

Analysis of Chemical and Phase Composition in Powder of U-Zr-Nb Post Hydriding-Dehydriding Process

Masrukan M^{a,*}, M.H Alhasa^a, Saga Octa D^a

^a Nuclear Fuel Technology Center (PTBBN) – BATAN, Puspiptek Area, Setu, South Tangerang, 15314, Indonesia

Corresponding author: *masrukan@batan.go.id

Abstract. To be used as a nuclear fuel, a fuel alloy must meet several requirements such as chemical, mechanical, physical, and neutronic compositions. The U-Zr-Nb powder is made from U-Zr-Nb ingots through a hydriding-dehydriding process and has a U composition that adjusts the weight of Zr, the composition of Zr remains at 6% while Nb varies by 2, 5, and 8 wt% (U-6Zr-2Nb, U-6Zr-5Nb and U-6Zr-8Nb). The powder obtained is then subjected to elemental and phase composition testing. Chemical composition and impurity contain testing uses Atomic Absorption Spectroscopy (AAS) and Ultraviolet (UV-Vis) spectroscopy, while phase analysis uses X-Ray Diffractometer (XRD). The purpose of the analysis of chemical composition and phase is to determine the constituent and impurity elements as well as the phases formed in the U-Zr-Nb alloy. The results of the analysis of U content in the U-6Zr-2Nb, U-6Zr-5Nb, U-6Zr-8Nb alloy powder samples were 89.307, 85.568, and 83.553 wt.%, while the Zr content analysis obtained successive results amounted to 6.220, 5.829, and 6.192. Meanwhile, in the analysis of Nb in U-6Zr-2Nb alloy powder, U-6Zr-5Nb, U-6Zr-8Nb obtained successive results amounted to 2.023, 5.04, and 8.155 wt%. The phase analysis results were obtained for each sample U-6Zr-2Nb, U-6Zr-5Nb, U-6Zr-8Nb contained U, and UO₂ compounds, where the U phase was the dominant phase. The highest γ U phase content is found in U-6Zr-5Nb, which is 92.108 %, and after the Nb content exceeds 5 %, the greater addition of Nb does not increase the number of \square U phases formed.

Keywords—Composition analysis; elements; phase; U-Zr-Nb powder.

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I. INTRODUCTION

Research and development of research reactor fuels continue to be carried out to obtain high-density fuels. Some uranium alloys are being developed to be used as research reactor fuels, including U-Mo, U-N, U-Zr alloys. The fuel replacement was carried out because the uranium silicide (U₃Si₂) alloy fuel is now relatively difficult to separate the Si element when it is recovered from uranium from U₃Si₂/Al waste and production [1]. In addition, U₃Si₂/Al fuels also have limitations due to difficult manufacturing, wherein the fabrication of U₃Si₂/Al fuels with densities greater than 5.2 U/cm³ a dog-bone will form at the end of the fuel element plate (FEP) [1], so U-Zr based alloys are candidates for fuel. The selection of U-Zr-based fuels is based on, among others: low neutron absorption cross-sections, good corrosion resistance, and high density [2].

The development of U-Zr alloys has been carried out by varying the Zr elements at 2, 6, 10, and 14 wt% and has been characterized and made into the mini of FEP [2]. Manufacturing and characterization of the mini of U-Zr FEP

at 6 wt% Zr (U-6Zr) shows acceptable results and meets requirements as nuclear fuel, such as thermal and mechanical properties [3], then prepared for irradiation tests in nuclear reactors using enrichment U of 19.75 wt% [4]. Research on making U-4Zr-2Nb fuel alloys with a density of 3.17 gU/cm³ have been carried out, and based on the results of irradiation tests, neutronic properties, and fabrication it is found that the alloy is good enough as a fuel until the alloy density reaches 8.0 gU/cm³ [5],[6].

To be used as fuel, an alloy must meet several chemical, mechanical, physical, and neutronic composition requirements. The chemical composition in the fuel will determine the characteristics of the fuel, which is related to the properties of the fuel, such as mechanical properties, physical properties, microstructure that is owned, and neutronic properties. Nuclear fuel is expected to have high purity/low impurity, especially the element content with a high neutron absorption cross-section such as elements B and Cd. The presence of elements B and Cd in high enough quantities will absorb neutrons to disrupt the reactor

operation. High microscopic neutron uptake intervals such as Cd and B, respectively, are 2.520 (50.) barn and 767. (8.) barn.

Similarly, the phase in a metal alloy affected the properties of the alloy, such as thermal properties, density, and corrosion. Phases can form if there are two or more metals combined and heated at a certain temperature. If one or more dissolves are dissolved into another metal, a solid solution is formed, while if one or two of the metals exceed the solubility limit in the other metal, a second phase is formed. The solubility of the solid and the second phase will be different so that it affects metal alloys formed, such as mechanical properties, thermal density, and corrosion. Solid solution alloys exhibit high strength, changes in density, and suppression or elimination of undesirable second phases. Increasing the strength of alloys through precipitation of a second phase from a supersaturated solid solution during aging (precipitation hardening) has been known to materials scientists for over a hundred years [7].

Pure uranium metal is a metal that has a high specific gravity, but the isotropy of γ U phase during irradiation is only stable at high temperatures, whereas at low temperatures, the α U phase structure has the potential to undergo swelling [8],[9]. Phase change α to γ (via β phase) cannot be resisted by quenching the pure α U phase at high temperatures, but the temperature range above where the α , β , and γ phases become stable so that alternatively requiring the addition of a combining agent [10]. U-Zr alloys in room temperature conditions dominated by the α U phase are in unstable conditions, so it is necessary to add metal to make it stable. One of the metal elements that can make stable conditions is the element Nb [11]. The presence of the element Nb will affect the nature of the metal alloy, especially expanding the phase γ that impacts the integrity of the lattice structure and stability at high temperatures. The metals Nb, Ti, Zr, Hf, and Re have high solubility in gamma uranium, and they do not form an intermetallic compound. However, Zr and Nb have a special advantage because of their low capture cross-section for thermal neutrons. The U-Zr-Nb alloys can stabilize the gamma phase (cubic). However, some different martensitic transformations have been found to occur, depending upon composition and cooling rate [12]. Therefore, in this study, an experiment was carried out to add the Nb element to the U-Zr alloy in several variations of the Nb composition. The purpose of this study was to determine the chemical composition and its effect on the formation of the U-Zr-Nb alloy powder phase.

The content of chemical elements and impurities were determined by Atomic Absorption Spectrometer (AAS), UV-Vis, and Inductively Coupled Plasma (ICP) techniques. Both of these analysis techniques were used because they have high selectivity and sensitivity and are simple. Meanwhile, to determine the phase formed by combining several metals, X-ray diffraction (XRD) techniques are used [11], [10].

II. MATERIALS AND METHOD

A. Materials and Equipment

In this experiment, several main materials and process equipment were used. The main ingredients of this experiment consisted of low-enriched U metal (depleted U), Zr metal (nuclear grade), and Nb metal. The auxiliary

materials consisted of hydrogen, argon, acetylene, chemical solutions for dissolution processes such as HF, HNO₃, TBP-Kerosene, and lamps for analysis according to the metal to be determined. The main ingredients of metal U, Zr, and Nb will be melted to become U-Zr-Nb, while argon gas auxiliaries are used for the smelting and hydriding processes. Other auxiliary materials, namely hydrogen gas, are used for the hydriding dehydriding process, converting U-Zr-Nb ingots into U-Zr-Nb powders, and acetylene gas for composition analysis using AAS.

For experimental purposes, this research utilized some equipment. This equipment are metal cutting machines (diamond cutting), melting furnaces, hydriding-dehydriding units, dissolution units, AAS test equipment units, and XRD units equipped with Hight Score software. The cutting machine was used to cut the U metal. The smelting ingot was used for XRD testing. The smelting furnace was used to melt the U, Zr, and Nb metals into U-Zr-Nb ingots. The hydriding-dehydriding unit was used to make powder from U-Zr-Nb ingots. Meanwhile, the dissolving unit was used to dissolve the U-Zr-Nb powder to analyze its composition. XRD equipment was used to analyze the phases that occur.

B. Methodology

The U-Zr-Nb alloy powder is made through the hydriding-dehydriding process of U-Zr-Nb ingots. U-Zr-Nb ingots are made from the metal smelting process U, Zr, and Nb in an electric arc melting furnace at a current of 150 A, which was equipped with water cooling. The composition of U-Zr-Nb powder made consists of Nb metal which is varied ie 2, 5, and 8 %wt Zr metal fixed at 6 wt% and U metal adjusts until the composition reaches 100 wt%. A variation of 2, 5, 8 %wt of Nb was chosen concerning the lowest temperature eutectic reaction region from which the formation of the stable phase. The hydriding-dehydriding process was carried out in the hydriding-dehydriding unit, where U-Zr-Nb ingot pieces were inserted into the retort tube in the hydriding-dehydriding unit. The hydriding process was started by heating the sample to a temperature of 450 °C and holding time 10 hours. The hydriding process was conducted by turning on the vacuum pump and opened the valve until the pressure reaches 14.46 psi. The next step was heating the sample at a temperature of 350 °C for 1 hour, then turned off the vacuum pump. Hydrogen gas filled the reservoir with initial pressure at approximately 14.80 psi. Afterward, it flowed into the retort tube, and the adsorption was recorded from the manometer every 15 minutes. The refilling of hydrogen gas (cycle II) was carried out when the gas pressure decreased, and the next refilling (cycle III) was carried out until the U-6Zr-5Nb ingot pieces appeared brittle. The last step hydriding process was to turn off the furnace then the temperature cooling down until it reached room temperature. After the hydriding process was finished, the dehydriding process was conducted to remove the hydrogen gas that attached to the brittle U-Zr-Nb sample by reheating at 450 °C to obtain the U-Zr-Nb sample which was brittle and easily crushed into powder. The powder obtained was partially subjected to composition testing using UV-Vis equipment for Zr levels, Nb using ICP, impurities using UV-Vis's spectrometers, and phase analysis using XRD equipment. U-Zr-Nb powder is first dissolved using HF solution and evaporated by heating then cooling in carrying

out chemical composition testing. The resulting solution was diluted in HNO_3 , then extracted a mixture of TBP and Hexan solutions in a ratio of 7: 3. The mixture of the two phases is allowed to stand for 5 minutes so that the water phase and organic phase separate well. The liquid phase solution containing Zr and Nb and U in the organic phase was analyzed using a UV-Vis spectrometer to determine the U and Zr content, while to determine the composition of the impurity element, the analysis was carried out using an atomic absorption spectrometer (AAS). The other powders are subject to phase testing using XRD equipment which is equipped with High score and GSAS software for data processing. In the implementation of testing using XRD, the

sample is inserted into the sample holder placed on the XRD device. XRD testing is carried out from an angle of 2θ by 20° to 80° . The test results were obtained in the form of a diffraction pattern curve between the angle of diffraction (angle 2θ) on the horizontal axis and the intensity of the light produced as a vertical axis. The data obtained is then processed using the Hight Score and GSAS software to determine the number of phases, composition, structure, and density. The scheme of the experiment process hydriding process is shown in Figure 1, while the scheme of the hydriding-dehydriding process to making U-Zr-Nb powder is shown in Figure 2.

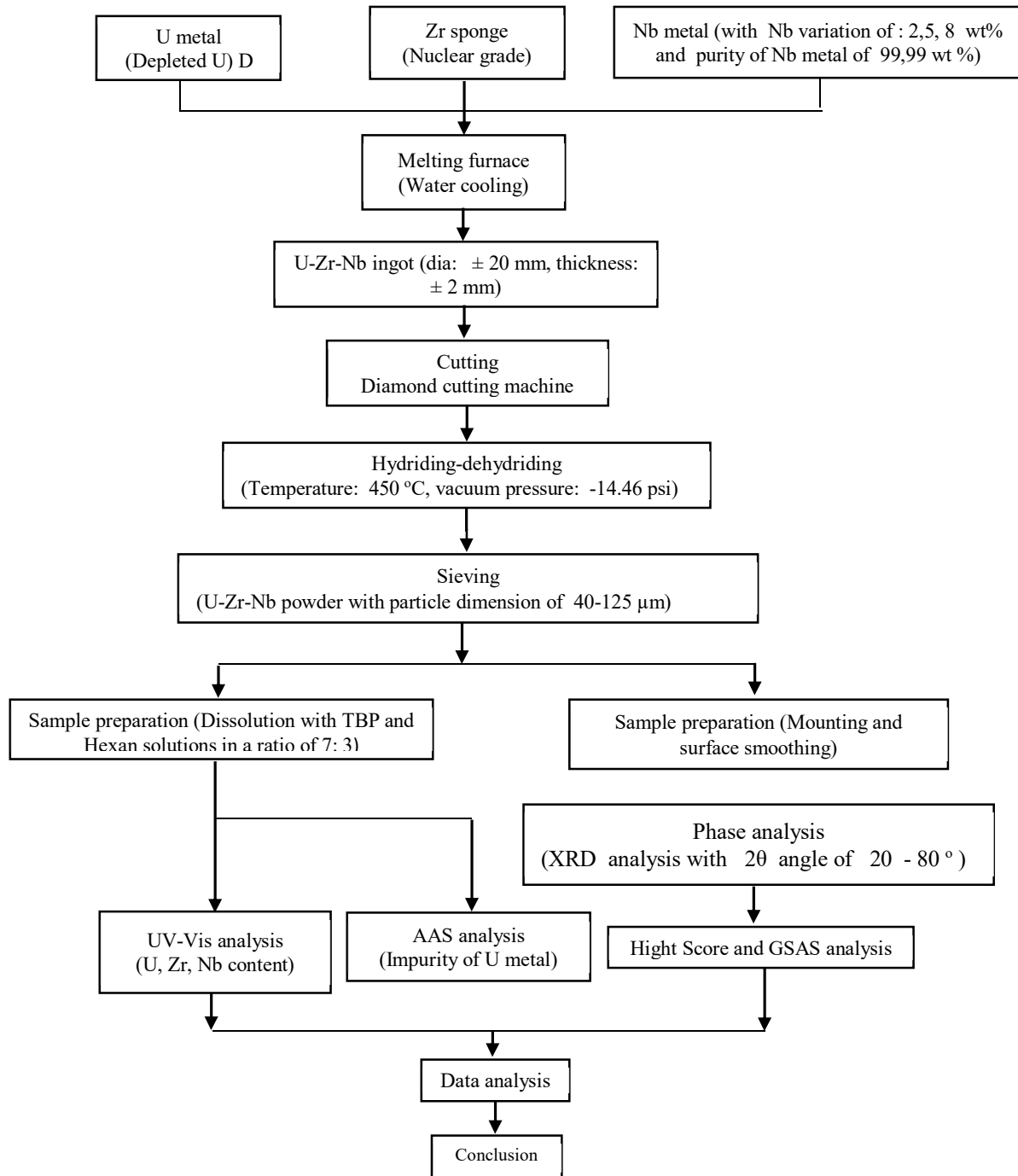


Fig. 1 Scheme of experiment process

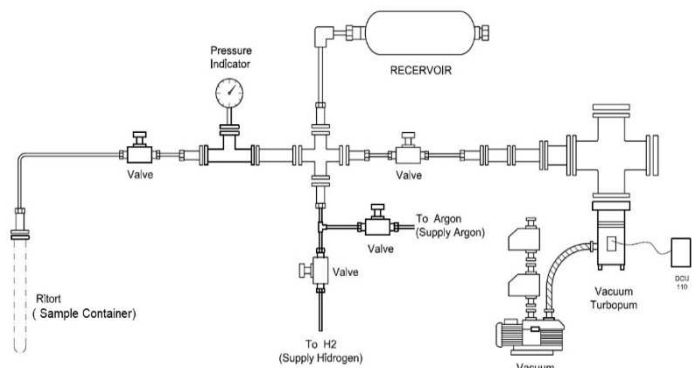


Fig. 2 The scheme of the hydriding- dehydriding equipment to making U-Zr-Nb powder

III. RESULTS AND DISCUSSION

A. Chemical Composition

The analysis of the chemical composition of the constituent elements in the U-Zr-Nb alloy is shown in Tables 1, 2, and 3. The results of the analysis of impurity elements are shown in Table 4. Table 1 contains the results of the analysis of the U-6Zr-2Nb powder. It seemed that the results of the analysis elements U, Zr, and Nb reached 89.307, 5.96, and 2.023 wt% with a recovery rate of 97.073, 99.333, and 101.150 % and an accuracy of 0.029, 0.007, and 0.012 %.

TABLE I
THE U, ZR, AND NB CONTENTS IN U-6ZR-2NB POWDER

Element	Content			
	Planned (wt%)	Test result (wt%)	Recovery (%)	Accuracy (%)
U	92	89.307	97.073	0.029
Zr	6	5.960	99.333	0.007
Nb	2	1.963	98.150	0.018

For the analysis of U-6Zr-5Nb powder elements as listed in Table 2, the U, Zr, and Nb analysis results were 85.568,

TABLE IV
CONTENTS OF IMPURITIES IN U-ZR-NB POWDER [13]

No	Element	U-6Zr-2Nb alloy (ppm)	U-6Zr-5Nb alloy (ppm)	U-6Zr-8Nb alloy(ppm)	Specification(ppm)
1	Ag	not detected	not detected	not detected	≤ 0.1
2	Al	243.65 ± 2.69	228.37 ± 3.32	322.24 ± 1.02	≤ 50.0
3	Ca	48.84 ± 1.86	62.64 ± 0.63	47.98 ± 0.15	≤ 50.0
4	Cd	not detected	not detected	not detected	≤ 0.2
5	Cu	38.04 ± 0.45	19.08 ± 0.1	37.00 ± 0.25	≤ 20
6	Co	not detected	not detected	not detected	≤ 75
7	Cr	28.57 ± 0.65	19.14 ± 0.0	22.58 ± 0.341	≤ 100
8	Fe	175.71 ± 3.43	141.92 ± 1.69	160.10 ± 3.16	≤ 71.48
9	Mg	8.51 ± 0.35	2.83 ± 0.0	9.91 ± 0.6	≤ 60.0
10	Mn	10.69 ± 0.52	12.69 ± 0.05	6.06 ± 0.071	≤ 10.0
11	Mo	23.66 ± 0.55	not detected	23.00 ± 0.73	≤ 50
12	Ni	23.66 ± 0.55	18.82 ± 0.25	not detected	≤ 30
13	Pb	not detected	not detected	3.32 ± 0.001	≤ 8.47
14	Si	not detected	not detected	not detected	≤ 60.0
15	Sn	13.41 ± 1.38	16.44 ± 1.41	14.16 ± 0.45	≤ 50.0
16	Zn	13.54 ± 2.17	10.27 ± 0.12	22.11 ± 0.28	≤ 100
17	Li	0.05 ± 0.0001	not detected < blank	0.04 ± 0	≤ 10

5.99, and 5.04 wt.% with a recovery rate of 96.144, 99.833, and 97.800 wt% and accuracy of 0.039, 0.002, and 0.008 wt.%. Meanwhile, for U-6Zr-8Nb, as shown in Table 3. The U, Zr, and Nb analysis results were respectively 83.553, 5.89, and 8.155 wt.% with recovery rates 97.155, 98.167, and 101.938 wt.% and the accuracy level 0.028, 0.018, and 0.019 %.

TABLE II
THE U, ZR, AND NB CONTENTS IN U-6ZR-5NB POWDER

Element	Content			
	Planned (wt%)	Test result (wt%)	Recovery (%)	Accuracy (%)
U	89	85.568	96.144	0.029
Zr	6	5.99	99.833	0.002
Nb	5	4.89	97.800	0.022

From the results of this analysis, it can be said that the results of the analysis of the elements U, Zr, and Nb are quite good. This is shown by the results of recovery in each of the analysis results obtained, namely between 96.144 to 99.833 % and a low level of accuracy, which is between almost 0.002 to 0.029 %. In making an alloy, the accuracy of U weight is important and needs to be considered because if the U content is different from what is planned, it will affect the amount of U used to affect the fuel performance in the reactor.

TABLE III
THE U, ZR, AND NB CONTENT IN U-6ZR-8NB POWDER

Element	Content			
	Planned (wt%)	Test result (wt%)	Recovery (%)	Accuracy (%)
U	86	83.553	97.155	0.028
Zr	6	5.890	98.167	0.018
Nb	8	7.770	97.125	0.028

In the analysis of impurity content, as shown in Table 4, it was found that some elements exceeded the specified requirements, namely elements of Al, Cu, Fe, and Mn.

This situation is caused by the elements being gained from the results of melting using a crucible made of metal, which has been used crucible for the melting process of other alloys so that some elements are analyzed. Other elements not included in the smelting result are suspected $\beta\epsilon\chi\alpha\upsilon\sigma\epsilon$ these elements are not widely used in the raw materials subject to the smelting process. However, these elements include elements that have a low neutron absorption look so that it is not a concern when used for nuclear fuel. The elements that have a high neutron absorption cross-section are elements B and Cd.

B. Phase analysis

The results of the phase analysis obtained from testing using XRD are shown in Figures 3 until 6. Figure 3 is a U-6Zr-2Nb diffraction pattern curve showing the formation of phases α , γ , and UO_2 compounds. Figure 3 and Figure 6 show curves that are dominated by phase α , which is 80.664 %, while phases γ and UO_2 are 16.893 % and 2.461%, respectively. Phases α formed respectively at an angle of 2θ at 36.835, 53.078, and 66.354 °. The γU phase was formed at an angle of 2θ at 35.493, 36.2 and 39.465 ° while the UO_2 compound is formed at an angle of 2θ at 28.298, 47.054 and 55,820 °. Uranium is very reactive with oxygen which forms the reaction of UO_2 compounds in the form of cubic structures. If the uranium metal is not conditioned under a vacuum or an inert gas atmosphere, the reaction can occur at room temperature. This is as explained in the reference [14].

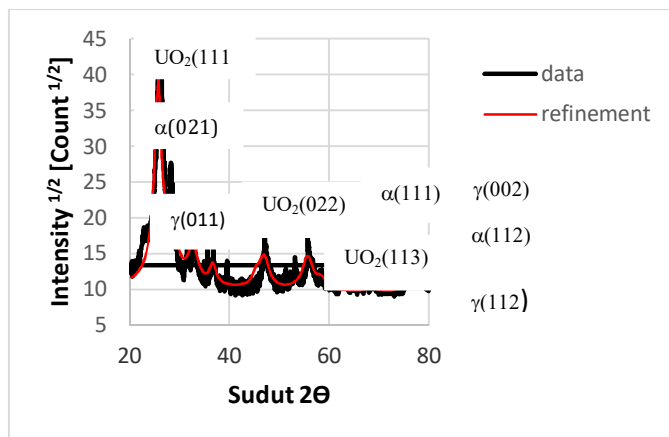


Fig. 3 Diffraction pattern of U-6Zr-2Nb powder. Testing result using XRD

Figure 4 is a diffraction pattern curve of U-6Zr-5Nb alloy powder showing that the γ phase, UO_2 compound, and α phase with a percentage of 7.855, 92.108, and 0.0359 %. The αU phase is formed at an angle of 2θ 36.835, 53.078, and 66.354 °. The γU phase is formed at an angle of 2θ 35.493, 36.2, and 39.465 ° while the UO_2 compound was formed at an angle of 2θ at 28.298, 47.054, and 55,820 °. An increase in the number of phases was seen in the γU phase, which was originally 16.899 to 92.108 %, while the UO_2 phase compounds decreased. This is caused by an increase in Nb content from 2 to 5 %. The presence of the Nb element causes the γ phase area to become wider. If the phase area becomes wider, the fuel becomes more stable. It is hoped that the U-Zr-Nb fuel will be in the γU phase because if it is in the γU phase, it will be stable.

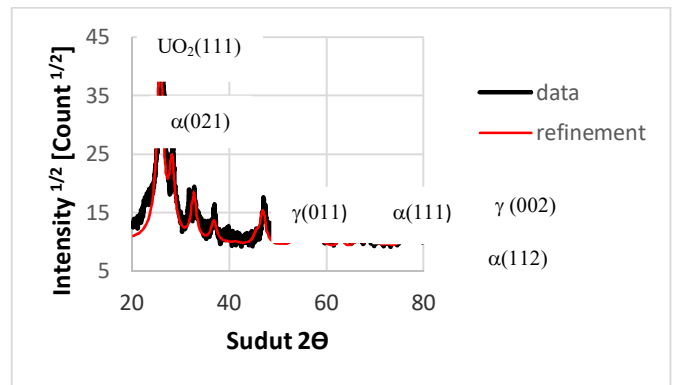


Fig. 4 Diffraction pattern of U-6Zr-5Nb powder. Testing results by using XRD

Figure 5 shows the U-6Zr-8Nb powder diffraction curve pattern shows that in the powder, there are αU , γU , and UO_2 compounds respectively 3.710, 35.665, and 60.623 %. Phases α formed at an angle of 2θ each at 36.835, 53.078, and 66.354. The γ phase is formed at an angle of 2θ each in 35.493, 36.2, and 39.465 °, while the UO_2 compound was formed at an angle of 2θ at 28.298, 47.054, and 55.820 °. As can be seen in Figure 5, the composition of the available phase percentage shows that the γU phase has decreased compared to the U-6Zr-5Nb powder, while the αU phase and UO_2 compound have increased. This means that after the Nb content exceeds 5 %, the greater addition of Nb does not increase the amount of phase γU , it can be said that the addition of Nb by 5 % will have the greatest impact on the change of phase α to phase γU .

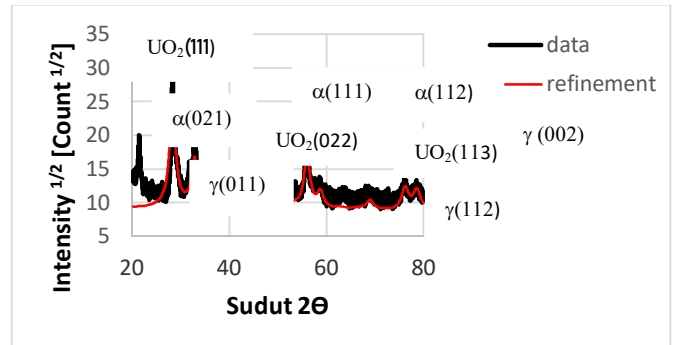


Fig. 5 Diffraction pattern of U-6Zr-8Nb powder. Testing result using XRD

When seen in Figure 6, which shows the relationship between the phases formed against the increase in Nb elements, it is seen that from the 2 to 5 % Nb content, there is an increase in the gamma phase while the alpha phase and UO_2 compound decrease, then the gamma phase decreases when the percentage of element Nb increases. It can be said that the addition of 5 % by weight of the Nb element into the U-6Zr alloy produced the largest amount of gamma phases formed.

Phase α is a phase consisting of U metal in which Zr and Nb are dissolved. The crystal structure of the phase formed is orthorhombic with a lattice parameter $a = 2.8580 \text{ \AA}$; $b = 5.8760 \text{ \AA}$; $c = 4.9550 \text{ \AA}$ and formed at relatively low temperatures, while the phase γU with a lattice parameter $a = 3.4480 \text{ \AA}$ is a metal U in which the metal Zr and Nb dissolve

and forms at higher temperatures and has a body center crystal structure cubic (bcc). Phase changes α to γ U phases are carried out by heating or adding metal elements at high temperatures then cooled to room temperature. Through the addition of metal elements at high temperatures, the γ U phase formed at high temperatures will remain even though the alloy temperature is cool. A change also follows this phase change in the crystal structure from the original phase α has an orthorhombic structure that will change to the γ U phase, which has a cubic structure. Meanwhile, UO_2 compounds are formed due to the oxidation of the U-Zr-Nb sample either during the hydriding-dehydriding process or after the hydriding-dehydriding process, where the U-Zr-Nb sample is stored in an open place and in contact with air.

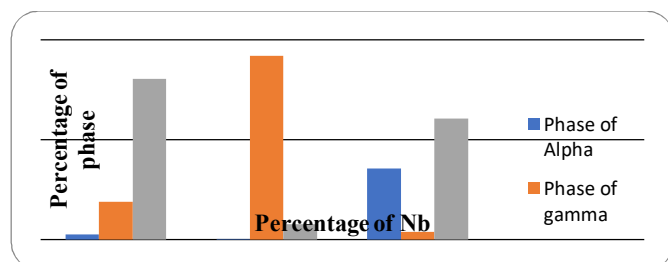


Fig. 6 Phase composition with Nb variation content in the U-Zr-Nb powder

When compared with U-6Zr-2Nb, U-6Zr-5Nb, and U-6Zr-8Nb alloys in the form of ingots (before hydriding-dehydriding), it can be seen that U-6Zr-2Nb alloy ingots have α U and γ U phases, ingot alloy U-6Zr-5Nb has α U, γ U, and δ 1 (UZr2) phases, U-6Zr-8Nb alloy ingots have α U, γ U, and δ 1 (UZr2) phases. Both U-6Zr-5Nb alloys, both ingots and powders have the same phases of U when in the same conditions. The α U phase appears at an angle of 2θ respectively at 35.4, 38.6, 52.05, and 65.01 $^\circ$, the γ U phase formed at an angle of 2θ respectively at 35.64, 59.8, and the 76 $^\circ$ while phase δ 1 (UZr2) is formed at an angle of 2θ at 26.67 $^\circ$. The results are not different from the two forms, namely powder, an ingot, where they produce the α U and γ U phases. Both of these results are also following previous research of making U-Zr-Nb alloys that obtained α U, γ U, and δ 1 (UZr2) phases [15],[16].

IV. CONCLUSION

The results of the analysis of U content in the U-6Zr-2Nb, U-6Zr-5Nb, U-6Zr-8Nb alloy powder samples were 89.307, 85.568, and 83.553 wt%, while the Zr content analysis obtained successive results amounted to 6.220, 5.829, and 6.192 wt%. Meanwhile, in the analysis of Nb in U-6Zr-2Nb alloy powder, U-6Zr-5Nb, U-6Zr-8Nb obtained successive results amounted to 2.023, 5.04, and 8.155 wt%. The recovery results on each U-6Zr-2Nb alloy powder, U-6Zr-5Nb, U-6Zr-8Nb between 96.144 to 99.833 %, and a low accuracy rate between 0.002 to 0.029 %. From the analysis of impurity elements, it was found that the elements Al, Mn, Cu, and Fe exceeded the requirements. The phase analysis results were obtained for each sample U-6Zr-2Nb, U-6Zr-5Nb, U-6Zr-

8Nb contained α U, γ U, and UO_2 compounds, where the γ U phase was the dominant phase. The highest γ U phase content is found in U-6Zr-5Nb, which is 92.108 wt%, and after the Nb content exceeds 5 wt%, the greater addition of Nb does not increase the number of γ U phases formed.

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