

Oxidation Resistant Graphite

July 2024

Timothy David Bragg, Michael Charles Barkdull





DISCLAIMER

This information was prepared as an account of work sponsored by an agency of the U.S. Government. Neither the U.S. Government nor any agency thereof, nor any of their employees, makes any warranty, expressed or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness, of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. References herein to any specific commercial product, process, or service by trade name, trade mark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the U.S. Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the U.S. Government or any agency thereof.

Oxidation Resistant Graphite

Timothy David Bragg, Michael Charles Barkdull

July 2024

Idaho National Laboratory Idaho Falls, Idaho 83415

http://www.inl.gov

Prepared for the U.S. Department of Energy Under DOE Idaho Operations Office Contract DE-AC07-05ID14517



Wednesday, July 17, 2024

Oxidation Resistant Graphite

Tim Bragg and Michael Barkdull



DOE ART GCR Review Meeting

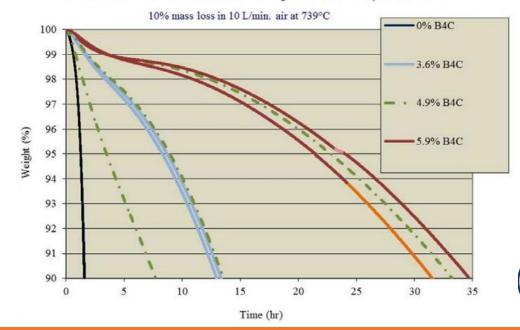
Hybrid Meeting at INL

July 16-18, 2024

Introduction

- Current Research:
 - Coat graphite with boron based materials to resist oxidation
 - Understand the coating mechanics
- Prior research:
 - Abnormally low rates of oxidation observed
 - Cause determined to be boron based doping
 - Prompted current research in oxidation resistance

Oxidation Performance of Graphite with B₄C Content





Why we are doing this

- Possibility of Air-Ingress Accident Scenarios puts reactors components at risk of oxidation
 - Oxidation reduces mechanical strength of graphite, compromising reactor core components
 - At high temperatures, graphite can quickly be compromised
 - Work in oxidation seeks to understand and mitigate risks of oxidation in reactors



Original work

- 2010's study on oxidation resistance
 - Graphite dopped with boron carbide
 - Graphite dopped in manufacturing
- Boron Carbide reduced mechanical strength loss after oxidation
- As fabricated strength decreased nearly 50% when dopped over 3.6%
- Strength decrease after oxidation was only 11% compared to 30% when not dopped

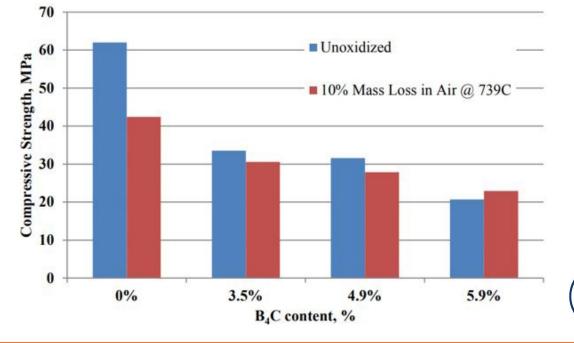


The benefits of doing this work

Oxidation decreases mechanical properties of graphite

 In the case of an air-ingress accident high oxidation can cause the core of the reactor to become structurally

unstable





Things decided last summer

- Use simple, post graphitization introduction of boron using a boron based coating
- Factors considered:
 - Source of Boron
 - Concentration
 - Heat treatment time

- Mechanism of application
- Sequential applications
- Heat treatment temperature



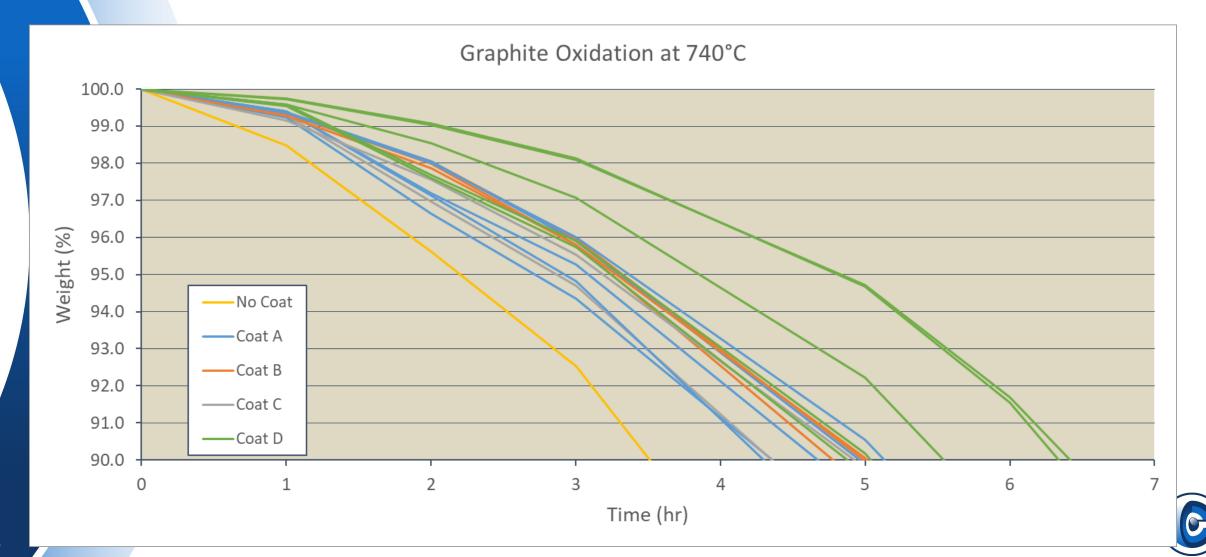
The work last summer

- What works and what stands out
- Wide Sweep
 - Various settings for the factors
 - Oxidized at 740 °C
- Initial imaging via SEM-EDS:
 - Attempt to determine boron content and location
 - Layered coating vs graphite structure
 - No clear indication of boron seen.



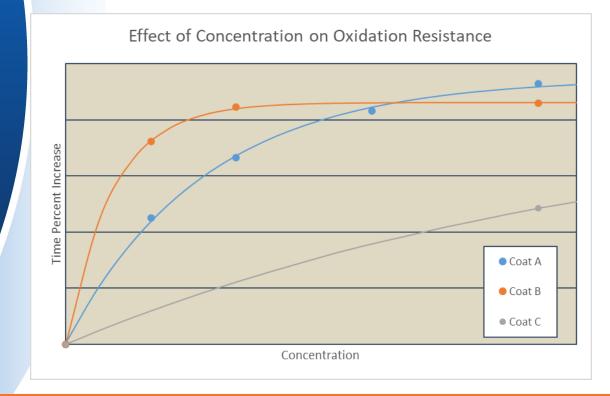


The results: Oxidation times



The results: Patterns

Time increase has an apparent exponential trend with concentration



 Difference in the oxidation distribution



Uncoated

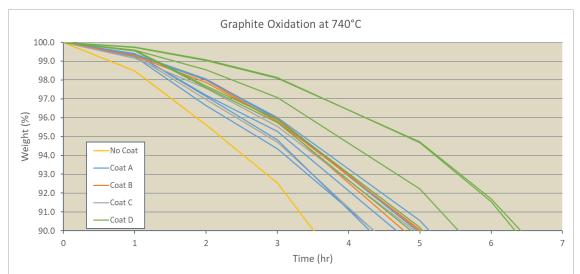


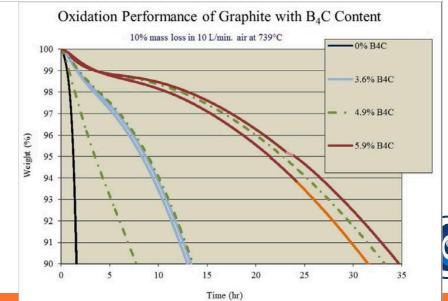
Coated



What we learned from the results

- All coating methods worked
- Option D seems to be the best option
- Has the ability to be scaled
- The results were in line with previous beliefs







What we are doing this summer

- We have set up a controlled application process
- Collecting data to feed into a computational model
- Investigating various with coating D compositions



The next steps

- Oxidation rate reduction achieved, need to determine why and how it works and need to determine what are the consequences:
 - Coating Characterization
 - Mechanism Determination
 - Strength Testing
- With this information the coating process can then be optimized and scaled up

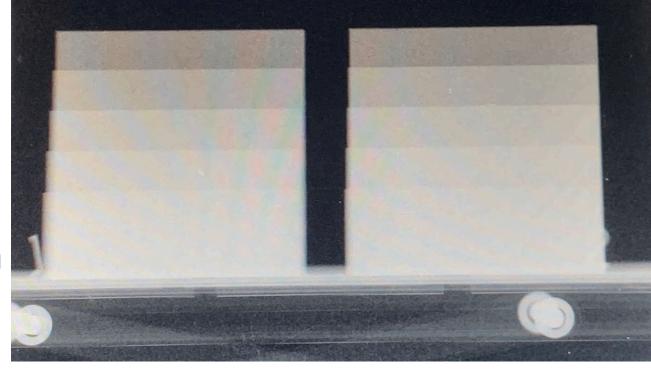


Imaging

 We are working with OSU to gather neutron imaging



- Initial boron content
- Boron burn off
- Boron penetration
- Final boron content





GRADE

$\times 10^{-3}$ 2114 **IG-110 IG-430 NBG-17 NBG-18 PCEA** 9.30 4.62 3.26 1.70 1.10 0.87 0.60 1.24 0.23 0.17 0.28 0.28 Min 8.45 2.68 2.84 1.47 0.85 0.51 10.27 5.62 3.72 2.02 Max 1.77 1.21 10.25 5.18 3.72 2.29 1.13 1.37 0.57 1.14 0.23 0.12 0.13 0.49 9.13 3.41 2.08 0.87 Min 3.24 0.60 11.02 6.15 4.11 2.54 1.30 2.16 Max

Modeling

- Working to set up parametric study
 - Boron concentration & Thickness
 - Heat treat & oxidation temp

 $D_{eff N2}$

 \overline{D}_{N_2Ar}

 $D_{eff\ Ar}$

 D_{ArN_2}

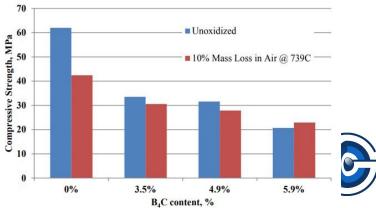
- Heat treatment & oxidation time
- Diffusion & concentration



Strength work

- Concern in oxidation stems from reduced graphite strength
- Two strength measures/tests will be explored:
 - 1. Split Disc
 - 2. Compressive
- Questions to be answered:
 - Is strength compromised due to the coating process?
 - Is there a shift in where the graphite is weak?





Conclusion

- Our efforts to mitigate graphite oxidation through advanced coating technologies
 - Initial research indicates promising results
 - Coating shows similar results to doped in fabrication
 - Goals:
 - Optimize these coatings by leveraging mathematical models
 - Conducting mechanical testing
 - Employing imaging techniques





ADVANCED REACTOR
TECHNOLOGIES PROGRAM

Thank you

Questions?

Thanks to Contributors:

W. Windes, R. Smith, A. Matthews, T. Yoder, A. Cunningham, M. K. Ames, J. Rufner, A. Salvador **Tim Bragg**

timothy.bragg@inl.gov



