SOLAR ENERGY
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Development of In-Situ Corrosion Kinetics and Salt Property Measurements

Salt Properties and Corrosion Kinetics on Metal Films

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START: We are not sure what the corrosion mechanism is and we cannot describe how to stop it.

After three years

Throughout the experiments, temperature, moisture and oxygen levels (contamination) have to be controlled very carefully.

- The electrochemical methods are applied to explore the thermodynamic and kinetic data of corrosion reactions in the molten salt.
- Ability to deposit, characterize and corrode Inconel / salt mixture under controlled environment (vacuum or controlled gas partial pressure) so that we can elucidate fundamental corrosion mechanisms. Nanoscale spatial resolution, sub second temporal resolution., at temps up to > 800 °C.
- In-situ TEM: Capability of real time measurements of Inconel corrosion by ternary salt mixture at grain-by grain level.
- Grain size of thin films of Inconel 625 grows with the increase of deposition temperature, and film with larger grain size (higher deposition temperature) is more corrosion resistant.
- O_2 and H_2O in glove box diffuse into salts and react with salts, and generate magnesium oxides and Cl_2. Cl_2 diffuse into alloy and corrode the film.
- In-situ NR: seven slab model to analyze NR data is developed.
- Work-in-progress: Ab initio molecular dynamics (AIMD) analysis based on neutron and X-ray experiments. And many others.

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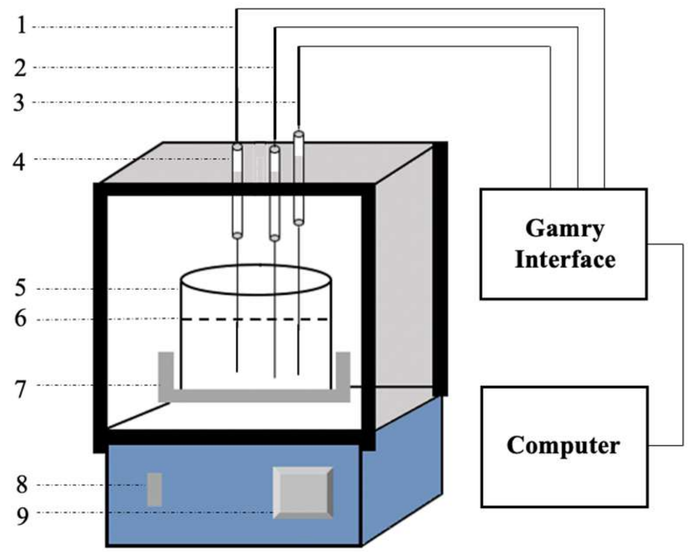
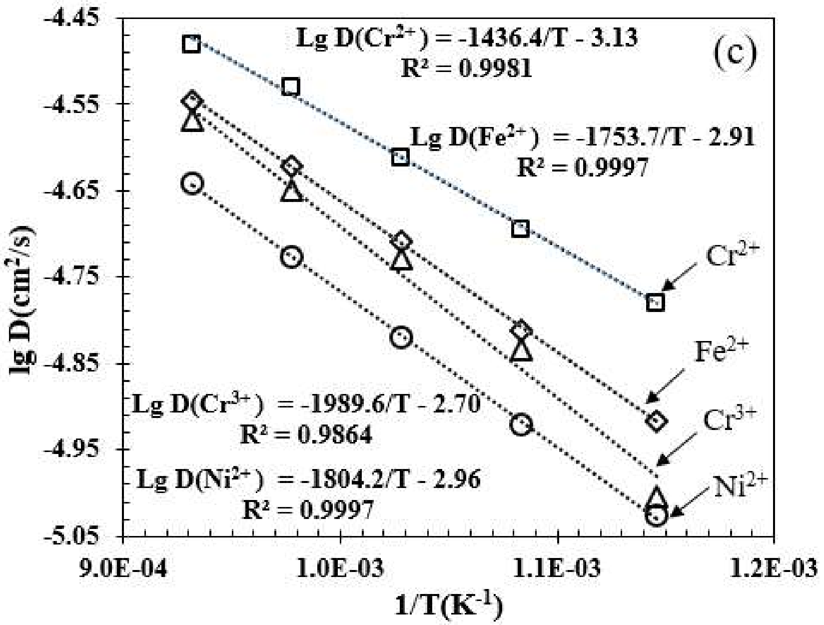
The electrochemical methods are applied to explore the thermodynamic and kinetic data of corrosion reactions in the molten salt.

Transport Kinetic Data: Diffusion Coefficient Measurement

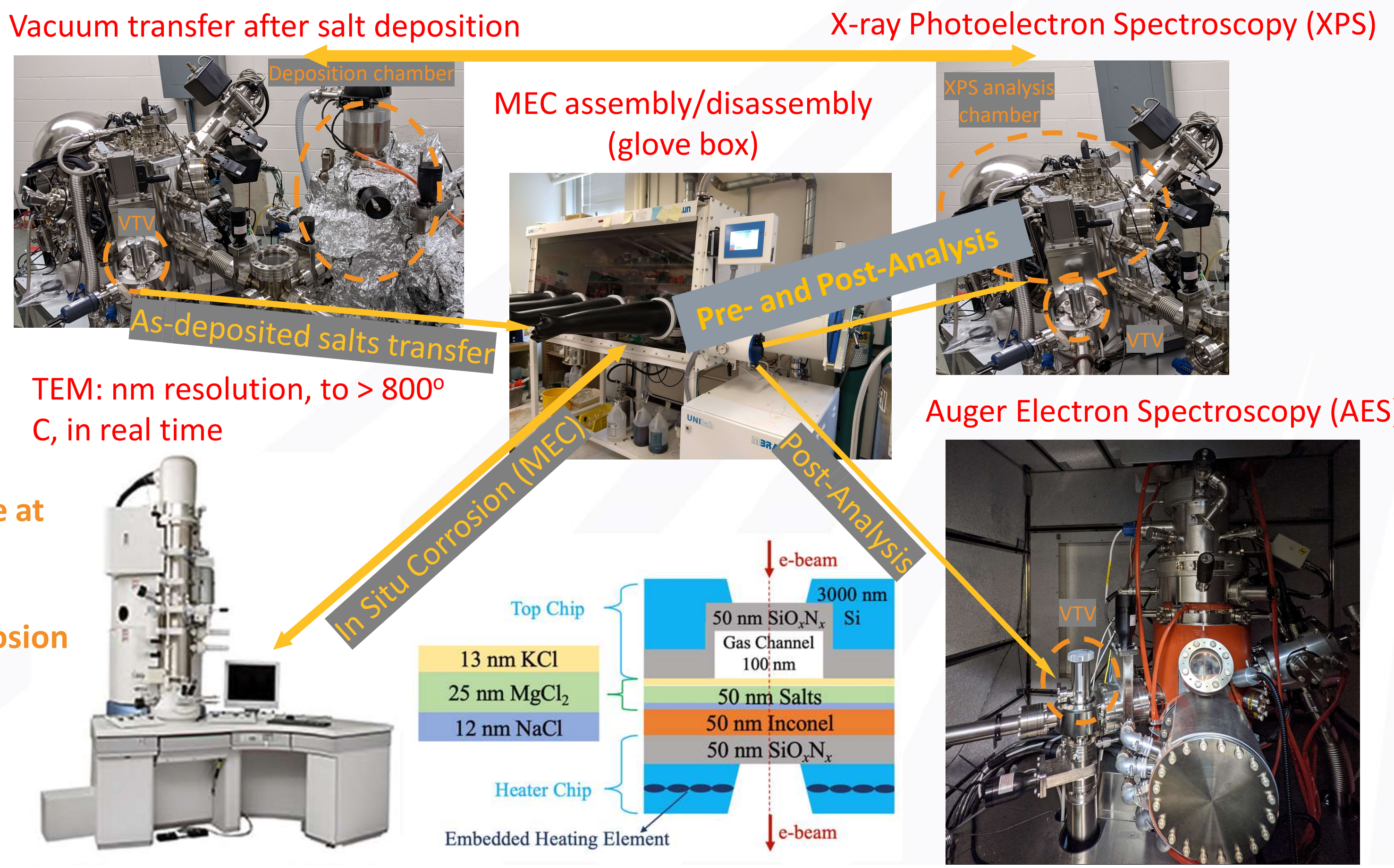




- 5 temperature (600-800°C) & 3 concentration (1.53×10^{-6} - 7.48×10^{-4} mol cm^{-3}). D ranged from 0.94 to 3.31 $\times10^{-5}$ cm^2s^{-1} with a descending order of $Cr^{2+} > Fe^{2+} > Cr^{3+} > Ni^{2+}$.
- We developed the correlation between temperature and the diffusion coefficient: It ascends with the rise of temperature and follows Arrhenius Law.

Ability to deposit, characterize and corrode Inconel / salt mixture under controlled environment (vacuum or controlled gas partial pressure) so that we can elucidate fundamental corrosion mechanisms. Nanoscale spatial resolution, sub second temporal resolution., at temps up to > 800 °C

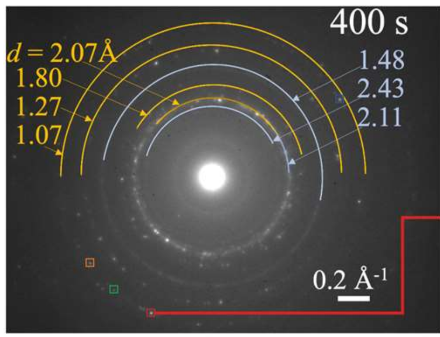
Controlled Environment Experimental Suite at RPI to Elucidate Fundamental High Temperature Corrosion Mechanisms



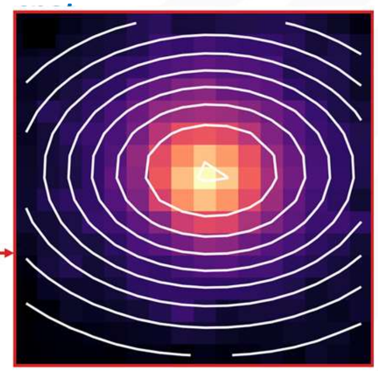
In-situ TEM: Capability of real time measurements of Inconel corrosion by ternary salt mixture at grain-by grain level

Measurements of corrosion rates in-situ to TEM

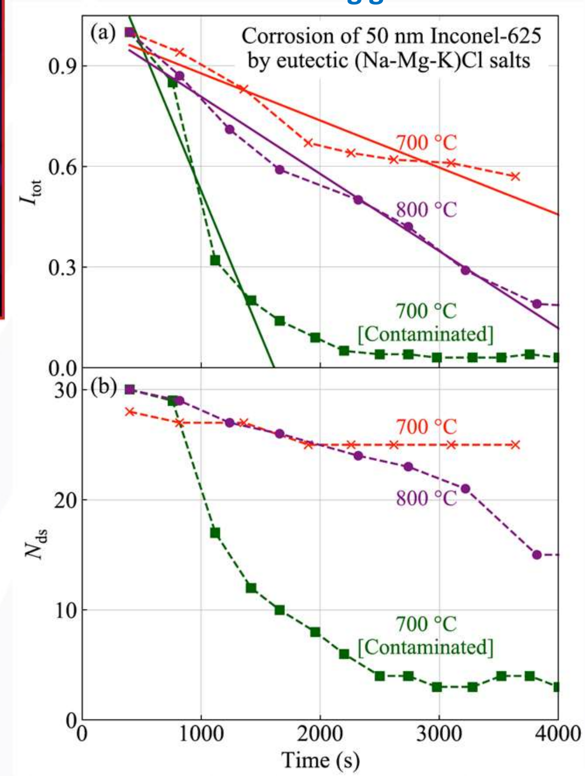
TEM diffraction pattern @700 C (Hydrated)



2D Gaussian fit of a single Inconel diffraction



Loss of Inconel diffraction intensity vs time at 700° C: Top – integrated intensity; Bottom – number of diffracting gains



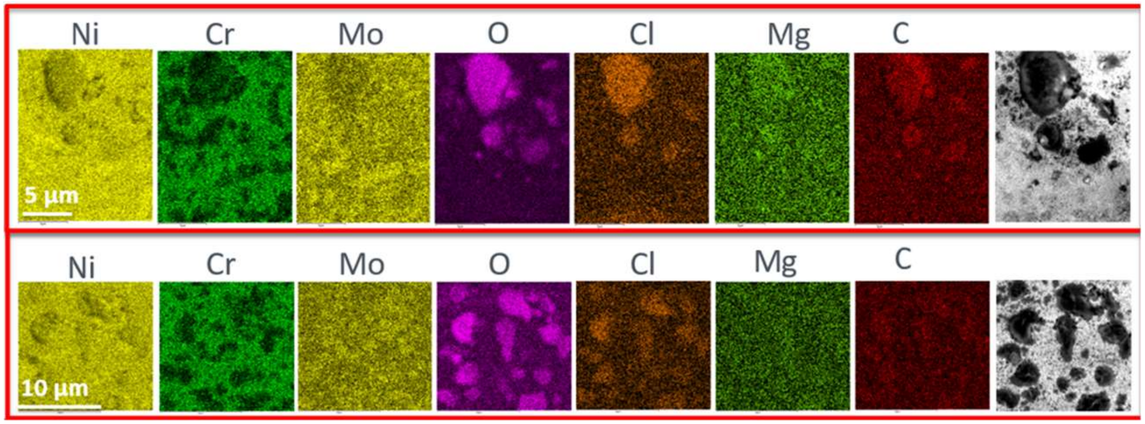
Ternary salt:
44.7 $MgCl_2$ – 25.8 KCl – 29.4 NaCl (mol.%)

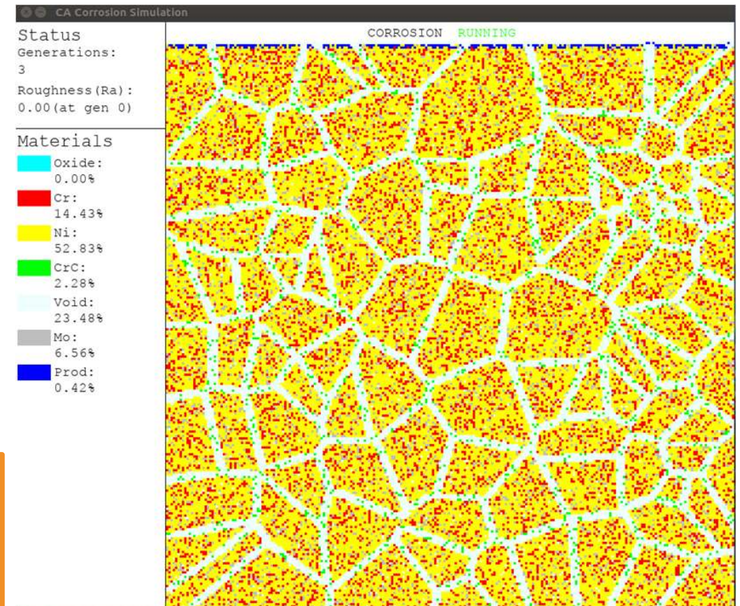
KCl-NaCl-$MgCl_2$ on Inconel

Extrapolation of bulk corrosion rates (hundreds of µm/year) to thin film corrosion rates (tens of nm /hour)

700° C (uncontaminated) → 220 µm/year
800° C (uncontaminated) → 350 µm/year
700° C (air exposed) → 1000 µm/year

Corrosion of Inconel 625 thin films





- ✓ EDS mapping results for Grain size ~ 76.9 nm. Severely attacked by Mg and also rich in Cl and O, resulting in the depletion of Ni and Cr.
- ✓ Grain size of thin films of Inconel 625 grows with the increase of deposition temperature, and film with larger grain size (higher deposition temperature) is more corrosion resistant.

$$MgCl_2 + H_2O \longrightarrow MgO + 2\,HCl$$
$$4\,HCl + O_2 \longrightarrow 2\,Cl_2 + H_2O$$
$$Cr + 2\,Cl_2 \longrightarrow CrCl_4$$

O_2 and H_2O in glove box diffuse into salts and react with salts, and generate magnesium oxides and Cl_2. Cl_2 diffuse into alloy and corrode the film.

START: We are not sure what the corrosion mechanism is and we cannot describe how to stop it.

Discussion

- **Salts:**
  - Mg and Cl are two most seen salt elements in the corroded films, in the form of magnesium oxides, Hydrogen chloride, and Chlorine.
- **Metal:**
  - Grain size of films grows with the increase of deposition temperature, and film with larger grain size (higher deposition temperature) is more corrosion resistant.
  - Cr is the most readily attacked element in the Inconel 625 film; most Energy Dispersive X-Ray Spectroscopy-EDS elemental mappings display Cr depletion (even at 550 °C)
  - Mo is the most corrosion resistant element of Inconel 625 film and is enriched in Cr-depleted area
  - limited protection from Oxide. Oxide type: NiO (weaker protection compared with Cr_2O_3) and very thin: ~2 nm.
  - Therefore increasing the proportion of Mo and decreasing Cr in alloy base may be one direction for material selection. For example: Haynes 242 and Hastelloy N