QUANTITATIVELY SIMULATING FISSION-ENHANCED DIFFUSION IN U-MO/AL BILAYER SYSTEMS BY SWIFT HEAVY ION IRRADIATION

J. Shi, H. Breitkreutz, W. Petry

Forschungs-Neutronenquelle Heinz Maier-Leibnitz (FRM II), Technische Universität München
Lichtenbergstr. 1, 85747 Garching, Germany

ABSTRACT

Swelling behaviour of the interdiffusion layer (IDL) that forms between the U-Mo fuel and the aluminium matrix during irradiation tests is one of the major research targets to improve the fuel's performance. Irradiation with swift heavy ions under appropriate conditions has been demonstrated to be a powerful tool to quickly reproduce certain effects of in-pile irradiation, especially a comparable IDL.

This paper presents new results obtained from several ion irradiation tests at the MLL accelerator in the past years, during which U-Mo/Al model systems were irradiated with high-energy (80 MeV) Iodine-127 ions in a temperature region up to 275 °C. An extensive data set was obtained for the relevant irradiation parameters, such as temperature, ion flux, and ion fluency. Post-irradiation examinations indicate a good agreement between the measured IDL thickness and calculations using a model based on in-pile irradiation results: the deviation at the centre of beam spots is within 10%. The fluency-to-fission-density conversion and the IDL growth correlation were thereby verified in a comprehensive manner enabling a quantitative interpretation of heavy ion irradiation experiments. The findings of this study notably extend the possible applications of this tool in nuclear fuel development.

1. Introduction

Uranium-molybdenum/aluminium (denoted as U-Mo/Al) fuel is the prime candidate for a new, high density research reactor fuel that is being developed to meet the requirements of the global conversion program from highly enriched uranium (HEU) fuels to fuels based on low-enrichment uranium (LEU). The addition of 7wt% to 10wt% of Mo to the U eliminates the anisotropy of the orthorhombic α-phase and stabilizes the desired cubic γ phase during irradiation test [1, 2]. Furthermore, the generated fission gases can be well accommodated up to a high fission density [3]. However, up to now, irradiation tests of this new fuel showed a promising but not yet fully sufficient irradiation behaviour. The build-up of an amorphous interdiffusion layer (denoted as IDL) due to ion-enhanced interdiffusion and thermal spiking phenomena occurring between the U-Mo and the surrounding Al matrix has been identified as the main reason for breakaway swelling and pillowing [4, 5, 6].

Previous studies revealed that addition of a small amount of Si in the Al matrix [7, 8] or application of a diffusion-barrier layer between U-Mo und Al [9, 10, 11] can effectively eliminate or greatly reduce the presence of IDL during irradiation. Nevertheless, the dynamics of IDL formation depending on different parameters have not been studied thoroughly. Advancing the understanding of its growth dynamics is therefore one of the primary research interests in this context.

Since the IDL growth is one of the deciding criteria for performance evaluation of U-Mo/Al based fuel, a model to predict IDL evolution is necessary. However, the highly intercorrelated radiation parameters in a reactor (in-pile irradiation) such as fission rate, burn-up, and temperature seriously limit this possibility. In addition, measuring fuel temperature during irradiation is extremely difficult in a reactor. For these reasons, accurate modelling of IDL growth using in-pile irradiation data is difficult and comes with notable uncertainties.

Under this circumstance, irradiation with swift heavy ions at a particle accelerator as an adequate out-of-pile technique has been applied to reproduce the irradiation damage caused by fission fragments during in-pile irradiation tests, e.g. to create IDL in U-Mo/Al based fuels. The feasibility and reliability of this technique have been confirmed by irradiation of U-Mo/Al samples with Iodine-127 ions at 80MeV during previous ion irradiation tests, whereby a qualitative comparison was presented as well [11, 12, 13].

Based on the data of several in-pile irradiation tests, an empirical model has been built and an IDL growth correlation to predict the thickness of the generated IDL between U-Mo und Al without protective measures was developed by Kim and Hofman [14] and later refined by Ye [21]. It is of outstanding interest if this correlation can be supported by the IDL growth behaviour during ion irradiation tests. Hence, Breitkreutz [15] has derived the equations to convert the ion flux and the ion fluency to fission rate and burn-up equivalents, respectively. The applicability has been preliminarily verified using the data from the 4/16 heavy ion irradiation test at the MLL accelerator at TUM [16].

In this paper, the results obtained from recent heavy ion irradiation tests on U-10Mo/Al bilayer fuel samples at the MLL accelerator are presented. The focus will be on the post-irradiation examination results, through which the IDL growth dynamics is further studied. This paper first presents heavy ion irradiation of monolithic U-Mo/Al systems in a relatively high temperature regime (140–275°C). It aims to strengthen the understanding of the influence of different irradiation conditions, e.g. temperature, fission rate and burn-up, on the enhanced IDL growth in U-Mo/Al based fuel, as well as the verification of the IDL growth model depending on the heavy ion irradiation parameters.

2. IDL growth models

The result of the RERTR-1 and -2 irradiation experiments in the ATR reported by Hofman and co-workers [17] shows the first observation of IDL growth in U-Mo/Al system from an irradiation test. A modified Arrhenius equation describing a dependency of the fission-enhanced interdiffusion on fission rate, temperature, and exposure time was developed to predict the thickness of IDL induced by in-pile irradiation [14]:

$$\mathrm{Y}^2 = \mathrm{A} \cdot \dot{f}^{\mathrm{p}} \cdot \exp(-\frac{\mathrm{q}}{T}) \cdot t \cdot f_{\mathrm{Mo}} \qquad \text{[Eq. 1]}$$

Where Y is the IDL thickness in µm, $A = 2.6 \cdot 10^{-8}\ \mathrm{\mu m^2 cm^{3p}} s^{p-1}$, $\dot{f}$ is the average fission rate during in-pile irradiation, $p = 0.5$ is the fission rate exponent. T is the normalization temperature in K, $q = 3850\mathrm{K}$ is the fit parameter for T, t is the irradiation time in s and f_{Mo} is the correction factor given by the mass content wt% of Mo in U-Mo. As mentioned above, the equation was derived using in-pile irradiation data; it is therefore very difficult to separate the contributions of temperature and fission rate since they are strongly correlated.

To assess a quantitative analysis of the IDL growth correlation, Breitkreutz [15] developed a conversion mechanism between heavy ion irradiation and the equivalent in-pile quantities, Fission rate and burn-up therefore correspond to a given ion flux and an ion fluency, respectively:

$$\dot{f} = \phi\xi/2 \qquad \text{[Eq. 2]}$$

$$n = \Phi\xi/2 \qquad \text{[Eq. 3]}$$

where ξ is the relative effect factor defined by the number of fission fragments that yield the same effect as one ion, its value can be obtained from SRIM/TRIM calculations.

3. Experiment description

The heavy ion irradiation was performed at the MLL accelerator in Garching, Germany. Unlike the in-pile irradiation, during which the fission products are generated randomly inside the fuel, the ion irradiation has a point source providing a steady ion beam consisting of Iodine-127 ions with 80 MeV. The beam can be further focused by magnetic quadrupole lens systems and then hits the sample surface perpendicularly.

3.1 U-Mo/Al bilayer

Instead of the commonly used dispersion fuels in a reactor, where fuel particles are dispersed in an aluminium matrix, the samples used for this study are monolithic U-10 wt% Mo (denoted as U-10Mo) foils, coated with a PVD-deposited thin layer of Al. In the following, this representative model is referred to as “U-Mo/Al bilayer”. This configuration enables a direct interpretation and a quantitative analysis of the IDL growth dynamics. 13 µm was chosen as the thickness of Al layer (ρ = 2.3 g/cm³ since the density of sputtered layer is generally lower than the raw material) according to SRIM simulations, The Bragg peak[1] is then targeted directly on the U-Mo/Al interface to eliminate the influence caused by different impinging directions of ions, as the fission fragments generated during in-pile irradiation first cross a UMo layer rather than an Al layer.

3.2 Ion Irradiation

3.2.1 Ion beam

This work summarizes the results of the ion irradiation campaigns in the year 2017. The ion beam was transmitted straight to the sample surface, the ions impinged the sample on the Al layer; no beam wobbling was used. With this approach the irradiation over the defined area has a Gaussian intensity distribution: The focusing system of a linear particle accelerator (usually shortened to “linac”) normally leads to a straight beam with an elliptical cross-section, its power density along radial axis follows a bivariate Gaussian distribution, whereby any cut through the centre brings up a univariate Gaussian distribution along the cut-line (Figure 1)

$$\phi(\mathrm{x}) = \phi_{\max} \cdot \exp\left[-\frac{1}{2} \cdot \left(\frac{x-x_0}{w}\right)^2\right] \qquad \text{[Eq. 4]}$$

where x is the position along the cut-line, x_0 is the position of beam centre and w is the Gaussian width. Once the IDL thickness distribution has been determined, the ϕ_{max} and the

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[1] A maximum energy deposition occurring at certain penetration depth during the ions travel through matter.

ion flux profile can be reconstructed with the approach proposed in [16]. The expected IDL thickness can be therefore directly correlated to a given ion flux:

$$Y(r) = (2.6 \cdot 10^{-8} \cdot exp(-\frac{3850\mathrm{K}}{T}) \cdot t \cdot f_{\mathrm{Mo}})^{1/2} \cdot (\frac{\xi}{2})^{1/4} \cdot \phi(r)^{1/4} \qquad \text{[Eq. 5]}$$

It should be noticed that the exponent fission rate exponent $p = 1/2$ was used to reconstruct the beam profile from the IDL thickness, which means its value cannot be verified later in the analysis. Based on this concept, a series of irradiation tests (“4/16 ion irradiation campaign”) have been successfully conducted at two different temperatures (383 K and 413 K) in 2016 [16].

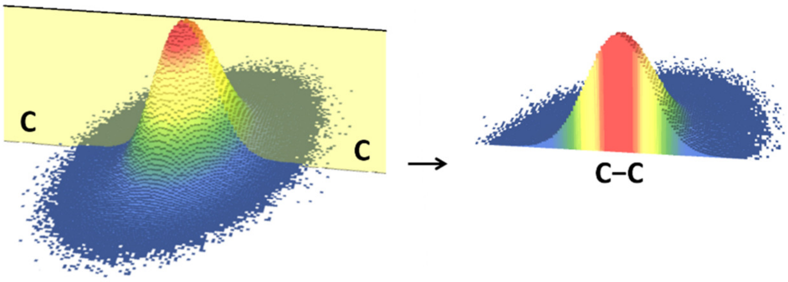


Figure 1. Visualization of a bivariate Gaussian distribution by simulation. A random cut C−C through centre leads to a univariate Gaussian distribution on the obtained cross-section.

3.2.2 Temperature

In this work, the temperature dependence of the interdiffusion behaviour between U-Mo and Al has been studied. Special attention has been paid to temperatures during preparation of samples, as only ion irradiation induced interdiffusion is desired. In-pile test results show that interdiffusion at the interfaces of U-Mo and Al at low temperatures (< 300°C) leads to an amorphous IDL [6]. Thus, the irradiation temperature has been varied up to 275°C by use of a PID temperature controller to maintain a constant temperature on the measurement point, which is set on the sample surface being irradiated. However, as explained in section 3.2.1, the ion beam has a bivariate Gaussian profile and therefore contributes an inhomogeneous heat flux. Nevertheless, the heating power of the ion beam is much lower compared to the external heating. Despite that the ion beam is focused onto a small area, the heat diffuses rapidly. Figure 2 shows a simulation of the temperature profile of sample S4_5 with the Al-surface irradiated by an ion beam of 2.3W (regular beam current, 200pnA I^{7+} at 80 MeV). The shape of the beam spot has been simplified to a circle with a Gaussian width of 0.5mm based on the measurements. The well-designed sample stage ensures good thermal contacts of interfaces of the sample stage. A deference of less than 10°C between the measurement point and the ion beam centre was calculated.

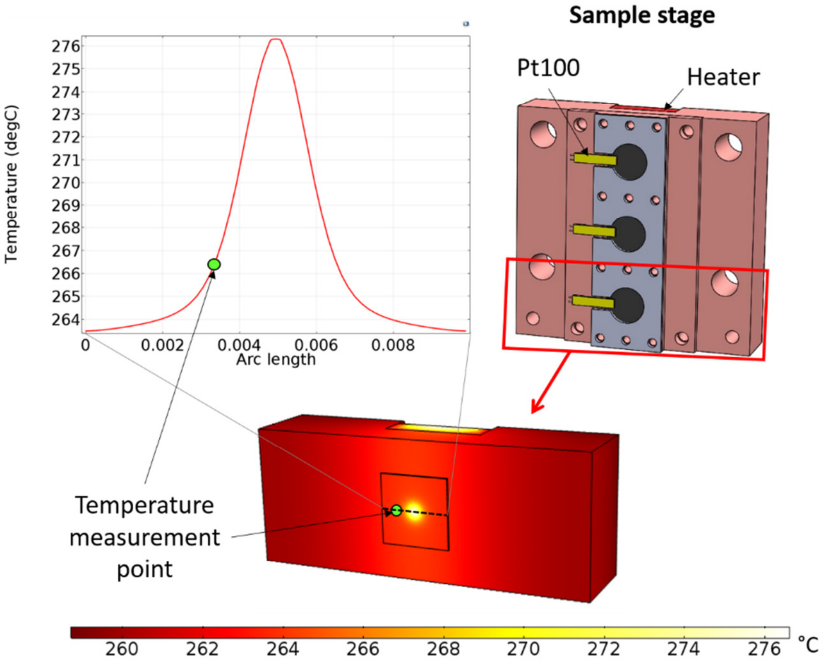


Figure 2. Temperature profile of sample S4_5 irradiated with a Gaussian ion beam. A temperature distribution along the black dashed line crossing the beam centre is shown. The temperature measurement point is located directly on the irradiated surface (marked with a green dot). The calculated temperature difference between the beam centre and the measurement point is less than 10°C.

3.3 Post-irradiation examination

After the ion irradiation, the geometrical information of the footprint left by the ion beam on the sample including the axes a, b and the angle θ between the major axis a and the horizontal line through the footprint centre were determined using optical microscopy (Figure 2). Destructive examinations were performed to determine the IDL thickness distribution along the cut line by embedding, polishing and scanning electron microscopy (SEM).

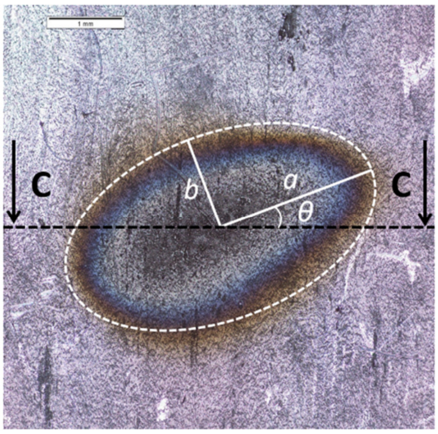


Figure 3. Geometrical measurement of the beam footprint on the sample S4_7. A cross-section C−C was made by vertically polishing the sample until reaching the cut line (black dashed line).

4. RESULTS AND DISCUSSION

During irradiation, a data set was obtained by varying the irradiation parameters; especially those significantly affecting the IDL growth, such as temperature and fluence. 5 samples were irradiated with an ion flux up to $2.4 \cdot 10^{13}\,\frac{\mathrm{ions}}{\mathrm{cm^2 s}}$. Fission density equivalents (burn-ups) of up to $n = 9 \cdot 10^{20}\,\frac{\mathrm{fiss}}{\mathrm{cm^3 s}}$ were reached within 11 hours, which correspond to several days of in-pile irradiation. The conditions of each irradiation experiment and the measurements of the beam footprint are listed in Table 1. Main focus was the investigation of the irradiation temperature.

Table 1 Irradiation conditions

Sample ID	S3_2	S3_4	S4_1	S4_5	S4_7
Final fluence in ions	9.50E+15	3.55E+15	5.13E+15	3.37E+15	4.29E+15
Irradiation time [h]	12.5	3.5	6.7	9,3	11
Temperature [°C]	140	180	200	275	220
Irradiation area [cm²]	0.079	0.069	0.041	0.075	0.071

4.1 SEM examination

Figure 4 gives an overview of a sample after irradiation. Non-irradiated areas were also analysed to see if a thermal IDL formed during fabrication and irradiation. As expected, no IDL has been found in the non-irradiated areas. The quantitative atom concentration ratio U/Al in the IDLs was identified between 1/3 and 1/4 by energy dispersive X-ray spectroscopy (EDX) and therefore corresponds to in-pile findings.

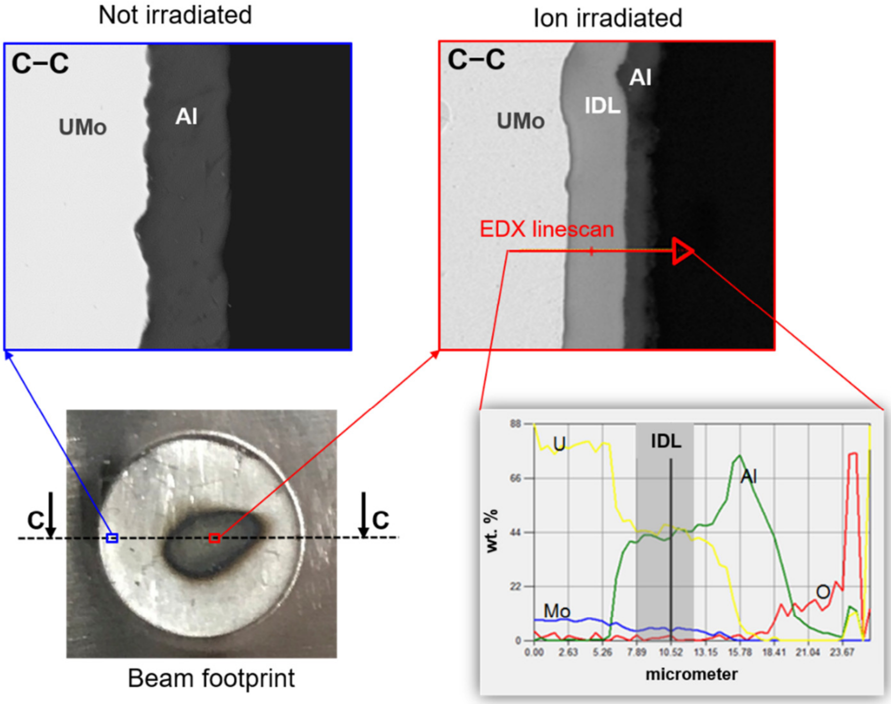


Figure 4. Sample S4_7 after ion irradiation and the corresponding BSE/EDX images at different locations.

4.2 Quantitative analysis of the IDL growth dynamics

As explained in section 3.2.1, the beam profile can be reconstructed with the help of the Gaussian width w and $Y_{\max}$, which can be determined by fitting on the measured IDL thickness along the cross-section. Fig. 5 shows that the data were well-fitted with the correlation $Y_{\max} \propto \phi^{1/4}$ obtained from [Eq. 5], Uncertainties of the parameters were small. The IDL thickness in the centre and at the border can be obtained from the fit-results. The centre flux $\phi_{\max}$ and the ion flux along the cut line are finally determined by means of the method explained in section 3.2.1. Thus, the expected IDL thickness can be easily calculated with [Eq. 5].

The deviation between the expected IDL thickness and the measured values is illustrated in Figure 6. The scattering for thin IDL can also be observed in the fit-results, which can be explained by the measurement uncertainties of the beam footprint and IDL thickness. It should also be kept in mind that in-pile an average deviation of 15% between measured and calculated IDL is found for [Eq. 1] [14, 15]. Therefore, it can be concluded that the prediction derived from [Eq. 1] based on in-pile data matches well with the measurement.

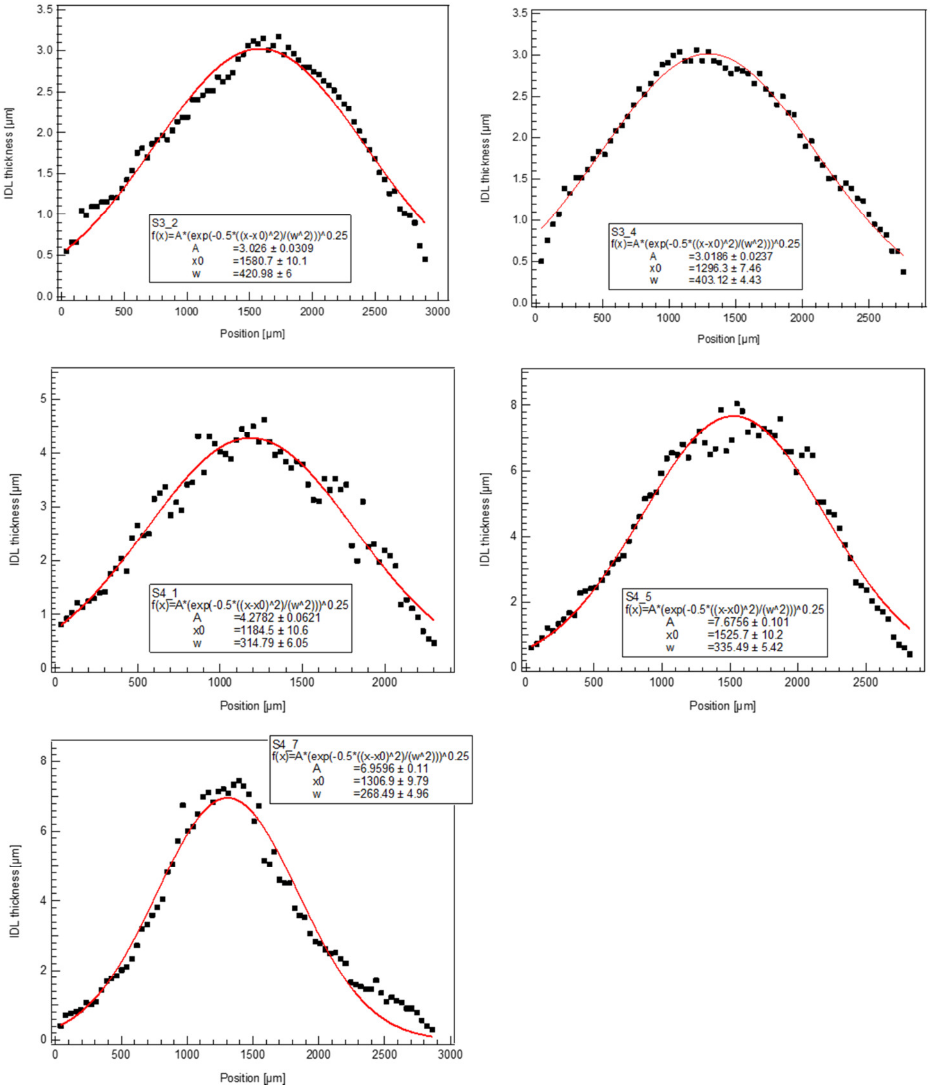


Figure 5. Measured IDL thickness of 5 samples and the fit results. The well-fitted data indicate a good applicability of the proportionality $Y \propto \phi^{1/4}$.

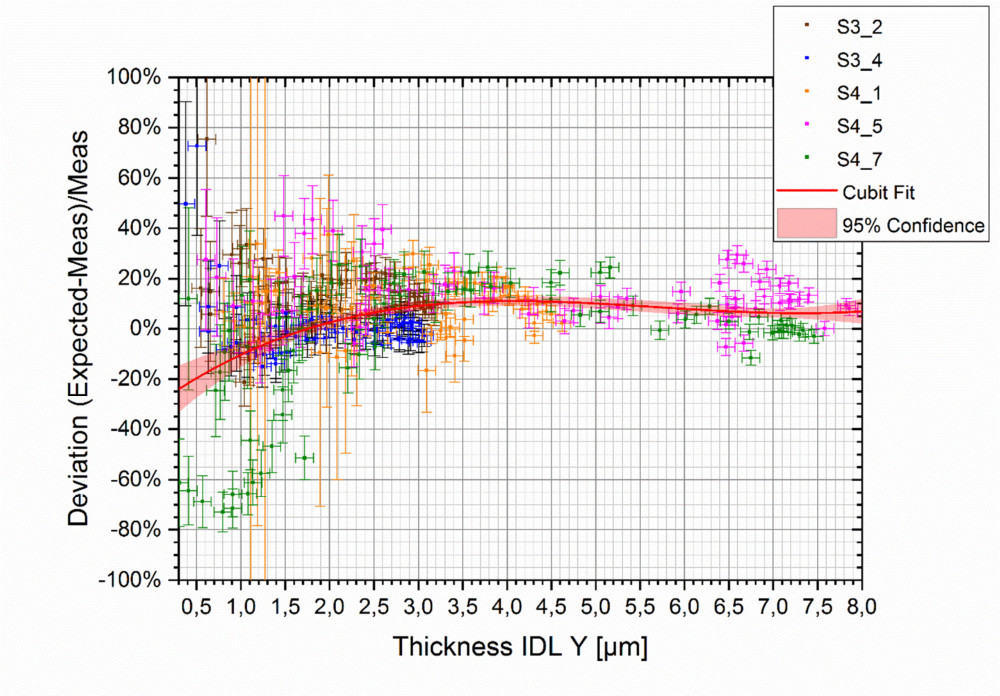


Figure 6. Deviation between the IDL thickness calculated with [Eq. 5] and the in SEM directly measured values of 5 samples irradiated at 140 °C, 180 °C, 200 °C, 220 °C and 275 °C, respectively. Y-axis is the deviation in percent.

Since the [Eq. 1] was developed with in-pile data, verification of the parameters presented in this equation with ion irradiation has been performed. The proportionality factor was studied by solving [Eq. 5] for *A*. In in-pile tests, this factor is assumed to be independent from the burn-up of the fuel, i.e. $A = 2.6 \cdot 10^{-8}$. A preliminary verification of this value has been carried out by ion irradiation in [15] with a fit result of $A = 2.86 \pm 0.1 \cdot 10^{-8}$. In this work, a very similar value $A = 2.88 \pm 0.19 \cdot 10^{-8}$ is obtained by fitting a constant (Figure 7). This matches very well with the one based on in-pile data.

An increased swelling rate has been observed in in-pile tests of SELENIUM plates [20] and previous tests, which leads to the assumption of an accelerated IDL growth at fission rates over $7 \cdot 10^{14}/(\mathrm{cm}^3 \cdot \mathrm{s})$ [22]. This would imply a change of the exponent of fission rate. In spite of the fact that the exponent p of the fission rate was already used to reconstruct the beam flux profile and its absolute value therefore cannot verified using the data based on the reconstruction. As shown in Figure 8, no significant trend of the exponent p of fission rate has been observed.

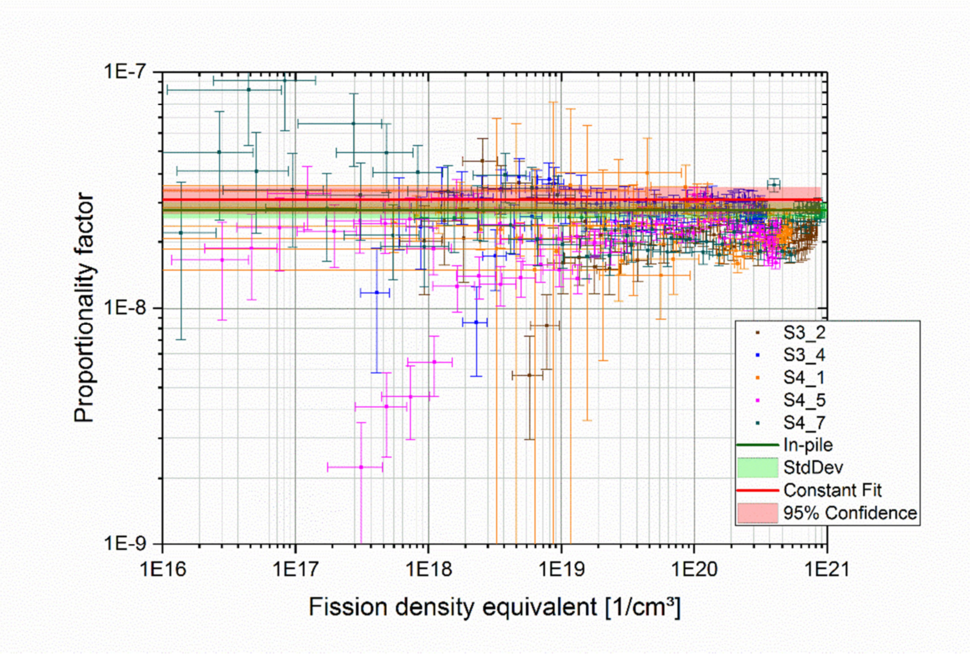


Figure 7. The proportionality factor $A = 2.88 \pm 0.19 \cdot 10^{-8}$ based on ion irradiation data is obtained by constant fit (red). The average value for in-pile is $2.6 \cdot 10^{-8}$ (green). The standard deviation of this constant (green marked area) was estimated as 15% as discussed above.

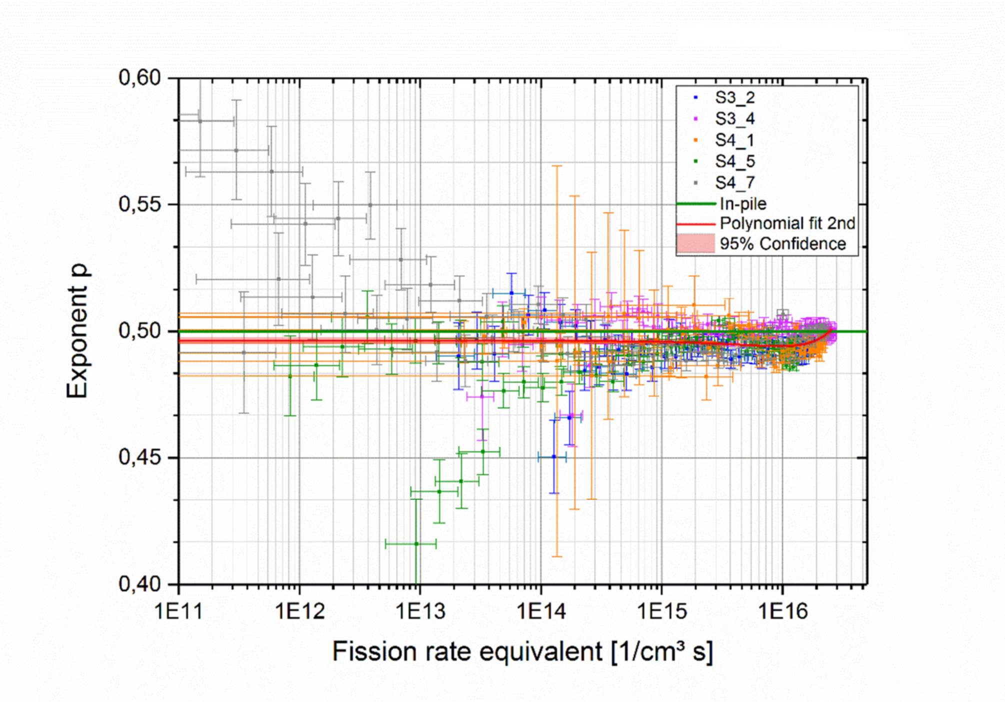


Figure 8. The exponent p of fission rate as a function of fission rate is shown. The fit line of in-pile data ($p = 0.5$) is highlighted with a green line.

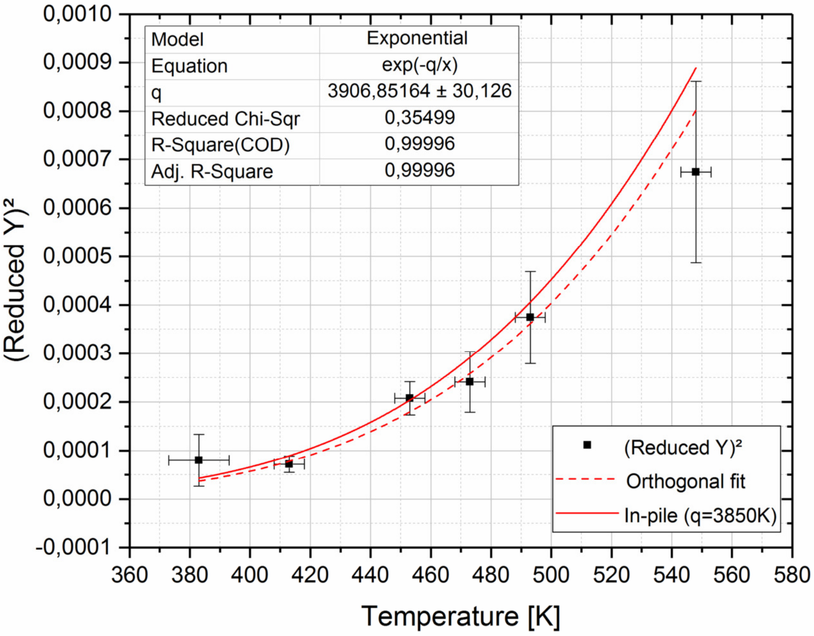
In this work, the samples were irradiated at different temperatures, the temperature-dependency of [Eq. 1] is now able to be verified. A reduced IDL thickness Y_{reduced} is therefore defined to eliminate the influences caused by fission rate and irradiation time:

$$Y^2_{\mathrm{reduced}} = \frac{Y^2}{A \cdot \dot{f}^{0.5} \cdot t \cdot f_{\mathrm{Mo}}} \qquad \text{[Eq. 4]}$$

Hence, a direct correlation of Y_{reduced} to temperature can be described as:

$$Y^2_{\mathrm{reduced}} = \exp(-q/T) \qquad \text{[Eq. 4]}$$

As illustrated in Figure 9, the reduced IDL thicknesses calculated with the data obtained from this work (140 °C – 548 °C) as well as the data of a sample from previous work (U3_5, 110 °C [16]) were plotted as a function of temperature. The temperature-dependency of the Arrhenius equation is verified by orthogonal-fitting the calculated Y^2_{reduced} based on the measured ion irradiation data with an exponential function (dashed curve). A good agreement is achieved comparing the temperature normalization *q* resulted from the following fit results (3907 ± 30 K) and the one obtained from in-pile data (3850 K).



Model	Exponential
Equation	exp(-q/x)
q	3906,85164 ± 30,126
Reduced Chi-Sqr	0,35499
R-Square(COD)	0,99996
Adj. R-Square	0,99996

Figure 9. Verification the Arrhenius-like temperature-dependence of the IDL growth. The temperature uncertainty of the sample irradiated at 110 °C from previous work [16] was above 10°C. After utilizing the improved heating/cooling system, this uncertainty has been reduced to 5°C in this work (see section 3.2.2).

5. Conclusions

Heavy ion irradiation with 80MeV Iodine-127 was conducted on U-Mo/Al bilayer systems at temperatures 140 °C, 180 °C, 200 °C, 220 °C and 275 °C, respectively. The ion-enhanced interdiffusion results in building-up of an IDL with very similar physical properties to the one generated during in-pile irradiation between the U-Mo and the Al layer. This was confirmed by the U/Al ratio identified by EDX (between 1/3 and 1/4).

After the irradiation, a bivariate Gaussian ion flux distribution was reconstructed, a large variety of fission rate and burn-up equivalents were therefore obtained. The measured data from the ion irradiation experiment was compared to the predicted results based on the model proposed by Hofman [14], which match well within the uncertainties of [Eq. 1] and the uncertainties of the experiment in this work. The applicability of the proportionality $Y \propto \phi^{\text{const.}}$ has been verified adequately through the well applicable fitting in Figure 5. The value of the proportionality factor was determined to $A = 2.88 \pm 0.19 \cdot 10^{-8}$, which is in good agreement with the in-pile ($A = 2.6 \cdot 10^{-8}$) and previous ion experiments ($A = 2.86 \pm 0.1 \cdot 10^{-8}$).

The abrupt increase in IDL growth at fission rates $\dot{f} > 7 \cdot 10^{14}\ \mathrm{fiss/(cm^3 \cdot s)}$ observed in the SELENIUM and previous irradiation tests [22] was not found in this work, as no significant trend of the exponent *p* of fission rate was found in Figure 8.

The temperature-dependency derived from [Eq. 1] was verified in the rage of 140−275°C, where the normalization temperature *q* was determined as 3907K ± 30K, which is very similar to the one derived from in-pile data (3850K). It is worth to mention that the U-7Mo was used in the modeling efforts of [Eq. 1], which differs from the one used in this work (U-10Mo) and may influence the comparability.

As a key parameter in irradiation-induced interdiffusion of different materials, temperature is expected to be varied in a wider range to provide more information including determination of the transition temperature between the ballistic mixing regime and temperature dependent mixing regime [23]. Further investigation of chemical composition and microstructure of the generated IDL is still ongoing aiming to allow for an enhanced understanding and comparability of in-pile and ion irradiation approaches.

Acknowledgements

The authors would like to thank the MLL staffs for their great support during our beam-times and also in the times in-between as well as all members of the working group “Hochdichte Kernbrennstoffe” of FRMII. The work was supported by a combined grant (FRM1318) from the Bundes-ministerium für Bildung und Forschung (BMBF) and the Bayerisches Staatsministerium für Bildung und Kultus, Wissenschaft und Kunst (StMBW).

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