

# THE EFFECT OF SI-RICH LAYER COATING ON U-MO VS. AL INTERDIFFUSION

HO JIN RYU\*, JAE SOON PARK, JONG MAN PARK, and CHANG KYU KIM

Research Reactor Fuel Development Division, Korea Atomic Energy Research Institute

1045 Daedeokdaero, Yuseong, 305-353, Daejeon – Republic of Korea

\*Corresponding author. E-mail : hjryu@kaeri.re.kr

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Si-rich-layer-coated U-7 wt%Mo plates were prepared in order to evaluate the diffusion barrier performance of the Si-rich layer in U-Mo vs. Al interdiffusion. Pure Si powder was used for coating the U-Mo plates by annealing at 900 °C for 1 h under vacuum of approximately 1 Pa. Si-rich layers containing more than 60 at% of Si were formed on U-7 wt%Mo plates. Diffusion couple tests were conducted in a muffle furnace at 560-600 °C under vacuum using Si-rich-layer-coated U-Mo plates and pure Al plates. Diffusion couple tests using uncoated U-Mo plates and Al-(0, 2 or 5 wt%)Si plates were also conducted for comparison. Si-rich-layer coatings were more effective in suppressing the interaction during diffusion couple tests between coated U-Mo plate and Al, when compared with U-Mo vs. Al-Si diffusion couples, since only small amounts of Al in the coating could be found after the diffusion couple tests. Si-rich-layer-coated U-7wt%Mo particles were also prepared using the same technique for U-7 wt%Mo plates to observe the microstructures of the coated particles.

**KEYWORDS :** Dispersion Fuel, Interdiffusion, Diffusion Couple Test, Diffusion Barrier

## 1. INTRODUCTION

Uranium-molybdenum (U-Mo) alloys have higher uranium density and better irradiation stability than existing research-reactor fuel compounds such as  $U_3Si$  and  $U_3Si_2$  [1]. In the microstructures of the dispersion fuels used in research reactors the fuel particles of uranium compounds are dispersed in an Al matrix. U-Mo/Al dispersion fuel is being developed for advanced research reactors to replace high-enriched uranium (HEU) fuel with low-enriched uranium (LEU) fuel under the international reduced enrichment for research and test reactors (RERTR) program [2]. Irradiation tests of U-Mo/Al dispersion fuel have shown that radiation-induced microstructural changes significantly influence the fuel's performance. The presence of interaction layers at the interfaces of the U-Mo particles and Al matrix during irradiation is one of the most challenging issues in the development of U-Mo/Al dispersion fuel [3]. Since the Al matrix is consumed by the interaction, the thermal conductivity of U-Mo/Al dispersion fuel decreases and the fuel temperature increases. In addition, the accumulation of radiation damage during irradiation induces amorphization of the interaction layers [4,5], and significant pillowing of the fuel plate may occur by the growth of fission gas voids primarily at the interaction layer/Al matrix interfaces.

While the use of large U-Mo particles with diameters

of 200-500  $\mu m$  has been proposed as a solution to mitigate the interaction problems [6,7], the addition of Si to the Al matrix has also been found to be a promising remedy to reduce the interactions [8]. Irradiation tests using Al-Si matrices instead of the Al matrix have shown reductions in the interaction layer growth [9]. Si-rich interaction layers are formed in the U-Mo/Al-Si dispersion fuel, and they decrease the interaction layer growth rate during irradiation. When the effects of Si content in the Al-Si matrix were investigated using irradiation tests of U-7 wt%Mo/Al-(0.2-4.8wt%)Si dispersion fuel, Al-Si with higher Si content showed more reduced interactions. However, the effect of Si on the interaction reduction decreases when the interaction layer increases since the Si content in the interaction layer becomes diluted. The dilution of Si in the U-Mo/Al-Si dispersion fuel can be delayed by increasing the Si content in the Al-Si matrix, but minimizing the total Si content is recommended for improving the reprocessing efficiency of the spent fuel. However, the optimum Si content has not yet been determined [10]. Moreover, there may be additional drawbacks in the use of Al-Si matrices. Al-Si binary alloy powder is not easily available on a commercial basis, and some commercial Al-Si powders contain additional alloying elements. In the Al-Si binary system, solid solubility of Si in Al is limited, and hence, hard Si particles can be precipitated. The conventional fabrication processes of

the dispersion fuel optimized for pure Al powder needs to be adjusted because the addition of Si to Al increases both the strength and brittleness of the fuel meat. Si in the interaction layer becomes diluted as the interaction layer grows and then loses its effect toward reducing the interaction. However, it is difficult to obtain high Si content in the interaction layer by interdiffusion between the U-Mo particle and Al-Si matrix during irradiation. Post-irradiation examination (PIE) of the IRIS-3 irradiation test using U-7 wt%Mo/Al-2wt%Si has shown that the Si content in the interaction layer is ~5 at% [11], while the PIE of the HANARO irradiation test using U-7 wt%Mo/Al-2 wt%Si has shown that the composition of Si content in the interaction layer is less than 11 at% [12].

However, the PIE of the BR1 reactor fuel–aluminum clad natural uranium rod with USi anti-diffusion layer–has shown that a thin USi layer of approximately 10  $\mu$ m limited the interaction between the uranium and aluminum cladding effectively during the irradiation [13]. This result suggests that a high Si content of more than 50 at% is very efficient as a diffusion barrier to reduce the interaction between the major diffusing components in the U-Mo/Al dispersion fuel, i.e., uranium and aluminum. Therefore, Si-rich-layer coatings on the U-Mo particles can be an alternative approach to solving the interaction problems. The Si-rich-layer coating method has many advantages, for example, a pure Al matrix can be used as in the conventional process conditions, and the total Si amount in the U-Mo/Al dispersion fuel can be reduced when Si is concentrated effectively at a thin layer on the surface of the U-Mo particles.

There are many methods to coat Si-rich layers on U-Mo particles, for example, solid state reactions, liquid phase reactions involving molten metals or salts, and gas phase deposition such as chemical vapor deposition or physical vapor deposition. Ryu et al. reported that a solid state diffusion method could form Si-rich coated layers on the surface of an atomized U-Mo particle or U-Mo plates [14]. Attempts to coat Si on a U-Mo particle have also been attempted elsewhere, and Si coating equipment has been developed in SCK-CEN, Belgium [15]. Therefore, it is necessary to estimate the performance of the Si-rich coating layer as a diffusion barrier. In this study, the microstructures and the diffusion barrier performance of Si-rich-layer coatings on U-Mo plates were examined by diffusion couple annealing tests.

## 2. EXPERIMENTAL PROCEDURES

Schematic illustrations of the Si-rich-layer coating processes and diffusion couple tests are shown in Fig. 1. Gamma-phase U-7 wt%Mo plates sliced from induction-melted ingots were obtained by heat treatment at 950 °C for 24 h. Pure Si powder (99%, -325 mesh, Sigma-Aldrich) was used for the coating by packing U-Mo plates in the

Si powder bed and annealing at 900 °C for 1 h under vacuum of approximately 1 Pa. Diffusion couple tests were conducted in a muffle furnace at 560 - 600 °C under vacuum using Si-rich-layer-coated U-Mo plates and pure Al plates. Diffusion couple tests were conducted in a muffle furnace at 560 °C. Diffusion couple tests using uncoated U-Mo plates and Al-(0, 2 or 5 wt%)Si plates were also conducted for comparison. Centrifugally atomized U-7 wt%Mo powder having a diameter of 200-500  $\mu$ m was used for the Si-rich-layer-coating tests. The microstructures of the diffusion couple specimens and coated particles were observed by scanning electron microscopy (SEM). Further, the elemental composition of the interaction layers was measured by energy dispersive X-ray spectroscopy (EDS), and standardless quantitative data were obtained by the ZAF correction algorithm of the EDAX Genesis X-ray microanalysis software.

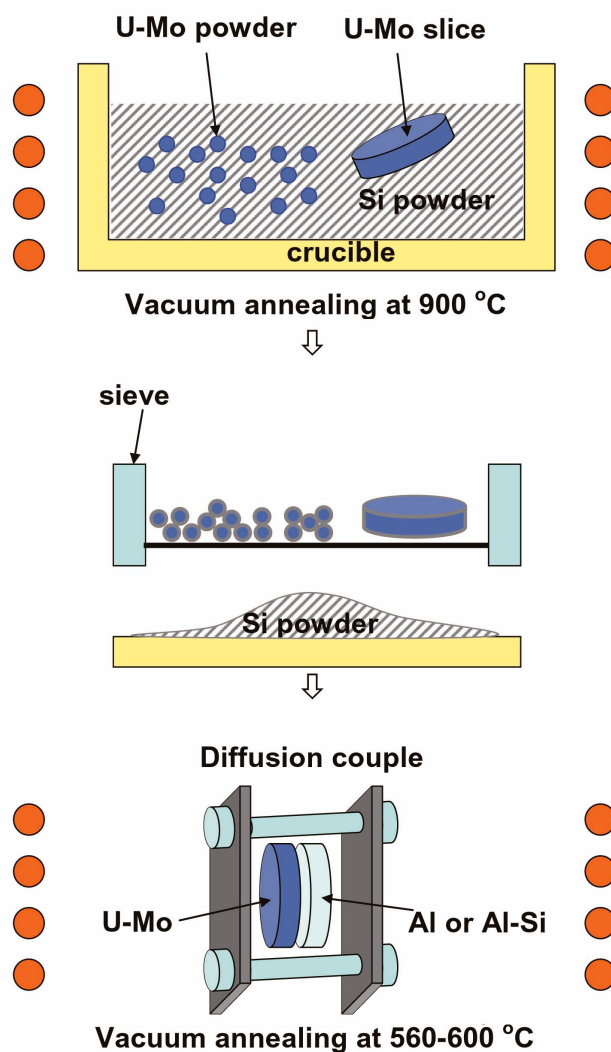


Fig. 1. Schematic Illustrations of Si-rich Layer Coating Processes and a Diffusion Couple Test

### 3. RESULTS AND DISCUSSION

#### 3.1 Diffusion Couple Tests Using Coated U-Mo Plates

Gamma-phase U-7 wt%Mo plates were annealed in a Si powder bed at 900 °C for 1 h under vacuum in order to conduct diffusion couple tests of coated U-Mo plates and Al plates. Si-rich layers with a thickness of approximately 5  $\mu$ m were formed on each U-Mo surface, as shown in Fig. 2. EDS measurements of the composition of the Si-rich layer suggest that the Si content is around 65 at%. Table 1 shows the composition of the Si-rich layer on the gamma-phase U-7 wt%Mo plates before the diffusion couple tests. Microstructural and compositional analyses showed that the Si-rich coated layers could be mixtures of two or more silicide phases. Many Si-rich phases were found in U vs. Al-Si or U-Mo vs. Al-Si diffusion couples. Leenaers et al. reported that USi and U(Al,Si)<sub>3</sub> layers were formed between the pure U slug and a molten Al-(11.2–11.5 wt%)Si alloy; the layers changed to USi<sub>2</sub> after 50 years of irradiation in the BR1 reactor [13]. Mirandou et al. reported that mixtures of U(Al,Si)<sub>3</sub> and U<sub>3</sub>Si<sub>5</sub> were formed after diffusion couple tests between U-Mo and Al-7.1 wt%Si alloy [16].  $\mu$ -XRD analyses showed that

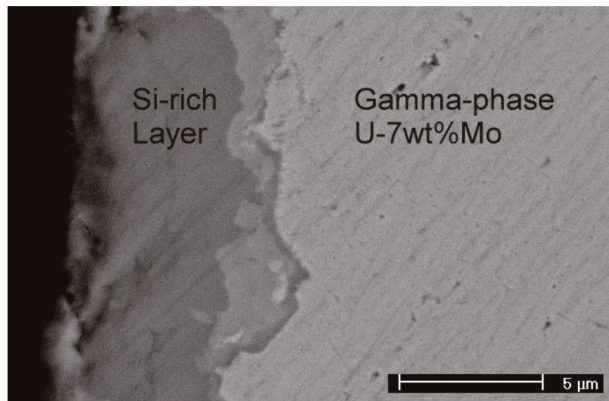


Fig. 2. A Cross-sectional Scanning Electron Micrograph of a U-7 wt%Mo Plate Showing a Si-rich Layer Formed by Annealing in a Si Powder Bed at 900 °C for 1 Hour Under Vacuum

**Table 1.** Compositions of a Si-rich Layer after Coating Treatment of a U-7 wt%Mo Plate Annealed in a Si Powder Bed at 900 °C for 1 Hour.

Element	Si-rich layer after coating (at.%)
Si	64.9
Mo	7.4
U	27.6

mixtures of U<sub>3</sub>(Al,Si)<sub>5</sub> and U(Al,Si)<sub>3</sub> are formed in a U-7 wt% Mo/Al-7 wt% Si diffusion couple [17]. The composition measured by EDS is similar to the compositions of U<sub>3</sub>Si<sub>5</sub> and USi<sub>2</sub>; however, more detailed X-ray or neutron diffraction studies are required to identify the crystallographic information of the Si-rich coated layers on the U-Mo particles or U-Mo plates.

Fig. 3 presents a comparison of the cross-sectional microstructures of uncoated U-7 wt%Mo plate/Al-5 wt%Si plate couples and coated U-7 wt%Mo plate/Al plate couples after annealing at 580 °C and 600 °C for 5 h each. Interaction layers were formed at the interface between U-Mo and Al-Si, and their thicknesses were approximately 50  $\mu$ m and 60  $\mu$ m at 580 °C and 600 °C, respectively, as shown in Fig. 3(a) and 3(c), respectively. However, the growth in the Si-rich layer in the coated U-Mo/Al diffusion couple was not considerable when compared with the growth in the interaction layer in the uncoated couples, as shown in Fig. 3(b) and 3(d). The Si-rich interaction layers formed in the U-Mo/Al-Si diffusion couple play the role of a diffusion barrier. However, the Si-rich coated layer exhibits better performance as a diffusion barrier. This improvement can be associated with the higher Si content in the layer. Fig. 4 shows the variations in the interaction layer thicknesses of the diffusion couples of uncoated U-7 wt%Mo plates against pure Al, Al-2 wt%Si, and Al-5 wt%Si plates at 560, 580, and 600 °C, respectively, up to 5 h. The changes in the thickness of the Si-rich coated layer of U-7 wt%Mo/Al diffusion couples have also been included in Fig. 4 for comparison.

Decomposition of  $\gamma$ -phase U-7wt%Mo into  $\alpha$ + $\gamma$  or  $\alpha$ + $\gamma'$  might occur during the diffusion couple tests at 560 °C and 580 °C. Gamma-phase decomposition at 560 °C is faster than that at 580 °C. However, as shown in Fig. 4, the interaction layers formed at 560 °C are always thinner than those formed at 580 °C, which means that the contribution of 20 °C difference in temperature might always be larger than the changes in diffusivity and reaction rate due to the gamma phase decomposition. The U-Mo/Al diffusion couple annealed at 600 °C for 5 h shows substantial interaction layer growth up to 275  $\mu$ m, while the diffusion couples of coated U-Mo vs. Al shows that the interaction layer thicknesses remained less than 20  $\mu$ m after annealing at 560, 580, and 600 °C up to 5 h. The diffusion couple for the coated U-7Mo plate vs. Al at 5 hours showed a thicker Si-rich layer at 560 °C than at 580 °C, while in the other test conditions, the Si-rich-coated-layers after diffusion couple tests of the coated U-Mo vs. Al did not show remarkable differences among them. It is difficult to judge whether the changes in the Si-rich layer originated primarily from a distinguishable mechanism, since there might be other sources of irregularity during the experiments. For example, no remarkable changes in the thickness of the Si-rich coated layer were observed in the diffusion couple annealed at



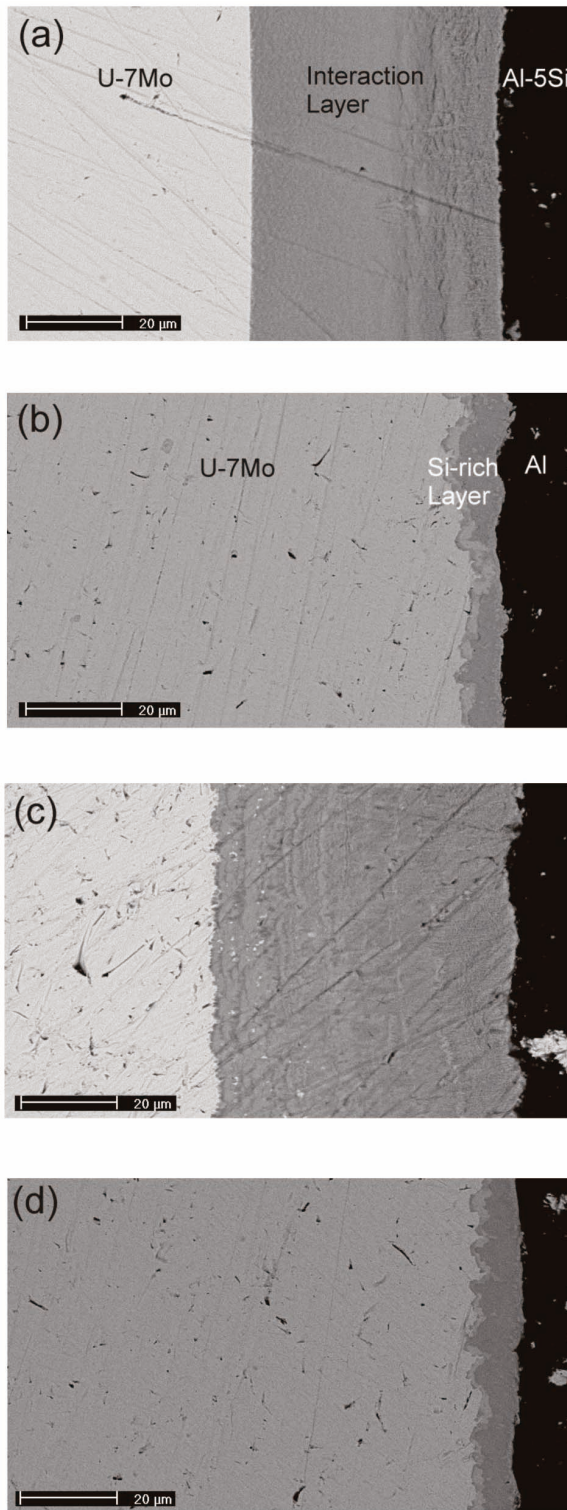
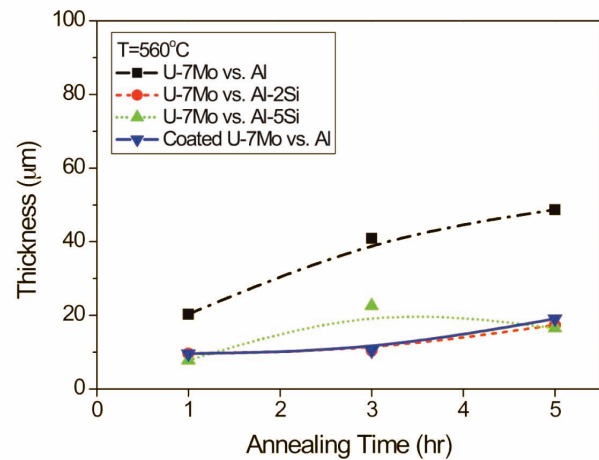
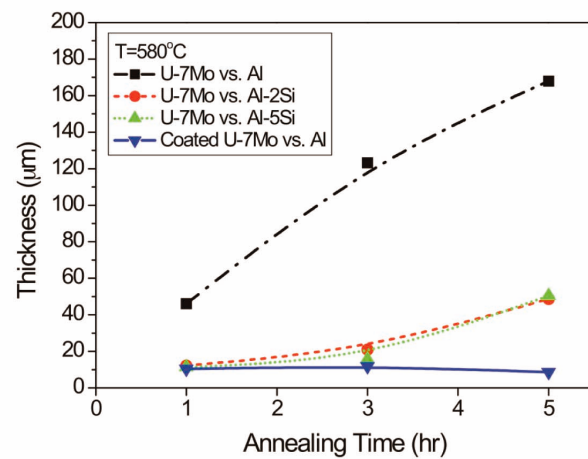


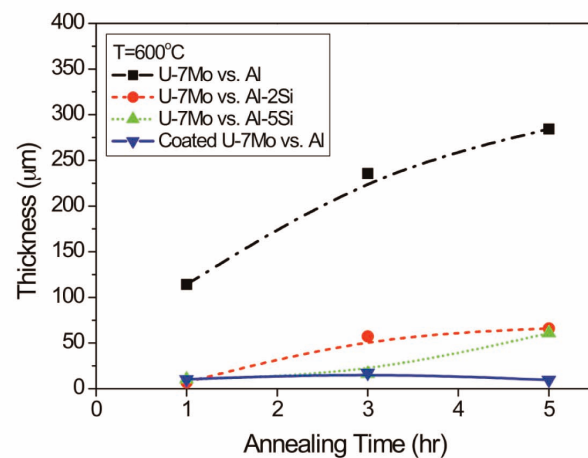
Fig. 3. Cross-sectional SEM Microstructures after Diffusion Couple tests of (a) an Uncoated U-7 wt%Mo Plate Against Al-5 wt%Si at 580 °C for 5 Hours, (b) a Coated U-7 wt%Mo Plate Against Pure Al Plates at 580 °C for 5 Hours, (c) an Uncoated U-7 wt%Mo Plate Against Al-5 wt%Si at 600 °C for 5 Hours, and (d) a Coated U-7 wt%Mo Plate Against Pure Al Plates at 600 °C for 5 Hours



(a)



(b)



(c)

Fig. 4. The Variation of the Interaction Layer Thicknesses in Diffusion Couples for Uncoated U-7 wt%Mo vs. Al-(Si) and the Si-rich Layer Thickness for Coated U-7 wt%Mo vs. Al Diffusion Couples Annealed at (a) 560 °C, (b) 580 °C, and (c) 600 °C

580 °C for 1, 3, and 5 h, as shown in Fig. 5.

Fig. 6 shows the X-ray elemental maps for the coated U-7Mo vs. Al diffusion couple annealed at 600 °C for 5 h; it clearly shows that the Si-rich layer remains while Al cannot be detected in the Si-rich layer after the diffusion couple annealing test. Further, the U-distribution does not show any remarkable changes, but a hint of the segregation of Mo at the interface with Al can be observed. When the compositions of Al and Si after the diffusion

couple test conducted at 600 °C for 5 h, shown in Table 2, are compared with those before the diffusion couple test, shown in Table 1, it can be seen that the Si content in the coatings is more than 60 at% without any severe dilution. This Si content is considerably higher than that in the case of the interaction layer of uncoated U-Mo/Al-Si diffusion couples, which is approximately 30 at% in U-Mo vs. Al-2 or 5 wt%Si diffusion couples, as shown in Table 2. The Al content in the Si-rich coated layers after

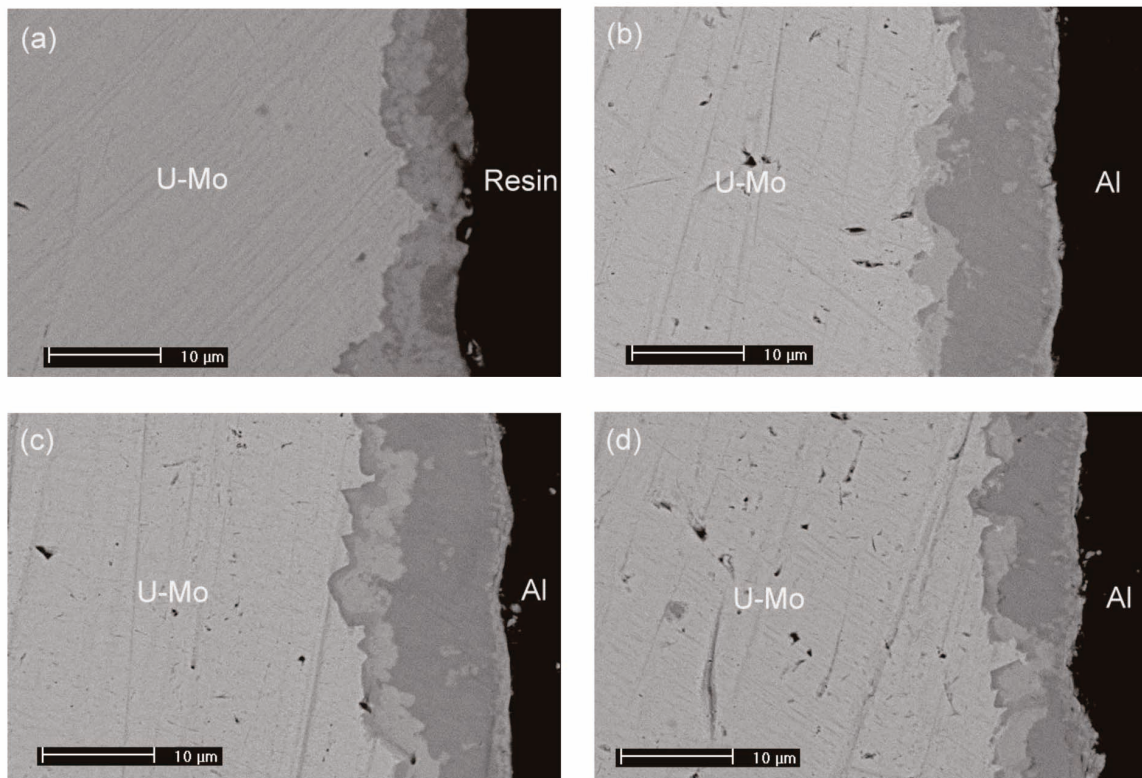


Fig. 5. Cross-sectional Microstructures of Si-rich Layers (a) in Coated U-7 wt%Mo and (b) in U-7 wt%Mo/Al Diffusion Couples Annealed 580 °C for 1 Hour, (c) 3 Hours, and (d) 5 Hours

**Table 2.** Compositions of a Si-rich Layer in Coated U-7 wt%Mo vs. Al Diffusion Couple and Compositions of Interaction Layers in Uncoated U-7 wt%Mo vs. Al-2 or 5 wt%Si Diffusion Couple Annealed at 600 °C for 5 Hours

Element	Si-rich layer in coated U-7Mo vs. Al (at%)	Interaction layer in U-7Mo vs. Al-2Si (at%)	Interaction layer in U-7Mo vs. Al-5Si (at%)
Al	4.8	51.4	49.6
Si	63.0	29.7	29.1
Mo	3.2	3.2	3.9
U	29.0	15.7	17.4



the diffusion couple tests was only approximately 5 at%, and this is considerably lower than that in the interaction layer, which is approximately 50 at% Al in U-Mo vs. Al-Si diffusion couples. This implies that the diffusion of Al is more prohibited by the Si-rich coated layers with higher Si content. The diffusion barrier performance of a U-Si layer with ~60 at% Si is much better than a U-Si-Al layer with ~30 at% Si under out-of-pile conditions. It has been estimated that dilution of the Si-rich layer during irradiation might be delayed by forming a thicker Si-rich layer in pre-irradiation annealing [10]. However, only a single Si-rich-layer thickness of about 5  $\mu\text{m}$  was used for the

diffusion barrier performance tests and the effect of Si-rich-layer thickness should be investigated in a future study.

### 3.2 Si-rich-layer Coating on a U-Mo Particle

When the same coating technique is applied to atomized U-7 wt%Mo particles having a diameter of 200–500  $\mu\text{m}$ , the coated U-Mo particles were recovered easily by sieve separation from the Si powder pack; the morphology of a coated U-Mo particle is shown in Fig. 7(a). The cross-section of the coated U-Mo particles comprises Si-rich

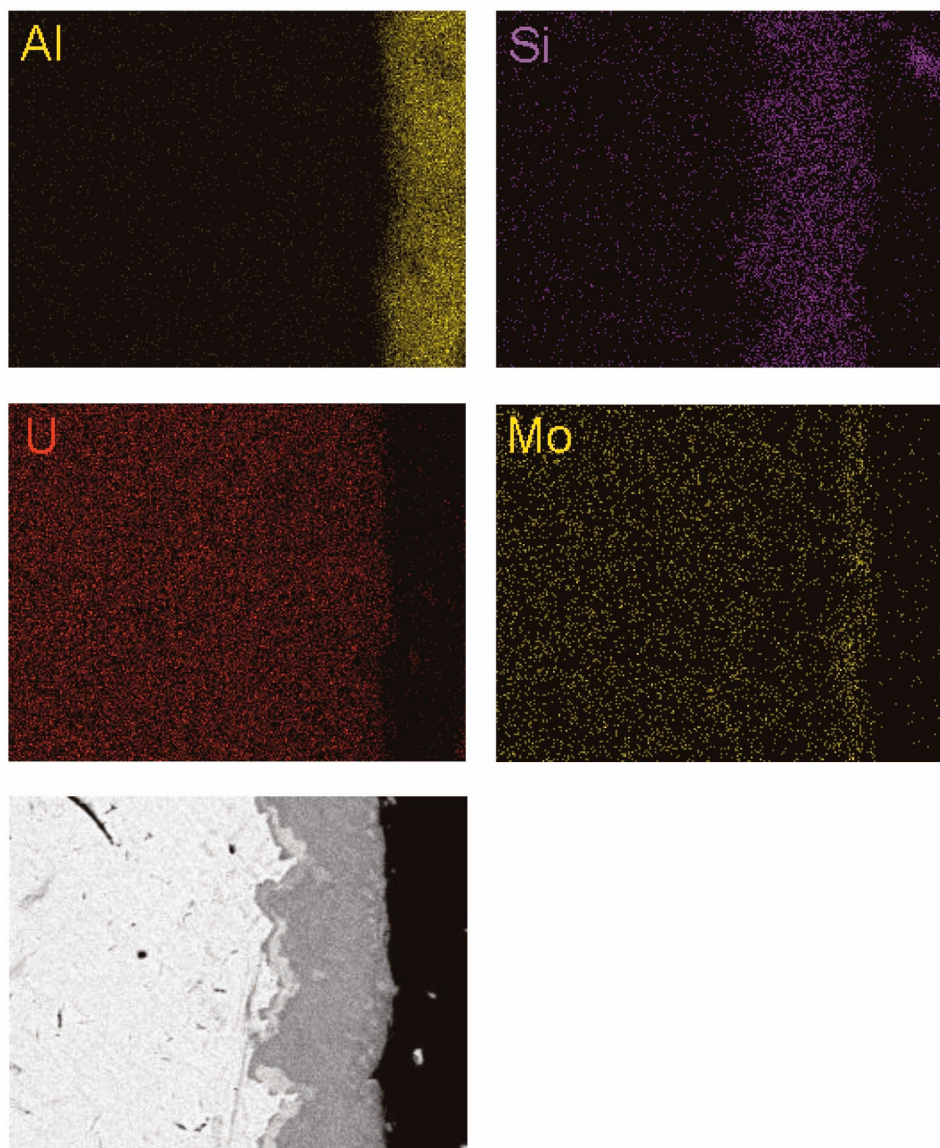


Fig. 6. X-ray Elemental Maps for Al, Si, U and Mo in a Coated U-7 wt%Mo vs. Al Diffusion Couple Annealed at 600 °C for 5 Hours

layers of approximately  $10\ \mu\text{m}$  at the peripheral regions, as shown in Fig. 7(b). The composition of the Si-rich layers on the surface of the atomized U-7 wt%Mo particles is shown in Table 3. The Si-rich coated layers consisted of a darker outer layer and a brighter inner layer. The Si content of the outer layer was around 60 at%, and the inner layer had lower Si content of approximately 50 at%. The Mo content of the outer layer was around 8 at%, and the

inner layer had a lower Mo content of approximately 2 at%. Further, an oxide layer remained at the outermost layer of the coated U-Mo particles. Since centrifugally atomized powders are passivated under an Ar gas atmosphere in the atomization chamber to prevent the spontaneous ignition of fine U-Mo powder, its surface is coated with a thin oxide layer [18,19]. Pre-existing oxide layers would hinder the uniform Si-rich coated layer formation on atomized U-Mo particles.

The theoretical mechanism for the effect of a Si-rich coated layer on the interdiffusion of U-Mo and Al is not yet fully understood. Atomistic modeling of the role of Si on the interaction of Al and U-Mo has revealed that the formation of Si-rich interfacial compounds inhibits Al diffusion to U-Mo [20]. Moreover, the reduced diffusion of Al is more noticeable in the layers with higher Si content. Since some differences exist in the diffusion mechanism between irradiation tests and out-of-pile diffusion couple annealing tests, irradiation tests should be conducted in order to evaluate the diffusion barrier performance under in-pile conditions. However, thermal annealing tests can still provide useful insights on the performance of Si-rich layers as a diffusion barrier. While low-power irradiation tests showed that the irradiation effect is considerably more dominant, high-power irradiation tests showed that the contribution of the thermal effect on the interaction layer growth was greater during irradiation because of higher fuel temperatures.

#### 4. CONCLUSIONS

Si-rich-layer-coated U-Mo plate and U-Mo particles were fabricated by Si powder pack annealing at  $900\ ^\circ\text{C}$  under vacuum. Si-rich layers containing more than 60 at% of Si were formed on U-7 wt%Mo plates and particles. Si-rich-layer coatings were more effective in suppressing the interaction during diffusion couple tests between coated U-Mo plate and Al, when compared with U-Mo vs. Al-Si diffusion couples; limited growth of the coating layer was observed and only small amounts of Al in the coating layer could be found after the diffusion couple tests.

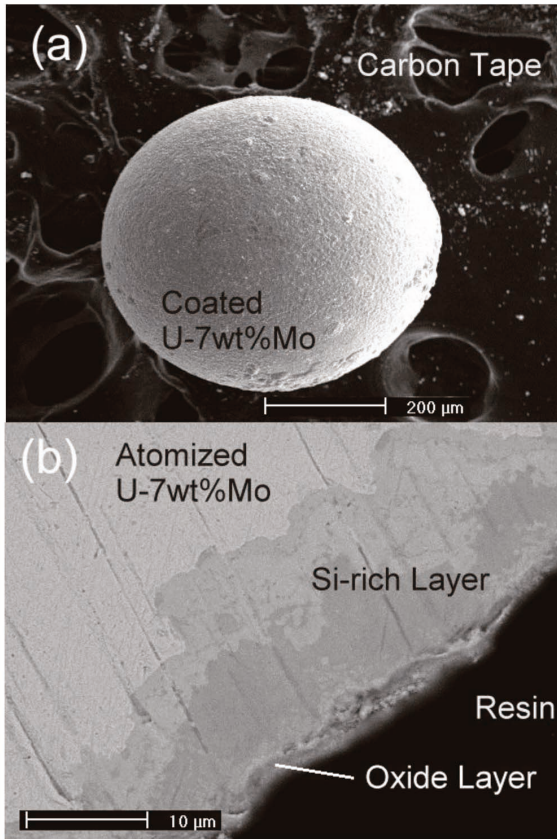


Fig. 7. (a) A Scanning Electron Micrograph of an Atomized U-7 wt%Mo Particle Annealed in a Si Powder Bed at  $900\ ^\circ\text{C}$  for 1 Hour Under Vacuum and (b) its Cross-sectional Micrograph

**Table 3.** Compositions of Coated Layers on the Surface of an Atomized U-7 wt%Mo Particle after Vacuum Annealing in a Si Powder Bed at  $900\ ^\circ\text{C}$  for 1 Hour

Element	Inner layer (at%)	Outer layer (at%)	Oxide layer (at%)
O	-	-	75.5
Si	50.2	61.7	3.9
Mo	1.5	7.7	1.0
U	48.3	30.6	19.6

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