Characterization of the Interaction Products in U-Mo/Al Dispersion Fuel from In-pile and Out-of-pile Tests

Ho Jin Ryu*, Yeon Soo Kim, G.L. Hofman
Argonne National Laboratory, 9700 S. Cass Ave, Argonne, IL 60439, USA
Assigned from Korea Atomic Energy Research Institute, Daejeon 305-353, Republic of Korea

D.D. Keiser
Idaho National Laboratory, P.O. Box 1625, Idaho Falls, ID 83415-6188, USA

ABSTRACT

The irradiation behavior of the interaction product in U-Mo/Al dispersion fuel during a pore formation resembles very closely that of an amorphous material, suggesting that the interaction product is amorphized and the structural instability of the interaction product is due to its amorphous character. However, no direct evidence as to whether it is amorphous or crystalline has been reported. Although extensive out-of-pile diffusion tests have been carried out to obtain metallurgical information of the interaction products, the interdiffusion behavior between U-Mo and Al is not fully understood and inconsistencies between out-of-pile and in-pile data still remain. In this paper, we analyzed all the available characterization results from both out-of-pile and in-pile tests to propose the most probable nature of the interaction product during irradiation and to establish the most consistent diffusion paths in the U-Mo-Al ternary system.

1. Introduction

U-Mo/Al dispersion fuel forms interaction products during irradiation as a result of the interdiffusion between U-Mo alloy and the Al matrix. The U-Al system has three intermetallic compounds of UAl_2, UAl_3, and UAl_4 uranium aluminides according to the U-Al phase diagram [1,2]. UAl_2 has a cubic Laves structure with a lattice parameter of 0.7766 nm. The crystallographic structure of UAl_3 is the $L1_2$ ordered simple cubic structure with a lattice parameter of 0.426 nm. UAl_4 is body-centered orthorhombic with lattice parameters of a=0.4397 nm, b=0.6251 nm, and c=1.3714 nm. In order to understand the reaction products of U-Mo/Al dispersion fuel, the Al-rich corner of a ternary phase diagram for the U-Mo-Al system is required. Only recently, efforts to establish the ternary phase diagram in the Al-rich corner of the U-Mo-Al system were undertaken [3].

Interaction product formation has been identified as the key issue in U-Mo/Al dispersion fuel development, particularly because of the porosity buildup problem observed in high power and burnup irradiation tests. Out-of-pile data from several experiments regarding metallurgical information on the interaction products of U-Mo/Al dispersion fuel have been carried out by using a diffusion-couple testing, casting, and a differential scanning calorimetry (DSC) [4-7]. In-pile test data are also available. However, the interdiffusion behavior between U-Mo and Al is still unclear due to inconsistencies between the out-of-pile and in-pile data as well as other inconsistencies among the various experiments.

A microstructural evolution of the porosity in the interaction product of U-Mo/Al dispersion fuel during irradiation resembles that of an amorphous material [8], suggesting that the interaction

product is amorphized during irradiation and the structural instability of the interaction product that causes a pore formation is due to its amorphous character. Although uranium compounds such as U_6Fe, U_3Si, U_3Si_2 are known to be amorphized during irradiation [9,10], there is no reference dealing with the radiation-induced amorphization of the interaction products in U-Mo/Al dispersion fuel.

In this paper, we analyzed the available characterization results from both out-of-pile and in-pile tests to clarify the inconsistencies and to propose the most probable nature of the interaction product during irradiation. We found that the interaction products in the U-Mo/Al dispersion fuel during irradiation were amorphous depending on the fuel temperature and irradiation condition. We also established the most consistent diffusion paths for the U-Mo-Al ternary system.

2. Data review

2.1. Out-of-pile tests

Compositions of interaction products have been obtained from diffusion couple tests of U-Mo vs. Al as marked in the ternary diagram shown in Fig. 1. In the diffusion couple tests of U-10wt%Mo vs. Al at 550°C for 5 hr and 25 hr, Ryu *et al.* observed three layered interdiffusion zone [4]. The atomic ratios of Al to (U+Mo) for 'L1', 'L2' and 'L3' (from the U-Mo side to the Al side) are 3.5, 4.4, and 7.6, respectively. Mirandou *et al.* also carried out diffusion couple tests of U-7wt%Mo vs. Al at 580°C for 4 hr and two layers were found in their diffusion couples [5]. Mazaudier *et al.* also observed an Al-rich phase $U_3Mo_4Al_{93}$ in U-(5 to 10wt%)Mo/Al diffusion couple tests at 600°C [3]. They noted that the thickness of the Al-rich phase increases with the Mo content in U-Mo alloy in the range of 5 – 10 wt%. This suggests that Mo promotes the formation of Al-rich compounds. Keiser *et al.* used diffusion couples of U-7Mo vs. Al annealed at 575 - 650°C [11]. In Table 1, the interaction phases observed by Keiser *et al.* for each test temperature are listed. Al-rich phases, $U_9Mo_1Al_{90}$ and $U_4Mo_3Al_{93}$, were observed. The higher-Al compounds (above UAl_4) were observed at a higher temperature (625°C) in their diffusion tests.

Table 1. Observed phases in U-7Mo/Al diffusion couples tests by Keiser et al. [11].

Temperature (^{o}C)	Time (hr)	Composition of observed phases (at%)			
		$(U,Mo)Al_2$	$(U,Mo)Al_3$	$(U,Mo)Al_4$	$(U,Mo)Al_{7+}$
575	0.5				$U_9Mo_1Al_{90}$
575	50	$U_{27}Mo_1Al_{72}$	$U_{22}Mo_3Al_{75}$		
575	50	$U_{28}Mo_1Al_{71}$	$U_{17}Mo_7Al_{76}$ $U_{22}Mo_3Al_{75}$		
625	50		$U_{22}Mo_3Al_{75}$	$U_{14}Mo_3Al_{83}$	$U_4Mo_3Al_{93}$

As can be seen in the SEM image of Fig. 2(a), the interaction product in U-Mo/Al dispersion fuel from an annealing test consists of several multiphase layers. Several researchers have also characterized the interaction products formed in out-of-pile dispersion or diffusion-couple tests of U-Mo vs. Al by using the X-ray diffraction (XRD) method [4,5,14]. Figure 2(b) shows the XRD patterns of the interaction layers of U-10wt%Mo/Al dispersion fuel annealed at 550°C for 40 hr [4]. The diffraction peaks of the UAl_3 and UAl_4 phases were observed.

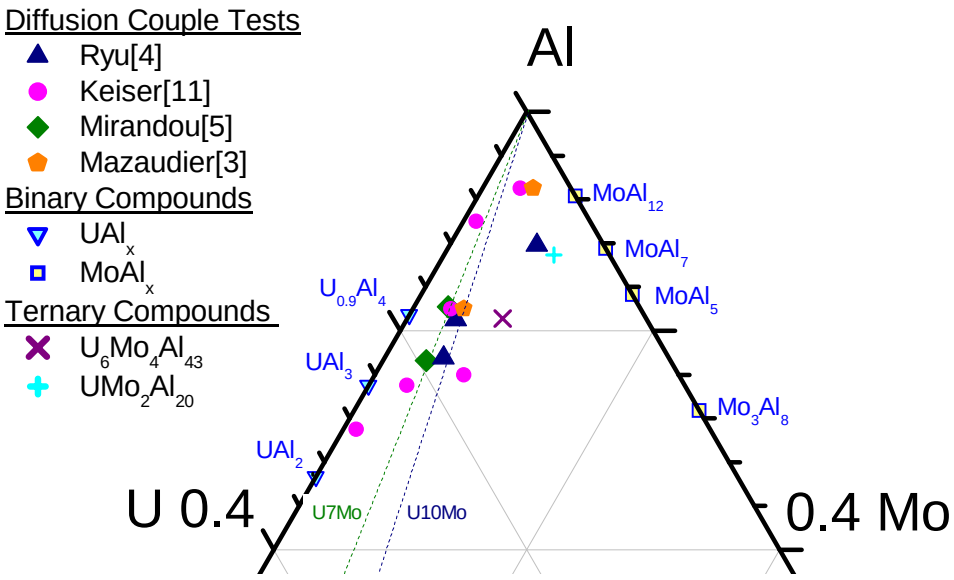


Fig. 1. U-Mo-Al ternary diagram showing observed ternary phases in out-of-pile U-Mo vs. Al diffusion couple tests [3-5,11]. The test temperature ranges 550-625°C, which means this ternary diagram is a composite of several isothermal sections.

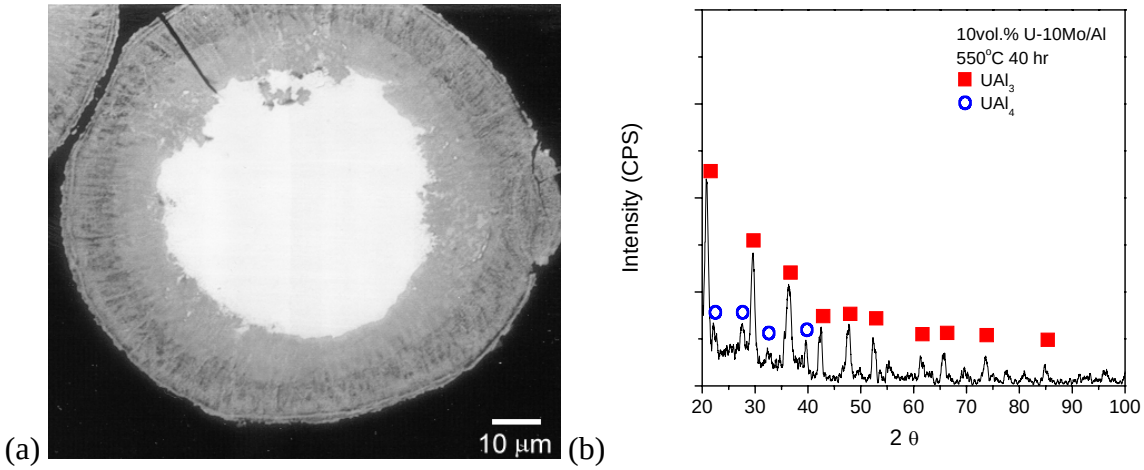


Fig. 2. (a) Scanning electron micrograph of interaction layers in U-10Mo/Al dispersion fuel annealed at 550°C for 25 hr, (b) XRD patterns of interaction layers in U-10Mo/Al dispersion fuel annealed at 550°C for 40 hr [4].

Mirandou *et al*. also reported UAl_3 and UAl_4 diffraction peaks from the interaction layers of a U-7wt%Mo vs. Al diffusion couple annealed at 580°C [5]. They also observed the minor peaks of the $U_6Mo_4Al_{43}$ and UMo_2Al_{20} phases. UMo_2Al_{20} has a cubic structure with a lattice parameter of 1.4506 nm [12]. $U_6Mo_4Al_{43}$ has a hexagonal structure with lattice parameters of 1.0966 and 1.7690 nm [13]. Keiser *et al.* also found $U_{11}Mo_7Al_{82}$ and $U_5Mo_6Al_{89}$ phases that are similar to $U_6Mo_4Al_{43}$ and UMo_2Al_{20} from annealed castings of U-Mo-Al alloys [6]. Palancher *et al*. also detected the Al-rich ternary phases, *i.e.*, $U_6Mo_4Al_{43}$ and UMo_2Al_{20}, in annealed U-7wt%Mo vs.

Al diffusion couples at 600°C for 10 hr [14]. Micro-XRD results using a synchrotron radiation revealed that each layer is composed of multiphase mixtures of UAl_3, UAl_4, $U_6Mo_4Al_{43}$, and UMo_2Al_{20}.

It is remarkable that no observation of a diffraction pattern for UAl_2 has been reported for the out-of-pile diffusion tests of U-Mo vs. Al. The reason may be that the formation of the UAl_3 phase in U-Mo vs. Al tests is thermodynamically more favorable that the UAl_2 phase. When the Al supply to UAl_3 from the Al side is less than that lost to U at the U-UAl_3 interface, however, UAl_3 transforms to UAl_2 because the Al loss overcomes the threshold energy for UAl_2 nucleation. A similar case was observed in an annealing test of U_3Si/Al and U_3Si_2/Al dispersion fuels at 600°C for 33 days with a 50-vol.% fuel loading in which the $U(Al,Si)_2$ phase formed only when the Al matrix is completely consumed [15]. In these fuels $U(Al,Si)_3$ is normally the phase to form as long as the Al matrix remains.

2.2. In-pile tests

Post-irradiation examination (PIE) results of the RERTR, IRIS-1 and -2, FUTURE, and KOMO-1 and -2 irradiation tests are available in terms of compositions of the interaction layers in irradiated U-Mo/Al dispersion fuel [16-19]. The temperatures for these tests were below 200°C; however, the fuel central region temperatures were above 200°C for some high power rods in the KOMO-2 test. Meyer *et al.* measured the compositions of the interaction layers of the irradiated fuel from the RERTR-3 irradiation test by using a wavelength dispersive X-ray spectroscopy (WDS) as shown in Fig. 3 [16]. Although the interaction zone observed by SEM appears to be a single homogeneous phase, compositions of the interaction zones irradiated in the IRIS-2 test varied in the interaction zone and by the location in the sample with a range of $(U,Mo)Al_{4.4}$ - $(U,Mo)Al_{5.8}$ [17]. The interaction products of the irradiated fuel from the IRIS-1 test had the composition range of $(U,Mo)Al_6$ - $(U,Mo)Al_7$ whereas those from the FUTURE irradiation test were in the range of $(U,Mo)_{3.3}$ - $(U,Mo)_{4.7}$ [18].

The PIE of the KOMO-2 irradiation test exhibited the similar relation of composition and fuel temperature [19]. Interaction layers in the cooler periphery of the rod-type fuel meat had a higher Al/(U+Mo) ratio than those in the hotter inner area of fuel meat as shown in Fig. 3.

Since the XRD method was first used for characterizing the interaction products formed during irradiation, no observation has ever been made to show the presence of a uranium aluminide with the Al/U ratio of 4 or higher. This is a contrasting feature to the out-of-pile diffusion-couple test results. Dubois *et al*. reported that they observed only U-Mo fuel and aluminum phases but no UAl_2, UAl_3 and UAl_4 peaks from the XRD tests of the IRIS-1 and IRIS-2 irradiation samples [20]. The measured volume fractions of the interaction zone of the IRIS-1 and IRIS-2 were 37% and 45%, respectively, i.e., large enough to yield a diffraction pattern .

Abundant data are available for the reaction of U (without Mo) in Al either with or without Si addition during irradiation. Dienst *et al*. [21] and Hofman [22] independently showed similar results that an interaction product forms in the UAl_x/Al dispersion fuel by the reaction of UAl_2 and UAl_3 with Al as shown in Fig. 4. Although they did not identify the interaction product by a

diffraction study, they assumed the interaction phase to be UAl_4 because UAl_4 is the only possible phase higher than UAl_2 and UAl_3 according to the U-Al phase diagram.

Richt *et al*. [23] reported on the XRD results of irradiated 48wt%U-49wt%Al-3wt%Si alloy fuel. An interaction layer was formed on the UAl_3 particles. This means that the interaction product must be UAl_4 or higher. However, they only detected that the UAl_3 phase had undergone an order-disorder transition. The UAl_4 phase was not detected. The absence of the diffraction pattern of UAl_4 from these tests is indirect evidence that the interaction product is not crystalline. Amorphization is due to a destruction of a lattice structure by irradiation damage (specifically, by fission fragment damage). In this situation, the interaction product remains as a non-equilibrium mixture of U and Al where the Al/U ratio of the interaction product does not need to be an integer, similar to the U-Mo/Al irradiation results discussed earlier. From this review, we found that a high Al-content interaction product, i.e., higher in Al-content than UAl_3, consistently tends to undergo an amorphization.

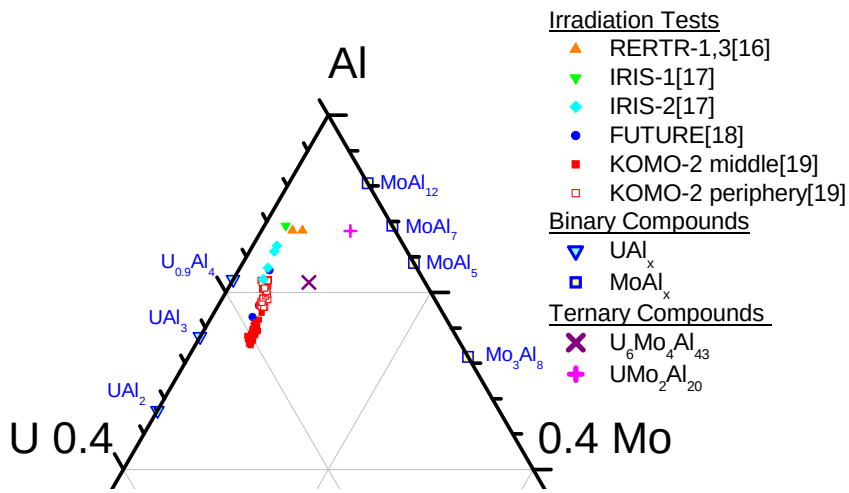


Fig. 3. U-Mo-Al ternary diagram showing observed ternary phase in U-Mo/Al irradiation tests.

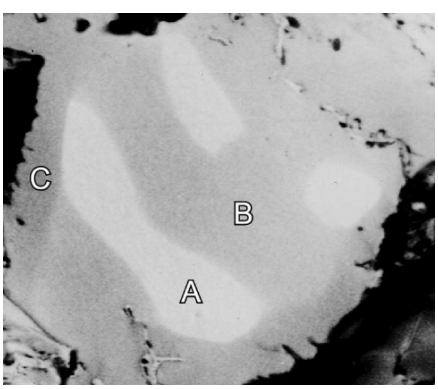
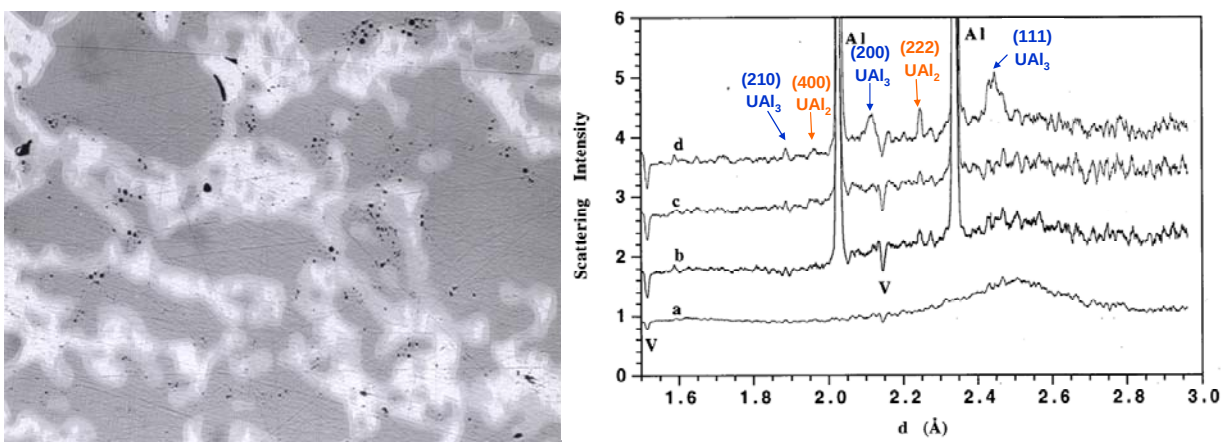


Fig. 4. A scanning electron micrograph of 93% enriched UAl_x/Al dispersion fuel after 60% U-235 burnup [22]. A is the UAl_2, B is the UAl_3, C is the interaction product.

Conlon and Sears reported on neutron diffraction analysis (NDA) results of rod-type U-Mo/Al dispersion fuel irradiated to 20% U-235 burnup [24]. It is noticeable that the amount of UAl_3 is predominant in the reaction product. They calculated the weight fractions of UAl_2 and UAl_4 by the Rietveld method to be 1 and 4 wt%, respectively. As Ryu *et al.* [25] reported, the fuel temperature of this type fuel with a similar power is much higher (the fuel centerline temperature ~400°C) than a plate type fuel (the fuel centerline temperature ~200°C). Indeed, the post-irradiation micrographs of Colon and Sears exhibited a complete consumption of the U-Mo

particles and Al matrix at the center of the fuel rod, which is also seen in Ref. 25. Comparing the burnup and the extent of interaction between U-Mo and Al with those of Ref. 25, the fuel centerline temperature of Ref. 24 can be estimated to be close to that of Ref. 25. Therefore, the finding of UAl_4 can be explained by the high fuel temperature at which the UAl_4 phase remains crystalline.

The diffraction patterns obtained from the U_3Si_2/Al dispersion fuel is another piece of evidence for an amorphization of the interaction product and the effect of the temperature on an amorphization [26]. U_3Si_2/Al dispersion fuel was irradiated to a 85% U-235 burnup (fission density of ~4.2 x 10^{21} /cm^3 fuel) in the ORR and post-irradiation annealed. The interaction layer remained with the same thickness after an annealing at 400°C for 700 hr as shown in Fig. 5(a), when compared with the as-irradiated thickness. Figure 5(b) shows the difference of the neutron diffraction patterns of the interaction products between the as-irradiated U_3Si_2/Al dispersion fuel and the annealed specimen. UAl_3 diffraction peaks of the as-irradiated fuel were too weak to be resolved in b and c of Fig. 5(b); however, they increased to distinguishable intensities after an annealing in d of Fig. 5(b). This result proves that the crystalline structure of an amorphous interaction product during irradiation could be restored by a high temperature annealing. Conversely, the amorphization of UAl_3 during irradiation seems to be due to the low fuel temperature ~90°C. The UAl_2 peaks marked in b and c of Fig. 5(b) is questionable because of their weak diffraction pattern.



(a) (b)

Fig. 5. (a) Optical micrograph of the 5.2 gU/cm^3 LEU U_3Si_2/Al dispersion fuel irradiated to a 85% U-235 burnup in the ORR and post-irradiation annealed at 400°C for 700 hr. (b) Neutron diffraction patterns of the U_3Si_2/Al dispersion fuel. (a: amorphized U_3Si_2 powder by neutron irradiation, b: low burnup (45% U-235 burnup) irradiated U_3Si_2/Al plate, c: high burnup (85% U-235 burnup) irradiated U_3Si_2/Al plate, d: annealed at 400°C for 700 hr after irradiation).

The inconsistency regarding the observation of UAl_3 among the test results is attributed to the difference in the irradiation temperature. Irradiation temperature is an important factor in determining whether the interaction product becomes amorphous or remains crystalline. In general, the lower the irradiation temperature, the higher the tendency for an amorphization is.

3. Results and Discussion

The out-of-pile diffusion test results can be summarized as follows: (i) The Al-rich compounds, i.e., an Al/(U+Mo) atom ratio greater than 4, are the result of multiphase mixtures of UAl_3, $U_{0.9}Al_4$, $U_6Mo_4Al_{43}$, and UMo_2Al_{20}. (ii) The higher the Mo content, the more likely is the formation of an Al-rich phase such as $U_6Mo_4Al_{43}$ and UMo_2Al_{20}. Indeed, this was confirmed by the test results that U-10Mo vs. Al tends to form an additional higher Al-content layer than U-7Mo vs. Al [4].

Based on the review, we developed a U-Mo-Al ternary phase diagram for the Al-rich region at the temperature range of 550 - 600°C as shown in Fig. 6(a). Because the ternary diagram contains test results at different temperatures, we suggest that it is a poly-thermal superimposed section. $(U,Mo)Al_3$ was believed to have a solubility of 3 - 4 at% Mo according to the XRD results and EPMA results. Figure 6(b) shows the probable diffusion paths of the U-Mo vs. Al diffusion couples. The diffusion path proceeds directly from Al to two-phase-field of $(U,Mo)Al_3$ and U_2MoAl_{20} when the Mo content is lower than that in U-7wt%Mo. However, the single phase U_2MoAl_{20} is included in the diffusion path when the Mo content is high enough to assist in the formation of the Al-rich phases.

The in-pile test results can be summarized as four points: (i) As the fuel temperature increases, the average Al/(U+Mo) atom ratio of the interaction products decreases. (ii) An interaction product with a higher Al-content tends to be amorphized more easily. (iii) As the test temperature decreases, the interaction product becomes amorphous more easily. (iv) Interaction products in the low temperature irradiated fuel samples are homogeneous, whereas those in high temperature irradiated fuel seem to be multi-phase.

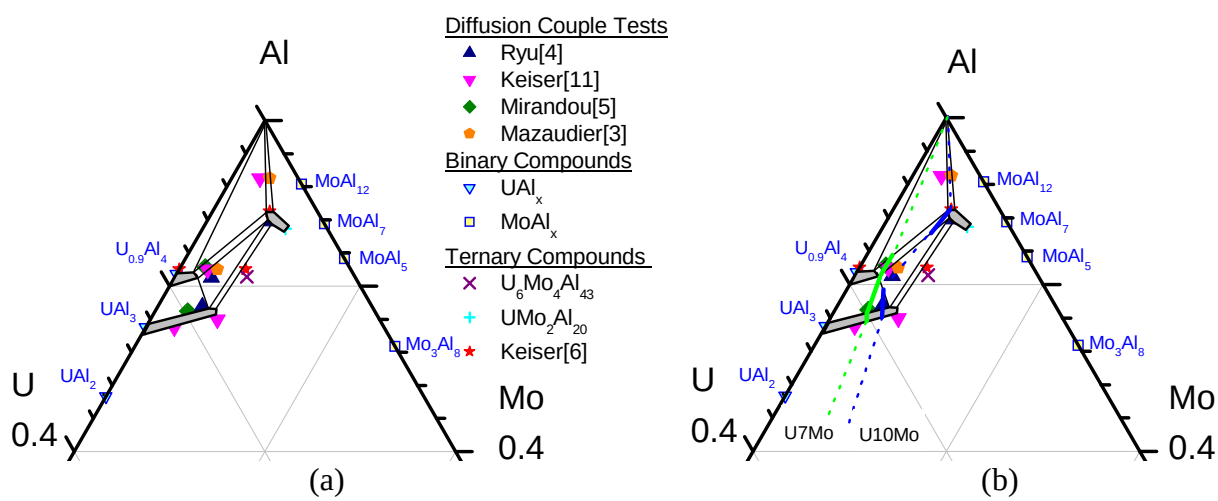


Fig. 6. (a) Schematic U-Mo-Al ternary diagram proposed in this study for 550-625°C. (b) U-Mo-Al ternary diagram showing diffusion paths in gamma phase U-Mo vs. Al diffusion couple.

Comparison between the out-of-pile and in-pile test results shows remarkable differences suggesting two important points:

- First, the composition of an interaction product has a relation with the fuel temperature. The fuel temperature for the IRIS-1 test is the lowest, that for the IRIS-2 is higher, and that for the FUTURE is the highest. This means that, the hotter the fuel, the lower the Al/(U+Mo) ratio for the irradiation tests is. Furthermore, the representative ratio for the out-of-pile tests ranges

from 3 to 4.4 in accordance with the phase diagram of the U-Al system, whereas the ratios of 3.3 to 7 were observed in in-pile tests. This difference can be explained when the interaction products were in a non-equilibrium state during irradiation. As stated in section 2.1, the Al/(U+Mo) ratio for the out-of-pile tests is thermodynamically determined. When the interaction product becomes amorphous at low temperatures, however, U, Mo and Al exist as a non-equilibrium mixture. In this situation, because of fission-fragment damage causing structural strain related to defects and disorder, the Al/(U+Mo) ratio can be higher than that possible for out-of-pile tests. On the other hand, when the fuel temperature increases, the amorphous product can restore crystallinity and the ratio has to decrease to 3 similar to the out-of-pile cases because the phase equilibrium of interaction product is dominated by thermodynamics at higher temperature. This is the reason why the rod-type U-Mo/Al dispersion fuel irradiated at a high temperature above 400°C showed crystalline UAl_3 diffraction peaks in post-irradiation examination.

- Second, the variation in the composition of the interaction product locally without showing phase boundaries suggests that the interaction product is not necessarily in a crystalline phase. Interaction layers developed in irradiated U-Mo/Al dispersion fuel did not show any evidence of the existence of ternary phases such as $U_6Mo_4Al_{43}$ and UMo_2Al_{20} found from the out-of-pile tests, which is another difference of the amorphous interaction product of U-Mo/Al dispersion fuel.

In order to satisfactorily explain the observations both from the out-of-pile and in-pile tests, the interaction products in some situations must be amorphous. The critical factor that determines whether an interaction product is amorphous or crystalline is believed to be the fuel temperature. There may exist a critical temperature above which an amorphization of a crystalline interaction product will not occur as illustrated in Fig. 7. The critical temperature may be proposed as a function of the fission rate, $\dot{f}$, and the composition of an interaction product, *x*, as follows:

$$T_c = f\left(\dot{f}, x\right) \tag{1}$$

where *f* is a function that increases as $\dot{f}$ increases and *x* increases. As discussed earlier, *x* is a function of the irradiation fuel temperature. The fission rate has an effect on the formation of the defects and disorders in the interaction products. In this regard, the critical temperature for an amorphization has similar variables as the critical damage rate function we developed for determining the growth of a pore into the interaction product [27]. The critical temperature for an amorphization of the interaction products in U-Mo/Al dispersion fuel was estimated to be around 300~400°C, by considering the results of the RERTR, KOMO and FUTURE irradiation tests. When more data are available, we may be able to complete this function.

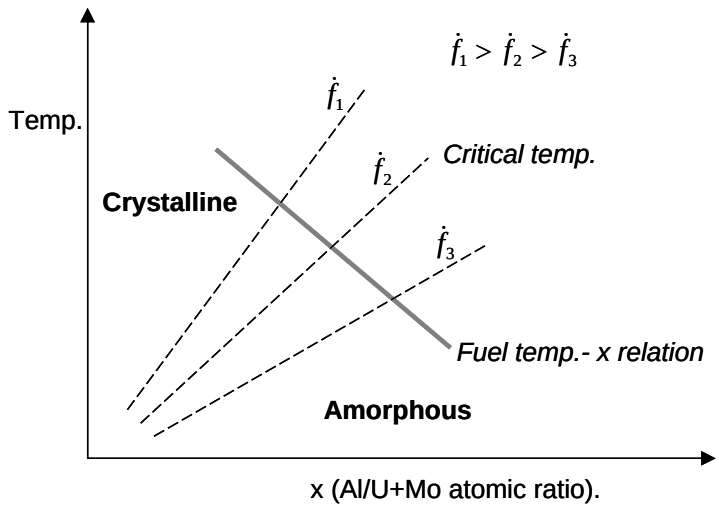


Fig. 7. A schematic diagram illustrating the concept of the critical temperature for amorphization of the interaction product during irradiation.

4. Conclusions

We maintain that the interaction products of U-Mo/Al plate-type dispersion fuel during irradiation are amorphous in order to explain the extensive post-irradiation examination results and to satisfy constraints therein.

We established the most acceptable diffusion paths in the Al-rich corner of U-Mo-Al ternary diagram by analyzing diffusion test results between U-Mo and Al. Diffusion paths of interaction products from out-of-pile and in-pile tests are different because the temperature regimes were different and the interaction products from the out-of-pile tests were thermodynamically determined whereas those of the in-pile tests were non-equilibrium mixtures of U, Mo and Al due to irradiation damage-induced amorphization.

Irradiation temperature is a critical factor in determining whether the interaction products become amorphous or not. We propose the concept of a critical fuel temperature to determine whether the interaction product is amorphous or crystalline as a function of the fission rate and the interaction product composition.

Acknowledgments

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