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Design and Fabrication of a Temperature-Controlled Fueled Molten Salt Capsule Irradiation Experiment

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I. INTRODUCTION

Although its commercial application may be a decade or more away, liquid-fueled Molten Salt Reactor (MSR) technology has recently attracted increased attention through several demonstration efforts underway in the U.S. and abroad. While most concepts build on the legacy of the Molten Salt Reactor Experiment (MRSE) [1], several new concepts under development propose salt compounds that have never been previously tested. While several efforts are underway across the world to develop irradiation testing capabilities for molten salts [2][3][4], none has demonstrated the ability to test enriched fuel bearing salt.

Idaho National Laboratory (INL) is developing a Molten salt Research Temperature-controlled Irradiation (MRTI) experimental capsule for the Neutron Radiography (NRAD) reactor. The experiment is envisaged to be versatile and able to host a variety of salt-wall material combinations. The first test is designed to contain actinide-bearing chloride salt, which previously has not been subjected to a neutron field. The main objectives of the experiment are to produce irradiated salt samples for Post Irradiation Examination (PIE), to evaluate salt behaviour under irradiation, and to test in-situ sensors and instrumentation.

The final design for the experiment was completed in 2022, and fabrication of the experiment is underway, with a prototype completed in February 2023. The salt is double encapsulated providing insulation and redundancy in the case of leakage. A gas-gap between the inner capsule and the outer container enables tuning of thermal conduction to the NRAD coolant by altering its thickness and its gas-mixture. An inert gas mixture ratio in the gas-gap ensures the configurations reached a targeted average salt temperature of 600°C, while avoiding salt freezing or exceeding material limits. A resistive heater placed within a thermowell controls the temperature of the experiment (both when the reactor is on or off). The selected configurations can contain over 10 cm³ of fuel-bearing chloride salt, which is sufficient for the purposes of the post-irradiation examination (PIE) and provides sufficient volume for two submerged thermocouple probes to

monitor salt temperature. The capsule wall material is Inconel 625 (IN625), and weld qualifications for the assembly have been developed. A glove-box laser weld technique was refined as part of this process to ensure a leak-tight assembly for the salt-bearing assembly and reduce the risks of impurities permeating into the salt.

This paper will provide an overview of the completed final design of the experiment, including schematics of Computer Aided Designed (CAD) model. It will then discuss the recent efforts to fabricate a prototype assembly to demonstrate the manufacturing process and conduct out-of-pile testing. This prototype was loaded with non-fuel bearing salt for simplicity. The next step towards irradiation of the fuel-bearing salt experiment will be highlighted as well.

II. EXPERIMENT OVERVIEW

The irradiation experiment is planned for the Neutron Radiography (NRAD) reactor at INL [5]. The reactor is 'TRIGA-type' reactor with UZrH fuel assemblies organized in a square lattice arrangement and surrounded by graphite blocks. An illustration of the reactor is shown in Figure 1. The MRTI experiment is currently planned for the F-1 position of the NRAD core.

While the irradiation test vehicle is expected to be flexible with the ability to irradiate a variety of salt samples, the current iteration described here has been fine-tuned in order to host an enriched uranium bearing chloride salt sample. The primary goal of the experiment is to provide irradiated salt samples that can be used to study the following during Post Irradiation Examination (PIE):

1. Understand and characterize the radiological source term mechanics of salts (i.e., transport of radionuclide to the plenum vs. precipitation/wall plating vs. remaining in solution).
2. Study the evolution of thermophysical properties with burnup as fission and activation products accumulate in solution in addition to gas bubbles and suspended nanoparticles.

3. Evaluate the impact of irradiation-induced (both from neutron damage and fission product generation). corrosion on materials interfacing with the salt.

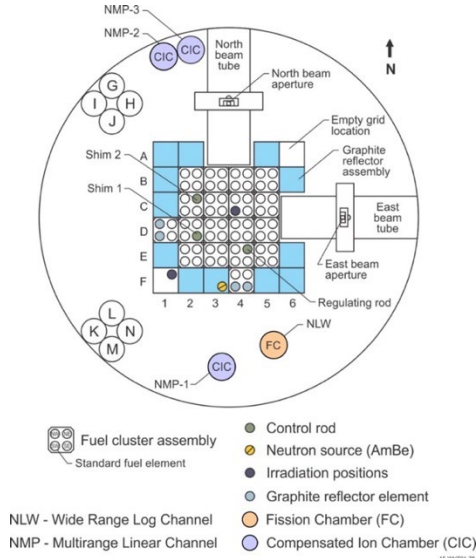


Figure 1. Overview of the NRAD Reactor core layout showing the various positions. Taken from [5].

Previous publications described parametric studies related to the experiment [6], followed by the description of the preliminary design [7]. This work will outline the final design specifications for the experiment along with the fabrication of a prototype assembly (with non-fuel salt). Detailed neutron transport and computation fluid dynamics simulations were conducted to simulate the performance of the experiment under irradiation. These analyses showed that the experiment was shown to be able to reach a targeted fission power density of above 20 W/cm^3 , while staying within thermal limits (limiting salt freezing and maximum heater temperature).

III. EXPERIMENT VEHICLE DESIGN

A. Salt-Bearing Capsule Design

The MRTI capsule design features a thin-walled IN625 tube design with a substantial plenum to account for both initial powder salt packing factor ($\sim 60\%$) and for the accumulation of fission gas and chloride gas during irradiation. The wall thickness is designed to allow for laser-based penetration and gas retrieval of the capsule plenum in PIE. IN625 was chosen as the capsule material for its strength at high temperature, and neutronics benefits over similar alloy Inconel 617. To isolate corrosion effects on IN625 alloy, the capsule is designed such that only IN625 is in contact with the molten salt during irradiation (no dissimilar metals and potential galvanic corrosion effects). Active instrumentation is used to record in-situ temperature in the molten salt and plenum region during irradiation. Type N thermocouples with IN625 sheaths were utilized for this design for high temperature operational range and superior irradiation resistance in comparison to Type K. Additionally an optical fiber

pressure sensor is utilized to measure changes in plenum pressure during irradiation. Due to size constraints regarding the optical fiber sensor, it is connected into the capsule plenum via a stainless-steel pressure extension tube.

Furnace-style heater designs were considered for initial heating of the fuel salt prior to irradiation. It was important during experimentation to maintain the fuel salt in the molten phase in order to avoid effects from salt radiolysis. For depleted uranium or low enriched uranium experiments furnace heaters are a reliable heating method. However, for higher enrichments involved in this experiment the fission heating of the molten salt would be too well insulated from the NRAD coolant water and would result in extreme temperature outside of the desired operational range of the experiment. Therefore a center cartridge heater is utilized in the MRTI capsule design in order to optimize heat transfer out of the capsule once the salt is self-heating. A high temperature Incoloy-800 sheathed 800 W heater is used as the specified heating element in the MRTI capsule. Due to the large plenum volume in the capsule only the bottom section is in contact with the salt and designed to be heated (bottom 7.6 cm). Input wattage in this heated region must be sufficient to fully melt the fuel salt and but only provide small amounts of supplemental power during irradiation. Because the heater sheath is not IN625, it is assembled into an IN625 thermowell in the axial center of the capsule.

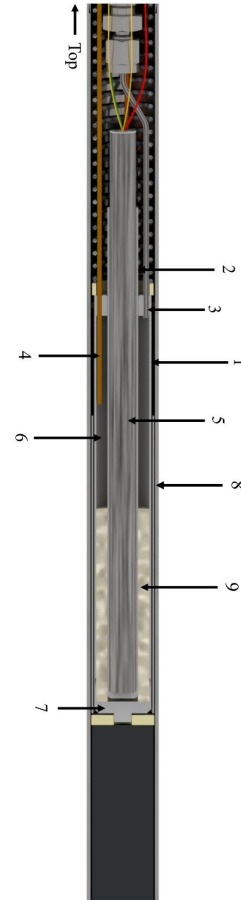


Figure 2. Half section view of MRTI capsule design. 1.) Capsule, 2.) Capsule heater thermowell, 3.) Pressure tube extension, 4.) Type-N thermocouple, 5.) Immersion heater, 6.) Plenum, argon gas, 7.) Capsule plug, 8.) Radiative Heat Shield, 9.) Molten salt volume

Heat transfer out of the capsule is controlled via an inert gas gap which is held via standoff geometries machined on the outer diameter of the capsule. Based on initial thermal hydraulic results this gas gap was designed as a 85% argon, 15% helium mixture in a total radial thickness of 0.76 mm. Further analysis utilizing STAR CMM+ revealed that at high temperatures – in the range of 1000°C – radiative heat transfer from the wall of the capsule becomes a driving mechanism of heat loss. Therefore, a radiative heat shield fabricated from 316 stainless steel (SS316) acts as an intermediate boundary between the capsule wall and the capsule containment, splitting the gas gap into two separate radial sections, and reducing radiative heat transfer.

Fuel salt with an argon atmosphere is sealed in the capsule by a combination of laser welding and induction braze procedures on the top of the capsule bulkhead and the bottom end cap.

B. Outer Can Design

The MRTI inner capsule is assembled in an outer can containment which ultimately seals the experiment from the NRAD coolant water and maintains the gas gap composition of the experiment. Outer can containment geometry is designed to resemble typical NRAD fuel pin geometries as to maintain prototypical cooling conditions on the outer diameter of the experiment. Similar to the NRAD fuel pins, the outer can containment and all welded fittings are machined from SS316L. The inner diameter of outer can containment is defined by the gas gap and capsule geometries. A top fitting on the outer can containment is welded to the outer can containment tube to interface with existing NRAD cluster assemblies shown in Section III,C.

To maximize heat generation rates of the fuel salt the midplane of the salt is aligned axially with fuel midplane of the NRAD reactor. A graphite spacer is designed with the correct height for the capsule placement at the desired axial position. Graphite was the chosen as the spacer material in order to maintain as much moderating material as possible when displacing water with the MRTI assembly. Because graphite is relatively thermally conductive a bisque-alumina is utilized as an insulative spacer between the capsule and graphite spacer. This bottom insulative spacer is designed with machined axially standoffs to create a gas gap between the capsule and most of the bisque-alumina material.

Above the capsule a SS302 compression spring holds the capsule and internals axially static during handling and irradiation, and accounts for any axial thermal expansion. To reduce potential loss of elasticity in the spring and additional axial heat loss in the in capsule a top insulative annular spacer is used between the spring and capsule. In this region above the capsule and within the inner diameter of the spring the pressure extension tube from the capsule is assembled onto the optical fiber pressure sensor via a compression swage fitting. Instrumentation and heating cabling is routed around the swage fitting and fed through a compression gland seal fitting at the top of the outer can containment assembly. Soft cabling from the heater is

potted via hermetic sealing compound at the compression gland fitting. Instrumentation and extension cabling is collected and reaches approximately 3 m to the top of the NRAD coolant pool.

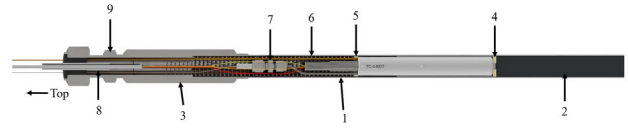


Figure 3. Cross-section view of the MRTI outer can containment design. 1.) Outer can geometry, 2.) graphite spacer, 3.) Outer can top fitting, 4.) Bottom insulation spacer, 5.) Top insulation spacer, 6.) compression springs, 7.) Swagelok fitting, 8.) Leadout potting cup, and 9.) Top compression seal fitting.

C. Experiment Assembly

The outer can containment MRTI experiment vehicle is designed to interface with typical NRAD cluster assembly hardware which interfaces with the NRAD core. To interface with the NRAD bottom cluster end fitting a male threaded bottom fitting is welded to the bottom of the outer can containment tube. Because the cluster assembly comprises four fuel pins, three other pins are required for the design. While graphite-filled or NRAD fuel pins have been considered for these cluster positions, ultimately aluminium 6061 mock pins were fabricated for the cluster assembly. In future experiments, these pins offer the opportunity for multiple MRTI experiment positions within a single cluster design. The cluster is held at the top by a lock-plate and bail assembly.

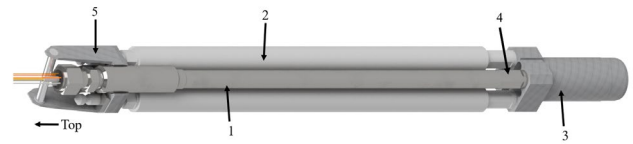


Figure 4. Isometric view of the MRTI cluster design. 1.) MRTI experiment vehicle, 2.) Aluminum pins, 3.) Bottom cluster end fitting, 4.) MRTI bottom fitting, 5.) Top cluster bail.

IV. PROTOTYPE FABRICATION

Experiment prototyping activities were conducted to improve assembly techniques and ultimately fabricate a scale surrogate-salt containing mock-up of the MRTI assembly. This assembly will then be leveraged for system commissioning testing of the MRTI heater temperature controller and expected outer can containment wall temperatures based on thermal hydraulic modelling.

A. Capsule Machining and Assembly

Capsule geometries were machined from IN625 bar for the inner capsule top and the capsule end cap. Bulkhead penetrations are drilled in the top of the inner capsule for braze assembly activities. High temperature nickel braze BNi-5 was identified as the material for the MRTI capsule with an operational temperature above 1000°C, and this material requires hole clearance of under 0.025 mm with the instrumentation and thermowell outer diameter. Induction brazing techniques were developed in accordance with American Society of Mechanical Engineers (ASME) Section IX qualification procedures for brazing IN625 sheath into IN625 bulkhead, as well as the

SS316 pressure extension tube into the bulkhead. This process was executed in one induction braze process using the high temperature BNi-5 braze. This braze is performed under purged flowing helium atmosphere at controlled pressure in order to reduce potential oxidation at the braze joint. Iterative parameter development and sample analysis was required to qualify the braze procedure for use in the experiment assembly.



Figure 5. Induction braze of an IN625 sheath instruments and thermowell in a IN625 bulkhead with prototypical thickness to MRTI capsule.

The capsule end cap is designed to be laser welded onto the capsule top. Laser welding is desirable for this application due to the thin wall of the capsule and the need to avoid potential salt melting during the welding process (laser welding provides control over local energy input in comparison to traditional hand welding techniques). An integral backer geometry with an internal interference fit is designed into the capsule wall to provide additional thermal protection of the salt during the welding process to avoid this unintended melting. This weld is performed under argon atmosphere and ultimately seals the argon plenum atmosphere in the capsule. This weld was also developed and qualified to ASME Section IX standards and testing.

Instrumentation, pressure tube extension, and the heater thermowell are induction brazed to the capsule top using the developed procedure. The compression swage fitting is installed on pressure extension tube and the fitting is capped with a union fitting or the pressure sensor is installed onto the fitting (pressure sensor is not required for surrogate salt commissioning test). From this assembly stage the capsule is filled with the surrogate LiCl-KCl salt powder using the same glovebox procedures which would be used for the fuel salt material to mock-up the assembly process. Because of leadout instrumentation a custom clamping assembly was designed to hold the capsule off the glovebox floor for salt loading. A custom metallic funnel is used to ensure catchment of salt in the capsule during loading. Once loaded, the clamp assembly also is designed to press the capsule end cap onto the capsule interference fit. Once the capsule is pressed it is ported into a laser welding glovebox and a weld is performed to seal the end cap to the capsule.

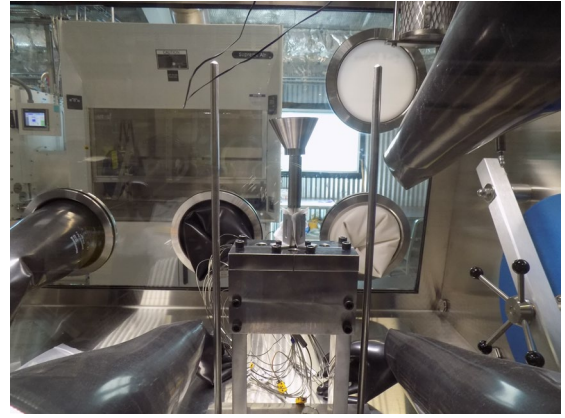


Figure 6. In-process capsule mounting, funnel and salt loading, and rails used for capsule end cap pressing.

B. Experiment Vehicle Assembly

With the completed assembly of the capsule the outer can containment assembly can be assembled. The outer can bottom fitting is pressed onto the outer can tube interference fit and welded onto the outer can tube with similar laser welding techniques for SS316L. The graphite spacer is first loaded into the outer can, followed by a pre-assembled combination of the capsule, radiative heat shield, and bottom and top insulative spacers.

Above the capsule and top insulative spacer the compression springs are slid over all instrumentation and leadouts. The outer can top fitting is then installed over the springs and pressed onto the interference fit on the outer can tube. This top fitting is then laser welded to the outer can tube. The compression seal gland fitting is then assembled over the instrumentation and onto the top can fitting. Gland internals of grafoil are carefully installed over all instrumentation and the heater cabling potting cup. The unsealed assembly is backfilled in a glovebox with the desired gas mixture. The final seal of the assembly occurs via installing the compression fitting manufacturer's instructions. After instrumentation and cabling connector installation is completed the MRTI outer can experiment vehicle is finished and inspected.



Figure 7. Completed MRTI surrogate salt experiment vehicle.

V. FUTURE WORK

Work is already underway to synthesize enriched UCl_3 to load in the final experiment assembly. Once this is completed, the fabrication process will be repeated in a controlled environment (due to the sensitive material) and loaded with enriched fuel bearing salt.

All the required documentation has also been submitted as part of the 'Experiment Safety Analysis' (ESA), and is currently under review. Once this is approved and the experiment is fully fabricated, the team can proceed to

installing the experiment within NRAD. Mimicking the layout of a TRIGA fuel cluster is expected to streamline this step. Initial zero-power testing in the reactor will then be conducted to check that the heater and all other systems (namely the controller and sensors) are operating as expected. After clearing these tests, the NRAD reactor power will be slowly ramped up and the irradiation can begin. The current plan is to commence irradiation around summer of 2023 timeframe. Around 30-days of irradiation (non-continuous) are planned. At the end of this, PIE activities can commence with the assembly transported to an INL hot cell where it will be disassembled. The initial plan is to conduct neutron radiography, gamma scan, and gas analysis. Future PIE could also include measurement of the irradiated salt properties and evaluating corrosion of salt-facing wall material.

VI. SUMMARY

The MRTI experiment design has been completed with the fabrication process demonstrated on a prototype assembly. The experiment is able to host salt samples around 10 cm³ of salt that will be kept at temperature using an immersion heater. Salt temperature will be monitored by submerged thermocouple sensors. The salt-bearing capsule is composed of IN625 alloy and is double encapsulated for defence in-depth. The second can is made of SS316. A gas-gap separating both contains a mixture of He-Ar gas that can be fine-tuned to control the experiment temperature.

A prototype assembly was fabricated and assembled to test the manufacturing process of the experiment. It was loaded with non-fuel bearing salt for this initial phase. Welding and brazing procedures for IN625 had to be established. The final experiment assembly was conducted in an inert environment with the recommended gas mix. The next step is to repeat this process with fuel-bearing salt that will then be loaded to the NRAD reactor for irradiation.

VII. ACKNOWLEDGEMENTS

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VIII. REFERENCES

- [1] R. E. Thoma, "Chemical Aspects of MSRE Operations," Oak Ridge National Laboratory, ORNL-4658, 1971.
- [2] N. D. Ezell, J. McDuffee, K. Smith and S. Raiman, "Initial Irradiation Testing of Chloride Salts for Molten Salt Reactors," in ANS Winter Meeting, Orlando, 2018.
- [3] P. R. Hania, "MSR Irradiation Program at NRG Petten," in MSR Workshop 2018, Oak Ridge, 2018.
- [4] C. W. Forsberg, P. F. Peterson, K. Sridharan, L. Hu, M. Fratoni and A. Kant-Prinja, "Integrated FHR Technology Development: Tritium Management, Materials Testing, Salt Chemistry Control, Thermal Hydraulics and Neutronics, Associated Benchmarking and Commercial Basis," NEUP Report, MIT-ANP-TR-180, 2018.
- [5] Bess, J. D. and Higgs, J. B. and Lell, R. M., "Neutron Radiography (NRAD) Reactor 64-Element Core Upgrade", Idaho National Laboratory, INL/EXT-13-29628, 2014.
- [6] A. Abou-Jaoude, J. Chandler, G. Core, K. Davies, C. Downey, W. Phillips, C. Tan and S. Wilson, "Conceptual Design of Temperature-Controlled Fuelled-Salt Irradiation Experiment to Support Demonstration of Advanced Nuclear Reactors". In Proc. International Mechanical Engineering Congress and Exposition, Virtual, 2021.
- [7] A. Abou-Jaoude, C. Downey, G. Core, K. Davies, W. Phillips, C. Tan, S. Yoon, and S. Wilson, "Design and Prototyping of a Fissile-Bearing Chloride Salt Irradiation Experiment", in *Transactions of the American Nuclear Society*, Virtual, Nov. 2021.