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International
Journal
Of Modern Engineering Research (IJMER)

Composition Analysis Of Elements, Density, And Thermal Properties Of The Pellet Of U-Zrh$_{1.55}$ Alloys

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ABSTRACT. *The pellet of U-ZrH$_{1.55}$ fuel is a potential replacement fuel for PWR UO$_2$ fuel that has been used. The pellet of U-ZrH$_{1.55}$ fuel is a potential replacement fuel for PWR UO$_2$ fuel that has been used. The aim of this research was to determine the composition of elements, density, and thermal properties of the pellet of U-ZrH$_{1.55}$. In the beginning, the U-ZrH$_{1.55}$ ingot was made from U and Zr metals contain 35%, 45% and 55% Zr by weight consequently. Next, the ingot was converted into powder using hydriding technique continued with milling. The U-ZrH$_{1.55}$ powder, then put into the mold (dies) and pressed at a pressure of 20 tons/cm^3 to form pellets. The U-ZrH$_{1.55}$ powder, then put into the mold (dies) and pressed at a pressure of 20 tons/cm^3 to form pellets. Pellets obtained were tested, its properties such as elemental composition, dimensions, density, and transition temperature testing. The results of elemental composition testing showed some impurity elements that exceed allowable limits such as elements Ni, Mg, Cd, Zr, and K. The result of density testing showed that the density decreases when the Zr content increases. Density values for the pellets of U-35ZrH$_{1.55}$, the U-45ZrH$_{1.55}$ and the U-55ZrH$_{1.55}$ respectively 9.9141 ; 7.9920; and 7.0359 g/cm^3. The result of temperature transition testing using DTA obtained that pellets of U-35ZrH$_{1.55}$ and U-55ZrH$_{1.55}$ have four times endothermic reaction stages while for U-35ZrH$_{1.55}$ have five times endothermic reaction stages. During endothermic reactions, all pellets undergo a phase change from the original phase $\alpha + \delta 1$ into phase γ at the end of the reaction.*

KEYWORDS: *Element, Composition, Density, Thermal Properties, Pellet, U-ZrH$_{1.55}$.*

Date of Submission:22-06-2019 Date of acceptance: 08-07-2019

I. INTRODUCTION

Energy usage in Indonesia is increasingly growing along with the growth of industries that require a substantial supply of electrical energy. The need for a substantial amount of additional energy must be immediately dealt with increasing the supply of electricity from various sources. Several types of renewable and non-renewable energy sources are available in Indonesia, such as fossil energy (oil), mine (coal), solar, wind, geothermal and nuclear materials. Continuation with and non-renewable use of fossil fuels and minerals (coal) will result in less depletion of fossil (oil) resources, while solar and wind energy is limited in applications. Therefore, it is necessary to develop new clean and efficient energy sources such as nuclear energy.

Development of nuclear fuel in Indonesia was developed by the Nuclear Fuel Technology Center (PTBN-National Nuclear Energy Agency (BATAN). In practice, the development of nuclear fuel is divided into two lines are fuel development of the power plant and research reactor. The development of power reactor fuel is aimed at among others to obtain prototype of PWR fuel element and HWR Cirene fuel beam while the development of research reactor fuel is aimed to obtain the new fuel with high density from an alloy based on UN, U-Mo, and U-Zr [1], [2].

PWR reactor fuel is generally made of ceramic oxide fuel UO$_2$, metal, and metal-ceramic (Cermet)[3]. A new fuel design has been proposed to replace UO$_2$ fuel with uranium hydride (U-ZrH$_{1.55}$), wherein the new fuel design it is advantageous to place hydrogen as a moderator directly in a fuel that allows the reactor to operate at a temperature relatively high (up to 750 ^{o}C) and has better thermal properties than ceramic fuel [4].

Meanwhile, the use of uranium for a fuel uranium hydride ($U\text{-}ZrH_{1.55}$) is relatively small when compared to other types of fuels, such as ceramic fuel. Meanwhile, the use of uranium for a fuel uranium hydride ($U\text{-}ZrH_{1.55}$) is relatively small when compared to other types of fuels, such as ceramic fuel. The advantages of this uranium hydride fuel have a direct impact on economic value. The investment values of hydride fuel are relatively lower and economical than ceramic type fuel [5].

The PWR design using $U\text{-}ZrH_{1.55}$ fuel is proposed by General Atomic from the USA and is known as the TPS (TRIGA Power System) reactor, wherein the design the reactor generates 64 MWt/16.4 MWe power and uses a low-enriched uranium 235U of 19.9% [6]. The development of uranium zirconium fuels in addition to obtaining PWR fuel is also intended can be further developed to fuel the fourth generation reactor. In this experiment, was performed making of $U\text{-}ZrH_{1.55}$ pellets at varies Zr content. It is assumed that various Zr content in $U\text{-}ZH_x$ alloy pellets will affect the properties possessed, such as density and temperature transition changes. This research aims to master the technology of making uranium zirconium hydride ($U\text{-}ZrH_{1.55}$) pellets which can be further processed into sintered pellets that meet the specification requirements as PWR fuel.

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Changes in lattice parameters can be derived from Vegard's law [7], which assumes that solid lattice parameters are linear atomically concentrated. Meanwhile, the thermal properties of nuclear fuel (in this case a temperature change transition) will also affect the performance of nuclear fuel. This research is aimed to obtain the elemental composition, density, and thermal properties of $U\text{-}ZrH_{1.55}$ fuel for PWR reactors. The results of this research are expected to be used as the material for consideration of future PWR reactor fuel development.

II. MATERIALS AND METHODS

The metals uranium (U) and Zr were cut into pieces and inserted into an electric arc smelting furnace for was melted. Melting process was conducted at 150 A and cooled with water cooling. Zr metal compositions that were melted respectively were 35%, 45% and 55% Zr. The U-Zr alloy smelting result is an ingot with a diameter of 15 mm and a thickness of 0.5 mm is then made powder by means of hydriding technique. The hydriding process begins by cutting the U-Zr ingot, and the U-Zr pieces are inserted into a glass container then fed into a retort tube on the hydriding unit for further hydriding process. The hydriding process was conducted at a temperature of 350 ^oC and vacuum condition (pressure 10^{-5} bar). The hydriding process produces a fragile and easily crushed $U\text{-}ZrH_{1.55}$ alloy by pounding it into a powder.

Part of powders have analyzed the composition, temperature transition and other parts were made become pellets. To make the pellet, a $U\text{-}ZrH_{1.55}$ powder is inserted into the mold with a certain weight and pressed with press pressure reaches 20 ton/cm^3 to become pellet $U\text{-}ZrH_{1.55}$. The pellet of $U\text{-}ZrH_{1.55}$ obtained further was measured the dimensions and its weight. From the dimensions and weight obtained can be known its density. Density measurements are carried out using a piece of pycnometer equipment. To measure the transition temperature of the pellets, some of the $U\text{-}ZrH_{1.55}$ powder to be pressed was taken for temperature transition testing using Differential Thermal Analysis (DTA) equipment. The DTA testing was conducted from 30 ^{o}C until 1000 ^{o}C.

III. RESULT AND DISCUSSION

In this discussion, a chapter will discuss among others: the results of chemical composition analysis, density testing, and thermal testing (temperature change transition).

3.1. Chemical Composition Analysis

The experimental results of the chemical analysis of pellet of U-$ZrH_{1.55}$ powder are shown in the form of figure and tables. Table 1 shows the results of chemical composition analysis using atomic absorption spectrometer equipment (AAS) to determine the impurities contained in the pellet of U-$ZrH_{1.55}$ alloys. Table 1 shows that some of the existing elements exceed the specifications /requirements for nuclear fuel, such as elements Ni, Mg, Cd, Zr and K. The Ni element is present in the pellets of U-35$ZrH_{1.55}$ and U-45$ZrH_{1.55}$ samples which both exceed the permitted specifications that are 51.8094 ug/g and 39.3718 ug/g, respectively, while the specification is 30.0 ug/g. For the Mg element, only in U-35$ZrH_{1.55}$, the amount is 76.3551 ug/g while the allowable specification is 50.0 ug/g. Other elements such as Cd are present on U-35$ZrH_{1.55}$ and U-35$ZrH_{1.55}$ where each of them is 0.4508 ug/g and 0.3667 ug/g while the specification is 0.2 ug/g. Meanwhile, the elements Zr and K are each found in pellets of U-35$ZrH_{1.55}$ and U-45$ZrH_{1.55}$ where the two elements are not wanted existence. The presence of impurity elements exceeding those specifications is thought to arise from the equipment used during the process from the process of cutting, smelting and crushing to powder. From the three test samples, only the sample of the pellet of the U-55$ZrH_{1.55}$ test is relatively small content of impurities (only Cd elements). From the existing impurity elements, the presence of Cd elements that need to be considered because it has a high microscopic cross-section compared to another element. The microscopic neutron absorption cross-section Cd is 2520. (50.) Barn [8]. If the value of microscopic neutron absorption cross-section such a high enough it will absorb a large enough amount of neutrons. This situation will cause the operation of the reactor to be disturbed [9]. If it is seen from among the samples test, it is seen that Co and Si elements in pellets of U-35ZrH_{155} and U-45ZrH_{155} alloys are detected, while U-55ZrH_{155} alloy is undetected. Meanwhile, Zn, Mo, V, and Sn elements that present in pellets of U-35ZrH_{155} and U-45ZrH_{155} alloys were detected, while in the U-55ZrH_{155} is undetected. In the U-$ZrH_{1.55}$ powder analysis, the results obtained as listed in Table 2. The results of the analysis listed in Table 2 show that the U content of each pellet of U-35$ZrH_{1.55}$; U-35$ZrH_{1.55}$ and U-55$ZrH_{1.55}$ powder respe-ctively 62.9100; 54.5645; and 44.1308 % weight. The difference is relatively small when compared with U content as planned e.g for U-35$ZrH_{1.55}$; U-35$ZrH_{1.55}$; and U-55$ZrH_{1.55}$ respectively of 3.0770; 0.7918; and 1.9310%. To become attention to the results of the U content analysis can be said that the results of the U content analysis approached the planned results.

Tabel 1. Chemical analysis result of impurity in U-$ZrH_{1.55}$ powder

No	Elemennts	U-35$ZrH_{1.55}$ alloy	U-35$ZrH_{1.55}$ alloy	U-55$ZrH_{1.55}$ alloy	PWR fuel specification
1	Al	0.0350 ug/g	0.0129 ug/g	0.3463 ug/g	≤ 50.0 ug/g
2	Ni	51.8094 ug/g	39.3718 ug/g	0.1100 ug/g	≤ 30.0 ug/g
3	Fe	7.6974 ug/g	7.8643 ug/g	0.326 ug/g	≤ 100.0 ug/g
4	Co	0.6616 ug/g	0.7288 ug/g	ttd	≤ 75.0 ug/g
5	Mn	2.5973 ug/g	2.3093 ug/g	0.0049 ug/g	≤ 10.0 ug/g
6	Pb	0.1597 ug/g	0.1652 ug/g	ttd	≤ 60.0 ug/g
7	Mg	76.3551ug/g	10.5672 ug/g	0.0379 ug/g	≤ 50.0 ug/g
8	Cu	14.3724 ug/g	10.0067 ug/g	0.0600 ug/g	≤ 20.0 ug/g
9	Cr	35.5799 ug/g	33.7893 ug/g	0.0797 ug/g	≤ 100.0 ug/g
10	Zn	-	-	0.4309 ug/g	≤ 100.0 ug/g
11	Cd	0.4508 ug/g	0.3667 ug/g	0.556 ug/g	≤ 0.2 ug/g
12	Mo	-	-	0.0294 ug/g	≤ 50.0 ug/g
13	Si	0.0858 ug/g	0.0082 ug/g	- ug/g	≤ 60.0 ug/g
14	V	-	-	-	≤ 5.0 ug/g
15	Ca	0.0301 ug/g	0.0269 ug/g	1.9281 ug/g	≤ 50.0 ug/g
16	Sn	- ug/g	- ug/g	0.0294 ug/g	≤ 50.0 ug/g
17	B	- ug/g	- ug/g	- ug/g	≤ 0.3 ug/g
19	K	5.8453 ug/g	3.4075 ug/g	- ug/g	- ug/g

Table 2. The analysis result of U content in U-$ZrH_{1.55}$ powder alloy

No.	Analysis result		
	U-35$ZrH_{1.55}$ (% weight)	U-45$ZrH_{1.55}$ (% weight)	U-55$ZrH_{1.55}$ (% weight)
1	62.641	51.678	43.8396
2	62.461	55.069	44.6955
3	62.736	55.484	45.9063
4	63.464	55.581	43.1298
5	63.247	55.011	43.0828
Average	62.910	51.678	44.131
Planned	65.000	54.564	45.000
The difference between the planned	3.077	0.792	0,869

Analysis result

3.2 Density

On the measurement of pellet dimensions manually obtained dimensions as shown in Table 3. Table 3 shows the results of height (T), diameter (D), weight (W) pellet and calculated density. Calculation of density based on pellet dimension size, and from calculation of density obtained density value pellet respectively are U-35$ZrH_{1.55}$, U-35$ZrH_{1.55}$, and U-55$ZrH_{1.55}$ is 6.70525; 5.5675; and 4.8420 g/cm^3. In the density measurement using pycnometer equipment, the density values for pellets of U-35$ZrH_{1.55}$; U-35$ZrH_{1.55}$ and U-55$ZrH_{1.55}$ respectively are 9.9141; 7.9920; and 7.0359 g/cm^3. Meanwhile, the theoretical density values for pellets of U-35$ZrH_{1.55}$, U-35$ZrH_{1.55}$ and U-55$ZrH_{1.55}$ respectively are 14.61, 13.4, and 12.163 g/cm^3. Meanwhile, the theoretical density values for U-35$ZrH_{1.55}$, U-35$ZrH_{1.55}$ and U-55$Zr_{H1.55}$ respectively are 14.61; 13.4; and 12.163 g/cm^3. Measurement of density either through manual by measuring dimensions and weight or by using pycnometer equipment shows that both of the values of U-ZrH1.55 density that obtained are lower when the Zr content is more height. This condition is caused by the lower Zr density than the U density, so when the Zr content gets larger then the density of the U-$ZrH_{1.55}$ alloy becomes lower. In the density measurement using pycnometer equipment, the densities U and Zr respectively are 19.000 g/cm^3 and 6.511 g/cm^3. The difference in density values obtained from the measurements using pycnometer equipment rather than manual measurement. This situation is due to the lack of accuracy of the manual manner resulting in less precise dimensions of the heights and diameter of the pellet. The result can affect the density calculation, which is not right as well. There are the different results obtained from density measurements using pycnometer equipment whereon measurement the equipment uses helium gas (He). Helium (He) gas that flows into the pellets gives the result of pellet density more height. This fuel is expected to have a high density approaching the theoretical density value.

Table 3. Dimension measurement result and calculation of pellets

Alloy	P (MP)	H (mm)	D (mm)	W (g)	ρ (g/cm^3) Average (Calcula-ted)	ρ (g/cm^3) Average (Calcula-ted)	ρ (g/cm^3) (Theoreti-cal)	Different with theo-retical density (%)
U-35$ZrH_{1.55}$	4.0000	13.6800	11.1800	9.2602	6.8989	6.7052	14.6100	58,5763
	4.0000	11.7500	11.2000	7.5341	6.5116			
U-35$ZrH_{1.55}$	4.5000	14.7600	11.2300	8.2543	5.6489	5.5675	13.4000	58.4514
	4.0000	9.0500	11.19	4.8802	5.4860			
U-55$ZrH_{1.55}$	4.0000	12.2700	11.20	5.8503	4.8420	4.8420	12.1600	60.1809

Information:

P = Pressure (MP), H = height of pellets, D = diameter of pellets
W = weight (g), ρ = density (g/cm^3)

Table 4. Density testing of pellets

No	Alloy	Density testing (with pycnometer)	Density calculation (theoretical)	Different with theoretical density
		ρ (g/cm^3)	ρ (g/cm^3)	(%)
1	U-35Zr$H_{1.55}$	9.9141	14.6100	32.1416
2	U-35Zr$H_{1.55}$	7.9920	13.4000	40.3582
3	U-35Zr$H_{1.55}$	7.0359	12.1600	42.1389

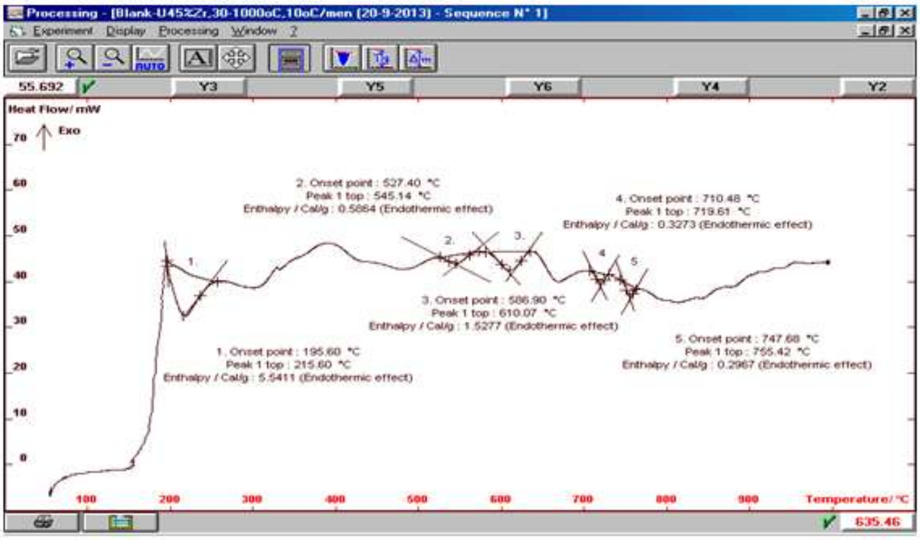
3.3. Transition Temperature Change

The results of the temperature change transition testing using Differential Thermal Analysis (DTA) equipment are shown in Figure 3. a, b, and c, which is a U-Zr$H_{1.55}$ DTA pellets thermogram with variations of Zr content of 35, 45, and 55%. Figures 2. a, b, and c show the formation of endothermic peaks. The measurement results are the amount of enthalpy, melting temperature and the formation of compounds in the presence of changes in heat flow.

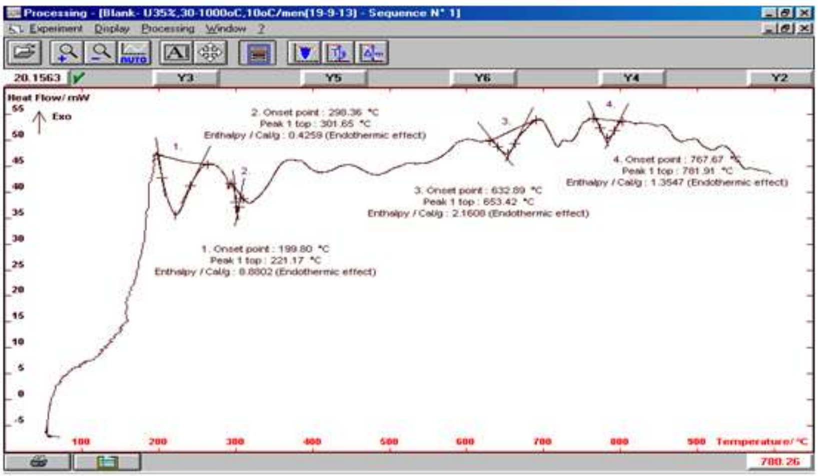
Pellets of U-35Zr$H_{1.55}$ experiencing chemical reactions process of four stages, where the first stage of endothermic reaction that occurs at temperatures 199.80-221.17 ºC without phase change, required heat of 8.8802 cal/g, the second stages of endothermic reaction at temperature 298.36-301.65 ºC no phase change occurs with required heat of 0.4259 cal/g. Both of the above reaction stages have α + δ1 phases. The third stages of endothermic reaction occurs at temperature of 632.89-653.42 ºC there is a phase change from α + δ1 to α + γ Zr that need of heat of 2.1608 cal/g, and the fourth stage occurs endothermic reaction at temperature of 767.67-781.91 ºC there is phase change as much as three times that of α + γZr become β + γZr, from β + γ Zr become γ + γZr, then from γ + γZr to γ that need of heat of 1.3547 cal /g. In the pellet of U-45ZrH1.55 alloy, the reaction takes place in the five stages of phase change, where the first stage reaction occurs at temperature of 195.60-215.60 ºC and no phase change which requires heat of 5.5411 cal/g, the second stages of endothermic reaction occurs at temperature 527.40-545.14 ºC and there is no phase change takes place heat of 0.5864 cal/g. Both of the above reaction stages have α + δ1 phases. The third stages of endothermic reaction occurs at temperature 586.90-610.07 ºC and there is phase change α + δ1 become α + γZr that need of heat equal to 1.5277 cal/g, fourth stages happened endothermic reaction at temperature 710,48-719.61 ºC accompanied by a three-phase change of α + γ Zr to become b + γZr , from b + γZr to γ + γZr, and from γ + γZr to γ by requiring heat of 0.3273 cal/g. The fifth stage, an endothermic reaction occurs at temperature 747.67 - 755.4 no phase change (fixed) and require heat of 0,2967 cal/g. In the fifth stages, the heat of the reaction is used to increase the temperature. Meanwhile, the U-55Zr$H_{1.55}$ alloy undergoes a reaction four stages, where the first stage of the endothermic reaction takes place at a temperature of 191.96-210.36 ºC and no phase change and require the heat of 16.6969 cal/g, the second stages of reaction endothermic that occurred at temperature 290.25–295.87 ºC no phase change with heat required of 0.2098 cal/g. The absence of phase change in the above process is due to the heat formed in the above two reactions is used to increase the reaction temperature. The third stages occur exothermic reaction at temperature 324.59-327.02 ºC and no change of phase with the release of heat equal to -0,3407 cal/g. The three reactions above have α+δ phases. The fourth stages happened endothermic reaction at temperature 742.24 -788.04 ºC happened change phase of four stages that are α+δ1 becomes α+ γZr, a+ γZr becomes β + γZr from β + γZr becomes γ+ γZr, and from γ+ γZr becomes γ with a need of heat equal to 8.5834 cal/g. Looking at the chemical reactions that occur and their relation to the phase change it can be said that the overall reaction in each U-Zr$H_{1.55}$ alloy requires different heat. Considering the U-Zr$H_{1.55}$ phase diagram[10], [11],[12], the required heat and the phase change occurring that of all three alloys in the U-Zr$H_{1.55}$ alloy that tested looked that the U-35Zr$H_{1.55}$ alloy requires the lowest heat to transform from phase α + δ1 to γ phase if it compared with the others. The three alloys that are U-35Zr$H_{1.55}$, U-45Zr$H_{1.55}$, and U-55Zr$H_{1.55}$ requiring reaction heat of respectively equals 12.8216 cal/g; 8.2792 cal/g; and 25.1494 cal/g. It can be said that when used as a fuel in terms of its thermal properties (transformation of heat changes), the U-35Zr$H_{1.55}$ alloy can be the best choice. In Table 5, can be seen the relationship between reaction temperature, phase change, and heat is needed or absorbed.

Table 5. The relationship between reaction temperature, phase change, and heat is needed or absorbed

No	Alloys	Phases	phase change temperature (°C)	Heat needed/ Released Cal/g	Information
1	U-35ZrH$_{1.55}$	a. α + δ1 b. α + δ1 c. a + γ d. γ	a. 199.80-221.17 b. 298.36-301.65 c. 632.89-653.42 d. 767.67-781.91	a. 8.8802 b. 0.4259 c. 2.1608 d. 1.3547	a. Endothermic b. Endothermic c. Endothermic d. Endothermic
2	U-45ZrH$_{1.55}$	a. α + δ1 b. α + δ1 c. a + γZr d. γ e. γ	a. 195.60-215.60 b. 527.40-545.14 c. 586.90-610.07 d. 710,48-719.61 e. 747.67 - 755.4	a. 5.5411 b. 0.5864 c. 1.5277 d. 0.3273 e. 0,2967	a. Endothermic b. Endothermic c. Endothermic d. Endothermic e. Endothermic
3	U-55ZrH$_{1.55}$	a. α + δ1 b. α + δ1 c. α + δ1 d. γ	a. 191.96-210.36 b. 290.25 – 295.87 c. 324.59-327.02 d. 742.24 - 788.04	a. 16.6969 b. 0.2098 c. - 0,3407 d. 8.5834	a. Endothermic b. Endothermic c. Exothermic d. Endothermic



a.



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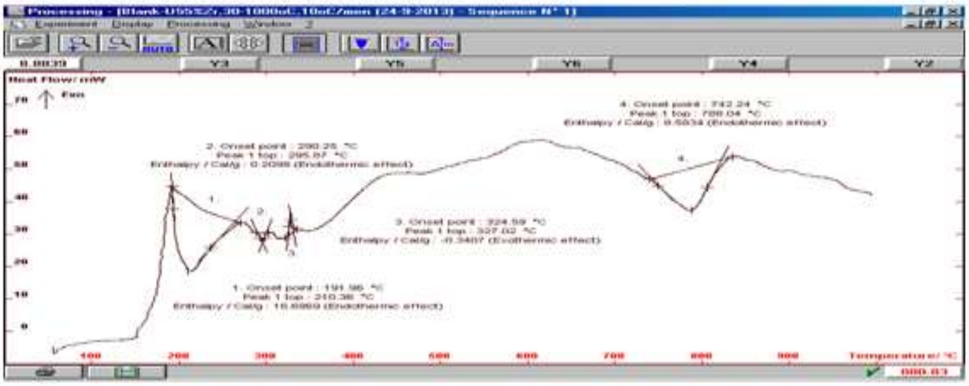

c.

Figure 1. The curve of a testing result of temperature change transition with DT equipment
a. $U\text{-}35ZrH_{1.55}$, b. $U\text{-}45ZrH_{1.55}$, and c.$U\text{-}55ZrH_{1.55}$

IV. CONCLUSION

The result of analysis of element composition, density and thermal properties of pellets of $U\text{-}35ZrH_{1.55}$, $U\text{-}35ZrH_{1.55}$, and $U\text{-}55ZrH_{1.55}$ can be concluded that the result of chemical composition test on all pellets samples obtained that for pellets of $U\text{-}35ZrH_{1.55}$ and $U\text{-}45ZrH_{1.55}$ contains some elements of impurities that exceed the allowable limits include elements Ni, Mg, Cd, Zr, and K, while for the pellet of $U\text{-}55ZrH_{1.55}$ only Cd elements that exceed the limit. Looking from the density, all tested pellets have a lower density value than the theoretical density either from manual measurement or using a pycnometer. The result of temperature transition testing using DTA obtained that pellets of $U\text{-}35ZrH_{1.55}$ and $U\text{-}55ZrH_{1.55}$ have four times endothermic reaction stages while for $U\text{-}35ZrH_{1.55}$ have five times endothermic reaction stages. During endothermic reactions, all pellets undergo a phase change from the original phase $\alpha + \delta 1$ into phase γ at the end of the reaction.

ACKNOWLEDGMENT

On this occasion, I would like to thank my friends in the field of BFBBR who have helped me do my research until becoming a manuscript. My acknowledgment especially to Mr. Suyoto, S.ST, Mr. Yatno D.A, Mr. Mugiyono, Mrs. Mujinem, and Mrs. Asminar.

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Masrukan Masrukan" Composition Analysis Of Elements, Density, And Thermal Properties Of The Pellet Of U-Zrh1.55 Alloys" International Journal of Modern Engineering Research (IJMER), vol. 09, no. 4, 2019, pp 01-07