**Multimodal In Situ X-ray and Electron Microscopy Reveals Temperature-Dependent Corrosion Mechanisms of Ni-20Cr Alloy in Molten Salt**

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**Introduction**

Understanding alloy behavior in molten salt environments plays an important role in developing sustainable energy systems. Specifically, a molten salt reactor (MSR) is considered as a safe and efficient nuclear power plant. However, due to the lack of oxide-based passivation and selective dissolution of alloy elements, the corrosion of structural materials arises safety and maintenance concerns. Previous studies showed the electrochemical dissolution of Cr in molten chloride salts forms cracks or voids due to the inevitable contaminations (H₂O and O₂) and the hygroscopic nature of the salt such as MgCl₂. There is a strong motivation to observe the real-time morphology change of the alloys in molten salt which is significant to better understand the interplay between the mass transport in the alloys and the salts, and the electrochemical reaction and the diffusion at the salt-metal interface. Additionally, the corrosion behavior varies because of the existence of the temperature gradient in the molten salt flow loop. This work used Ni-20Cr alloy and molten KCl-MgCl₂ (50-50 mol.%) as a model system to study the influence of temperature on the molten salt corrosion characterized by in situ synchrotron X-ray nanotomography and transmission electron microscopy without cleaning and air-exposure to preserve the chemical states.

**Experiment Setup**

In situ synchrotron X-ray nanotomography experiment was conducted in the full-field X-ray imaging beamline (FX, 18-ID) with a microfocus X-ray source at National Synchrotron Light Source II (NSLS-II). The Ni-20Cr and MgCl₂-KCl were sealed in double cavity in Ar-filled glovebox. The temperatures were 500, 600, and 800°C. The same samples were characterized by transmission electron microscopy without contacting the air or water at Idaho National Laboratory.

**3D Morphology Evolution at Varied Temperatures**

- **500°C**
  - Core (Cr)-shell (Ni) structure formed after corrosion
  - Cr dissolution formed Cr²⁺ in the salt

**Local Morphological Change**

- Corrosion occurs at the salt-metal interfaces in all three temperatures
- Grain boundary corrosion was observed at 500 and 600°C
- At 800°C, the morphological evolution in molten salt corrosion is similar to the one in a percolation deaingloying, forming a bicontinuous porous structure
- Corrosion progresses into the grains, forming larger voids and cracks along grain boundaries

**Elemental Distribution and Chemical Oxidation - 600°C**

- Time-dependent development of corrosion mechanism

**Summary and Future Work**

- At lower temperatures (500-600°C), while the reaction starts as a grain boundary corrosion, with a prolonged reaction time under an elevated temperature, the molten salt corrosion propagates into the grains
- A bicontinuous porous structure formed at a higher temperature (800°C), following the percolation deaingloying mechanism
- A core-shell structure formed in the alloys after corrosion, with an enriched Ni surface
- The Cr²⁺ species formed after corrosion
- Further analysis is underway to discern the kinetics mechanisms and the interplay between the mass transport (long-range and surface diffusion) and the chemical reaction in molten salt corrosion and how they may lead to the formation of different morphologies

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**References**