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**IRIS-TUM: MICROSTRUCTURE OF THE NON-IRRADIATED PLATES**

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**ABSTRACT**

During the IRIS-TUM irradiation experiment five large scale UMo dispersed plates have been irradiated to high burn ups. Ground powder has been used, the U235 enrichment was ~50%. The test included three different kind of plates: without Si addition (<0.1wt%) with ~7gU/cc and ~8gU/cc and with 2.1wt%Si addition with ~8gU/cc, respectively. Post irradiation examination data have already been presented.

Now, in addition to this previous work, powder used for preparing these plates has been characterized by XRD. Samples taken from the middle and the edge of the fuel zone of each kind of the three non-irradiated spare plates have been examined using SEM/EDX and XRD. The samples were provided by AREVA-CERCA.

In case of no Si addition to the matrix (7gU/cc and 8gU/cc) the usual microstructure of ground powder dispersed in Al has been observed: The UMo particles with irregular shapes of different sizes up to ~150µm contain oxide stringers and an oxide layer. The Al matrix contains considerable porosity.

In case of 2.1wt% Si inside the matrix, in addition to the usual microstructure, a thinSi rich diffusion layer (SiRDL – 1-2µm thicknes) around some UMo particles has been observed. The growth of the SiRDL depends on the presence of nearby Si particles (size: 2-10µm). A more uniform growth of the SiRDL could be expected for a more homogenous Si distribution.

XRD results show that the original powder consists mainly of two phases:  $\gamma$ -UMo and UO<sub>2</sub>, with a poor crystallization state of the  $\gamma$ -phase. After the fabrication of the plates, there is an obvious  $\gamma$ -phase destabilization. Nevertheless, SEM pictures show that this destabilization did not lead to a significant intergranular propagation of the interaction process during fabrication.

## 1 Introduction

In the framework of the IRIS-TUM irradiation campaign - a cooperation between the Technische Universität München (FRM II), the Commissariat à l'Energie Atomique (CEA) and AREVA-CERCA - five full size test fuel plates containing ground UMo powder in an Aluminum matrix have been irradiated under severe conditions, i.e. a maximum heat flux of 260W/cm<sup>2</sup> and a maximum burn up between 56.3% and 88.3% (LEU equivalent). The irradiation took place in the OSIRIS test reactor of CEA at Saclay. Although the plates suffered from a large swelling at the end of the irradiation campaign, none of them showed a cladding failure and no fission products were released [1]. The results of the post irradiation examinations on these plates have been presented recently [2,3,4,5].

For this paper samples taken from the middle and the edge of the fuel zones of the non-irradiated spare fuel plates have been examined using SEM/EDX and laboratory scale XRD. The results will be compared to the in-pile irradiated state and to results obtained on non-irradiated samples containing atomized powder and ground powder of different elementary compositions [6,7]. Furthermore the evolution of the UMo phases during hot rolling will be discussed.

## 2 Sample preparation

In order to improve the irradiation performance of UMo/Al dispersion fuels which suffered from excessive swelling due to the build-up of a large interaction layer at the UMo/Al interface during former irradiation test [8,9] four different actions that have shown to be efficient in limiting the plate swelling have been taken up in the IRIS-TUM irradiation campaign:

1. Addition of 2wt%Si to the Al matrix [10]
2. Oxidation of the UMo powder [10]
3. Increase of the Mo content to 8wt% to further stabilize the  $\gamma$ -UMo phase
4. Use of ground UMo powder instead of atomized powder [11,12]

Point 1-3 aim at limiting the build up of the undesired UMo/Al interdiffusion layer (IDL) which is regarded as the cause for the excessive plate swelling while point 4 - compared to atomized powder - induces more porosity into the meat which takes up the plate swelling at the beginning of the irradiation. Furthermore, the fission gas behavior seems to be improved when using ground powder [5]. Since in the case of dispersed fuel it is only possible to convert the FRM II from the use of highly enriched Uranium (HEU) to an enrichment of  $\approx 50\%$  (MEU) [13] it has been decided to use an enrichment of 49.3% for the IRIS-TUM plates. The plates were produced by AREVA-CERCA, the main features are given in Table 1. The plates can be categorized in the following way where X, Y and Z are running numbers:

- 800X: 8.5gU/cm<sup>3</sup>, no Si addition to the matrix
- 850Y: 8.3gU/cm<sup>3</sup>, 2.1wt% Si addition to the matrix
- 700Z: 7.3gU/cm<sup>3</sup>, no Si addition to the matrix

Plate number	8001	8002	8501	8503	7002	7003
Uranium density [gU/cm <sup>3</sup> ]	8.5	8.4	8.3	8.3	7.3	7.3
Meat Porosity [vol%]	8.1	7.9	9.0	8.9	6.5	6.4
Si in Al content [wt%]	0.07	0.07	2.1	2.1	0.07	0.07
Vol% of Al in the meat	38.2	38.0	38.7	38.6	45.0	45.2
Meat thickness [mm]	0.49	0.49	0.49	0.49	0.54	0.54
Mo in UMo [wt%]	8.1	8.1	8.1	8.1	8.2	8.2

Table 1: Parameters of the six irradiated IRIS-TUM plates as shown in [1].

In addition to the six plates depicted in Table 1 three identical twin plates – one of each kind – named 8003, 8502 and 7001 have been produced and remained non-irradiated. From each of these plates two samples have been taken from the middle of the fuel zone and from a zone close to the picture frame, respectively – see Fig. 1. Cross sections have been prepared from each sample and have been examined using SEM/EDX and laboratory scale XRD. Furthermore, samples from the original ground UMo powder provided by AREVA-CERCA have been examined using SEM/EDX, XRD and laser granulometry.

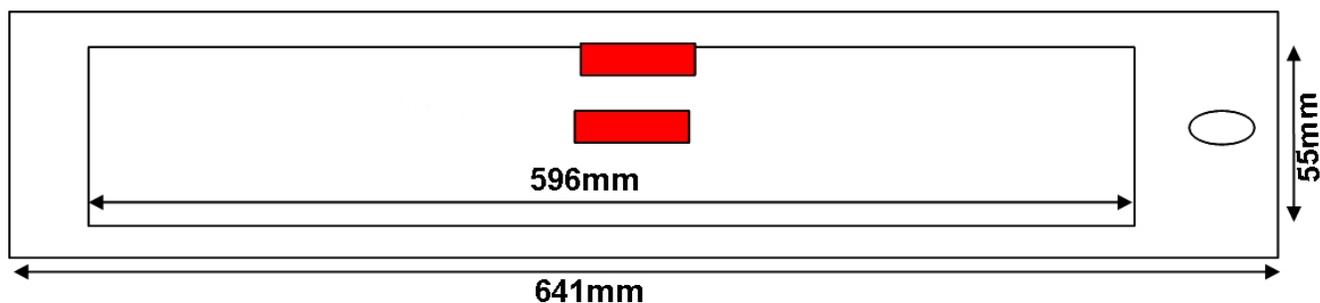


Fig. 1: Sampling positions at the fresh fuel plates. The size of the samples (red) was 28x10mm<sup>2</sup>.

### 3 Results and discussion

#### 3.a UMo ground powder – State before plate fabrication

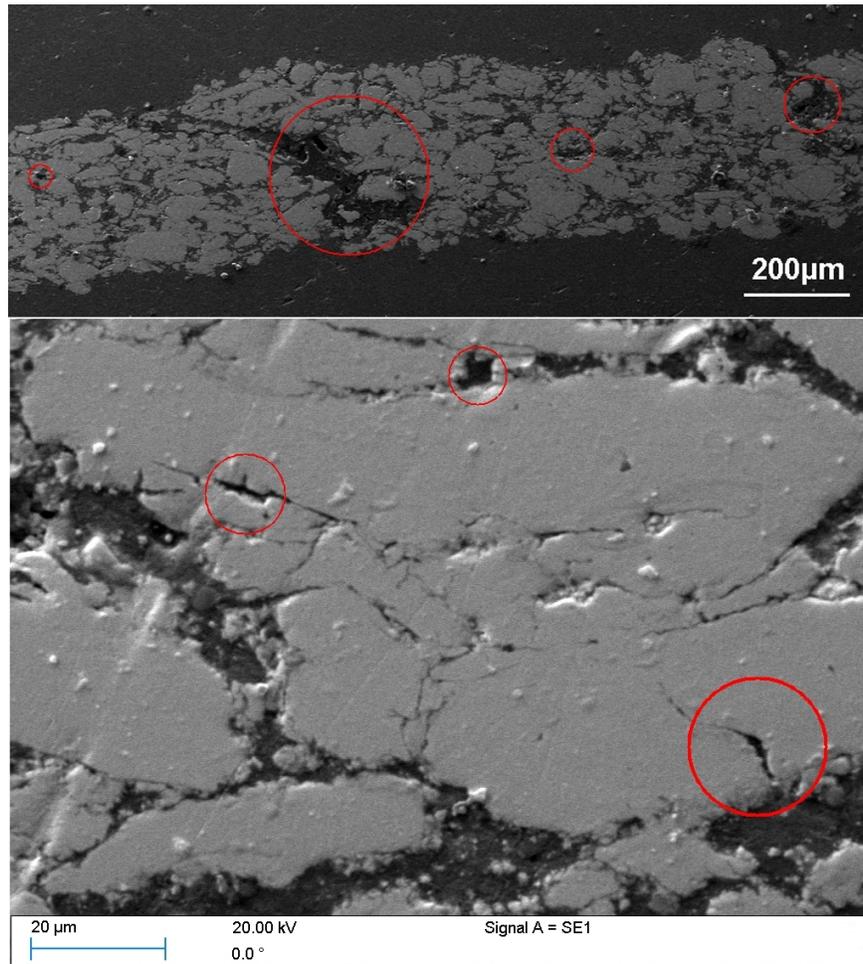
Samples of the ground UMo powder used for the IRIS-TUM plate production have been examined. They were sieved by AREVA-CERCA in order to obtain two classes: fine particles (0-40µm diameter) and coarse ones (40-125µm diameter). The two sieved powders were characterized by :

- laser granulometry to determine the size distribution of the particles,
- laboratory scale XRD to determine the phase composition.

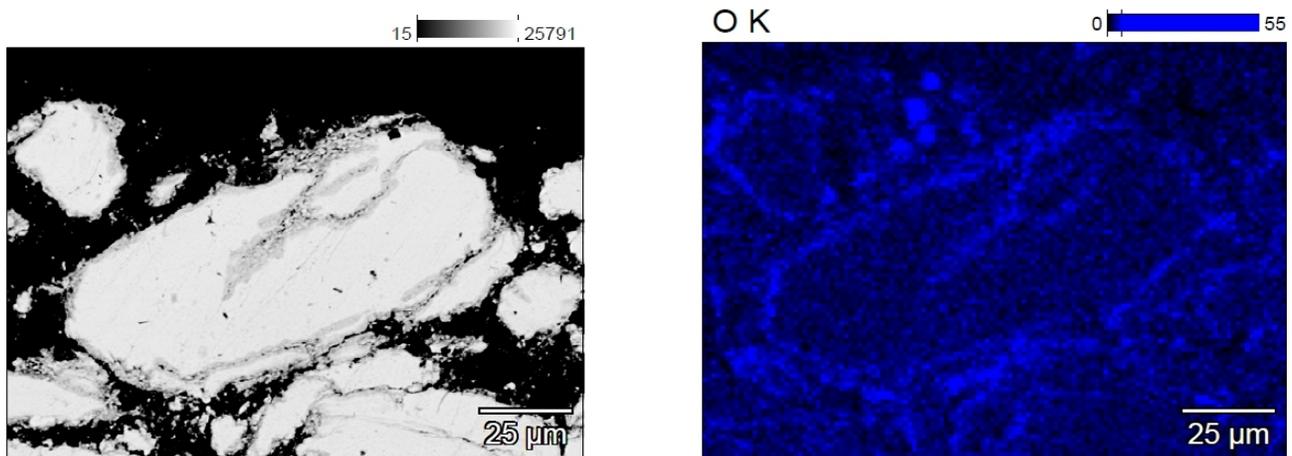
Granulometry measurements showed that a significant amount of very fine particles characterized by a size of a few µm to 10 µm were present in the [0-40 µm] class, while, in the [40-125 µm] class, some particles larger than 125 µm were also present. In both cases, XRD analysis evidenced two phases: γ-UMo and UO<sub>2</sub>. It can be concluded that the grinding process must have accumulated many defects inside the UMo because the measurement revealed broader reflection peaks and a higher background noise compared to a XRD analysis of atomized UMo powder. The γ-UMo was therefore initially in a bad state of crystallization.

#### 3.b IRIS-TUM plate samples – SEM/EDX analysis

Samples taken from the non-irradiated IRIS-TUM plates have been examined using SEM/EDX. The typical microstructure of UMo/Al plates prepared with ground UMo powder has been found within all three samples: UMo particles of different size up to ~150µm and irregular shape are dispersed inside the Al matrix. The particles contain cracks and sometimes look even “folded” due to the high mechanical forces applied during grinding. Furthermore, the particles often contain oxide stringers and are in general oxidized at the interface UMo-Al. Also a considerable amount of porosity is visible inside the matrix (compare Fig. 2 and Fig. 3). The surface percentage of porosity, UMo and Al have been determined for each of the three samples by analyzing SE-SEM pictures, results are given in Table 2. It is noteworthy that the amount of UMo and Al determined by image analysis is consistent with the values given in Table 1 according to [1], although the values are not the same (surface % vs. vol. %). As expected, the UMo fraction is higher in the plates 8003 and 8502 compared to plate 7001. However, the percentage of porosity could not be determined precisely by image analysis.



*Fig. 2: Low (upper part) and high (lower part) resolution picture of the typical as prepared matrix state of the IRIS-TUM samples. The irregular size and shape of the ground UMo particles, the presence of porosity (red circles) and the high UMo loading are noteworthy.*



*Fig. 3: BSE image (left) and EDX map on the Oxygen K-alpha line (right) of the same area. The UMo particles are oxidized and contain oxide stringers due to the grinding process.*

No signs of granular  $\gamma$ -phase destabilization could be observed inside all three as-fabricated IRIS-TUM plates. An UMo-matrix interaction occurred only on plate 8502 – the one with 2.1wt% Si inside the matrix.

Here, in addition to the features described above Si precipitates (size 1-5 $\mu$ m) are distributed in the matrix (compare Fig. 4). Moreover, a Silicon rich layer (SiRDL) formed at the interface UMo/Al (compare Fig. Fig. 5). It must be pointed out, that the UMo grains are not completely covered with the SiRDL: in fact - and in contrast to what was observed on atomized powder with the same or higher Si content inside the matrix [14, 6] - only a minor part of the UMo particles are covered by the SiRDL.

It is worth noting that fine UMo particles (typical size: a few  $\mu$ m) and sharp edges of bigger particles reacted especially often and readily with the Si inside the matrix – an example can be seen in Fig. 5.

The Si/(U+Si) content of the SiRDL inside the IRIS-TUM sample has been determined to be 42at%. This value is slightly lower than values obtained previously on samples with 5wt% (Si/(U+Si)=50at%) and 7wt%Si (Si/(U+Si)=54at%) addition to the matrix (compare Table 3). Since the production parameters of the IRIS-TUM plates and the samples examined before are comparable, it can be expected that the SiRDL will also consist of stoichiometric  $USi_2$  [6,7].

Plate	UMo [surf.%]	Al [surf.%]	Porosity [surf.%]
8003	68 $\pm$ 4	26 $\pm$ 5	5 $\pm$ 5
7001	54 $\pm$ 12	45 $\pm$ 12	2 $\pm$ 1
8502	69 $\pm$ 6	28 $\pm$ 5	2 $\pm$ 1

Table 2: UMo, Aluminum and porosity surface % as determined using image analysis on SEM secondary electron pictures.

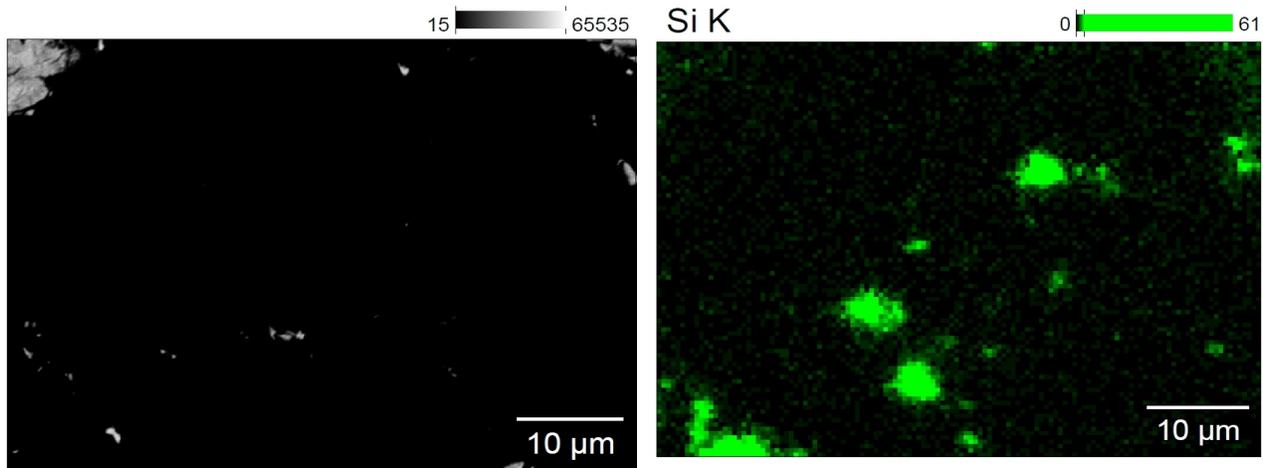


Fig. 4: IRIS-TUM 8503 (2.1wt%Si inside the matrix): BSE image (left) and EDX map on the Si K- $\alpha$  line (right) of the same area. The size of the Si precipitates inside the matrix varies between 1-5 $\mu$ m.

	IRIS-TUM, 8502	5wt%Si [6]	7wt%Si [6]
Si/(U+Si) (at%)	42	50	54

Table 3: Si content of the SiRDL found on IRIS-TUM samples containing 2.1wt%Si inside the matrix compared to previous work on similar samples[6].

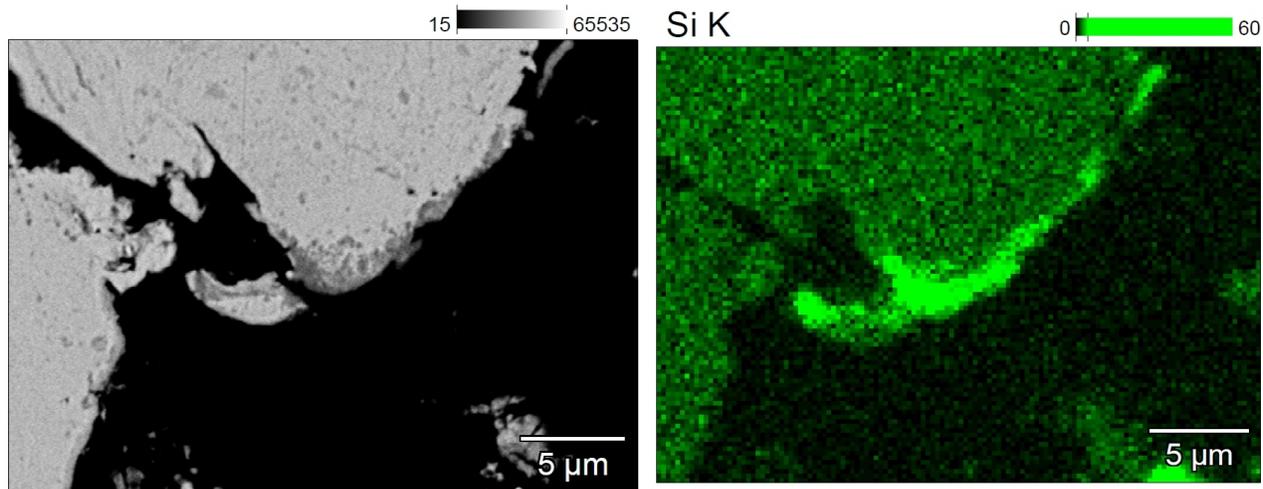


Fig. 5: IRIS-TUM 8503 (2,1wt%Si inside the matrix): BSE image (left) and EDX map on the Si K - $\alpha$  line (right) of the same area. At some spots a thin SiRDL formed at the interface UMo-Al.

### 3.c IRIS-TUM plate samples – XRD analysis

Samples from plates 8003 - pure Al matrix - and 8502 - 2,1wt%Si inside the Al matrix - have been examined using laboratory scale XRD and Rietveld analysis. Results are given in Table 4 together with values obtained on comparable samples during other studies. The values obtained for the composition of the two examined IRIS-TUM plates are identical within the limitations of the method. No signs of Si or Si-containing phases have been found inside the plate with Si addition to the matrix due to the small overall Si content. Despite the fact that no granular  $\gamma$ -phase destabilization of the UMo has been observed by SEM, a high amount of  $\alpha$ -U has been found inside the samples ( $\alpha / (\alpha+\gamma)$  =44 and 43%, respectively for the two samples examined). These values are higher than values obtained on samples containing atomized powder (U8Mo atom:  $\alpha / (\alpha+\gamma)$ =17% [18]), but similar to values obtained on other samples containing ground powder (U8Mo  $\alpha / (\alpha+\gamma)$ =32% [18] and U8Mo1Ti  $\alpha / (\alpha+\gamma)$ =35% [7]. It is noteworthy that this  $\gamma$ -phase destabilization occurring on samples containing ground UMo can be diminished by adding some wt% Nb or Pt to the UMo [7, 15, 16, 17].

Plate	Al (wt%)	$\alpha$ -U (wt%)	$\gamma$ -U-Mo (wt%)	UO <sub>2</sub> (wt%)	$\alpha / (\alpha+\gamma)$ (%)
IRIS-TUM 8003	51	19	24	6	44
IRIS-TUM 8502	45	20	27	9	43
U8Mo [18]	10	20	43	27	32
U8Mo atom. [18]	22	13	62	3	17
U8Mo1Ti [7]	60	11	20	9	35
U8Mo1,5Nb [7]	50	12	29	9	29
U8Mo3Nb [7]	62	9	25	4	26
U8Mo1Pt [7]	65	6	20	9	23

Table 4: Phase composition of different ground UMoAl samples as determined by laboratory scale XRD and Rietveld analysis.

The lattice parameters of the  $\alpha$ -U as determined by Rietveld analysis of the measured XRD diagrams are given in Table 5. The well-known orthorhombic structure of  $\alpha$ -U (symbol Cmcm) has been used during the Rietveld analysis. However, compared to the lattice constants given in literature [19], the crystal lattice of the  $\alpha$ -U inside ground UMo/Al samples is in general slightly deformed: the b-parameter is contracted while the a- and c-parameters are stretched.

Plate	a (Å)	b (Å)	c (Å)	$\gamma$
IRIS-TUM 8003	2.8603	5.8543	4.9651	90°
IRIS-TUM 8502	2.8637	5.8543	4.9651	90°
U8Mo1Ti [7]	2.8640	5.8506	4.9642	90°
U8Mo1,5Nb [7]	2.8642	5.8542	4.9706	90°
U8Mo3Nb [7]	2.8599	5.8542	4.9732	90°
U8Mo1Pt [7]	2.8615	5.8478	4.9601	90°

Table 5:  $\alpha$ -U cell parameters of different ground UMo powders as determined by laboratory scale XRD and Rietveld analysis. Orthorhombic structure, symbol Cmcm. For comparison: the lattice parameters of pure  $\alpha$ -U are  $a=2,8536\text{Å}$ ,  $b=5,8698\text{Å}$ ,  $c=4,9555\text{Å}$ ,  $\gamma=90^\circ$ , symbol Cmcm, e.g. [19].

The Mo content of the remaining  $\gamma$ -UMo phase has been calculated using the following expression [20]:

$$a(\text{Å})=3,4878-0,0034[\text{Mo}](\text{at}\%)$$

Calculated values are given in Table 6. It is noteworthy, that the calculated Mo content for the two IRIS-TUM samples of 11,8 and 11,1wt%, respectively is higher than the value expected from the production parameters given in [1] (8wt%Mo). This behavior is consistent with the values calculated from the  $\gamma$ -UMo lattice parameter of other samples prepared with ground powder.

However, a certain uncertainty on the calculated Mo content has to be considered. Comparing the expression used for this calculation with that determined by A.E. Dwight

$$a(\text{Å})=3,4808-0,00314[\text{Mo}](\text{at}\%) \quad [21]$$

or another by S.T. Konobeevsky

$$a(\text{Å})=3,481-0,00333[\text{Mo}](\text{at}\%) \quad [22]$$

the error can be estimated to be about 0,5 wt%. Even with such an uncertainty, the Mo content of the  $\gamma$ -phase remains still significantly higher than the mean alloy composition (8 wt%).

Plate	a (Å)	Mo (at%)	Mo (wt%)
A5 matrix	3.4027	25.0	11.8
Al+2,1Si	3.4077	23.6	11.1
U8Mo [18]	3.4	27.0	13.0
U8Mo1Ti [7]	3.4007	25.6	12.2
U8Mo1,5Nb [7]	3.4007	25.6	12.2
U8Mo3Nb [7]	3.3964	26.9	12.9
U8Mo1Pt [7]	3.4017	25.3	12.0

Table 6:  $\gamma$ -UMo lattice parameter as determined by laboratory scale XRD and Rietveld analysis and corresponding calculated Mo content.

### 3.d XRD analysis – Discussion of the results

In this section the phase behavior of ground UMo will be discussed. First, the main results are summed up:

- XRD analysis of the ground UMo powder prior to plate production revealed only the presence of  $\gamma$ -UMo and UO<sub>2</sub>. However, the  $\gamma$ -UMo lattice structure has accumulated many defects during the grinding process and was in a bad crystalline state.
- XRD analysis of two as-fabricated plates (with and without Si addition) revealed the presence of  $\alpha$ -U besides  $\gamma$ -UMo in the samples. The ratio  $\alpha / (\alpha + \gamma)$  was about 43- 44% for each of them.
- Regarding the lattice constants of the  $\alpha$ -U inside the samples it is noteworthy that they differ somewhat from the lattice constants of  $\alpha$ -U given in literature: the b-parameter is contracted while the a- and c-parameters are stretched.
- By calculating the Mo content of the remaining  $\gamma$ -UMo from its lattice constant a higher Mo content than expected has been found: 11-12wt%Mo instead of 8wt% as expected.

On the first view, the phase transformation occurring in ground UMo powder upon plate production of the initial  $\gamma$ -phase into  $\alpha$ -U and  $\gamma$ -UMo enriched in Mo resembles strongly to the decomposition of  $\gamma$ -stabilized UMo according to the TTT diagram that occurs upon annealing for several hours at temperatures between 375°C and 550°C:  $\gamma$ -UMo decomposes first into  $\alpha$ -U and  $\gamma$ -UMo enriched in Mo. Later, as the  $\alpha$ -U content increases U<sub>2</sub>Mo appears beside  $\gamma$ -UMo. Thermal equilibrium is reached when all  $\gamma$ -UMo has been transferred into a mixture of  $\alpha$ -U + U<sub>2</sub>Mo [23, 26]. However, a detailed view on the data obtained on the IRIS-TUM samples excludes this simple interpretation:

- It is unlikely that the temperature of the fuel plates reached temperatures between 375°C and 550°C for more than 1-2h. Indeed this would be necessary to induce the  $\gamma$ -UMo decomposition according to the TTT diagram. .
- Already the beginning of  $\gamma$ -UMo decomposition according to the TTT diagram ( $\gamma$ -UMo(1)  $\rightarrow$   $\gamma$ -UMo(2) +  $\alpha$ -U where UMo(2) has a higher Mo content than UMo(1)) leads to the formation of a cellular decomposition product [24], which has not been observed on the IRIS-TUM samples.
- The solubility of Mo in  $\alpha$ -U is <1at% [25, 26]. Under this condition and the fact that over 40% of the UMo decomposed into  $\alpha$ -U (Table 4), the rise from the expected  $\sim$ 18at% to the measured  $\sim$ 25at% Mo inside the  $\gamma$ -UMo is too small to assume that the  $\alpha$ -U can be free of Mo. However, two  $\alpha$ -U like phases are known with Mo contents of 2,95-6,20at%Mo ( $\alpha'$ , orthorhombic structure) and 7,20-11,18at%Mo ( $\alpha''$ , monoclinic structure) [27, 28].
- The measured cell parameters do not fit to values reported in pure  $\alpha$ -U in literature: the b-

parameter is contracted while the a- and c-parameters are stretched. However, this is characteristic of the  $\alpha'$ -UMo phase reported in literature [29]. This phase is basically orthorhombic  $\alpha$ -U with a lattice distortion due to the presence of Mo atoms on lattice sites [30].

Good results during the Rietveld analysis have been obtained using the orthorhombic structure of  $\alpha$ -U (Space group Cmc<sub>2</sub>m). No line splitting compatible with a monoclinic structure has been observed. Therefore it seems reasonable to conclude that the  $\alpha'$ -phase is present (instead of the  $\alpha''$ -phase).

Thus, the UMo particles inside the as-fabricated IRIS-TUM plates seem to correspond to a mixture of ~40%  $\alpha'$ -UMo with a Mo content of ~6at% and of ~60%  $\gamma$ -UMo further enriched in Mo. Complementary examinations using TEM could help to check these assumptions. It must be pointed out that this mixture of phases is still far from the thermal equilibrium state which would be  $\alpha$ -U and U<sub>2</sub>Mo [26].

By comparing the lattice parameters of the  $\alpha$ -U phase obtained on other samples containing ground UMo powder and taking into account the fact that the  $\gamma$ -UMo in these plates is also enriched in Mo (compare Table 5 and Table 6) one can conclude that this behaviour is a general feature of this type of fuel plates.

The phase transformation from  $\gamma$ -UMo to  $\alpha'$ -UMo+ $\gamma$ -UMo during the plate production occurred most likely due to the annealing of the lattice defects during the hot-rolling step that were induced by the previous grinding before.

#### 4 Conclusions

Samples taken from three non-irradiated IRIS-TUM plates have been characterized using SEM/EDX and laboratory scale XRD. In addition to the features well known from fuel plates prepared with ground powder – a high amount of porosity plus oxidized and deformed UMo particles with oxide stringers and of irregular size and shape – a SiRDL has been found at the interface UMo-Al inside the fuel plate with 2.1wt%Si addition to the Al matrix. However, the SiRDL is visible only at a minor part of the UMo particles and the Si content is lower than what has been found during previous examinations [6].

By comparing the XRD results of the ground UMo powder prior to plate production to the results obtained on samples taken from the fuel plates one finds that  $\gamma$ -UMo of a poor crystalline state seems to have decomposed into  $\alpha'$ -UMo+ $\gamma$ -UMo enriched in Mo. This behaviour is most likely due to the annealing of lattice defects during the hot-rolling step and not to the normal  $\gamma$ -UMo  $\rightarrow$   $\alpha$ -U+ U<sub>2</sub>Mo decomposition according to the TTT diagram after annealing for several hundred hours. In order to check this evolution scheme, TEM+EDX characterizations should be performed.

It is worth noting that no signs of  $\alpha$ -U or  $\alpha'$ -U like phases have been found during the post irradiation examinations of the IRIS-TUM plates [3]. The phase transformation  $\gamma$ -UMo(1)  $\rightarrow$   $\alpha'$ -UMo+ $\gamma$ -UMo(2) has therefore been reversed. This effect has been observed before after in-pile and heavy ion irradiation [18, 7, 31, 32]

#### 5 Acknowledgments

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