Corrosion Characterization Using Micro-CT 3D Reconstruction

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Background

Monofrax® K-3, an electro-fused chromium alumina refractory, is used in the joule-heated ceramic melters for Hanford nuclear waste vitrification. Corrosion at the melt line critically limits melter life. A laboratory-scale bubbled crucible melt test methodology was developed to assess corrosion behavior based on glass melt composition, temperature, and duration. Two analytical methods were compared: traditional optical microscopy cross-section analysis and micro-computed tomography (micro-CT) for 3D reconstruction of corrosion depth in 18 corroded samples. Micro-CT demonstrates enhanced sensitivity in capturing asymmetric corrosion behavior, providing valuable insights into K-3 corrosion dynamics.

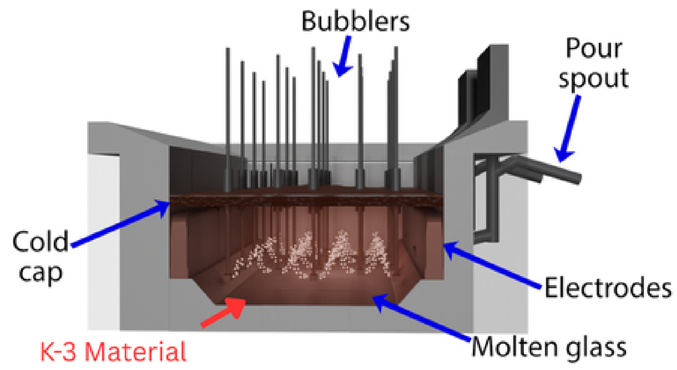

Figure 1. Schematic of joule-heated glass melter. As seen in brown, the K-3 lining is in constant contact with waste glass.

Objective

Compare and quantify variation between two corrosion characterization methodologies.

1. Optical imaging of cross sectioned sample (ASTM C621).
2. Micro-CT scanning of sample -> 3D reconstruction.

Refractory Corrosion Test

Lab scale crucibles are used to submerge a sample in simulated molten waste glass. A test matrix varying temperature, glass composition, and duration of test is evaluated.

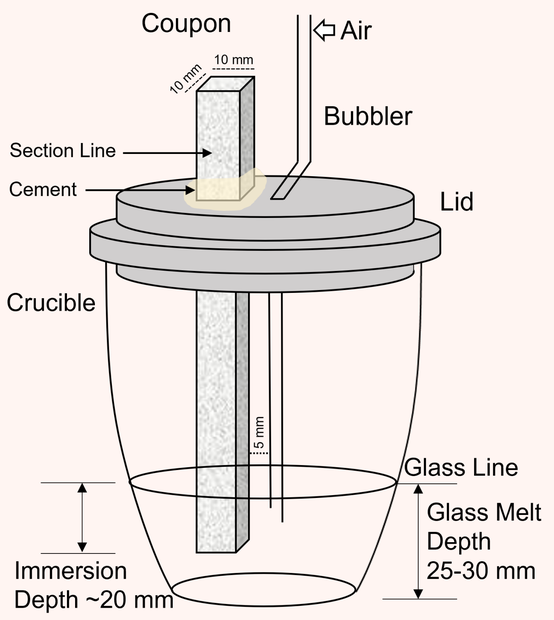

Figure 2. Schematic of laboratory scale crucible test setup.

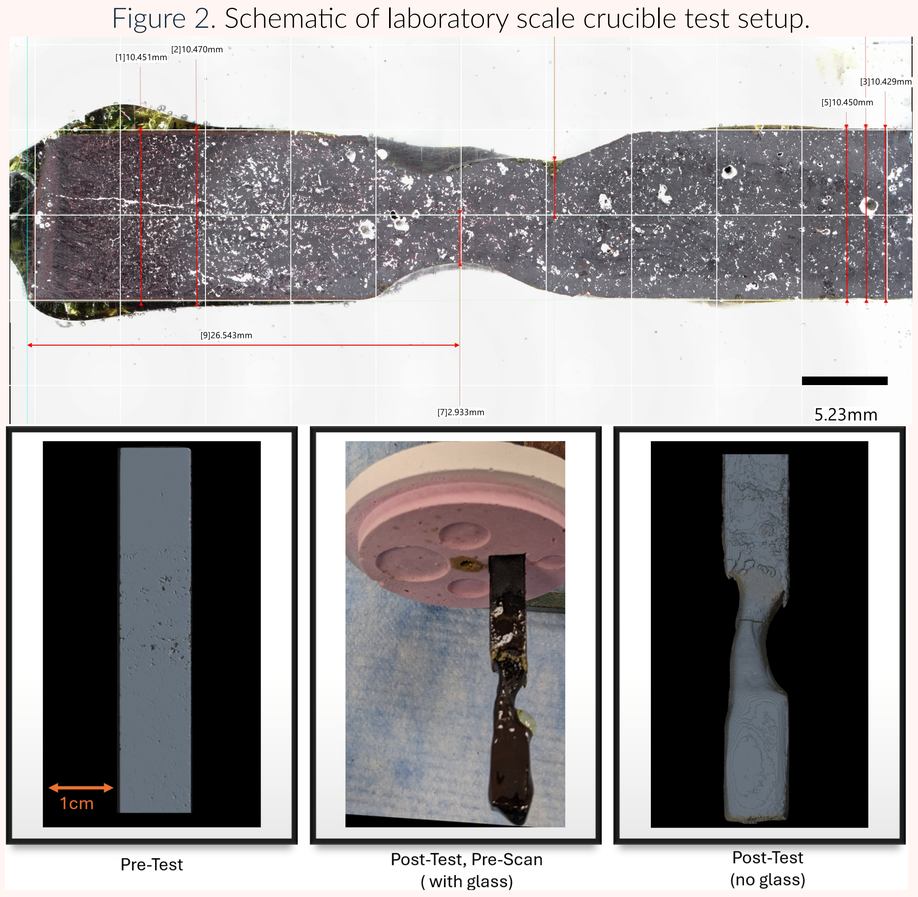

Figure 3. Optical measurement (top) and micro-CT reconstruction (bottom) (LORPM14R1 1150C 3 Day)

Design

1. 18 K-3 samples spanning 7 glass compositions, trial times of 1D-6D, and temperature range $1050C - 1200C$ evaluated.
2. Depth of max corrosion measured via optical and micro-CT methods recorded.

Micro-CT Processing

1. Micro-CT scan captures 2000 slices encompassing $8cm$ of physical sample distance, for a voxel size of $40\mu m$
2. Using vector operations, the individual slices are rotated to be aligned about Z
3. The binary image profile is segmented via a thresholding algorithm to remove glass remnants as seen in Figure 3.
4. The sample edge in each slice is converted to an outline, and bounding boxes are fit to each outline, which are converted to numerical data. The minimum box width is the site at which maximum corrosion has occurred.

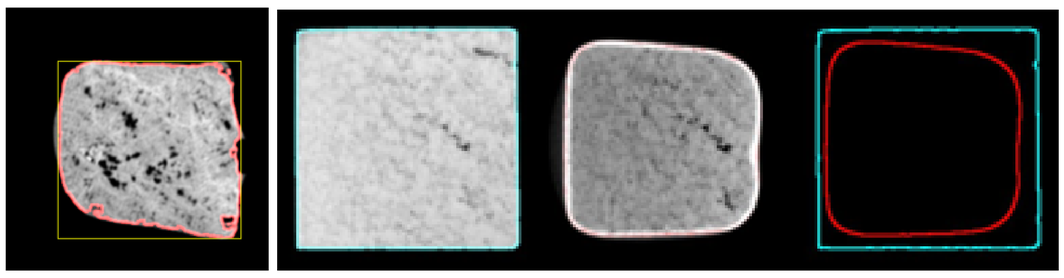

Figure 4. Visual representation of neck depth measurement. Bounding boxes shown at right, pre and post treatment samples shown at middle left and right, respectively.

5. The python library 'PyVista' is used to generate a mesh of the surface of the sample, producing a 3D reconstruction seen in Figure 3.

Optical Imaging Method

- Samples are bisected vertically and mounted in epoxy, and finally the surface is polished.
- Using the width of the coupon, a center line is drawn.
- Due to the bubbling, the corrosion depth (measured as minimum distance from center line) is measured on both bubbling and non bubbling sides. (see optical image in Fig. 3)

Results and Discussion

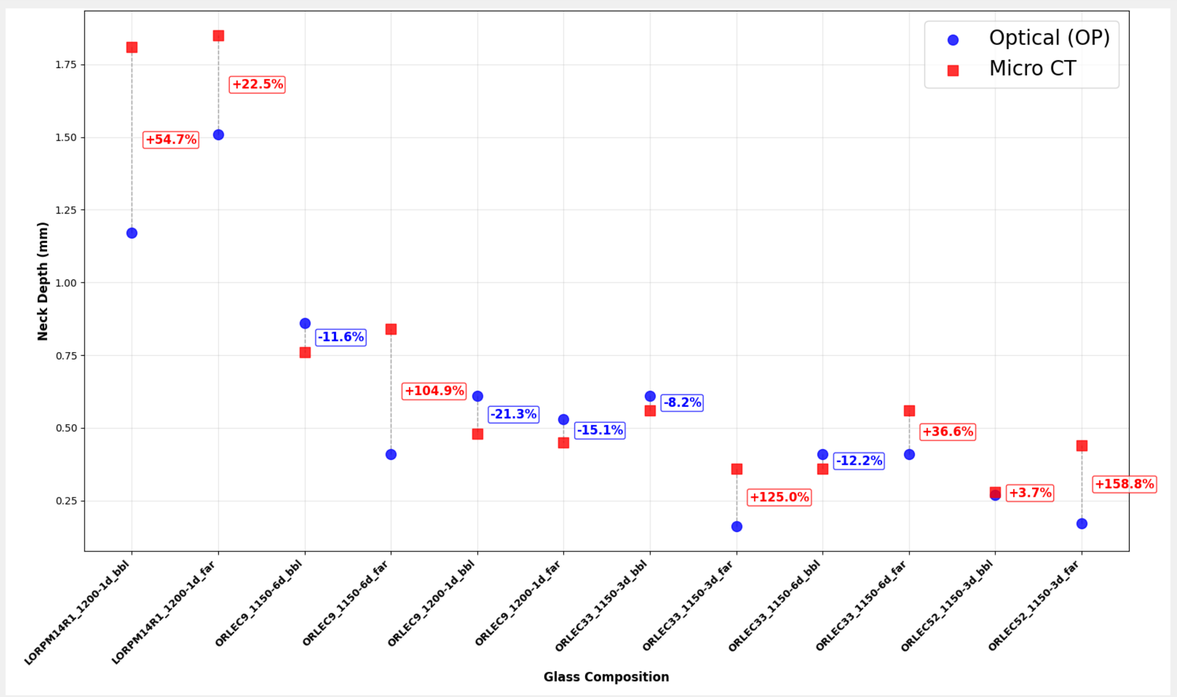

Figure 5. Summarized neck depth data across 12 samples displaying non-trivial corrosion.

- Many samples presented negligible corrosion, likely due to insufficient duration spent in furnace, and lower temperatures.
- There is a 47.9% mean percentage difference across the 12 plotted samples.
- Variation in neck depth is primarily due to the optical method only utilizing a discrete planar neck, whereas the micro-CT method can measure a neck across the entire volume of the sample.
- Further work will involve generation of a larger data set and varied experimental conditions, namely increased temperature and test duration.

References and Acknowledgments

1 Jin, T. et al., 2023. Glass-contact refractory of the nuclear waste vitrification melters in the United States: a review of corrosion data and melter life. International Materials Reviews, 68(8), pp.1135-1157. 2 ASTM C621-09 (2022). Standard Test Method for Isothermal Corrosion Resistance of Refractories to Molten Glass.

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