

Urania

Jurnal Ilmiah Daur Bahan Bakar Nuklir

Beranda jurnal: <http://jurnal.batan.go.id/index.php/urania/>



STUDY OF FISSION GAS BUBBLES AND INTERACTION LAYER ON IRRADIATED U_3Si_2 -Al DENSITY OF 4.8 gU/cm^3

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(Naskah diterima: 29-05-2022, Naskah direvisi: 19-06-2022, Naskah disetujui: 28-06-2022)

ABSTRACT

STUDY OF FISSION GAS BUBBLES AND INTERACTION LAYER ON IRRADIATED U_3Si_2 -Al DENSITY OF 4.8 gU/cm^3 . Uranium-silicide compound fuel dispersed in aluminium matrix (U_3Si_2 -Al) have been used in a large number of research reactors around the world because of its excellent behavior under irradiation. This fuel also provides high uranium density with typical fuel loading up to 4.8 gU/cm^3 to compensate for the reduced fissile amount in LEU. To improve the density of current U_3Si_2 -Al (2.96 gU/cm^3) used in Indonesian GA Siwabessy Multipurpose Research Reactor, U_3Si_2 -Al dispersion fuel plate with density of 4.8 gU/cm^3 ($U^{235} \sim 19.75\%$) had been irradiated in RSG GAS for 175 days at 15 MW power to burnup level of approximately 40%. The characterization was performed using SEM-EDS and optical microscope to study microstructure of the irradiated fuel, largely the fission gas bubbles and the interaction layer between U_3Si_2 fuel and Al matrix. The average diameter of the bubbles with diameter from 0.06 to $0.55 \mu\text{m}$ was $0.21 \mu\text{m}$. The interaction layer was identified as $U(Al,Si)_{2,3}$ with thickness of approximately $1.5 \mu\text{m}$. The relatively small fission gas bubbles and the interaction layer didn't cause swelling on the fuel and the overall performance of the fuel plate was very good.

Keyword: LEU, uranium-silicide, post-irradiation examination, interaction layer, fission gas bubbles.

ABSTRAK

STUDI GELEMBUNG GAS FISI DAN LAPISAN INTERAKSI U_3Si_2 -Al PASCAIRADIASI DENSITAS $4,8 \text{ gU/cm}^3$. Bahan bakar senyawa uranium-silisida yang terdispersi dalam matriks aluminium (U_3Si_2 -Al) telah digunakan di sejumlah besar reaktor riset di seluruh dunia karena sifatnya yang sangat baik selama diiradiasi. Bahan bakar ini juga memiliki densitas uranium yang tinggi yang pada umumnya mencapai $4,8 \text{ gU/cm}^3$ untuk mengimbangi pengurangan jumlah fisil dalam LEU. Untuk meningkatkan densitas U_3Si_2 -Al ($2,96 \text{ gU/cm}^3$) yang saat ini digunakan dalam Reaktor Serba Guna GA Siwabessy Indonesia, pelat elemen bakar dispersi U_3Si_2 -Al dengan densitas $4,8 \text{ gU/cm}^3$ ($U^{235} \sim 19,75\%$) telah diiradiasi di RSG GAS selama 175 hari pada daya 15 MW hingga tingkat pembakaran sekitar 40%. Karakterisasi dilakukan menggunakan SEM-EDS dan mikroskop optik untuk mempelajari struktur mikro bahan bakar pascairadiasi, terutama gelembung gas fisi dan lapisan interaksi antara bahan bakar U_3Si_2 dan matriks Al. Diameter rata-rata gelembung dengan ukuran diameter dari $0,06$ hingga $0,55 \mu\text{m}$ adalah $0,21 \mu\text{m}$. Lapisan interaksi teridentifikasi sebagai $U(Al,Si)_{2,3}$ dengan ketebalan sekitar $1,5 \mu\text{m}$. Gelembung gas fisi dan lapisan interaksi tidak menyebabkan pengembangan pada bahan bakar dan kinerja pelat bahan bakar secara keseluruhan sangat baik.

Kata kunci: LEU, uranium silisida, uji pascairadiasi, lapisan interaksi, gelembung gas fisi.

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INTRODUCTION

Since 1978 multiple research reactor operating countries have signed agreements for the conversion of HEU (High Enriched Uranium) fuel to LEU (Low Enriched Uranium) fuel in research reactors. LEU defined as enriched uranium containing less than 20% of the isotope ²³⁵U, while HEU is uranium containing 20% or more of the isotope ²³⁵U [1]. The use of LEU in research reactor reduces the proliferation risks associated with HEU since LEU cannot be used for the manufacture of nuclear explosive devices without transmutation or further enrichment while HEU is relatively easier to use in nuclear weapon.

While the use of LEU shortened the lifetime of fuel inside the reactor, the LEU fuel with higher uranium density was developed under RERTR (Reduced Enrichment for Research and Test Reactors) program, both on existing type and new type of LEU of fuel. Fabrication techniques have been developed to maximize the uranium density. Three main HEU fuels when RERTR program begin in 1978 were UAl_x -Al dispersion fuel, U_3O_8 -Al dispersion fuel, and $UZrH_x$ alloy fuel, and all had successfully developed to have higher uranium density with LEU [2] [3]. In 1988, the US Nuclear Regulatory Commission approved the use of a new high density LEU fuel consisting of uranium silicide (U_3Si_2) dispersed in a matrix of aluminum up to a density of 4.8 gU/cm³. Numerous research reactors around the world have made successful conversions to this fuel [4] [5]. This silicide fuel provides high uranium density, relatively simple and inexpensive to fabricate, and show excellent behavior under irradiation [6].

The 30 MW Multipurpose Research Reactor G.A. Siwabessy (RSG-GAS) BATAN since operated in August 1987 already use LEU U_3O_8 -Al dispersion fuel with density 2.96 gU/cm³ and enrichment 19.75% ²³⁵U [7] [8], in accordance with the RERTR Program. But in 1999 the whole reactor core of the RSG-GAS converted from using U_3O_8 -Al fuel into using U_3Si_2 -Al fuel domestically produced to improve the RSG-GAS performance [9] and to benefit better safety margin offered by U_3Si_2 -Al [10]. Until now, RSG-GAS use U_3Si_2 -Al fuel with uranium density 2.96 g/cm³.

The success in producing and utilizing the 2.96 gU/cm³ U_3Si_2 -Al fuel brought BATAN to develop U_3Si_2 -Al fuel with higher uranium density of 4.8 g/cm³. U_3Si_2 -Al fuel with density

4.8 gU/cm³ had been successfully fabricated in PTBBN BATAN with fabrication process that is almost similar with U_3O_8 -Al fuel [11].

The uranium-silicide fuel plate is produced by melting uranium and silicon (92.5% and 7.5% by weight) together to make U_3Si_2 ingot, followed by comminution (milling) to produce a powder. The silicide powder is mixed with aluminum powder with ratio according to the uranium density, and then pressed into a powder metallurgical compact. The compact is then clad by a frame and two cover sheets, and welded on some spot on the four side, before hot and cold rolling to produce a fuel plate with thickness of 1.3 mm [4] [12].

Three full size U_3Si_2 -Al fuel plates with uranium density of 4.8 g/cm³ with code CBBJ249, CBBJ250, and CBBJ251 had been irradiated in the reactor in 2008. The three plates inserted in a fuel bundle on position 3, 7, and 19 while the others 18 plates were dummy [13]. Irradiation conducted at 15 MW power [14]. The irradiation time was 87 days for CBBJ 249, 175 days for CBBJ251, and 263 days for CBBJ250 [15]. Kadarusmanto reported that the amount of burned ²³⁵U calculated with ORIGEN2 was 18.41% for CBBJ249, 36.71% for CBBJ251, and 55.16% for CBBJ250 [15]. To investigate the microstructure of U_3Si_2 -Al dispersion fuel with density 4,8 gU/cm³ after irradiated at relatively medium reactor conditions and fuel burnup estimated to be ~40%, metallography examination was performed on CBBJ251 fuel plate. This paper reports the results of metallography examinations that were performed using SEM and optical microscope and discusses the observed microstructural changes in the irradiated U_3Si_2 -Al fuel, particularly the interaction layer (IL) between U_3Si_2 particles and Al matrix in and the fission gas bubbles.

There have been numerous studies on the effect of irradiation to the microstructural changes of U_3Si_2 -Al fuel. U_3Si_2 , as well as other uranium-silicide compounds such as U_3Si , are known to be amorphized during ion and neutron irradiation [16], [17]. Numerous PIE test also showed the formation of fission gas bubble and interaction layer (IL) between U_3Si_2 particles and Al matrix in U_3Si_2 -Al fuel. The gas fission product atoms that are not soluble in fuel particle diffuse and cluster to form gas bubbles [18]. Most fission gas bubbles are distributed in the fuel particles,

while some also formed in near interfaces of IL layer [19]. J. Gan reported the microstructural characterization using TEM for U_3Si_2 -Al (~75% enrichment) dispersion fuel irradiated in Advanced Test Reactor (ATR) for 105 days to high fission density of $5.4 \times 10^{27} \text{ f/m}^3$. Irradiation and fission of the fuel resulted in the amorphization and development of circular fission gas bubbles mostly in the fuel particle, with size of 10-1000 nm and volume fraction in the fuel was ~11%. High concentration of gas bubble also formed in narrow region of IL near either interface of fuel/IL or interface of IL/Al matrix. The thickness of the IL layer was approximately $5 \mu\text{m}$ [19].

The gas bubble of the insoluble fission gases Xe and Kr [20] formed in the fuel particles can increase the fuel volume and contribute to the fuel meat swelling, as well as solid fission products. At very high burnup, swelling can cause a large deformation of fuel plate which may even disturb the flow of coolant [18]. Kim [21] reported that fuel temperature and fission density during irradiation contributes to size formation of fission gas bubbles. At very high temperature and high fission density, the large bubbles forms, some of which can interconnect and can result in fuel failure by pillowing. Kim also suggested based on several data available that bubble with size of $5 \mu\text{m}$ are considered as a threshold of being large bubble [22]. Higher density compounds such as U_3Si exhibit undesirably high swelling rates at low or moderately high fission densities compared to less dense compounds such as U_3Si_2 and USi that appear to have a more stable swelling behavior [17].

Other issues that occur in uranium-silicide fuel (U_3Si , U_3Si_2 , and USi) dispersed in Al matrix is the interaction layer (IL) that formed between the fuel particles (U and Si) and the matrix (Al) by interdiffusion [21]. The layer may change the volume of the fuel meat by consuming the fuel particles and matrix and decreases fuel thermal conductivity. Many studies have identified the layer as a single intermetallic compound $U(Al,Si)_3$, that has composition intermediate between UAl_3 and USi_3 [21],[23], [24]. For IL of U_3Si_2 , the Al to Si ratio is approximately 3.5, and the composition of the compound lies on the tieline between U_3Si_2 and Al on ternary phase diagram.

Fission can also occur in the ILs because the presence of U [18]. The ILs of irradiated U_3Si_2 -Al fuel had been previously reported to be amorphous [25]. Kim et al [21] studied the ILs growth data in U_3Si -Al, U_3Si_2 -Al, and USi -Al dispersion fuels from both out-of-pile and in-pile tests. The growth rate is linear with the U/Si ratio in the fuel particle (highest for U_3Si -Al and the lowest for USi -Al), though the reduction of U with burnup did not seem to decrease the IL growth rate.

METHODOLOGY

Uranium silicide U_3Si_2 -Al fuel plate with code CBBJ251 was irradiated in RSG-GAS reactor core for 175 days. After irradiation, the fuel plate was discharged from the core to the reactor pool for cooling down for certain period of time. Then, the fuel plate was transferred to the Radiometallurgy Installation (RMI) hot cell facility for detailed post irradiation examination (PIE) through underground transfer channel. According to Kadarusmanto [15] the amount of burned ^{235}U from CBBJ251 fuel plate calculated with ORIGEN2 was 36.71%. Meanwhile, based on measurements and calculations with gamma scanning by PTBBN BATAN, the amount of burned ^{235}U was 39.42%. Nondestructive test for the plate was conducted first before cutting the fuel for the destructive test. Visual inspection and imaging with x-ray radiograph showed no anomalies (such as defects, blister, damage, and significant dimensional changes) were found [26].

After the nondestructive test, the plate was cut at the center (as depicted in Fig. 1) with dimension 25 mm x 20 mm using a punching-device. The sample was cut again into smaller pieces of 3 mm x 3 mm for metallography sample using a low-speed precision cutter. The sample was prepared by mounting the sample inside a ring using mixture of epoxy and conductive filler, followed by mechanical wet grinding with up to grit 2400 SiC paper and mechanical polishing with $1 \mu\text{m}$ diamond paste. Ultrasonic cleaning was conducted for about 15 minutes before examination using optical microscope. After that, the sample was sputter-coated with Au for microstructural characterization with SEM-EDS. For a comparison, unirradiated U_3Si_2 -Al fuel plate sample was also prepared and characterized using SEM-EDS.

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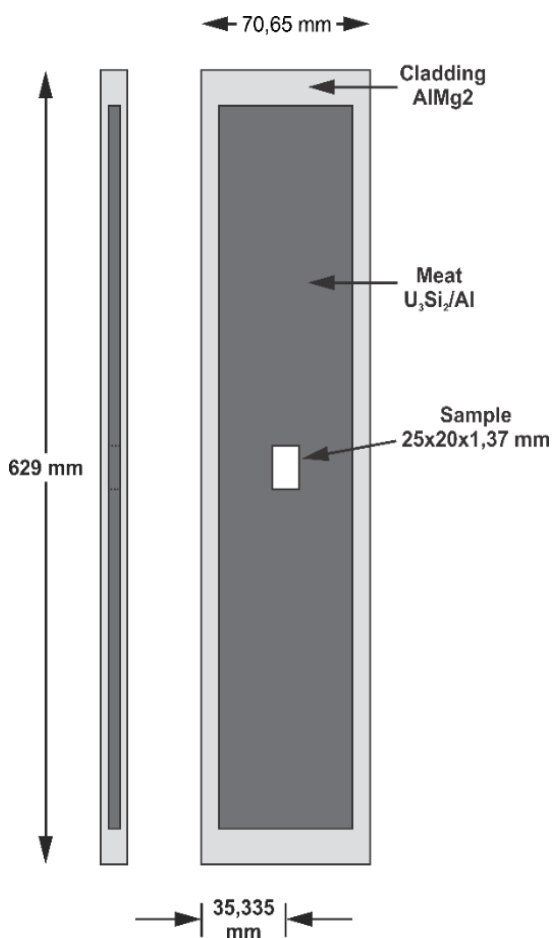


Figure 1. Sampling position in CBBJ251 fuel plate using punching-device. The sample dimension of the fuel plate was based on non-destructive test result [26]

RESULTS AND DISCUSSION

a. Unirradiated U_3Si_2 -Al fuel plate

The micrograph of unirradiated U_3Si_2 -Al fuel plate using SEM (Fig 2) shows U_3Si_2 fuel particles (light), Al matrix, and AlMg2 cladding (dark). The U_3Si_2 particles have a complex and irregular geometry and large variation in size. Multiple cracks are clearly visible on the fuel particles (Fig 3) that probably caused by deformation during compacting and rolling during fuel plate fabrication. It is known that intermetallic U_3Si_2 have a brittle characteristic and tetragonal crystalline structure [19], [27].

Fig 3 shows some gray contrast along the cracks in the fuel particles, that probably is a penetration of Al into the cracks resulted from fuel plate compacting and rolling during fabrication [19]. Keiser Jr et al reported that U-Al-Si phase is formed along the cracks in the relatively larger fuel particles and around the smaller ones, that appeared as medium contrast phase [28]. Mirandou et al did report based on XRD and SEM result that low amount of $U(Al,Si)_3$ phase can be formed in U_3Si_2 -Al fuel during fabrication process [29]. As seen in Fig 4, there is no visible formation of ILs at the interface of matrix and fuel particle, either around small or large particles, even with high magnification SEM image. It has been suggested that other phases that could possibly present as a secondary phase in U_3Si_2 are U_{ss} , U_3Si , and USi . These phases have different properties with U_3Si_2 and can affect the performance of the fuel [22]. Scratches in the surface resulted during sample preparation are still visible (Fig 3).

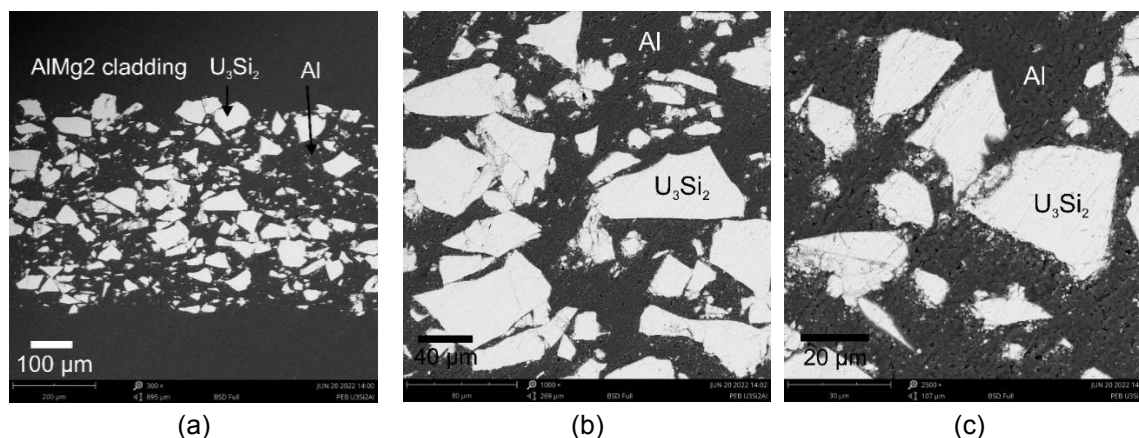


Figure 2. Backscatter electron SEM image of unirradiated U_3Si_2 -Al fuel showing unirradiated U_3Si_2 fuel meat inside Al matrix, magnification of 300x (a); 1000x (b); and 2500x (c).

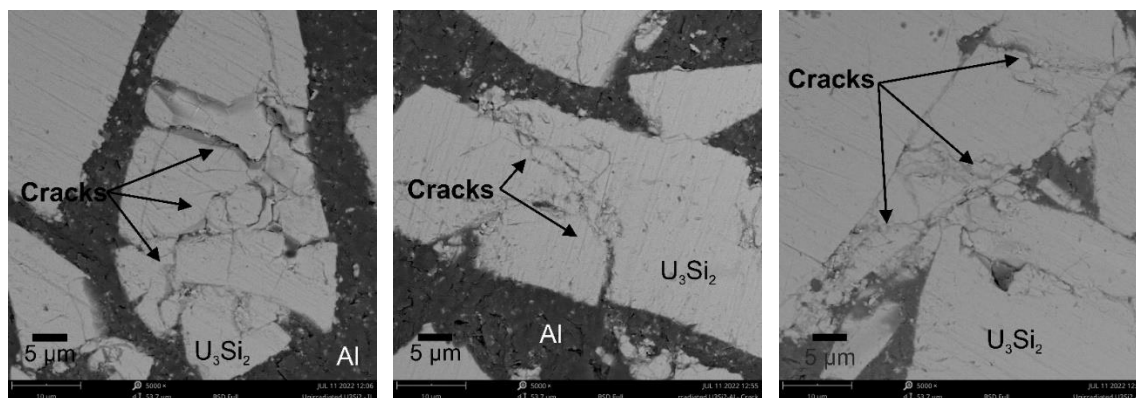


Figure 3. Backscatter electron SEM image with magnification 5000x shows cracks at unirradiated U_3Si_2 fuel particles

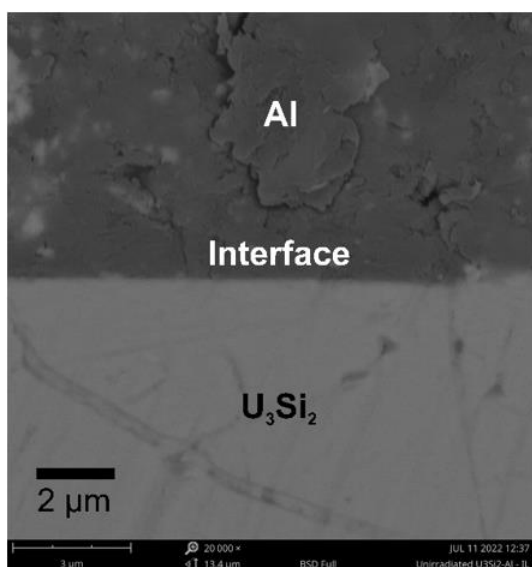


Figure 4. Backscatter electron SEM image with magnification of 20000x shows interface of U_3Si_2 fuel and Al matrix, where no visible interaction layer observed.

b. Irradiated U_3Si_2 -Al fuel plate

The optical microscope image of irradiated U_3Si_2 -Al fuel plate with magnification up to 100x are shown in Fig 5. In the optical microscope image, ILs are clearly visible as a gray area at the interfaces of fuel (dark) and matrix (light). However, fission gas bubbles are hardly visible in this image due to resolution limit.

Backscatter image SEM image of the irradiated U_3Si_2 -Al fuel particles is shown in Fig 6. Irradiation of U_3Si_2 -Al fuel in the reactor for 175 days resulted in formation of ILs and fission gas bubbles. The thickness of the IL measured using ImageJ software is approximately $1,5 \mu\text{m}$. Note that this value is obtained from the measurement of the average thinnest ILs because the thinnest ILs are more likely lies on the equator of the fuel particle [21]. Many literature studies identified the IL as an intermetallic compound of $U(Al,Si)_3$.

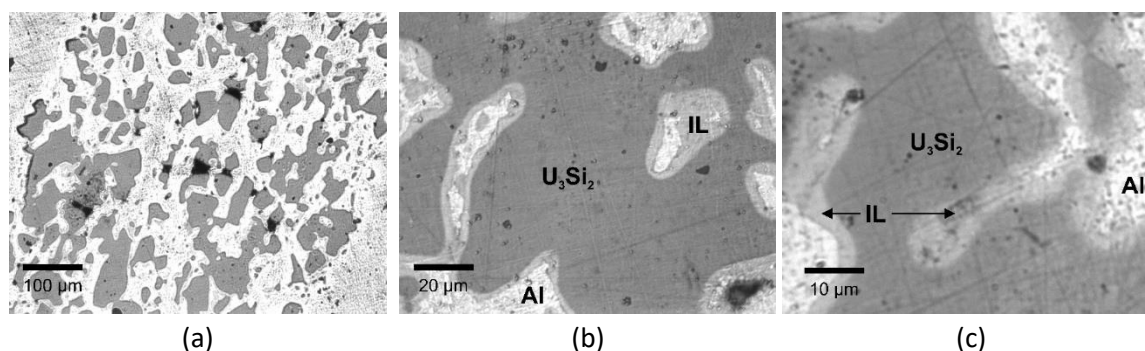


Figure 5. Optical microscope image of the irradiated U_3Si_2 -Al fuel with magnification 10x (a); 50x (b), and 100x (c).

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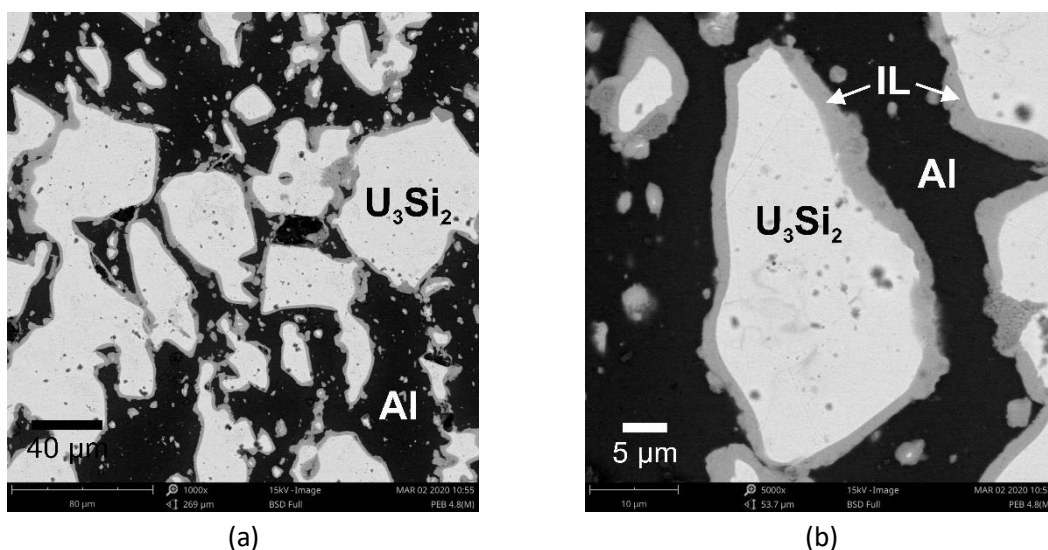


Figure 6. SEM image of irradiated U_3Si_2 showing interaction layers (ILs) at matrix and fuel interfaces, magnification of 1000x (a); and 5000x (b)

Fig 7(a) and 7(b) shows more detailed fission gas bubble in the fuel particle. The tetragonal U_3Si_2 transform into amorphous structure after irradiation even at a very low fission density, therefore the bubbles have a circular shape to reduce its surface energy in an amorphous material [19]. The cause of amorphization is the damage to crystal structure by highly fission fragments. The bubbles distributed in the fuel particle and no bubbles are found in the ILs. Kim [21] suggests that fission-gas bubbles only form in

the IL at extremely high fission densities. For LEU fuel, fission-gas bubbles do not form in the IL because the maximum fission density in the ILs is $3,6 \times 10^{21}$ f/cm³. High fission density also affects the formation of large bubbles, as well as high fuel temperature [22]. In Fig 6, there are no interconnections between the bubbles observed in the fuel particles. D. D. Keiser Jr stated other factor that affect fission gas behaviour such as matrix stress, burnup, fuel properties, fission gas properties, grain size, etc [28].

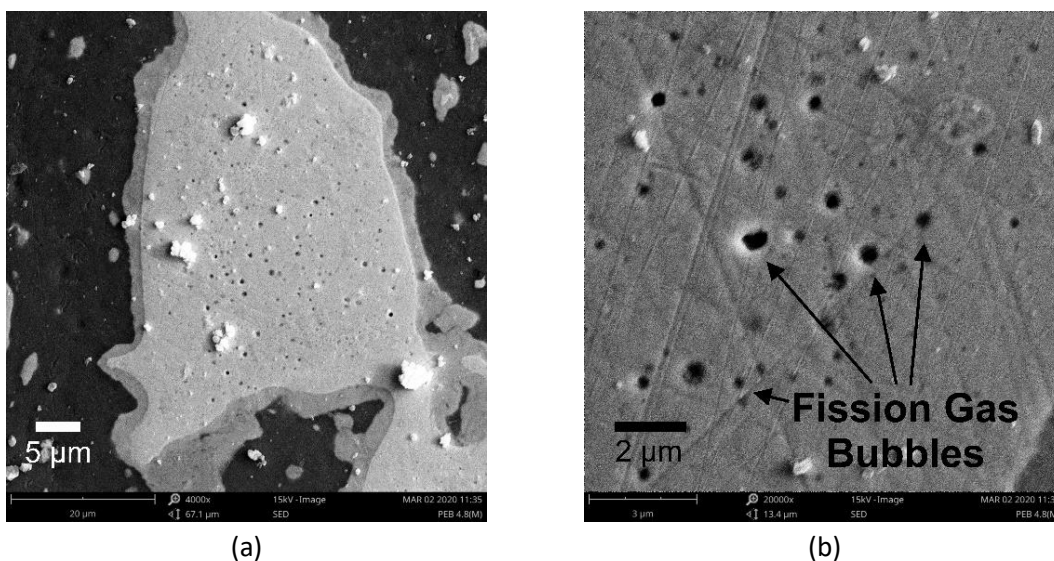


Figure 7. Secondary electron SEM image of the fission gas bubble inside the fuel particle with magnification of 4000x (a); and 20000x (b).

Image analysis of the fission gas bubbles presented in Fig 7(b) were performed using ImageJ software, as shown in Fig 8(a). The smallest bubble diameter that can be measured with ImageJ was 60 nm. Bubbles smaller than 60 nm diameter were hardly measured due to resolution limit. From the 41 bubbles count with bubble diameter from 60 nm to the largest of $0,55 \mu\text{m}$, the

average size of the bubbles was $0,21 \mu\text{m}$. This size are considered stable and small compared to $5 \mu\text{m}$ threshold by Kim [22], and therefore did not lead to fuel swelling, in accordance with the result of the previous non-destructive test [26]. Diameter size distribution of the bubbles can be seen in Fig 8(b).

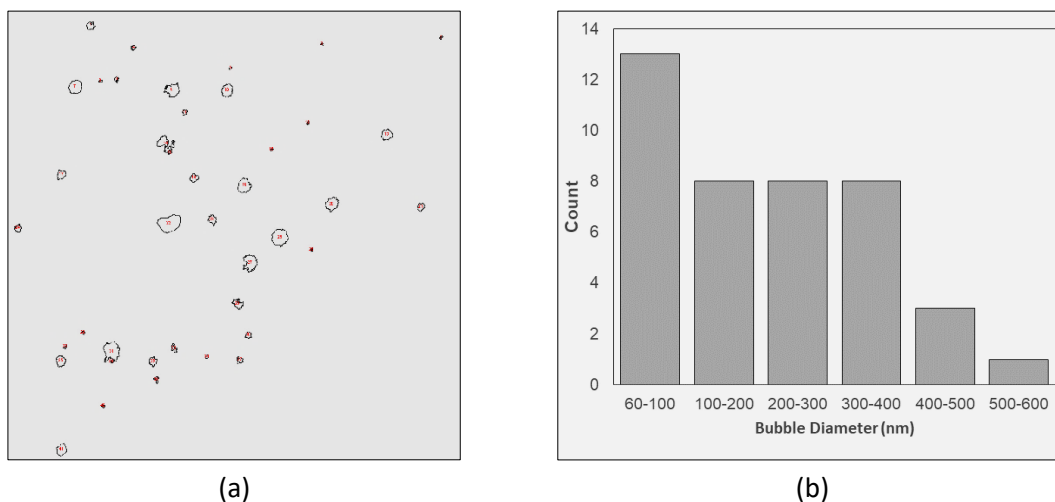


Figure 8. ImageJ size analysis of the bubbles and its diameter size distribution.

EDS point measurements were conducted at two different spots on the IL, as shown in Fig 9. The result reveals that the two spots of IL have identical composition that consist of U, Al, and Si as shown in Table 1. The $(\text{Si}+\text{Al})/\text{U}$ ratio is ~ 2.3 which is lower than the exact stoichiometry i.e. 3. Kim [21] suggests that this deviation is because the IL is amorphous, and the U, Al, and Si atoms exist in a mixture without crystalline restriction of stoichiometry. The Al/Si ratio in this study is ~ 7.6 . In the study reported by Leenaers [30], the IL with composition of 22.6 wt% Al, 4.7 wt% Si and 52.5 wt% (corresponding with 68.3 at% Al, 13.7 at% Si, and 18 at% U) were interpreted as $U(\text{Al},\text{Si})_{4.6}$ with an Al/Si ratio of 5. In this study, based on EDS measurement result and similar

interpretation with Leenaers, the IL has composition of $U(\text{Al},\text{Si})_{2,3}$ with a low Si content, as shown in U-Al-Si ternary diagram in Fig.10. However, more research is required to confirm this result.

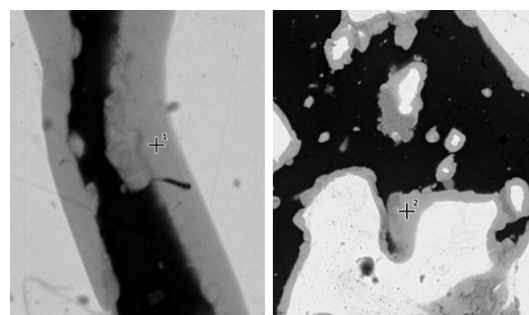


Figure 9. EDS point measurement locations at the IL

Table 1. EDS point measurements result of U, Al, and Si element at the interaction layer (at%).

Spot	U	Al	Si	Al/Si	$(\text{Si}+\text{Al})/\text{U}$
1	30.13	61.79	8.08	7.65	2.32
2	29.6	62.25	8.15	7.64	2.38

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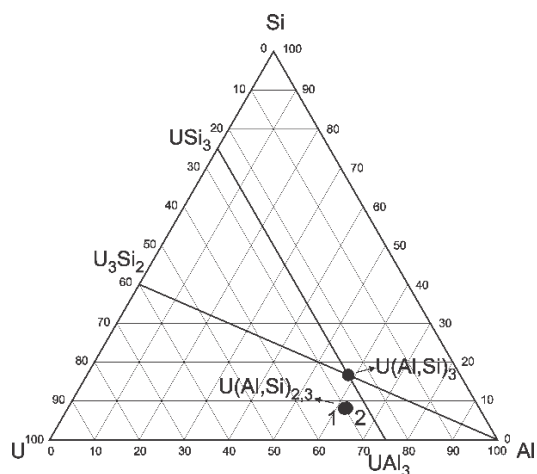


Figure 10. Ternary U-Al-Si phase diagram shows the formed IL of $U(Al,Si)_{2,3}$ in the current study.

CONCLUSIONS

U_3Si_2 -Al fuel plates with uranium density of 4.8 g/cm³ showed a good performance after irradiation in RSG-GAS reactor core for 175 days at 15 MW power and fuel burnup about 40%. The fuel showed a good integrity, and no detrimental changes were found in the microstructure. Fission gas bubble that formed in the fuel particles have a circular shape. From the 41 bubbles count with bubble diameter from 60 nm to the largest of 0.55 μ m, the average size of the bubbles was 0.21 μ m. The bubbles did not lead to fuel swelling, consistent with previous non-destructive test result. There are no interconnections between the bubbles observed in the fuel particles.

An interaction layer (IL) between the Al cladding and the U_3Si_2 fuel particles was formed. The ILs have thickness of approximately 1.5 μ m and visible through optical microscope. There are no fission gas bubbles observed in the IL. Based on EDS measurement, the IL have composition of $U(Al,Si)_{2,3}$, where the (Si+Al)/U ratio is \sim 2.3 and Al/Si ratio is \sim 7.6.

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