July 26, 2023

Rebecca Smith
ART Graphite Researcher
Idaho National Laboratory

Oxidation Activities

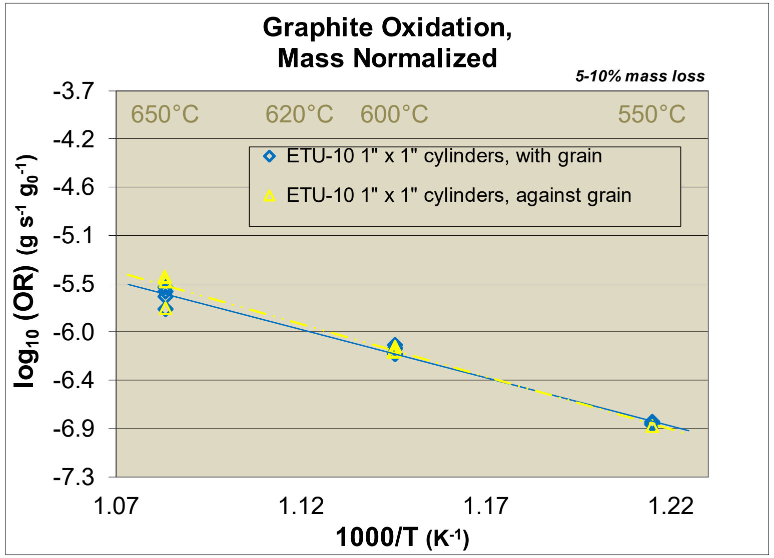
oxidation rate, penetration/lathing, and strength after oxidation work

DOE ART Gas-Cooled Reactor (GCR) Review Meeting
Virtual Meeting
July 25 – 27, 2023

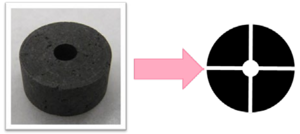
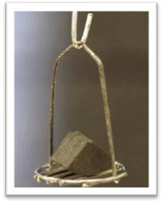
ART
Advanced Reactor Technologies

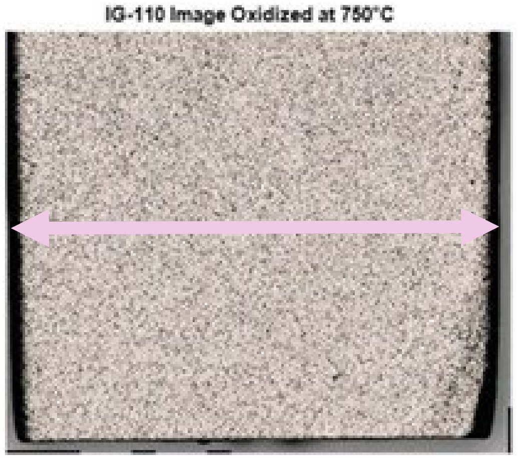
Introduction:
Recent and Current Oxidation Studies

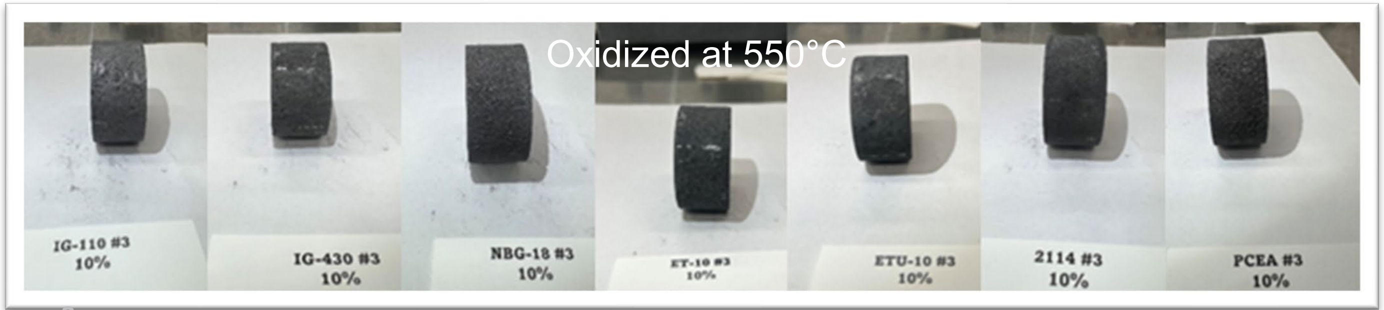
- Background
- 3 Areas of Investigation
  - Arrhenius Rate Analysis
  - Strength after Oxidation
  - Penetration Depth Studies
- Conclusion





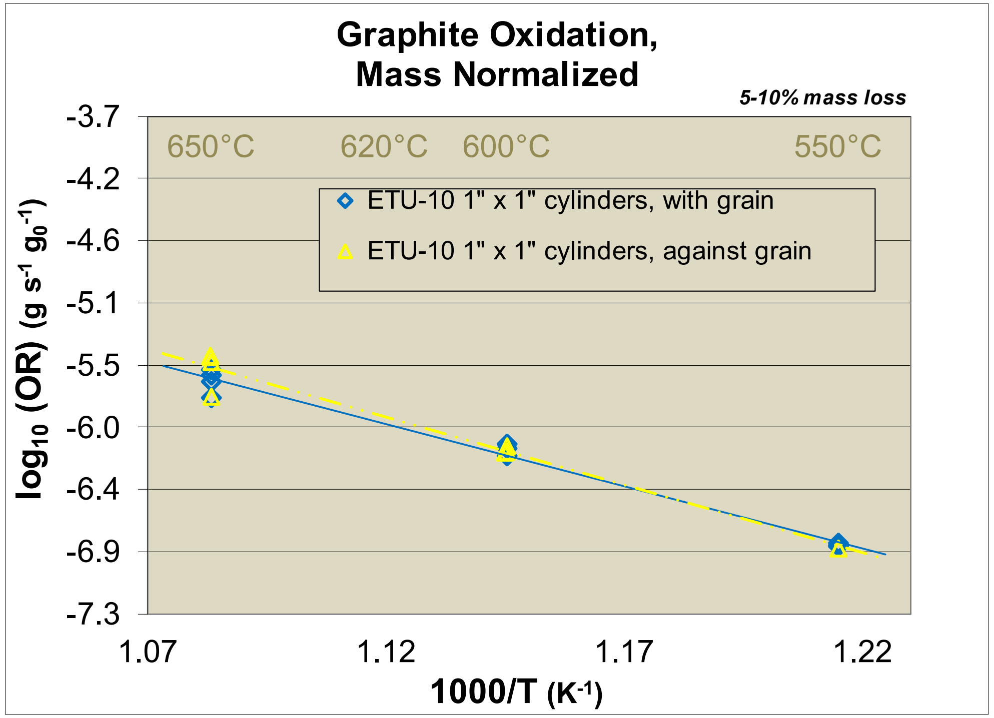




Acute and Chronic Oxidation

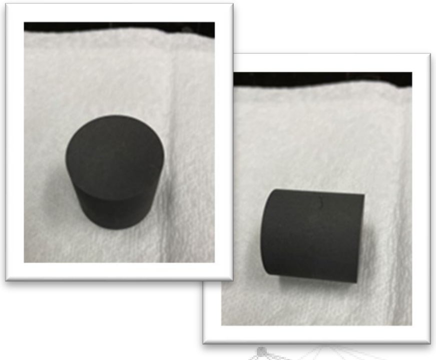
Background – Rate Behavior

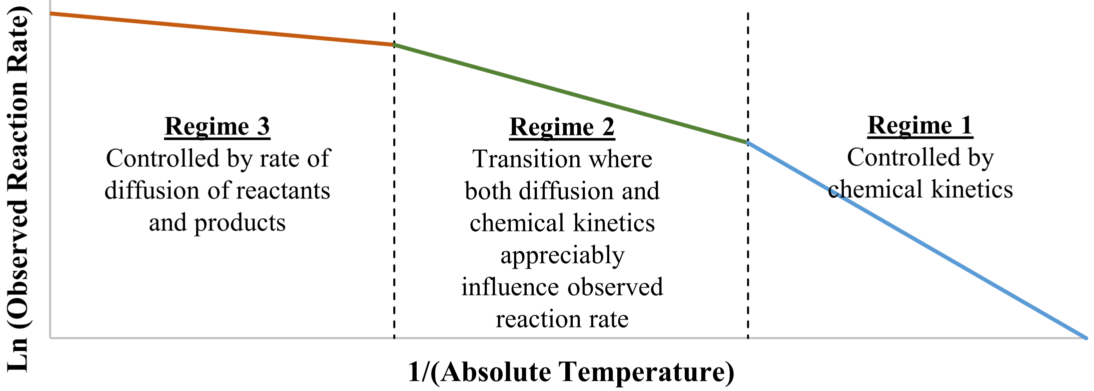
Little (if any) rate difference observed in WG v. AG data to date – *maybe* slight effect at higher T with increase in diffusion influence



- Arrhenius Rate Analysis:
  - ASTM D7542 Assumes Regime 1 up to ~750°C
  - Dependent on the graphite grade microstructure
  - Uniform oxidation presumed

Arrhenius Relationship
$k = A \bullet \exp(-E_a/RT)$

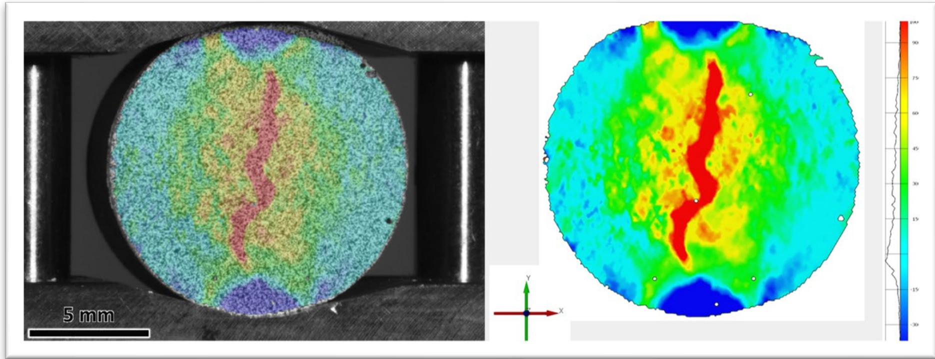


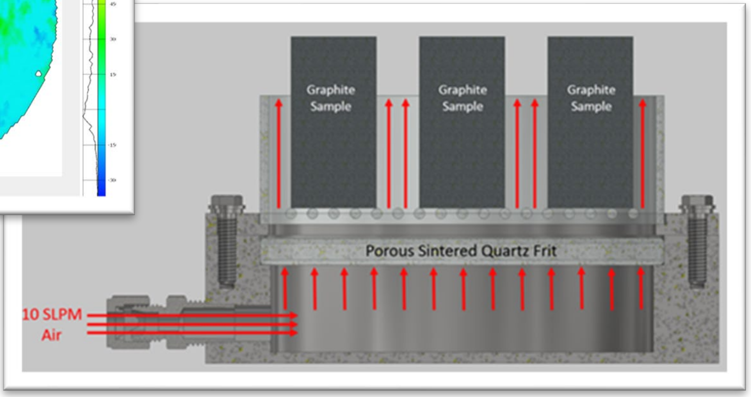


Temperature Dependence at Constant Air Flow Rate

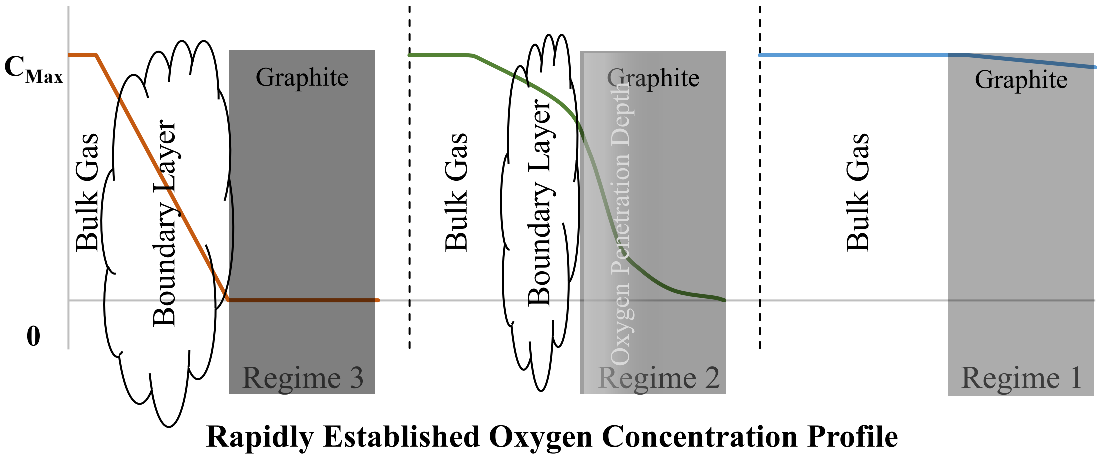
Adaptations
- to investigate effects of irradiation
- to induce oxidation resistance

Background – Strength Behavior

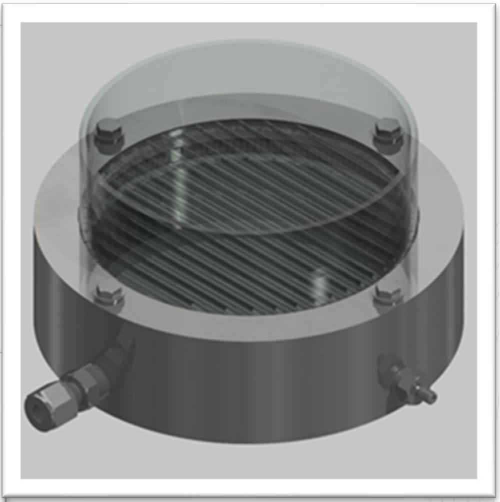




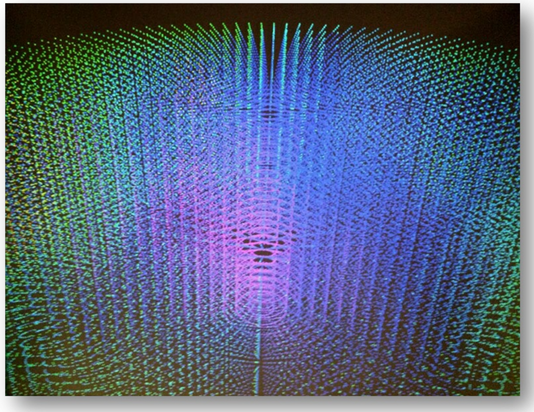
- Implications for Modeling, ASME Code, Testing Standards:
  - Graphite performance changes with oxidation
  - Dependent on the graphite microstructure <u>and</u> gas transfer
  - Local variations in conditions → non-uniform oxidation at all temperatures tested



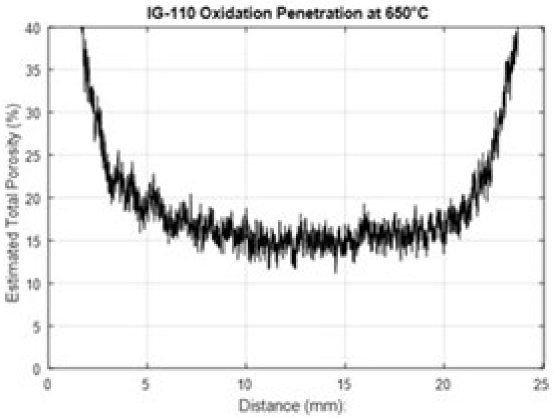
Theoretical Infinite Slab

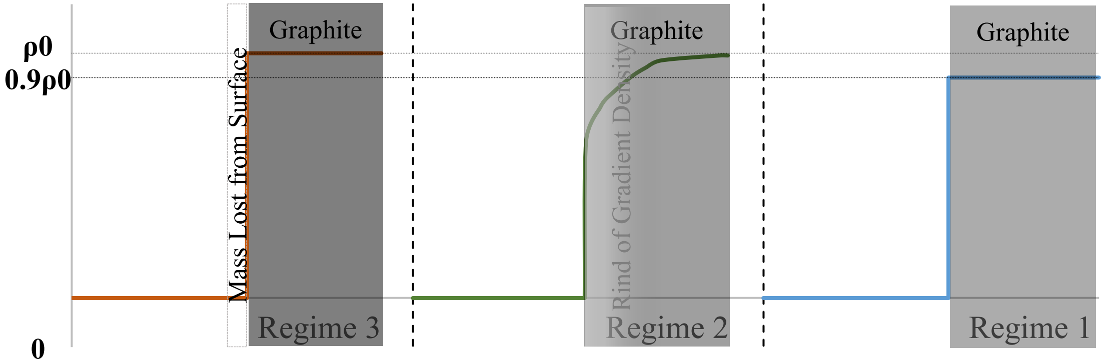


Background – Density Profile



- Observed Damage from Oxidation
  - Strong temperature dependence
  - Regime 2 density gradient over all temperatures tested
  - Inconsistent post-oxidation properties at (moderate) T = 650°C





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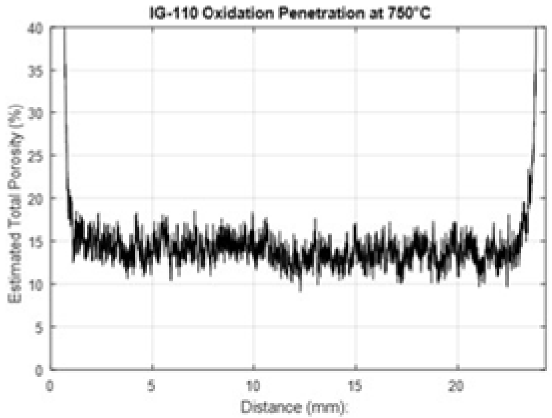


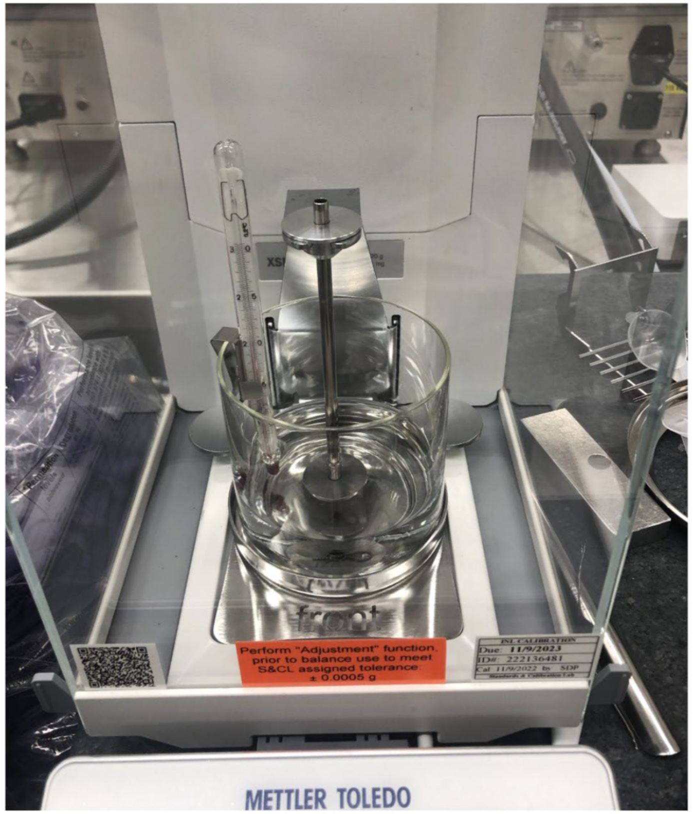
Density Profile is essential to understanding results of post-oxidation strength tests

Penetration Measurements (aka Lathing Study)

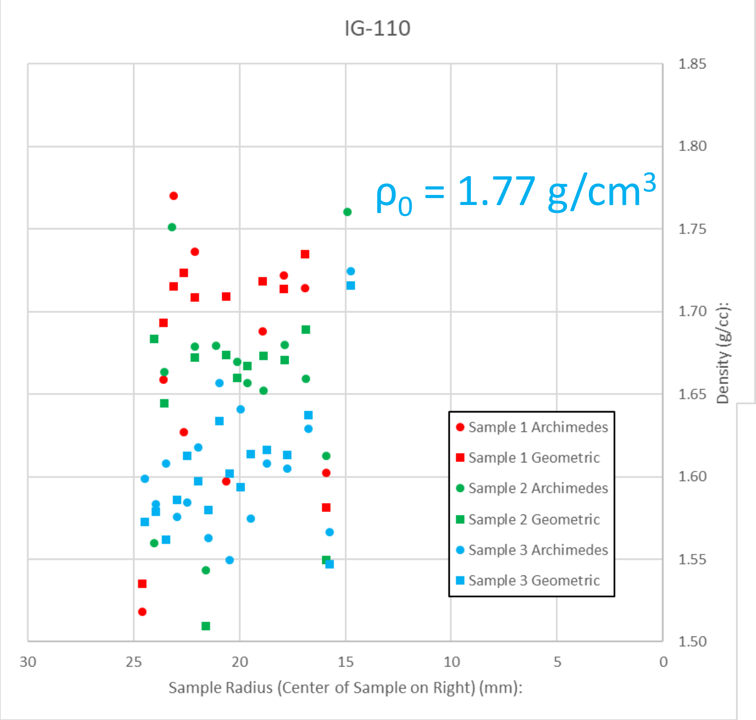
- 3 mass loss values: 2%, 6%, and 10%
- 550°C oxidation temperature
- Hole in sample center for precision machining
- 50 mm initial sample diameter
- Archimedes and geometric density analysis

Machined in 1 mm and 2 mm steps

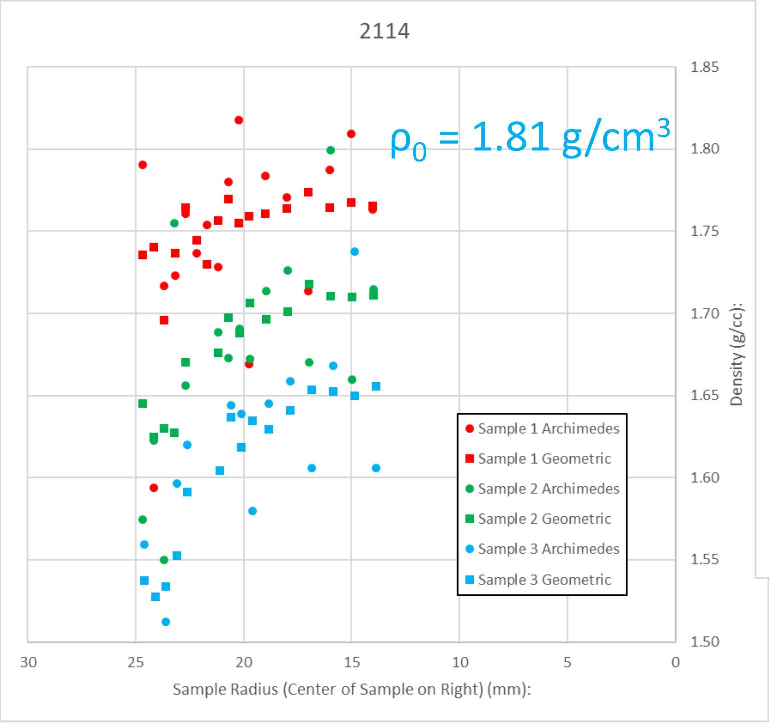
Archimedes measurements follow ASTM C20, “Standard Test Methods for Apparent Porosity, Water Absorption, Apparent Specific Gravity, and Bulk Density of Burned Refractory Brick and Shapes by Boiling Water.”

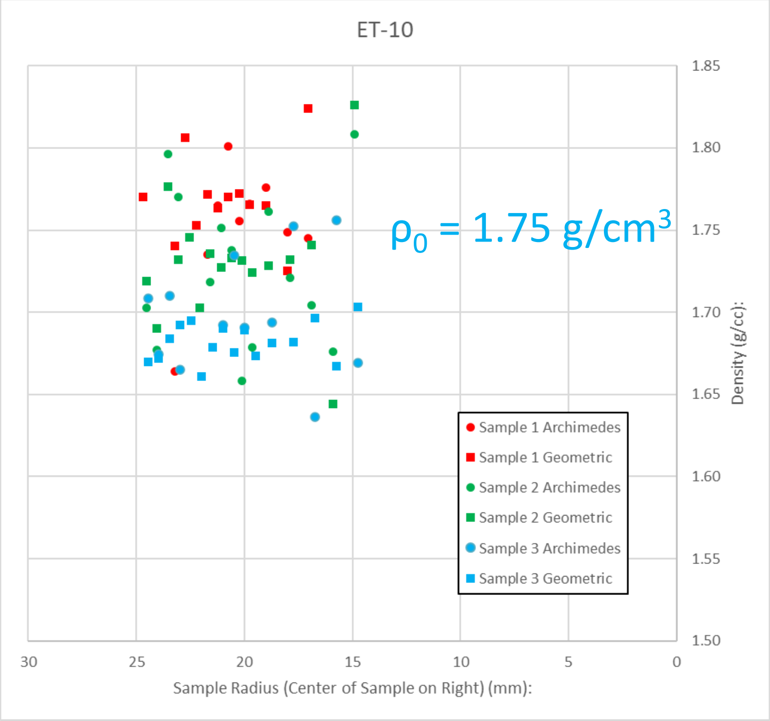


Penetration Measurements



With 18 Machining Steps Completed

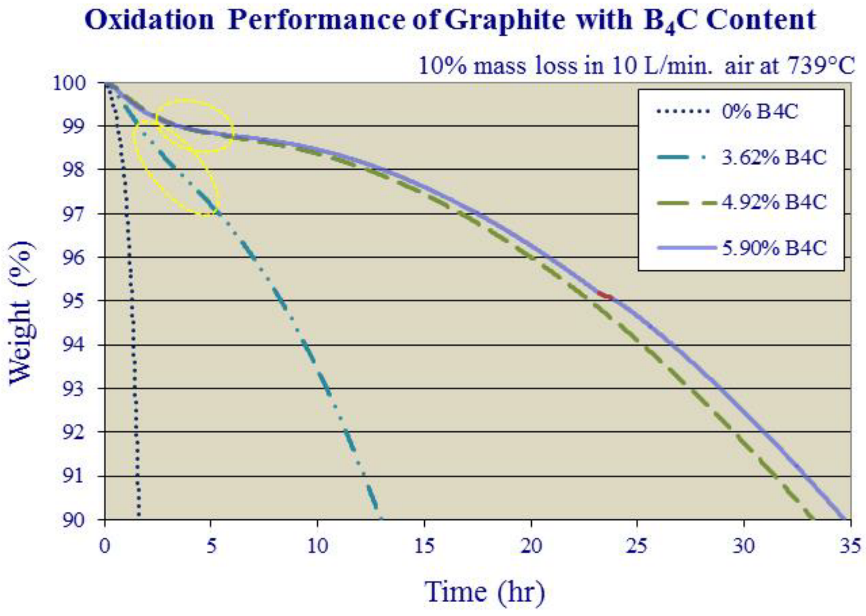


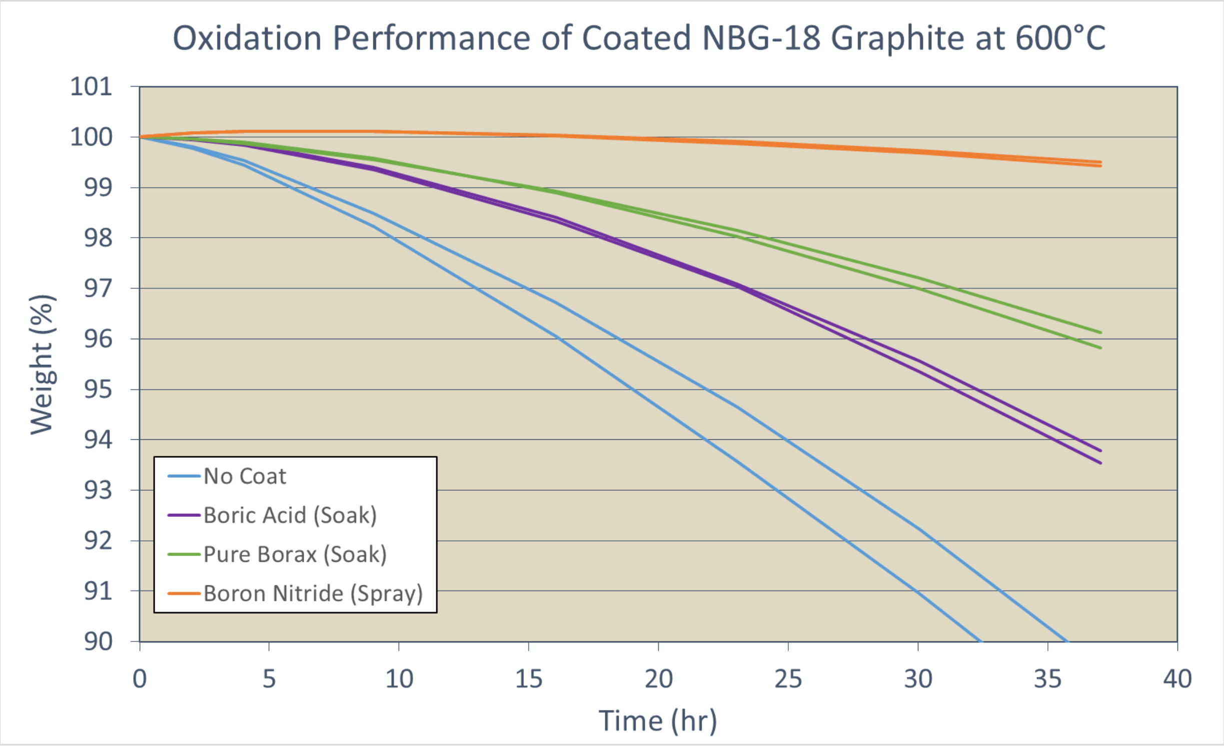


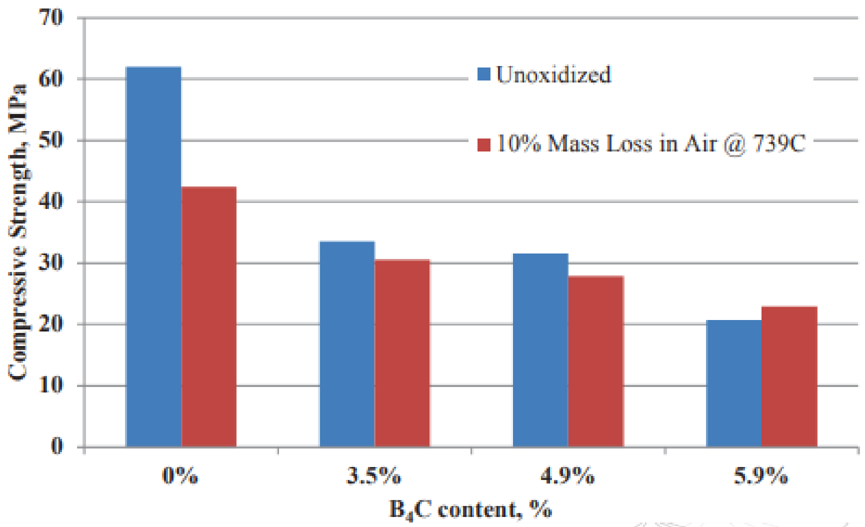
Mass Loss From Oxidation:
Sample 1, 2%
Sample 2, 6%
Sample 3, 10%

Oxidation Resistant Graphite Coating Development

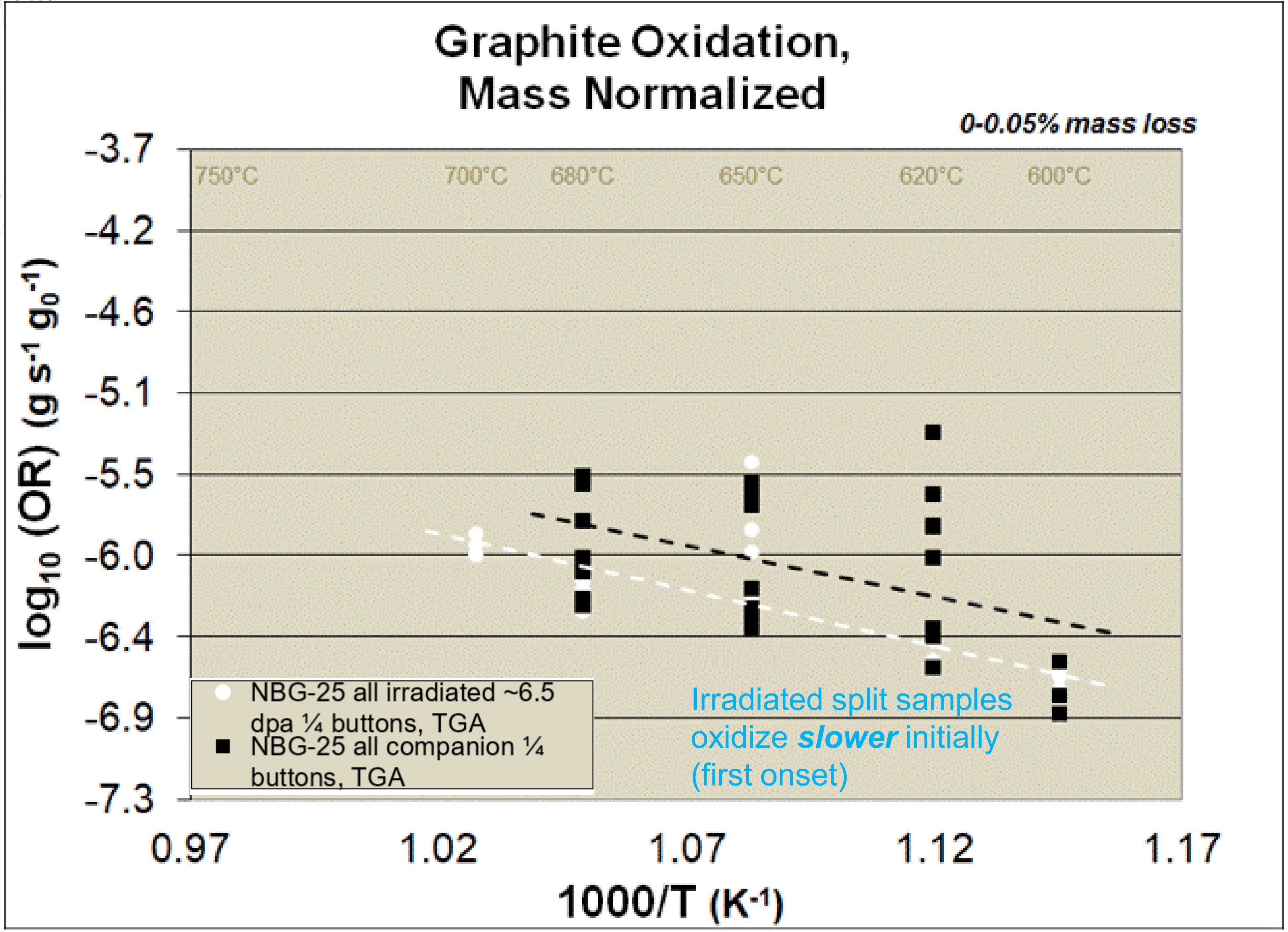
- Why Coat:
  - Increased resistance to oxidation
  - Manufacturing limitations
  - Maintain graphite strength



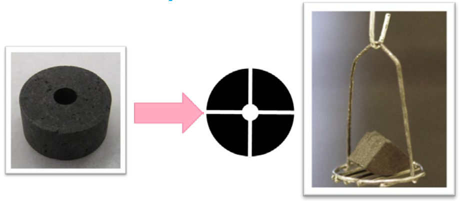


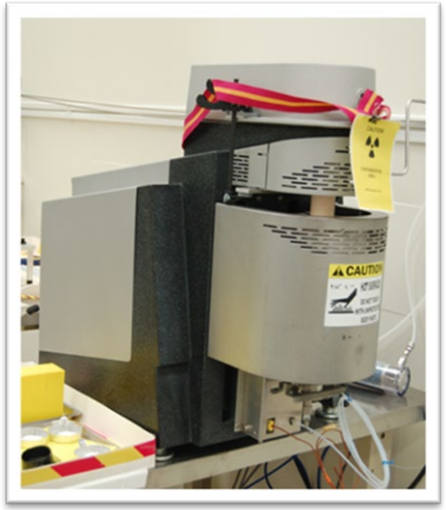


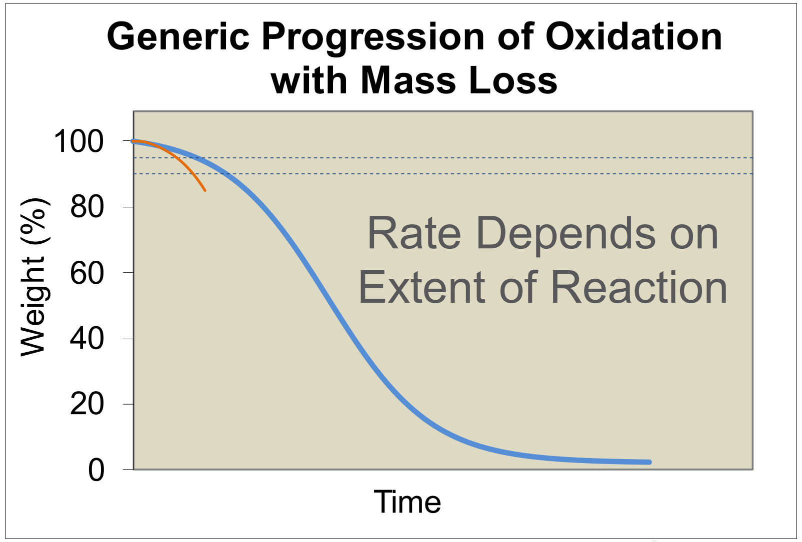
Irradiated v. Unirradiated Graphite Oxidation - Rate Behavior



Irradiated split sample oxidation ***accelerates faster*** with reaction progression, 2X or 3X at the 5-10% mass loss at ~6.5 dpa

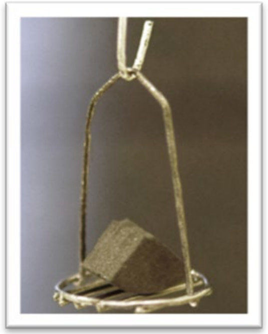


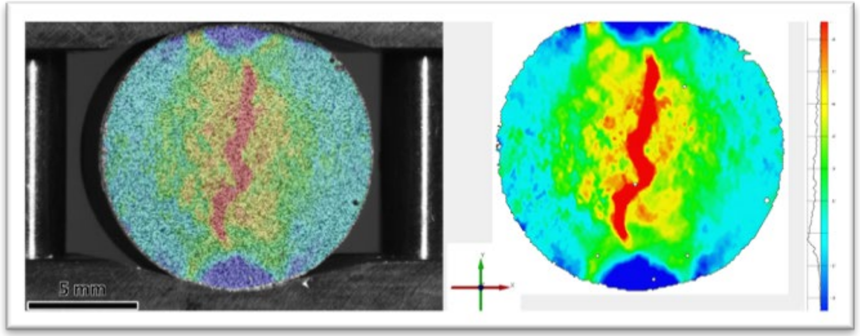


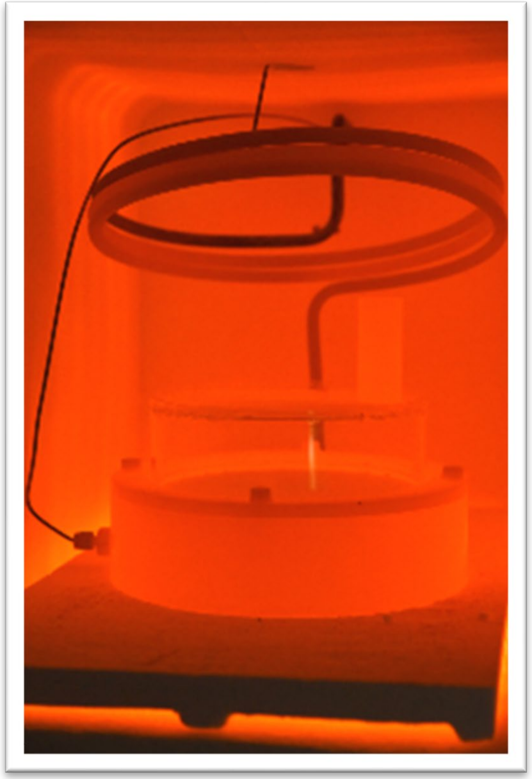


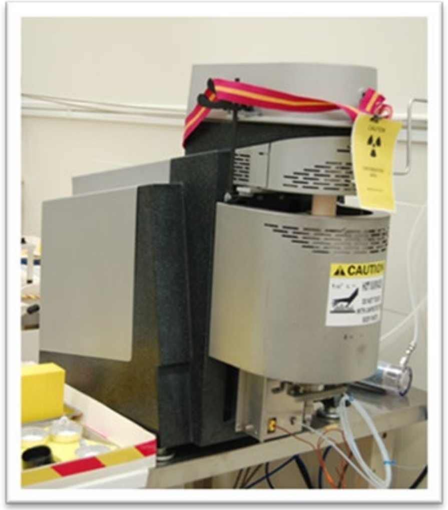
Conclusion:
Oxidation Studies Moving Forward

- 3 Areas of Investigation
- ASTM Standards in Development
- Documentation to support ASME Code









Recent & Pending Publications

- Smith, R., and W. Windes, "Performance of Graphite Oxidation with Environment and Specimen Geometry Variations," http://doi.org/10.1520/STP163920210134, ASTM International book on the Selected Technical Papers (STP 1639) *Graphite Testing for Nuclear Applications: The Validity and Extension of Test Methods for Material Exposed to Operating Reactor Environments* (ISBN:978-0-8031-7725-3) December 2022.
- Paul, R., C. Contescu, and N. Gallego, "A Microstructural Modeling-Based Approach to Graphite Oxidation Beyond ASTM D7542," http://doi.org/10.1520/STP163920210080, ASTM International book on the Selected Technical Papers (STP 1639) *Graphite Testing for Nuclear Applications: The Validity and Extension of Test Methods for Material Exposed to Operating Reactor Environments* (ISBN:978-0-8031-7725-3) December 2022.
- Paul, R., C. Contescu, N. Gallego, R. Smith, J. Bass, J. Kane, A. Tzelepi, and M. Metcalfe, "On the thermal oxidation of nuclear graphite relevant to high-temperature gas cooled reactors," https://doi.org/10.1016/j.jnucmat.2022.154103, 573 (2023) *Journal of Nuclear Materials*.
- Cai, L., R. Smith, A. Matthews, D. Cottle, W. Chuirazzi, F. Xu, B. Gross, C. Chen, R. Latta, and W. Windes, "Determining the Oxidation Behavior of Matrix Graphite," submitted in June 2023 to *Journal of Nuclear Materials* for peer review.

Questions?

Rebecca Smith **Idaho National Laboratory**

rebecca.smith@inl.gov (208) 526-3874

Idaho National Laboratory