

Determining the Impact of Magnesium Addition for Corrosion Control of Select Alloys in a Molten Salt Reactor

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Prepared for the Canadian Nuclear Society
2024 Graduate Student Scholarship Award

THE UNIVERSITY OF NEW BRUNSWICK

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List of Acronyms and Symbols

List of Abbreviations

CANDU[®]: CANadian Deuterium Uranium reactor

CNER: Centre for Nuclear Energy Research

EDM: Electrical Discharge Machining

FIB: Focus Ion Beam

ICP-MS: Inductively Coupled Plasma-Mass Spectrometry

MSR: Molten Salt Reactor

SEM-EDS: Scanning Electron Microscopy/Energy-Dispersive X-ray Spectrometry

SSR-W: Stable Salt Reactor-Wasteburner

UNB: University of New Brunswick

WATSS: Waste to Stable Salt

XRF: X-Ray Fluorescence

List of Units and Chemical Names

BN: Boron Nitride

MW_e: Megawatts Electric

MW_{th}: Megawatts Thermal

YSZ: Yttria - Stabilized Zirconia

1. INTRODUCTION

1.1 Background Information

Molten Salt Reactors (MSRs), a Generation IV nuclear reactor concept, provide low-pressure and high-temperature operation, while corrosion control and salt environment material compatibility remain challenging. This study continues static molten salt immersion experiments from previous research to evaluate long-term corrosion mechanism of molten chloride salt on 316 Stainless Steel and Nimonic[®] Alloy PE16, and to investigate using magnesium metal addition for corrosion control at 550°C. Significant apparatus design improvements and seal performance were accomplished. The experiments demonstrated Cr depletion and MgO formation on the alloy surface and these are recognized as major corrosion mechanisms of Nimonic[®] Alloy PE16 in a molten eutectic NaCl and MgCl₂ salt mixture. Mg-metal addition reduces molten salt impurities, but intermetallic compound formation between Mg and Ni causes dealloying. This research contributes to optimizing the experimental framework the molten chloride salt corrosion experiments and recommends selected alloys for compatibility in MSRs for research and development purposes.

1.2 Project Scope

Around 2016, the Centre for Nuclear Energy Research (CNER) at the University of New Brunswick (UNB) began developing its molten salt research capabilities, with continuous progress in apparatus acquisition and research projects, and the renovation project of the molten salt laboratory within the Advanced Nuclear Reactors Laboratory (ANRL) that was under construction between 2024 and 2025.

The research project conducted in this work investigated molten chloride salt corrosion experiments under static immersion at CNER to (1) develop and improve the molten chloride salt static immersion experiment environment control, specifically to eliminate oxygen and moisture ingress from atmosphere; (2) evaluate the long-term corrosion mechanisms of molten chloride salts on 316 stainless steel and Nimonic[®] Alloy PE16; and (3) study the effectiveness of magnesium for corrosion control under 550°C with an argon purge, in order to support the molten salt reactor and small modular reactor industrial development.

2. LITERATURE REVIEW

2.1 Molten Salt Reactor: Moltex Energy Canada SSR-W

Chloride salt-based molten salt reactors are considered to be a good choice for a fast neutron molten salt reactor or a burner-type reactor to recycle legacy spent fuel and consume long-term radioactive waste as transmutation of minor actinides [1], [2]. Moltex Energy Canada's SSR-W is one of the selected small modular reactor concepts by New Brunswick Power for advanced SMR deployment. The SSR-W concept mainly contains three sections, a 300 MW_e chloride-salt fast-neutron-spectrum burner-type stable salt reactor (SSR) (see Figure 1), a WASTE-TO-STABLE-SALT (WATSS) process conversion unit to recycle CANDU[®] spent fuel to MSR fuel, and a GridReserve[®] system to store excess heat from the nuclear island and prepare for energy peak demands using solar-salt thermal energy storage. This innovative concept combines nuclear and renewable energies to provide flexible and reliable clean energy on demand, helping with climate change issues and increasing energy demand [3].

The SSR-W utilizes a combination of chloride salts as fuel salt (which includes NaCl-MgCl₂-XCl₃, where X = uranium, transuranic actinides and lanthanides) contained in the fuel pins instead of a loop-type conventional molten salt reactor design [2]. The coolant salt is a eutectic mixture of NaCl and MgCl₂, which removes heat from the fuel pins and assemblies via continuous flow through the intermediate heat exchanger. Core coolant temperatures range between 500 and 625°C [4]. This research project aims to support the first-of-the-kind SSR-W reactor concept via research and development on chloride coolant salt structural material compatibility, material integrity, and corrosion control. The schematic diagram Figure 1 in shows the reactor core geometry, fuelling machine, coolant and fuel salts locations [4], [5], [6].

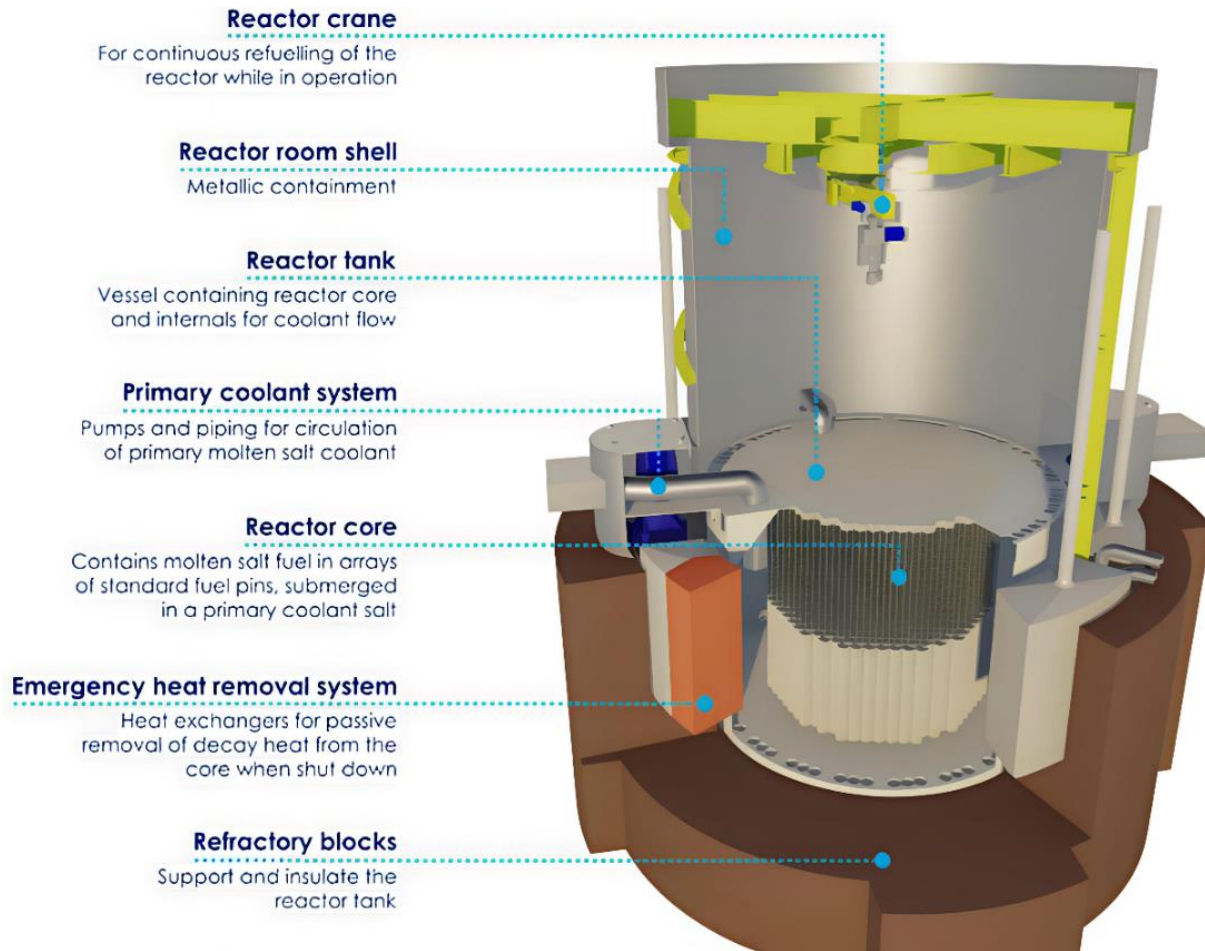


Figure 1. Schematic Diagram of Moltex Energy Canada SSR-W [6].

2.2 Alloy Specification

The static molten chloride salt immersion corrosion experiments are conducted on 316SS and Nimonic® Alloy PE16, and micro-XRF analysis of the alloy compositions are listed as Table 1. These materials have been selected since they are the proposed materials of choice for the reactor vessel, piping and major reactor internal components (316SS) and the reactor fuel pins (Nimonic Alloy PE16).

Table 1. Material Composition (wt.%) Determined by Micro XRF.

	316 Stainless Steel	Nimonic[®] alloy PE16
Al	0.15	1.03
Co	0.54	-
Cr	16.44	15.66
Cu	0.46	-
Fe	68.59	35.08
Mn	1.44	-
Mo	1.97	3.20
Ni	9.67	43.20
S	0.02	0.34
Si	0.62	0.27
Ti	-	1.22
V	0.09	-

3. EXPERIMENT OVERVIEW

The testing apparatus preparation procedures are established; experimental conditions are summarized and post-corrosion sample analysis methods are presented in the Experimental Methods section.

3.1 Previous Molten Salt Corrosion Test Overview at CNER

Testing trials of static immersion molten chloride salt corrosion experiments were conducted on 316SS at CNER with varying experiment times up to 500 hours. Corrosion test atmospheres were controlled at 550°C with continuous argon purging. Molten chloride salt mixtures of NaCl and MgCl₂ were pre-melted in the inert glovebox furnace to ensure eutectic mixing. Metal coupons were fabricated via EDM cutting and polished to 1200 grit. Yttria-stabilized zirconia (YSZ) crucibles were employed as the molten salt corrosion test vessel, and crucibles were contained in a 316SS corrosion retort. Four coupons were used in each trial, two coupons with metallic Mg added for corrosion control, and the other two as reference samples without the added Mg. The assembly process of the salts and coupons in the corrosion retort occurred in a glovebox under Ar atmosphere where the retort vessel was hand-tightened. The assembled apparatus was then transferred out of the glovebox and connected with a continuous Ar purge line and externally heated by a high-temperature furnace during the corrosion test. These initial trials identified significant requirements for

improving the corrosion retort seal performance and oxygen control in the purge gas.

In terms of molten chloride salt corrosion results, significant indications of intergranular corrosion were identified after 500 hours exposure on 316SS without Mg corrosion control, with an average corrosion-affected depth of 105 μm (Figure 2), while intergranular corrosion-affected depth was reduced significantly (Figure 3) when Mg was included for corrosion control [7]. Continuous development on corrosion retort seal performance showed that the extent of molten chloride salt corrosion on 316SS is greatly reduced when better oxygen and moisture-control are maintained in the environment, at 550°C with continuous Ar purging.

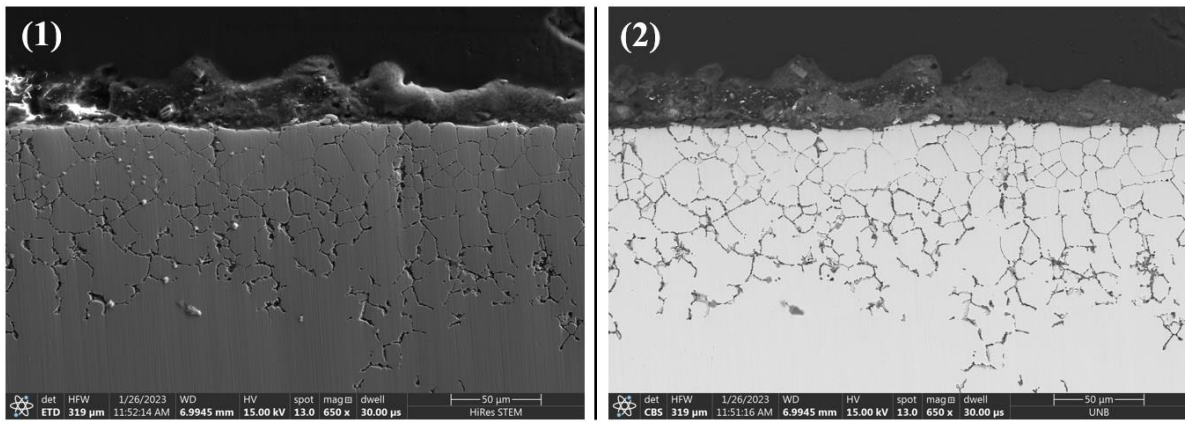


Figure 2. SEM Images of Previous CNER 316SS 500-Hours Corrosion Test without Mg Metal Addition [7].

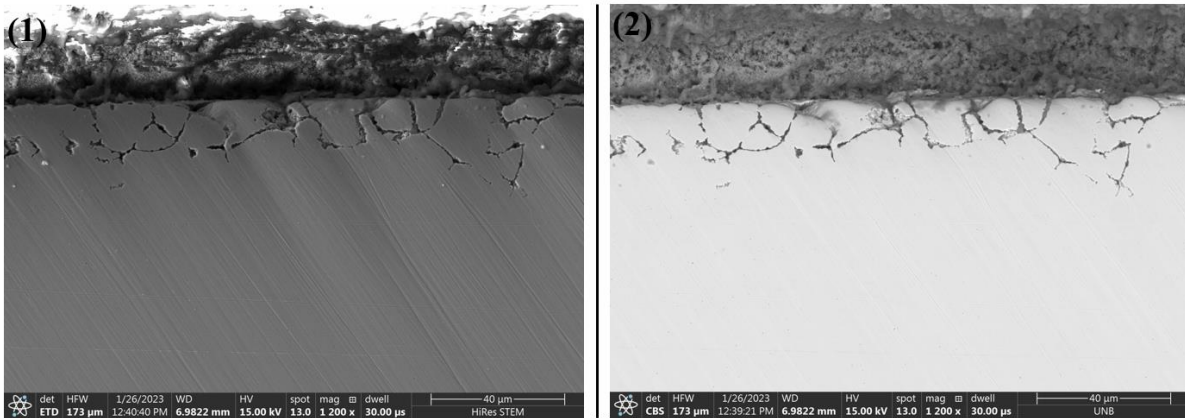


Figure 3. SEM Images of Previous CNER 316SS 500-Hours Corrosion Test with Mg Metal Addition [7].

3.2 Apparatus Preparation

Corrosion retort seal challenges were recognized when reviewing the previous results at CNER, and the key milestones for experiment preparation include five major sections:

- 1) Corrosion retort preparation (fabrication, non-leakage testing, cleaning, dehydration).
- 2) Test coupons and coupon holder preparation (fabrication, polishing, cleaning, and dehydration).
- 3) Crucible preparation (cleaning and dehydration).
- 4) NaCl and MgCl₂ salt preparation (dehydration and mixing).
- 5) Mg ribbon preparation (weighing and assembling).
- 6) Assembly of the experimental setup.

In terms of the apparatus and chemicals prepared for the static molten chloride salt corrosion experiments, yttria-stabilized zirconia (YSZ, 30ml, Catalog No. CC30AZ, with Compositions listed in Table 2) crucibles and boron nitride (BN, 30ml, >99 wt.%) crucibles are utilized as molten salt containers to determine the inert characteristic of crucible materials. Both types of crucibles are made by Almath UK [8], [9]. Ultra-dry MgCl₂ (99.99% metals basis, Catalog No. 042850-22) and dry NaCl (99.99% metals basis, Catalog No. 087605-30) were purchased from ThermoFisher Scientific. The eutectic chloride salt compositions are 44.438-55.562 wt.% (56.578-43.422 mol%) for NaCl-MgCl₂ respectively, which decreases the melting temperature of the mixture to around 450°C. Corrosion tests set the temperature to ~100°C higher than the melting temperature to ensure even mixing [7], [10], [11], [12]. High-purity metallic Mg ribbon (99.99 wt.%, Catalog No. AA1281114) was acquired from Alfa Aesar, and ultra-high purity Ar gas (Grade 5, 99.9999%, CAS No. 7440-37-1) sourced from Linde Canada Inc [13], [14]. The salt storage and pre-treatment, corrosion test apparatus assembly, test vessel sealing, and post-corrosion test vessel unsealing are all conducted in the Ar-environment glovebox (MBRAUN UNIlab pro, H₂O content <1 ppm, O₂ content <1 ppm, Figure 4).

Table 2. YSZ Crucible Composition List [7].

Weight Percent (wt.%)								
ZrO ₂ + HfO ₂	Y ₂ O ₃	SiO ₂	Fe ₂ O ₃	TiO ₂	Al ₂ O ₃	Na ₂ O	CaO	Loss on ignition (L.O.I)
85.8	13.7	0.1	0.003	0.1	0.25	0.01	0.01	0.6



Figure 4. MBRAUN Glovebox Setup.

The experimental setup was assembled in the glovebox (MBRAUN UNIlab pro, H₂O content <1 ppm, O₂ content <1 ppm) under an argon environment. The coupon assembly setup and corrosion retort orientation are shown in Figure 5 and Figure 6.

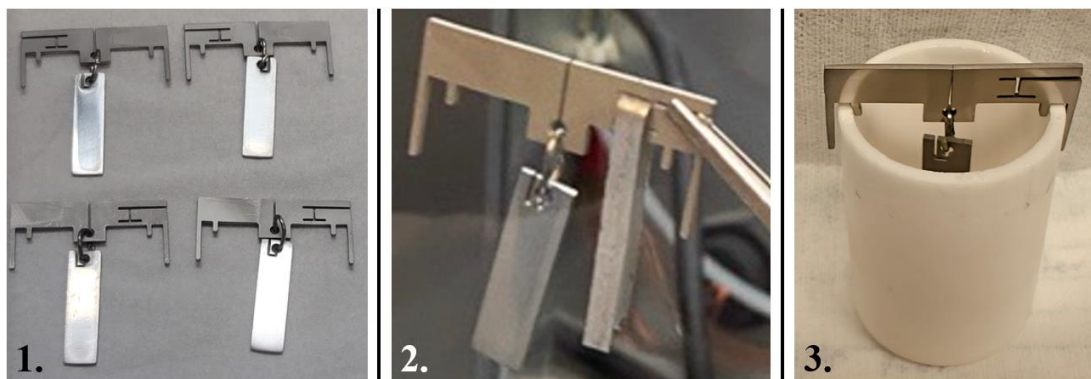


Figure 5. (1) Coupon Sets, (2) Set with Mg Ribbon, (3) Crucible Set Demo.

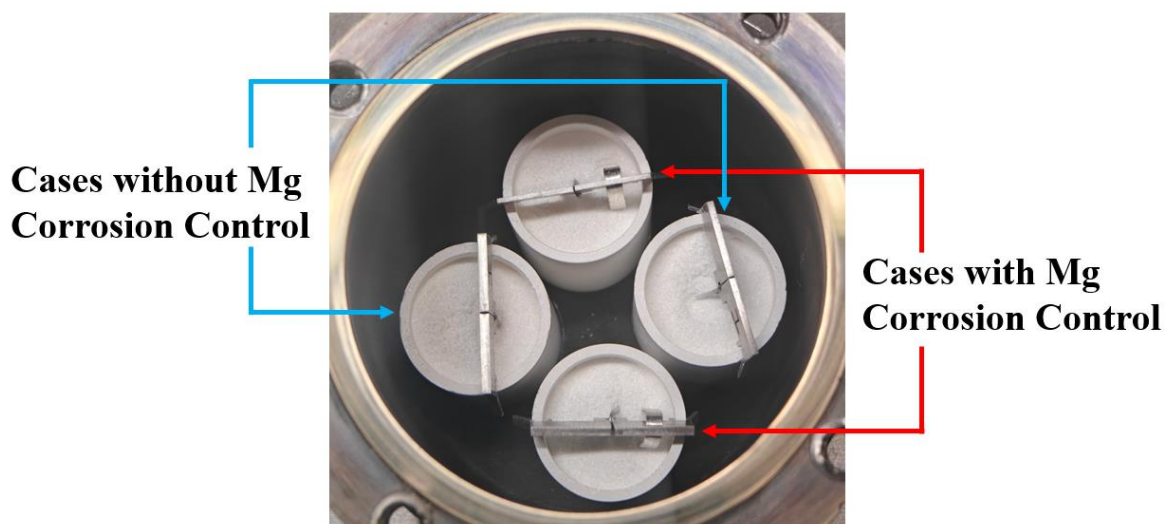


Figure 6. Corrosion Retort Orientation.

When the retort preparation was completed, the argon inlet and outlet valves on the retort lid were closed, and the retort transferred to the high-temperature furnace (Lindberg/MPH 56000 Series Crucible Furnace, Model No. 56611) for static molten salt immersion corrosion experiment. The process flow diagram and lab setup are in Figure 7.

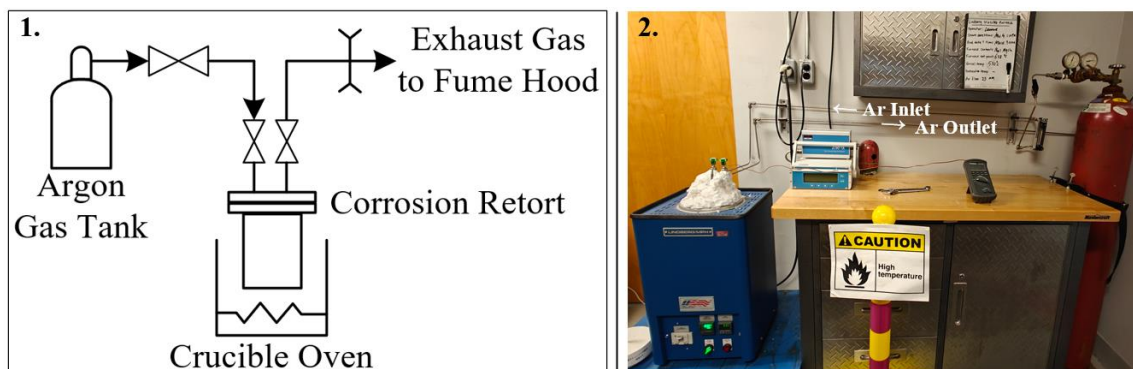


Figure 7. Molten Salt Static Corrosion Experiment (1) Process Flow Diagram, (2) Lab Setup.

3.3 Trial Condition Summary

The five trials of static molten chloride salt immersion corrosion experiments were performed to evaluate the long-term corrosion mechanisms of molten chloride salts and investigate the effectiveness of Mg corrosion control under 550°C with Ar purging and are summarized in Table 3 and Table 4.

Table 3. Static Molten Salt Immersion Corrosion Experiment Test Matrix (Part 1).

Trial #	Temp. (°C)	Pressure (atm)	Time (hrs)	Test Coupon	Salt Composition	Salt Mass (g)
1	550	1 atm, Ar gas purge	500	316SS	44.438- 55.562 wt.% NaCl-MgCl ₂	25g per crucible. 100g per trial.
2			500	316SS		
3			500	PE16		
4			1000	PE16		
5			500	316SS & PE16		

Table 4. Static Molten Salt Immersion Corrosion Experiment Test Matrix (Part 2).

Trial #	Crucible	Salt Pre-Melt	Metal Sample Sandblast Pre- Treat
1	YSZ	Yes	Yes
2	BN	Yes	Yes
3	BN	Yes	Yes
4	BN	No	No
5	BN	No	No

The selection of the crucible material for the remainder of this work was decided based on the results from the first two trials. Analysis of the post-corrosion crucible materials found that the YSZ crucibles produced unstable defects in a high-temperature environment where oxygen and moisture are well controlled and contributed oxygen anion to the molten chloride salt corrosion test (by reduction of ZrO_2 to $\text{Zr} + \text{O}^{2-}$), resulting in future experiments being performed with boron nitride crucibles.

Salt pre-melting was done in the first three trials, while this process was eliminated in subsequent trials since the pre-melt salt block required size-reduction to a powdery form, which is not only produces a powder but may also introduce impurities during size reduction. In the first three trials, prior to polishing, the metal coupons were subjected to a sandblasting to remove EDM cutting residues. This step was eliminated in the fourth and fifth tests because the sandblasting may have introduced fine silica particles that contaminated the metal samples.

3.4 Post-Corrosion Sample Analysis

Post-experimental analysis is critical for proper interpretation of the experiments. After the completion of experiments, the major steps in post-corrosion sample analysis include:

- 1) Visual inspection of coupons, crucibles, and salts before methanol cleaning.
- 2) Methanol cleaning and drying of post-corrosion coupons.
- 3) Mass difference comparison of pre- and post-corrosion coupons.
- 4) Preparation of SEM-EDS coupon samples.

5) Post-corrosion salt sample preparation for ICP-MS salt composition analysis.

The post-experiment cross-section and surface analysis was performed via SEM-EDS (ThermoFisher Scientific Scios 2 DualBeam). The electron microscopic analysis, with elemental identification and quantification, supports interpretation of the molten salt corrosion mechanisms and diffusion / migration of elements. Post-corrosion salt samples are prepared for ICP-MS (Agilent 7700x Quadrupole ICP-MS) analysis to understand the dissolution of metal species into molten salt.

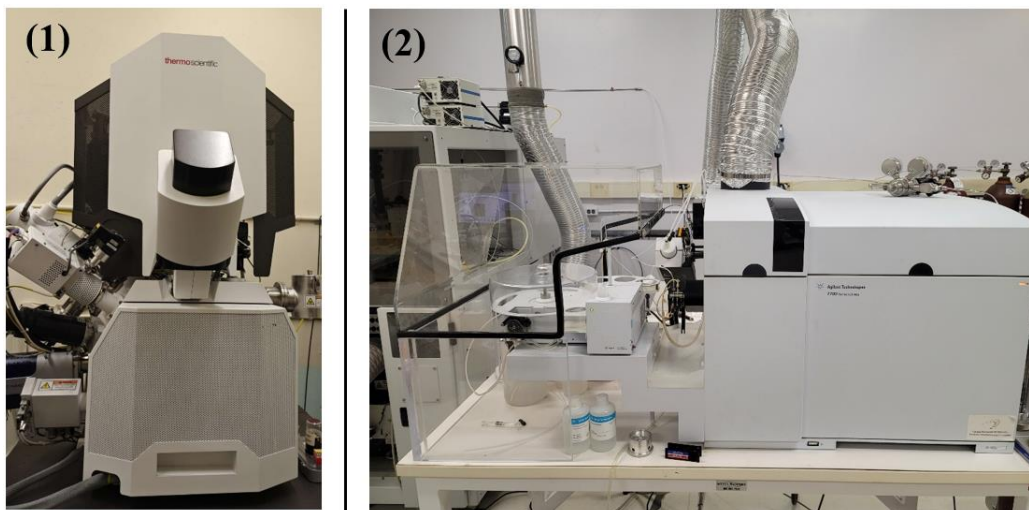


Figure 8. Analytical Instruments (1) SEM-EDS, (2) ICP-MS.

4. RESULTS

Five trials of static molten chloride salt immersion experiments were conducted; major outcomes of Nimonic[®] Alloy PE16 with and without Mg addition are summarized in this section. Improvements were implemented in an iterative fashion, based on results from each of the previous trial.

4.1 Nimonic[®] Alloy PE16 Without Mg Addition

Nimonic[®] Alloy PE16 without Mg metal addition examined in the 1000-hour corrosion experiment. SEM-EDS surface mapping (Figure 10 and Figure 11) and FIB cross-section analysis (Figure 12 and Figure 13) show that the corrosion mechanisms are described by significant Ni mobility from the metal matrix to the surface, Si impurity-driven corrosion forming a silicide corrosion product layer, Cr depletion along the grain boundaries, and MgO formation below the metal surface (approx. 5 μm deep on average) along the grain boundaries. The non-uniform corrosion layer formation is shown in the SEM mapping of the surface sample in Figure 9.

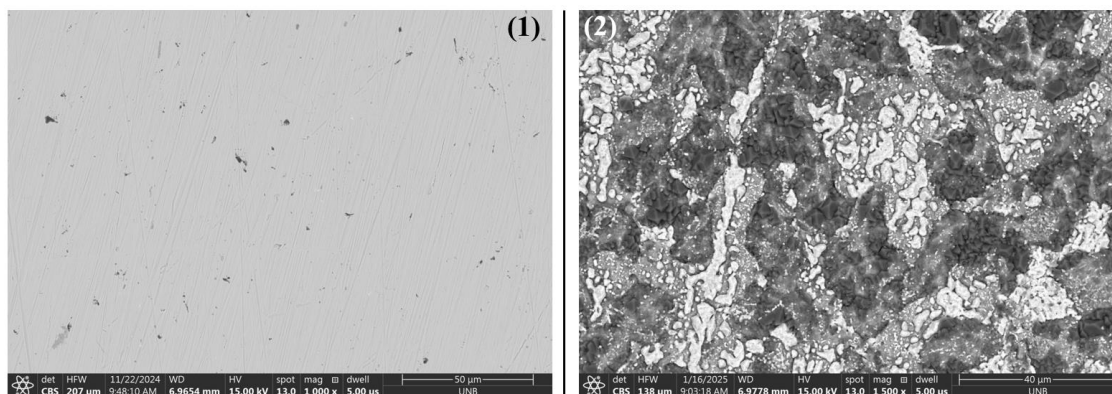


Figure 9. SEM Images of Nimonic® Alloy PE16 Surface Samples (1) Reference, (2) Post-Corrosion 1000-Hours without Mg Addition.

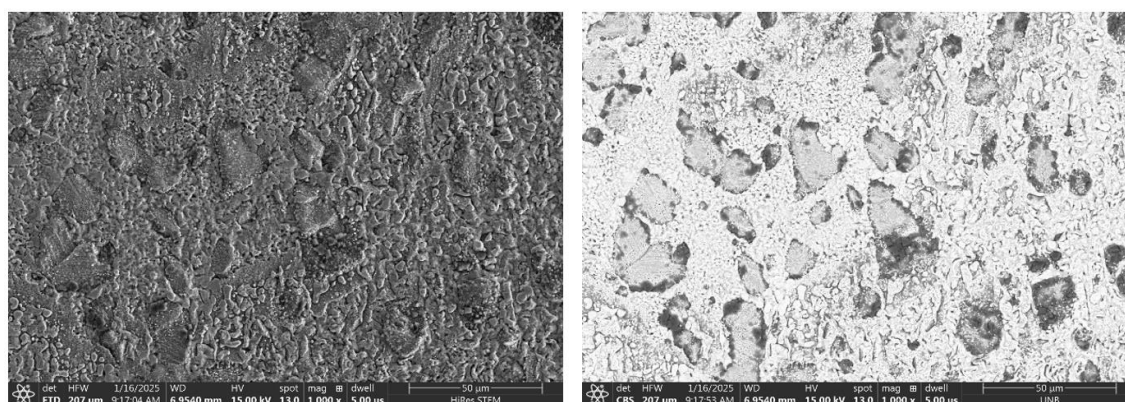


Figure 10. SEM Image of Trial 4 Nimonic® Alloy PE16 Surface Sample without Mg Addition.

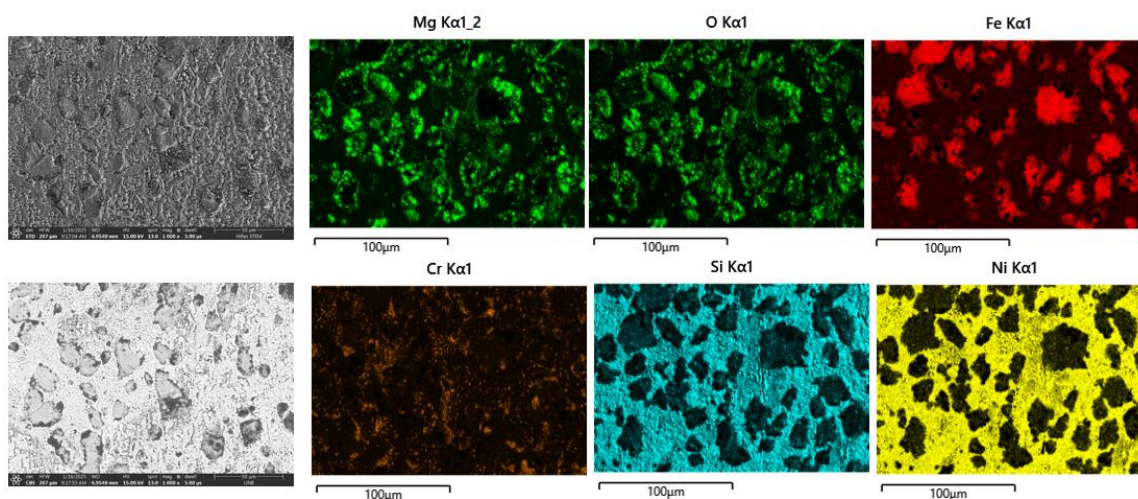


Figure 11. EDS Mapping of Trial 4 Nimonic® Alloy PE16 Surface Sample without Mg Addition.

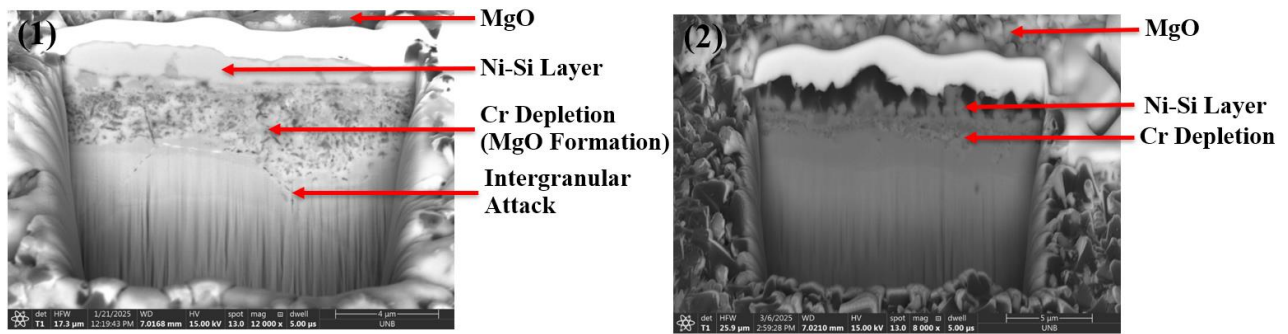


Figure 12. Nimonic® Alloy PE16 FIB Cross-section SEM without Mg Addition, (1) 1000 hrs, (2) 500 hrs.

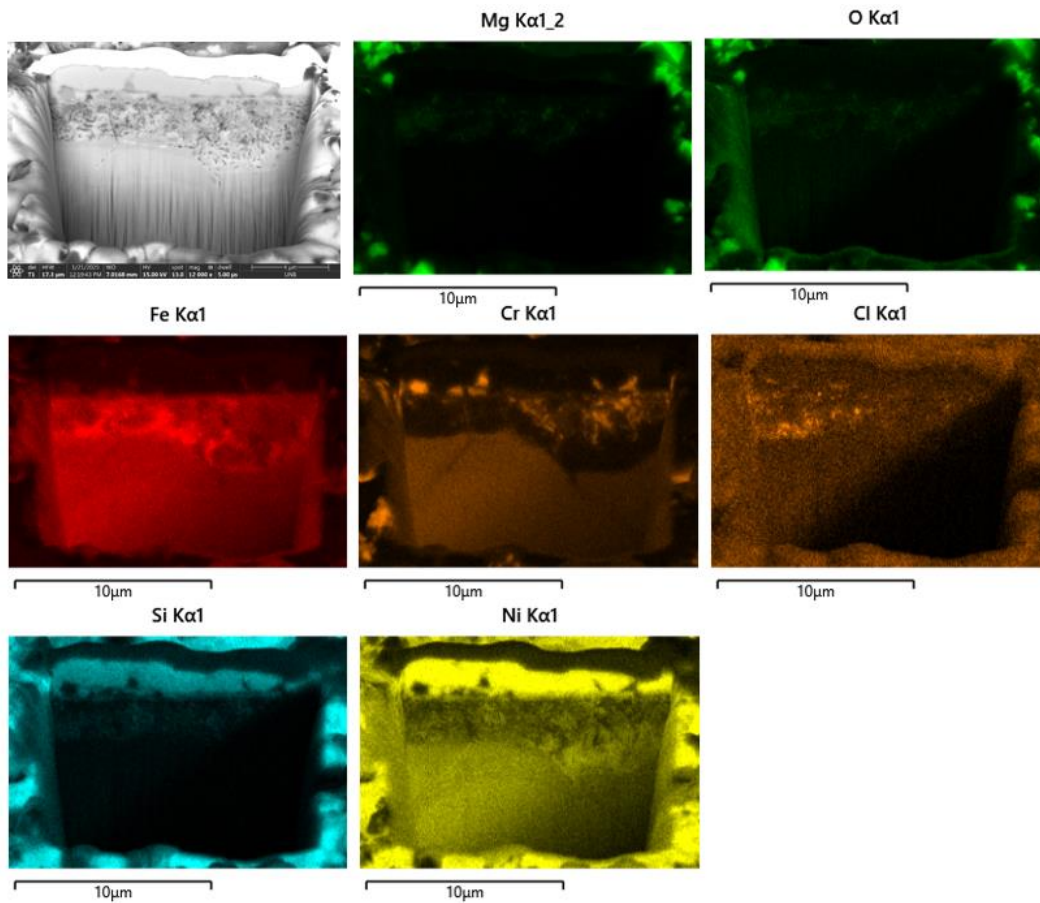


Figure 13. SEM-EDS Mapping of Trial 4 Nimonic® Alloy PE16 FIB Cross-Section Sample without Mg Addition.

4.2 Nimonic® Alloy PE16 with Mg Addition

The large- and localized-scale cross-sections of the Nimonic® Alloy PE16 coupon samples with Mg metal addition are shown in Figure 14 (1) and Figure 14 (2) respectively. A MgO layer formed above the base metal and no significant corrosion effects nor elemental species leaching from the base metal are identified below the MgO deposit. Minor pitting corrosion was witnessed on the coupon surface, but the alloy generally maintained its integrity.

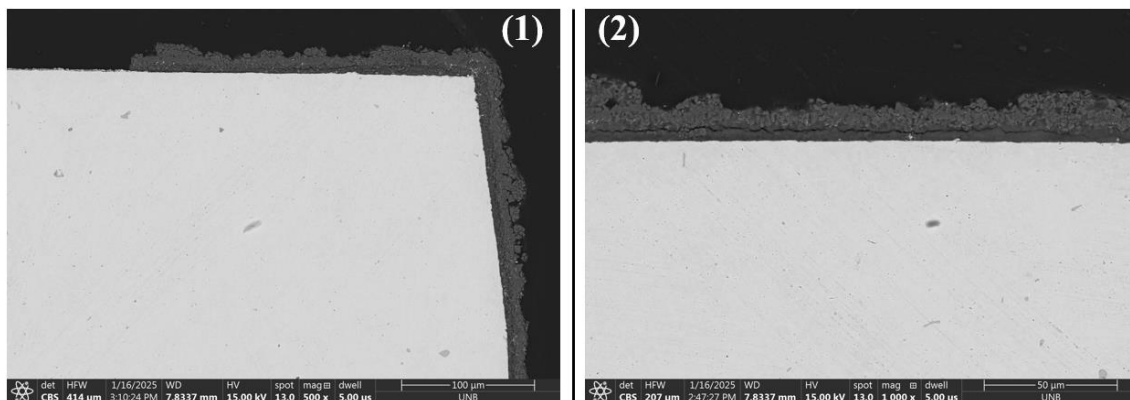


Figure 14. Nimonic® Alloy PE16 Coupon Cross-Section with Mg Addition, 1000 hrs.

4.3 ICP-MS Elemental Composition Analysis on Post-Corrosion Salts

Table 5 is the ICP-MS composition analysis results of post-corrosion salt samples from Nimonic® Alloy PE16 for 1000 hour exposures, with and without added Mg. Comparing the two 1000-hour trials, Mg added as a corrosion control method reduces Cr, Mn, and Fe depletion, while an increase in Ni concentration when Mg is present shows the effect of Ni-Mg interaction and dealloying.

Table 5. ICP-MS Analysis of Post-Corrosion Salt from Molten Chloride Salt Immersion Test on Nimonic® Alloy PE16.

Elemental Composition of Dissolved Elements from Nimonic® Alloy PE16 (ppb)			
Element	Baseline	1000 hours (No Mg Addition)	1000 hours (Mg Addition)
Cr	1.4	4.9 ± 0.1	1.3 ± 0.1
Mn	0.2	3.0 ± 0.2	1.5 ± 0.3
Fe	31.0	79.3 ± 3.5	48.9 ± 14.9
Ni	0.6	1.1 ± 0.2	4.5 ± 1.4

5. CONCLUSION

This research project expanded upon the research capabilities on molten salt corrosion testing and analysis at the Centre for Nuclear Energy Research of the University of New Brunswick. The work has contributed to understanding molten chloride salt corrosion mechanisms and innovating corrosion control to support the research and development in molten salt reactor material compatibility studies and selection for the future nuclear technology leadership in New Brunswick. Five trials were conducted at 550 °C, and the experimental procedures and analytical methods were continuously improved based on the trial results. It is foreseeable that more improvements in apparatus compatibility, preparation and analysis procedures can be implemented, and systematic experimentation and examination of molten salt corrosion will be beneficial to the field.

By experimenting and analysing the molten chloride salt static immersion corrosion tests and results on 316SS and Nimonic[®] Alloy PE16, the corrosion behaviour was determined via the SEM-EDS analysis on coupon samples, while alloy dissolution quantified by ICP-MS salt composition analysis. The effectiveness of Mg addition corrosion control was also illustrated. With the ongoing progress in methodology development and in-depth sample analysis, the research project objectives can be met, and challenges that arose during this work may be resolved. This contribution consists of optimizing of the experimental framework in the field of molten chloride salt corrosion and provides recommendations for the compatibility of selected metal alloys for the molten salt reactor research and development.

6. ACKNOWLEDGEMENT

I would like to express my sincere gratitude to CNER and UNB faculties and staff for their leadership, mentorship, and guidance: Dr. William Cook, Mr. Chester Morries, Mr. Steven Cogswell, Dr. Christopher Alcorn, Mr. Adon Briggs, Dr. Christopher McFarlane, Mr. Brandon Boucher, Dr. Jan-Hendrik Poehls, Dr. Ben Loder, Dr. Lihui Liu, Dr. Dennis Connor, and Ms. Monishola Aransiola for their technical support and contributions.

I acknowledge and greatly appreciate the financial contributions and partnerships from the Government of New Brunswick, Mitacs, Moltex Energy Canada, and the University of New Brunswick and the Canadian Nuclear Society for the Graduate Scholarship Award.

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