

IDENTIFIKASI BAHAN KELONGSONG PADUAN ZrNbMoGe HASIL UJI HIDRIDASI 400°C - 1 JAM MENGGUNAKAN TEKNIK DIFRAKSI

IDENTIFICATION ON ZrNbMoGe ALLOY OF 400°C-1 HOUR HYDRIDATION TEST FOR CLADDING MATERIALS BY USING DIFFRACTION TECHNIQUE

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ABSTRAK

Sintesis paduan zirkonium yang mengandung unsur Nb (ZrNbMoGe) telah dilakukan di PSTBM-BATAN. Dalam reaktor PWR/PHWR, bahan ini bersentuhan langsung dengan cairan pendingin, yakni: H₂O demin yang bisa memicu reaksi antara Hidrogen dan kandungan zirkonium yang reaktif dalam paduan. Uji hidridasi dalam aliran gas H₂ beratmosfir gas argon dilaksanakan untuk memprediksi kelakuan paduan ZrNbMoGe pada temperatur tinggi. Peristiwa hidridasi Zr-H sangat dihindari karena peristiwa *blistering* yang mampu merapuhkan bahan. Sampel paduan dengan kandungan 2%Nb, 0,5%Mo, 0,5%Ge and Zr dalam kesetimbangan telah diuji hidridasi dalam aliran gas H₂ pada temperatur 400°C selama 1 jam. Pengukuran pola difraksi menggunakan difraktometer sinar-X dengan target Cu. Puncak baru muncul pada sudut $2\theta=20,95^\circ$. Interpolasi matematis dilakukan via regresi polinomial orde-2. Identifikasi dan pencocokan profil menggunakan telusuran JCPDS "search and match". Hasil memperlihatkan bahwa fasa hidridasi (400°C-1 jam) sangat dekat dengan struktur kristal ϵ -ZrH₂. Kristalografi fasa tersebut memiliki parameter kisi $a= b= 3,52 \text{ \AA}$ dan $c= 4,45 \text{ \AA}$ (struktur tetragonal) dengan refleksi pertama bidang (201) pada sudut $2\theta=21,60^\circ$. JCPDS juga menampilkan fasa lain berstruktur ortorombik pada sudut $2\theta=20,91^\circ$ dengan parameter kisi $a= 9,183 \text{ \AA}$, $b= 22,47 \text{ \AA}$ and $c= 4,24 \text{ \AA}$ dengan bidang refleksi (220). Fasa ini diduga merupakan lapisan anti-hidrid (*passivatif*) yang mampu menghalangi penggetasan bahan kelongsong akibat proses hidridisasi dalam reaktor.

Kata kunci : teknik difraksi, paduan ZrNbMoGe, temperatur tinggi, hidridasi.

ABSTRACT

Synthesis of a new niobium containing zirconium alloy of ZrNbMoGe has been developed in PSTBM-BATAN. In PWR/PHWR reactor, this materials face with liquid coolant i.e. H₂O demin that may trig a reaction between hydrogen and containing zirconium. Hydrization tests in argon atmosphere were recently carried out looking for hydrogen effect in material. Hydrogenisation must be avoided due to causing brittle of the material via blistering of Zr-H compound system. Sample with 2%Nb, 0.5%Mo, 0.5%Ge and Zr in balance content has been hydridized by flowing H₂ at temperature of 400 °C for an hour. Effects of the test by time-high temperatur dependence on the zirconium content were investigated by X-ray diffractometer. The observed peak at $2\theta=20.95^\circ$ was mathematically interpolated by orde-2 polynomial regression. Identifying peak was performed by X-ray diffraction technique and then it conformed 'search and match' by JCPDS tracing. Results show that new phase in 400°C-1 hour hydridation sample was close to crystal structure of ϵ -ZrH₂. The crystallographic data emphasize that new phase

with lattice parameter of $a= b= 3.52 \text{ \AA}$ and $c= 4.45 \text{ \AA}$ (tetragonal) is (201) plane or $2\theta=21.60^\circ$. Also, the JCPDS traces another new orthorhombic phase of $\text{Nb}_3\text{H}_3\text{O}_9$ at angle of $2\theta=20.91^\circ$ with lattice parameter of $a= 9.183 \text{ \AA}$, $b= 22.47 \text{ \AA}$ and $c= 4.24 \text{ \AA}$ conforming to (220) plane. Hypothesized, this is an anti-hydride layer (passivity) that can overcome hydridation process of cladding materials in the reactor.

Keywords : diffraction techniques, ZrNbMoGe alloy, high temperature, hydridation

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INTRODUCTION

During the ongoing operation of PWR-type reactors, the mechanical properties of zirconium alloy cladding material used, can be degraded with respect to the combination of oxidation process, hydridation and embrittlement due to radiation. Corrosion on the outer surface of the cladding caused by the cooling water may take the form of hydrogen absorption capable of forming a second phase in the form of hydride phase. For Zircalloy-4 the rate of the absorbed free hydrogen from corrosion reaches 10-20%. At room temperature, the solid solubility limit of hydrogen in Zircalloy-4 is less than 10% wt ppm, but reaches 100% wt ppm at the reactor operating temperatures of around $320 \text{ }^\circ\text{C}$ ¹⁾. In the zirconium metal cladding material, it was found that hydrides have formed the δ -hydride phase having a face centered cubic structure. The phase type and the quantity of hydrogen, in addition of being influenced by the ambient temperature and the hydrogen content, the cladding materials are also highly influenced by the composition of the alloy element cladding material. Hydrogen and hydride induced degradation of the mechanical and physical properties of Zirconium-based alloys. Also, corrosion of nuclear fuel cladding results into the generation of hydrogen. With operation time increase, the hydrogen concentration in the cladding may exceed its terminal solid solubility and brittle zirconium hydrides may precipitate. Indeed hydrides are present in high burnup fuel cladding, which is therefore more susceptible to failure²⁾

Knowledge of hydrogen concentration is needed when assessing the response of zirconium alloys to hydrogen. The hydrogen concentration is critical to the tests; too much hydrogen may cause hydrides to be present at the peak temperature reached before the lower test temperature is attained, while too little hydrogen may provide insufficient hydrogen for hydride precipitation during testing and give a false, low crack velocity³⁾.

Theorem of hydrogen entering the cladding is clearly established by literatur⁴⁾.

In this research, the initial study consists of phase identification of the hydride formed in the new ZrNbMoGe alloy⁵⁾ made for PWR reactor cladding materials. Identification of the hydride phase in the cladding material can be accomplished by hydrides test and phase identification using X-ray diffraction measurements (XRD-610). ZrNbMoGe cladding materials have been developed at PTBIN with some variation of the alloy composition⁶⁾ and, and the authors were capable of achieving good mechanical properties such as high hardness and strength as well as good corrosion resistance in the cooling water environment and high temperature corrosion due to oxidation. Some tests related to the structural integrity and crack resistance was carried out for the analysis of the integrity of the alloy material. The type and quantity of hydride phase in the cladding material will be the initial data to measure the resistance of the cladding material from hydridation which is an important parameter in maintaining the integrity of the cladding material. Some characteristics of ZrNbMoGe have been observed and reported, i.e.: crystal structure and hardness⁷⁾, tensile strengths⁸⁾, residual stresses⁹⁻¹⁰⁾ and oxidation resistant¹¹⁾.

METHODOLOGY

The alloy samples used in the the hydration test are obtained from ingots synthesized by melting the alloy elements in the electric arc furnace. Alloying elements consist of sponge zirconium (Zr) nuclear grade with a purity of 99.97%, Niobium (Nb) in the form of rod/wire 99.6% and Molybdenum (Mo) and Germanium (Ge) in the form of sponge with purity of each of the above 99 %. The composition of the test sample in weight per cent (wt%) are: 97% Zr, Nb 2%, 0.5% Mo and 0.5% Ge. Smelting was carried out at temperatures above 1850°C within the confinement of argon gas to

prevent oxidation, and re-smelting carried out four times to produce a homogeneous sample. Furthermore, the cutting process is carried out in order to obtain square disc-shaped ingot samples with a 1.0 x 1.0 cm cross section and a thickness of 1.0 mm and continued with rinsing the samples with acetone. Samples that were mounted inside the test equipment are shown in Figure 1.

The testing apparatus consists of the following main components, namely a quartz test tube, a heating furnace, a thermocouple, hydrogen and argon gas cylinders. Samples are mounted on or inserted into the sample boat made of a ceramic material and then put in a test tube. Next, hydrogen gas containing 30% H₂ and argon gas are allowed to flow into the quartz tube in which the sample was confined, and then heated to a temperature of 400° C for 1 hour in a furnace. After this testing procedure is concluded, the sample was allowed to cool down until it reached room temperature and then the characterization of the microstructure and diffraction test for the analysis phase were carried out.

Measurements of the X-ray diffraction pattern of the test sample was carried out utilizing the following measurement parameters: wavelength of Cu-K (1.5405 Å), an angular range of $5^{\circ} \leq 2\theta \leq 75^{\circ}$, a step counting of $\Delta 2\theta = 0.05^{\circ}$, preset time 2 seconds, performed in the step-counting mode. Alloy crystal structure analysis was performed using the Rietan (Rietveld Analysis) program [10].

RESULTS AND DISCUSSION

In Figure 2 is shown the diffraction pattern of the alloy material ZrNbMoGe neutron scattering scanning results using the High Resolution Powder Diffractometer (HRPD) at PTBIN -BATAN in Serpong, with a wavelength of 1.8195Å. The significant penetration power of the neutron beam made it possible to have a satisfactory peak to background ratio in the neutron beam intensity pattern profile.

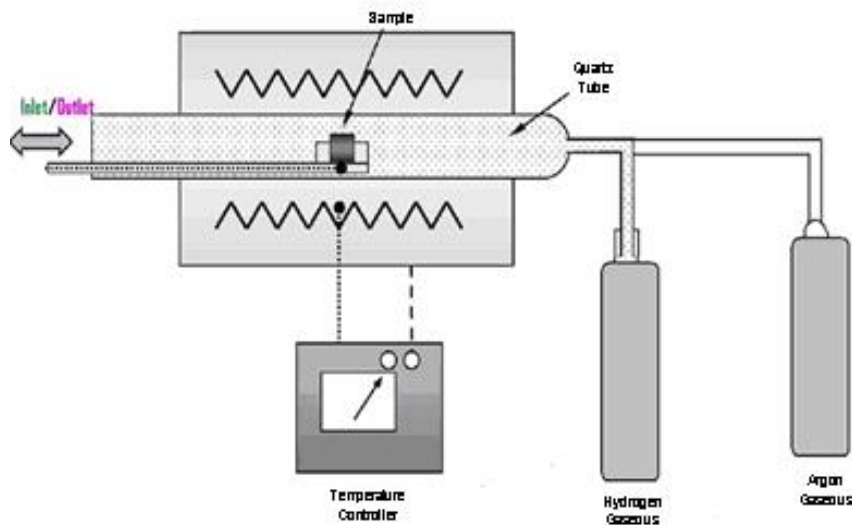


Figure 1.
Schematic of hydridation test on the materials.

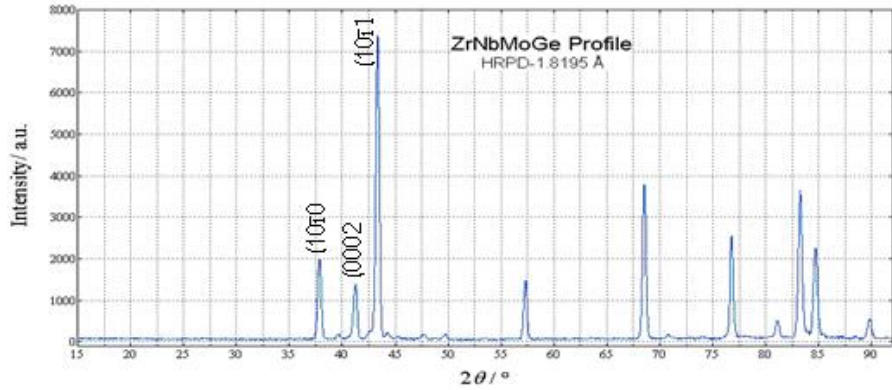


Figure 2.

High Resolution Powder Diffractometer (HRPD) neutron diffraction pattern of ZrNbMoGe alloy before hydridation using a wavelength of 1.8195 Å.

The difference in the intensity of the diffraction peaks is very clear and sharp, and as shown in the diagram the reflection intensity appears as pointy needles. The whole pattern of the diffraction peak is dominated by the zirconium cladding phase as the primary matrix material. The first three main peak of the dominant phase are from the (10 0), (0002) and (10 1) diffraction planes appearing at successive 2θ diffraction angles of 37.85° , 41.30° and 43.34° . The highest intensity of 7,374,663 counts was observed for the (10 1) diffraction plane

belonging to the zirconium phase. Initial diffraction pattern showed only four phases are formed before the sample is subjected to hydration treatment, namely: the zirconium (Zr) hexagonal structured phase, the Zr_3Ge tetragonal structured phase, the orthorhombic structured ZrGe phase and the cubic structured $ZrMo_2$ phase, as was reported previously by Parikin *et al.* [7]. Hydride phase does not appear in the profile of the diffraction pattern in the angular range of $5^\circ \leq 2\theta \leq 35^\circ$, and the curves lines are very smooth as shown in Figure 2.

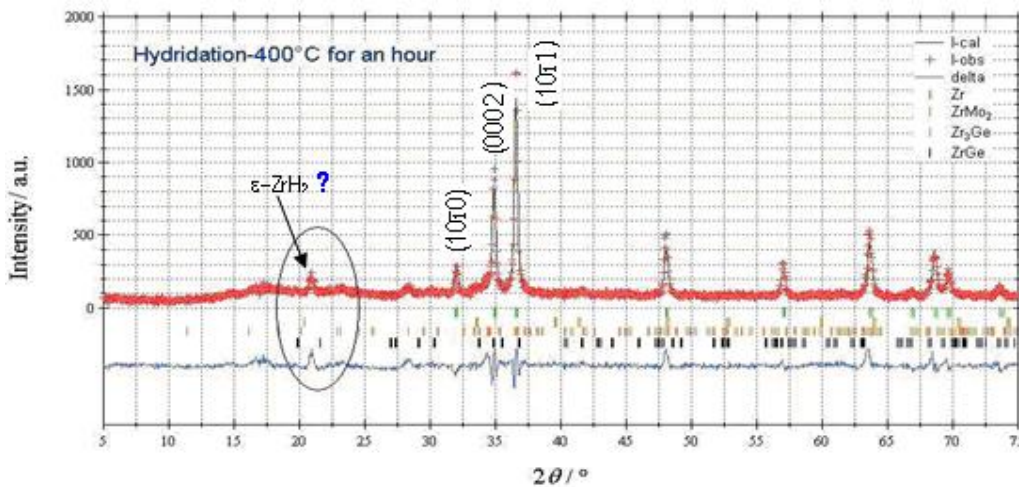


Figure 3.

Diffraction pattern with Rietveld refinements on ZrNbMoGe alloy resulting of X-ray Diffractometer (XRD) scanning, 1.5405Å in wavelength.

Hydridation test treatment for one hour at a temperature of 400 °C is suspected to cause the material to be contaminated by hydrogen gas. Figure 3 shows the pattern of the four-phase profile refinement process to trace the materials' crystalline phase; This pattern is the result of Rietveld Analysis code refinement of the X-ray diffractometer

measurement with Cu-K α target ($\lambda = 1.5405 \text{ \AA}$) [12]. The main peak of the first three phases of zirconium have been indexed as the (10 0), (0002) and (10 1) diffraction planes, and appear to be shifted to the left at the diffraction angles $2\theta = 32.00^\circ$, 34.90° and 36.55° respectively, which is caused by

the deviation in the wavelength of the diffractometer.

In the diffraction diagram shown in Figure 3, a foreign peak appears at approximately $2\theta = 20.00^\circ$ angular position, which does not belong to either one of the four phases present in the materials: the hexagonal zirconium (Zr) phase (green), the cubic $ZrMo_2$ phase (orange), the tetragonal Zr_3Ge phase (pink) and the orthorhombic $ZrGe$ phase (black). Considering that during

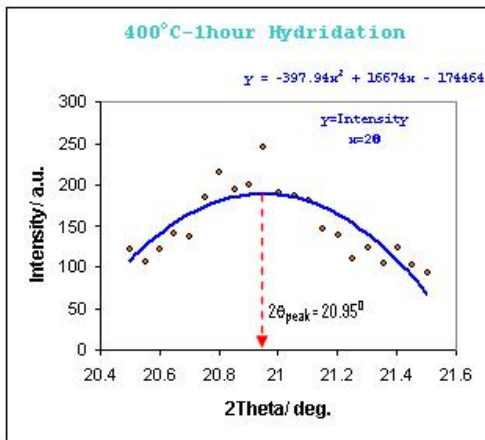


Figure 4.

2nd order of polinomial interpolation on a peak plane of suspected hydride phase resulting of X-ray Diffractometer (XD-610) scanning.

The analysis of the peak positions is then concentrated in the angular diffraction range of $20.50^\circ \leq 2\theta \leq 21.50^\circ$, in order for the curve fitting process to be done through the process of diffraction data interpolation. The results of the analysis of 2nd order polynomial curve give the tendency of X-ray scattering data to follow the linear equation:

$$y = -397.94 x^2 + 16674 x - 174464 \quad (1)$$

where y is the intensity (au) and x = is the 2θ diffraction angle ($^\circ$). From the equation of the curve, further differentiation of level-1 is carried out to obtain:

$$(dy/dx) = -795.88 x + 16674 \quad (2)$$

which is the gradient of the curve line. According to a mathematical theorem, a

the treatment of the sample, 30% H_2 hydrogen gas and argon gas simultaneously flowed into the quartz tube and heated to a temperature of $400^\circ C$ for 1 hour, it is safe to assume that a new phase was formed in the form of crystalline zirconium hydride (ϵ - ZrH_2). This particular hydride phase is to be avoided because it causes embrittlements of the cladding material and would ultimately risk failure of reactor operation.

curve reaches its peak, when the curve crosses the abscissa point on the same zero line gradient. Therefore, when $(dy/dx) = 0$, then one obtains the value of $x = 20.95^\circ$, or the peak's diffraction angle is located at the position $2\theta = 20.95^\circ$. This mathematical procedure to search for the diffraction peak's angular position is illustrated in Figure 4.

Figure 5 shows a JCPDS reference table [13] by incorporating elements of Zr and H atoms as the main constituents of the crystal. Provided that the nearest diffraction angle of ϵ - ZrH_2 phase is $2\theta = 21.622^\circ$, as measured by X-ray diffraction at a wavelength of 1.5418 \AA . However, recalculation using a wavelength of 1.5405 \AA through Bragg law formulation [14]:

$$\lambda = 2 d_{hkl} \sin \theta \quad (3)$$

then the angular shift of this diffraction plane is at the reflection angle $2\theta = 21.60^\circ$ and has an interplanar distance of $d = 4.11 \text{ \AA}$.

Further observations on the angle of this peak is compared with the results of interpolation angle $2\theta = 20.95^\circ$, then the angular shift difference ($\Delta 2\theta$) amounted to 0.65° . This figure has an uncertainty of ± 0.01 which is large enough so that it is doubtful that the diffraction peaks belongs to the hypothesized phase. However, because in the hydridation test, the sample is heated to a temperature of $400^\circ C$ for 1 hour and flushed with both hydrogen gas and argon gas in a sealed quartz tube, the largest likelihood is that the materialized compound is a zirconium hydride compound. In this case the tetragonal ν - ZrH_2 phase with lattice parameters $a = b = 3.52 \text{ \AA}$ and $c = 4.45 \text{ \AA}$ is the nearest candidate among an array of probable hydride compounds that could have been formed.

Wavelength: 1.5418

2θ	Int	h	k	l	2θ	Int	h	k	l
21.622	10	2	0	1	113.845	2	3	3	3
24.941	8	1	1	0	122.333	<2	4	4	0
26.689	10	1	0	1	126.314	<2	1	1	5
32.475	100	1	1	1	133.512	4	2	0	5
36.130	30	2	0	0	135.473	2			
40.486	16	0	0	2	136.496	4	4	0	4
51.954	16	2	2	0	141.321	4	6	1	0
55.415	20	3	0	0	143.385	2			
58.407	<2	2	1	2	156.781	4	4	2	4
59.146	2	3	0	1					
62.641	25	3	1	1					
67.865	10	2	2	2					
68.600	10	1	1	3					
73.662	<2	3	1	2					
76.588	2	4	0	0					
79.945	<2	4	0	1					
85.657	6	2	2	3					
87.777	6	4	2	0					
91.089	8	3	1	3					
98.710	2	2	0	4					
101.428	6	2	1	4					
107.981	4	4	0	3					
110.191	2	2	2	4					

Sys: Tetragonal S.G. P4/mmm (139)
 a: 3.52 b: 4.45 c: 0.8974
 Ref: Ibid.
 Deleted by Duplicate Program. Mwt: 93.24 Volume[CD]: 110.17

Figure 5. Table JCPDS for ε-ZrH₂ hydride phase tetragonal structure.

Wavelength: 1.54071

2θ	Int	h	k	l	2θ	Int	h	k	l
7.869	100	0	2	0	41.362	3	1	10	0
12.520	39	1	2	0	42.462	4	4	4	0
15.80	14	0	4	0	43.691	15	3	8	0
18.499	11	1	4	0	46.306	4	4	6	0
19.321	4	2	0	0	48.101	14	1	10	1
20.908	7	2	2	0	48.566	5	0	12	0
23.764	16	0	0	0	49.610	5	1	12	0
23.965	18	0	1	1	50.223	7	3	8	1
24.998	63	2	4	0	51.313	4	2	10	1
25.703	10	1	6	0	51.313	4	4	8	0
25.985	11	1	1	1	52.452	7	2	0	2
26.715	6	1	2	1	52.452	7	5	4	0
27.750	9	2	5	0	52.745	7	2	12	0
28.464	6	0	4	1*	53.477	6	1	5	2
30.261	12	3	2	0	53.953	5	1	13	0
30.784	21	2	6	0	55.076	6	2	4	2
31.841	15	0	8	0	59.301	6	5	5	1
32.592	8	1	5	1	59.979	6	4	9	1
32.938	7	2	3	1	60.368	6	2	7	2
33.329	7	3	4	0	60.586	6	6	1	0
33.329	7	1	8	0					
37.488	4	2	8	0					
37.948	21	3	6	0					
39.221	3	4	0	0					
40.084	8	0	10	0					

Sys: Orthorhombic S.G. Pmmn (58)
 a: 9.183 b: 22.470 c: 3.775 A: 0.1087 C: 0.1680
 Ref: Nedjar, R., Borel, M., Raveau, B. Mater. Res. Bull., 20, 1291 (1985)
 Dx: 3.630 Dm: 3.699 SS/POM: F₂₀ = 1119.031 (87)
 Color: White
 ~Not permitted by space group.
 Silicon used as an external stand. PSC: ofP60 Mwt: 425.74
 Volume[CD]: 778.94

Figure 6. Table JCPDS for Nb₃H₃O₉ phase orthorhombic structure, which is an anti-hydride phase which hinders the formation of zirconium hydride (Zr-H) due to the production of H₂ gas in the core (cupola) reactor.

Another possible explanation of the reflection peak is the phase Oxonium Niobium Oxide (H₃ONb₃O₈ or Nb₃H₃O₉), by an inspection of the JCPDS reference tables these type of compounds are to be found at the diffraction angle $2\theta = 20.91^\circ$, having orthorhombic symmetry with lattice parameters $a = 9.183 \text{ \AA}$, $b = 22.47 \text{ \AA}$ and $c = 3.775 \text{ \AA}$, belongs to the (220) diffraction plane with an interplanar distance of 4.24 \AA . This is in accordance with the calculation formula d_{hkl} through the Bragg angle of the diffraction peaks obtained from interpolation results $2\theta = 20.95^\circ$ and $d_{hkl} = 4.2366 \text{ \AA}$ or rounded up to 4.24 \AA . Figure 6 shows the JCPDS table scan results of this compound.

The first hypothesis is that the process that occurs in laboratory

experiments is less than perfect so there is still moisture from the air which accompanies hydrogen gas and oxidize the surface of the test sample in a test tube. The second hypothesis when the tube is being opened in order to remove the sample, the temperature was still at a sufficiently elevated level, such that the test samples experienced a spontaneous oxidation during cooling in the open air in the hygroscopic laboratory space. But this purely accidental occurrence raises a new hypothesis, that the Nb₃H₃O₉ phase are compounds that can protect the surface of the cladding materials in order for the alloy not to be easily hydridated by the production of hydrogen gas (H₂) in the nucleus (cupola) reactors similar to the passive (Cr_xO_y) layer in stainless steel. This compound is a

salt that can be decomposed into H_3O^+ and stoichiometric Nb_3O_8^- . When the group oxonium (H_3O^+) encountered the H_2 gas in the fuel elements' cupola system, it would be neutralized into H_2O and 3 H^+ ions. These ions are then recaptured by niobium oxide (Nb_3O_8^- or NbO_3^-) on the surface of the cladding material to form Hydrogen Niobium Oxide (Nb_3O_8^- atau NbO_3^-) compounds in a repetitive mode.

CONCLUSION

From the discussions and arguments presented above it can be concluded that: X-ray diffraction to identify the new phase ϵ - ZrH_2 or $\text{Nb}_3\text{H}_3\text{O}_9$ are still in an intense discussion stage at this point (debatable). The identification is based on the reflection angle $2\theta = 20.95^\circ$ mathematical interpolation results, when shifted to the diffraction angle $2\theta = 21.60^\circ$ belonging to the tetragonal ϵ - ZrH_2 phase with lattice parameters $a=b=3.52\text{\AA}$ dan $c=4.45\text{\AA}$ with an interplanar distance of $d = 4.11\text{\AA}$; but when the reflection shifted to the diffraction angle $2\theta = 20.91^\circ$ then it should be identified as the orthorhombic $\text{Nb}_3\text{H}_3\text{O}_9$ phase with lattice parameters $a=9.183\text{\AA}$ $b=22.47\text{\AA}$ dan $c=3.775\text{\AA}$, associated with the (220) diffraction plane and an interplanar distance of 4.24 \AA . In hydridation test where the sample is heated to a temperature of 400 ° C for 1 hour and flushed with hydrogen- and argon gas in the sealed quartz tube, the compound that will be formed with the highest probability is the zirconium hydride ϵ - ZrH_2 phase compound, which is capable to embrittle the cladding. The $\text{Nb}_3\text{H}_3\text{O}_9$ phase compound is an anti-hydride salt compound that is similar to the passivative (Cr_xO_y) layer in stainless steels and can be decomposed into H_3O^+ and Nb_3O_8^- . oxonium (H_3O^+) cluster to neutralize the H_2 gas in the fuel elements cupola system which is then dissociated into H_2O and H^+ ions.

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