

Synthesis of Nuclear Fuel Based on U-6Zr-xNb Alloy (x = 0, 1, 4, and 7 wt.%): Corrosion Product Analysis and γ -U Phase Stability in Acidic and Alkaline Environment

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Abstract

The U-6Zr alloys, considered as nuclear fuel, must endure mechanical, thermal, and corrosive loads. Corrosion resistance is crucial during reactor operation and storage, as environmental conditions can trigger material degradation. Niobium (Nb) addition is expected to suppress corrosion. The U-6Zr-xNb alloys (x = 0, 1, 4, and 7 wt.%) were fabricated using arc-melting and tested in acidic and alkaline environments. XRD analyses before corrosion revealed that x = 0 and 1 wt.% alloys consisted of a single α -U phase, while x = 4 wt.% showed both α -U and γ -U phases. At x = 7 wt.%, only the γ -U phase was present, but after corrosion, XRD showed the formation of α -U and UO₂ phases for x = 0 and 1 wt.%, α -U, γ -U, and UO₂ phases for x = 4 wt.%, and only γ -U phase for x = 7 wt.%. Electrochemical tests demonstrated that E_{corr} increased and corrosion CR decreased with higher Nb content. Thus, Nb effectively enhanced corrosion resistance of U-6Zr alloys. Among all compositions, U-6Zr-7Nb alloy showed the lowest corrosion rate and highest resistance in both environments, suggesting its strong potential as a nuclear fuel candidate.

Keywords

U-6Zr-xNb, Corrosion Product, γ -U Phase, Acidic, Alkaline

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1. INTRODUCTION

The oxidation mechanism of uranium metal, especially in its interaction with water, is strongly influenced by oxygen (O₂) and hydroxide ions (OH⁻). However, several studies on the corrosion mechanism of uranium metal have focused on the formation of uranium hydride (UH₃), which is produced during the corrosion reaction. The formation of this hydride is influenced by various factors, such as temperature, hydrogen pressure, oxidation by water, and the specific reaction mechanism that affects the corrosion rate (Banos et al., 2018; Guyadec et al., 2010; Mohammadian et al., 2026). Uranium metal has a strong affinity for oxygen, making it readily oxidized in air, even under Ultra High Vacuum (UHV) conditions (Banos and Scott, 2020). During the initial reaction stage, the adsorbed water undergoes complete dissociation, with complete dissociation being the dominant process (Banos and Scott, 2020; El-Wazery et al., 2022). The adsorption process follows the Langmuir chemisorption mechanism (Banos et al., 2016) forming oxides, which mark the beginning of the formation of a continuous oxide layer.

One of the fundamental components of the reactor is the moderator, which can be light water (H₂O) or heavy water (D₂O). The moderator slows down fast neutrons from energies of a few MeV to thermal neutrons (~0.025 eV) and also acts as a coolant to absorb heat (Dobrzynski et al., 1994). The moderator should ideally be free of impurities, but in practice, impurities are still present, which can make the environment acidic or alkaline. In addition, impurities can capture neutrons and become radioactive through (n, γ) reactions. The decomposition of water by nuclear radiation is also often facilitated by the presence of ionic impurities. Therefore, reactor fuel must be able to withstand corrosive environments.

Because uranium is the fuel for nuclear reactors and given the very low natural abundance of fissile uranium isotopes, enrichment technologies are necessary. Several studies have explored this approach by combining uranium metal with transition metals or oxides, such as U₃Si₂-Al alloys (Kim and Hofman, 2011; Nelson et al., 2018). U-ZrHx alloys (Simnad, 1981), uranium oxides (Olander, 2009), and U₃O₈-Al oxide composites (Durazzo et al., 2017). Recently, U-Mo and U-

Zr-based uranium alloys have been developed as candidates for future reactor fuels (Xie et al., 2019; Snead et al., 2019; Janney and Hayes, 2018; Aitkaliyeva, 2022). Although U-Mo alloys exhibit excellent irradiation performance, the high cost and limited availability of molybdenum have encouraged the development of U-Zr alloys as a more economical alternative, owing to the greater abundance of zirconium.

U-Zr-based alloys are chosen for several reasons, including high density, low neutron absorption cross section, domestic availability, high thermal stability, good thermal conductivity, ease of fabrication, absence of intermetallic compounds, and high solidus temperature (Zhou et al., 2021; Carmack et al., 2009). However, at room temperature, U-Zr alloys exhibit an unstable α -U phase. Therefore, alloying strategies are needed to stabilize the γ -U phase. One such strategy is the development of U-Zr-Nb alloys. The addition of Nb to U-Zr alloys can convert the α and δ phases into the γ phase through substituting and increasing the Nb content improves the stability of the γ phase (Zhou et al., 2018). Ternary uranium-zirconium-niobium (U-Zr-Nb) alloys are promising nuclear fuels for research reactors, high-flux reactors (HFRs), and fast breeder reactors. These alloys offer many advantageous properties, including higher thermal conductivity and density, ease of fabrication, and good compatibility with fuel cladding (Ferreira et al., 2018). Both Zr and Nb exhibit low microscopic neutron absorption cross sections and high solubility in the γ -U phase. The addition of Nb dissolved in γ -U can significantly improve the corrosion resistance under rapidly cooled γ -U conditions and help stabilize γ -U at low temperatures (Wu et al., 2016). U-Zr-Nb alloys also exhibit good mechanical, physical, and thermal properties (Kaity et al., 2018; Banos and Scott, 2020; Kaity et al., 2020).

Since corrosion directly determines fuel performance, it is an important focus in nuclear fuel development. Among the most significant factors affecting the corrosion of uranium alloys are solution pH, aeration, alloying element content, and impurities. Therefore, studies addressing pH, aeration, and alloying elements such as Nb are needed to evaluate their impact on the corrosion resistance of U-Zr-based alloys. Basically, the main objective of this paper is to present a new perspective on the stability of the γ phase and its corrosion resistance, which focuses more on corrosion products through experimental and analytical data. Therefore, the supporting data is more directed towards phase stability based on the results of qualitative and quantitative crystal structure analysis of the U-6Zr-xNb alloy, while its correlation with corrosion resistance and corrosion products is also discussed. This finding ultimately aims to see the potential of the U-Zr-Nb alloy as a promising candidate for nuclear reactor fuel.

In this study, the addition of Nb to U-Zr alloys, which normally exhibit an unstable α -U phase, was employed to promote γ -phase stabilization and improve structural stability. Furthermore, Nb addition is expected to enhance corrosion resistance, thereby contributing to improved mechanical and thermal performance. Previous studies have reported the influence of Nb

on phase stability and corrosion behavior of uranium-based alloys. However, the relationship between γ -phase stabilization, corrosion resistance, and corrosion products in U-Zr-Nb alloys remain insufficiently explored. Therefore, this work presents a preliminary investigation that provides additional insight into the role of Nb in modifying the microstructural stability and corrosion behavior of U-6Zr-xNb alloys. Phase stability was evaluated through qualitative and quantitative crystal structure analyses, while its correlation with corrosion resistance and corrosion products was examined using experimental data. The main contribution of this study lies in improving the understanding of how Nb addition influences γ -U phase stability and corrosion performance in U-Zr-Nb alloys, thereby supporting their potential application as candidate nuclear reactor fuels.

2. EXPERIMENTAL SECTION

2.1 Materials

The metallic raw materials used in this study included low-enriched uranium metal supplied by the American National Laboratory (99.78% purity), nuclear-grade zirconium (Zr) metal (Aldrich, 99.9% purity), and niobium (Nb) sheet (Aldrich, 99.8% purity). Chemical reagents included HNO₃ for the uranium metal pickling process, mounting resin for sample preparation during microstructural analysis, and ultra-high-purity (UHP) argon gas as the protective atmosphere during alloy synthesis.

2.2 Alloy Preparation

The U-6Zr-xNb alloys (x = 0, 1, 4, and 7 wt.%) were fabricated using an arc-melting furnace under an ultra-high-purity argon atmosphere. Prior to melting, the chamber was evacuated to approximately 10⁻⁶ mbar using a combination of a rotary pump and a diffusion pump to minimize contamination. The melting temperature was maintained at approximately 1400 °C to ensure complete melting and homogeneity. Each alloy button was remelted six times to improve chemical homogeneity, producing ingot samples with a diameter of approximately 20 mm and a thickness of approximately 5 mm (weighing approximately 15 g each). The ingots were subsequently sectioned using a Struers low-speed cutting machine and mounted in resin for further characterization.

2.3 Electrochemical Corrosion Test

Prior to corrosion testing, the alloy samples were ground to a 1500-grit finish, mounted in resin, and connected to a copper wire for electrical contact. The exposed surface area of each working electrode was measured accurately. Electrochemical measurements were carried out using a standard three-electrode corrosion cell connected to a GAMRY Reference 600 potentiostat. The alloy sample served as the working electrode, a platinum wire as the counter electrode, and a saturated calomel electrode (SCE) as the reference electrode.

Testing was performed in both acidic (HNO₃ solution) and alkaline (NaOH solution) environments at room temperature. The electrochemical evaluation sequence consisted of open

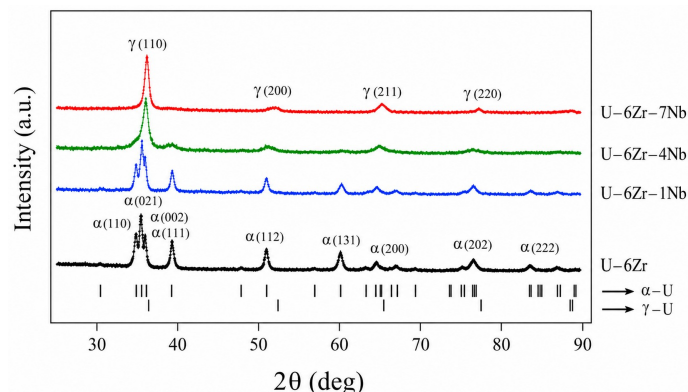


Figure 1. XRD Patterns of U-6Zr-xNb Alloys ($x = 0, 1, 4$ and 7 wt.%)

circuit potential (OCP), linear polarization resistance (LPR), and Tafel polarization measurements. LPR measurements were executed over a potential range of -0.02 V to $+0.02$ V vs. OCP at a scan rate of 0.05 mV/s. Data analysis was conducted using Gamry Echem Analyst DC105 software to calculate the corrosion current density (I_{corr}) and corrosion rate (CR).

2.4 Characterization

Phase identification and crystal structure characterization of the U-6Zr-xNb alloys were performed using a PANalytical Empyrean X-ray diffractometer with a "Cu-K" α anode ($\lambda = 1.5406 \text{ \AA}$). The diffraction profiles were recorded over a 2θ range of 25 – 90° with a step size of 0.02° . Quantitative phase analysis was conducted using the Rietveld refinement method implemented in GSAS (General Structure Analysis System) software.

Microstructural features and elemental distributions were evaluated using a field-emission scanning electron microscope equipped with an energy-dispersive X-ray spectrometer (FE-SEM-EDS; Phenom Pharos G1 Desktop SEM-FEG, Thermo Scientific). Prior to SEM observation, mounted samples were sequentially ground using SiC abrasive papers up to 1500 grit and subsequently polished with $1 \mu\text{m}$ diamond paste. The polished specimens were then chemically etched using a solution consisting of 3 M HNO_3 and 1 M HF mixed at a volume ratio of $1:1$ to reveal the grain boundaries and phase morphology. EDS analysis was conducted to quantify elemental composition and verify chemical homogeneity across the alloy matrix.

3. RESULTS AND DISCUSSIONS

3.1 Condition Before Corrosion

Figure 1 shows the ingots synthesized for U-6Zr-xNb alloys ($x = 0, 1, 4$, and 7 wt.%), which were successfully produced using the arc-melting method. U-6Zr, U-6Zr-1Nb, U-6Zr-4Nb, and U-6Zr-7Nb alloys are uranium-based solid solutions, as the alloys are dominated by the uranium crystal structure. Figure 1 shows the XRD results used to determine the phase formation of U-6Zr-xNb alloys ($x = 0, 1, 4$, and 7 wt.%).

XRD measurements of U-6Zr-xNb alloys ($x = 0, 1, 4$, and 7 wt.%) were carried out at room temperature within the diffraction angle range of $2\theta = 25$ – 90° . For the alloy composition $x = 0$, the diffraction pattern corresponds to the orthorhombic α -U phase, consistent with JCPDS card no. 96-900-8585. The diffraction peaks of the α -U phase were observed at 2θ values of 30.49° , 34.93° , 35.57° , 36.18° , 39.51° , 51.17° , 60.34° , 65.33° , 77.02° , and 85.05° , corresponding to the (020), (110), (021), (002), (111), (112), (131), (200), (202), and (222) crystal planes. For the alloy composition $x = 1$ wt.%, the diffraction pattern also corresponds to the α -U phase with an orthorhombic structure, containing U, Zr, and Nb elements. In contrast, the alloy composition $x = 4$ wt.% exhibits two phases: α -U with an orthorhombic structure and γ -U with a cubic structure. Furthermore, the alloy composition $x = 7$ wt.% shows a diffraction pattern consistent with the γ -U phase with a cubic structure, containing U, Zr, and Nb. The diffraction peaks of the γ -U phase match the values reported in the JCPDS card no. 96-900-8557, with peaks observed at 2θ values of 36.43° , 52.47° , 65.56° , and 77.39° , corresponding to the (110), (200), (211), and (220) crystal planes. The phase identification results for all compositions of U-6Zr-xNb alloys are in close agreement with previous reports (Olander, 2009). The XRD data were further analyzed using the GSAS software to determine phase composition and crystal structure (Toby, 2001; Idris and Osman, 2013). Figure 2 presents the XRD pattern refinements of U-6Zr-xNb alloys ($x = 0, 1, 4$, and 7 wt.%), with the reliability factors Rwp, Rexp, Rp and GoF (χ^2) meeting the refinement criteria. These analyses were complemented by surface morphology observations using BSE-SEM, which distinguishes phases based on contrast differences. According to the refinement results obtained using the GSAS software, the incorporation of Nb atoms, the presence of Nb atoms in this structure can alter all the structural parameters of the sample, as presented in Table 1. Figures 2(a) and 2(b) show the refined XRD patterns for the $x = 0$ (U-6Zr) and $x = 1$ wt.% (U-6Zr-1Nb) alloys, respectively, both of which exhibit a single-phase Bragg diffraction peak corresponding to the α -U structure. The BSE-SEM image of the $x = 1$ wt.% alloy confirms this result, as the surface morphology shows no observable contrast differences across the sample surface, indicating single-phase homogeneity. Confirmation of the results should be possible using SAED (selected area electron diffraction) analysis in transmission electron microscopy (TEM), considering that TEM/SAED test data are ideal for supporting XRD analysis results because they provide direct visual confirmation at the nanoscale. In addition, these methods complement and verify the crystallinity data of the alloys being tested. However, this test has not been possible due to the limited testing facilities for radioactive materials.

Figure 2(c) shows the XRD pattern refinement for the alloy composition $x = 4$ wt.% (U-6Zr-4Nb), which exhibits Bragg diffraction peaks corresponding to two phases, namely α -U and γ -U. The BSE-SEM image of the $x = 4$ wt.% alloy reveals surface morphology with visible contrast differences. Light-gray and dark-gray regions appear on the surface, indicating

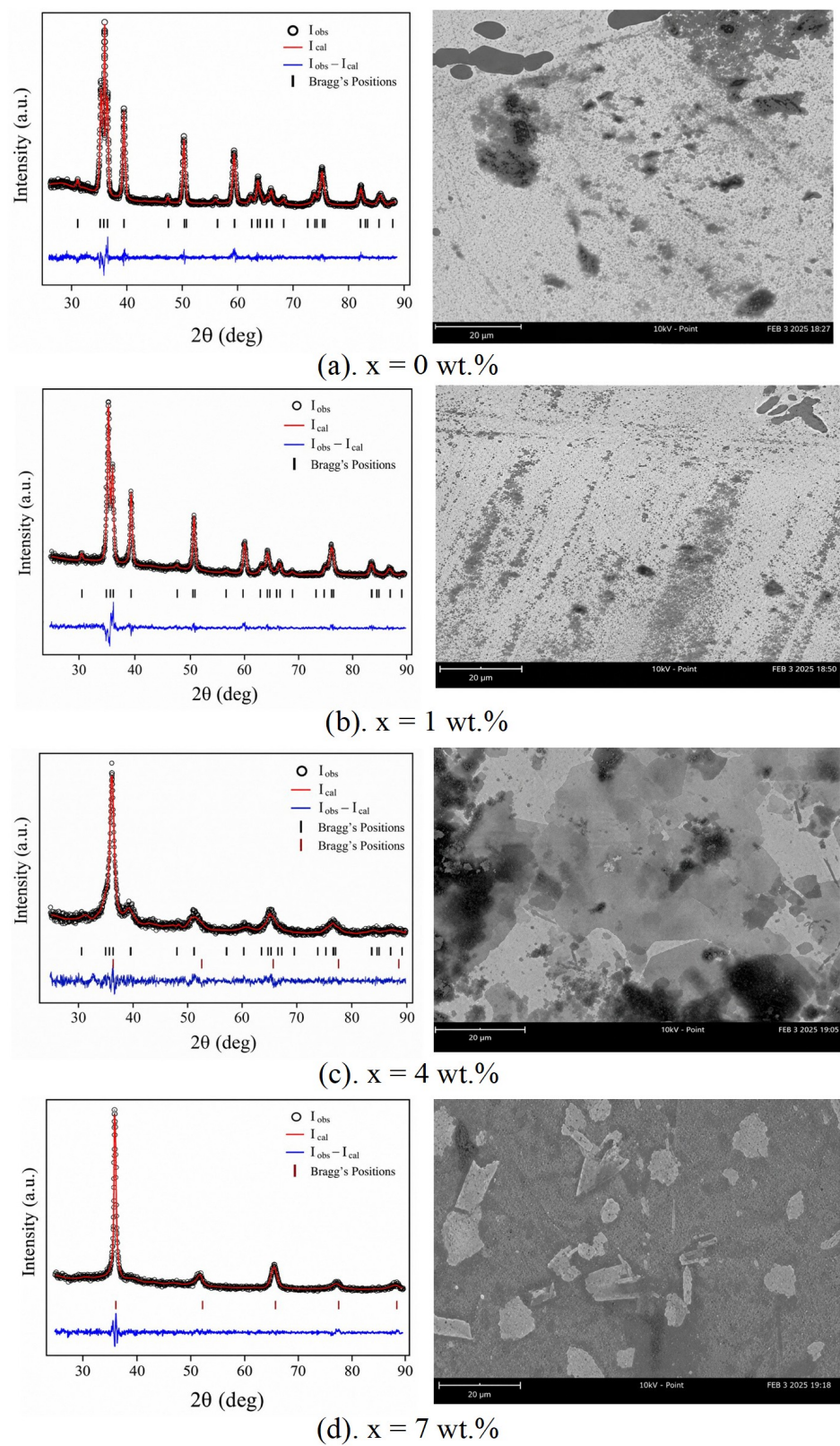


Figure 2. Refinement Result of X-Ray Diffraction Pattern and BSE SEM Image of U-6Zr-xNb Alloys

Table 1. Crystal Structure Parameters, Fit Criteria (R_p , R_{exp} , R_{wp}), and Goodness of Fit (GoF or χ^2) on U-6Zr- x Nb Alloy ($x = 0, 1, 4$ and 7 wt.%)

Composition	U-6Zr	U-6Zr-1Nb	U-6Zr-4Nb	U-6Zr-7Nb
Temperature, T (K)	300 K	300 K	300 K	300 K
Rad. wavelength, λ (Å)	1.5406	1.5406	1.5406	1.5406
Sample form	Ingot	Ingot	Ingot	Ingot
Phase 1 $\alpha - U$				
Crystal system	Orthorhombic	Orthorhombic	Orthorhombic	–
Space group	C m c m (63)	C m c m (63)	C m c m (63)	–
Mass fraction (wt.%)	100	100	9.57	–
Unit cell a (Å)	2.8615(1)	2.8668(4)	2.9268(3)	–
dimension b (Å)	5.8720(3)	5.8503(9)	5.7458(7)	–
c (Å)	4.9740(2)	4.9734(6)	4.9645(3)	–
Unit-cell volume, V (Å ³)	83.57(1)	83.41(3)	83.48(2)	–
Atomic density, (g·cm ⁻³)	18.799	18.436	18.063	–
Phase 2 $\gamma - U$				
Crystal system	–	–	Cubic	Cubic
Space group	–	–	I m $\bar{3}$ m (229)	I m $\bar{3}$ m (229)
Mass fraction (wt.%)	–	–	88.81	100
Unit cell a (Å)	–	–	3.5036(2)	3.4961(1)
dimension b (Å)	–	–	3.5036(2)	3.4961(1)
c (Å)	–	–	3.5036(2)	3.4961(1)
Unit-cell volume, V (Å ³)	–	–	43.01(9)	42.73(6)
Atomic density, (g·cm ⁻³)	–	–	17.709	17.755
Agreement Indices				
GoF, χ^2	1.074	1.261	1.378	1.109
R_{wp}	0.0887	0.1168	0.1151	0.1179
R_{exp}	0.0919	0.1311	0.1351	0.1241
R_p	0.0690	0.0935	0.0888	0.0920

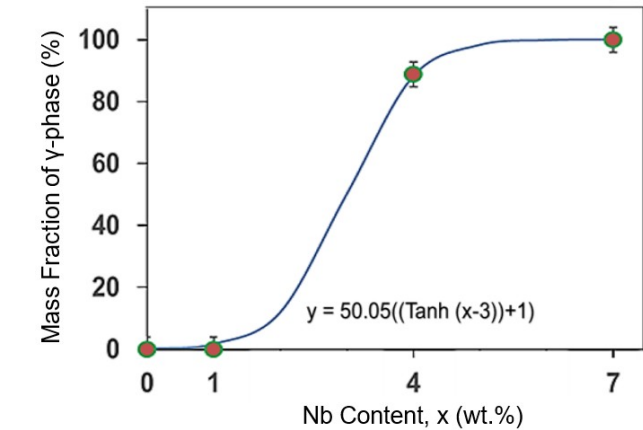


Figure 3. Mass Fraction of γ -U Phase of U-6Zr- x Nb Alloys ($x = 0, 1, 4$ and 7 wt.%)

the presence of multiple phases. Meanwhile, Figure 2(d) shows the XRD pattern refinement for the alloy composition $x = 7$ wt.% (U-6Zr-7Nb), which displays Bragg diffraction peaks corresponding to a single γ -U phase. The BSE–SEM image of

the $x = 7$ wt.% alloy confirms this result, as the surface morphology shows no observable contrast differences, appearing uniformly light gray across the sample surface. According to the refinement results obtained using the GSAS software, the incorporation of Nb atoms, the presence of Nb atoms in this structure can alter all the structural parameters of the sample. The refinement results of U-6Zr- x Nb alloys ($x = 0, 1, 4$, and 7 wt%) XRD pattern show R_{wp} values ranging from 0.0887 to 0.1179 and χ^2 values from 1.074 to 1.378. This results show that the X-ray diffraction pattern has a very good fitting quality according to the fit criteria (R_{wp}) and goodness of fit (χ^2) (Slater 1964). R_{wp} is the ratio of the weight difference between the observed and calculated XRD patterns (the ideal value of $R_{wp} < 10\%$). Meanwhile χ^2 (chi-squared) is the ratio of the observed XRD pattern that is comparable to the expectation (ideal value $\chi^2 < 1.3$). From refinement result, it can be seen that for the alloy composition $x = 0$ to $x = 4$ wt.%, the lattice parameter a increases, while the lattice parameters b and c appear to decrease. Likewise, the unit cell volume and atomic density appear to decrease with increasing Nb atomic content in the structure. This indicates that there has been an increase and decrease in interatomic bonds. It is known that

Table 2. Crystal Structure Parameters, Fit Criteria (R_{wp}), and Goodness of Fit (χ^2) on U-6Zr- x Nb Alloys ($x = 0, 1, 4$, and 7 wt.%) After Corrosion in Acidic (pH = 5.61), and Alkaline (pH = 7.28 Environments)

pH	pH = 5.61				pH = 7.28			
Composition (x)	0Nb	1Nb	4Nb	7Nb	0Nb	1Nb	4Nb	7Nb
Temperature, T (K)	300 K							
Wavelength, λ (Å)	1.5406							
Sample form	Ingot							
Phase 1	α - U							
Crystal system	Orthorhombic							
Space group	C m c m (63)							
Mass frac. (wt.%)	89.65	90.19	3.13	–	90.51	92.06	2.72	–
Unit cell a (Å)	2.8624(2)	2.8655(3)	2.8842(1)	–	2.8609(1)	2.8659(2)	2.8809(2)	–
dimension b (Å)	5.8692(4)	5.8509(7)	5.8178(3)	–	5.8715(3)	5.8555(5)	5.8141(5)	–
c (Å)	4.9757(3)	4.9662(5)	4.9561(2)	–	4.9733(2)	4.9857(3)	4.9854(3)	–
V (Å ³)	83.59(1)	83.26(2)	83.16(8)	–	83.54(1)	83.66(1)	83.50(8)	–
ρ (g·cm ^{–3})	18.695	18.433	18.023	–	18.705	18.418	18.067	–
Phase 2	γ - U							
Crystal system	Cubic							
Space group	I m $\bar{3}$ m (229)							
Mass frac. (wt.%)	–	–	96.61	100	–	–	96.41	100
Unit cell a (Å)	–	–	3.5363(4)	3.4801(5)	–	–	3.4725(3)	3.4937(6)
dimension b (Å)	–	–	3.5363(4)	3.4801(5)	–	–	3.4725(3)	3.4961(1)
c (Å)	–	–	3.5363(4)	3.4801(5)	–	–	3.4725(3)	3.4961(1)
V (Å ³)	–	–	44.22(1)	42.15(1)	–	–	41.87(1)	42.73(6)
ρ (g·cm ^{–3})	–	–	16.869	17.257	–	–	16.869	17.047
Phase 3	UO ₂							
Crystal system	Cubic							
Space group	F m $\bar{3}$ m (225)							
Mass frac. (wt.%)	10.35	9.81	0.26	–	9.49	7.94	0.87	–
Unit cell a (Å)	5.4485(3)	5.4485(1)	5.4452(7)	–	5.4400(5)	5.4257(7)	5.3560(1)	–
dimension b (Å)	5.4485(3)	5.4485(1)	5.4452(7)	–	5.4400(5)	5.4257(7)	5.3560(1)	–
c (Å)	5.4485(3)	5.4485(1)	5.4452(7)	–	5.4400(5)	5.4257(7)	5.3560(1)	–
V (Å ³)	161.75(2)	161.75(1)	161.45(6)	–	160.99(4)	159.72(6)	153.64(1)	–
ρ (g·cm ^{–3})	11.088	11.087	11.090	–	11.140	11.242	11.230	–
Agreement Indices								
GoF, χ^2	1.085	1.186	1.245	1.182	1.071	1.067	1.228	1.088
R_{wp}	0.0739	0.1174	0.1016	0.1154	0.0863	0.0835	0.1129	0.0756
R_{exp}	0.0770	0.1278	0.1134	0.1255	0.0893	0.0862	0.1251	0.0789
R_p	0.0575	0.0931	0.0801	0.0908	0.0668	0.0647	0.0877	0.0502

the atomic radii of U, Zr, and Nb are 175 pm, 155 pm, and 145 pm (Vollath, 1986; Valance et al., 2020), respectively. It appears that the atomic radius of Zr and Nb is much smaller compared to the atomic radius of U. In other words, the atomic radius of U > Zr > Nb. Thus, with the increasing number of Nb atoms, the total atomic radius will be smaller, so that it is very possible that with the increase in lattice parameter a , and the decrease in lattice parameters b and c , the structure can transform from an orthorhombic system to a cubic system. The phase transformation from α -U to γ -U from the results of this XRD pattern refinement can be seen in Figure 3 which shows the growth of the γ -U phase along with the increasing Nb content in the sample.

According to the assumption that the single phase of α -U occurs at composition $x = 0$, while the single phase of γ -U occurs at composition $x = 7$ wt.%, so the growth of the γ -U

phase follows the composition $x = 7$ wt.%. According to the mathematical calculations by fitting the mass fraction of γ -U phase growth from the refinement results of the XRD pattern shows that the growth of the γ -U phase follows a hyperbolic tangent pattern, where according to the calculation results, the mass fractions of the α -U and γ -U phases at alloy compositions $x = 0, 1, 2, 3, 4, 5, 6$, and 7 wt.% are 100: 0, 98.20: 1.80, 88.17: 11.93, 49.95: 50.05, 11.83: 88.17, 1.70: 98.30, 0.15: 99.85 and 0: 100%, respectively. This means that for Nb content of 1 wt.% ($x = 1$), Nb is able to form γ -U phase with composition of U-6Zr-7Nb of 1.80% and α -U phase with composition of U-6Zr of 98.20%. For Nb content of 2 wt.% ($x = 2$), Nb is able to form γ -U phase with composition of U-6Zr-7Nb of 11.93% and α -U phase (U-6Zr) of 88.17%, and so on until for Nb content of 6 wt.% ($x = 6$), Nb is able to form γ -U phase with composition of U-6Zr-7Nb of 99.85%. Thus, Nb with certain

Table 3. Results of Calculations of I_{corr} , E_{corr} , and Corrosion Rate from the Results of Tafel Curve Measurements on U-6Zr-xNb Alloys ($x = 0, 1, 4$ and 7 wt.%)

pH	Nb (wt.%)	I_{corr} ($\mu A/cm^2$)	E_{corr} (mV)	CR (mpy)
5.61	0	0.8074	-0.4557	0.2389
	1	0.7754	-0.3996	0.2333
	4	0.2361	-0.2102	0.0744
	7	0.1323	-0.1785	0.0416
7.26	0	2.9717	-0.4768	0.8795
	1	1.2989	-0.2956	0.3908
	4	0.5924	-0.2455	0.1868
	7	0.46153	-0.1927	0.14527

composition limits is able to stabilize the γ -U phase. According to Alexander Landa who has succeeded in making a model related to this stability using the CALPHAD method or a first-principles approach such as Density Functional Theory (DFT). An integrated approach between DFT and CALPHAD is used to map the Gibbs free energy and heat of formation of this alloy. The γ -U phase is stable at high temperatures (above 1048 K to a melting point of 1405 K). Thermodynamic calculations show that Niobium (Nb) substitution stabilizes the γ -U phase with a body-centered cubic (BCC) structure at high temperatures and maintains its metastable phase at room temperature by suppressing the martensitic transformation (Landa et al., 2020; Duong et al., 2016; Liu et al., 2012). Thus, Nb with certain composition limits is able to stabilize the γ -U phase. Uranium metal has high density but low mechanical properties and high anisotropy unless combined with other elements (Ghoshal et al., 2013).

The addition of alloying elements such as Nb, Zr, and Mo increases the stability of γ -U phase, which has better mechanical properties and physical symmetry (Ghoshal et al., 2013; Bacon et al., 2019). U-Zr alloys show good dimensional stability during thermal cycling and retain the γ -U phase over a wider temperature range. However, U-Zr-Nb alloys are more resistant to swelling than either U-Nb or U-Zr alloys (Martin et al., 2016).

3.2 Condition After Corrosion in Acidic and Alkaline Environment

Figure 4 presents the XRD patterns of U-6Zr-xNb alloys ($x = 0, 1, 4$, and 7 wt.%) after corrosion in acidic (pH 5.61) and alkaline (pH 7.28) environments. The XRD results reveal a clear effect of Nb addition on phase evolution with increasing Nb-content. The U-6Zr and U-6Zr-1Nb alloys were dominated by the α -U phase with an orthorhombic crystal structure, while a secondary UO_2 phase was also detected after corrosion. In contrast, the U-6Zr-4Nb alloy contained a mixture of α -U, γ -U, and UO_2 phases, whereas U-6Zr-7Nb exhibited a single γ -U phase with no detectable UO_2 phase. These observations indicate that Nb addition promotes γ -U phase stabilization and reduces corrosion-product formation.

The XRD observations were further supported by the EDS

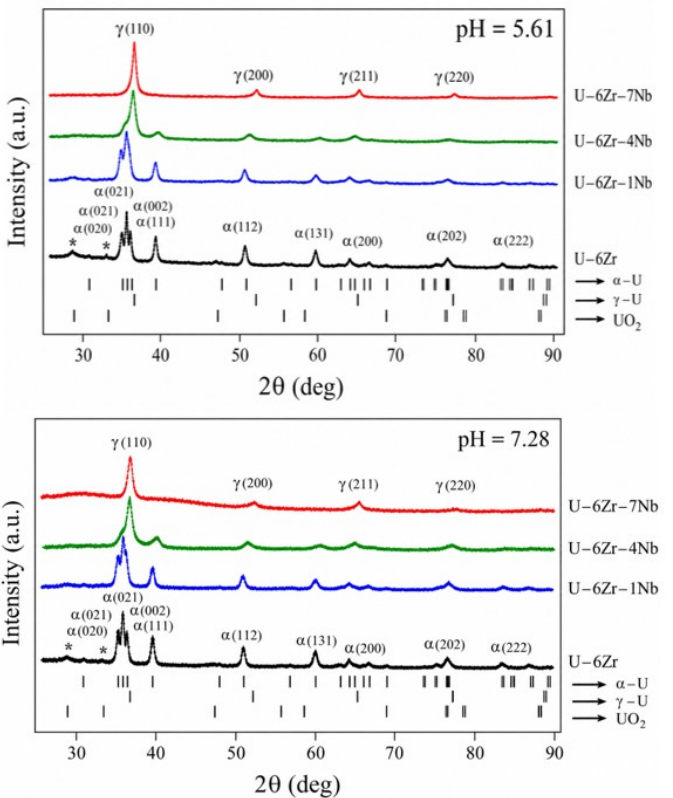
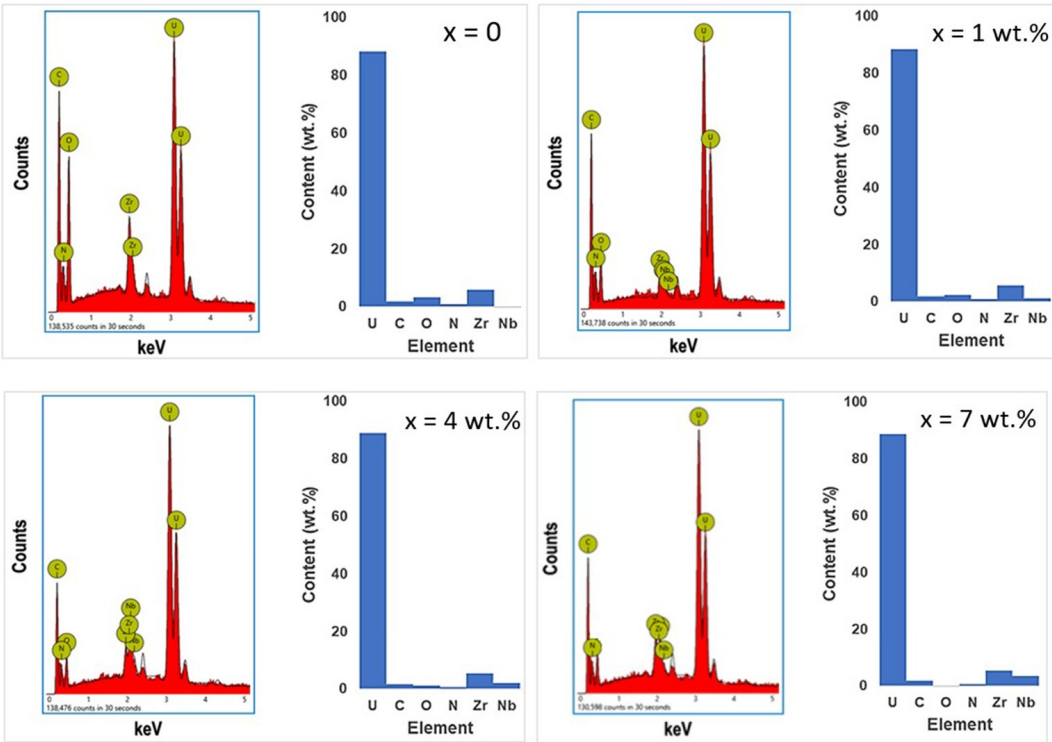
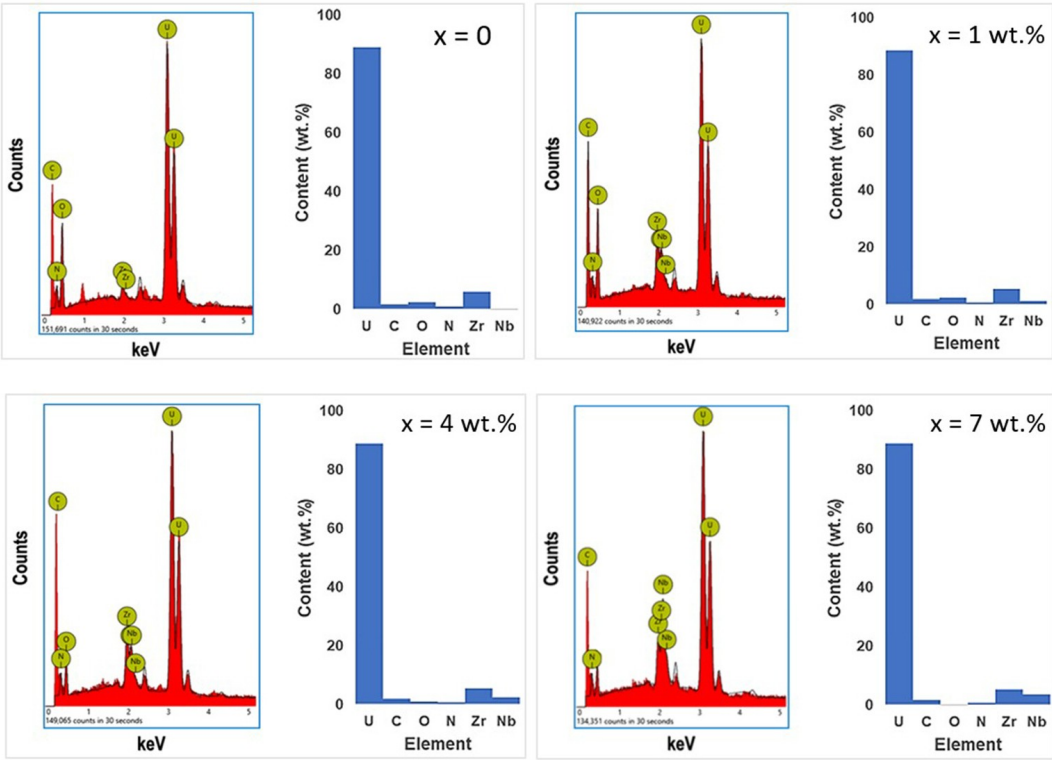


Figure 4. XRD Patterns of U-6Zr-xNb Alloys ($x = 0, 1, 4$, and 7 wt.%) After Corrosion in Acidic (pH 5.61) and Alkaline Environments (pH 7.28)

results shown in Figure 5. Oxygen concentrations decreased from approximately 20 wt.% in U-6Zr to 16 wt.% in U-6Zr-1Nb and 2 wt.% in U-6Zr-4Nb. No oxygen was detected in the U-6Zr-7Nb alloy, suggesting either the absence of oxide formation or the presence of oxide levels below the EDS detection limit. The agreement between XRD and EDS indicates a progressive reduction in oxidation with increasing Nb content. The presence of carbon and nitrogen detected by EDS is attributed to sample preparation procedures, specifically carbon coating and nitric-acid-based etching.



(a)



(b)

Figure 5. Results of Elemental Analysis of U-6Zr-xNb Alloys (x = 0, 1, 4, and 7 wt.%) After Corrosion in Acidic Environment (pH = 5.61) (a); and Alkaline Environments (pH = 7.28) (b)

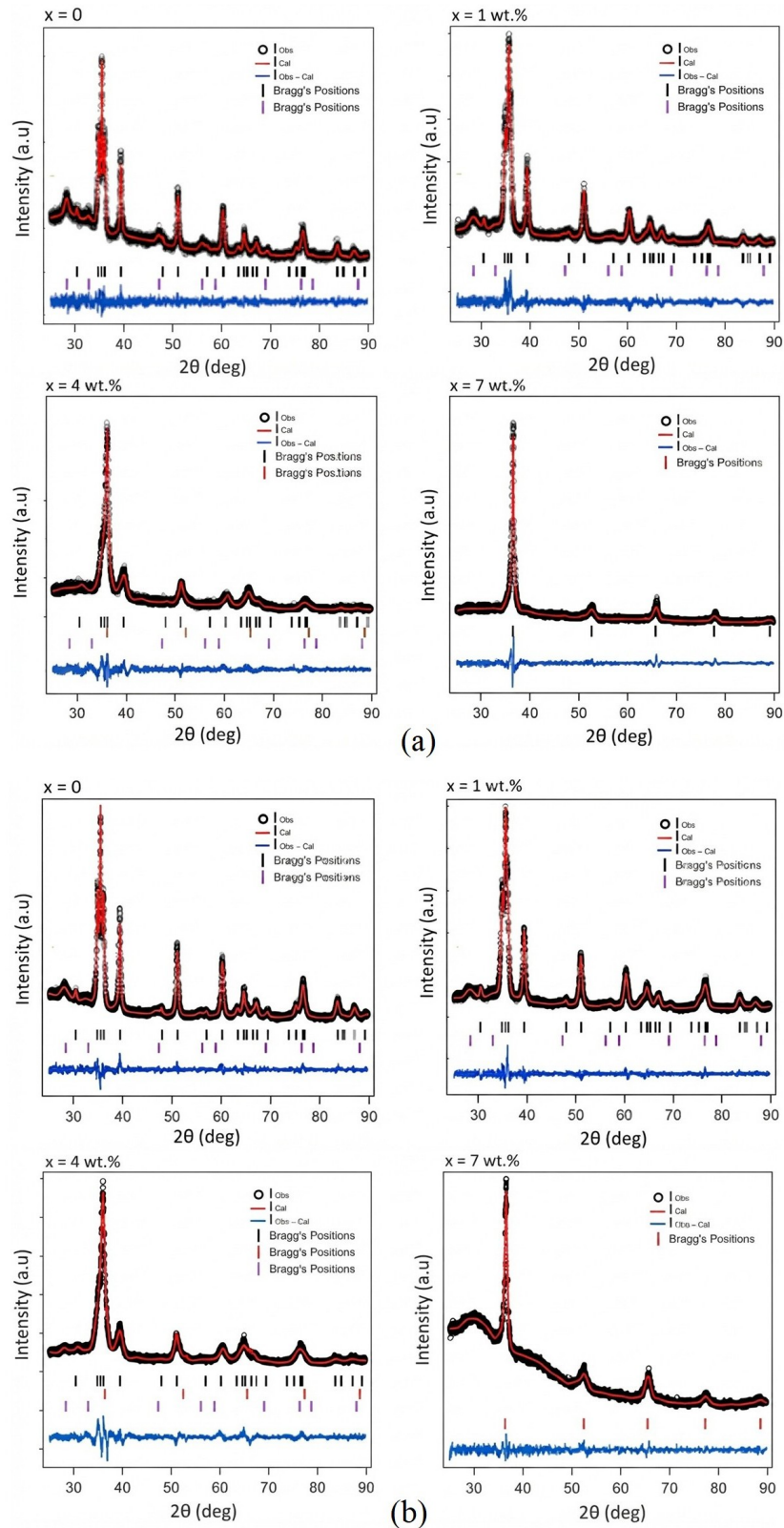


Figure 6. Refinement Result of X-ray Diffraction Pattern of U-6Zr-xNb Alloys After Corrosion in Acidic Environment (pH = 5.61) (a); and Alkaline Environments (pH = 7.28) (b)

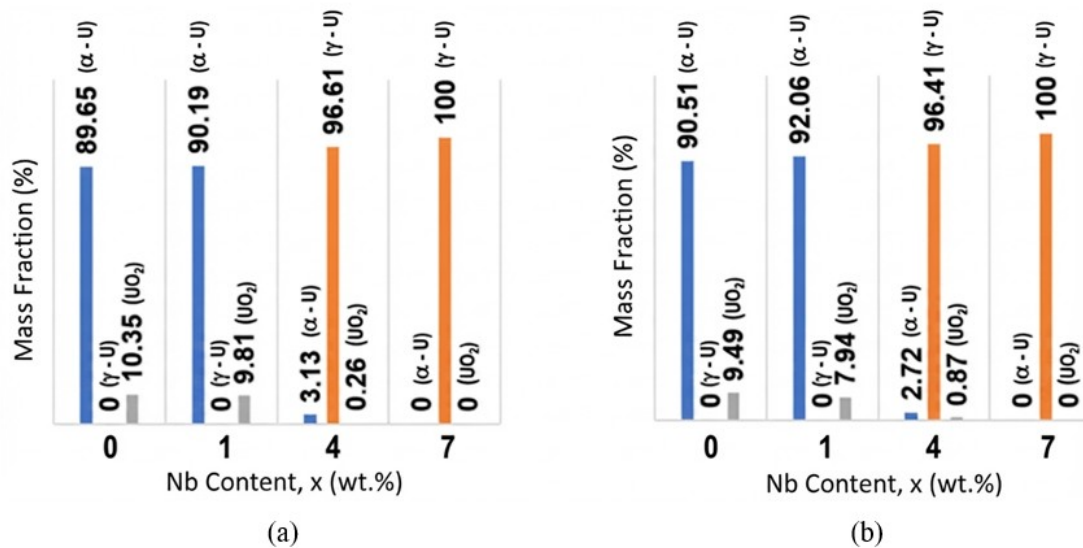


Figure 7. Mass Fraction of Corrosion Products of U-6Zr-xNb Alloys (x = 0, 1, 4, and 7 wt.%) in Acidic Environment (pH = 5.61) (a); and Alkaline Environments (pH = 7.28) (b)

To quantify the effect of Nb on corrosion-product formation, Rietveld refinement was performed using the GSAS software, and the results are presented in Figure 6 and Table 2. The refinement quality was satisfactory for all samples, with average Rwp and χ^2 values of 0.102 and 1.175 under acidic conditions, and 0.089 and 1.114 under alkaline conditions, respectively. The refinement enabled quantitative determination of the α -U, γ -U, and UO_2 phase fractions, as summarized in Figure 7.

Under acidic conditions, the UO_2 fraction decreased from 10.35 wt.% in U-6Zr to 9.81 wt.% in U-6Zr-1Nb and further to only 0.26 wt.% in U-6Zr-4Nb. No detectable UO_2 phase was observed in U-6Zr-7Nb. A similar trend was obtained under alkaline conditions, where the highest UO_2 fraction was observed in the U-6Zr (9.49 wt.%) and gradually decreased with increasing Nb addition. The consistency of this trend in both corrosion environments demonstrates that Nb has a strong influence on suppressing uranium oxidation regardless of the solution chemistry.

An important observation is that the reduction in UO_2 formation occurred simultaneously with the gradual transformation of α -U into γ -U phases. The U-6Zr-4Nb alloy, which contained both α -U and γ -U phases, exhibited only a very small UO_2 fraction, whereas the fully γ -U U-6Zr-7Nb alloy showed no detectable UO_2 phase. This correlation suggests a strong relationship between γ -U stabilization and resistance to oxidation.

Overall, the combined XRD, EDS, and GSAS refinement results demonstrate that increasing Nb content significantly reduces oxide formation and promotes γ -U phase stabilization. The progressive decrease in oxygen concentration and UO_2 fraction provides direct evidence that Nb suppresses uranium

oxidation during corrosion. These findings explain the improved corrosion performance observed in the Nb-containing alloys and indicate that both phase stability and oxide suppression play important roles in controlling the corrosion behavior of the U-6Zr-xNb alloy system.

3.3 Corrosion Resistance in Acidic and Alkaline Environment

Corrosion rate measurements in this study were performed using electrochemical methods, specifically by determining the polarization resistance. Figure 8 and Table 3 summarize the electrochemical corrosion behavior, including potentiodynamic curves and the relationships between Nb content and I_{corr} , E_{corr} , and CR of U-6Zr-xNb alloys (x = 0, 1, 4, and 7 wt.%), in acidic (pH 5.61) and alkaline (pH 7.28) environments. A clear influence of Nb addition on the corrosion response was observed in both media. As the Nb content increased from 0 to 7 wt.%, the corrosion current density (I_{corr}) decreased from 0.8074 to 0.1323 $\mu\text{A cm}^{-2}$ in the acidic solution, accompanied by a reduction in corrosion rate from 0.2389 to 0.0416 mpy. Simultaneously, the corrosion potential (E_{corr}) shifted from -0.4557 to -0.1785 V, indicating a lower tendency for electrochemical degradation. Similar behavior was observed in the alkaline environment, where I_{corr} decreased from 2.9717 to 0.4615 $\mu\text{A cm}^{-2}$ and the corrosion rate decreased from 0.8795 to 0.1453 mpy with increasing Nb content.

The electrochemical results are consistent with the structural characterization described in the previous section. The alloys exhibiting lower UO_2 fractions also showed lower I_{corr} values and lower corrosion rates. In particular, the U-6Zr-7Nb alloy exhibited the lowest corrosion rate and no detectable UO_2 phase by XRD, suggesting that suppression of uranium oxida-

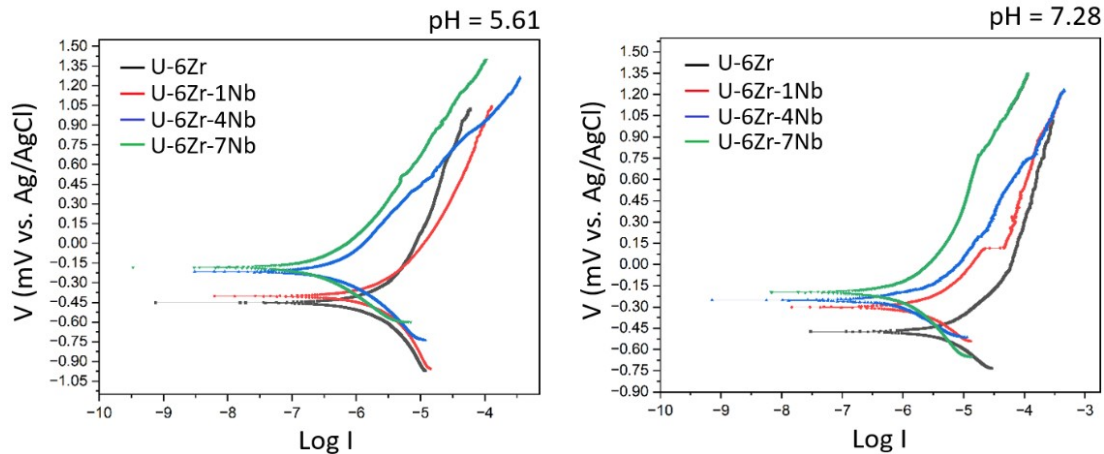


Figure 8. Potentiodynamic Polarization Curve of Corrosion Rate Tests on U-6Zr-xNb Alloys (x = 0, 1, 4 and 7 wt.%) in Acidic Environment (pH = 5.61) and Alkaline Environments (pH = 7.28)

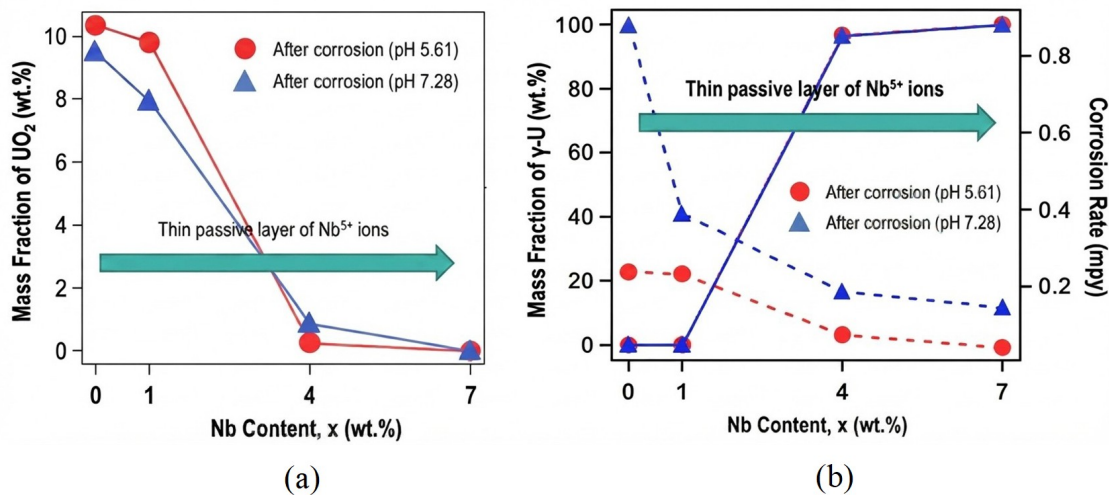


Figure 9. Reduction of Corrosion Products Due to the Delamination Process of Nb^{5+} Ions on The Uranium Metal Surface, (a) Mass Fraction of UO_2 vs Nb Content, (b) Mass Fraction of γ -U (Solid Lines) and Corrosion Rate (Dashed Lines) vs Nb Content

tion is a major factor contributing to the improved corrosion resistance. The strong correlation between oxide formation and electrochemical performance indicates that the corrosion behavior of the investigated alloys is closely governed by the extent of oxidation occurring during exposure to the corrosive media.

Another important observation is the strong relationship between phase constitution and corrosion resistance. The U-6Zr and U-6Zr-1Nb alloys, which remained dominated by the α -U phase, exhibited relatively high corrosion rates and significant UO_2 formation. In contrast, the emergence of the γ -U phase in U-6Zr-4Nb coincided with a substantial reduction in both UO_2 fraction and corrosion rate. Further Nb addition to 7 wt.% resulted in a fully stabilized γ -U phase and the lowest corrosion rate among all investigated alloys. These observations suggest that γ -U stabilization plays an important role

in reducing oxidation susceptibility and improving corrosion resistance.

Figure 9 shows that the addition of Nb from 1 wt.% to 4 wt.% causes a sharp decrease in the corrosion rate, which is the main phase in the formation of the passive layer of Nb_2O_5 and ZrO_2 , which is highly protective against corrosion attacks (Gomozov et al., 1974; Kelly et al., 2001; Wu et al., 2016). The Nb_2O_5 layer acts as a corrosion inhibitor due to its several superior properties, especially its stability and corrosion resistance. The protection mechanism involves the formation of a highly stable passive layer on the metal surface, preventing direct contact between the base metal and the corrosive environment. The Nb_2O_5 layer is naturally occurring and can create a dense and non-reactive passive layer. This layer acts as a physical barrier, effectively preventing oxygen and other aggressive ions from reaching the metal surface, thus halting the

corrosion process. Nb_2O_5 is a very thermodynamically stable oxide, even under extreme conditions. This stability ensures that the protective layer does not easily decompose or dissolve in various chemical environments. Nb_2O_5 has good dielectric properties, which means it does not conduct electricity. This can help prevent electrochemical corrosion, where electrons flow from the anode to the cathode. The Nb_2O_5 dielectric layer can disrupt the electrochemical circuitry necessary for corrosion. The Nb_2O_5 layer has good mechanical strength and strong adhesion to metal substrates. This makes it resistant to physical damage and friction, which can open cracks in the protective layer. Although such surface films were not directly detected by XRD, they may exist in quantities below the detection limit or in partially amorphous forms. Their presence could reduce oxygen transport to the alloy surface and suppress further oxide growth, thereby decreasing electrochemical activity. This interpretation is supported by the observed reduction in oxygen concentration, the lower UO_2 phase fraction, and the progressive decrease in I_{corr} with increasing Nb content.

The present findings are consistent with previous studies reporting enhanced corrosion resistance in Nb-containing uranium alloys (Masrukan et al., 2024; Weirick, 1975; Masrukan et al., 2025; Sermsrithong et al., 2022). However, the current work further demonstrates a direct correlation between γ -U phase stabilization, reduced UO_2 formation, and improved electrochemical performance in U-6Zr-xNb alloys.

A limitation of the present study is the absence of direct surface characterization techniques such as X-ray photoelectron spectroscopy (XPS) and transmission electron microscopy (TEM). Therefore, the formation of Nb-rich passive films remains a proposed interpretation rather than a directly verified observation. Nevertheless, the experimental data consistently demonstrate that increasing Nb content improves the corrosion resistance of U-6Zr-xNb alloys. Future studies employing XPS and TEM are recommended to further clarify the corrosion-protection mechanisms.

4. CONCLUSIONS

U-6Zr-xNb alloys ($x = 0, 1, 4$, and 7 wt.%) were successfully fabricated by conventional arc-melting method and evaluated for corrosion in acidic ($\text{pH} = 5.61$) and alkaline ($\text{pH} = 7.28$) environments. Before corrosion, the $x = 0$ (U-6Zr) and $x = 1$ wt.% (U-6Zr-1Nb) alloys exhibited a single α -U phase with an orthorhombic structure (space group $Cmcm$, no. 63). At $x = 4$ wt.% (U-6Zr-4Nb), two phases were formed, α -U and γ -U. At $x = 7$ wt.% (U-6Zr-7Nb), a single γ -U phase with a cubic (bcc) structure (space group $Im\bar{3}m$, no. 229) was identified. After corrosion, UO_2 was detected in $x = 0, 1$, and 4 wt.% alloys, while no UO_2 was detected in $x = 7$ wt.% alloy. The UO_2 mass fraction decreased with increasing Nb content. In addition, Nb promoted the transformation of α -U into γ -U phase. Electrochemical corrosion tests in both acidic and alkaline environments demonstrated that the corrosion rate decreased with increasing Nb content. The improved corrosion resistance is attributed to the combination of γ -U phase stabilization and

formation of a thin Nb-rich passive film, likely to consist of a thin passive Nb_2O_5 layer on the uranium alloy surface. Although very thin, this protective layer significantly suppresses corrosion in both environments. Overall, Nb addition promotes γ -U phase stability and enhances corrosion resistance, demonstrating the potential of U-6Zr-xNb alloys as promising candidates for advanced nuclear reactor fuel applications.

5. ACKNOWLEDGEMENT

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