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Effect of Ni Addition on the Density, Microstructure, Hardness, and Phase Stability of U-7Mo-xNi Alloys

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ABSTRACT

The Reduced Enrichment Research and Test Reactor (RERTR) program required a transition from high enriched uranium (HEU) to low enriched uranium (LEU). The most promising nuclear fuel with LEU is uranium-molybdenum (U-Mo). U-Mo has high density and can act as γ -U stabilizer, but under irradiation test, showed undesirable interaction with aluminum (Al) matrix. To stabilizing γ and minimize interaction with Al, Ni was added to U-7Mo. The study began with fabrication of U-7Mo-xNi alloy using an electric arc melting furnace under an argon atmosphere. The resulting U-7Mo-xNi were characterize through microstructural observation using optical microscope, density measurement using automatic gas pycnometer, hardness testing using Vickers, and phase identification using x-ray diffraction (XRD) then compared to U-7Mo. The results show that U-7Mo-xNi has lower density than U-7Mo, with 17.42 for U-7Mo, 16.77 g/cm³ for U-7Mo-1Ni, 16.45 g/cm³ for U-7Mo-2Ni and 16.42 g/cm³ for U-7Mo-3Ni. The average grain size also shows a decrease from 20.97 μ m to 14.41 μ m, which leads to an increase in hardness based on Hall-Petch relations. The results of hardness test show the higher value than U-7Mo with value 240.4 HV, 313.87 HV, 340.51 HV and 357.47 HV for U-7Mo-xNi as Ni composition in order. For XRD analysis, peaks detected at 2θ angle of 37.0°, 53.6° and 66.5° same as U-7Mo peaks and identified as γ -U phase, but in U-7Mo-xNi has a minor peak at 2θ angle of 40.8° identified as Mo. The addition of Ni to U-7Mo improve the hardness of the alloy and the γ -U phase still be maintained. The dendritic structure and minor peaks in U-7Mo-xNi indicate the need for further homogenization and characterization.

Keywords: Density, microstructure, hardness, phase, U-Mo-xNi alloy.



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INTRODUCTION

The operational sustainability of research reactors is highly dependent on the availability of reliable nuclear fuel. In principle, both research reactors and power reactors require nuclear fuel that meet certain requirements to ensure their safety and efficiency during operation [1]. Indonesia has three research reactors that play an important role in research on nuclear physics, material testing, new fuel development, and the production of radioisotopes for medical and industrial needs. One of these research reactors is the Reaktor Serba Guna – Gerrit Augustinus Siwabessy (RSG-GAS), located at B.J. Habibie Science and Technology Complex (KST), National Research and Innovation Agency (BRIN), Indonesia. This multi-purpose research reactor has a thermal power of 30 MW and an average thermal neutron flux of 2×10^{14} n/cm²s [2].

Its operations began in 1987 using oxide dispersion fuel (U₃O₈-Al) then U₃Si₂-Al with a uranium density of 2,96 gU/cm³ and reach maximum uranium density of 4,8 gU/cm³ [3]. RSG-GAS became one of the first reactors in the world to use Low Enriched Uranium (LEU) as its nuclear fuel, namely U-235 19.75%, following the Reduced Enrichment Research and Test Reactor (RERTR) program, which required a transition from High Enriched Uranium (HEU) to LEU in 1978 [4] [5]. The nuclear fuel elements and control rods for this reactor are produced independently by PT INUKI (Persero).

Although there is an obligation to transition from HEU to LEU, these fuel candidates must be capable to use under high burn-up condition with minimal irradiation damaged [6]. To achieve high burn-up is by increasing density, which can also extend the time of fuel in the reactor core. Therefore, development of nuclear fuel was carried out using uranium-molybdenum (U-Mo) that can be increased to 8 gU/cm³ [4][7].

Molybdenum is an element classified as a transition metal in group 6 and period 5 of the periodic table [8]. This metal has a BCC (Body Centered Cubic) crystal structure and high solid solubility, so the γ phase structure of the U-Mo alloy relatively resistant to irradiation. The equilibrium point of the γ -U

phase structure in solid form is at a temperature of $575^\circ\text{C} < x < 1285^\circ\text{C}$, depending on its composition. With its high binding energy, Mo metal allows for the expansion of the γ -U phase region [1][9][10].

The physical properties of Mo are considered ideal for alloying with uranium as nuclear fuel, but research by Jana et al. states that a eutectoid reaction occurs in U-Mo alloys formed through the discontinuous precipitation (DP) mechanism. This mechanism forms a layered structure of α -U and γ -U-Mo phases, where the α -U phase worsens the mechanical properties of the alloy, such as susceptibility to distortion and swelling. Under irradiation test, U-Mo fuel particles showed undesirable interaction with aluminum (Al) matrix [11]. The layer between U-Mo and the Al matrix will cause fracturing through significant volumetric expansion during reactor operations [12].

There are several solutions, including fuel coating, use of monolithic fuel and adding third element to U-Mo alloy [13]. Among these, adding third element as method was selected. Transition metals such as silicon (Si), zirconium (Zr) niobium (Nb), titanium (Ti), chromium (Cr) and nickel (Ni) are potential as third element of U-Mo alloys because ability to minimize the formation of interaction layer and stabilize γ -U phase on U-Mo alloys [14][12][15]. In this study, Ni was added to U-7Mo alloys because can act as γ -U stabilizer and to be a grain refiner which can increase the hardness of the alloy [15][16]. Ni as grain refiner is important for U-7Mo, which has ductile materials.

Therefore, this study was conducted to analyze the effect of Ni addition to U-7Mo by comparing the density, microstructure, hardness level, and phase formed of U-7Mo-xNi and U-7Mo alloys as a candidate for nuclear fuel for RSG-GAS.

METHODOLOGY

The detailed procedure follows the methodology developed in the author's undergraduate thesis work [21]. The U-7wt%Mo-x.wt%Ni (U-7Mo-xNi) alloys (x=1, 2 and 3) are using depleted U with a purity of 98,8% U content, Mo granule with a purity 99% and Ni metal sheet with a purity 99%.

The production of the alloy began with the pickling U. The pickling process was conducted to clean the surface of U from

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oxide layer and other impurities using HNO_3 with concentration of 60% at temperature 70°C . The other metals (Mo and Ni) are cleaned with alcohol. After that, all metals are analyzed to determine their U content and being weighed according to composition variations as shown in Table 1.

The melting process was carried out using the electric arc melting technique with a current setting of 150 A in an argon (Ar) media. To ensure the homogeneity of the alloy, each sample was melted in 30 seconds and repeated five times.

The analysis included measuring the density of U-7Mo-xNi alloy using an automatic gas pycnometer Ultrapyc 1200e. The hardness of the alloy was counted by Vickers Hardness test, while phase identification was conducted using X-Ray Diffraction (XRD) SmartLab Rigaku and analyzed using HighScore software.

The metallographic observed microstructure began with mounting, grinding, polishing, and etching. The surface of each sample was taken using optical

microscope (Nikon Ehipot) in center position with 200x magnification. Then, the grain size was counted using AxioVision software. The overall experimental workflow of this study is illustrated in Figure 1.

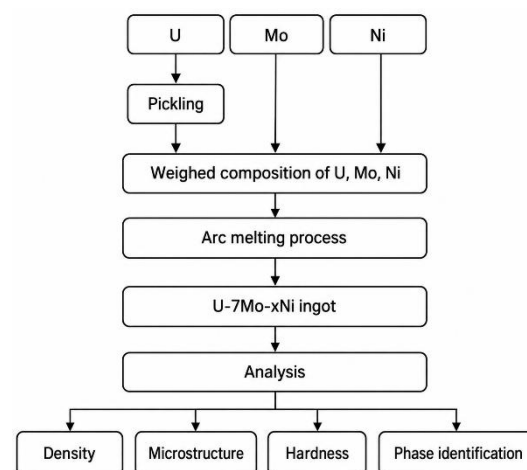


Figure 1. Flowchart of the research process

Table 1. Mass composition of U-7Mo-xNi alloy fabrication

Alloy	U		Mo		Ni	
	Mass (g)	Content (wt.%)	Mass (g)	Content (wt.%)	Mass (g)	Content (wt.%)
U-7Mo-1Ni	9.2030	91.47	0.7001	6.96	0.1084	1.08
U-7Mo-2Ni	8.9529	90.90	0.6819	6.92	0.1960	1.99
U-7Mo-3Ni	9.1682	89.29	0.7147	6.96	0.3035	2.96

RESULT AND DISCUSSION

Figure 2 shows the U-7Mo-xNi after melting process using electric arc furnace under an argon atmosphere. The ingot appears relatively compact and rounded shape, indicates that the melting process proceeded properly.

Density Result Testing

The density of the ingots was measured using an automatic gas pycnometer, and the results are presented in Table 2 and Figure 3. The density of the alloy decreases as the Ni content increases. The U-7Mo-xNi has a lower density than U-7Mo, which has a density of 17.42 g/cm^3 [7]. Theoretically, it can be explained by the rules of mixtures, which Ni has a density of

approximately 8.9 g/cm^3 , lower than U (19.07 g/cm^3) and Mo (10.22 g/cm^3) causes a decrease in the total of alloy's density [16][4][8]. Ni as third element contributes to the density of ingot, this is like addition of Si to U-7Mo [4]. The decrease in density is possibly to microporosity and the purity of the metals less than 100%.

Microstructure Observation Result

Microstructure of the ingots in central position was observed using optical microscope at 200x magnification. The result shows that the addition of Ni significantly reduces grain size, which is smaller than average grain size of U-7Mo, shown in Figure 4.

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Table 2. Density, Grain Size and Hardness Measurement of U-7Mo-xNi Alloy

Alloy	Density (g/cm ³)	Average Grain Size (μm)	Hardness (HV)
U-7Mo	17.42*	41.38 ± 1,8	240.4
U-7Mo-1Ni	16.77	20.97 ± 6,5	313.87 ± 5.5
U-7Mo-2Ni	16.45	16.71 ± 5,5	340.51 ± 4.9
U-7Mo-3Ni	16.42	14.41 ± 5,1	357.47 ± 6.7



Figure 2. U-7Mo-Ni ingot

Ni precipitates formed in grain boundary [15]. The formation of precipitates is one of the factors that cause grain growth restricted, resulting the decrease in grain size. The alloy still has a dendritic structure in the center of the ingot. This dendritic structure is the result of the relatively rapid cooling rate. Supardjo et al. and Suryaman et al. also observed the dendritic structure in the U-7Mo based alloy using the same technique [17].

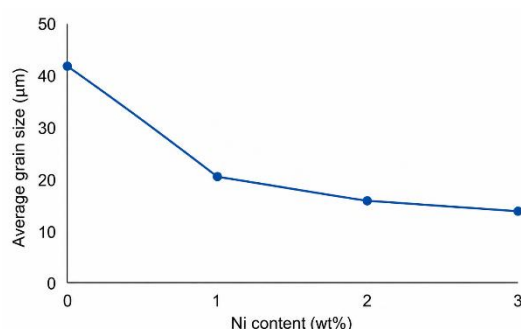


Figure 3. Average Grain Size of U-7Mo-xNi

Hardness Result Testing

As shown on Table 2, the Vickers test results indicate that the hardness of the alloy increases as the average grain size decreases. The increase of hardness is due to Ni as grain refinement because can restricted dislocation within a grain. In previous study by Supardjo et al. the addition

of Si to the U-7Mo alloy also showed an increase in hardness as the Si content increased, the result then compared to U-7Mo-xTi which exhibited the same trend. The addition of the third element to U-7Mo based alloy, such Ni, Si and Ti contributes to an increase in the alloy's hardness [4].

The relationship between microstructure and hardness in alloys can be explained by the Hall-Petch relations [18],

$$\sigma = \sigma_0 + k d^{-0.5} \quad (1)$$

Where σ denotes the yield strength, d corresponds to the grain size, and σ_0 and k are material-dependent parameters. This indicates an increase in yield strength due to a reduction in grain size, which also affects hardness. As the grain size becomes finer ($d^{-0.5}$ increase) the number of grain boundaries increase. Therefore, decreasing the grain size will restrict dislocation movement, which will result in increased hardness [19][20].

Phase Identification Testing

Figure 5 shows the XRD results, where the peaks are detected at 2θ angle of 37.0° , 53.6° , 66.5° , and minor peaks at 40.8° . By analyze using HighScore plus the diffraction peaks indicates that U-7Mo-xNi has dominant γ -U phase according to COD database. The result also comparing with XRD results of U-Mo alloy by Octadamailah et al., as shown in Figure 6, which has angles of approximately 37° , 53° , 66° and 78° correspond to the (011), (002), (112) and (022) plane, identified as γ -U phase [10][7].

The U-7Mo-xNi diffraction peaks slight shift to the left as shown in Figure 7, indicates the diffraction peaks toward lower 2θ as the Ni content increase. According to Bragg's Law ($n\lambda = 2d \sin\theta$), this shift peaks, increasing the interplanar spacing and lattice parameter leads to larger unit cell, which may decrease the density.

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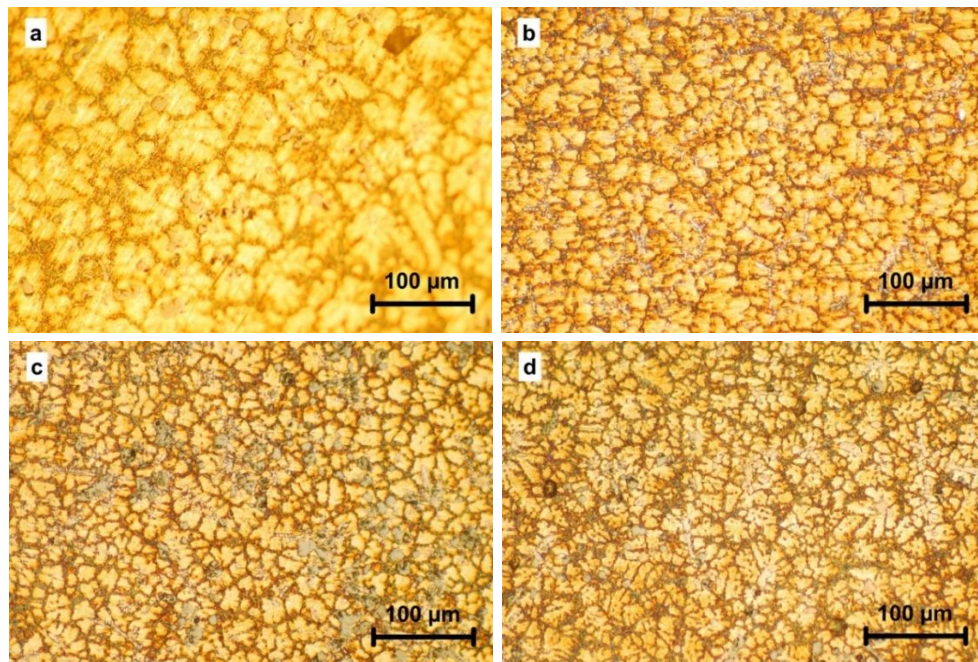


Figure 4. Microstructure of U-7Mo (a); U-7Mo-1Ni (b); U-7Mo-2Ni (c); and U-7Mo-3Ni (d).

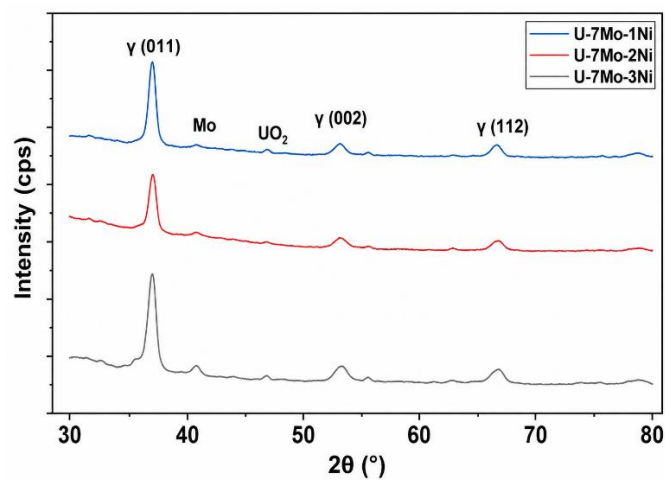


Figure 5. XRD results of U-7Mo-xNi

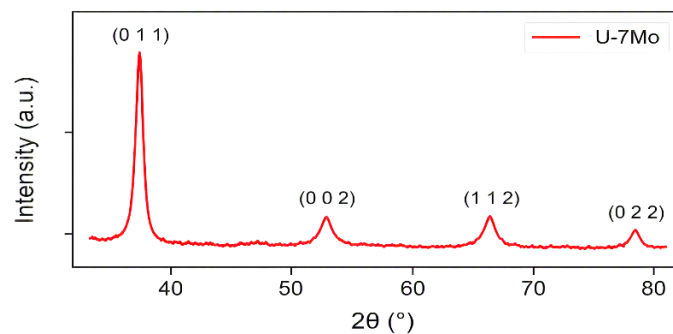


Figure 6. XRD results of U-7Mo [7]

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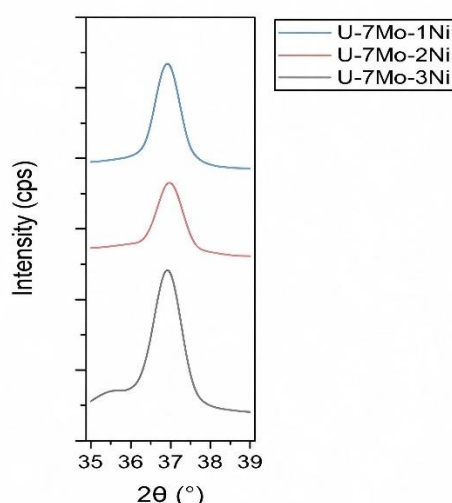


Figure 7. XRD patterns of U-7Mo-Ni at 2θ angle of 37.0°

In the context of nuclear fuel, the γ -U phase is preferred because greater stability during irradiation compared to the α -U phase of uranium alloys [9]. In this study, Mo and Ni act as γ -U phase stabilizers by maintaining the stability of BCC γ -U [17][7][15]. The XRD results show that the dominant phase of U-7Mo-xNi is BCC γ -U, indicating that the γ -U phase can be maintained even with addition of Ni. A minor peak also observed at 40.8° indicate a peak of Mo.

CONCLUSION

The addition of Ni to U-7Mo alloys decreased the density and grain size while increasing hardness, without changing the dominant γ -U phase. The density decreased from 17.42 g/cm^3 for U-7Mo to 16.42 g/cm^3 for U-7Mo-3Ni, while the average grain size decreased from 41.38 to $14.41 \text{ }\mu\text{m}$ and hardness increased from 240.4 to 357.47 HV . XRD results confirmed that the γ -U phase remained dominant in all U-7Mo-xNi alloys, although the dendritic microstructure and minor Mo peak indicate that further homogenization is required. Among the Ni-containing alloys, U-7Mo-1Ni retained the highest density, whereas U-7Mo-3Ni exhibited the finest grain size and highest hardness. Therefore, no single composition can yet be considered optimum; however, U-7Mo-1Ni offers the best balance between maintaining high density and improving mechanical characteristics.

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