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CORROSION BEHAVIOR OF Zr-10Mo ALLOYS IN NIOBIUM-DOPED LACTATE RINGER'S SOLUTION

Vita Yuliana Prastika[1], Pradoto Ambardi[1] and Djoko Hadi Prajitno[1,2]
[1,2]*Teknik Metalurgi - Universitas Jenderal Achmad Yani*
Jl. Terusan Jenderal Gatot Subroto, Bandung, 40285
[2]*Pusat Sains dan Teknologi Nuklir Terapan - BATAN*
Jl. Tamansari 71, Bandung 40132
E-mail: vitaaprastika@gmail.com

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ABSTRACT

CORROSION BEHAVIOR OF Zr-10Mo ALLOYS IN NIOBIUM-DOPED LACTATE RINGER'S SOLUTION. This research discusses the corrosion behavior of Zr-10Mo alloys in Niobium-doped lactate ringer's solution. The addition of Niobium of 1%, 3% and 5% on Zr-10Mo alloys aims to study the effect of adding Niobium to the hardness, microstructure and corrosion resistance of Zr-Mo alloys. The alloys are made by melting the materials of Zr, Mo and Nb with a certain percentage of weight using single arc melting furnace in Argon atmosphere with high purity and Tungsten eletrodes. The alloys (as cast) are subjected to hardness testing using C-scale rockwell, microstructure using optical microscopy, compounds and phases in the alloys (as cast) using XRD and corrosion testing using polarization method in lactate ringers' solution. The hardness value increases by the addition of Niobium, Zr-10Mo-1Nb alloy of 34 HRC, Zr-10Mo-3Nb alloy of 38 HRC and Zr-10Mo-5Nb alloy of 41.5 HRC. The basketweave-shaped microstructure with the addition of Niobium creates relatively smaller grain size. The value of corrosion rate decrases by the addition of Niobium at the immersion times of 0 minute, 60 minutes and 120 minutes, Zr-10Mo-1Nb alloy of 4.208 mpy, Zr-10Mo-3Nb alloy of 3.538 mpy and Zr-10Mo-5Nb alloy of 2.813 mpy, included in the excellent category (1-5 mpy).

Keywords: Niobium, Zr-10Mo Alloy, Hardness, Corrosion Resistance, Lactate Ringer

ABSTRAK

PERILAKU KOROSI PADUAN Zr-10Mo DALAM LARUTAN RINGER LAKTAT YANG DI DOPING NIOBIUM. Dalam penelitian ini dibahas mengenai perilaku korosi paduan Zr-10Mo dalam larutan ringer laktat yang di doping Niobium. Penambahan unsur Niobium antara 1%, 3% dan 5% pada paduan Zr-10Mo bertujuan untuk mempelajari pengaruh penambahan unsur Niobium terhadap kekerasan, struktur mikro dan ketahanan korosi paduan Zr-Mo. Paduan dibuat dengan cara meleburkan material Zr, Mo dan Nb dengan persentase berat tertentu menggunakan *single arc melting furnace* dalam atmosfer Argon dengan kemurnian tinggi dan elektroda Tungsten. Paduan (*as cast*) dilakukan pengujian kekerasan menggunakan *rockwell* skala C, struktur mikro menggunakan mikroskop optik, senyawa dan fasa pada paduan (*as cast*) menggunakan XRD serta pengujian korosi dengan metode polarisasi dalam larutan ringer laktat. Nilai kekerasan meningkat dengan penambahan unsur Niobium, paduan Zr-10Mo-1Nb sebesar 34 HRC, paduan Zr-10Mo-3Nb sebesar 38 HRC dan paduan Zr-10Mo-5Nb sebesar 41,5 HRC. Struktur mikro berbentuk *basketweave* dengan penambahan unsur Niobium ukuran butir relatif lebih kecil. Nilai laju korosi menurun dengan penambahan unsur Niobium dalam waktu imersi 0 menit, 60 menit dan 120 menit, paduan Zr-10Mo-1Nb sebesar 4,208 mpy, paduan Zr-10Mo-3Nb sebesar 3,538 mpy dan paduan Zr-10Mo-5Nb sebesar 2,813 mpy, termasuk kategori *excellent* (1-5 mpy).

Kata kunci: Niobium, Paduan Zr-10Mo, Kekerasan, Ketahanan Korosi, Ringer Laktat

INTRODUCTION

Zirconium is mainly used as a structural material in nuclear applications due to its low absorption of neutrons. Recently, zirconium-based materials have gained attention as structural materials for aerospace, chemical and biomedical applications because of their resistance against corrosion and wear [1]. The combination of acceptable strength, high corrosion resistance and good biocompatibility has made zirconium alloys suitable for biomaterials for orthopedic implants. Zirconium has been known as non-toxic and non-allergic [2]. Alloy-based Zr has recently been used as orthopedic biomaterials [3-5], such as artificial knee joints [3]. However, the mechanical reliability of Zr is not sufficient to be used as structural biomaterials. Using Mo as Zr alloy element because it is an effective reinforcement element for Zr [6,7] and this shows low cytotoxicity [6,8] and low magnetic weakness [6]. Molybdenum (Mo) or Niobium (Nb) elements have been added to the Zr matrix to develop binary alloys and show a strengthening effect, low magnetic susceptibility and or low modulus [7,9-11].

Zr and its alloys are also known as bioactive metallic biomaterials which are very good because they can form apatite layers like bone on the surface in the body of living creatures. In general, Zr exhibits high mechanical strength, high toughness against fracture and good corrosion resistance, so that it can act as a structural material. As a result, Zr alloy is an ideal material developed for the replacement of biological hard tissues. However, to date, reports of the application of Zr alloys in the replacement of biological hard tissues are still very rare. The microstructure and magnetic susceptibility of Zr-Mo alloys as cast have been studied and it was found that Zr alloys are useful for medical devices that use Magnetic Resonance Imaging (MRI) [3, 12].

This study aims for the behavior of corrosion resistance of alloy biomaterials Zr-10Mo-xNb (x = 1%, 3% and 5%) as-cast condition in the ringer's lactate solution by polarization method, as well as for developing orthopedic biomaterials such as artificial knee joints.

EXPERIMENTAL METHOD

Materials and Instruments

This study uses materials consisting of Zirconium sponge with 99% purity (Aldrich Chemical), Molibdenum rod with 99.8% purity (Goodfellow) and Niobium with 99.9% purity (ESPI Metals), and ringer's lactate solution (PT. Widatra Bhakti).

The equipment used in this study consists of Mettler Toledo AL204 Analytical Scale to weigh the materials according to the composition in Table 1, single arc melting furnace of PSTNT BATAN to melt the materials into alloys, hardness rockwell scale-C Krisbow KW0600121 to obtain the hardness value of the alloy, Optical microscope Eclipse LV150 to analyze the microstructure of alloy, X-Ray Diffraction (XRD) PANalytical X'Pert PRO PW3040/x0 to analyze compounds and phases in alloy, "Gamry Framework" software to study tafel polarization and corrosion rate of corrosion test results by the polarization method referring to ASTM G59 standard, corrosion testing using the polarization method with counter electrode in the form of platinum wires, reference electrode in the form of reference electrode Ag-AgCl Fisher ScientificTMAccumetTM, and work electrodes in the form of alloy to be tested.

Table 1. Composition of alloy (in %wt)

Alloy	Zr		Mo		Nb	
(as cast)	%	g	%	g	%	g
Zr-10Mo-1Nb	89	13.35	10	1.5	1	0.15
Zr-10Mo-3Nb	87	13.05	10	1.5	3	0.45
Zr-10Mo-5Nb	85	12.75	10	1.5	5	0.75

Method and Procedure

The materials are weighed with the composition in Table 1 with a total weight of 15 grams per alloy, then the melting process is conducted using single arc melting furnace, the melting process uses Tungsten electrode (W) given an electric current with a voltage of 230 V and current of ±110 A so as to produce an arc that will melt the materials in a copper crucible hearth which is flowed with water as a cooler, the process is carried out in the atmosphere of Argon UHP 99.99% gas with high purity which aims to protect the melted materials from oxidizing. The melting process of each alloy is carried out 4 times to get homogeneous alloy results, melting results such as buttons. Each alloy (as cast) is prepared to form a cylindrical shape of ±15 mm diameter and ±4 mm thick.

Each alloy undergoes initial characterization in the form of hardness test using hardness rockwell scale-C referring to ASTM E18 with indentation in the form of a diamond cone, minor load of 10 kg, major load of 140 kg and total load of 150 kg. Metallographic examination is conducted at the upper alloy surface, each alloy is prepared first with the sanding process using 60-2000 mesh sandpaper, polishing process uses a velvet cloth and ZrO_2 polishing powder as the abrasive particle and the etching process uses etching solution with the composition of 5% H_2SO_4, 10% HF, 30% HNO_3 and 55% H_2O, the etching is conducted by immersion. The initial XRD testing is conducted on the upper alloy surface, after obtaining initial characterization, each alloy is subjected to corrosion testing using the polarization method referring to ASTM G59 standard in ringer's lactate solution. This testing uses three types of electrodes namely reference electrode (Ag-AgCl), counter electrodes (platinum wire) and work electrode (alloy to be tested), the three electrodes are connected to the potentiostat and the computer with "Gamry Framework" software. Polarization curves are obtained in the potential range of -350mV (initial) to +350mV (final). Corrosion testing in ringer's lactate solution is carried out by varying the immersion time of each alloy i.e. 0 minute, 60 minutes and 120 minutes.

After corrosion testing by the polarization method, the alloy is subjected to final characterization by XRD testing.

Ringer's lactate solution is used as the corrotion medium as it resembles ion, the concentration and pH of human body, where the composition of ringer's lactate solution can be seen in Table 2.

Table 2. Chemical composition of ringer's lactate solution.

Reagent	Composition(g/mL)
Sodium Lactate ($C_3H_5NaO_3$)	1.55 g/500 mL
Sodium Chloride (NaCl)	3.0 g/500 mL
Potassium Chloride (KCl)	0.15 g/500 mL
Calcium Chloride ($CaCl_2.2H_2O$)	0.1 g/500 mL
Water for injection	500 mL
Osmolaritas	274 mOsm/L
Na^+	130.3 mEq/L
K^+	4 mEq/L
Lactate (HCO_3^-)	27.7 mEq/L
Cl^-	109.4 mEq/L
Ca^{++}	2.7 mEq/L

RESULTS AND DISCUSSION

Based on the results of hardness testing using the C-scale rockwell method in Table 3, the hardness test results data on each alloy ara obtained.

Table 3. Effect of Nb on the hardness Vickers of Alloys.

Alloy (*as cast*)	Hardness Value *Rockwell* C (HRC)
Zr-10Mo-1Nb	34
Zr-10Mo-3Nb	38
Zr-10Mo-5Nb	41.5

The addition of Niobium element from 0 to 9% by Nb weight in Zirconium-based alloys can increase the hardness value and reduce the modulus young value [13]. The addition of Niobium element of 1%, 3% and 5% increases the alloy hardness of Zr-10Mo-1Nb by 34 HRC, Zr-10Mo-3Nb by 38 HRC and Zr-10Mo-5Nb by 41.5 HRC. This is due to the addition of Molybdenum element which has a strengthening effect on Zirconium, the radius of Zirconium atom (0.155 nm) and Molybdenum (0.139 nm) shows the substitutional solid solution and has too high electronegativity difference, the electronegativity of Zirconium atom (1.33) is smaller than Molybdenum (2.16) so that it will form a second phase of Mo2Zr which has a higher hardness than pure Zirconium and the β phase lattice parameter in the alloy decreases by increasing the Niobium element content so that internal stress occurs, and the radius of Niobium atom (0.145 nm) is smaller than Zirconium (0.155 nm) showing that substitutional solid solution occurs in the alloy.

With the increase in hardness, it is inversely proportional to the young modulus in zirconium alloys. The value of modulus young has decreased and is close to natural bone modulus young (10-30 GPa)[2], zirconium alloys show promising potential for new biomaterials from a relatively low modulus young point of view (closer to natural bone modulus young) compared to biomaterials alloy Ti-6Al-4V (107.2 GPa) and Stainless Steel 304 (173.3 GPa)[2], thus Zr-10Mo-xNb alloy (x= 1%, 3% and 5%) in this study is appropriate to be used as a new implant.

The results of metallographic examination with 200x magnification can be seen in Figure 1.

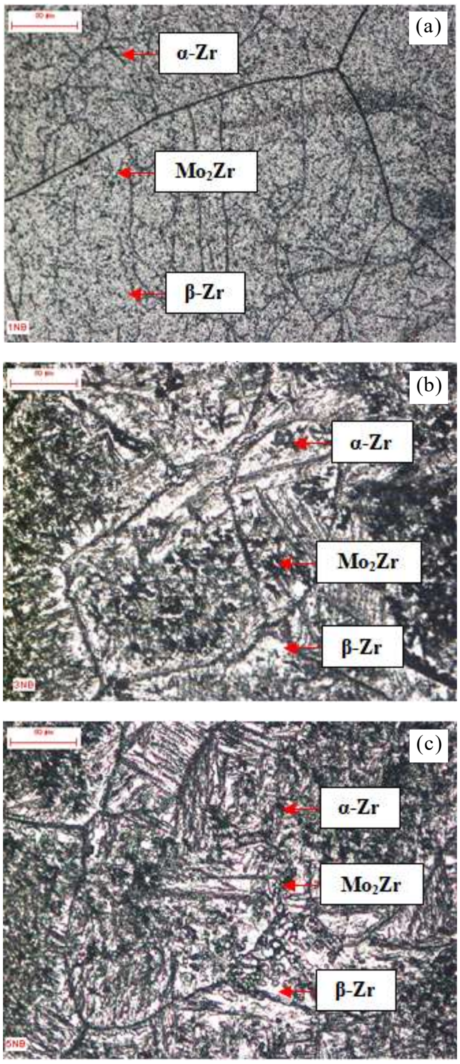

Figure 1. Microstructure of Zr-Mo-Nb alloys (a). Zr-10Mo-1Nb; (b). Zr-10Mo-3Nb and (c). Zr-10Mo-5Nb

The microstructure in basketweave-shaped Zr-10Mo, Zr-10Mo-1Nb, Zr-10Mo-3Nb and Zr-10Mo-5Nb alloys contain black dendritic α-Zr phase in the white β-Zr phase [14,16] and Mo2Zr intermetallic compound in the α-Zr phase [17]. α-Zr phase is black dendritic at the β-Zr grain boundary, α-Zr phase that grows from a number of different nucleation areas in β-Zr grain area where the form of those plates intersect and do not have time to extent forming the basketweave structure, this is because the insoluble Niobium element in β-Zr phase serves as the nucleation site of α-Zr phase during the colling time with the final basketweave structure [16]. In the Zr-10Mo-xNb alloy (x= 1%, 3% and 5%), the transformation mechanism of β-Zr phase into α-Zr phase + Mo2Zr intermetallic compound starts from the early cooling stage from the melting of Zirconium alloy into β-Zr phase, the next stage is the α-Zr nuclei grow and are trapped in the β-Zr grain and Mo2Zr intermetallic compound grows in the α-Zr phase.

With the addition of higher Niobium, it will cause more nuclei centers so as to refine the grain size into relatively smaller size [14]. In Figure 1, it appears that the grain size and relatively small grain size refinement occur by the addition of higher Niobium, as Niobium serves as a nucleating agent.

Zr-10Mo-5Nb alloy has smaller grain size compared to Zr-10Mo-1Nb and Zr-10Mo-3Nb alloys, so Zr-10Mo-5Nb alloy has higher hardness value, as the 5% content of Niobium element results in more nucleation centers compared to the Niobium element with less than 5% content in Zr-10Mo-xNb alloy. Therefore, it is known that the smaller the grain sizes in the alloy microstructure, the higher the alloy hardness. This is in accordance with Hall-Petch equation [15], as follows:

$$\sigma y = \sigma i + k / \sqrt{d} \quad \ldots\ldots\ldots\ldots\ldots\ldots \quad (1)$$

where:

σy = Material yield stress
σi = The stress needed to move the dislocation
k = Constant
d = Grain size

Based on the results of corrosion testing with the polarization method, the tafel polarization curve is obtained in Figure 2.

Table 4. Data of constant Tafel, corrosion potential and corrosion rate with different immersion time. Niobium is one of betha stabilizers or beta stabilizer element for zirconium alloys, β phase has better corrosion resistance compared to α phase, in this study the addition of Niobium element in Zr-10Mo binary alloy is conducted as it can stabilize and increase β phase, so the addition of Niobium element by 1%, 3% and 5% can increase corrosion resistance, Niobium element is also more reactive to form a passive layer compared to Molybdenum element (18).

Based on the curve of the effect of Niobium on the corrosion rate, it shows that the addition of higher Niobium (Nb= 1%, 3% dan 5%) in Zr-10Mo-xNb alloy results in a lower corrosion rate so that corrosion resistance increases. The corrosion rate in Zr-10Mo-1Nb alloy of 4.208 mpy, Zr-10Mo-3Nb alloy of 3.538 mpy and Zr-10Mo-5Nb alloy of 2.813 mpy can be seen in Table 4, the decrease in the corrosion rate produced is in the range 1-5 mpy and included in the excellent category, the addition of Niobium tends to increase more β phase, thereby increasing the corrosion

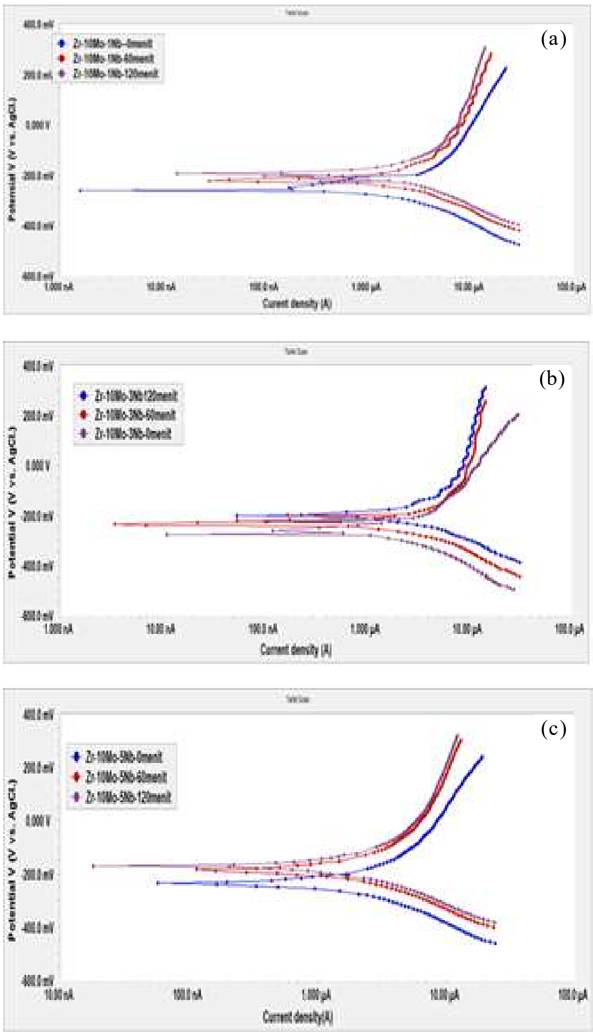

Figure 2. Tafel polarization curve of Zr-Mo-Nb alloys. (a). Zr-10Mo-1Nb; (b). Zr-10Mo-3Nb and (c). Zr-10Mo-5Nb

Table 4. shows the results of processing the tafel polarization curve for each alloy.

Alloy (*as cast*)	Remark	Immersion time		
		0 mins	60 mins	120 mins
Zr-10Mo-1Nb	I_{corr} (µA)	4.339	4.009	4.640
	E_{corr} (mV)	-256.4	-216.6	-194.0
	Corrosion rate (mpy)	4.790	4.426	4.208
Zr-10Mo-3Nb	I_{corr} (µA)	5.250	3.950	3.910
	E_{corr} (mV)	-259.0	-220.0	-201.0
	Corrosion rate (mpy)	4.757	3.578	3.538
Zr-10Mo-5Nb	I_{corr} (µA)	3.780	3.370	3.100
	E_{corr} (mV)	-235.0	-186.0	-170.0
	Corrosion rate (mpy)	3.429	3.053	2.813

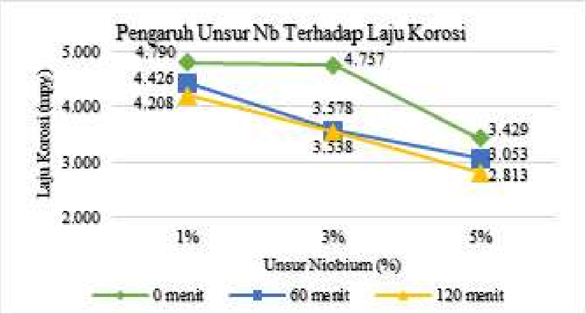

Figure 3. Effect of Niobium on the corrosion rate.

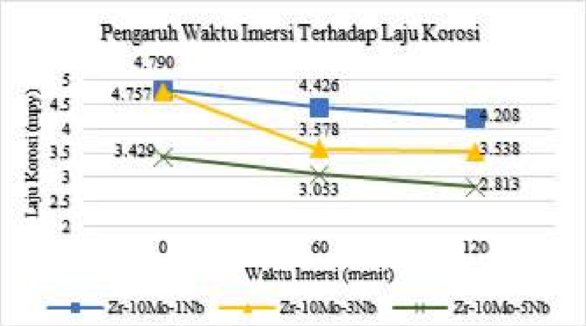

Figure 4. Effect of immersion time on the corrosion rate.

resistance of Zr-10Mo-xNb alloy (x= 1%, 3% and 5%), this is proven by the decreased corrosion rate and the formation of a stable ZrO_2 passive layer and NbO_2 passive layer.

The more addition of Niobium in the alloy and the longer the immersion time causes the corrosion potential to increase in the positive direction and the corrosion current decreases, so that the corrosion rate decreases due to the formation of a more stable passive layer and has better corrosion resistance.

Based on the curve of the effect of immersion time on the corrosion rate, it shows that an immersion time of 0 minute, 60 minutes and 120 minutes decreases the corrosion rate, this is due to the formation of ZrO_2 and NbO_2 passive layers which are more stable in longer immersion time, Zr-10Mo-1Nb alloy during immersion time of 0 minute has a corrosion rate of 4.790 mpy, immersion time of 60 minutes has a corrosion rate of 4.426 mpy and 120 minutes has a corrosion rate of 4.208 mpy, Zr-10Mo-3Nb alloy during 0-minute immersion time has a corrosion rate of 4.757 mpy, immersion time of 60 minutes has a corrosion rate of 3.579 mpy and 120 minutes has a corrosion rate of 3.538 mpy and Zr-10Mo-5Nb alloy during 0-minute immersion time has a corrosion rate of 3.429 mpy, immersion time of 60 minutes has a corrosion rate of 3.053 mpy and 120 minutes has a corrosion rate of 2.813 mpy, the decrease in corrosion rate produced with an immersion time of 0 minute, 60 minutes and 120 minutes is in the range of 1-5 mpy and categorized as excellent.

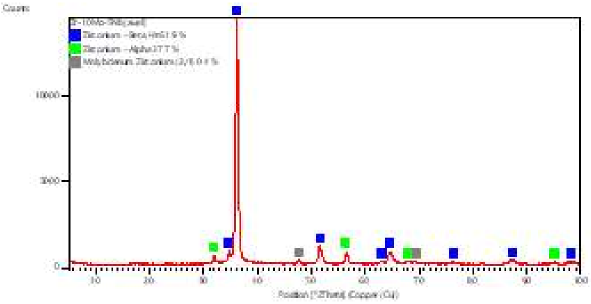

Figure 5. XRD pattern of Zr-10Mo-5Nb alloy before corrosion testing.

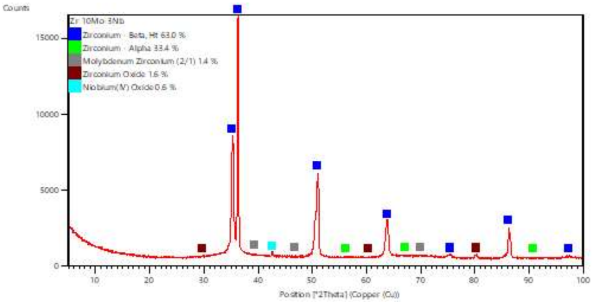

Figure 6. XRD pattern of Zr-10Mo-3Nb alloy after corrosion testing.

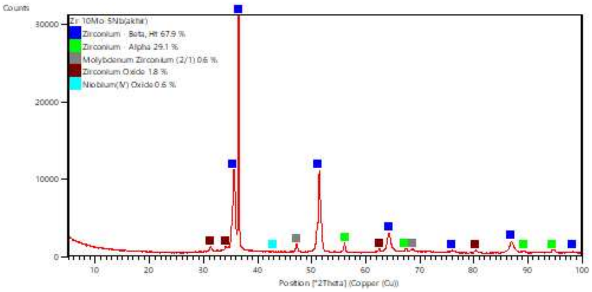

Figure 7. XRD pattern of Zr-10Mo-5Nb alloy after corrosion testing.

In Figures 5 to 7, the XRD pattern of the alloy (as cast) before and after corrosion testing by the polarization method is obtained.

Based on the XRD patterns it can be seen that the phases formed in Zr-10Mo-3Nb and Zr-10Mo-5Nb alloys are α-Zr phase, β-Zr phase and Mo2Zr intermetallic compound. The highest peak is by β-Zr phase, while α-Zr phase and Mo2Zr intermetallic compound have a lower peak, this shows that the intensity of β-Zr phase is higher than α-Zr phase and Mo2Zr intermetallic compound. After corrosion testing with the polarization method on the alloys, ZrO_2 oxide compound and NbO_2 oxide compound are formed as passive layers.

Zr-10Mo-5Nb alloys has lower corrosion rate compared to Zr-10Mo-3Nb, this is proven by the formation of more ZrO_2 oxide compound than NbO_2 oxide compound in Zr-10Mo-3Nb alloy in XRD patterns. The number of ZrO_2 oxide compound is greater than NbO_2, this is because Zirconium has a lower Eosel (Eosel= -1.45) compared to Niobium (Eosel= -1.099), so

Zirconium is more reactive and more easily oxidized compared to Niobium, whereas Molybdenum is less reactive to form Mo-oxide compound than Zirconium and Niobium because it has a higher Eosel (Eosel= -0.2), it takes longer immersion time to form Mo-oxide compound. The reaction of the formation of a passive layer is as follows:

Oxidation: $Zr \longrightarrow Zr^{4+} + 4e$
Reduction : $O_2 + 2H_2O + 4e \longrightarrow 4OH^-$

Total reaction : $Zr + O_2 + 2H_2O \longrightarrow Zr(OH)_4$
$Zr(OH)_4 \longrightarrow ZrO_2 + 2H_2O$

Oxidation: $Nb \longrightarrow Nb^{4+} + 4e$
Reduction : $O_2 + 2H_2O + 4e \longrightarrow 4OH^-$

Total Reaction : $Nb + O_2 + 2H_2O \longrightarrow Nb(OH)_4$
$Nb(OH)_4 \longrightarrow NbO_2 + 2H_2O$

In the presence of ZrO_2 oxide compound and NbO_2 oxide compound which are passive, the alloys will have better corrosion resistance compared to single ZrO_2 oxide compound.

CONCLUSION

From the results of the discussion, it can be concluded as follows: Addition of Niobium (Nb= 1%, 3% and 5%) in Zr-10Mo alloy increase hardness, Zr-10Mo-1Nb alloy has hardness of 34 HRC; Zr-10Mo-3Nb alloy has hardness of 38 HRC and Zr-10Mo-5Nb alloys has hardness of 41.5 HRC, and the addition of Niobium decreases the corrosion rate and increase corrosion resistance, corrosion rate of Zr-10Mo-1Nb alloy is 4.208 mpy, Zr-10Mo-3Nb alloy is 3.538 mpy and Zr-10Mo-5Nb alloy is 2.813 mpy. Increased immersion time of 0 minute, 60 minutes and 120 minutes increases corrosion resistance and decreases corrosion rate in Zr-10Mo-xNb alloy (x= 1%, 3% and 5%), this is due to the formation of more stable ZrO_2 and NbO_2 passive layers. The results of corrosion testing with the polarization method on Zr-10Mo-xNb alloy (x = 1%, 3% and 5%) have a corrosion rate in the range of 1-5 mpy with excellent category. Initial XRD test results on Zr-10Mo-5Nb alloy before corrosion testing contain α-Zr phase, β-Zr phase and Mo_2Zr intermetallic compounds, while the final XRD test results on Zr-10Mo-3Nb and Zr-10Mo-5Nb alloys after corrosion testing contiain α-Zr phase, β-Zr phase, Mo_2Zr intermetallic compound, ZrO_2 and NbO_2 oxide compounds. The formation of ZrO_2 and NbO_2 passive layers increases corrosion resistance in alloys.

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