for coatings deposited at 4, 5, and 6 kV, respectively. Among the examined circumstances, the 5 kV coating had the smallest crystallite size, showing that this sputtering voltage provided enough ion energy to improve surface adatom mobility while inhibiting excessive grain development.

The refined crystallite structure indicates the effect of sputtering voltage on the coating growth mechanism. These results demonstrate that sputtering voltage significantly influences the nucleation and growth behavior of the Ni–Cr coating.

The W-H analysis found positive microstrain for the Zr-4 substrate ($+6.44 \times 10^{-4}$) and negative microstrain for all Ni–Cr coatings (-3.83×10^{-4} , -5.35×10^{-4} , and -0.65×10^{-4} for the 4, 5, and 6 kV coatings, respectively). Negative microstrain values in magnetron-sputtered coatings imply compressive lattice strain caused by energetic particle bombardment (Abadías et al., 2018; Borowski and Mysliwiec, 2025). The coating deposited at 5 kV displayed the greatest magnitude of compressive microstrain and the smallest crystallite size, indicating that this deposition condition promoted simultaneous lattice distortion and crystallite refinement. In contrast, the lower magnitude of compressive microstrain observed at 6 kV could be attributed to partial relaxation of lattice distortion caused by enhanced atomic diffusion and localized grain growth during sputtering, resulting in a larger crystallite size than that obtained at 5 kV (Abadías et al., 2018). These findings suggest that the sputtering voltage regulates the balance of crystallite refinement and lattice strain development during coating growth.

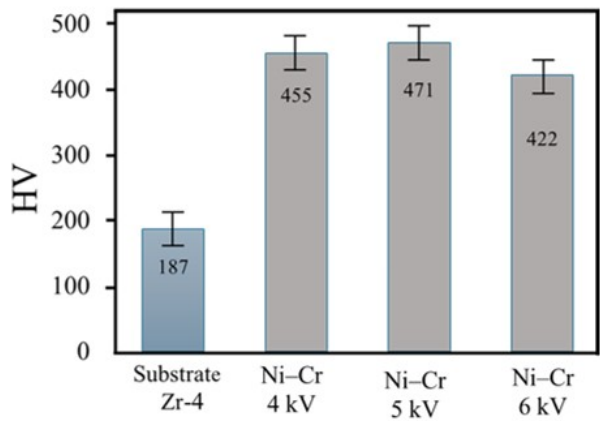


Figure 8. Vickers hardness values of the uncoated Zr-4 substrate and Ni-Cr coatings obtained using the Vickers indentation method

Table 5. Average Vickers Hardness (HV) of the Uncoated Zr-4 Substrate and Ni-Cr Coatings Deposited at Different Sputtering Voltages

Sample	HV	Standard deviation
Substrate Zr-4	187.63	5.06
Ni-Cr 4 kV	455.97	25.24
Ni-Cr 5 kV	471.11	10.93
Ni-Cr 6 kV	422.48	36.50

3.4 Vickers Hardness Test

The microhardness data (Table 5 and Figure8) show a considerable improvement in the mechanical performance of the Zr-4 substrate following Ni-Cr coating deposition. The untreated Zr-4 substrate had a hardness of 187.63 ± 5.06 HV, while the coated samples had significantly higher hardness values of 455.97 ± 25.24 HV, 471.11 ± 10.93 HV, and 422.48 ± 36.50 HV at sputtering voltages of 4, 5, and 6 kV, respectively. These hardness levels are comparable to recent findings for Ni-Cr-coated Zr-4 substrates. Wicaksono et al. (2026) found that Ni-Cr oxide coatings applied via electroplating and heat treatment improved the hardness of zirconium alloys from 167.2 ± 9.2 HV to 418.2 ± 43.5 HV, 510.8 ± 40.3 HV, and 627.1 ± 72.3 HV. Although the coating fabrication technique and strengthening mechanisms differ, both investigations clearly show that Ni-Cr-based surface modification significantly increases the surface hardness of zirconium alloys. The higher hardness suggests that Ni-Cr deposition improves resistance to indentation-induced plastic deformation.

The 5 kV sputtering voltage resulted in the maximum hardness (471.11 ± 10.93 HV), suggesting the best mechanical reaction among the coatings. Similarly, Wicaksono et al. (2026) verified increased load-bearing capacity in Ni-Cr-coated Zr-4 substrates, indicating that Ni-Cr surface modification improves

Table 6. Nanoindentation-Derived Mechanical Properties of the Uncoated Zr-4 Substrate and Ni-Cr Coatings Deposited at Different Sputtering Voltages

Sample	Hardness H (GPa)	Young's Modulus E (GPa)	H/E	H ³ /E ² (GPa)
Substrate Zr-4	3.87 ± 0.84	105.85 ± 10.74	0.0364 ± 0.0067	0.00567 ± 0.00329
Ni-Cr 4 kV	8.29 ± 1.22	183.86 ± 23.41	0.0451 ± 0.0044	0.0171 ± 0.0048
Ni-Cr 5 kV	8.76 ± 1.48	195.57 ± 30.24	0.0449 ± 0.0042	0.0181 ± 0.0053
Ni-Cr 6 kV	7.30 ± 2.08	189.45 ± 37.48	0.0379 ± 0.0048	0.0113 ± 0.0057

mechanical performance Overall, coating densification, crystallite refinement, and better interfacial integrity all contributed to the Ni-Cr coating’s increased hardness.

In contrast, the 4 kV coating had a reduced hardness due to its non-uniform coating morphology and inadequate surface coverage, as shown by uneven grain distribution and localized substrate exposure observed by EDS. Although the coating thickness increased with sputtering voltage, the hardness evolution was not directly proportional to thickness. Despite having the highest coating thickness (about 9.1 μ m), the 6 kV coating had a lower hardness of 422.48 ± 36.50 HV. This drop in hardness is associated with a minor rise in crystallite size, a reduction in interdiffusion layer thickness, and worsening of surface shape as shown in SEM images. These findings indicate that raising the sputtering voltage from 5 to 6 kV changed the film formation mechanism, resulting in a less favorable microstructure for hardness augmentation due to lower interfacial strengthening and increased structural heterogeneity.

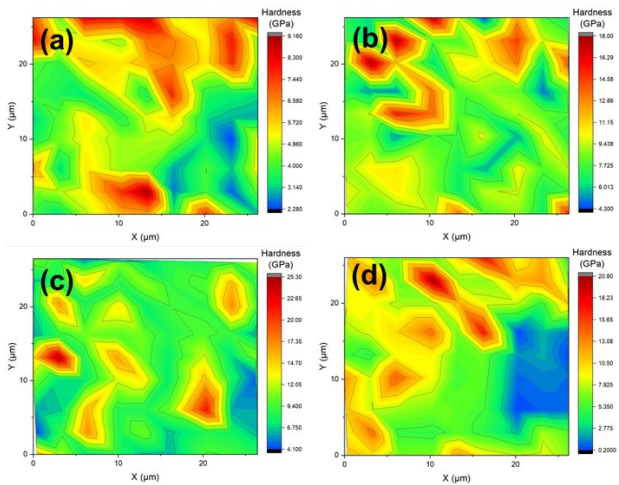


Figure 9. Spatial Distribution of Nanoindentation Hardness for the (A) Uncoated Zr-4 Substrate and Ni-Cr Coatings Deposited at (B) 4 kV, (C) 5 kV, and (D) 6 kV

3.5 Nanoindentation Mechanical Properties

The two-dimensional hardness distribution maps (Figure 9) show how sputtering voltage affects the spatial homogeneity of mechanical characteristics in the deposited coatings. These discoveries are consistent with the quantitative nanoindenta-

tion results presented in Figure 11a and Table 6. The average hardness increased significantly from 3.87 ± 0.84 GPa for the uncoated Zr-4 substrate to 8.29 ± 1.22 GPa, 8.76 ± 1.48 GPa, and 7.30 ± 2.08 GPa for coatings deposited at sputtering voltages of 4, 5, and 6 kV, respectively. The considerable increase in hardness following Ni-Cr deposition is consistent with the findings of Sidelev et al. (2019) who identified hardness as one of the important mechanical criteria governing the suitability of sputtered protective coatings for ATF claddings. While their study primarily compared different coating compositions, the current work shows that optimizing the sputtering voltage within the Ni-Cr coating system is equally important, as the 5 kV deposition condition produced the highest hardness and the most homogeneous distribution. Nanoindentation yields consistently greater hardness readings than the Vickers method because it probes a considerably smaller deformation volume, where the indentation size effect is more prominent (Metzger et al., 2017). Furthermore, for thin-film systems, the larger plastic deformation zone generated during Vickers indentation may extend into the Zr-4 substrate, causing the measured hardness to reflect the combined response of the coating and substrate, resulting in lower apparent hardness values than those obtained by nanoindentation (Ul-Hamid, 2021).

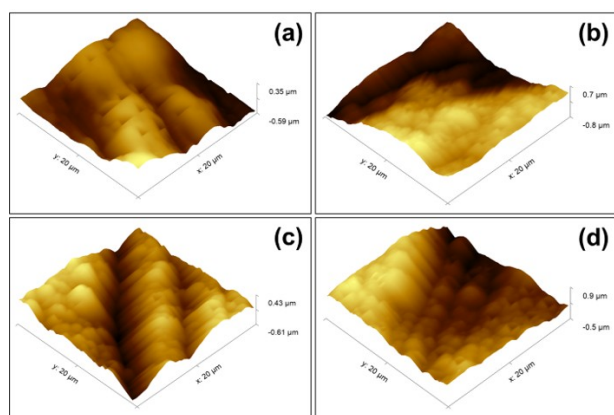


Figure 10. Three-Dimensional Surface Topography Obtained by In-Situ Scanning Probe Microscopy (SPM) Over a Scanning Area of $20 \times 20 \mu\text{m}^2$ for (A) Uncoated Zr-4 Substrate and Ni-Cr Coatings Deposited at (B) 4 kV, (C) 5 kV, and (D) 6 kV

The three-dimensional surface topography (Figure 10) obtained with the integrated Scanning Probe Microscope (SPM) clarifies the effect of sputtering voltage on the surface morphology of the deposited coatings. Measurements were taken over a $20 \times 20 \mu\text{m}^2$ scanning region. The uncoated Zr-4 substrate (Figure 10a) has an uneven surface with a vertical height range of roughly -0.59 to $0.35 \mu\text{m}$, indicating surface relief after polishing and nanoindentation scanning. Following Ni-Cr deposition, the coatings have different topographical properties depending on the sputtering voltage. The coating deposited at 4 kV (Figure 10b) has a wider vertical height variation (-0.80

to $0.70 \mu\text{m}$), indicating that the deposited layer has not yet acquired a fully uniform surface shape. The 5 kV coating (10c) has a narrower height range (-0.61 to $0.43 \mu\text{m}$) and a more homogeneous surface profile, indicating a denser and uniform coating. The higher kinetic energy of sputtered species, together with surface diffusion, promotes adatom mobility and compact film growth (Borowski and Mysliwiec, 2025; Han et al., 2024). At 6 kV (Figure 10d), the surface exhibits the greatest vertical fluctuation (-0.50 to $0.90 \mu\text{m}$), indicating the formation of more prominent surface undulations. Excessive ion bombardment may cause surface irregularity at 6 kV, leading to localized defect formation and re-sputtering under high-energy deposition conditions. However, these mechanisms were not directly investigated in the current study (Abadias et al., 2018; Borowski and Mysliwiec, 2025). These findings show that sputtering voltage has a considerable influence on the surface mechanical response of Ni-Cr coatings.

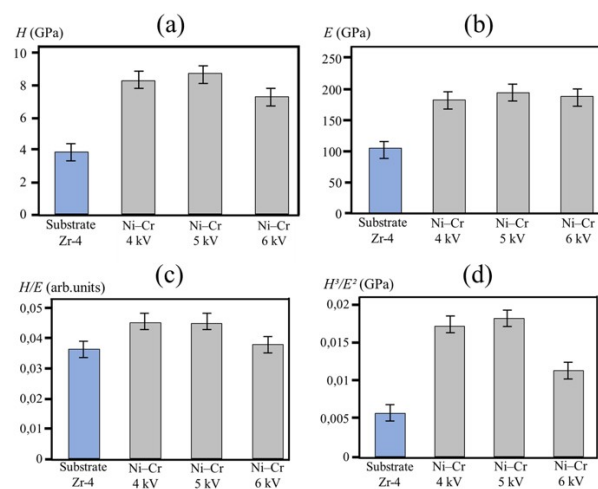


Figure 11. Nanoindentation-Derived Mechanical Properties of the Uncoated Zr-4 Substrate and Ni-Cr Coatings Deposited at Sputtering Voltages of 4, 5, and 6 kV: (A) Hardness, (B) Young's Modulus, (C) H/E Ratio, and (D) H^3/E^2 Ratio

The increase in hardness was accompanied by a significant improvement in Young's modulus (Figure 11b and 6), which increased from 105.85 ± 10.74 GPa for the uncoated Zr-4 substrate to 183.86 ± 23.41 , 195.57 ± 30.24 , and 189.45 ± 37.48 GPa for the coatings deposited at 4, 5, and 6 kV, respectively. The higher Young's modulus implies that the Ni-Cr coatings significantly increased the elastic stiffness of the Zr-4 substrate, making it more resistant to elastic deformation under contact loading. Among the coated samples, the 5 kV coating had the highest Young's modulus, indicating that this deposition setting resulted in the stiffest coating. The maximum Young's modulus found at 5 kV may relate to the creation of a reasonably compact coating morphology and improved interfacial properties, as shown by SEM-EDS analysis. In contrast, the decrease in hardness and Young's modulus observed for

Table 7. Corrosion Rate Values of the Uncoated Zr-4 Substrate and Ni–Cr Coatings Deposited at Different Sputtering Voltages

Sample	E _{corr} (V)	I _{corr} (A/cm ²)	Corrosion rate (mm/year)
Substrate Zr-4	$(-3.063 \pm 0.032) \times 10^{-1}$	$(8.087 \pm 0.194) \times 10^{-7}$	$(9.193 \pm 0.221) \times 10^{-3}$
Ni–Cr 4 kV	$(-8.580 \pm 0.020) \times 10^{-2}$	$(3.222 \pm 0.297) \times 10^{-7}$	$(3.663 \pm 0.338) \times 10^{-3}$
Ni–Cr 5 kV	$(1.760 \pm 0.078) \times 10^{-2}$	$(1.956 \pm 0.115) \times 10^{-7}$	$(2.224 \pm 0.131) \times 10^{-3}$
Ni–Cr 6 kV	$(-5.930 \pm 0.010) \times 10^{-2}$	$(2.400 \pm 0.152) \times 10^{-7}$	$(2.729 \pm 0.173) \times 10^{-3}$

the 6 kV coating was accompanied by an increase in crystallite size and a less homogeneous surface morphology, implying that increasing the sputtering voltage from 5 to 6 kV altered the coating growth behavior and reduced its mechanical efficiency.

To assess the coatings’ deformation behavior, the H/E and H^3/E^2 ratios were computed, as shown in Figure 11 c-d and 6. The H/E ratio (elastic strain tolerance) rose from 0.0364 ± 0.0067 for the uncoated Zr-4 substrate to 0.0451 ± 0.0044 , 0.0449 ± 0.0042 , and 0.0379 ± 0.0048 for coatings deposited at 4, 5, and 6 kV. The coatings produced at 4 and 5 kV had virtually identical H/E values, indicating similar resistance to elastic strain. Although the 5 kV coating had the maximum hardness, its slightly lower H/E ratio was due to the concomitant increase in Young’s modulus, and the standard deviations overlapped, indicating that the difference between the two coatings is unlikely to be substantial. The H/E ratios obtained in the current investigation are still less than 0.1, indicating that further tuning may be necessary to improve resistance to crack formation under severe mechanical loading. Sidelev et al. (2019) observed that magnetron-sputtered Ni–Cr coatings had greater H/E and H^3/E^2 ratios than pure Cr coatings, indicating improved cracking resistance. However, the H/E values remained less than 0.1, indicating that additional optimization of the deposition process is required to improve coating toughness.

In contrast, the H^3/E^2 ratio, which is widely regarded as an indicator of resistance to plastic deformation (Figueiredo et al., 2024), reached its highest value for the 5 kV coating (0.0181 ± 0.0053 GPa), followed by the 4 kV (0.0171 ± 0.0048 GPa) and 6 kV (0.0113 ± 0.0057 GPa) coatings, whereas the uncoated substrate exhibited the lowest value (0.00567 ± 0.00329 GPa). The higher H^3/E^2 value at 5 kV indicates superior resistance to plastic deformation. This is consistent with the trend reported by Sidelev et al. (2019) who identified Sidelev et al. (2019) as a reliable parameter for assessing the mechanical durability of sputtered protective coatings for nuclear fuel claddings. Overall, the nanoindentation results show that the 5 kV deposition condition provided the best mechanical performance within the investigated voltage range, demonstrating the potential of Ni–Cr coatings as a surface modification approach for improving the mechanical performance of Zr-4 alloys used in nuclear cladding applications.

3.6 Potentiodynamic Polarization Test

According to corrosion test results (Figure 12 and Table 7), the uncoated Zr-4 substrate had the greatest I_{corr} of $(8.087$

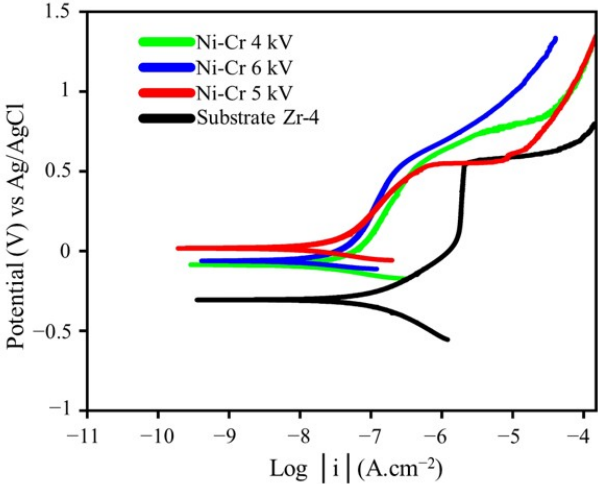


Figure 12. Nanoindentation-Derived Mechanical Properties of the Uncoated Zr-4 Substrate and Ni–Cr Coatings Deposited at Sputtering Voltages of 4, 5, and 6 kV: (A) Hardness, (B) Young’s Modulus, (C) H/E Ratio, and (D) H^3/E^2 Ratio

$\pm 0.194) \times 10^{-7}$. The highest corrosion rate $(9.193 \pm 0.221) \times 10^{-3}$ mm/year showing strong electrochemical activity in the 4000 ppm H₃BO₃ and 2.5 ppm LiOH solution. Following Ni–Cr deposition, the I_{corr} value and corrosion rate dropped, showing the coating layer’s increased corrosion resistance. This improvement could be attributed to the Ni- and Cr-containing phases detected by XRD, which can diminish the electrochemical activity of the substrate surface. In particular, Cr inclusion enhances the creation of a persistent and adherent Cr₂O₃ passive coating, which inhibits anodic dissolution and corrosion propagation (Sidelev et al., 2022). As a result, the Ni–Cr layer acted as a protective surface modification layer, increasing the corrosion resistance of the Zr-4 substrate in the examined aqueous environment. This finding is consistent with the findings of Golgovici et al. (2024), who discovered that chromium coatings deposited on Zircaloy-4 significantly improved corrosion resistance by forming a dense Cr₂O₃ passive layer, effectively suppressing anodic dissolution and limiting electrolyte penetration. Although their study used pure Cr coatings and the current study used Ni–Cr coatings, both studies found that chromium-containing coatings can improve the electrochemical stability of Zr-4 substrates by lowering corrosion current density and improving surface protection behavior.

The Ni–Cr coating deposited at 4 kV had a thickness of 7.9 μ m, an interdiffusion zone of 1.2 μ m, and a corrosion rate of $(3.663 \pm 0.338) \times 10^{-3}$ mm/year, indicating improved corrosion resistance compared to the uncoated Zr-4 substrate. However, its performance was inferior to that of the 5 kV sample. This observation is consistent with the current investigation, which found that the best corrosion performance at 5 kV was related with the thickest interdiffusion layer and refined crystallite structure rather than the maximum coating thickness.

Ni-Cr coating applied at 5 kV had the lowest Icorr (1.956 ± 0.115) $\times 10^{-7}$. The corrosion rate was $(2.224 \pm 0.131) \times 10^{-3}$ mm/year, suggesting the most effective corrosion prevention among the examined conditions (Fan and Huang, 2013; Singh and Marya, 2019). Similar behavior has been observed in sputtered Ni-Cr-based coatings, that increased coating compactness and interfacial stability improved corrosion resistance. Wicaksono et al. (2026) discovered a similar tendency when they tested Cr-Ni coated zirconium alloys made by electroplating and heat treatment. They found that enhanced coating densification and oxide layer stability considerably reduced corrosion rate, implying that corrosion resistance was influenced not only by coating composition but also by coating integrity and microstructural evolution. This observation is consistent with the current investigation, which found that the best corrosion performance at 5 kV was related with the thickest interdiffusion layer and refined crystallite structure rather than the maximum coating thickness. The 6 kV sample had a thicker coating (9.1 ± 0.15 μm), but a thinner interdiffusion zone (1.16 ± 0.1 μm) and a higher corrosion rate ($2.729 \pm 0.173 \times 10^{-3}$ mm/year). This finding shows that excessive bombardment at higher sputtering voltages caused lattice distortion, residual stress, and microstructural flaws, compromising coating integrity (Abadias et al., 2018). As a result, the corrosion resistance of the Ni-Cr coating was determined not only by coating thickness, but also by microstructural compactness and coating-substrate interfacial stability. Among the evaluated deposition circumstances, the 5 kV sample provided the best corrosion protection performance in the tested aqueous environment.

4. CONCLUSION

This work examined the effect of sputtering voltage on the microstructure, mechanical characteristics, and corrosion behavior of DC-sputtered Ni-Cr coatings formed on Zr-4 substrates. The findings show that sputtering voltage is crucial in determining crystallite size, coating morphology, and coating/substrate interface properties, which all influence coating performance. The coating deposited at 5 kV had the best microstructural, mechanical, and corrosion properties, with the smallest crystallite size (7.75 nm), thickest interdiffusion layer (2.48 ± 0.16 μm), compact and homogeneous microstructure, improved hardness, and lowest corrosion current density and rate. These data show that adjusting the coating microstructure, rather than increasing coating thickness, is the most important aspect in improving the protective efficacy of sputtered Ni-Cr coatings. These data show that adjusting the coating microstructure, rather than increasing coating thickness, is the most important aspect in improving the protective efficacy of sputtered Ni-Cr coatings. This study used SEM, XRD (Williamson-Hall), SEM-EDS, and electrochemical investigations to show a clear association between sputtering voltage, microstructural evolution, and the mechanical and corrosion performance of Ni-Cr coatings. The results give significant recommendations for optimizing sputtering settings for Ni-Cr coatings under the laboratory conditions explored in this study, as well as demon-

strating their promise for ATF cladding applications. Because the current study was limited to room-temperature characterization, future research should include high-temperature oxidation and corrosion testing under simulated reactor and LOCA conditions, irradiation performance evaluation, and advanced microstructural characterization to determine the long-term suitability of Ni-Cr coatings for nuclear fuel cladding.

5. ACKNOWLEDGEMENT

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