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In-Cell Thermal Creep Frame Capability: Out-of-Cell Construction and Subsized Sample Testing

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ABSTRACT, SUMMARY, AND FOREWORD

The accelerated qualification of in-core materials at relevant reactor conditions is necessary to rapidly develop performance data and enable licensing processes critical for advanced nuclear reactor demonstrations. In order to take advantage of higher efficiencies, advanced reactor designs will operate at significantly higher temperatures relative to the traditional light-water reactor fleet. This increase in steady-state operating temperature has a significant impact on the long-term mechanical behavior of reactor components under constant load, making the study of the geometrical and mechanical integrity of in-core components essential during typical and off-normal conditions. These higher temperatures result in aggressive environments which enhance thermally driven processes such as creep, and increase the risk of creep failures. Existing American Society of Mechanical Engineers (ASME) Section III Div 5 materials often have inadequate creep lifetimes to perform the necessary structural functions under reactor relevant conditions. Due to this, there is significant need for the capability to assess constant load thermal creep behavior of neutron irradiated advanced reactor materials.

Another challenge in qualifying in-core materials is the limited availability of neutron irradiated bulk material or samples for thermal creep testing. To respond to the challenges of using American Society for Testing and Materials (ASTM) standard sized samples, it is necessary to address the feasibility of using subsized specimen geometries for thermal creep behavior of advanced reactor materials. Therefore, the U.S. Department of Energy Office of Nuclear Energy (DOE-NE) National Reactor Innovation Center (NRIC) prioritizes the development of a thermal creep testing infrastructure for multiple subsize specimens to accelerate the demonstration and deployment of advanced reactor concepts. This report describes the activities for the construction of a thermal creep testing capability at Idaho National Laboratory (INL). The overall project consists of conceptual design, out-of-cell demonstration, and in-cell demonstration phases.

During fiscal year (FY)-2022, the team has procured the materials necessary to fabricate the conceptual design presented within the (FY)-2021 report. This includes the fabrication of clevises to hold multiple thicknesses of subsized tensile samples, procurement of furnaces to heat tensile samples during experimentation, and construction of structural frames to hold each of the box furnaces. The fabrication of each aspect of the creep frame is in accordance with the original design space identified for the operational capacity of the creep frame for different advanced reactor relevant materials with the consideration of the feasible operation in the hot cell at the Fuels and Applied Science Building (FASB) at INL.

Initial testing has also been performed to compare the behavior of subsized specimens using the clevises developed for use in the new creep frames with previous creep testing performed at INL for Alloy 617 using ASTM Standard sized creep specimens. Creep tests performed at 800 °C, 80 MPa were done using the same type of creep frames as those used for the ASTM Standard sized samples for two different subsized sample thicknesses: 0.75 mm thick and 1.0 mm thick. The behavior of these subsized samples will be used to verify the appropriate operation of our newly constructed creep frame.

For FY-2023, the out-of-cell demonstration and final installation of the in-cell creep frame is planned.

ACKNOWLEDGEMENTS

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ACRONYMS

ASTM	American Society for Testing and Materials
ASME	American Society of Mechanical Engineers
ATS	Applied Test System
DOE	U.S. Department of Energy
DOE-NE	U.S. Department of Energy Office of Nuclear Energy
FASB	Fuels and Applied Science Building
FY	fiscal year
HFEF	Hot Fuels Examination Facility
IASCC	Irradiation Assisted Stress Corrosion Cracking
INL	Idaho National Laboratory
LMP	Larson-Miller parameter
MFC	Materials and Fuels Complex
NRIC	National Reactor Innovation Center
SEM	Scanning Electron Microscope

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In-Cell Thermal Creep Frame Capability: Out-of-Cell Construction and Subsized Sample Testing

1. INTRODUCTION

The U.S. Department of Energy Office of Nuclear Energy (DOE-NE) National Reactor Innovation Center (NRIC) has funded the development of a thermal creep testing infrastructure for subsized, irradiated specimens to accelerate the demonstration and deployment of advanced reactor concepts. This report describes the activities for the construction of a thermal creep testing capability at Idaho National Laboratory (INL) to allow for post-irradiation creep testing to be performed within INL hot cells. The overall project consists of conceptual design, out-of-cell demonstration, and in-cell demonstration phases.

The current momentum of the advanced reactor development requires accelerated materials qualification for licensing and final deployment of these reactors. Since these reactors operate at temperatures above 500°C, the core internal components—including the fuel cladding alloys—must exhibit excellent geometrical stability and mechanical integrity for increased long-term operation. Therefore, the high-temperature and long-term mechanical integrity of in-core components must be assessed to progress materials qualification for advanced nuclear reactors.

For temperatures of relevance to advanced reactor designs, materials performance is governed primarily by thermal creep. As depicted in Figure 1, stainless steels and high strength nickel alloys are operating well within the creep regime at temperatures greater than 500 °C [1]. Therefore, the thermal creep advanced reactor materials must be assessed for qualification purposes. For example, the Fluoride-salt-cooled, High-temperature Reactor being developed by Kairos Power will be utilizing 316 stainless steel as a structural material and will have a reactor outlet temperature of 650 °C. This material/temperature condition is clearly within the regime where thermal creep behavior dominates. Determination of engineering level creep parameters, such as the onset of tertiary creep, fracture time, and Larson-Miller parameter (LMP) is critical, as well as the steady-state thermal creep parameters to ensure safe reactor operation. Neutron irradiation will also have a large effect on the mechanical properties of structural components, including changes to the material yield stress, capacity for uniform elongation, embrittlement, and many others. Microstructural damage due to incident neutrons as well as transmutation products during the course of irradiation can dramatically impact the reliable lifetime of reactor components, and may have an appreciable effect on creep behavior. Understanding the role neutron damage plays in the creep behavior of structural materials will be an important aspect of ensuring safe operation throughout the lifetime of the reactor.

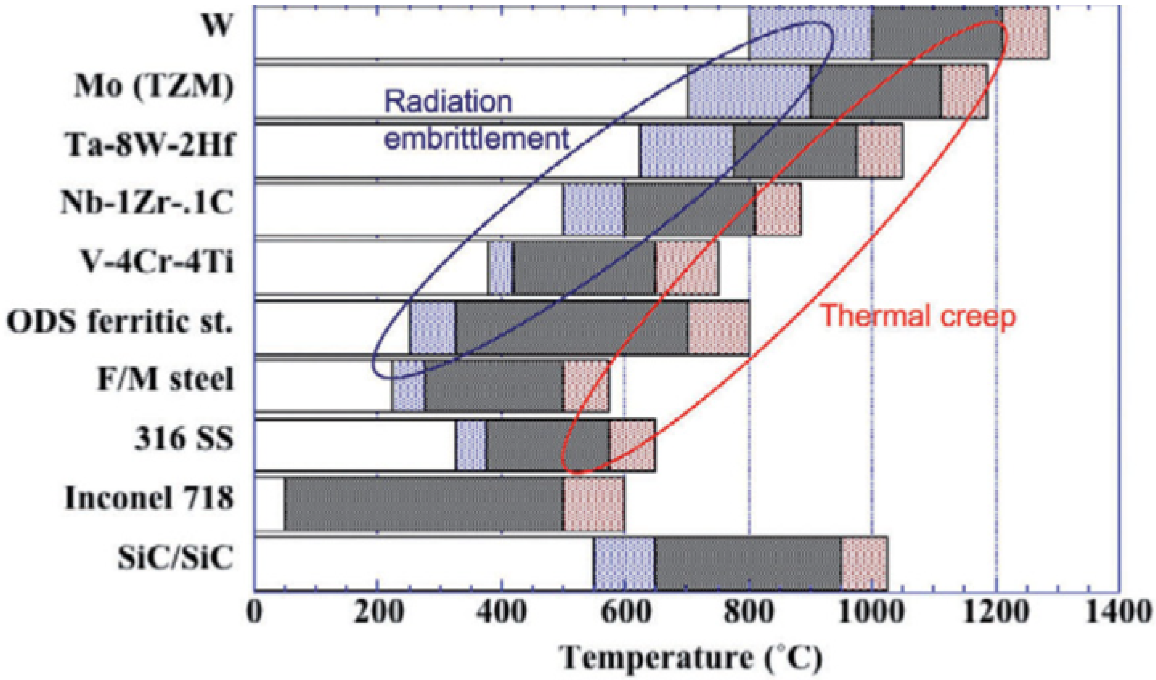

Figure 1: Operating temperature window of structural materials in advanced reactor concepts.

Conventional thermal creep tests are performed based on the American Society for Testing and Materials (ASTM) International Standard. While the ASTM Standard is developed to generate reliable and reproducible data, the large specimen geometry results in several challenges for testing neutron irradiated material. The large sample size inherently increases the footprint of the testing equipment required. This is difficult in specialized testing facilities which require testing to be performed within a hot cell, which will be limited in physical space. This space limitation will typically prohibit the ability to run multiple samples simultaneously. This is particularly important when it comes to thermal creep testing since tests can run continuously for thousands of hours. Significant gains in testing efficiency can be made if simultaneous testing of multiple samples can be accomplished. Additionally, ASTM Standard creep specimens are prohibitively large to fit within typical irradiation capsules. The size of one sample alone could eliminate certain testing positions within a reactor and will limit the total number of samples which can be irradiated to a unique set of conditions (combination of material, temperature, dose, etc.). In order to produce enough samples for effective post-irradiation examination testing within an appropriate timeframe and budget, significantly smaller samples are typically irradiated, as shown in Figure 2.

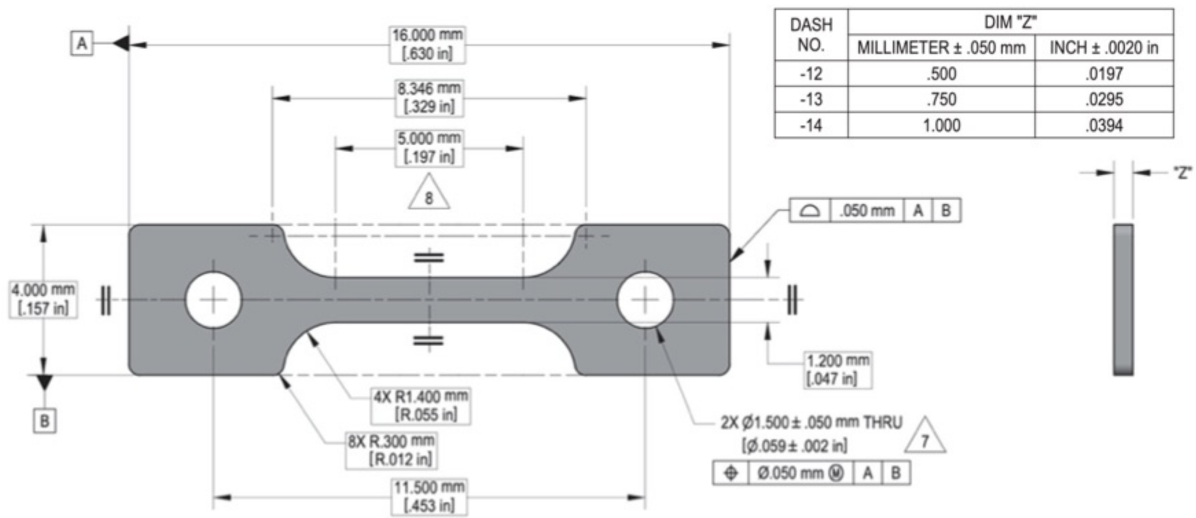

Figure 2: Subsize specimen design (INL drawing: DRW-606738).

In order to generate reliable thermal creep data from these subsized tensile specimens, specialized infrastructure beyond what is available for testing ASTM Standard sized specimens is required. A facility capable of performing thermal creep tests on flat subsized specimens is needed to address the challenges of ASTM Standard sized specimen testing. To overcome infrastructural challenges, NRIC has supported the construction of hot cell creep frames located in facilities at INL.

This report describes the activities related to the construction of an in-cell thermal creep test facility for small-scale specimens in the irradiation-assisted stress-corrosion cracking (IASCC) hot cell located at the Fuels and Applied Science Building (FASB) at INL. The whole project includes three stages: (1) conceptual design; (2) out-of-cell demonstration tests; and (3) in-cell demonstration testing. During the current fiscal year (FY)-2022, materials and equipment required to construct the conceptual design outlined in a previous report [2] were procured. Frames for conducting out-of-cell testing have been constructed, and testing plans have been developed to assess the operation of the newly constructed frames. Preliminary testing of flat subsized tensile specimens on ASTM creep frames has also been completed for two sample thicknesses: 0.75 mm and 1.0 mm.

2. PROCUREMENT AND FABRICATION OF CREEP FRAME COMPONENTS FOR OUT-OF-CELL TESTING

The procurement and fabrication of components necessary for out-of-cell testing of the creep frame were based on the conceptual designs reported previously [2]. Of particular importance for out-of-cell were the following requirements:

- Ability to perform creep tests in the temperature range of 600-800 °C
- Ability to control testing temperatures within ±5 °C
- Ability to test subsized specimens
- Small footprint of the test frame to ensure fit within limited hot cell space

These requirements were fulfilled primarily through the procurement of three MTS box furnaces which can be loaded into independent frames and the fabrication of clevis grips which were made to specifically hold different thicknesses of flat tensile samples (shown in Figure 2).

2.1 Sample Heating for Out-of-Cell Creep Testing

Three Series 653 high-temperature clamshell box furnaces were purchased and delivered to INL. Each furnace has three zones to ensure adequate control of the temperature surrounding tensile samples during the course of the creep test. Figure 3 shows an example of the furnace procured by INL.

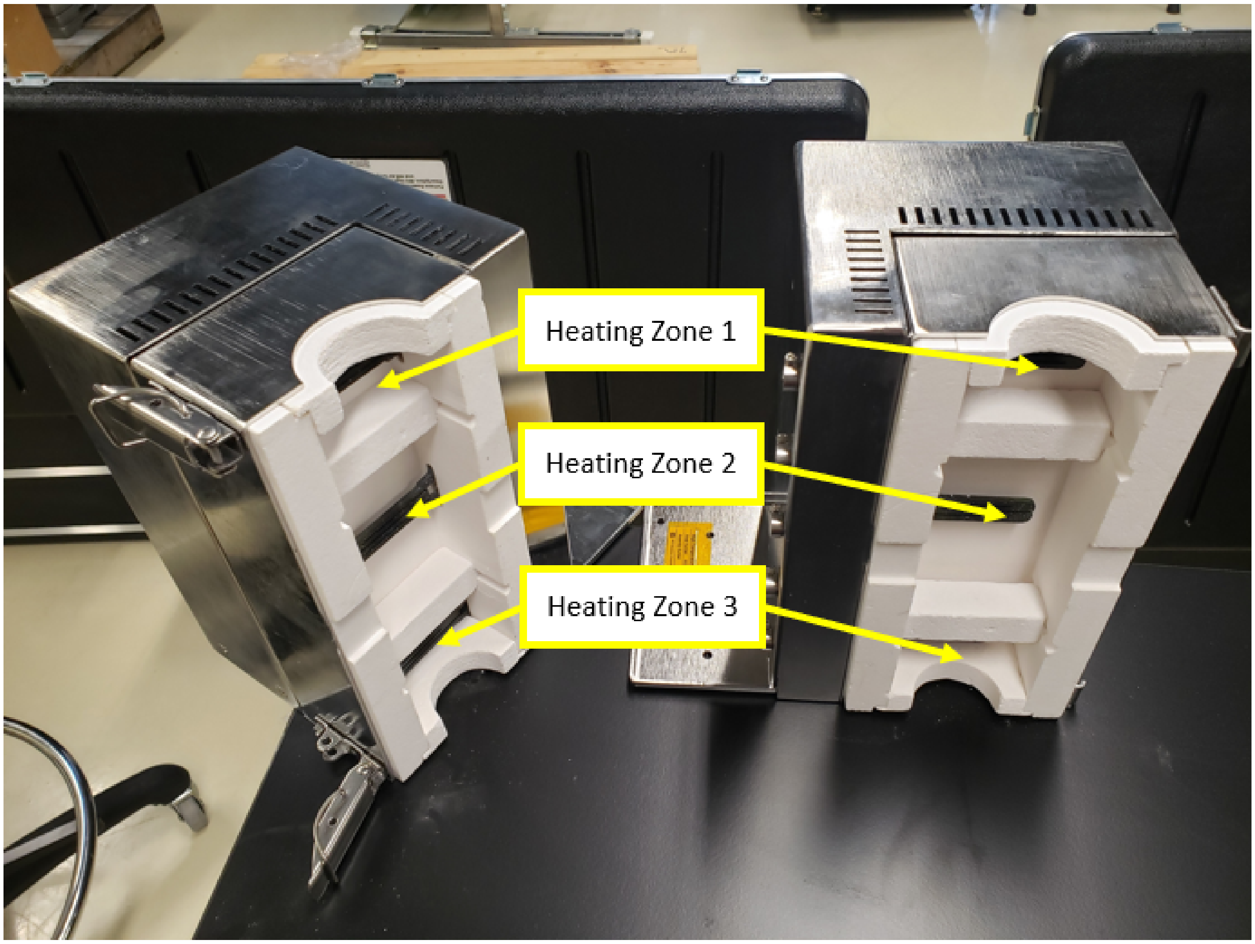

Figure 3: MTS Series 653 high-temperature furnace.

The furnaces have a minimum stable temperature of 100 °C and a maximum reported stable temperature of 1400 °C while no sample is loaded within the furnace. However, these operational temperatures will depend on the sample and clevises located within the furnace, meaning that an actual creep testing temperature of 1400 °C will not be possible. Using MTS 680 low cycle fatigue grips, typical maximum operating temperature is 1050 °C with a 75 minute ramping time to reach maximum temperature [3].

A furnace bake-out routine has been initiated on all furnaces to verify initial functionality and determine a maximum operating temperature while utilizing the custom grips fabricated for this creep frame. A ramp and hold routine similar to that shown in Table 1 will be used to assess the functionality outlined above. This is a projected bake-out routine and is subject to change at the discretion of the principal investigator based on the behavior of the system or the sample which is currently being tested. Since the primary temperature range for testing was determined to be 600-800

°C in the conceptual design of the creep frame, particular emphasis will be made to determine furnace operation within this temperature range. Temperature will be recorded for all three zones of the furnace (e.g., top, middle, bottom) to determine any axial temperature gradients. The bake-out routine is meant to be used as a preliminary verification of the creep frame set-up. Recording of temperatures for each zone in the furnace will be verified using an instrumented specimen, which has been demonstrated as a robust technique in the hot cell at the Materials and Fuels Complex (MFC) Hot Fuels Examination Facility (HFEF). This procedure will be adapted to coincide with the newly built frames. For each furnace zone, a constant temperature offset is determined at the desired testing temperature. Then, these offsets are used to set the temperature of the zone controllers for non-instrumented samples. The verification of temperatures and axial temperature gradients will include significantly longer times at the discretion of the principal investigator. This calibration step will also be used to determine temperature variance during steady-state operation.

Table 1. Temperature routine to determine functionality of MTS Series 653 high-temperature furnace for creep frame applications.

Target Temperature [°C]	Ramp Time [min]	Hold Time [min]
100	10	20
200	10	20
300	10	20
400	10	20
500	10	20
550	10	20
600	10	60
650	10	20
700	10	60
750	10	20
800	10	60
850	10	20
900	10	20
950	10	20
1000	10	60
MAX	—	Overnight

Operation of the furnaces and newly fabricated load frames will follow a similar research and development test control plan which is already in place at INL for the tensile testing of reactor materials [4]. Modifications to the test control plan will be made to reflect the use of subsized specimens and any differences between the newly fabricated frames and creep testing frames currently in operation at INL. The design from the conceptual phase for a single MTS furnace set-up is shown in Figure 4.

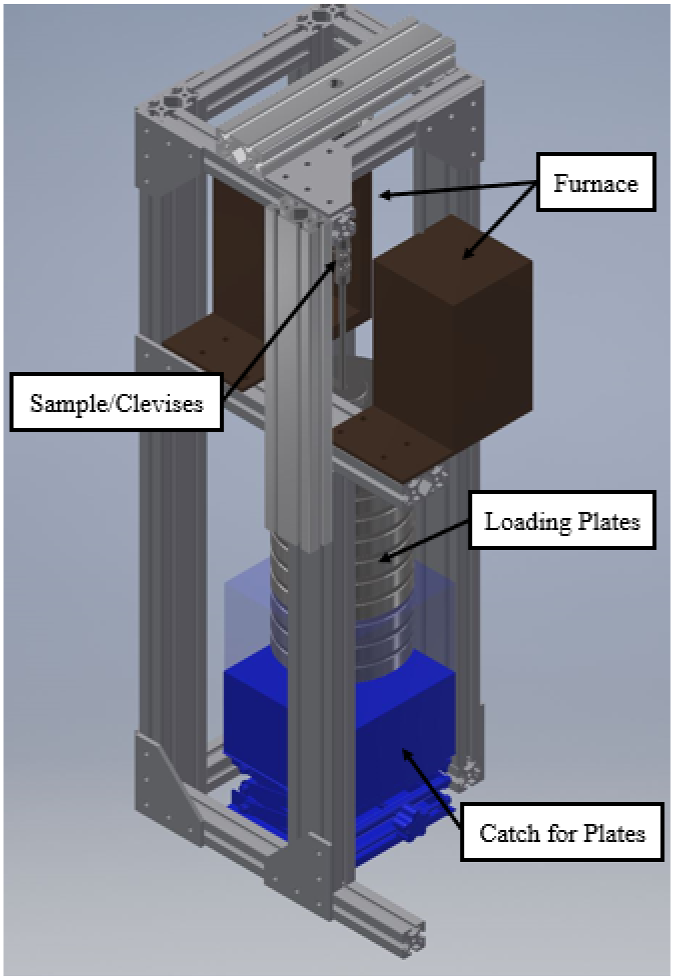

Figure 4: Computer aided design drawing of single furnace frame set-up concept for new INL creep frame construction.

Figure 5 shows three constructed creep frames based on the design shown in Figure 4. One of the three creep frames (left) is loaded with the MTS Series 653 high-temperature furnace and temperature controller, staging for bake-out procedure.

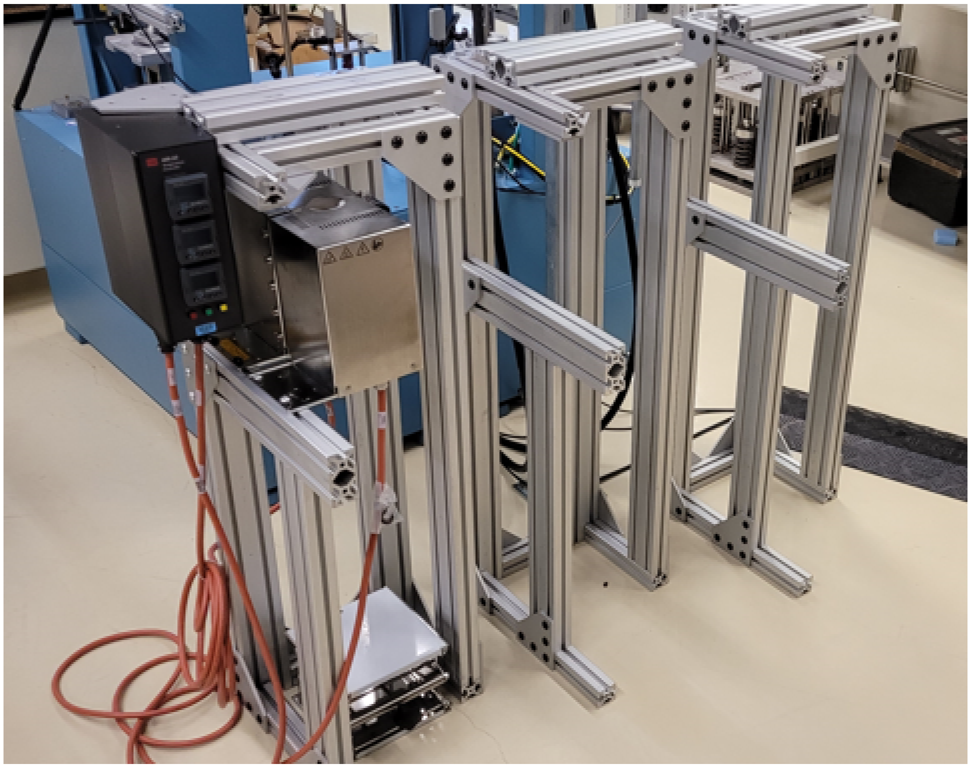

Figure 5: Newly constructed creep frames at INL to hold MTS Series 653 high-temperature 3-zone furnaces and temperature controllers.

Figure 6 shows the relative size of the newly constructed creep frames with the Applied Test Systems (ATS) creep frame system which has been used extensively at INL to perform creep tests on ASTM Standard size samples. It can be seen from Figure 6 that the newly constructed creep frames are significantly smaller than the ATS frames, allowing INL to fit several of the new frames within the FASB utility cell.

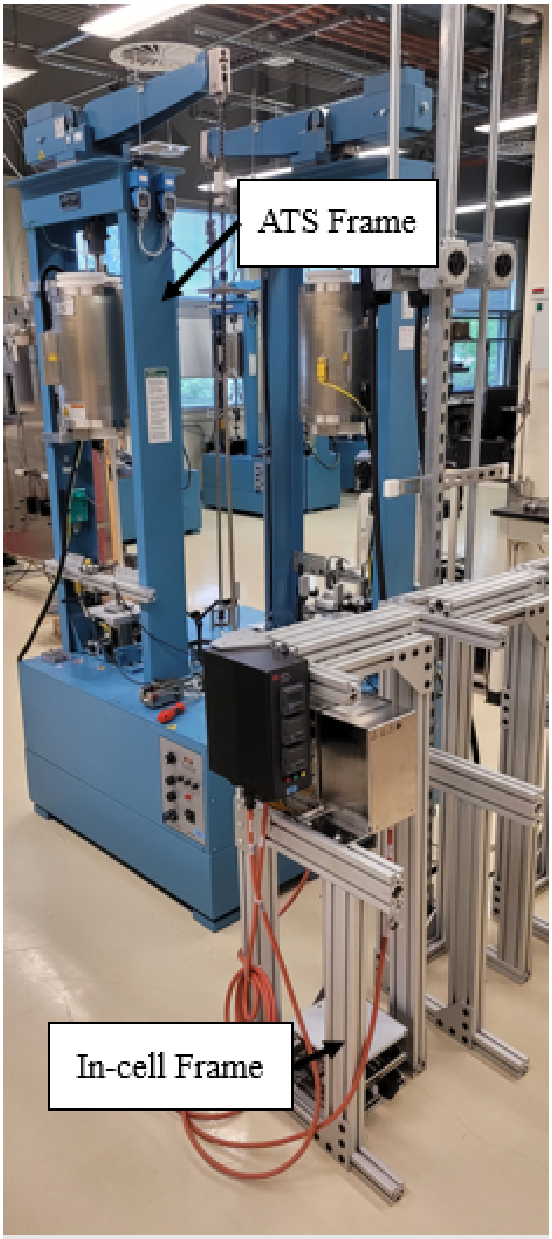

Figure 6: Size comparison between ATS creep frame and newly constructed creep frame for in-cell use.

In anticipation of this newly constructed creep frame moving inside the utility hot cell at FASB, the Scanning Electron Microscope (SEM) which was previously contained in the utility cell has been removed. Figure 7 shows the inside of the utility cell before (left) and after (right) the removal of the SEM to make room for the newly constructed creep frames.

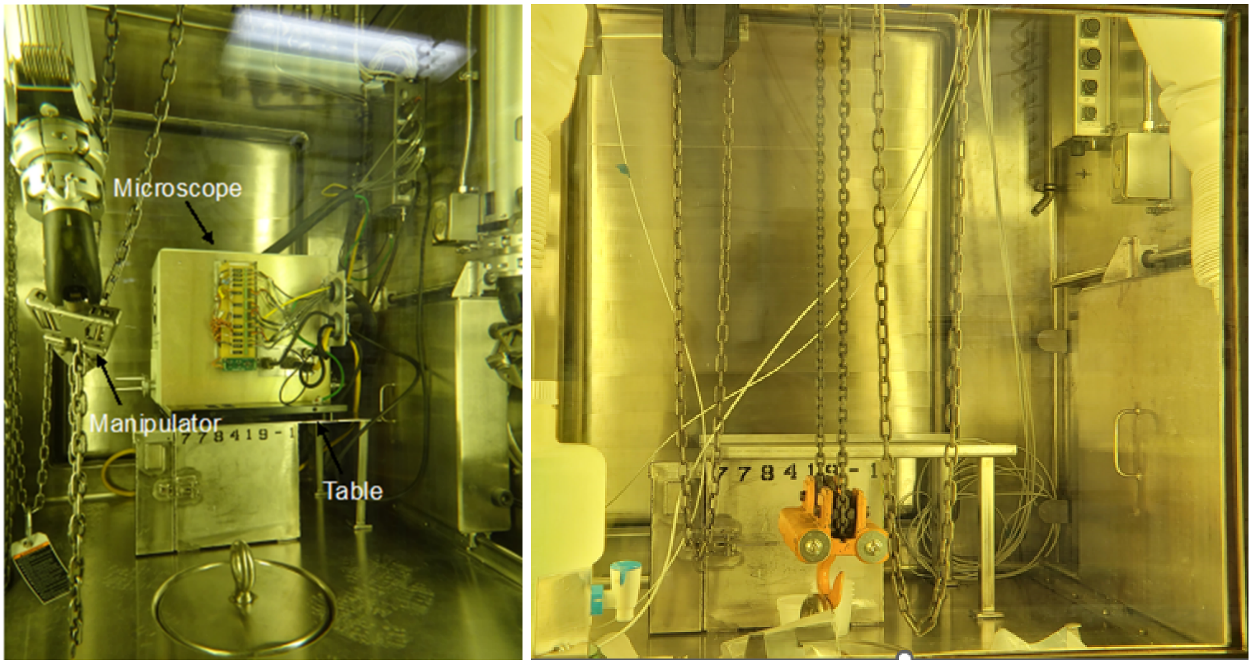

Figure 7: FASB utility cell before (left) and after (right) removal of the SEM to make room for the newly constructed creep frames.

2.2 Load Application for Out-of-Cell Creep Testing

For load application to the flat tensile samples of the type shown in Figure 2, INL has developed a set of custom drop-in fixtures which can be made to hold varying thicknesses of flat subsized samples: 0.5 mm, 0.75 mm, and 1.0 mm. Clevis grips for these three specimen thicknesses have been fabricated using heat treated Alloy 718, which was selected due to its high-temperature mechanical properties. This will minimize unwanted deformation in the clevis structure and isolate deformation to the creep specimens while at high temperatures. Fabricated clevises are shown in Figure 8. Each clevis set is specifically designed for a single sample thickness, to ensure that the line of uniaxial tension passes through the center of the sample. These clevises were also designed such that they will fit within the MTS box furnaces detailed in section 2.1.

Figure 8: Unloaded (left) and loaded (right) flat subsized specimen clevises for newly constructed creep frame.

Through the development of clevises with the capability to test multiple thicknesses of samples, the newly built creep frames will be able to test a small variety of sample geometries and will unlock additional flexibility within neutron irradiation campaigns. For irradiation testing programs utilizing the thinner flat tensile sample geometry, a larger number of samples can be irradiated and tested, resulting in more testing data and accelerating the pathway toward material qualification.

3. SUBSIZED SPECIMEN FEASIBILITY TESTING

In order to assess the functionality of this new creep frame capability, it is necessary to compare its operation to expected creep results. This is being performed in two stages. The first stage consisted of comparing the creep behavior of flat subsized specimens (of the same dimension which will be used on the newly fabricated creep frame) using the clevises outlined in section 2.2 with ASTM Standard size round tensile samples. Extensive work was performed at INL on Alloy 617, which was chosen as the baseline material for this comparison. The subsized testing was performed on the same type of system which was used to collect the ASTM Standard size sample creep behavior, the only difference being the clevises used and the tested sample geometry.

The second stage will consist of comparing the subsized specimen behavior of samples tested on the larger ATS frames with samples tested on the newly constructed creep frames at the same temperature/load condition.

3.1 Alloy 617 ASTM Standard Size Sample Creep Rupture Testing

Work performed at INL for creep behavior of Alloy 617 was done in support of the development of the ASME Alloy 617 Code Cases through the INL Advanced Reactor Technologies [5]. Cylindrical creep specimens were machined in accordance with standard ASTM E139 [6]. Samples had a nominal 6.35 mm reduced section diameter and a gauge length of 32 mm. A drawing of the ASTM Standard size sample is shown in Figure 9.

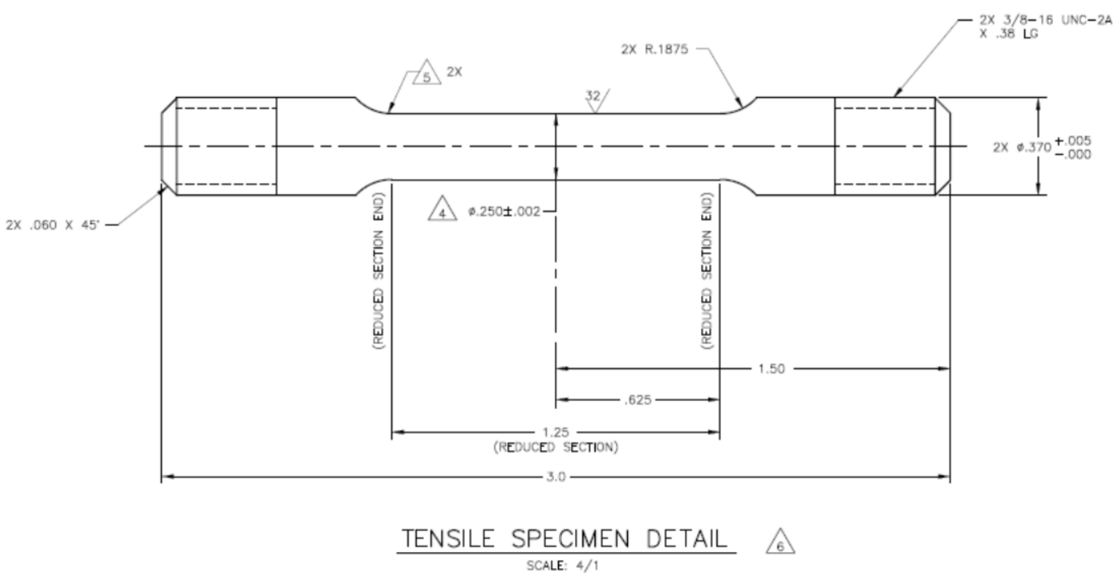

Figure 9: Example of ASTM Standard size round tensile specimen used in creep testing of Alloy 617 at INL.

Creep tests were performed using a dead weight loading configuration and testing temperatures were measured using a type K thermocouple near the gauge section of the creep specimen to ensure the temperature was controlled to within ±3 °C of the target test temperature. Dual averaging linear variable differential transformer transducers were used to monitor creep strain to a resolution higher than 0.01% strain. The testing condition of 800 °C with a dead weight load of 80 MPa was selected as the creep condition to generate a total elongation as a function of time plot to compare the

ASTM Standard size specimens of Alloy 617 to the subsized specimen geometry. The primary metric of comparison for these initial tests was the time to rupture, which is shown diagrammatically in Figure 10.

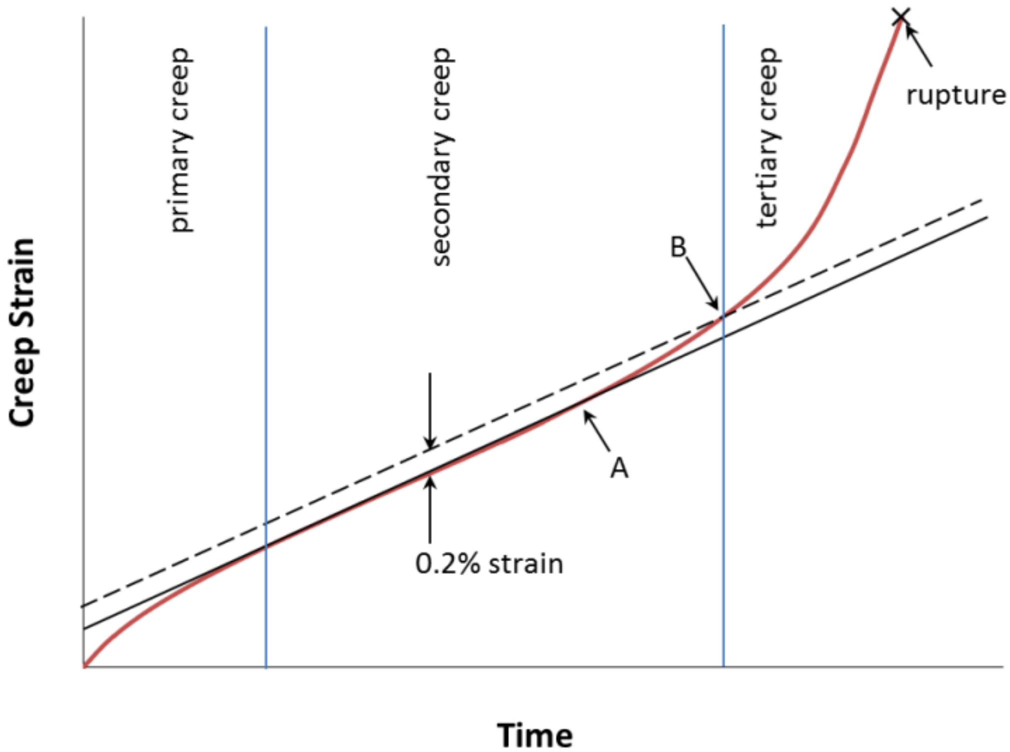

Figure 10: Typical creep curve showing the three stages of creep, minimum creep rate, and 0.2% offset to onset of tertiary creep.

For two separate ASTM Standard size samples tested at 800 °C, 80 MPa the time to rupture was measured to be 1138 hours and 1208 hours respectively. These tests will be referred to as ASTM 1 and ASTM 2.

3.2 Alloy 617 Subsized Sample Creep Rupture Testing

Two subsized samples have been tested on the same type of ATS creep frames as the 617 ASTM Standard size samples. One test was performed using a 0.75 mm thick flat subsized tensile bar and the other test used a 1.0 mm flat subsized tensile bar. Each of these tests utilized the clevises fabricated by INL specifically to hold these subsized specimens. Creep rupture tests were run at 800 °C, 80 MPa to match the conditions used for the ASTM Standard size tests. The results for the time to rupture under these testing conditions are summarized in Table 2.

Table 2. Rupture time at 800 °C, 80 MPa for ASTM Standard size and flat subsized specimens.

Sample	Rupture Time [hours]	% Elongation

ASTM 1	1138	54.4
ASTM 2	1208	53.7
0.75 mm	664	12.7
1.0 mm	1311	13.7

The time to rupture for the 1.0 mm sample aligned well with the previous data collected using ASTM Standard size samples under the same conditions. However, the time to rupture for the 0.75 mm thick subsized sample was half that of the ASTM Standard size samples. It is not possible to draw distinct conclusions based on the limited number of tests performed since creep testing typically shows a large variance in time to rupture and a factor of two difference falls within the error of this type of experiment. Additional testing of the 0.75 mm thick sample type is needed before proper conclusions about its use with our creep frame set-up can be made. A plot showing the creep data for both the ASTM Standard size and flat subsized specimens is shown in Figure 11.

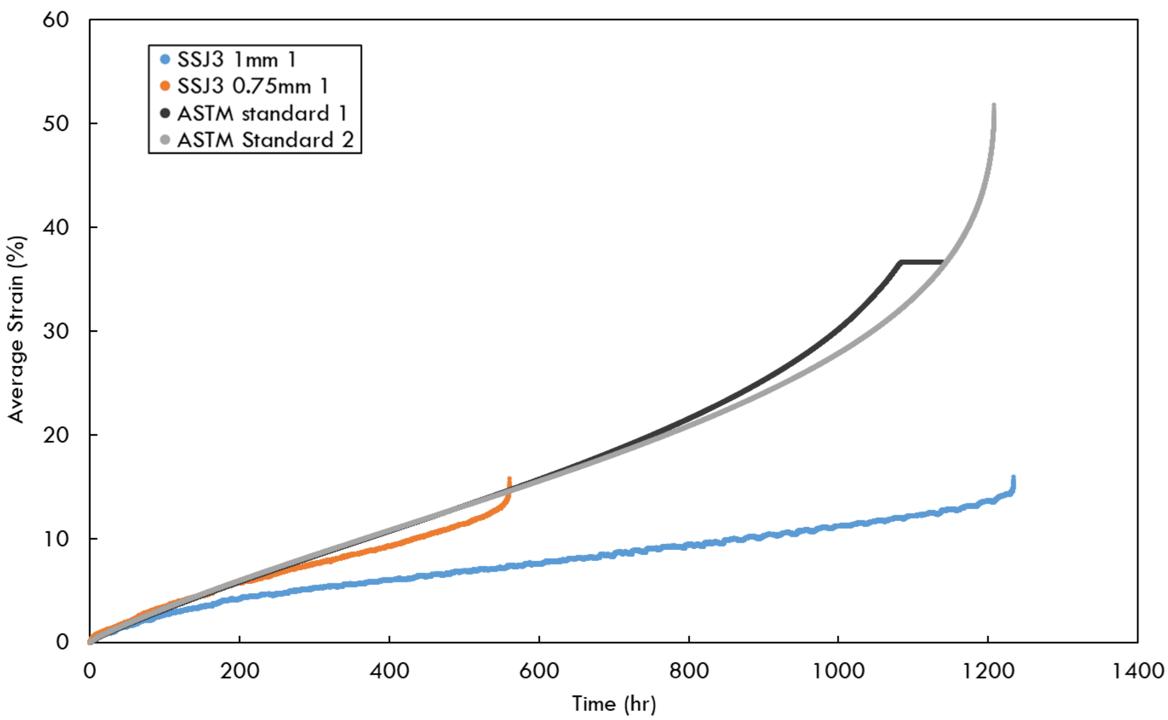

Figure 11: Strain as a function of time for Alloy 617 samples creep tested at 800 °C, 80 MPa.

Optical examination of both subsized specimens shows clear creep damage along the entire length of the gauge section. Visible damage was confined to the gauge section, and did not extend into the loading section of the sample shoulders or the tensile heads, which is to be expected. Optical images of the 1.0 mm thick and 0.75 mm thick flat subsized specimens after conclusion of the creep test are shown in Figure 12 and Figure 13 respectively.

Figure 12: Optical image of 1.0 mm thick flat subsized specimen after creep testing at 800 °C, 80 MPa until creep rupture.

Figure 13: Optical image of 0.75 mm thick flat subsized specimen after creep testing at 800 °C, 80 MPa until creep rupture.

White substance visible in near the sample shoulders and tensile heads is milk of magnesia, which is used in the heads of the grips as an anti-seizing agent.

It was observed that for both of the subsized specimens, the percent of elongation at rupture was significantly lower than the ASTM Standard size samples. It is important to note that the percent of elongation is based on post-test specimen measurement (fracture surfaces are put back together and length measured), not based on the creep curves shown in Figure 11. It is not possible to make a definitive conclusion on this behavior due to the limited data, although this phenomenon will be inspected during subsequent testing of subsized specimens on both the ATS creep frames and the newly constructed creep frames. Upcoming creep testing will include one Alloy 617 sample which is 0.5 mm in thickness and a second sample with a 0.75 mm thickness to duplicate the previous test. Both samples will be run on the larger ATS test frame at 800 °C, 80 MPa, just like the previous set of tests. After bake-out of the newly constructed frames is completed, a 1.0 mm thick Alloy 617 sample will be tested at 800 °C, 80 MPa to be compared with the data already presented here.

4. SUMMARY

During FY-2022 three creep frames to hold MTS Series 653 high-temperature furnaces were constructed out-of-cell to demonstrate functionality in creep testing prior to being moved into the utility cell in FASB. These frames were built according to the conceptual design performed for this project during FY-2021. Testing plans have been developed to assess the operation of the newly constructed frames and to calibrate the temperature zones within the MTS furnaces prior to installation within the utility cell. As part of the verification process for the behavior of the newly constructed creep frames, two creep tests using flat subsized Alloy 617 specimens were performed on ATS creep frames using custom built clevis. The two samples were 0.75 mm and 1.0 mm thick respectively. Samples were tested at 800 °C, 80 MPa and compared with ASTM Standard size samples previously tested at INL. Initial results show that the measured rupture time for the subsized specimens fall within the typical for creep testing, which is encouraging.

During FY-23, the following activities are planned: (a) calibration of the newly constructed creep frames and execution of out-of-cell demonstration experiments; (b) the identification of the facility needs and work controls for the in-cell installation; and (c) the installation of the creep frame in the IASCC utility cell at FASB.

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