U.S. DEPARTMENT OF ENERGY | Office of NUCLEAR ENERGY

OAK RIDGE National Laboratory

Molten Salt Reactor PROGRAM

Graphite Salt Studies

Nidia C. Gallego
Oak Ridge National Laboratory

Annual MSR Campaign Review Meeting 16-18 April 2024

Purpose:
Assist in the near-term deployment of MSRs by help defining the safe working envelope for nuclear graphite

We acknowledge Financial Support from Advanced Reactor Technology Program – DOE-NE

This research used resources at the High Flux Isotope Reactor, a US DOE Office of Science User Facility operated by the ORNL

Current Research Focus of Graphite-Salt Studies at ORNL:

- Understanding salt intrusion (***penetration depth and salt distribution***) in a wide range of graphite grades (various microstructures) as a function of temperature, pressure and time.
- Studying wetting behavior of salt on graphite surfaces to develop predictive models for salt intrusion
- Studying wear and erosion behavior of graphite in molten salt
- Working with the **ASTM** community to develop standards to measure the effect of salt intrusion on graphite properties
- Working with the **ASME** Community to develop the needed knowledge to address the gaps in the ASME code.

Team Effort – Contributors

Nidia Gallego

Jisue Moon

Cristian Contescu

Yuxuan Zhang

Jim Keiser

Tomas Grejtak

Lianshan Lin

David Arregui-Mena

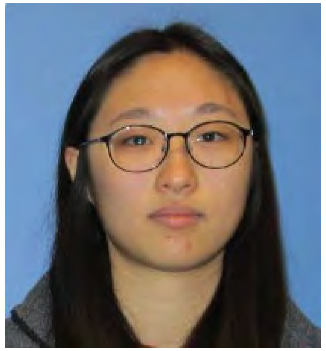
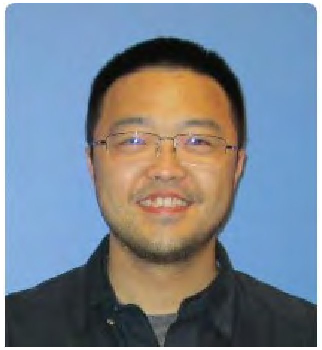
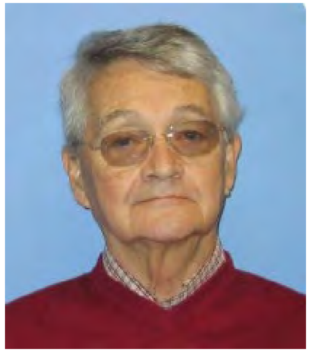
Adam Willoughby

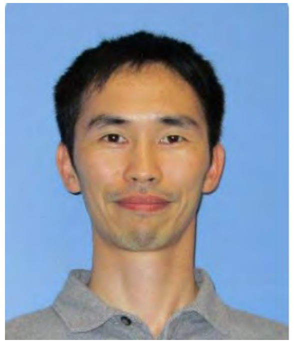
Jun Qu

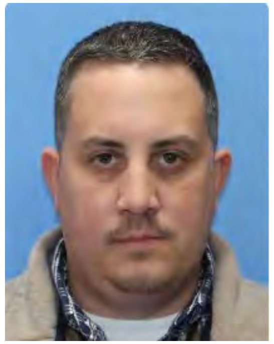
J. Wilna Geringer

New postdoc will join in a few weeks

Many others internal and external collaborators



Impact / Accomplishments

- 8 ORNL Technical Reports
- 5 Journal Publications
- 1 book chapter
- Many abstracts and presentations

- Participation in ASTM and ASME committees

Intrusion Studies

- Continued the use of neutron imaging to study the effect of P, T and t on salt penetration and distribution for several graphite grades
- Continued contact angle measurements and currently evaluating the effect of graphite surface properties, temperature and time on wetting behavior.

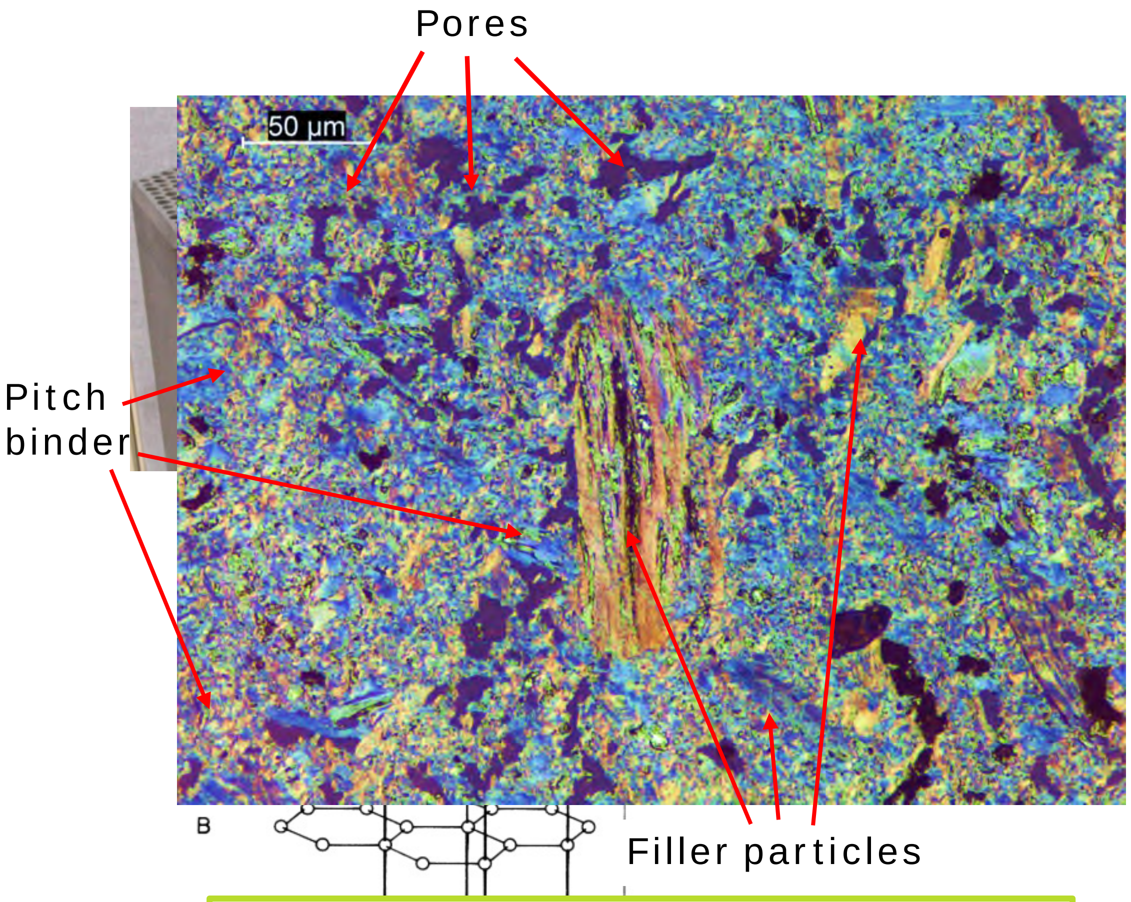
Wear Studies

- Completed proof of principle studies of graphite pin on SS flat
- Commissioned glovebox and tribometer;
- Initiated graphite on graphite wear testing under controlled environment

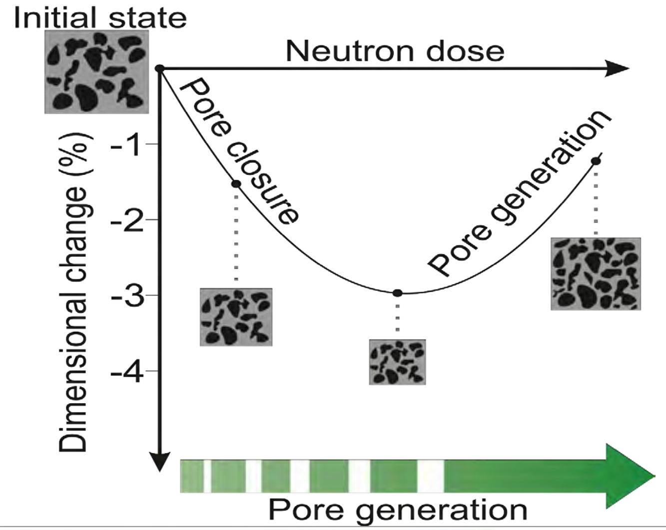
ASME / ASTM

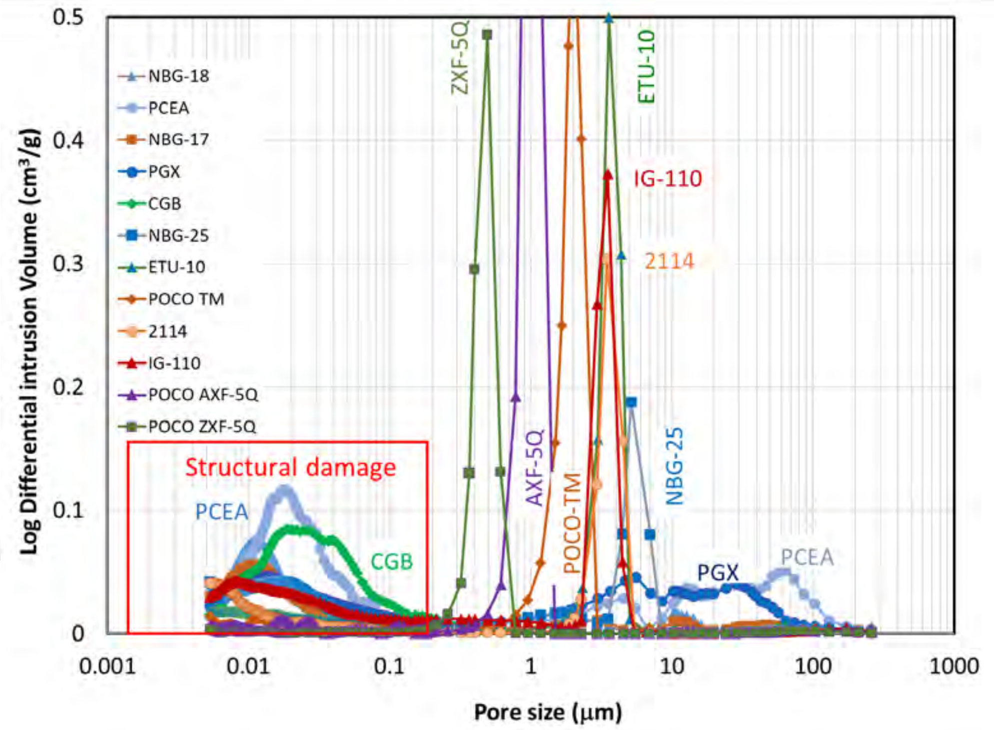
- Participation in ASME
- Participating in ASTM

Manufactured Graphite and its Porous Structure



Manufactured Graphite has about 20 % porosity





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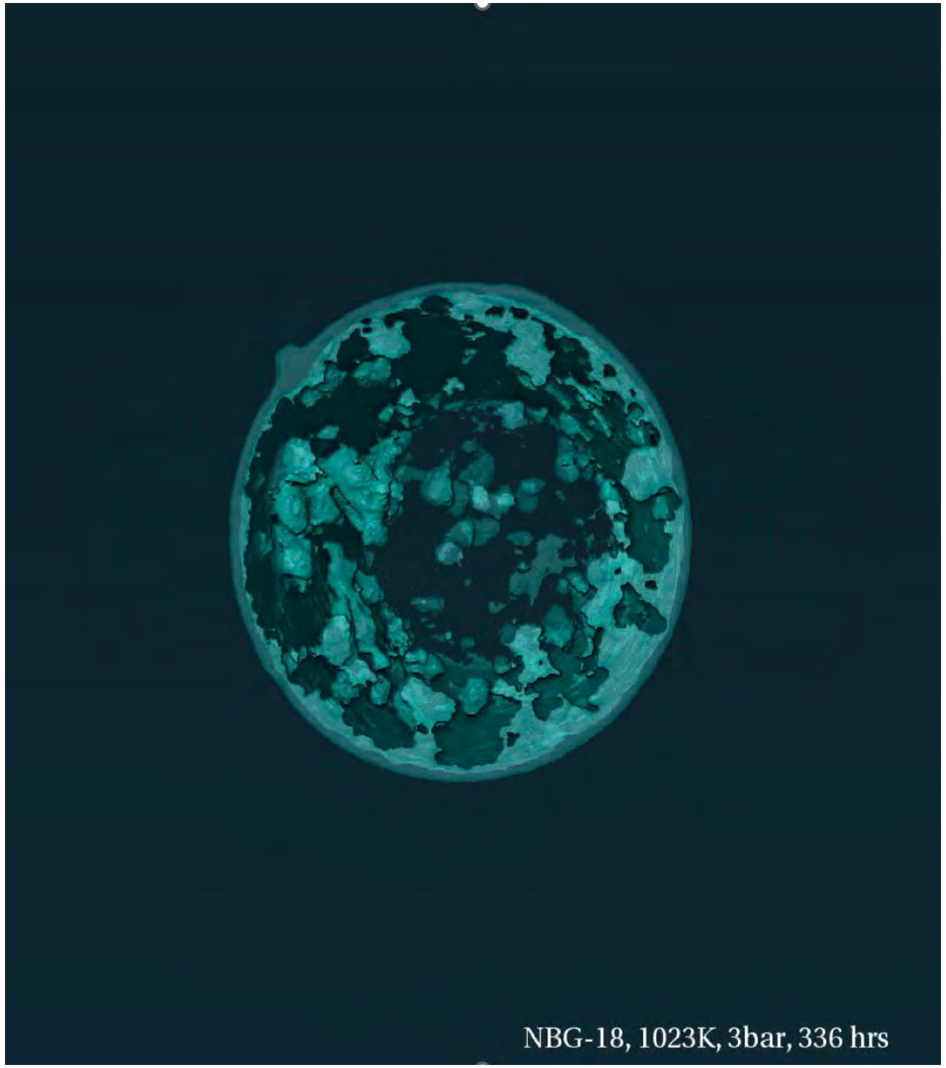
What does Porosity in Graphite Mean to MSRs?

- **Salt intrusion into pores?**
- **Effect of that salt intrusion on graphite properties? (mechanical, thermal)**
- **Chemical Interaction between salt and graphite?**
- **Edge sites and porosity for tritium retention of fp retention?**

Understanding salt intrusion and salt wetting behavior on graphite surfaces

Salt intrusion into graphite porous structure



- Built capabilities for salt intrusion studies (FLiNaK, < 10 bar, < 750ºC) and conducted measurements on a wide range of graphite grades and intrusion conditions.
- Demonstrated and implemented the use of neutron imaging to study intrusion and determine salt penetration and distribution.
- On-going studies of the effect of **<u>P</u>**ressure, **<u>T</u>**emperature and **<u>t</u>**ime for a wide range of graphite grades/microstructures.

Carbon 213 (2023) 118258

Contents lists available at ScienceDirect

Carbon

journal homepage: www.elsevier.com/locate/carbon

ELSEVIER

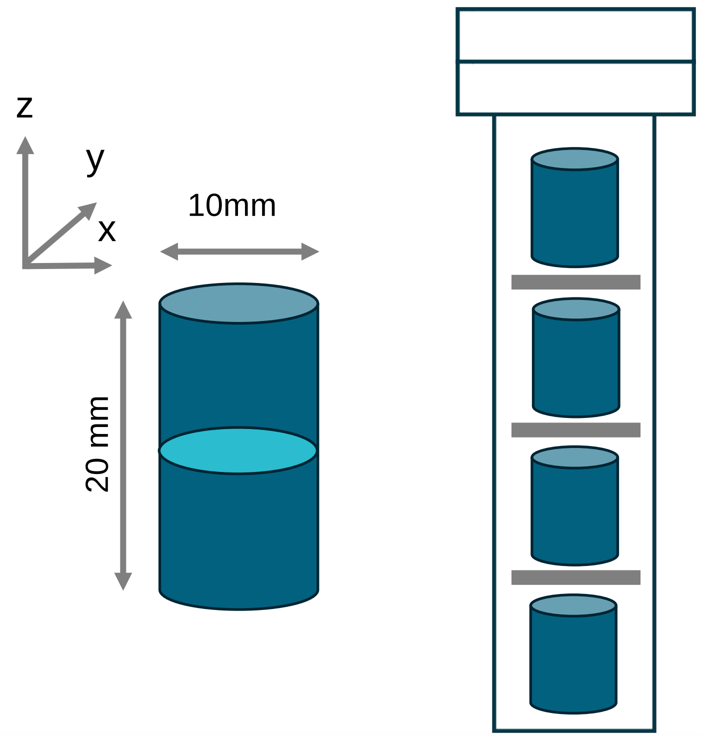
A neutron tomography study to visualize fluoride salt (FLiNaK) intrusion in nuclear-grade graphite

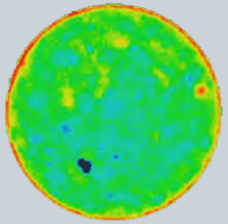
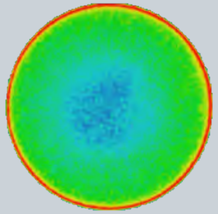
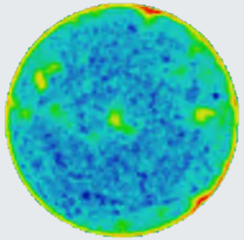
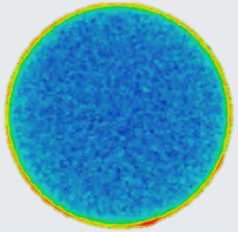
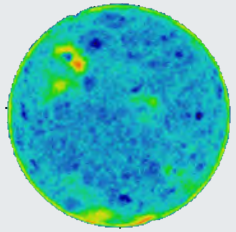
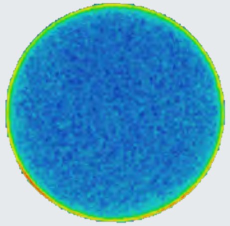
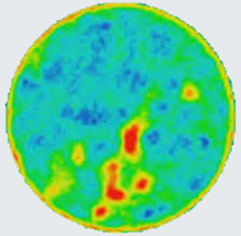
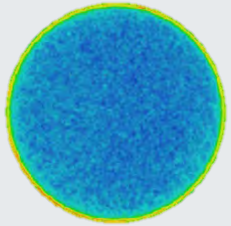
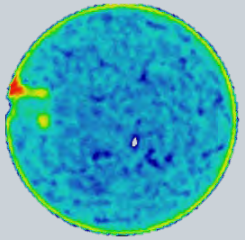
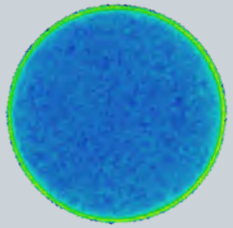
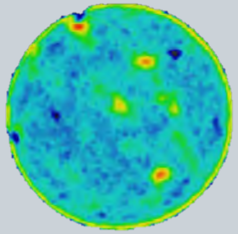
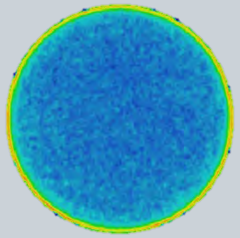
Jisue Moon [a,*], Nidia C. Gallego [b,**], Cristian I. Contescu [b], James R. Keiser [c], Dino Sulejmanovic [c], Yuxuan Zhang [d], Erik Stringfellow [d]

Summary results so far: effects of P, T t

	650ºC		750ºC	
	12 hrs	2 weeks	12-20 hrs	2 weeks
7 bar		✓		
5 bar		✓	✓	✓
3 bar		✓	✓	✓
	Graphite Grades: IG-110; 2114; ETU-10; NBG-25, NBG-18; PCEA			

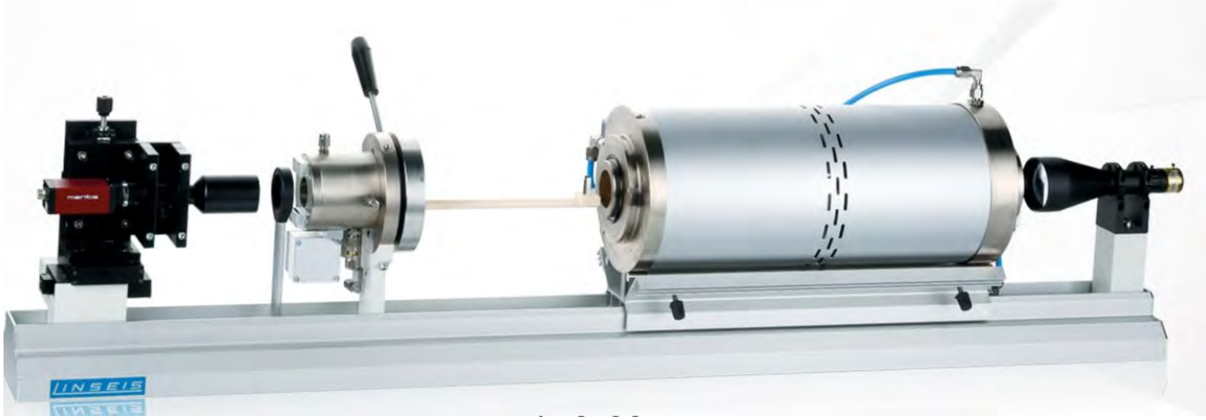
- Neutron Imaging Beamline CG-1D (ORNL's HFIR)
- Image resolution ~ 75 μm

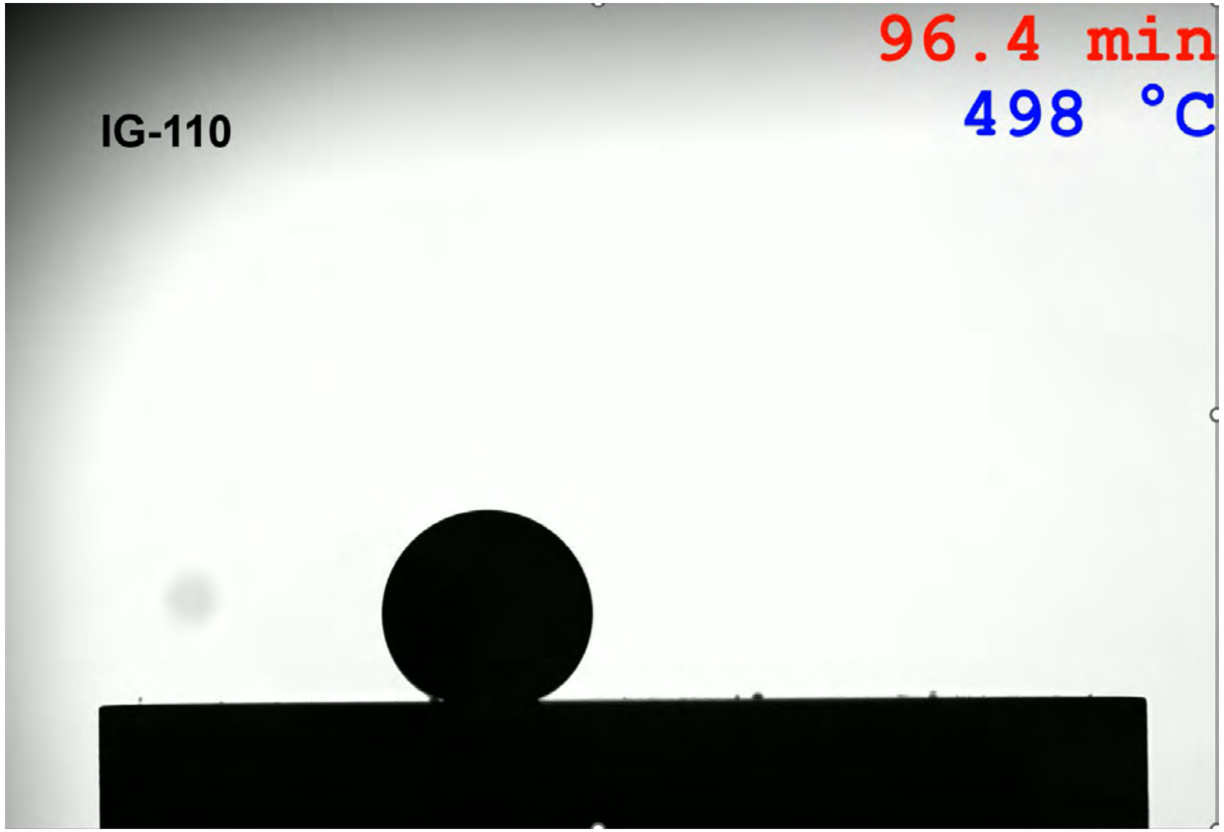


	650ºC			750ºC			
	12 hrs	2 weeks		12-20 hrs		2 weeks	
		NBG-18	IG-110	NBG-18	IG-110	NBG-18	IG-110
7 bar							
5 bar							
3 bar							

Can we predict salt intrusion?

Understanding salt wetting behavior on graphite





High temperature contact angle measurements

Contact angle measurement condition

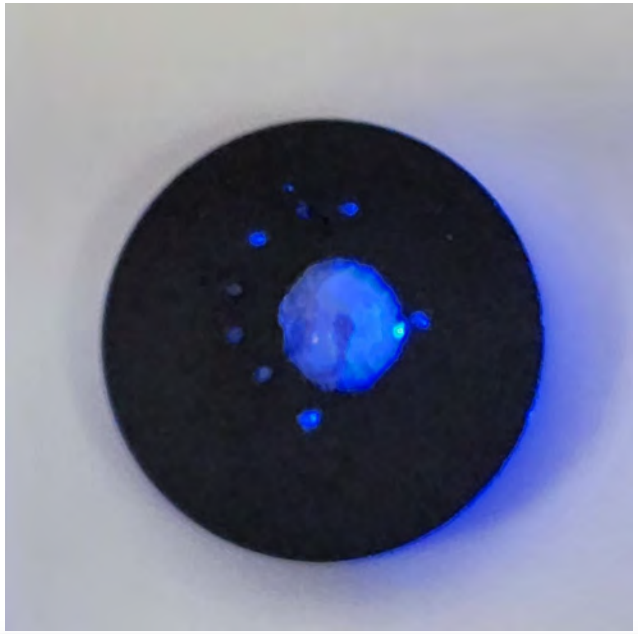
- Salt : 3mm diameter salt pellet (~8 mg)
- Graphite disc sample: 10mm diameter with 2mm thickness

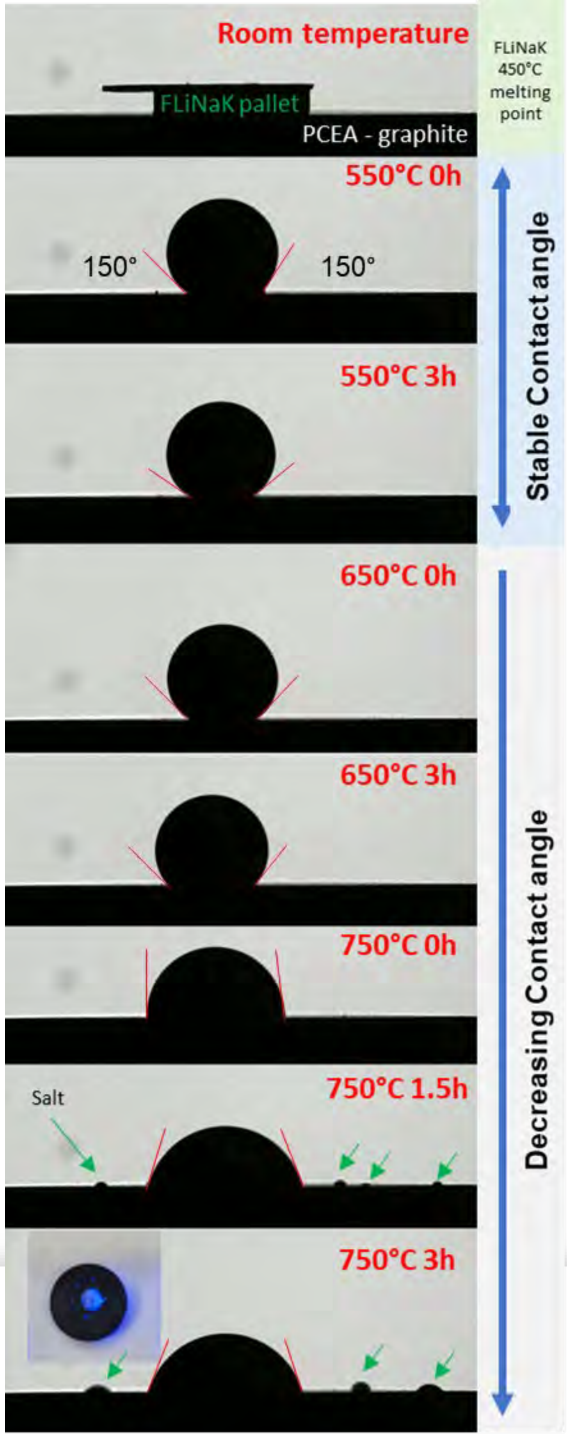
Salt properties

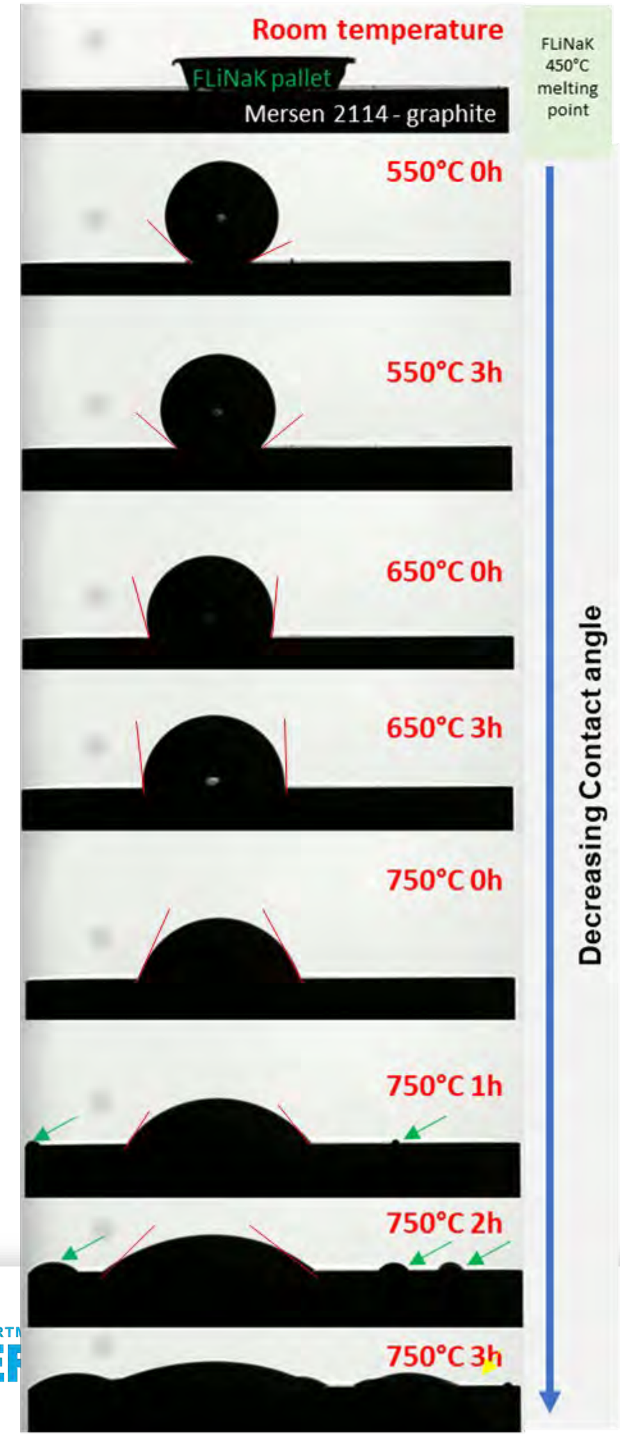
- FLiNaK Melting point 454 ^{o}C

Diffusion through open pores from the surface

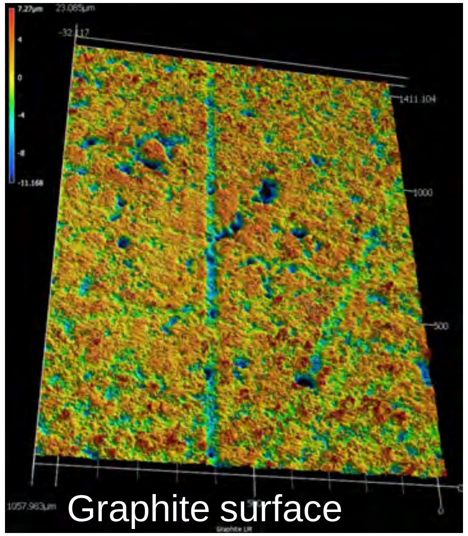
- At higher temperature (small droplets were formed close to the main FLiNaK drop

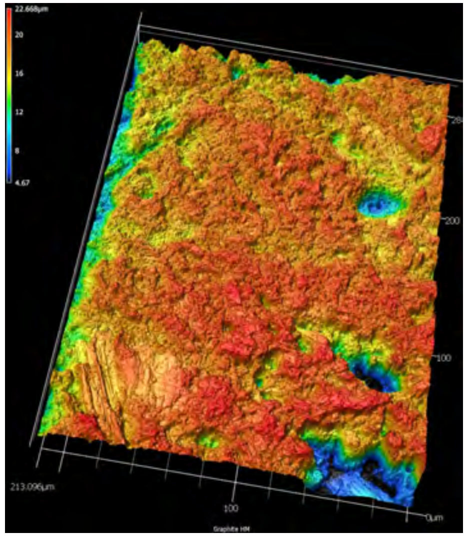


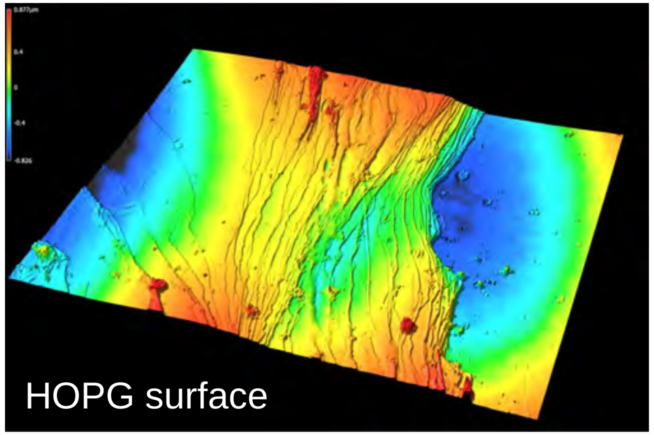


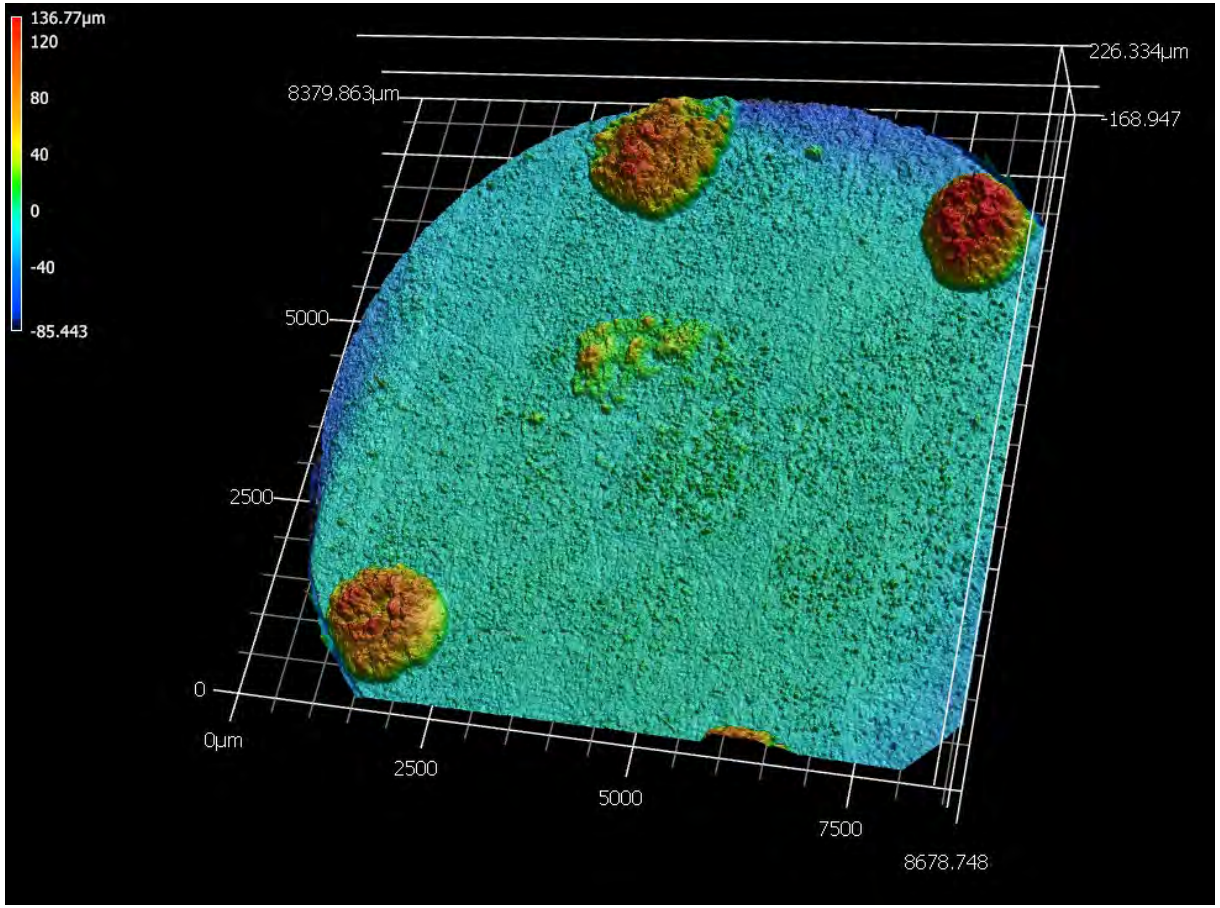


Wetting behavior of molten salt on graphite surface



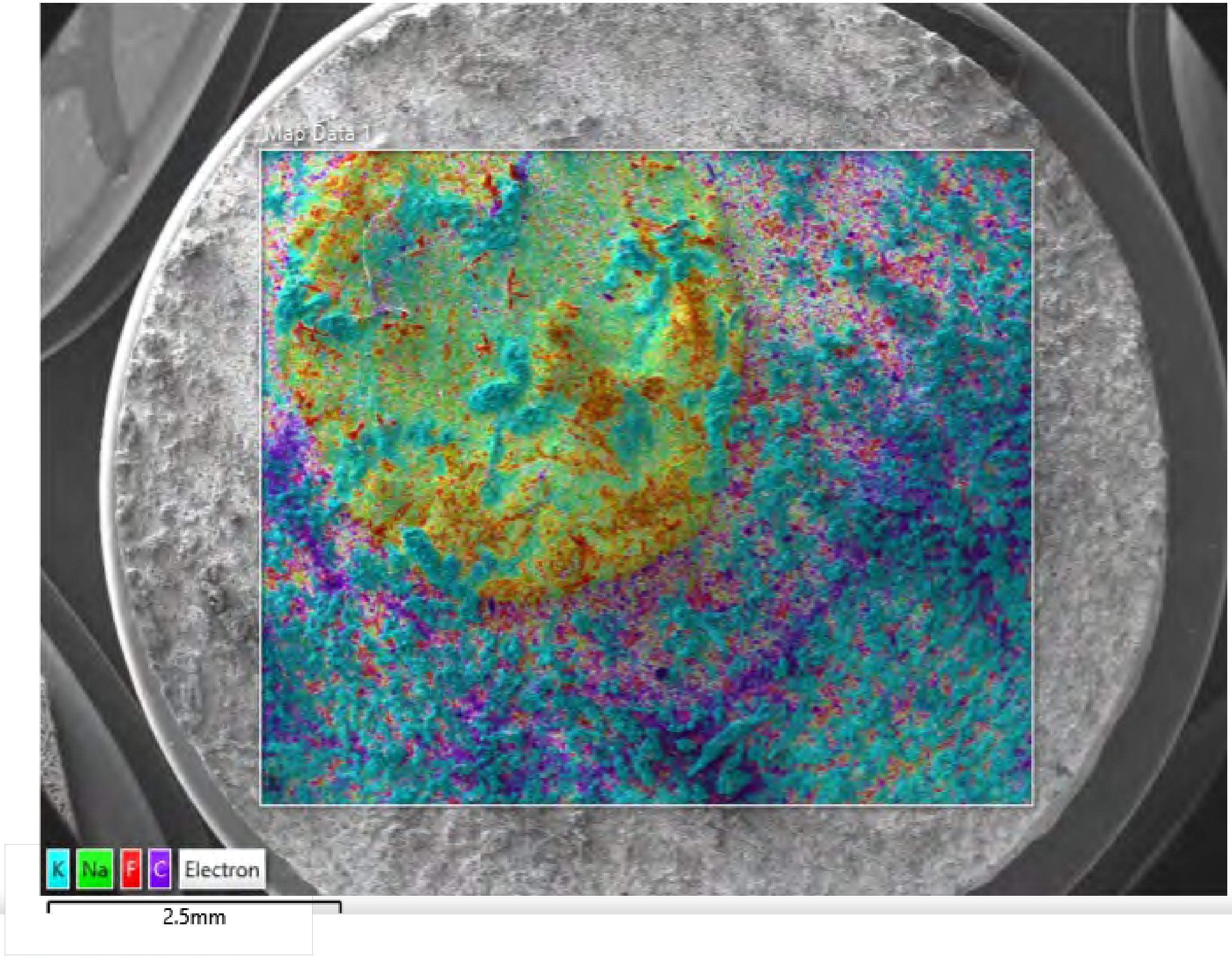




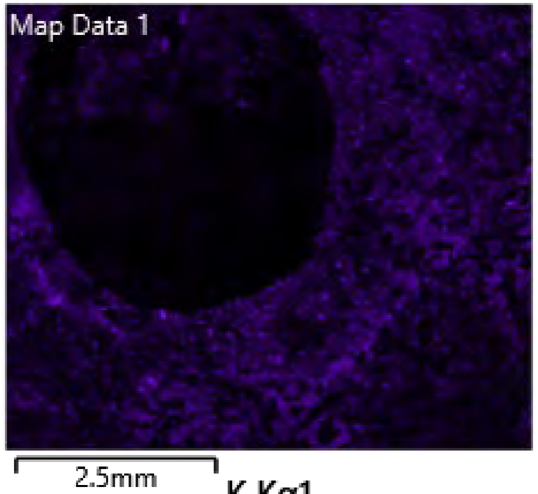


Overview EDS combined elemental map – Sample surface

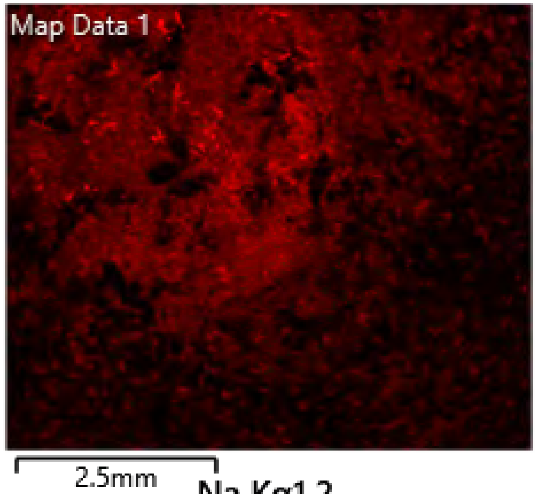
- The surface of the samples can be characterized with EDS to get a glance at the interactions between salt and the sample



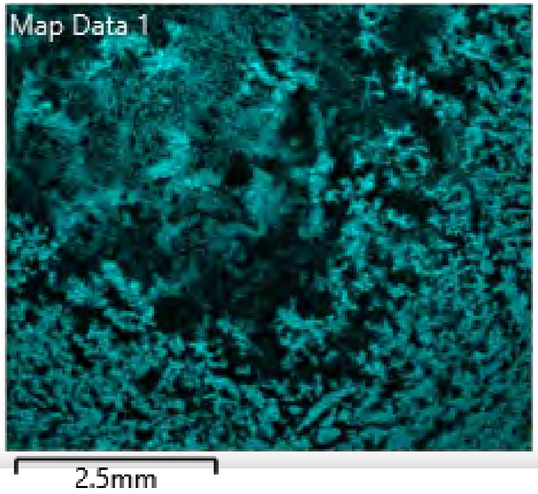
C Kα1,2



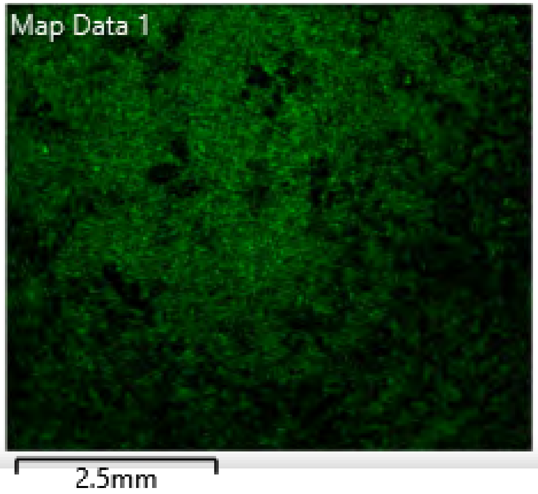
F Kα1,2



K Kα1



Na Kα1,2

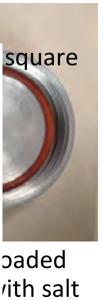


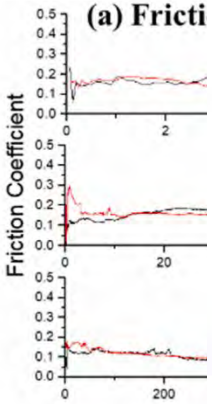
Summary / on-going activities

- Salt intrusion happens but it is highly dependent on temperature, pressure, time and graphite grade
- Salt distribution and penetration depth is highly dependent on pore structure
- On-going work to further analyze the data collected on effect of intrusion time, and additional neutron imaging time has been approved
- Continue the evaluation of contact angle measurements and the effect of other variables (graphite grade, surface finish, pre-treatment, moisture content, salt impurities…)

Understanding wear properties of graphite in a molten salt

Graphite on 316 stainless steel in molten FLiNak salt





Wear 522 (2023) 204706

Contents lists available at ScienceDirect

Wear

journal homepage: www.elsevier.com/locate/wear

ELSEVIER

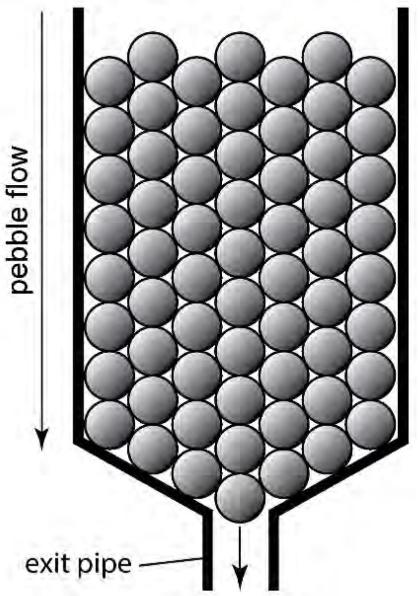
Tribocorrosion of stainless steel sliding against graphite in FLiNaK molten salt☆

Xin He [a], Chanaka Kumara [a], Dino Sulejmanovic [a], James R. Keiser [a], Nidia Gallego [b], Jun Qu [a,*]

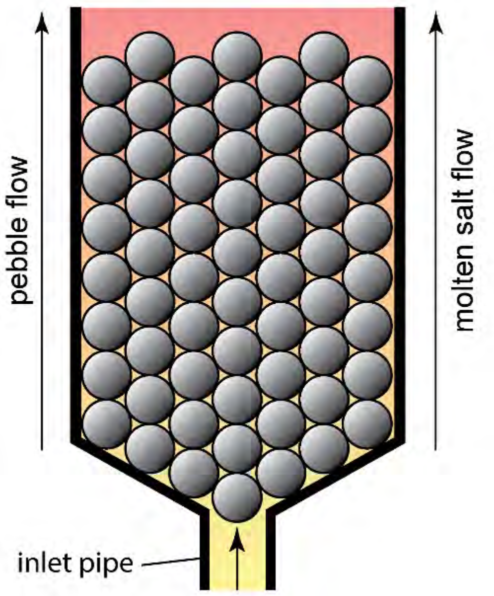
[a] *Materials Science and Technology Division, Oak Ridge National Laboratory, Oak Ridge, TN, 37831, USA*
[b] *Chemical Sciences Division, Oak Ridge National Laboratory, Oak Ridge, TN, 37831, USA*

Tribological characterization of graphite in dry argon and molten salt environment

High temperature gas-cooled reactor



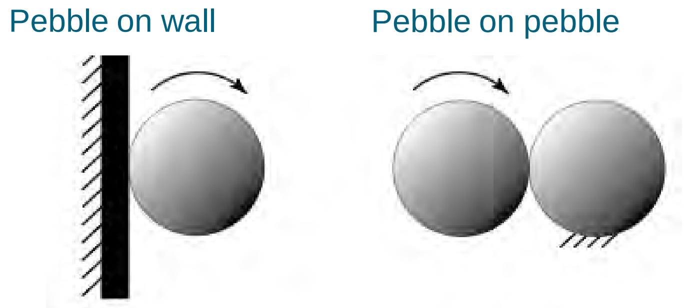
Molten salt reactor



- Rector contains thousands of pebbles that circulated during operation.
- Each pebble passes through multiple times before it is finally discharged.
- In **HTGR**, the pebbles flow downwards, while in **MSR** the pebbles and molten salt flow upward.
- ***Pebble flow results in their abrasion and dust contamination.***
- <u>**Environment:**</u> argon or helium gas, temperatures up to 650 °C.

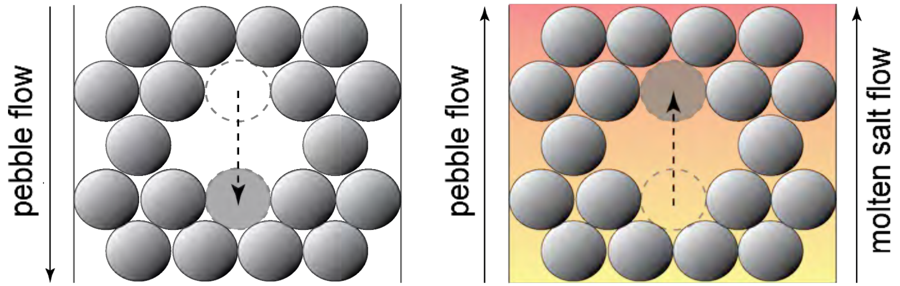
Contact modes

Sliding & Rolling



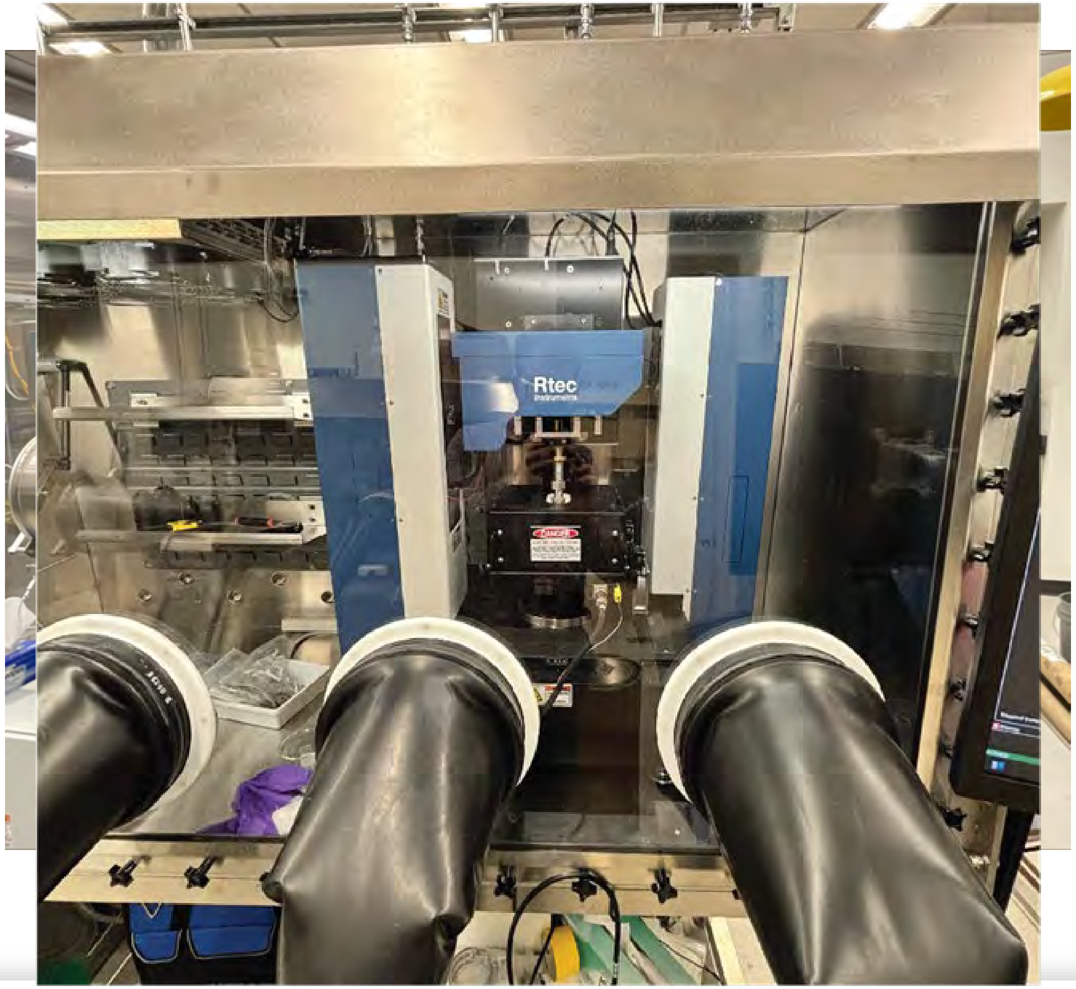
Pebbles slide and roll against other pebbles and the graphite wall during circulation in the reactor which results in their abrasion.

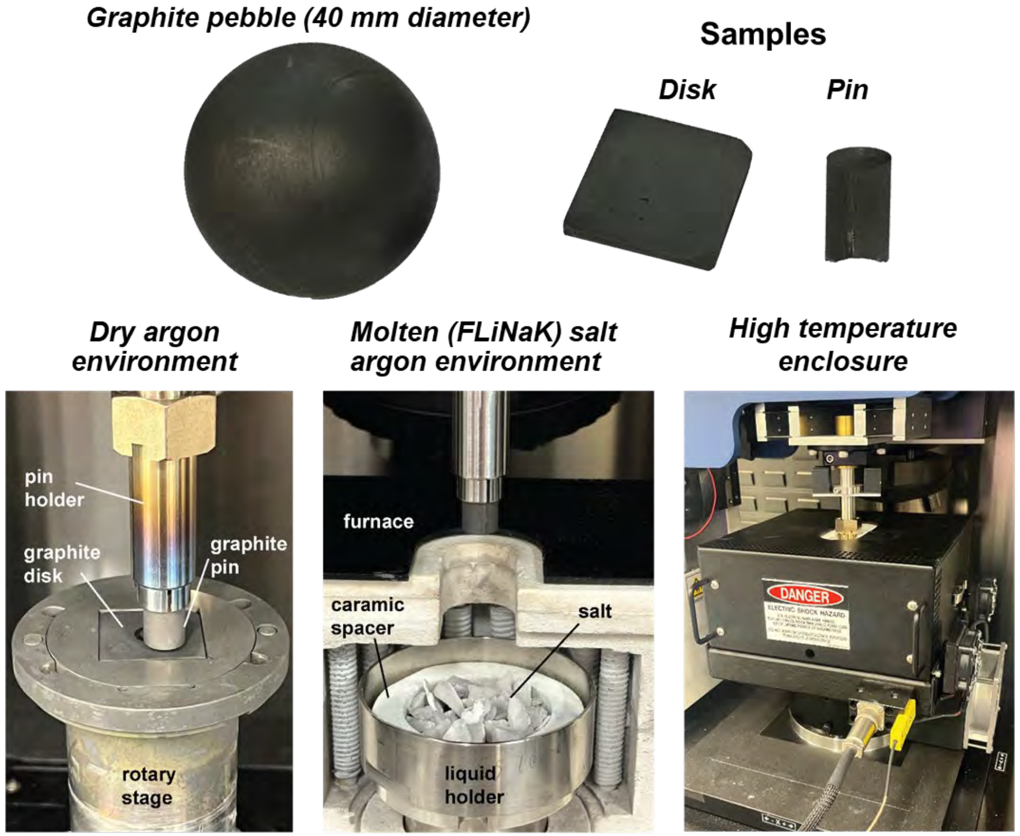
Impact



A pebble could move through the void and collide with an opposing pebble at higher contact loads causing an **impact**.

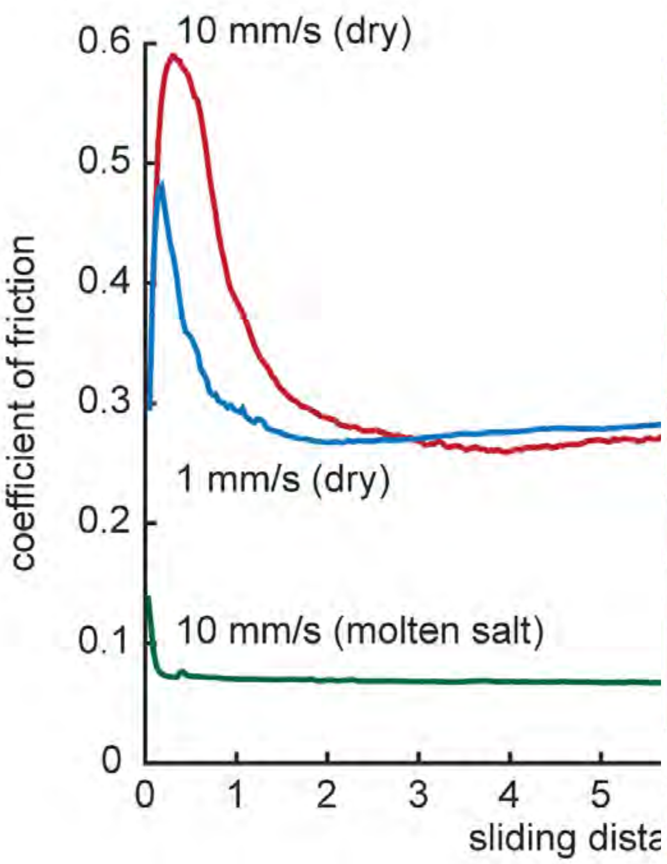
Fully commissioned glovebox and tribometer will enable measurements under more controlled environment

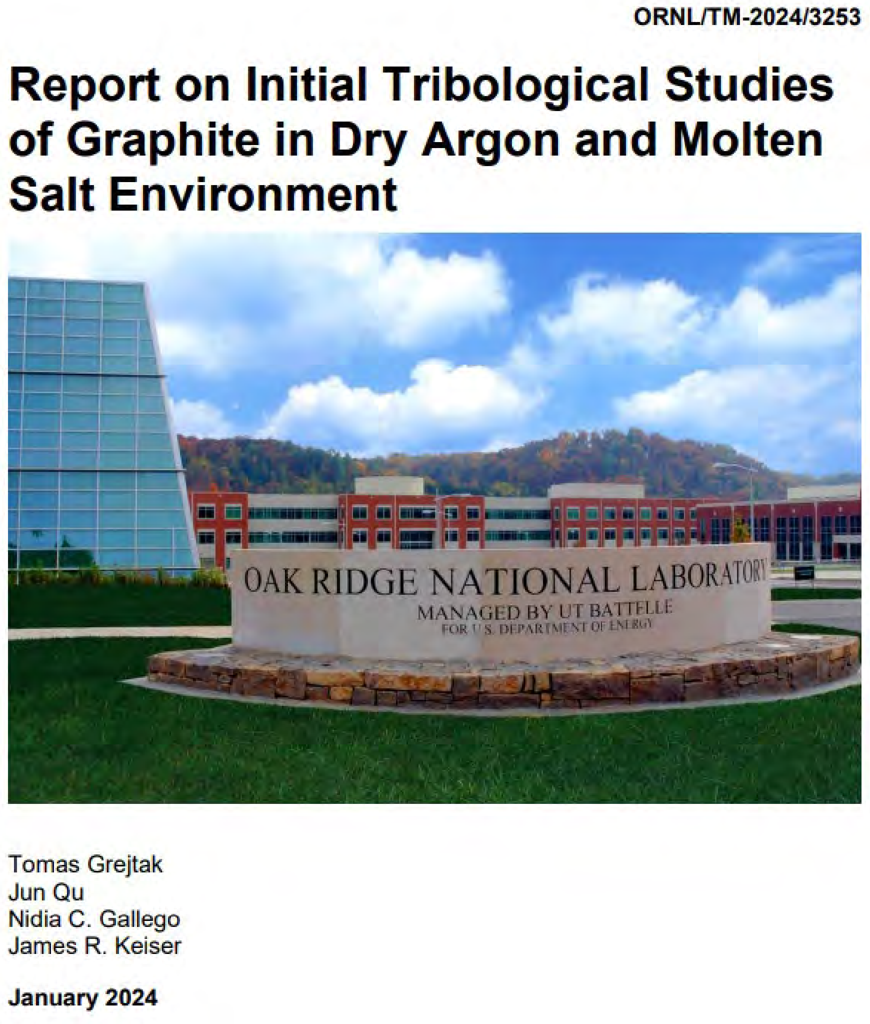




*Thanks to Kairos for providing the graphite pebbles

Graphite-on-graphite: dry argon vs molten FLiNaK salt at 650°C




ORNL/TM-2024/3253

Report on Initial Tribological Studies of Graphite in Dry Argon and Molten Salt Environment

Tomas Grejtak
Jun Qu
Nidia C. Gallego
James R. Keiser

January 2024

liding speed mm/s)	Maximum COF	Steady state COF
	0.47	0.28
0	0.58	0.27
0	0.14	0.07

n salt lubrication significantly es friction and wear.

Summary - Wear

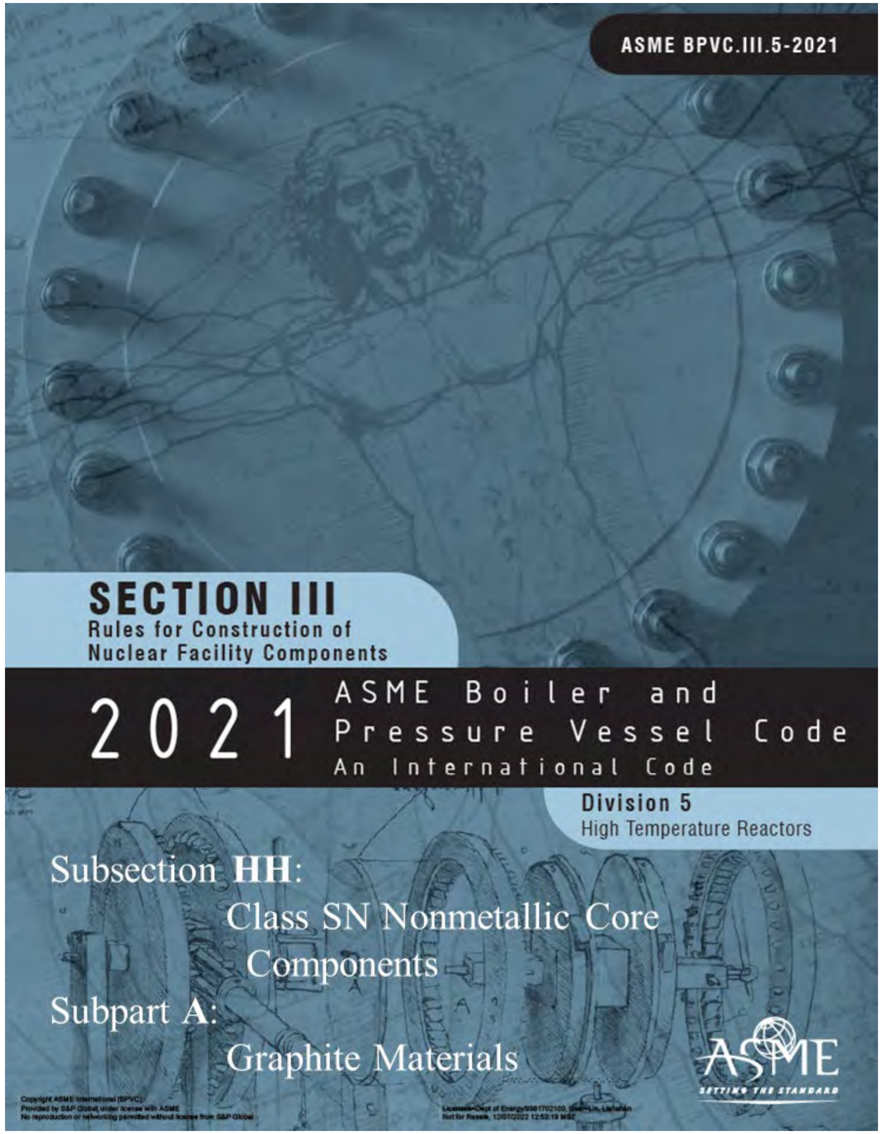
- Initial scoping studies of the wear behavior of graphite in molten salts were completed and published.
- New facilities have been installed and will allow us to continue our studies under more control environments.

ASME SEC III Division 5
High Temperature Reactors

The current HHA does not address any coolant salt interactions with graphite.

Chemical attack, salt infiltration and retention as well as wear and erosion aspects need to be incorporated in the design rules.

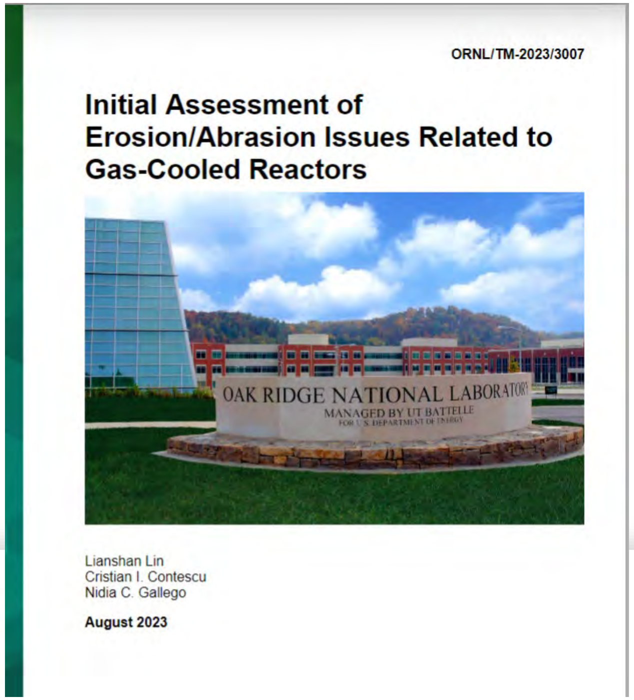
Standards:
D02.F0 on Manufactured Carbon and Graphite Products

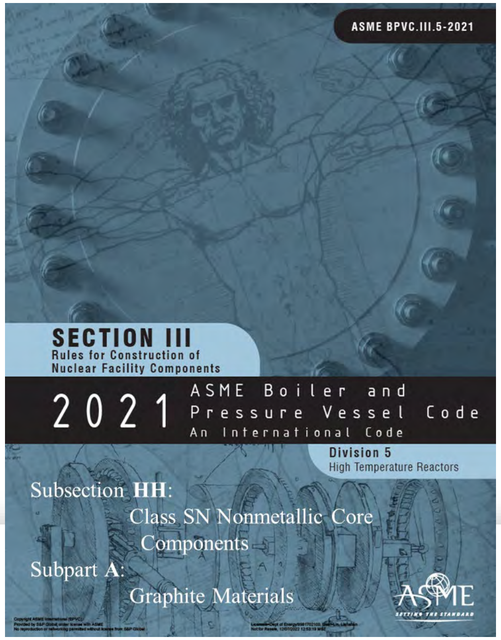


ASME SEC III Division 5
High Temperature Reactors

Record 23-2484 submitted and approved

Proposal to edit sections HHA-3140 and HHA-3143 to address applicability of the sections to MSRs





Record 23-2484

Proposal to edit sections HHA-3140 and HHA-3143 to address applicability of the sections to MSRs.

CURRENT TEXT

HHA-3140 SPECIAL CONSIDERATIONS (23)

Assessment of Graphite Core Components comprising the Graphite Core Assembly shall include consideration of the effects of thermal oxidation, irradiation, abrasion and erosion, fatigue, and buckling. The rules for oxidation in HHA-3141 and abrasion and erosion in HHA-3143 are specific to high temperature gas-cooled reactors.

HHA-3143 Abrasion and Erosion

(a) Abrasion shall be evaluated if there is relative motion between Graphite Core Components, Graphite Core Components and interfacing components, or Graphite Core Components and the fuel of a pebble bed reactor.

(b) Erosion shall be evaluated in areas where the mean gas flow velocity in the cross section of the channel exceeds 330 ft/sec (100 m/s).

PROPOSED TEXT

HHA-3140 SPECIAL CONSIDERATIONS

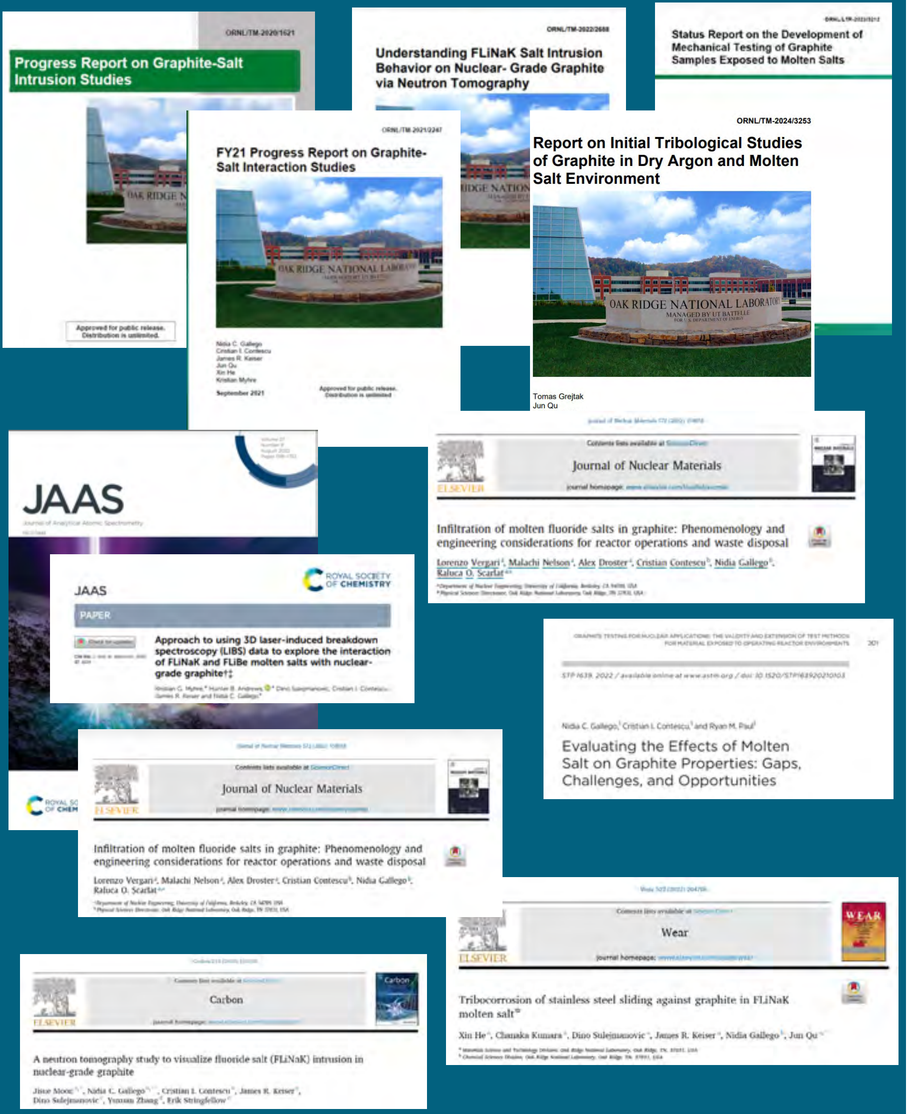
Assessment of Graphite Core Components comprising the Graphite Core Assembly shall include consideration of the effects of thermal oxidation, irradiation, abrasion and erosion, fatigue, and buckling. ~~The rules for oxidation in HHA 3141 and abrasion and erosion in HHA 3143 are specific to high temperature gas cooled reactors.~~

HHA-3143 Abrasion and Erosion

(a) Abrasion shall be evaluated if there is relative motion between Graphite Core Components, Graphite Core Components and interfacing components, or Graphite Core Components and the fuel of a pebble bed reactor.

(b) ~~Erosion shall be evaluated in areas where the mean gas flow velocity in the cross section of the channel exceeds 330 ft/sec (100 m/s).~~ The designer shall determine the value of the mean fluid flow velocity, above which, an evaluation of erosion is necessary and justify the adequacy of the value in the Design Report. The effect of any debris in the fluid shall be considered.

Publications



Grejtak T, Qu J, Nidia Gallego, Keiser J "Report on Initial Tribological Studies of Graphite in Dry Argon and Molten Salt Environment" ORNL/TM-2024/3253 (January 2024)

Gallego N, Lin L, Contescu C "Status Report on the Development of Mechanical Testing of Graphite Samples Exposed to Molten Salts" ORNL/LTR-2023/3212 (December, 2023)

Gallego N, Braatz J, Contescu C, Sulejmanovic D, Keiser J, Zhang Y "Graphite-Salt Interactions: Summary of FY23 Activities" ORNL/TM-2023/3144 (October, 2023)

Lin L, Contescu C, Gallego N "Initial Assessment of Erosion/Abrasion Issues Related to Gas-Cooled Reactors" ORNL/TM-2023/3007 (September, 2023)

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* All available at OSTI

Thank you

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