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Simulated Fission Gas Behavior in Silicide Fuel at LWR Conditions

Nuclear Engineering Division

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ABSTRACT

As a promising candidate for the accident tolerant fuel (ATF) used in light water reactors (LWRs), the fuel performance of uranium silicide (U_3Si_2) at LWR conditions need to be well-understood. However, existing experimental post-irradiation examination (PIE) data are limited to the research reactor conditions, which involve lower fuel temperature compared to LWR conditions. This lack of appropriate experimental data significantly affects the development of fuel performance codes that can precisely predict the microstructure evolution and property degradation at LWR conditions and therefore evaluate the qualification of U_3Si_2 as an AFT for LWRs. Considering the high cost, long timescale, and restrictive access of the in-pile irradiation experiments, this study aims to utilize ion irradiation to simulate the in-pile behavior of the U_3Si_2 fuel. Both *in situ* TEM ion irradiation and *ex situ* high-energy ATLAS ion irradiation experiments were employed to simulate different types of microstructure modifications in U_3Si_2. Multiple PIE techniques were used or will be used to quantitatively analyze the microstructure evolution induced by ion irradiation so as to provide valuable reference for the development of fuel performance code prior to the availability of the in-pile irradiation data.

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1. Ion Irradiation Experiments Plan

Have advantages in thermal conductivity and heavy metal density, U_3Si_2 has been considered as a promising candidate as an accident tolerant fuel (ATF) materials used in light water reactors (LWRs). In order to evaluate the qualification of U_3Si_2 as an ATF applied in LWRs, its fuel performance during steady state operation as well as its accident tolerance at different accident scenarios must be precisely assessed. However, the existing in-pile experimental data of U_3Si_2 are limited to the research reactor conditions, which usually involve low fuel temperature/high burnup[1,2] or very high temperature (> 1000°C)[3]. The lack of experimental data measured at LWR conditions makes it challenging to develop fuel performance code that can predict the behaviors of U_3Si_2, especially the fission gas behavior, with confidence. Although in-pile neutron irradiation experiments will provide credible references for the development of advanced fuel performance codes, it takes long time to perform post-irradiation examinations (PIEs) of neutron-irradiated samples and make the results available to the community. Thus, it would be of great merits to utilize ion irradiation to simulate the radiation effects induced by neutrons and then to have the experimental data available for fuel performance code development prior to the release of in-pile data.

This project aims at investigating the fuel performance of U_3Si_2 at LWR conditions, especially the amorphization threshold and fission gas behavior, based on the existing ion irradiation and post-irradiation examination (PIE) capabilities at Argonne National Laboratory (ANL). Three sets of irradiation experiments were planned for this project. Using the U_3Si_2 fuel samples provided by Idaho National Laboratory[4], the amorphization threshold measured by Birtcher et al.[5] is first re-examined using the *in situ* capability of the IVEM-Tandem facility. Then, the high-energy ion irradiation generated by the Argonne Tandem Linac Accelerator System (ATLAS) is adopted to emulate the fission fragments with energy of approximately 100 MeV so as to validate the TEM amorphization threshold experiment. In addition, the morphology of Xe bubbles will also be investigated based on ATLAS irradiated samples.

1.1 *In Situ* Irradiation Experiments on Amorphization Threshold

Birtcher et al. measured the amorphization threshold of arc-melted U_3Si_2 using *in situ* TEM irradiation[5]. As a different batch of U_3Si_2 specimens that were fabricated by another technique (cold pressing and sintering) will be used in this project, a validation experiment will first be performed using IVEM-Tandem. Both arc melted and sintered specimens are irradiated to investigate the influence of the conditioning and grain size of the samples. In addition, as the LWR application is the focus of this project, higher study temperatures will also be considered here.

Due to the validation nature of this experiment, the experiment conditions are duplicating the previous experiment to a large extent. A 1.0 MeV Kr ion beam is used to irradiate the TEM specimens instead of the 1.5 MeV Kr ion beam in the original experiments due to the change of accelerator setup at IVEM-Tandem. Assuming 50 nm sample thickness, SRIM gives an average damage of 2.1×10^{-15} dpa per 1 ion/cm^2. Three ion irradiation temperatures will be taken in to account: room temperature, 150°C, and 300°C. The former two temperatures were chosen to replicate Birtcher et al.'s results[5], the latter one was chosen to expand this threshold to LWR conditions. The amorphization will be identified by analyzing the electron diffraction patterns of the specimens.

In this experiment, the target maximum damage level will be 8 dpa, which approximately takes 7 hours to achieve assuming a typical IVEM-Tandam flux of 1.56×10^{11} ion/cm^{-2}·s. For those higher temperatures, the amophization threshold could be very high (>>8 dpa), or even cease to exist. In that case, a higher ion flux might be used to achieve higher target doses. Even higher dose may be realized by ATLAS irradiation that will be discussed in the next section.

Table 1.1: Summary of IVEM-Tandem experiment plan

ID	T(°C)	dpa	Burnup	Ion Source	Dose rate (ion/cm^{-2}·s)	Target Dose (ion/cm^{-2})	Irradiation Time (h)	Comments
1-1	R.T.	2.0	0.004%	1.0 MeV Kr	1.56×10^{11}	9.52×10^{14}	1.7	Arc-melted
1-2	150	2.0	0.004%	1.0 MeV Kr	1.56×10^{11}	9.52×10^{14}	1.7	Arc-melted
1-3	300	8.0	0.017%	1.0 MeV Kr	1.56×10^{11}	3.81×10^{15}	6.8	Arc-melted
1-4	R.T.	2.0	0.004%	1.0 MeV Kr	1.56×10^{11}	9.52×10^{14}	1.7	Sintered
1-5	150	2.0	0.004%	1.0 MeV Kr	1.56×10^{11}	9.52×10^{14}	1.7	Sintered
1-6	300	8.0	0.017%	1.0 MeV Kr	1.56×10^{11}	3.81×10^{15}	6.8	Sintered

1.2 ATLAS Irradiation Experiments on Amorphization Threshold

As a physics research facility, ATLAS is capable of producing heavy ions with energy level of GeV. For material research applications, only the first set of linacs are taken use of, which provides an up to 1 MeV/amu acceleration capability (~100 MeV for heavy ions like Xe). Therefore, the kinetic energy of fission fragments can be replicated. Meanwhile, the high energy ions create a deep damage profile, which help eliminate the surface effects that compromise the interpretation of irradiation effects of ion irradiation experiment with conventional energy level.

Here, 80 MeV Xe ions will be used to irradiate the U_3Si_2 specimens. The SRIM code[6,7] was used to calculate the damage profile and ion range, as shown in Figures 1.1 and 1.2. The Xe range is ~6 micron, which is much deeper than conventional ion source (less than 1 micron).

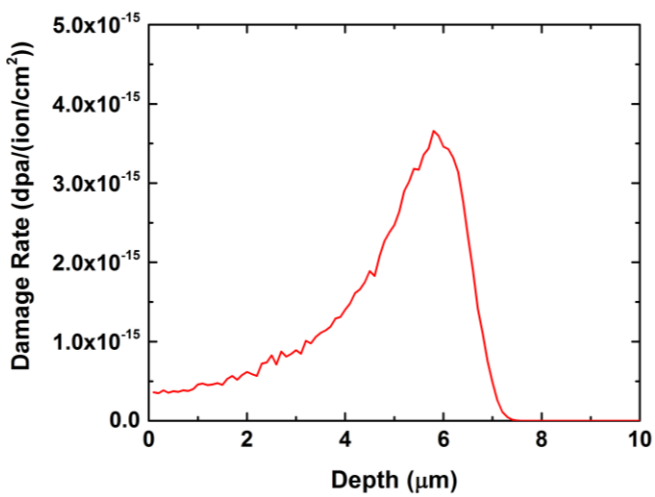

Figure 1.1 SRIM simulated damage profile of 80 MeV Xe ions

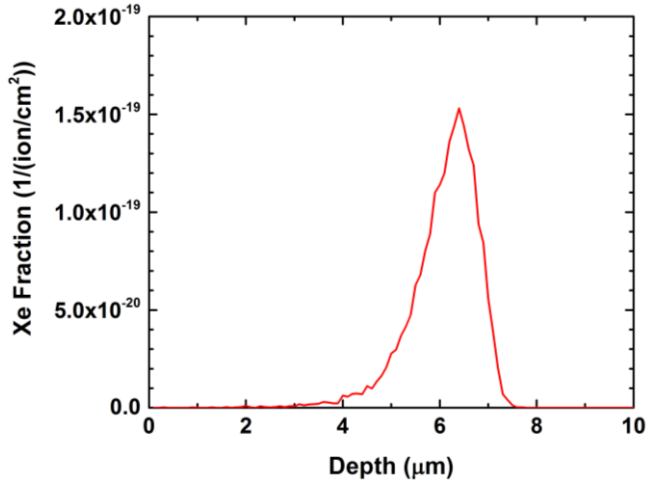

Figure 1.2 SRIM simulated Xe fraction profile of 80 MeV Xe ions

In order to further validate the amorphization threshold measured by IVEM and expand it to LWR condition, three temperatures (150°C, 300°C, and 450°C) and a peak irradiation dose of 8.0 dpa are selected to form the irradiation condition matrix. For an 8.0 dpa peak damage, a 0.5 dpa to 8.0 dpa damage profile can be found throughout the radiation damage range. Due to the surface effects coupled with IVEM experiments, changes in the amorphization threshold are possible. At ATLAS, a peak ion beam flux is approximately 9.2×10^{11} ion/cm^2·s[8]. In that case, it takes less than an hour to approach 8.0 dpa peak dose.

Since 80 MeV Xe ions create a deep damage profile with different dpa values at different depth. FIB samples of approximately 10 μm×10 μm×10 μm blocks will be prepared from the irradiated specimens. The blocks will be investigated using synchrotron X-ray diffraction at Advanced Photon Source (APS). A 1 μm×10 μm beam size will be used to perform the depth scan. Thus, different dpa regions in the same specimens can be analyzed separately, giving more detailed information of the amorphization threshold.

Table 1.2 Summary of ATLAS experiment plan for amorphization investigations

ID	T(°C)	dpa	Burnup	Ion Source	Dose rate (ion/cm^{-2}·s)	Target Dose (ion/cm^{-2})	Irradiation Time (h)	Comments
2-1	150	8.0	0.001%	80 MeV Xe	9.2×10^{11}	2.76×10^{15}	0.83	Sintered
2-2	300	8.0	0.004%	80 MeV Xe	9.2×10^{11}	2.76×10^{15}	0.83	Sintered
2-3	450	8.0	0.017%	80 MeV Xe	9.2×10^{11}	2.76×10^{15}	0.83	Sintered

1.3 ATLAS Irradiation Experiments on Bubble Morphology

ATLAS ion irradiation source is also used here to implant Xe into U_3Si_2 to simulate the evolution of Xe bubbles in the fuel at LWR conditions. 80 MeV Xe is used in this part of the experiments. According to the SRIM calculation[6,7], 1 fission/cm^3 results in around 1.69×10^{-18} dpa. Thus, a 5% typical LWR burnup leads to 2411 dpa. Assuming each fission reaction produce 0.24 Xe atoms, 5% burnup gives a 0.72% Xe fraction in U_3Si_2.

According to the SRIM calculation shown in Figure 1.2, assuming the Xe diffusion is limited in the 4 to 7 micron range, the average Xe fraction induced by 1 ion/cm^2 is 7×10^{-20}. Therefore, 1.03×10^{17} ion/cm^2 is required to replicate the Xe fraction in 5% burnup U_3Si_2 fuel, which typically takes 31 hours at ATLAS. This also gives a peak dpa around 350, making the samples also qualified for amorphization examination at intermediate dose condition. Three temperatures will be used: 300°C, 450°C, and 600°C, which respectively simulate the temperature conditions near the surface and the center of the fuel pellet in LWRs[3]. It is also worth mentioning that the peak target dpa of ion irradiation is only approximately seventh of the actually damage of that in in-pile-irradiated specimens, which is supposed to involve insufficient radiation effect (e.g. radiation-enhanced diffusion, RED, of Xe). A possible solution is to use other high-energy fission product ions (such as Zr and I) to create the rest of the damage.

The target Xe content and irradiation temperatures are selected to better replicate the typical conditions in LWRs. The conditions adopted now are within the range of the in-pile irradiation plan. Multiple material characterization techniques, such as SEM, TEM, and synchrotron scattering will be used to investigate the bubble morphology in those samples.

Table 1.3 Summary of ATLAS experiment plan for Xe bubble morphology

ID	T(°C)	dpa	Burnup	Ion Source	Dose rate (ion/cm^{-2}·s)	Target Dose (ion/cm$^{-2)}$	Irradiation Time (h)	Comments
3-1	450	350	5%	80 MeV Xe	9.2×10^{11}	1.03×10^{17}	31.1	Sintered
3-2	600	350	5%	80 MeV Xe	9.2×10^{11}	1.03×10^{17}	31.1	Sintered
3-3	300	350	5%	80 MeV Xe	9.2×10^{11}	1.03×10^{17}	31.1	Sintered

2. *In Situ* TEM Ion Irradiation Experiments

The U_3Si_2 ingots were first made by arc melting. Then the arc-melted U_3Si_2 was ball milled to form fine powder. The powder was then cold pressed and sintered to fabricate U_3Si_2 fuel pellets (see Ref. [4] for more details). Both arc-melted and sintered samples were investigated using the *in situ* TEM ion irradiation experiments. The TEM samples were made by focused ion beam (FIB). The *in situ* TEM experiments were performed on the IVEM-Tandem facility at Argonne National Laboratory (ANL) working at 300 kV. In this report, only room temperature irradiation results are reported, experiments at elevated temperatures will be performed and reported in a future report.

2.1 Amorphization Threshold of Arc-Melted U_3Si_2 Samples

The arc-melted U_3Si_2 sample was first irradiated at room temperature to replicate Birtcher et al.'s results [5]. The sample was made by arc melting the mixed uranium and silicon powders. The arc-melted sample has grain size larger than the typical FIB sample size (~10 μm). As a result, there is only one grain in the investigated sample.

TEM observation of as-received sample shows the large U_3Si_2 grain with some stacking faults in it (see Figure 2.1a). The electron diffraction pattern also indicates the crystalline state of the material (see Figure 2.1b).

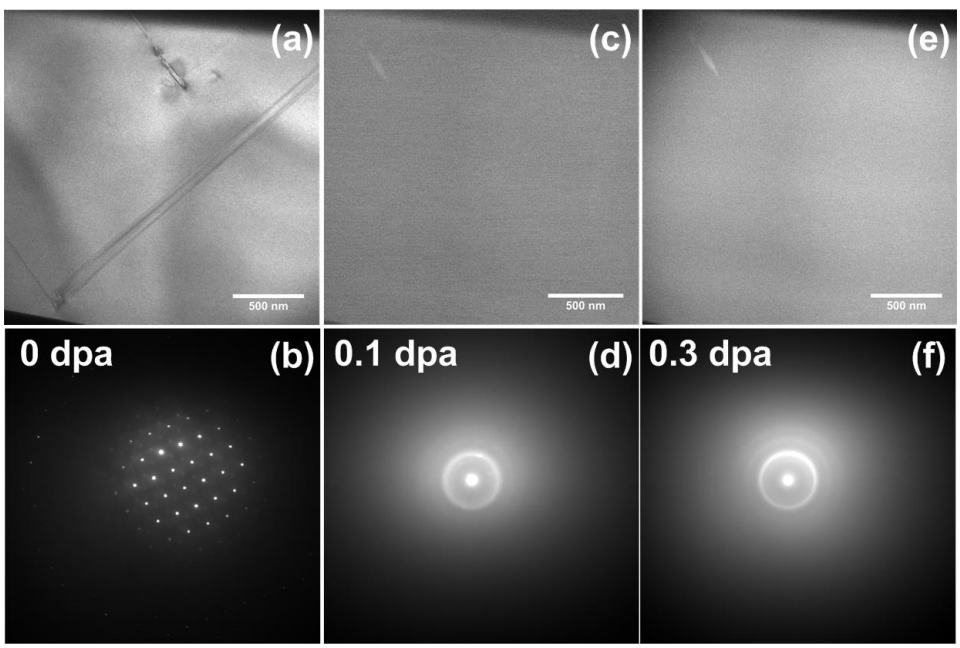

Figure 2.1 Amorphization of arc-melted U_3Si_2 irradiated at room temperature

1 MeV Kr ions were used to irradiate the arc-melted U_3Si_2 sample. At 0.1 dpa, the majority of the U_3Si_2 phase has already become amorphous. According to the diffraction pattern (Figure 2.1d), the diffraction spots can barely be distinguished, whereas the ring structure contributed by the amorphous phase is dominant. In the bright field image (Figure 2.1c), due to the amorphization, the stacking faults disappeared because of the loss of long distance order. As the radiation dose increases to 0.3 dpa, the sample is fully amorphous as shown in Figure 2.1f. After amorphization, due to the loss of long distance order, there is little contrast in the bright

field image. The only feature is a bright area probably originating from the mass-thickness contrast of a small precipitate (Figure 2.1e).

The 0.3 dpa full amorphization threshold determined in this study is consistent with Birtcher et al.'s measurement.

2.2 Amorphization Threshold of Sintered U_3Si_2 Samples

The sintered U_3Si_2 sample was also irradiated at IVEM-Tandem using 1 MeV Kr ions. The sintered sample contains smaller grains so that there exist multiple grains in a single FIB specimen. As shown in Figure 2.2, there are at least four matrix grains and one precipitate in the specimen. According to the manufacturer[4], three phases exist in the sintered U_3Si_2 sample, the primary phase U_3Si_2 and two secondary phases, USi and UO_2. Four grains (G1~G4) were selected for further investigation in this sample, as shown in Figure 2.2. Based on the diffraction patterns, G1, G2, and G4 are U_3Si_2, while G3 is a UO_2 precipitate. In the as-received sample, a series of voids exist in the materials due to the sintering procedure (see voids in G1, Fig. 2.2).

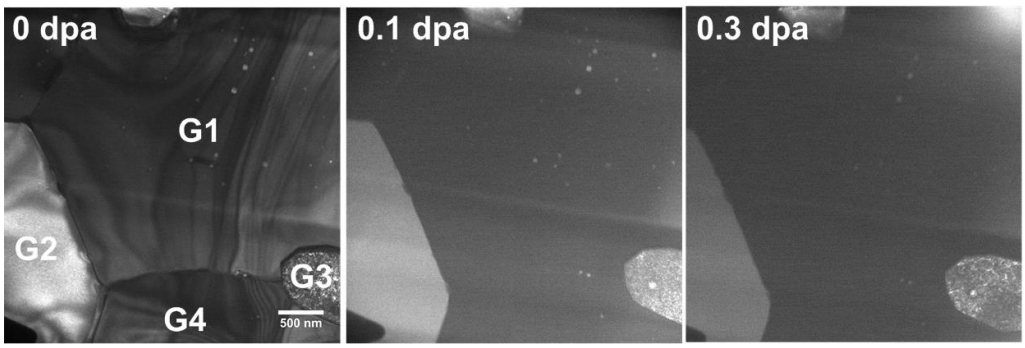

Figure 2.2 Bright field images of the sintered U_3Si_2 irradiated at room temperature

After only 0.1 dpa Kr irradiation, G1 and G4 becomes fully amorphous. Due to the loss of crystal structure, the diffraction contrast (dislocations, bending contour, etc.) in G1 and G4 disappears. Meanwhile, G2 only becomes partially amorphous, while the original diffraction pattern can still be distinguished. At 0.3 dpa, G2 also becomes fully amorphous. The difference in amorphization threshold may come from the different crystallographic orientations of those grains. Also, after amorphization, the contrast between G2 and G1/G4 still remains. This is probably due to the different thickness during the FIB preparation, which may also originate from the crystallographic orientation difference and consequent preferential ion etching. Additionally, the UO_2 precipitate, G3, maintains its fluorite structure throughout the experiment.

Therefore, the the matrix phase in the sintered U_3Si_2 sample has the consistent amorphization behavior as the arc-melted U_3Si_2 sample.

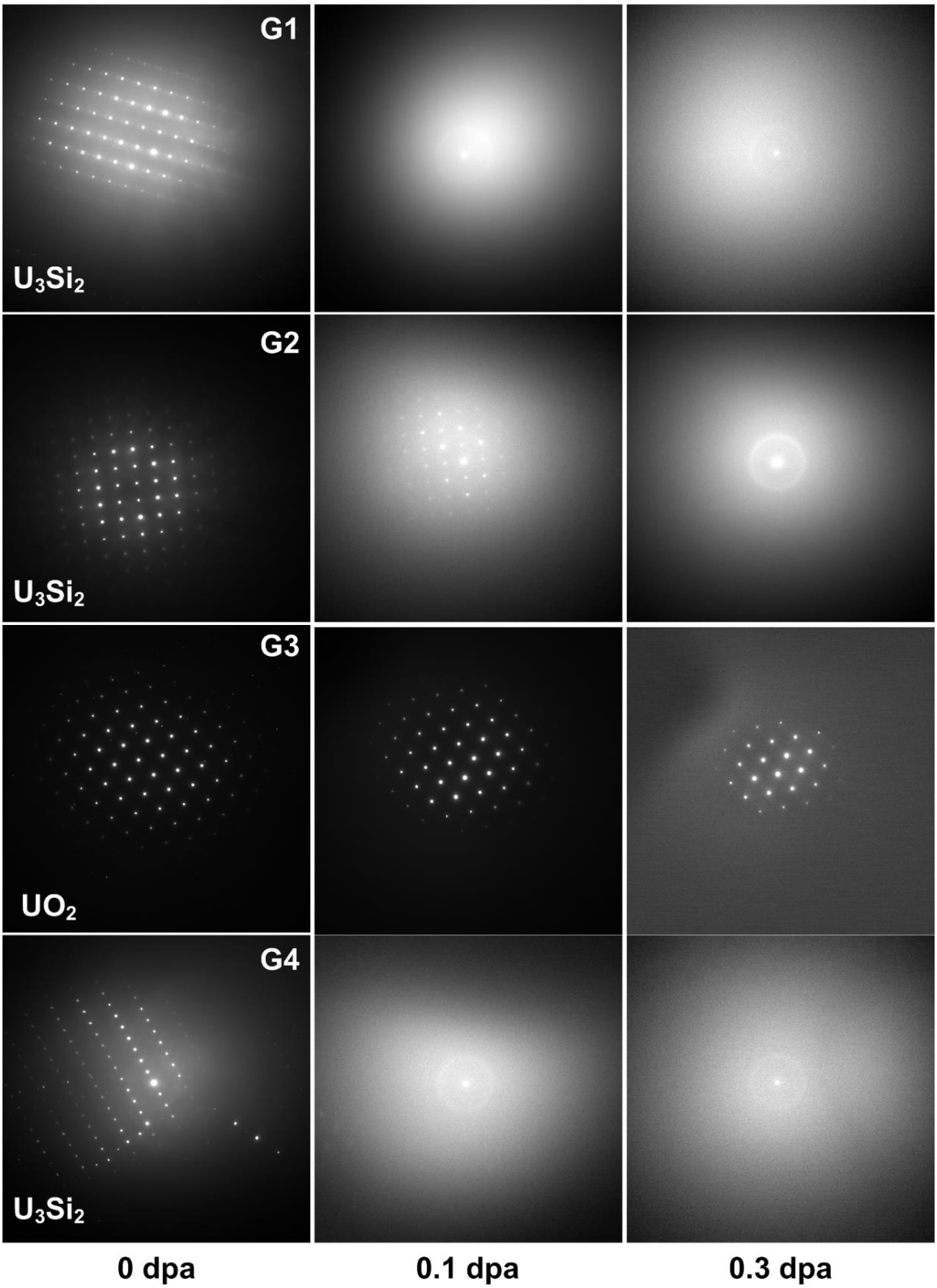

Figure 2.3 Diffraction pattern evolution of the four grains in the sintered sample

3. ATLAS High-Energy Ion Irradiation Experiments

The high-energy Xe ion irradiation experiment was performed for U_3Si_2 samples at ATLAS. ATLAS is a superconducting linear accelerator (linac) facility for heavy ions at energies in the vicinity of the Coulomb barrier. The ion irradiation experiment involved in this study does not need to use all the acceleration capability of ATLAS. Instead, the ion irradiation can be conducted right after the PII linac and before the booster linac. After the PII linac, ATLAS is capable of accelerating heavy ions up to 1 MeV/amu, which is over ~100 MeV for heavy ions such as Xe. An ion irradiation chamber has been established there, which is the foundation of the high-energy ion irradiation experiments discussed in this report (see Figures 3.1 and 3.2).

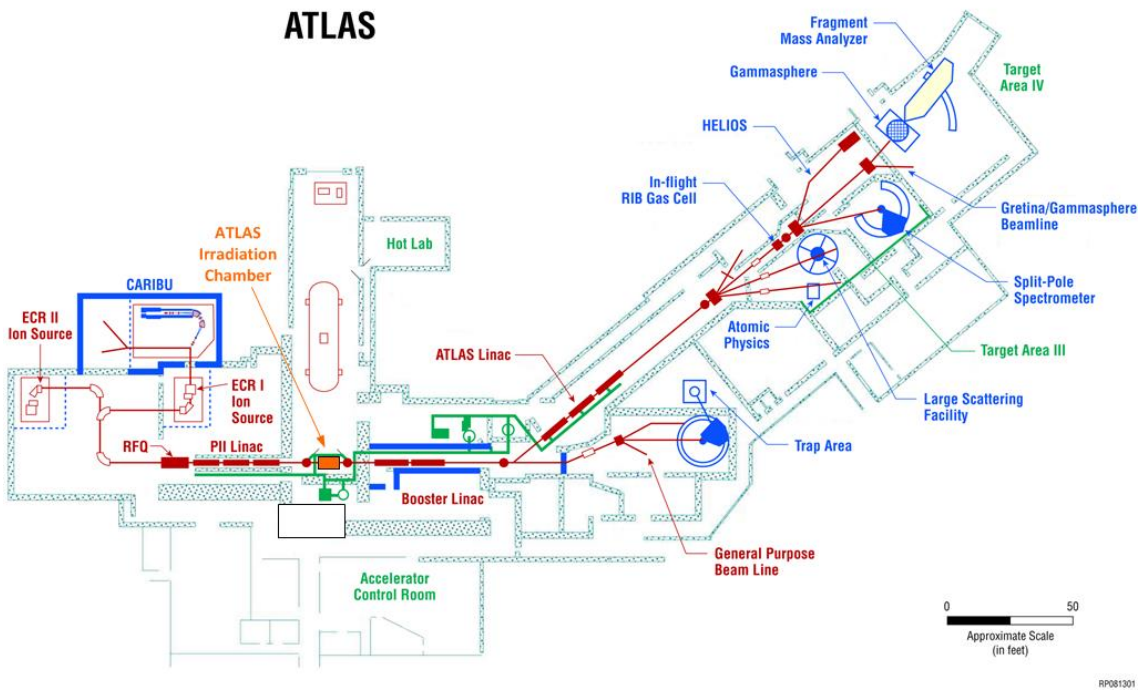

Figure 3.1 The location of the ion irradiation chamber shown in the ATLAS floorplan

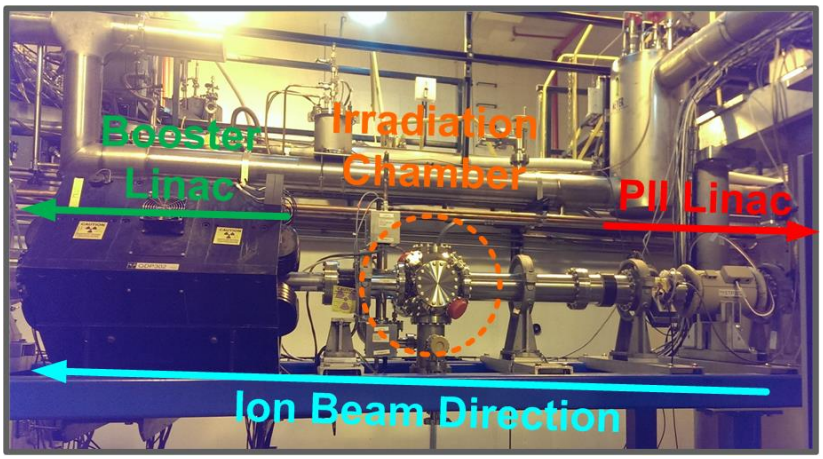

Figure 3.2 The ion irradiation chamber established between the PII linac and the booster linac at ATLAS

3.1 Design of the Ion Irradiation Stage for ATLAS

In order to utilize the limited ATLAS beam time efficiently, a new ion irradiation stage was designed. Considering the fact that it takes over 10 hour for the pumping system at ATLAS to have the ion irradiation chamber get sufficient vacuum level (~10^{-6} torr), the new design aims at minimizing pumping-venting times during each beam session. Therefore, a 1-D vertical manipulator was used to establish a multi-story ion irradiation stage, as shown in Figure 3.3.

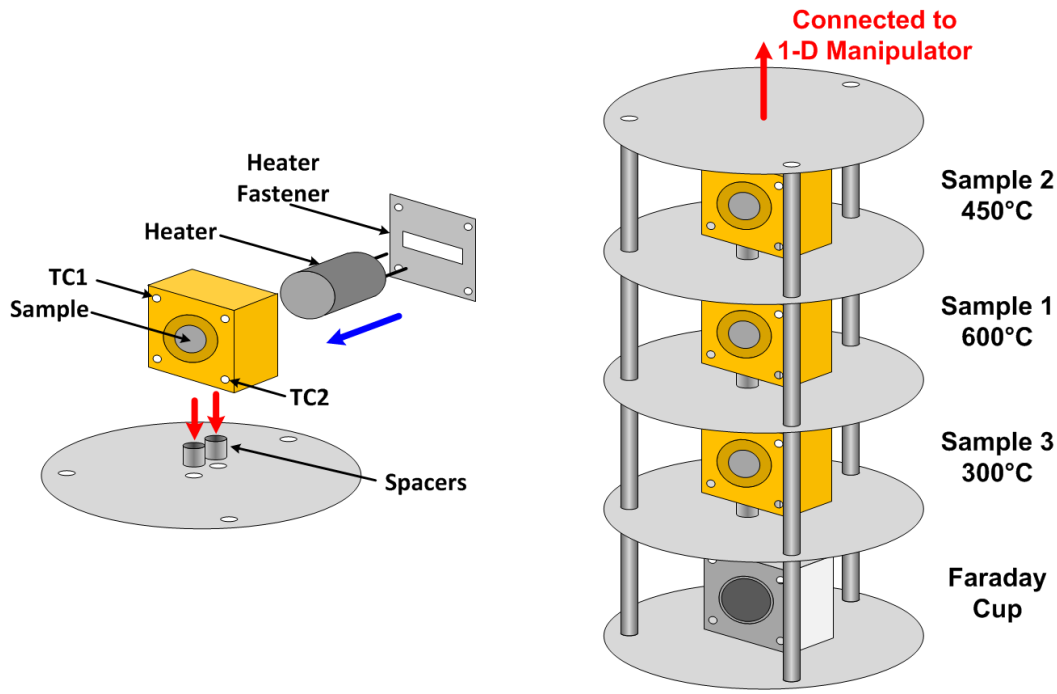

Figure 3.3 Multi-story design of irradiation stage with independent heating systems

As discussed before, U_3Si_2 samples were planned to be irradiated at various temperatures at ATLAS. In order to irradiate those samples at different temperature without venting the chamber and changing the sample, each story has its independent heating system. The frame of the irradiation stage is made of austenitic steels. On each story, the sample is mounted on a copper block using high-temperature high-vacuum capable silver paste. Inside the copper block, a molybdenum-clad cartridge heater that is capable of heating up to 1200°C in ultra-high vacuum is embedded. The copper is selected to minimize the temperature difference. The entire copper block with the sample and the heater is mounted on a 316 steel plate. Two eighth-inches 316 steel spacers are put between the block and the plate to insulate the heat. Three stories of those structures are serially connected together with another story containing a Faraday cup, which is used to tune the ion beam. 316 steel spacers were used between plates to ensure the spacing. The entire four-story irradiation stage is then connected to a 1-D manipulator.

On each copper block, two K-type thermocouples are mounted on diagonal positions (see Figure 3.4) to provide redundancy. This design can support up to 4 thermocouples per sample if necessary. The Faraday cup has a bias voltage input wire and a readout signal wire. Meanwhile, each cartridge heater needs two power supply wires that can handle up to 15 V voltage and 20 A electric current. The heaters can work in both AC and DC mode. In order to reduce the feedthrough requirement, DC power supply was selected so that the ground can be shared by three heaters. Therefore, six pairs of K-type thermocouples, two wires for the Faraday cup, and four power supply cords need to be connected through feedthroughs. Two 2.75'' CF flange thermocouple feedthroughs, each of which has four pair of

K-type ports, were used. The Faraday cup wires were also connected to the thermocouple feedthrough, occupying a pair of thermocouple signals. Besides, a 4.5'' CF flange 5-contact power supply feedthrough was used for the power supply wires, as shown in Figure 3.5.

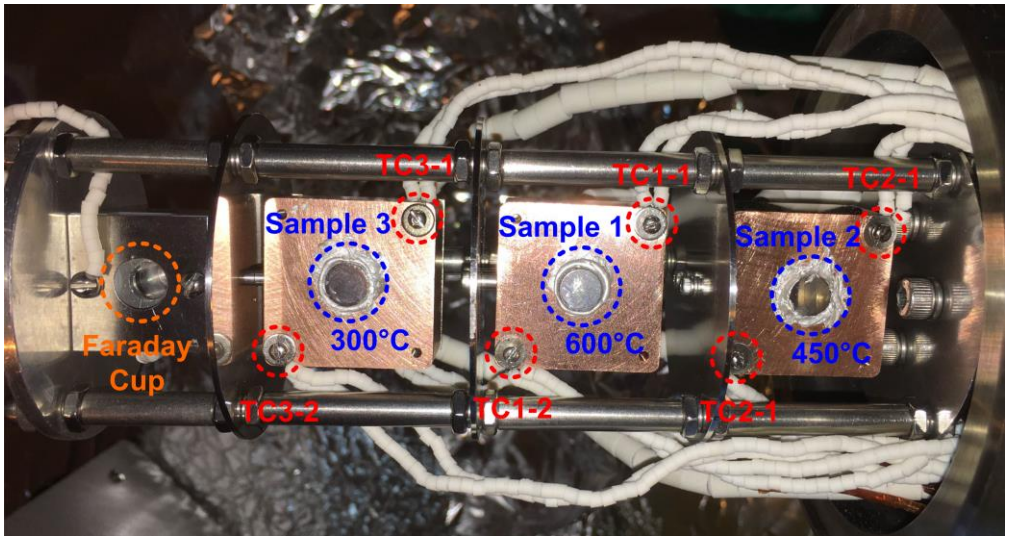

Figure 3.4 Three U_3Si_2 samples mounted on the irradiation stage

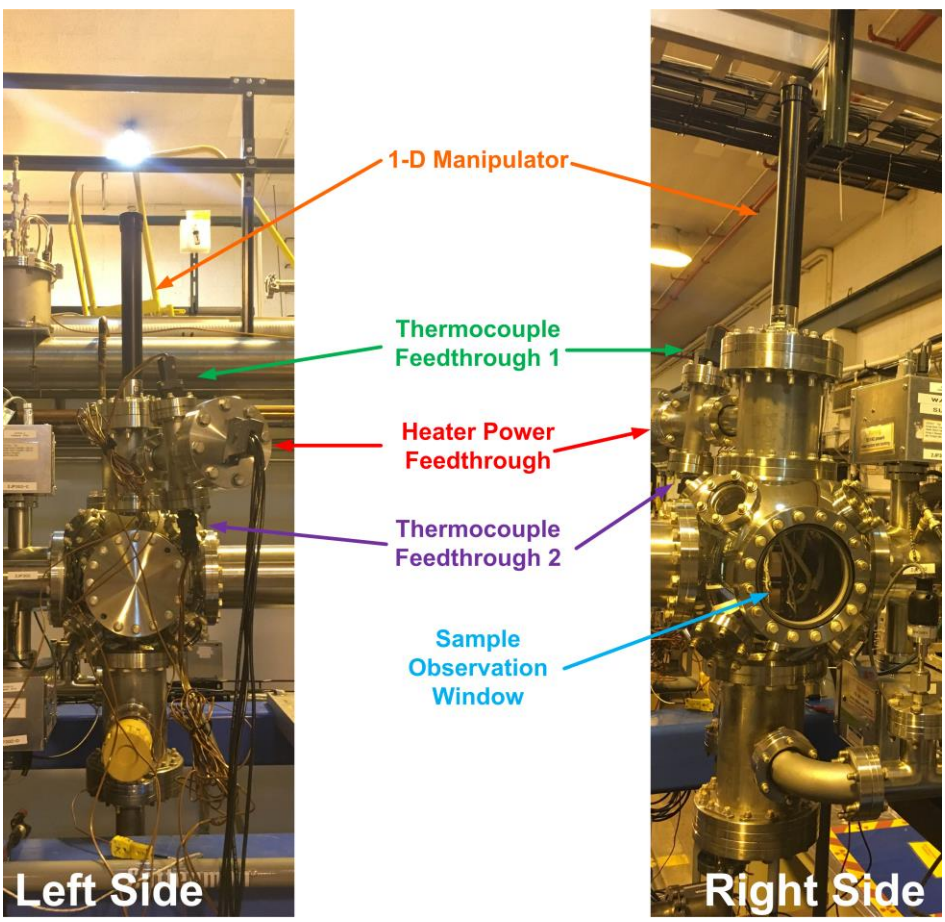

Figure 3.5 The design of the irradiation stage, notice the three feedthroughs

As the U_3Si_2 samples experienced up to 600°C high temperature in the chamber, all the wires inside the chamber are naked copper wires insulated by ceramic beads. In order to precisely control the irradiation temperatures of those samples, a DC power supply with a PID temperature controller is used (see Figure 3.6).

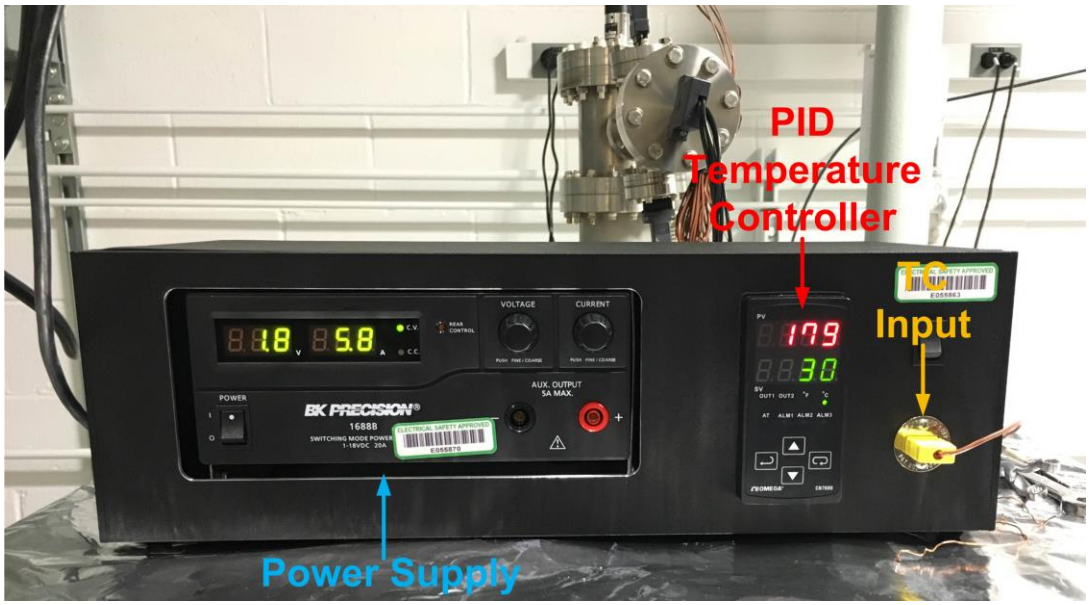

Figure 3.6 Power supply with PID temperature control

3.2 Ion Irradiation Experiment

The first ATLAS ion irradiation experiment was done for U_3Si_2 as planned in Section 1.3. Three different irradiation temperatures, 300°C, 450°C, and 600°C were used for three sintered U_3Si_2 samples. The beam current was maintained at around 100 particle nA. The beam profile was assumed to have a 2-D Gaussian shape. The beam profiler before the ion irradiation chamber showed that the σ_y is approximately twice as σ_x. The σ_y at the ion irradiation chamber was measure to be 4.4 mm using the Faraday cup and the 1-D manipulator. Thus, the peak equivalent burnup is as high as 22%, whereas the edge area on the sample has an equivalent burnup as low as 4%. Therefore, the targets listed in Section 1.3 were achieved. Meanwhile, the temperature control system proved to work properly. The entire temperature history of samples irradiated at 600°C and 300°C is illustrated in Figure 3.7. At 300°C, the radiation heating effect is still prominent. Each time the beam was blocked for fine tuning and returned, there was an approximately 30°C fluctuation. At 600°C, the radiation heating effect is negligible, the fluctuation is within 10°C throughout the irradiation.

3.3 Prospective Post-Irradiation Examination Efforts

Multiple material characterization techniques will be used to investigate the ATLAS irradiated U_3Si_2 specimens. First of all, FIB will be used to mill the irradiation surface to reveal cross section so that large fission gas bubbles can be identified in SEM images. Then, TEM samples will also be prepared by FIB for later TEM investigation. As the dpa values are very high in those samples, the TEM specimens can first be used to confirm the fact that U_3Si_2 maintains crystalline at LWR temperatures. In addition, the morphology of intragranular and intergranular bubbles will also be well studied, while the size distribution and number density will also be measured based on the TEM images. Micro-cube samples will also be prepared from those irradiated sample for synchrotron X-ray investigations to reveal both the phase evolution as well as the strain induced by fission gas bubbles.

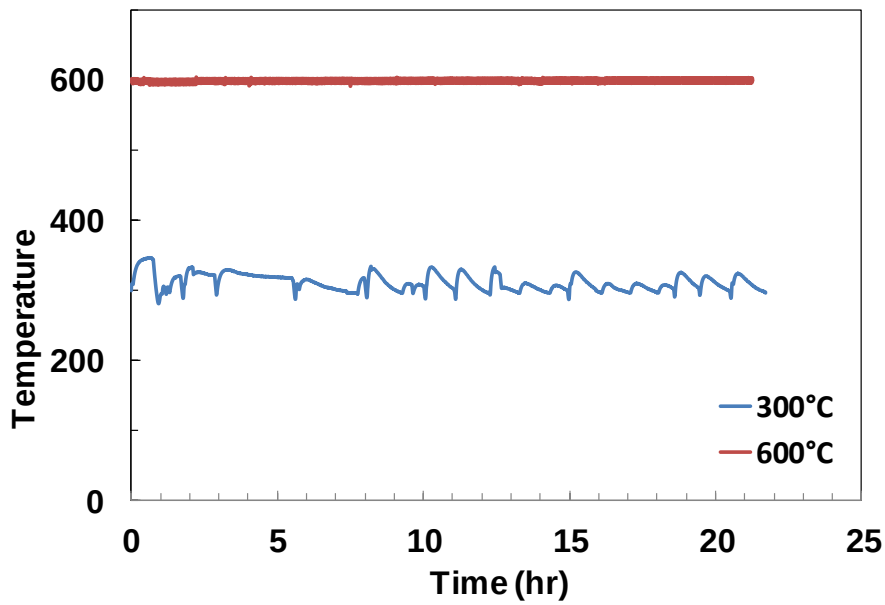

Figure 3.7 Temperature record of ion irradiation sample

4. Conclusions

In this study, a systematic ion irradiation plan was made so as to investigate the amorphization threshold and fission gas behavior in U_3Si_2 at LWR conditions by using heavy ion irradiation to simulate in-pile neutron irradaition. The irradiation plan includes both *in situ* TEM observation and ex-situ high-energy Xe implantation. *In situ* TEM Kr ion irradiation experiments at room temperature show that recently developed sintered U_3Si_2 pellet has similar amorphization behavior to arc-melted U_3Si_2 ingots. Meanwhile, a multi-story ion irradiation stage was desinged and establish for the ATLAS high-energy ion irradiation experiments at various elevated temperature. The sintered U_3Si_2 specimens were successfully irradiated by 80 MeV Xe ions at 300°C, 450°C, and 600°C. The rest of the ion irradiation experiments and corresponding PIE characterization will be performed in the next fiscal year.

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