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MICROSTRUCTURAL CHARACTERIZATION OF THE E-FUTURE FRESH FUEL PLATES

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ABSTRACT

Within the framework of the LEONIDAS-US Partnership, the first experiment - called E-FUTURE - required to qualify U-Mo dispersion LEU fuel for the BR2 and the RHF high European reactors has been launched. The E-FUTURE test consists of 4 full sized plates made of U(Mo) atomized powders dispersed in Al with Si matrix (4 and 6 wt% Si). Three different pre-irradiation heat treatments have been applied in the aim of developing a Si-rich diffusion layer on the U(Mo) particles before the irradiation.
In this paper, the microstructural analyses (optical, SEM, EPMA) performed by CEA and SCK•CEN on the E-FUTURE fresh fuel plates are presented and discussed.

1. Introduction

Due to its high uranium density and its good stability under irradiation, U(Mo) alloy fuel is considered as one of the most promising fuel candidates for the HEU to LEU fuel conversion of high performance research reactors. For more than 10 years, a considerable effort has been devoted worldwide to the development of U(Mo) alloy dispersion fuel, mostly to mitigate the unsatisfactory in-pile behaviour of the atomised U(Mo) dispersed in pure Al at high heat fluxes, as it has been evidenced in past experiments such as the IRIS2 and FUTURE irradiations [1, 2]. A large part of the instability of atomised U(Mo)/Al fuel is attributed to the behaviour of the fission gas and the properties of the U(Mo)-Al interaction layer (IL) formed during the irradiation. The poor fission gas retention of the IL causes large pores to develop between the IL and the Al matrix, leading to excessive swelling or

pillowing of the fuel plates. It is well established that in mild operating conditions, typically the OSIRIS operating conditions, the addition of Si to the Al matrix of U(Mo) dispersion fuel has a positive effect, resulting in a better behaviour of full size plates under irradiation by stabilising the U(Mo)-Al interaction layer and slowing down its formation (cf. IRIS3 experiment [3]). It is now appropriate to verify that a similar improved behaviour of optimised U(Mo)-Al(Si) fuel is reproduced in more demanding operating conditions, fully representative for high power research reactors such as BR2, RHF and JHR.
In that context, the LEONIDAS Advanced Technology Group was formed in 2009 [4] to plan and execute experiments to qualify U(Mo)-Al(Si) dispersion LEU fuel for the conversion of the BR2 and RHF high flux European reactors. The LEONIDAS group is a collaboration between AREVA-CERCA, CEA, ILL and SCK•CEN. The US GTRI program has also partnered with the LEONIDAS group to accelerate work towards the common goals. Within the framework of the LEONIDAS-US Partnership [5], a first experiment, called E-FUTURE, has been designed to be the initial step to demonstrate the optimised U(Mo)-Al(Si) dispersion fuel performance at high heat fluxes, near 500 $W.cm^{-2}$. The objective of the E-FUTURE irradiation is to finalise and validate the choice of the optimal fabrication conditions in terms of Si content in the Al matrix and heat treatment.
In preparation for the E-FUTURE irradiation, microstructural analyses (optical, SEM, EPMA) were performed by CEA and SCK•CEN on as-fabricated fuel plates. Results of this characterization prior to the irradiation are presented in this paper and are helpful to better understand the in-pile microstructure evolution and therefore to fine-tune eventually the fabrication conditions for the next LEONIDAS qualification experiment of U(Mo)-Al(Si) dispersion fuel.

2. E-FUTURE Plates Design

2.1. Choice of Si content and thermal treatment conditions

The beneficial effect of the addition of small quantities of Si to the Al matrix on the rate of formation of U(Mo)-Al(Si) interaction layer and on the in-pile stability of this phase against the formation of large, instable fission gas pores has been clearly evidenced in various irradiation experiments, either on full size plates irradiations such as IRIS3 [6] and IRIS-TUM [7, 8] or on mini-plates tests such as RERTR-6, -7A, -9A, -9B [9, 10]. For example, the IRIS3 experiment has demonstrated that 2.1wt%Si was sufficient to improve significantly the in-pile fuel behaviour, since no abnormal swelling was observed contrary to the plates with Al-0.3wt%Si in mild operating conditions (peak heat flux was about 200 $W.cm^{-2}$). One of the conclusions drawn from RERTR experiments [10] is that a minimum of 4wt% Si is considered necessary to sustain the positive in-pile Si effect under more severe irradiation conditions. As a good compromise between a sufficiently high Si concentration in the Al to sustain its beneficial effect and the lowest possible Si content for fuel back-end (reprocessing) issues, E-FUTURE was designed with two matrix Si concentrations of 4wt% and 6wt%.
It is now also concluded from PIE results that the presence of a Si-rich layer at the U(Mo)-Al interface prior to the irradiation would be at least beneficial to avoid formation of UAl_4 known to be deleterious under irradiation. But as the silicon has a very low solubility in the aluminum, Si will be present in the form of precipitates. Si diffusion to the U(Mo)-Al interface by thermal activation can be promoted with appropriate thermal treatments (temperature, time) during fabrication. Elevated temperatures (500°C or more), as used in the fabrication of fuel plates with Al6061 cladding are considered too severe for softer Al alloys cladding such as AlFeNi or AG3NE and could lead to an excessive blister rejection ratio. Moreover, at those temperatures, γ–U(Mo) decomposition is significant [11, 12, 13] with apparition of a $U_6Mo_4Al_{43}$ phase, considered as an unstable phase as derived from ion irradiation

experiments [14]. Finally, 3 kinds of thermal treatments have been selected for the E-FUTURE fabrication:

- a relatively low annealing temperature of 425°C during 2h; quite similar to the IRIS3 reference fabrication conditions. In the IRIS3 experiment, a high Si concentration was measured in the Si-rich layer prior to the irradiation with the presence of $U(Al,Si)_3$ and U_3Si_5 phases, in good agreement with out-of-pile experiments [11].
- two annealing treatments at a medium temperature of 475°C during respectively 2h and 4h with the aim of varying the pre-irradiation layer thickness and maybe its composition [11] by thermally enhancing the Si diffusion towards the surface of the U(Mo) particles.

2.2. E-FUTURE plates fabrication

The E-FUTURE plates were fabricated by AREVA-CERCA with atomised U7Mo powder dispersed in Al matrix with either 4wt% or 6wt% Si. Two kinds of cladding - AlFeNi and AG3NE - were used, as regular claddings of respectively RHF and JHR for AlFeNi and BR2 for AG3NE. The plates have a high uranium loading of 8 $gU.cm^{-3}$ and an uranium enrichment of about 19.6 % ^{235}U. The as-fabricated porosity is low, comprised between 1.2 and to 2.6 vol%, in good agreement with previous fabrications made with atomised U(Mo) powders [15]. The main characteristics of the fabrication conditions of the E-FUTURE plates are summarized in Table 1, in which one can also find the selected plates for the irradiation test and those chosen for the fresh fuel plates microstructural analysis.

Si content in Al matrix	Thermal treatment	Cladding	Plate Id.	Selected for…
4wt%	425°C 2h	AlFeNi	U7MC4111	In-pile in BR2
		AlFeNi	U7MC4112	Fresh fuel examination
	475°C 2h	AG3NE	U7MC4201	Fresh fuel examination
		AG3NE	U7MC4202	In-pile in BR2
6wt%	425°C 2h	AG3NE	U7MC6101	Fresh fuel examination
		AlFeNi	U7MC6111	In-pile in BR2
	475°C 4h	AG3NE	U7MC6301	In-pile in BR2
		AlFeNi	U7MC6311	Fresh fuel examination

Table 1: E-FUTURE fabrication matrix

3. Characterization plan

The LEONIDAS Group defined the fresh fuel features to be studied in priority on the four plates as follows:

- the main characteristics of the meat such as the U(Mo) particle distribution, the porosity, etc.
- the meat/cladding interface to investigate the eventual presence of Mg_2Si precipitates,
- the fuel particle microstructure and crystal structure (eventual decomposition of the γ phase),
- the U(Mo)-Al(Si) matrix interaction layers (thickness, composition…),

- the Si precipitate size variation and their distribution in the Al matrix.

The AlFeNi cladding plates were preferentially characterized by the CEA when the AG3NE plates were analyzed by the SCK•CEN. The characterization tools and methods used by the CEA and by the SCK•CEN were different in certain cases. Table 2 recaps the applied analysis strategies in both laboratories. The main differences can only be found in the quantifications of U(Mo)-matrix interaction layer thicknesses and compositions.

Two types of samples were cut and prepared from each plate:
- two cross-section samples, one cut perpendicular to the rolling direction, one parallel to this direction, characterized with OM, SEM and EDS or EPMA,
- one sample embedded and polished parallel to the meat/cladding interface, down to the middle part of the meat, for XRD measurements.

	CEA	SCK•CEN
Meat characteristics	OM	OM
Meat/cladding interface	OM + SEM and X-Ray maps (SEM-EDS)	OM + SEM and X-Ray maps (SEM-EDS+EPMA)
U(Mo) particles ▪ microstructure ▪ structure	 OM XRD	 OM XRD
U(Mo)/matrix interaction layers ▪ thickness ▪ composition	 SEM + image analysis (method 1) SEM-EDS (point analyses)	 SEM + image analysis (method 2) EPMA (linescans)
Si particles distribution in the matrix	OM X-ray maps (SEM-EDS)	OM X-ray maps (SEM-EDS+EPMA)

OM: Optical Microscopy – SEM: Scanning Electron Microscopy – EDS: Energy dispersive Spectroscopy
EPMA: Electron Probe MicroAnalysis – XRD: X-Ray Diffraction

Table 2: Characterized parameters and means used by CEA and SCK•CEN.

Using the cross-section samples, it was confirmed that the relative orientation of the cutting plane and the rolling direction has no significant influence on the microstructural characteristics to be studied.
The thicknesses of the as-fabricated interaction layers (called SiRDL, for "Si rich diffusion layers") formed at U(Mo)-matrix interfaces were measured by image analysis, on SEM images in back-scattered electrons (BSE) mode, using two different methods:
- Method 1: this method consists in detecting the boundaries of the SiRDL (thanks to its grey level range), measuring the distance between these two boundaries, perpendicular to the SiRDL skeleton at 50 to 200 different points and calculating its mean, minimum and maximum values. It was applied on U(Mo) particles taking into account the following criteria: particle diameter > 20 µm (in order to limit edge cut effects, leading to an apparent widening of the SIRDL), relatively round shape, SiRDL without marked intergranular propagation.
- Method 2: the SiRDL thickness is measured at four fixed locations (cross-wise) on 20 randomly chosen particles.

The main difference between these two methods lies in the statistical weight represented by areas without any SiRDL. In method 1, this weight is low because such areas were not selected for measurements and only a few points, in the selected areas, present no SIRDL. In method 2, areas without any or with very little SiRDL are randomly taken into account and are thus more often measured, resulting in lower average thicknesses.

4. Results

4.1. Main characteristics of the meat

All the cross-sections were carefully examined by OM and no particularities were noticed: the U(Mo) particles were regularly distributed in the meat and few porosities were observed, in accordance with global density measurements performed at AREVA-CERCA: see Figure 1, for example.

4.2. Meat-cladding interface

In the AlFeNi cladding plates (AlFeNi contains less than 1% Mg), OM examinations did not evidence any precipitates aligned at the meat-cladding interface. However, a few associations of Si and Mg were observed on X-Ray maps, only in the plate U7MC4112, which tends to indicate that some Mg_2Si precipitates are present at this interface.
In the AG3 cladding plates (AG3 contains 2.8% Mg), Mg_2Si precipitates are clearly visible, aligned on the meat-cladding boundary of the U7MC6101 plate, it is less clear for the U7MC4201 plate which received the higher temperature heat treatment.

4.3. U(Mo) particles microstructure and structure

OM examinations clearly evidenced signs of γ-U(Mo) phase decomposition for all the studied plates. These signs were logically more pronounced in samples coming from the plates which were heat treated at 475°C, as illustrated by Figure 1, which compares two plates respectively annealed at 425 and 475°C. At 425°C, only an apparent broadening of cell boundaries (probably due to a minor γ phase decomposition) is observed, whereas at 475°C, the γ phase decomposition is obvious (compare encircled particles). It is also worth noting that Si particles in the matrix are quite easily observed in plates annealed at 425°C (some of them are indicated by pink stars in Figure 1a), whereas they are significantly less numerous in those annealed at 475°C (see section 4.5.). This could be interpreted as an indication that small Si particles have already reacted with U(Mo).

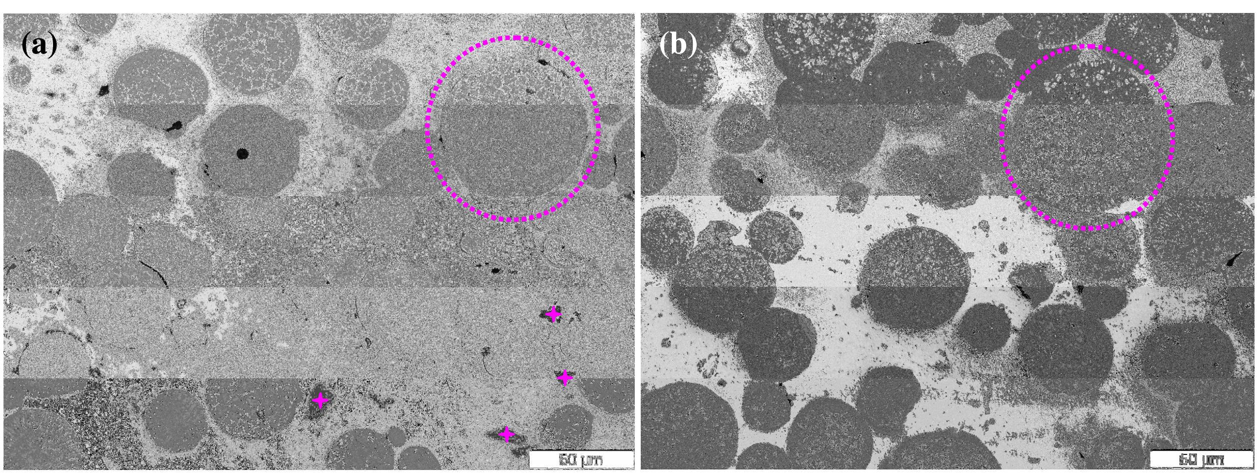

Figure 1: Comparison of the microstructures observed by OM :
(a) plate #4112 (4%Si, 425°C – 2h, AlFeNi), (b) plate #6311 (6%Si, 475°C – 4h, AlFeNi)

XRD measurements confirmed that a partial γ-U(Mo) phase decomposition has occurred in all the plates, with a more pronounced effect in the plates annealed at 475°C: see Figure 2. The volume fraction of interaction phases being low (see section 4.4.), only a $U(Al,Si)_3$ phase was observed slightly above the background, especially in the plate U7MC6311: cf. dotted purple line in Figure 2. Rietveld refinements of the diffractograms were performed with the TOPAS 4 software (commercialized by Bruker AXS). Results are only indicative, due to an overestimation of the Al content and a lack of detection of the minor phases (U_2Mo…), when studying this type of samples by conventional XRD [16]. The results do show that the amount of α-U phase approximately doubled between the plates annealed 2 hours at 425°C and the U7MC6311 plate, annealed 4 hours at 475°C.

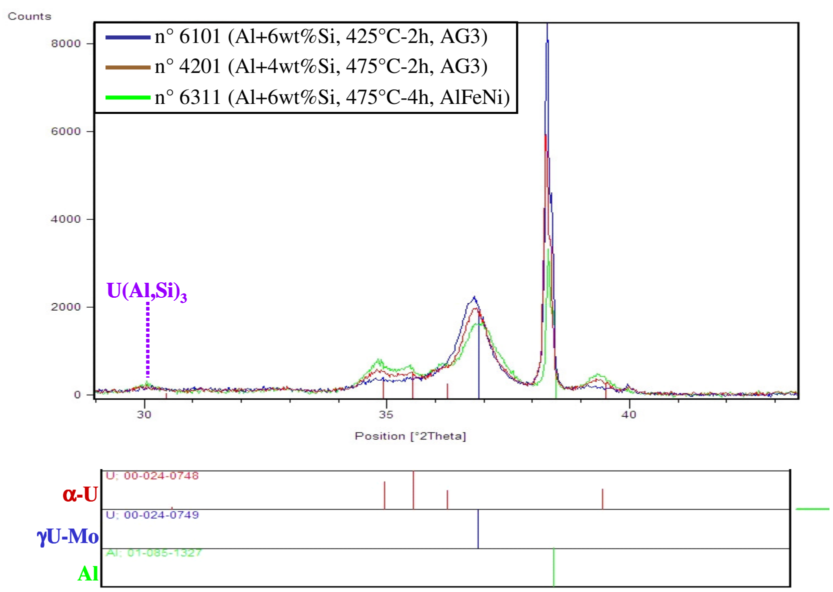

Figure 2: XRD results on plates #6101, #4201 and #6311

4.4. U(Mo)/matrix interaction layers

All the observations performed by OM and by SEM on the 4 plates led to the same conclusions, i.e:

- almost all U(Mo) particles present interaction areas at some points on their periphery,
- the SiRDL is often not present on the entire particle circumference and its thickness is irregular,
- intergranular propagation of the interaction is observed inside certain particles.

Figure 3 gathers typical views of each sample. SiRDL thicknesses appear qualitatively larger in plates annealed at 475°C, compared to those annealed at 425°C. Si particles are difficult to evidence in SEM standard imaging modes. Some of them can be observed in Figure 3d (pink stars).

Plate Id.	Method	Mean value (µm)	Maximum value (µm)
U7MC4112	1	0.7	4.5
U7MC6101	2	0.6	3.6
U7MC4201	2	0.8	3.6
U7MC6311	1	1.3	7.9

Table 3: Mean and maximum SiRDL thicknesses measured on each plate.

Table 3 recaps the values of the mean and maximum SiRDL thicknesses measured on each plate. As previously explained (cf. section 3), the mean values are not directly comparable, since they are determined with 2 different methods (they seem to differ by a factor of the order of 2, for a given annealing condition). With the method 1, an approximate doubling of these values is to be noticed, when increasing the annealing conditions from 425°C, 2h to 475°C, 4h.

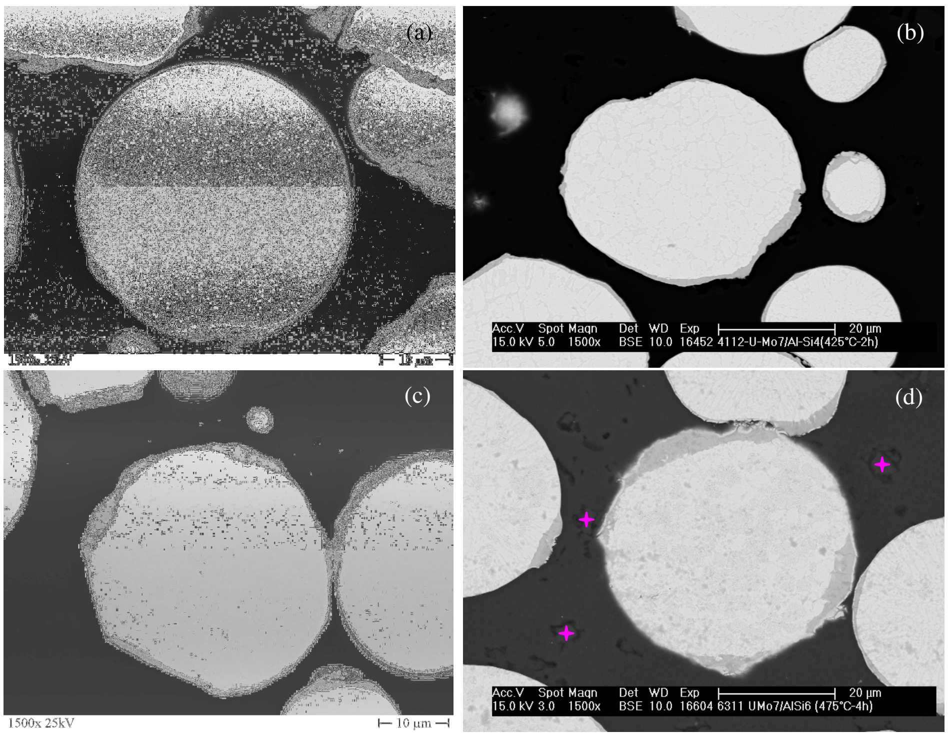

Figure 3: SEM micrographs (BSE mode) of (a) plate #6101 (6%Si, 425°C-2h, AG3NE), (b) plate #4112 (4%Si, 425°C-2h, AlFeNi), (c) plate #4201 (4%Si, 475°C-2h, AG3NE), (d) plate #6311 (6%Si, 475°C-4h, AlFeNi)

Table 4 gathers the results of Si content measurements in the SiRDLs. Point analyses by EDS were performed on plates with AlFeNi cladding, on homogeneous parts of the SiRDL (i.e. with no intergranular propagation) with a thickness of at least 2.5 µm, in order to avoid as far as possible a contribution of the surrounding materials. EPMA linescans were performed on plates with AG3 cladding. In this case, the composition of the sample was measured over a line in steps of approximately 1 µm. An average Si concentration is calculated over the thickness corresponding to SiRDL.

Plate Id.	Method	Mean value (at%)	Minimum value (at%)	Maximum value (at%)
U7MC4112	EDS	50	42	55
U7MC6101	EPMA	38	13	53
U7MC4201	EPMA	33	23	42
U7MC6311	EDS	36	25	50

Table 4: Si content measurements in SiRDL, on each plate.

4.5. Si particle distribution in the Al(Si) matrix

In Figures 1a and 3d, some Si particles were already pointed out (cf. pink stars). In fact, on both OM and SEM images, it was difficult to perform quantitative image analysis of these particles. Only qualitative information is given. In all cases:

- only relatively large Si particles are observed: their mean size is of the order of 5 µm, the largest ones reaching 20 µm,
- these particles often do not present a fully cohesive interface with the matrix, but this may be in part related to sample preparation and the brittle nature of the Si particles,
- there is no clear correlation between the presence of such Si particles at the closest U(Mo) boundary and the formation of a SiRDL.

These observations tend to indicate that the smallest Si particles probably disappeared during the annealing of the plates and led to the SiRDL development, while the largest remaining ones probably did not play a major role in the interaction process.

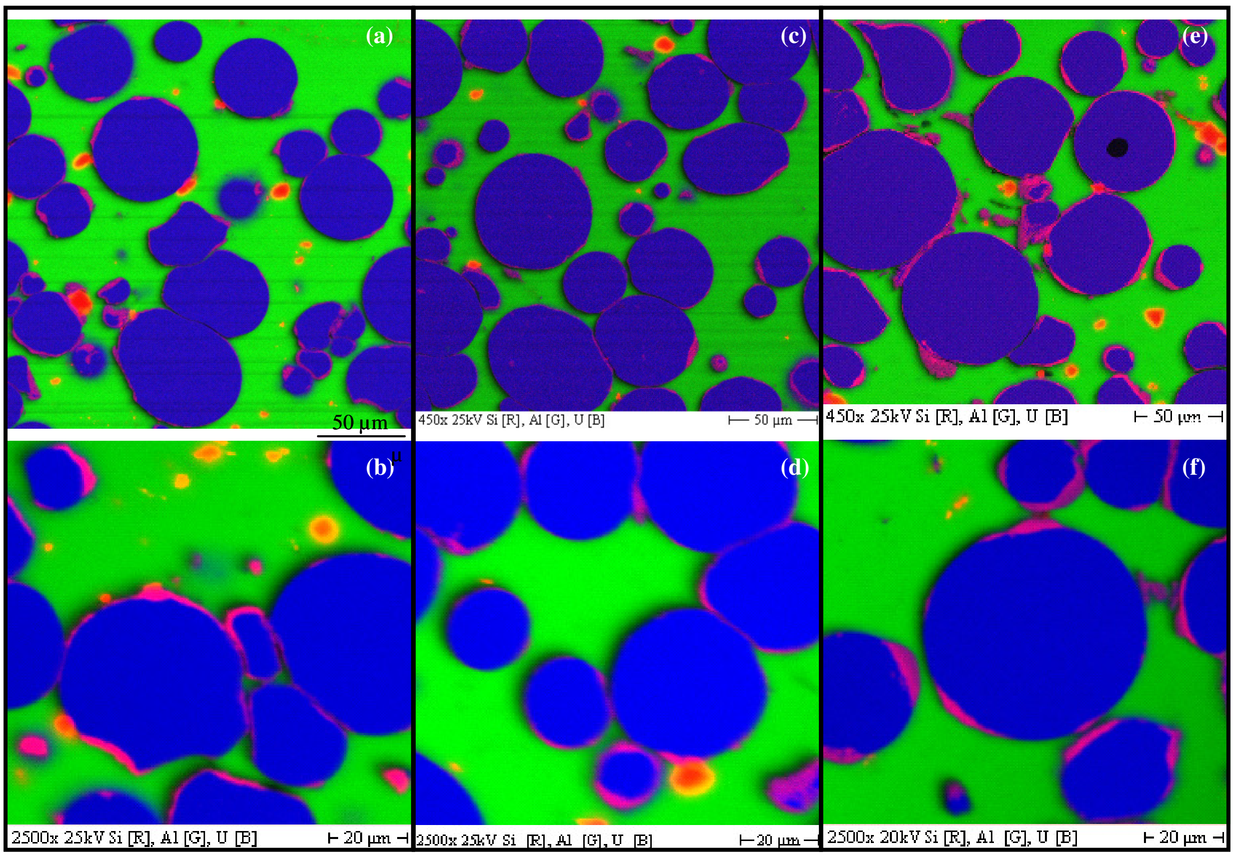

Figure 4: Si X-ray maps (EPMA) on 3 of the studied plates, at two magnifications (a,b) plate #6101 (6%Si, 425°C-2h, AG3NE), (c,d) plate #4201 (4%Si, 475°C-2h, AG3NE), (e,f) plate #6311 (6%Si, 475°C-4h, AlFeNi)

Figure 4 shows typical Si X-Ray maps obtained by EPMA on three of the studied plates, illustrating the 3 different annealing conditions. All these maps are relatively similar: irregular SiRDLs surrounding U(Mo) particles are clearly evidenced and few Si particles in the Al matrix are detected. However, the thickness of the SiRDL and the covered fraction of the U(Mo) particle surfaces appear higher in the plate U7MC6311, annealed at 475°C 4h . The number of remaining Si particles is conversely lower, compared to the plate U7MC6101, annealed at 425°C 2h (which has the same original Si content). The plate U7MC4201 is characterized by markedly less Si particles, in accordance with its lower Si content.

5. Discussion

5.1. Comparison with IRIS3 plate status before irradiation

IRIS3 and E-FUTURE plates were fabricated by AREVA-CERCA, using the same process with the same kind of atomised U(Mo) powders. In the framework of the IRIS3 experiment, two plates with an Al matrix added with 2.1wt% Si matrix were irradiated [3]. Two main differences in the production process should be noted between IRIS3 and the E-FUTURE:

- the IRIS3 plates were fabricated with an Al-Si alloy powder, whereas the E-FUTURE plates were fabricated with a mixture of Al and Si powders,
- the IRIS3 plates underwent a blister anneal treatment at 425°C for less than 2 hours.

The first point leads to major differences in terms of distribution of Si particles in the matrix: in the IRIS3 plates, Si particles had a typical size of one to a few µm and they were nearly uniformly distributed in the aluminum matrix (cf. Figures 4 and 5).

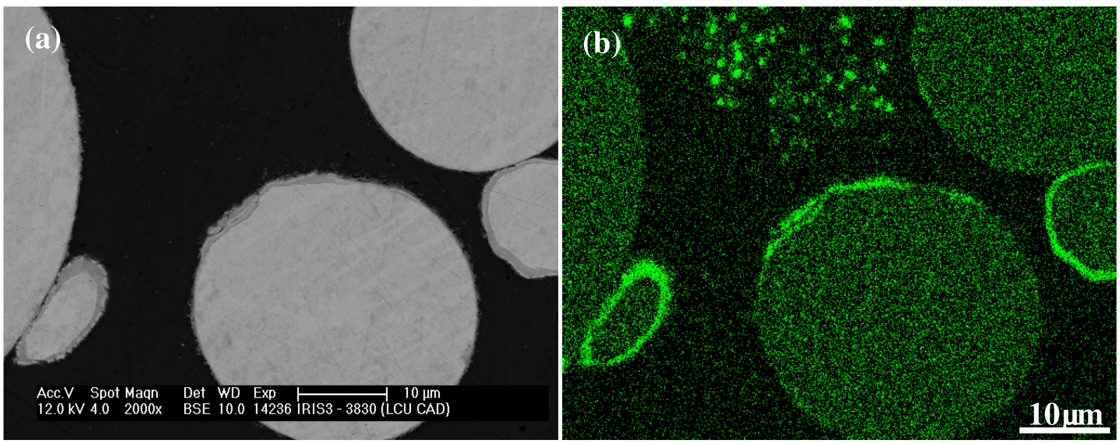

Figure 5: IRIS3, with Al+2wt%Si, as fabricated plate
(a) SEM micrograph (BSE mode), (b) Si X-ray map (EDS)

After the blister test, a thin and discontinuous SiRDL (thickness ≤ 1 µm) was present around many U(Mo) particles (cf. Figure 5) with in certain cases a depletion effect in the matrix (absence of Si particles in a zone of about 5 to 10 µm in width around the U(Mo) particle). According to pointwise EDS analyses, the Si enrichment of the observed SiRDL was of the order of 50at%.

These data allow to conclude that the IRIS3 plates and the E-FUTURE plates heat treated at 425°C do not show important differences in their SiRDL characteristics, except the fact that these layers are present around almost all the U(Mo) particles in E-FUTURE (even if they are discontinuous) and are probably slightly thicker. Concerning the Si particles still remaining in the matrix after the annealing step, the 2 types of plates are significantly different:

- in the IRIS3 plates, numerous small Si particles are still present in the matrix,
- in the E-FUTURE plates, only a few large precipitates (mean size ~ 5 µm) are present.

These differences could have an impact on the irradiation behaviour of the E-FUTURE plates, since less Si will be available at the direct vicinity of the U(Mo) particles. Indeed, previous results showed that Si particles played a beneficial role on the in-pile SiDRL stability only when they were very close to U(Mo) particles [7].

5.2. Comparison with RERTR plates state before irradiation

The results of these analyses can be compared to the results presented by D.D. Keiser on the production of a number of fuel plate samples with different annealing temperatures and times [17]. In the latter study, plates were produced using AA5052 Al alloy, which is similar to AG3NE in composition. The plate matrices were produced with different Si contents, the rolling of the plates was done at 425°C. And various heat treatments were applied.
The closest comparison can be made in the case of the 475°C/4h anneal of the 6w%Si plate, since for the other RERTR plates the report does not contain results on exactly the same anneals/Si content combinations. A similar microstructure of the fuel/matrix interaction layers is described and Si concentrations in the SiDRL varying between 26 and 41 at% are measured, which is comparable to the results in this study. In the thicker layers, probably related to decomposition regions, the report mentions slightly lower Si concentrations, which is also in agreement with our conclusions above. Regarding the interaction layer thickness, the INL report shows that layers between 1 and 3 µm thickness, with an average of 1.5µm are found, which again is very comparable to the values shown in Table 3 for the U7MC6311 plate.
Comparing the interaction layers formed in this study with the as-fabricated microstructure of the RERTR (mini-)plates, which are produced at higher rolling temperature (500°C), shows that the microstructures are highly similar. Particularly the U7MC6311 fuel plate has a microstructure that is highly comparable, as was also concluded in [17]. Keeping in mind the RERTR-9A miniplates that had received an additional HIP-step at 500°C [18], the microstructures of the RERTR-9A plates show that short exposures to temperatures of 450-500°C yields promising SiRDLs (highest Si content), which is what was attempted in the E-FUTURE fuel plates.

6. Conclusions

The E-FUTURE experiment is currently testing in BR2 four full sized plates made of U(Mo) atomized powders dispersed in Al with Si matrix (4 and 6 wt% Si). Two of them are made with AlFeNi cladding, the two others are made with AG3NE.
During the fabrication of the plates, three different heat treatments have been applied in the aim of developing different kinds of microstructure and Si-rich diffusion layer on the U(Mo) particles before the irradiation.
The main conclusions of the Prior Irradiation Examinations made by both CEA and SCK•CEN on the fresh fuel plates can be summarized as follows:

- Uniform distribution of U(Mo) particles in the meat for all plates without large porosities,
- Alignments of Mg2Si precipitates are clearly visible only on the meat-AG3NE cladding interface,
- Partial γ to α–U(Mo) transformation of the fissile particles is evidenced by XRD for all the samples, but it is all the more pronounced as the temperature of the heat treatment is high,
- A Si-rich pre-formed layer is observed on almost all U(Mo) particles (in all the plates) but this layer is not uniform in thickness and always discontinuous, with localised larger interactions (certainly to be linked to local γ-U(Mo) decomposition),
- The thickness of this Si-rich layer and the covered fraction of the U(Mo) particles surface appear higher with the temperature, inversely the Si concentration of the pre-formed layer decreases as its thickness increases,
- Qualitatively, Si particles appear larger and less numerous as the temperature of the heat treatment increases.

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