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IRRADIATION BEHAVIOR OF ATOMIZED AND GROUND U(MO) DISPERSION FUEL.

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ABSTRACT

In the framework of the IRIS-3, FUTURE and IRIS-TUM irradiation programs, several full size plates containing atomized (IRIS-3 and FUTURE) or ground (IRIS-TUM) U(Mo) dispersion fuel in a aluminum matrix, with and without addition of silicon, have been irradiated in the OSIRIS reactor [1] (IRIS-3, IRIS-TUM) and in the BR2 reactor [2] (FUTURE). The microstructural analyses of the fuels from these irradiation projects were performed at the hot laboratory (LHMA) of SCK•CEN in Mol, Belgium.
Some results of the PIE have been reported previously [3, 4, 5]. Recent electron probe microanalysis results on the ground U(Mo) fuel provide further insight in the effect of adding silicon to the aluminum matrix and reveal some distinct differences between ground and atomized fuel, particularly concerning the fission gas behavior.

1 Introduction

Being one of the better advanced fuel candidates for the conversion of high performance research reactors from HEU to LEU fuel, a lot of effort has been put in the development and fabrication of U(Mo) dispersion fuel. However, some past irradiation campaigns, e.g. IRIS2 and FUTURE [2] have shown that the classical atomised U(Mo) fuel dispersed in a pure Al matrix, cannot withstand high operation conditions. A large part of this instability of atomised U(Mo) fuel is attributed to the behavior of the fission gas generated and the properties of the U-Mo-Al interaction layer (IL) that forms during the irradiation. PIE have revealed the existence of fission gas nanobubbles on a superlattice in the U(Mo) fuel cells and have shown that the IL becomes amorphous during irradiation [6].
One of the attempts to improve the behavior of atomised U(Mo) dispersion fuel, is the addition of silicon to the Al matrix [7]. It is believed that this should impede the growth of the IL. From the PIE on IRIS3 [4] and RERTR6 [8], it was concluded that the addition of silicon only helps when the Si particles were close to the U(Mo) kernels prior to the irradiation or if a Si rich layer was formed around the metallic fuel particle during production.
In the IRIS3 irradiation campaign, also fuel plates containing ground U(Mo) fuel have been irradiated (IRIS-TUM) [3]. Compared to atomised fuel, ground fuel behaves differently under irradiation and it is not completely understood why.

For the IRIS-TUM experiment, several fuel plates have been irradiated [1]. The result on optical microscopy and scanning electron microscopy on some of the fuel plates, have already been briefly presented [3]. In the current paper, more detailed results including EPMA on the fuel plate containing ground U(Mo) dispersed in an Al matrix and ground U(Mo) in an Al-2.1 wt% Si matrix, will be presented and compared to the PIE results obtained on the atomised fuel.

2 Irradiation of Atomised U(Mo) Fuel

2.1 The FUTURE irradiation (no Si addition).

In the framework of qualifying U(Mo) dispersion fuel, two experimental fuel plates have been irradiated in the FUTURE irradiation rig in the BR2 reactor at SCK•CEN [2] in 2004. The meat of the fuel plates consisted of U-7 wt% Mo atomized powder with an enrichment of 19.8% ^{235}U dispersed in an Al matrix (Al5). The plates had a loading density of ≈ 8.5 g U cm^{-3}. The cladding of the fuel plates was an AG3NE Al-Mg alloy (2.81 wt% Mg). The fuel plates were submitted to a maximum heat flux of 353 W/cm^2 while the cladding surface temperature was kept below 130°C. The fuel was irradiated for two cycles leading to a maximum burn-up of approximately 33% ^{235}U (2.8 ×10^{21} fissions/cm^3 U(Mo)). After the second irradiation cycle, visual inspection and profilometry of the fuel plates revealed a noticeable increase of the plate thickness and the irradiation was stopped.

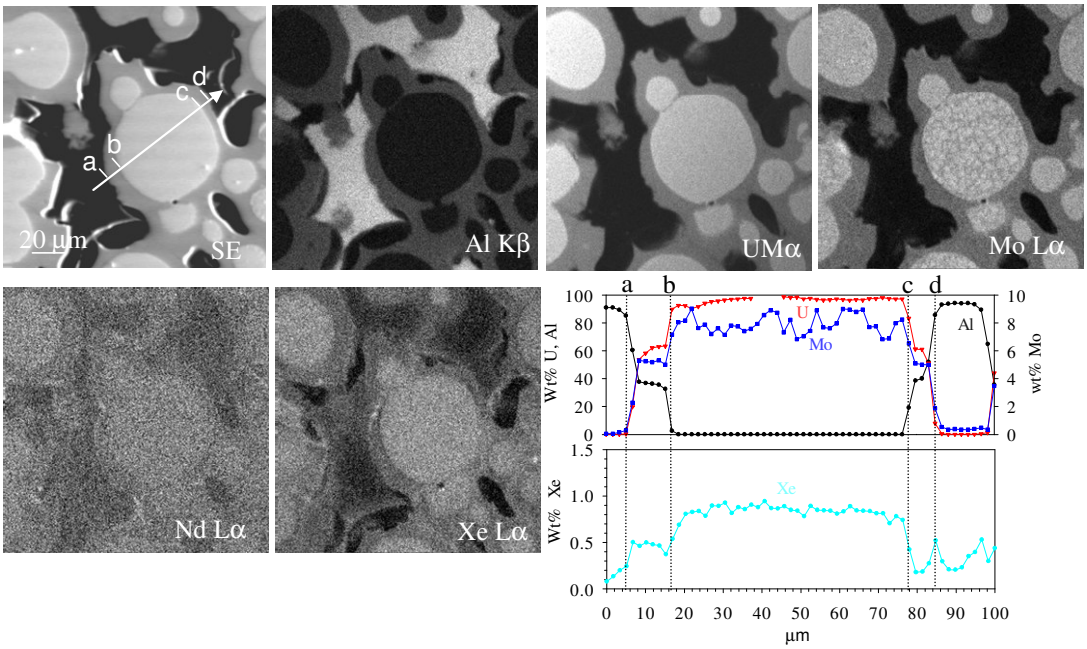


Figure 1 Quantitative linescan on an U(Mo) fuel kernel in one of the FUTURE plates.

From the PIE, it became clear that the unstable behavior of the fuel plates was related to the fission gas generated in the fuel kernels, which had accumulated at the interface between the IL and the matrix in large voids (fig.1). Later on, in a unique TEM investigation [6], it was found that the fission gases precipitate in nanobubbles which lie on an ordered lattice inside the fuel cells. With the continuous interaction between the fuel and the matrix, more and more of these nanobubbles get incorporated into the IL. The TEM investigation further showed that the IL becomes amorphous during the irradiation. In such an environment, the fission gas bubbles can move to the outer surface of the IL where they are stopped by a crystalline matrix. As such, the fission gas will accumulate at this interface. The resulting large amounts

of FP's at the interface of the IL and the matrix lead to the formation of large voids, unacceptable swelling and eventually failure of the fuel plate.

2.2 The IRIS3 irradiation (with Si addition).

As introduced higher, one of the attempts to improve the irradiation behavior of the atomised U(Mo) dispersion fuel, is to add silicon to the Al matrix. It is believed that the addition of a small quantity of silicon would reduce the formation of the interlayer. For this test, 4 plates were irradiated in the OSIRIS reactor in the IRIS-3 experiment [1]. The cladding of these plates is made of AG3NE alloy (2.81 wt% Mg). Destructive PIE results are currently still limited to one plate but fuel plate swelling data has shown that the plates with only 0.3 wt% Si in the IRIS-3 irradiation have pillowed, much like the FUTURE plates. Its meat consists of U7.3wt%Mo particles dispersed in an aluminum matrix to which 2.1 wt% Si has been added. The fissile material density is 7.8-8.0 g U_{tot}/cm^3 and the uranium enrichment is 19.8% ^{235}U. The fuel plate was kept in the reactor during 7 irradiation cycles (130.6 full power days) and submitted to a heat flux of maximum 200 W/cm^2, while the surface cladding temperature is kept below 85 °C. At its EOL, the plate had an average burn up of 48.8 % ^{235}U (3.4×10^{21} fissions/cm^3 U(Mo)) with a peak burn up at the maximum flux plane of 59.3 % ^{235}U (4.1×10^{21} fissions/cm^3 U(Mo)).

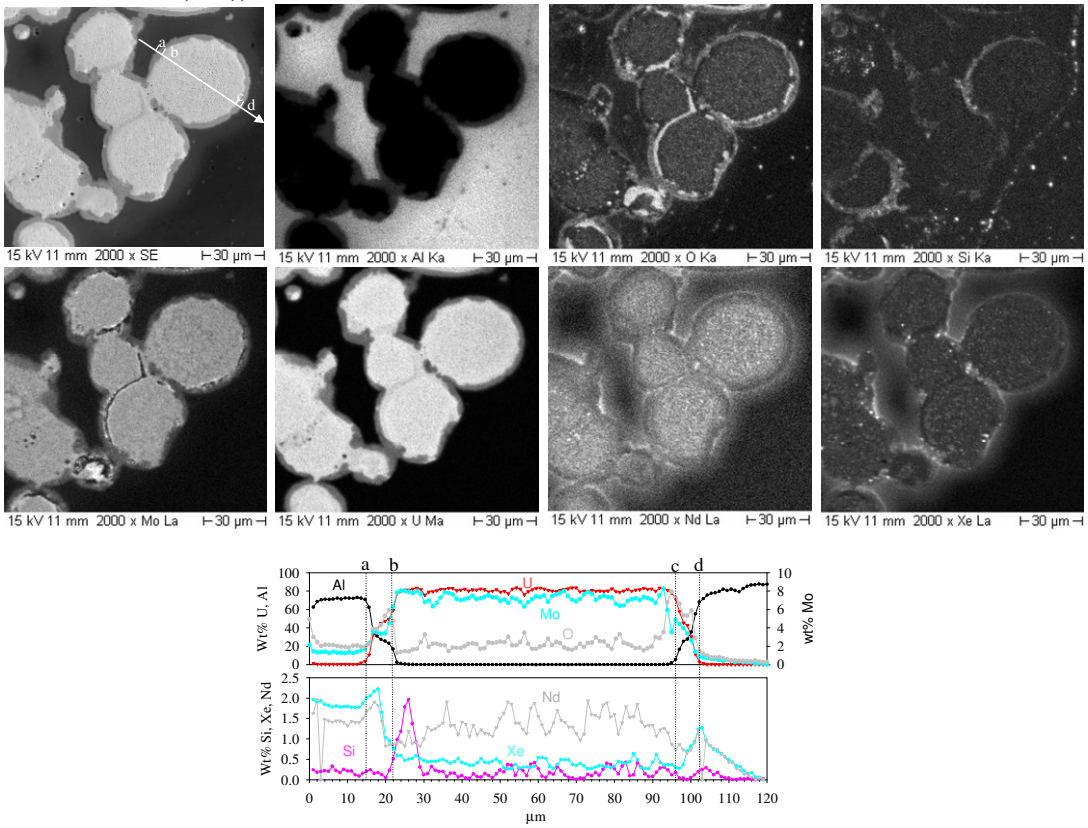


Figure 2 X-ray maps and a linescan over an U(Mo) kernel located at the meat-cladding-frame interface.

The PIE [4] showed that the addition of 2.1 wt% Si to the Al matrix seems to have a positive result on the thickness of the interaction layer, but only if there was close contact between the silicon particle and the UMo fuel at the beginning of the irradiation. The EPMA X-ray maps and quantitative linescan presented in fig.2 summarises these observations well. It should be noted that the position at which these maps are recorded is at the meat-cladding-frame interface, where a slight oxidation of the fuel, induced during the rolling process, is expected.

The BU at this position is 59.3 % ^{235}U. Other measurements at different positions show similar results.
It is observed in the X-ray maps that the asymmetry in the IL thickness is related to the presence of Si at the interface with the U(Mo) fuel kernel. At those positions where a Si particle was near to the fuel at the start of the irradiation, little IL has grown. This should be seen between point 'a' and 'b' but the effect is very local as it depends on the close proximity of a Si particle in the matrix and the U(Mo) kernel. As is visible in the U and Mo maps, there are several places where the kernel appears to 'protrude' into the IL, i.e. where no IL is formed, which is the result of such a Si particle in contact with the kernel. However, the line scan has unfortunately missed those locations. Better measurements of this effect are shown in [4]. Towards point 'c', a large increase is measured in the oxygen signal. This oxide layer is an UO_x layer (depleted in Mo) while between 'c' and 'd', an U-Mo-Al(-O) IL surrounding the fuel kernel is seen.
The increase in the Si signal after 'b' reflects the silicon rich layer, as observed in the X-ray maps. At point 'a' and 'd' the typical fission product (Xe and Nd) halo is measured. Inside the fuel particle, the Nd signal increases to a constant value of approximately 1.3 wt% , while the Xe signal decreases. This is related to precipitation of Xe into bubbles, which get opened during sample preparation. The gas is thus removed during polishing and as such a lower content is measured for Xe. It should also be noted that the Xe distribution shows some patches containing nanobubbles [4].

3 Irradiation of Ground U(Mo) Fuel

Fuel plates U8MV8002 and U8MV8503 are two out of the six full-size AlFeNi cladded fuel plates containing ground U(Mo) powder, that have been irradiated in the OSIRIS reactor in the framework of a contract between the CEA and the TUM [3].
The plates have a meat loading of ~8.4 g U_{tot}/cm^3 and a uranium enrichment of approximately 49.5 % ^{235}U. The meat consists of U8.1wt%Mo particles dispersed in either a pure (A5) aluminum matrix (plate U8MV8002) or an Al matrix to which 2.1 wt% Si (plate U8MV8503) is added. The cladding of the fuel plates is AlFeNi alloy (1 % Fe, 1 % Ni and 1 % Mg). The fuel plates were kept in the reactor during 5 irradiation cycles (90.6 full power days). At their EOL the plates have a maximum burn-up of respectively 21.7 % ^{235}U (54.3 % ^{235}U LEU equivalent, 4.14×10^{21} fissions/cm^3 U(Mo)) for plate U8MV8002 and 22.3 % ^{235}U (55.8 % ^{235}U LEU equivalent , 4.22×10^{21} fissions/cm^3 U(Mo)) for plate U8MV8503.
Similar OM and SEM observations (briefly presented in [3]) could be made on samples from fuel plates U8MV8002 and U8MV8503, even though the latter specimen contains 2.1 wt% silicon in the Al matrix.

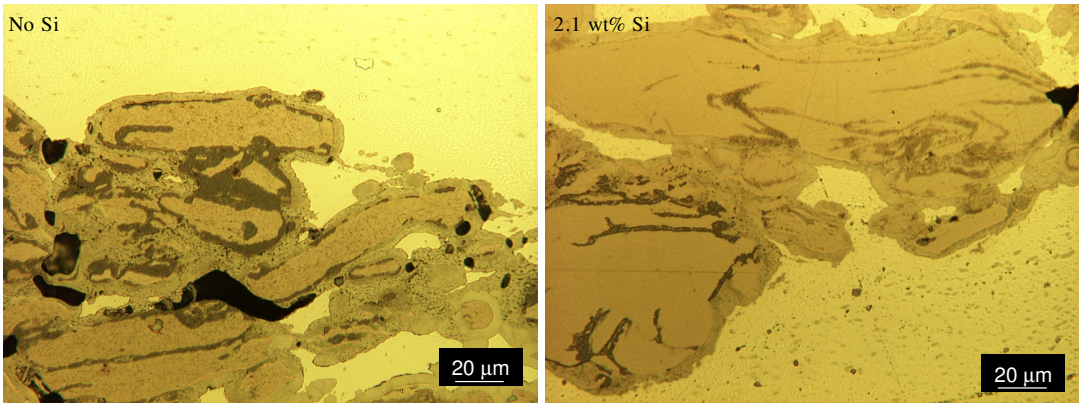


Figure 3 OM image of fuel particles at the meat-cladding interface in fuel plate U8MV8002 with no Si (left) and U8MV8503 with 2.1 wt% Si added to the matrix (right).

On the OM images, a thick oxide layer on the metallic fuel kernel surface was clearly observed as well as UO_x stringers inside the fuel particles (fig.3).

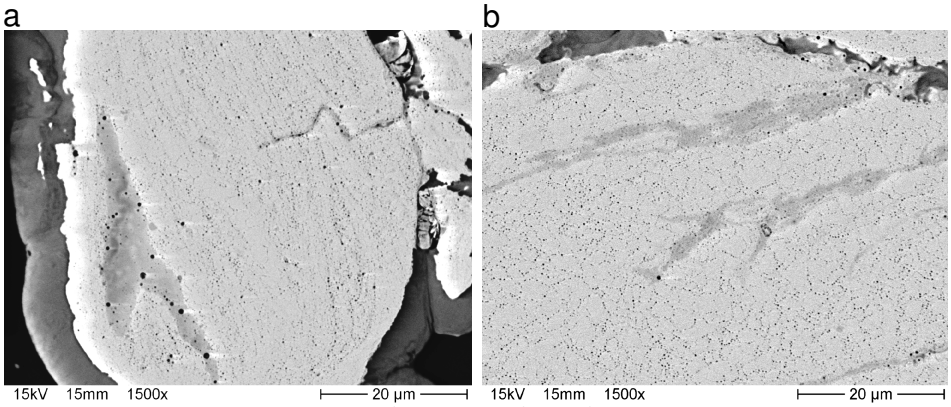


Figure 4 Backscattered electron images of irradiated ground U(Mo) fuel.

Concerning the behavior of the fission gases, in both fuel plates the fission gas bubbles moved towards defect structures such as stress lines (fig.4a) or grain boundaries (fig. 4b). Regarding the addition of Si to the matrix it was concluded that this has a small, but positive result on limiting the growth of the interaction layer.

3.1 The IRIS-TUM irradiation (no Si addition)

Figure 5 shows the secondary electron (SE) image and X-ray maps recorded at the transition of the meat with the cladding at a position where the burn-up equals 22.5 % ^{235}U or 55.3 % ^{235}U LEU equivalent.

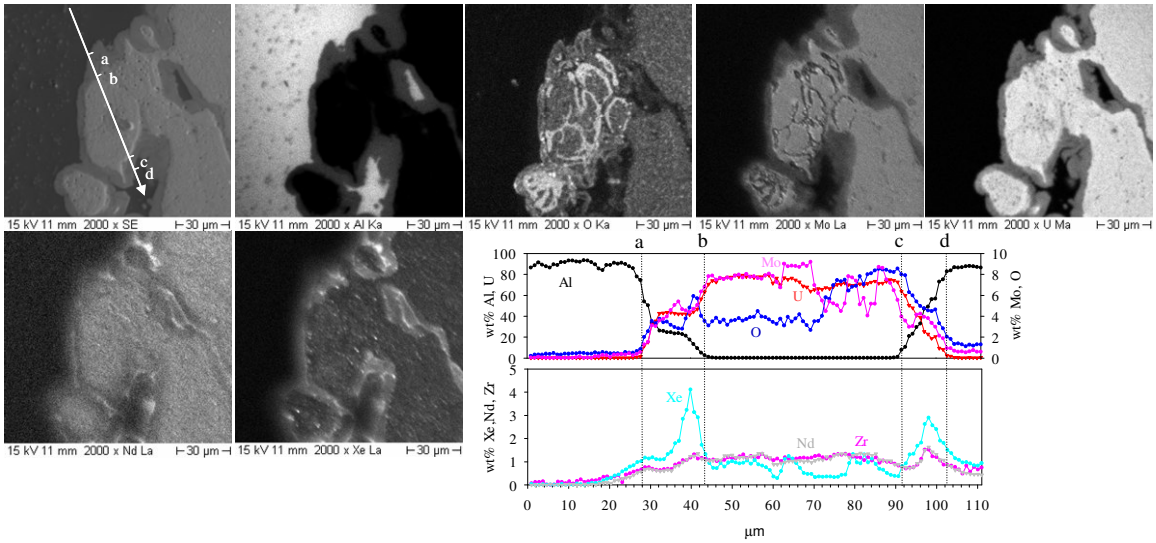
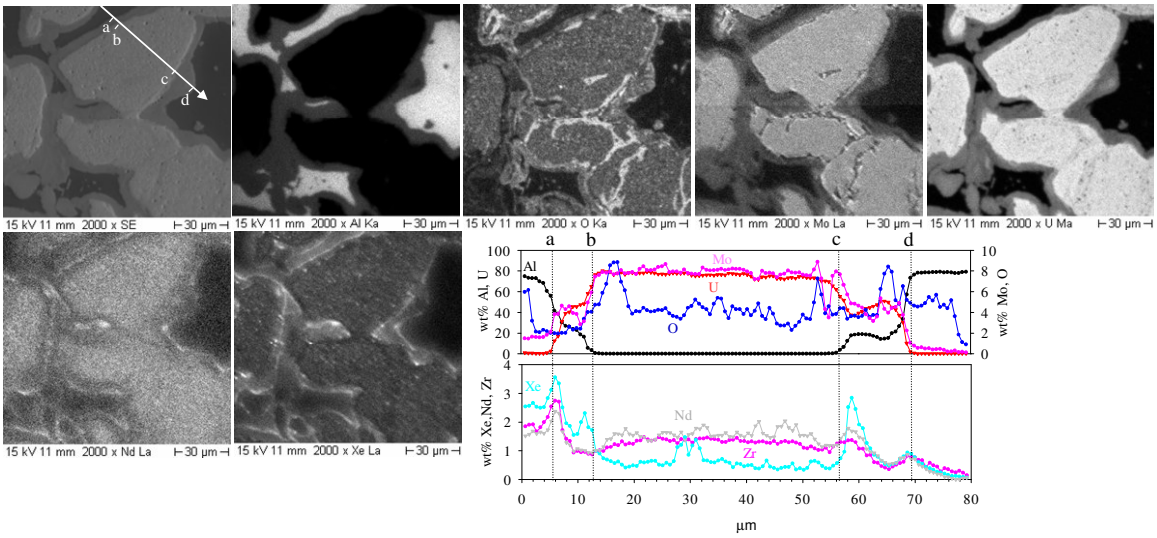


Figure 5 Linescan over a ground U(Mo) kernel in an Al matrix, located at the meat-cladding interface of fuel plate U8MV8002.

The oxygen map shows that the fuel kernel under investigation contains stringers with higher amounts of oxygen which are typical for the meat-clad interface. However, UO_x stringers can also be found inside most ground particles throughout the meat. From the U and Mo maps, it

can be seen that these stringers contain U but are depleted in Mo. Although, at higher magnification, it can be observed that Mo is present as particles in these oxygen stringers.
The Al, U and Mo maps show that an Al-U-Mo interaction layer has grown around each fuel kernel. The Xe and Nd maps illustrate that xenon has precipitated into bubbles inside the fuel kernel, while at the interface of the interaction layer and the cladding or matrix, the typical fission product halo has formed. The IL itself also contains an amount of fission products.
The quantitative linescan (indicated on the SE map of figure 5), shows between points 'a' and 'b' an IL with a composition of $(U,Mo)Al_3$ (corrected for BU). It should be noted that this is not a defined compound. The stoichiometry of the IL is quoted to allow comparison to other papers.
In-between 'b' and 'c', the interior of the fuel kernel is measured. In the first part, the U and Mo signals are rather constant. As the linescan proceeds more towards point 'c', a slight increase followed by a steep drop in the Mo signal is measured and this is mirrored by an increase in the oxygen signal. Between 'c' and 'd', again an $(U,Mo)Al_3$ IL is measured. Please note that after point 'd', in the pure Al matrix, still an amount of Mo is measured (~0.7 wt%), resulting from the injection of Mo as fission product into the matrix, similar to the Nd and Zr evolution.
The measurements of the fission products show in the IL, located in-between 'a' and 'b', two fission product concentration peaks (one at 30 µm and one at 40 µm). Considering the width of the IL at this position, the two peaks are probably two fission product halos indicating that there is or was a second fuel kernel in the neighborhood. The xenon signal in the IL is however higher than that the Nd or Zr signal.

Fig.6 gives the secondary electron (SE) image and X-ray maps over a fuel kernel close to the former analysed position in the fuel plate but located in the middle of the meat. Also here, the



fuel kernels contain UO_x stringers which are depleted in Mo.
Figure 6 Linescan over a ground U(Mo) kernel in an Al matrix, located in the meat of fuel plate U8MV8002.

Xe is precipitated in bubbles and an enrichment of Xe at the fuel/IL boundary and at the IL/matrix interface is seen. The linescan defined over the fuel particle (see SE map), shows between 'a' 'b' and 'c' 'd' an IL having a composition around $(U,Mo)Al_3$ (corrected for BU). It is again noticed that in the Al matrix, prior to point 'a', the Mo signal is around 1.5 wt%.
The UO_x layer as observed in the X-ray maps is seen in the linescan as a peak in the O signal close to points 'b' and 'c'. There is no noticeably effect on the other signals which is probably related to the small sizes of the UO_x patches.

The strong delineation of Xe observed in the X-ray maps is also reflected in the linescan : an increased intensity for Xe, Nd and Zr is found in the IL at point 'a' (near the interface of the IL with the matrix), while a second (solely) Xe peak is located in the IL at 'b' (near the interface of the IL with the fuel kernel). A nearly similar observation is made in the IL at 'c' and 'd', but here an increased intensity of Xe, Nd and Zr is seen in both locations. Considering the larger width of the IL between 'c' and 'd', this indicates that it concerns a two halos, meaning that there is or was a second fuel kernel in the neighborhood.
It should however again be noted that in the IL the concentration of Xe is higher than that of Nd (or Zr).

3.2 The IRIS-TUM irradiation (with Si addition)

Fig. 7 shows the secondary electron (SE) image and X-ray maps recorded in the middle of the meat at a position in fuel plate U8MV8503 where the BU equals 23.1 % ^{235}U or 57.2 % ^{235}U LEU equivalent.

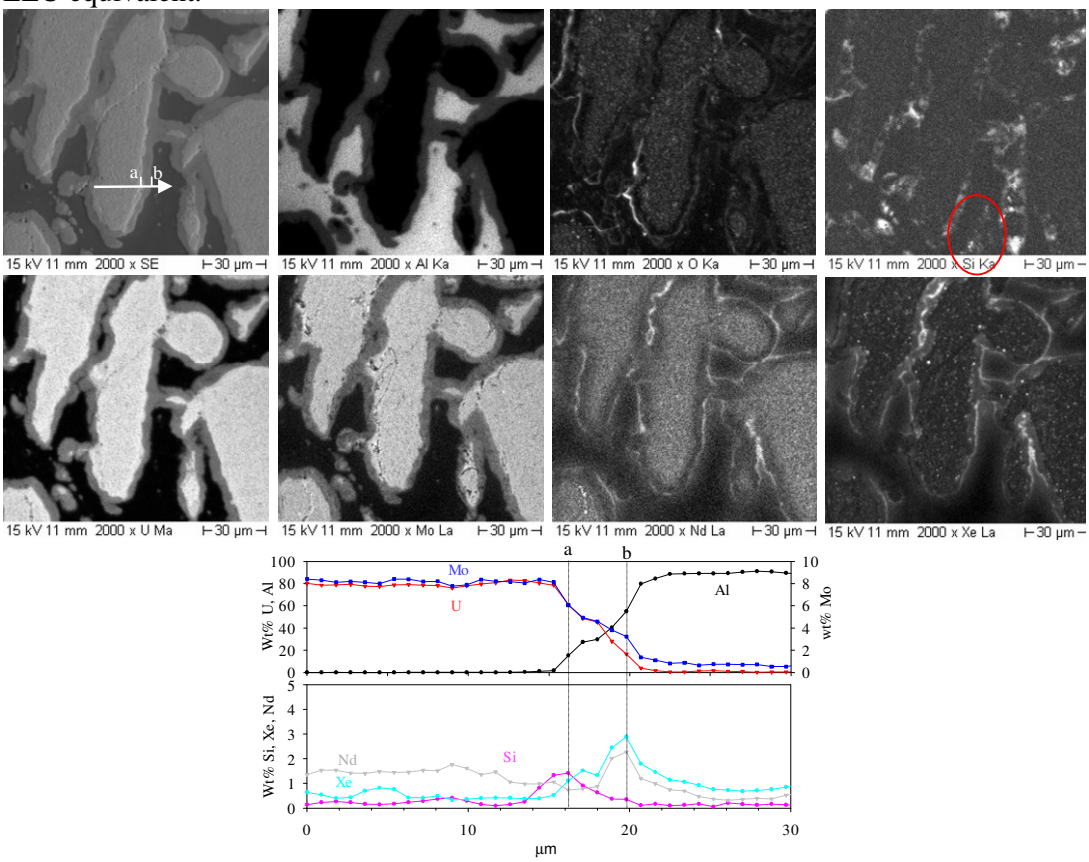


Figure 7 Linescan over a ground U(Mo) kernel in an Al 2.1 wt%Si matrix, located in the meat in fuel plate U8MV8503.

From the silicon X-ray map it is immediately clear that most of the Si in the matrix has disappeared except for some isolated parts (e.g. in the red circle) that are sufficiently far from any fuel particle. From the Al, U, Mo and Si X-ray maps, it is seen that an interaction layer has grown around the fuel kernels and that it contains Si. Similar to the atomised fuel, the amount of Si in the IL is not homogenous.
The increased concentration of oxygen at the interface of the IL and the matrix, seen in the oxygen X-ray map should be considered an artifact. It concerns cracks that are most probably introduced during sample preparation.

The Xe and Nd maps shows that this fission gas has agglomerated into larger bubbles inside the fuel particles. At the interface of U(Mo) fuel kernel with the IL larger xenon bubbles can be found. The typical fission product halo can be observed at the interface of the IL with the matrix.
The quantitative linescan shows the formation of a silicon rich layer near point 'a'. Between 'a' and 'b', an IL having a composition of $(U,Mo)Al_4$ is calculated (corrected for BU). At 'b' the fission product halo is measured.

Figure 8 shows the secondary electron (SE) image and X-ray maps recorded at the transition of the meat with the cladding in fuel plate U8MV8503 at a position where the burnup equals ~13 % ^{235}U or 32.5 % ^{235}U LEU equivalent.

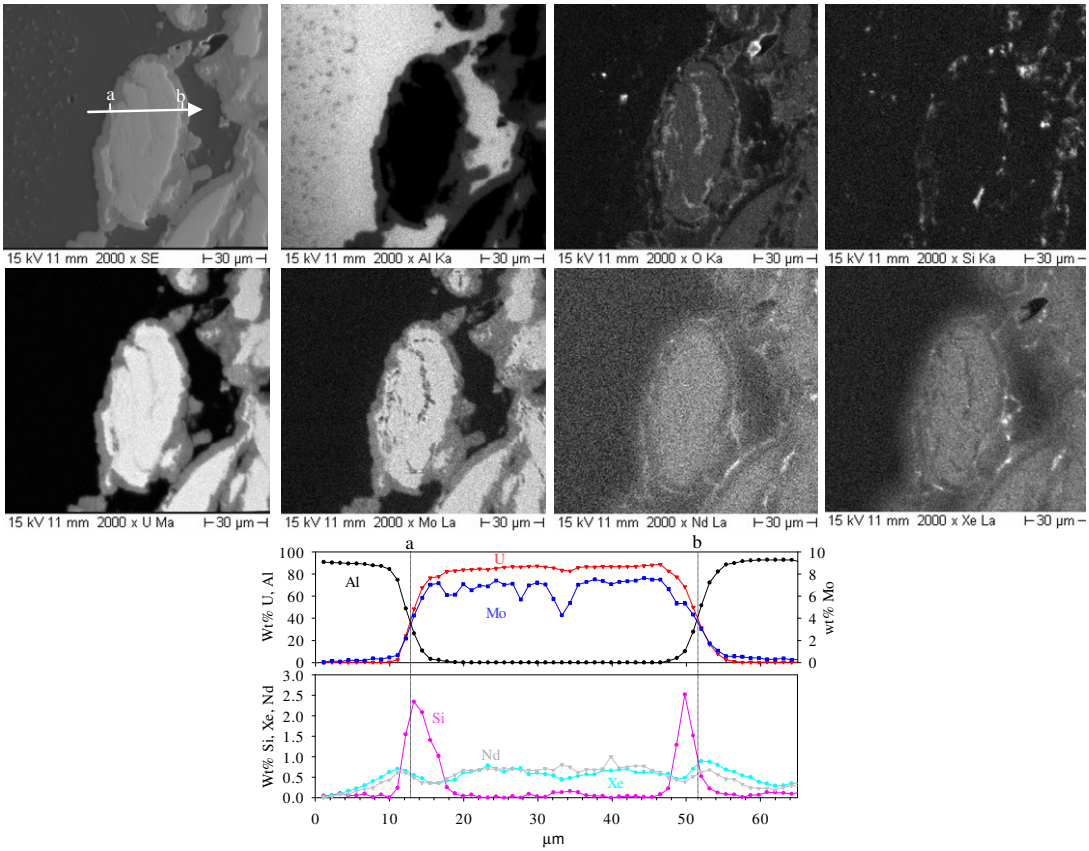


Figure 8 Linescan over a ground U(Mo) kernel in an Al 2.1 wt%Si matrix, located at the transition meat cladding in fuel plate U8MV8503.

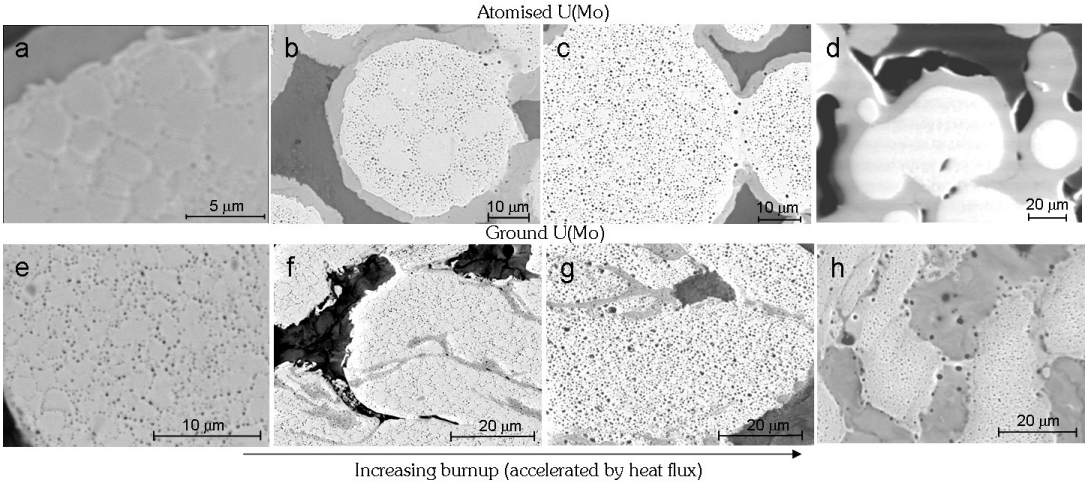
The oxygen map again shows inside the fuel kernel the UO_x stringers which contain precipitated, metallic Mo. The xenon map shows that this fission product has not yet agglomerated into bigger bubbles, as can be expected at lower burnup, but it is interesting to see that the gas is not detected in the UO_x stringers.
The linescan defined over this fuel particle shows the influence of Si on the formation of the IL. In 'a' and 'b', an abrupt change from matrix to fuel is recorded indicating that hardly any IL has been created. At both 'a' and 'b' a Si rich layer has formed due to the interaction of the Si particles and U(Mo) fuel. The Si peaks are located just inside the fuel kernels, rather than on the interface.

4 Discussion

Overall, the irradiation induced microstructural changes to atomised and ground fuel are relatively similar. However, some differences can be discerned. These mainly concern the oxidation of the fuel particles prior to the irradiation and the behavior of the fission gas during irradiation.

The growth of an oxide layer on the metallic particles lying at the meat-clad interface is seen for both ground and atomised fuel. This oxide layer is only formed at the outer side of the fuel plate and is related to the fabrication procedure (hot rolling). The presence of such an oxide layer on the fuel kernel could alter the properties of the IL, but it seems not to prevent or reduce its growth.
The UO_x stringers inside the fuel kernels are only observed in the ground fuel. It is therefore assumed that these are formed during the grinding process of the fuel particles. The presence of these oxide stringers in the fuel kernel could change the properties of the ground fuel, but this will be a local effect only. It is seen that in these stringers, Mo can be found as metallic precipitate. As Mo is not soluble in UO_x, one can indeed expect that during the oxidation of the fuel (grinding process or rolling process), molybdenum precipitates. With the presence of Al and U in the meat, the oxygen potential will stay low enough to keep Mo in a metallic form during irradiation.
The general effect of the oxides on the fuel plate behaviour appears to be minimal, but it is difficult to assess to what extent the oxide alters the properties of the interaction layer.

The most striking difference between atomised and ground fuel is in the behavior of the fission gas during irradiation. The evolution of the microstructure for both types of fuel with



increasing burnup is illustrated in figure 9.

Figure 9 Evolution in microstructure of atomised and ground fuel, with increasing burnup.

At low burnup, it has been measured [6] that the fission gas in the atomised fuel is precipitated into nanobubbles, which align in an ordered lattice structure. As the burnup increases, the growing nanobubbles will agglomerate first at the cell boundaries (these cells are typical for atomised fuel) (fig.9a). Even at higher burnup, one can still observe these patches of nanobubbles in the atomised fuel (fig.9b), but the number and size of the bubbles on the cell boundaries increases and the zones around the boundaries become wider. After a certain BU, a stable and homogenous distribution of bubbles of nearly similar sizes is seen

(fig.9c), although this will also be influenced by variations in the microstructure of the kernels, for which the cell structure can be very different.
The fact that the nanobubble lattice exists up to higher burnup, is considered one of the reasons that fuel plates containing atomised fuel, irradiated under specific conditions, will show void formation. The growing layer resulting from the interaction between the Al matrix and the U(Mo) fuel, will incorporate the nanobubbles present in the fuel cells. In this amorphous IL, the bubbles can not be contained in a regular lattice and will agglomerate at the interface of the IL and matrix and eventually form large voids (fig.9d). The growth of the IL, and hence the incorporation of the nanobubbles in it, is accelerated by a higher operating power.

For ground fuel, the xenon gas will also precipitate in nanobubbles, but due to the high defect concentrations (dislocations (cold work) and grain boundaries) in the ground particles, the nanobubbles will readily concentrate around these defects (fig.9e) and a regular nanobubble lattice will only form in local, defect free areas of the U(Mo) lattice. With increasing burnup, the bubbles on the grain boundaries will coalesce and grow (fig.9f). At higher burn up, a coarsening to a stable, more homogenously dispersed bubble structure is observed (fig.9g).
In the ground fuel, there will be fewer nanobubbles present that can be incorporated in the IL during its growth. This means that, compared to atomised fuel, less fission gas will be transported out of the fuel through the IL to the interface with the matrix, thus reducing the possibility that at this location voids are created.
However, the PIE on the IRIS-TUM plates described in this paper, indicate that the xenon fission gas bubbles in the fuel kernel accumulate at the interface fuel-interaction layer (fig.9h), since they are too large to get incorporated into the IL. The upcoming PIE on the swollen IRIS-TUM plates U8MV8501 and U8MV7003, irradiated to higher burnup [3], might show void creation at the IL-fuel interface.
This accumulation of bubbles at the fuel-IL interface is also observed by AECL in U7Mo fuel, manufactured by a different method, and irradiated to high burnup in the Canadian NRU reactor [9] (fig. 10).

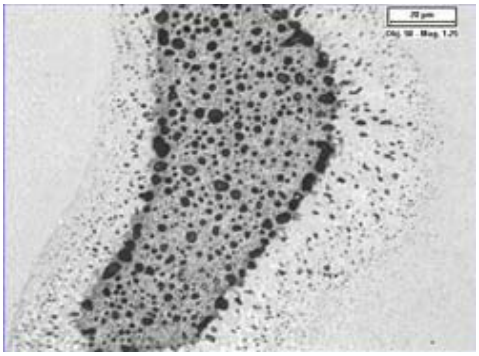


Figure 10 Image of U7Mo fuel after 80 at% burnup (from [9]).

The effect of adding silicon to the matrix, is similar in both fuel types. Only at those positions on the fuel kernel surface where there was close contact with the silicon particles prior to the irradiation, a silicon rich layer has formed. It is this type of layer that limits the growth of the U-Mo-Al interaction layer [10].
Those Si particles not in close contact to the fuel kernel, will gradually be incorporated by the growing IL. The apparent larger amounts of Si found in the IL of ground fuel compared to the atomised fuel, is the consequence of the larger surface area of the ground fuel particles. Due

to this, relatively more IL is formed and as such almost no matrix and hence silicon particles remain.

5 Conclusions

Comparison of the results from the PIEs on atomised and ground fuel shows that one of the major differences found after irradiation between both types of fuel is the behavior of the fission gas. The larger lattice stress due to defects present in ground fuel disturbs the formation of the nanobubble lattice and leads to larger fission gas bubbles at lower burnup. This could explain why this type of fuel withstands higher operating conditions better. On the other hand, the creation of voids at higher burnup at the interface of the fuel and the interaction layer in ground fuel could be possible. Upcoming PIE on ground fuel plates exhibiting swelling should confirm or refute this.

The effect of adding silicon to the matrix, in order to limit the growth of the U-Mo-Al interaction layer, is the same for both types of fuel. A positive result on the thickness of the interaction layer is only seen if there was close contact between the silicon particle and the U(Mo) fuel at the beginning of the irradiation.

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