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Understanding graphite– salt interactions: a multidisciplinary approach

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Oak Ridge National Laboratory



U.S. DEPARTMENT
of **ENERGY**

ORNL IS MANAGED BY UT-BATTELLE LLC
FOR THE US DEPARTMENT OF ENERGY



Team Effort – Contributors



Nidia C. Gallego



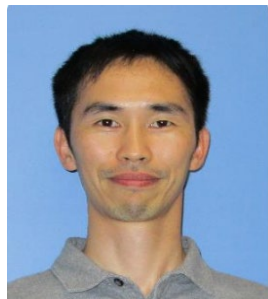
Jisue Braatz



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David Arregui-Mena



Lianshan Lin



GS Jung



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Jun Qu



Yuxuan Zhang



Cristian Contescu



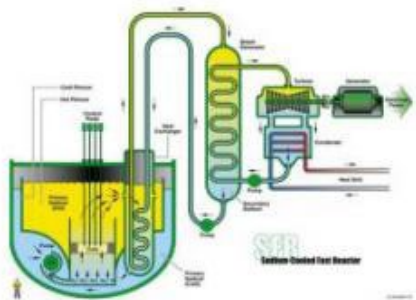
Wenqi Li

INGSM 2024
UC Berkeley, CA

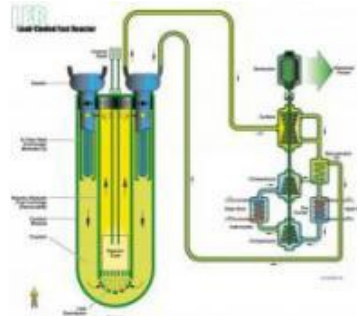


Kairos Power
(Alameda, CA)
visit 2024

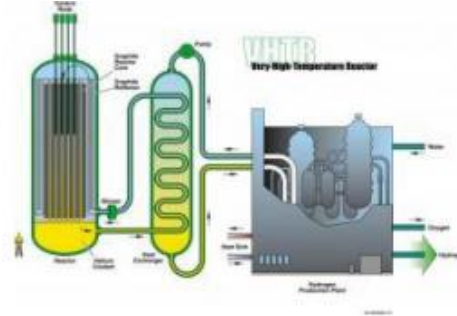
Graphite plays a critical role in several GenIV-type Advanced Reactor concepts, including gas-cooled and molten salt reactors.



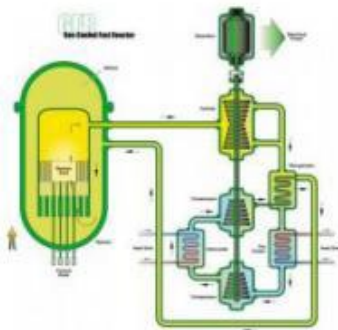
Sodium Fast Reactor



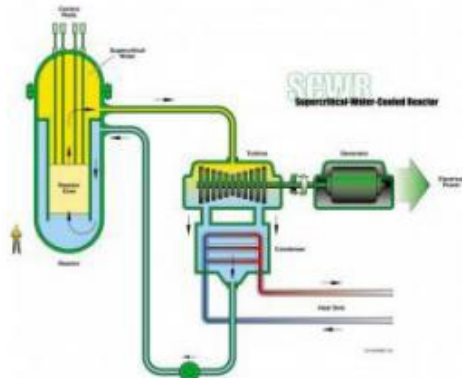
Lead Fast Reactor



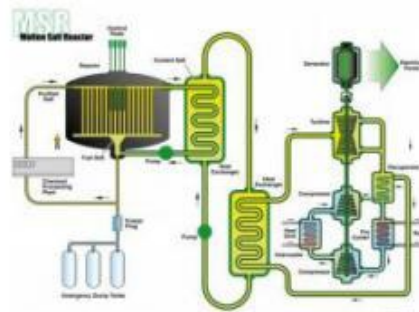
Very High Temperature Reactor



Gas Cooled Fast Reactor



Supercritical Water Cooled Reactor

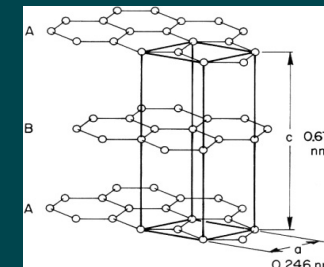


Molten Salt Cooled Reactor

15

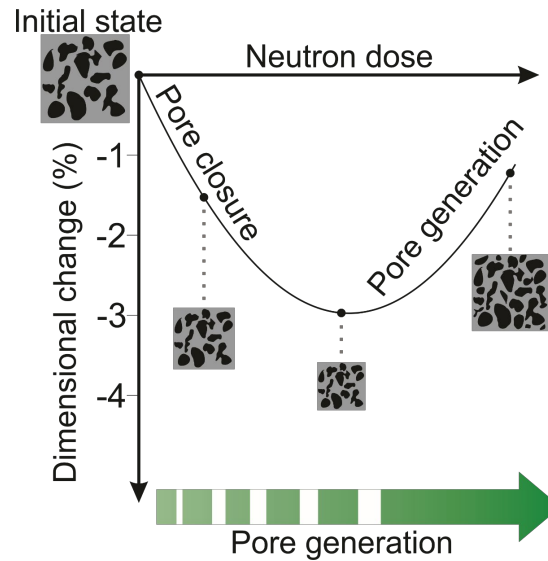
Why graphite?

- Good neutron moderator for thermal reactors
- Low neutron absorption – enhances chain reaction
- High thermal conductivity



Effect of fast neutron irradiation and its relationship with microstructure

- Dimensional changes, structural damage
- Change in mechanical and thermal properties



Graphite plays a critical role in several GenIV-type Advanced Reactor concepts, including gas-cooled and molten salt reactors.

Environment Effects

Gas-cooled reactors

- ❖ Chronic oxidation
- ❖ Acute oxidation

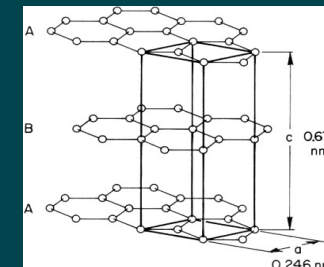
Fluoride salt-cooled reactors

Physical

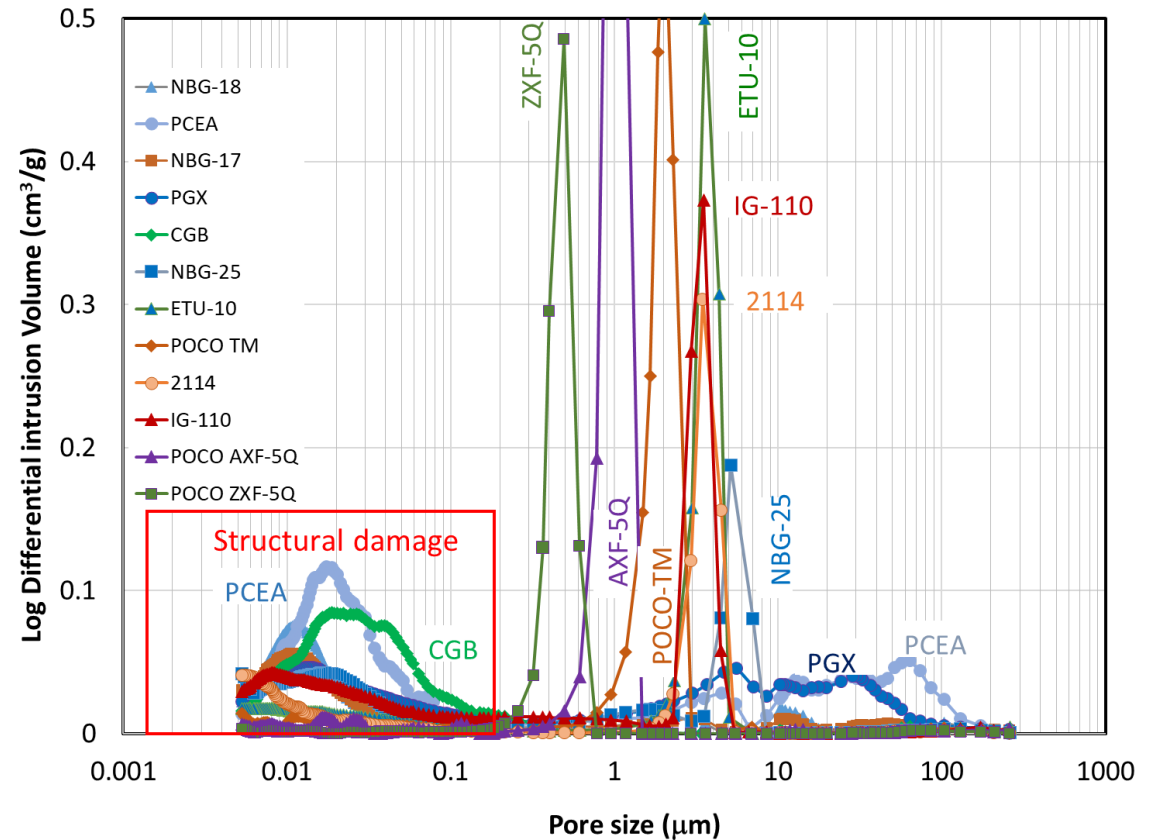
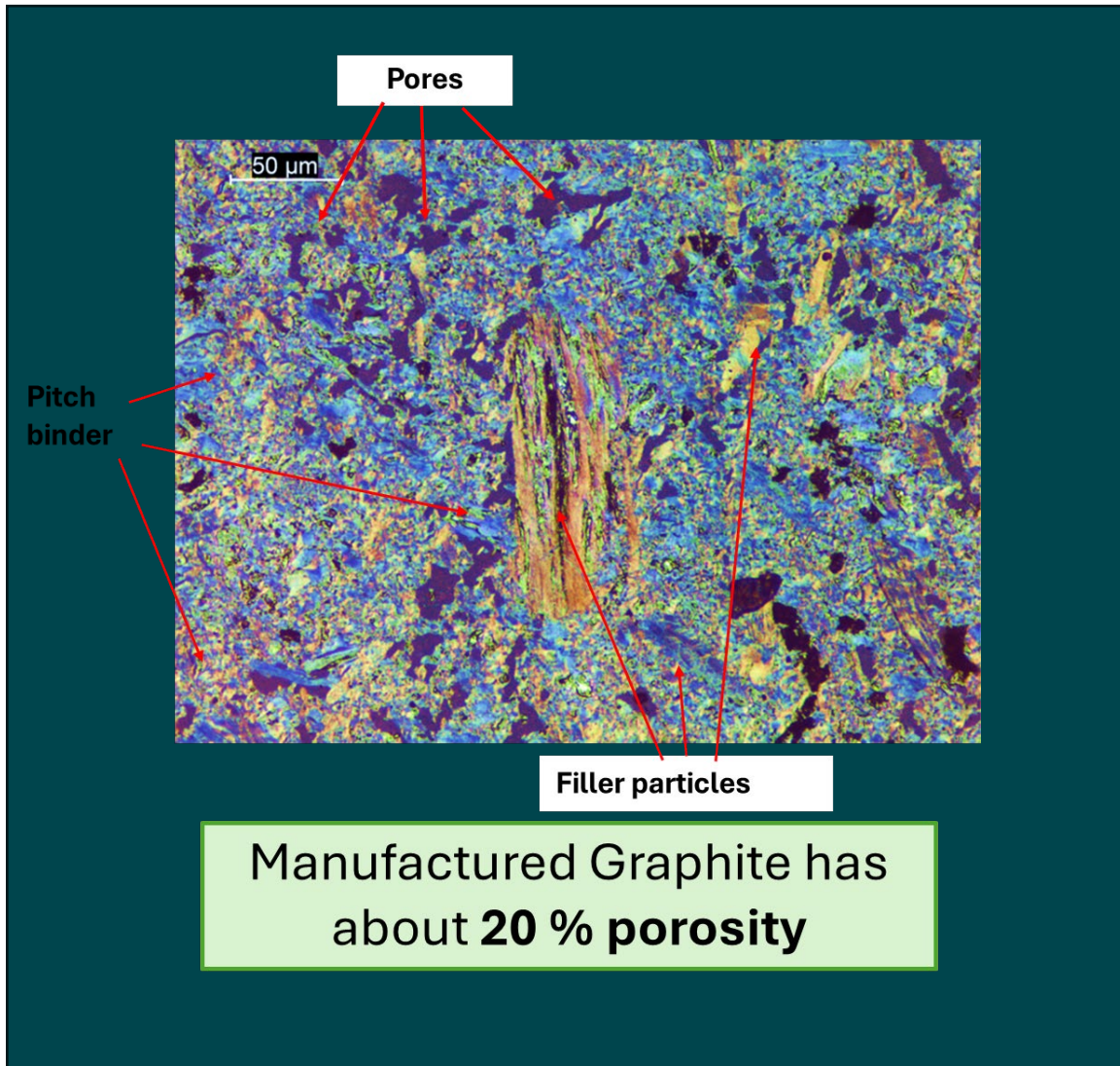
- ❖ Salt intrusion into graphite pores and effect on mechanical/ thermal properties; effect of/on irradiation damage; what if salt is fueled
- ❖ Wear and abrasion
- ❖ Erosion
- ❖ Effect of potential heating/cooling cycles

Chemical

- ❖ Chemical Interactions: Fluorination, intercalation, effect on properties of graphite
- ❖ Effect of salt impurities
- ❖ Effect of graphite dust in salt
- ❖ Absorption of fission products
- ❖ Galvanic corrosion



Graphite pore network (**size, shape and connectivity**) varies significantly between graphite grades



Graphite pore size distribution obtained from mercury intrusion measurements

Objective:

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 - Graphite Program under the GCR
Campaign

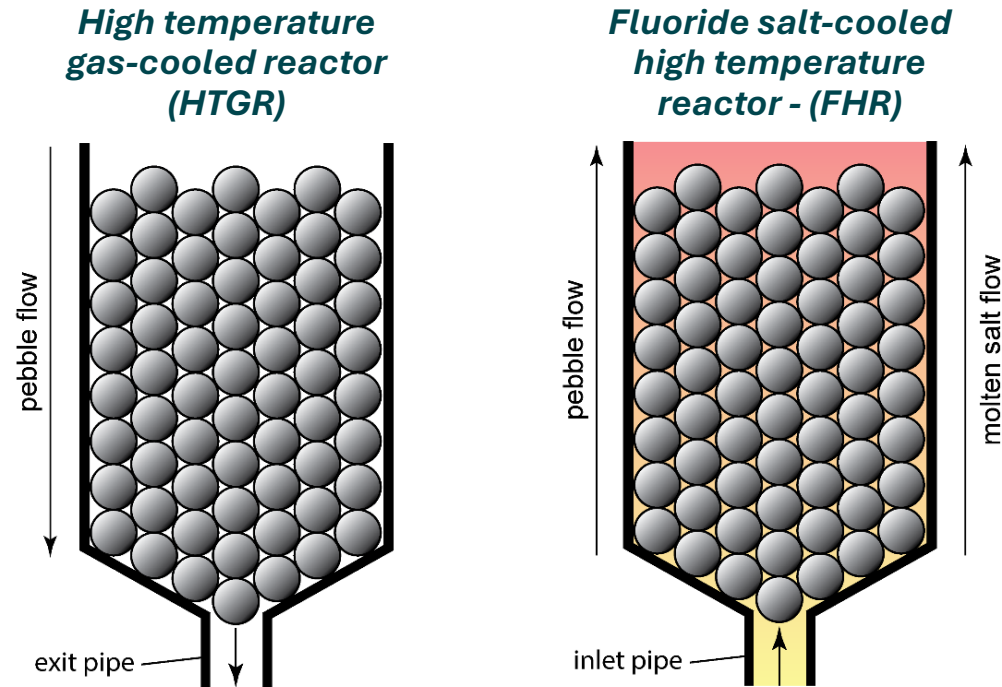
This research used resources at the High
Flux Isotope Reactor, a US DOE Office of
Science User Facility operated by the ORNL

Current Research Focus of Graphite-Salt Studies at ORNL:

- Understanding physical interactions: salt intrusion, penetration depth and salt distribution, as a $F(T, P, t)$ and graphite microstructures.
- 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.

Tribological Studies - Understanding wear properties of graphite

Tribological characterization of graphite in dry argon and molten salt environment

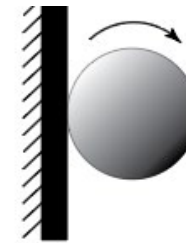


- Reactor contains thousands of pebbles that circulated during operation.
- Each pebble passes through multiple times before it is finally discharged.
- In **HTGR**, the pebbles flow downwards, while in **FHR** the pebbles and molten salt flow upward.
- **Pebble flow results in their abrasion and potential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

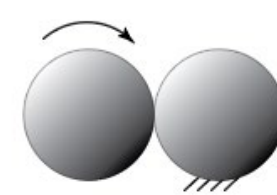
Contact modes

Sliding & Rolling

Pebble on wall

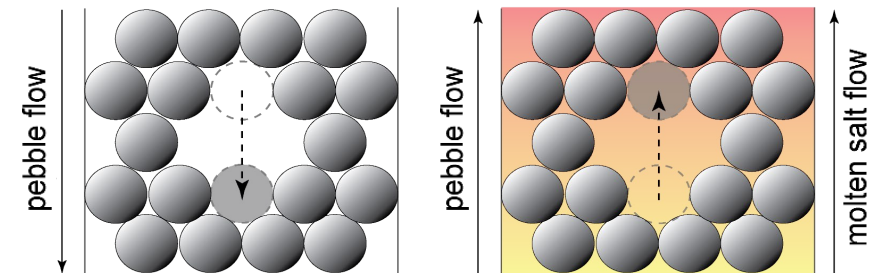


Pebble on pebble



Pebbles slide and roll against other pebbles and the graphite wall during circulation in the reactor which may result in **their abrasion and dust contamination**.

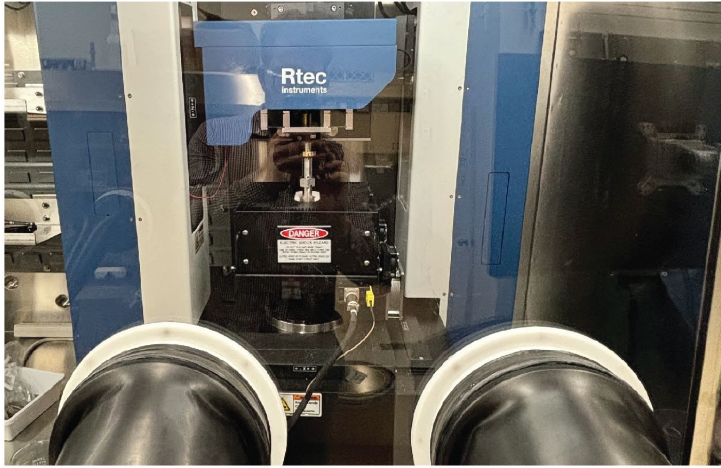
Impact



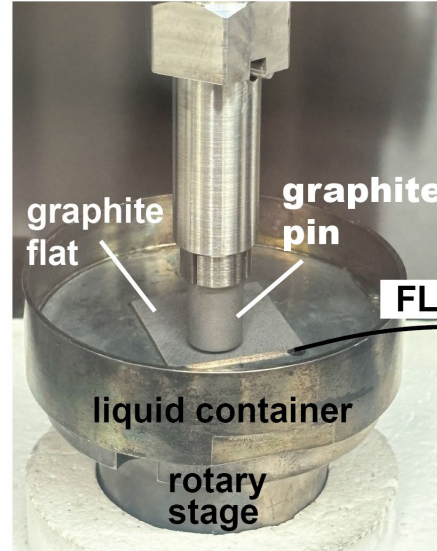
A pebble could move through the void and collide with an opposing pebble at higher contact loads causing an **impact**.

Friction and wear characterization – sliding mode

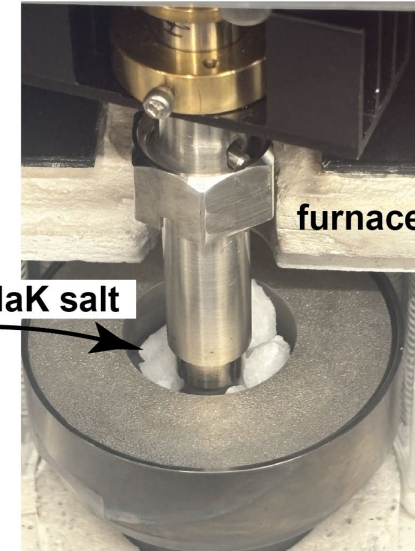
Tribometer placed in a glovebox with a controlled Ar atmosphere



Dry argon



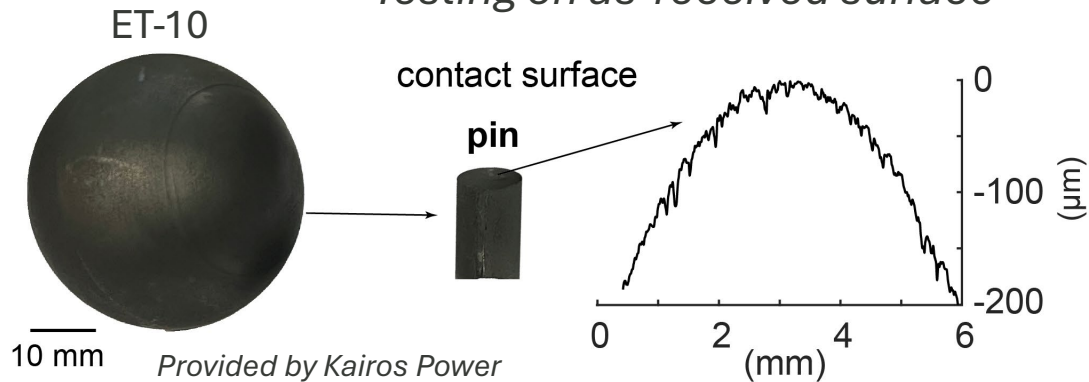
FLiNaK



After the test



Testing on as-received surface



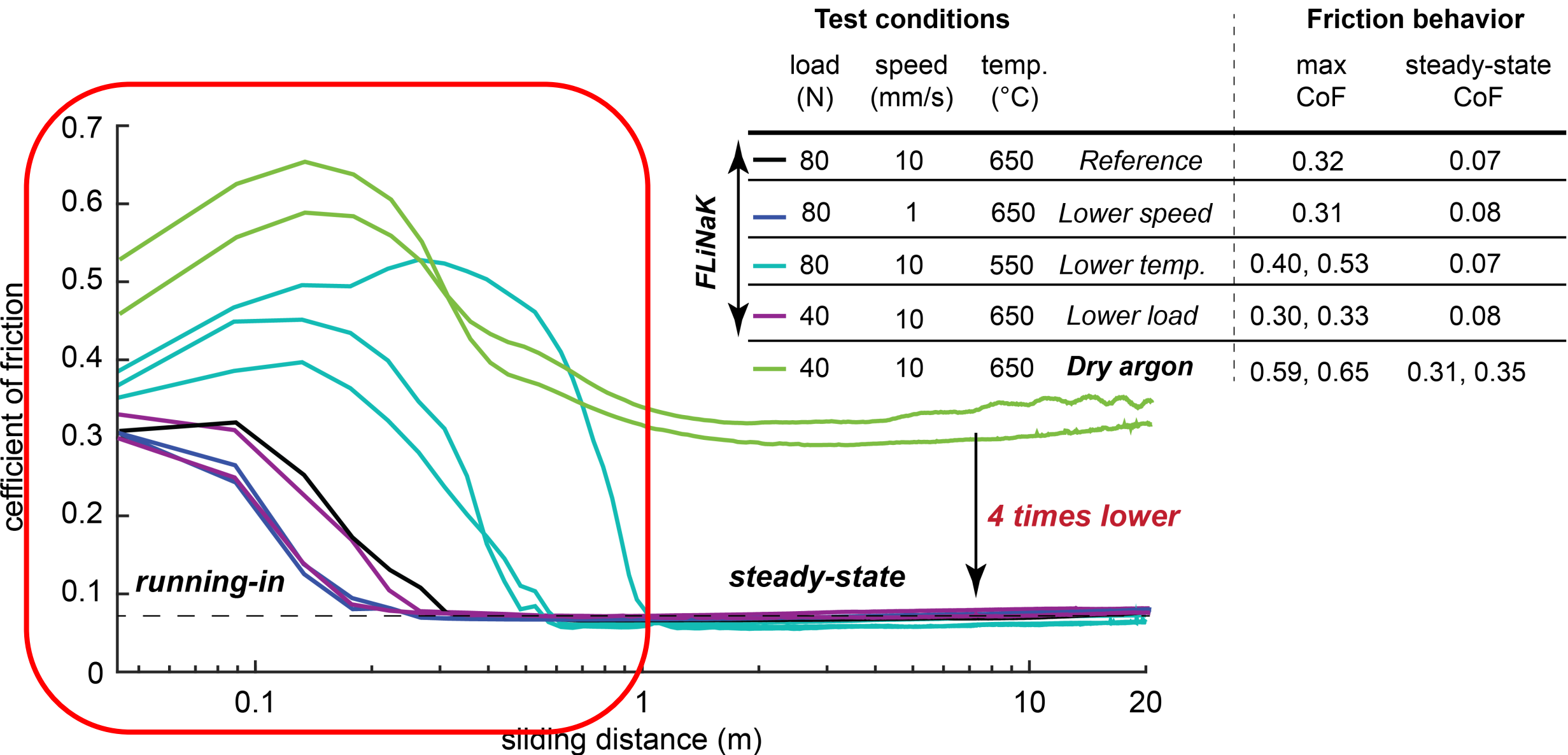
Prepared with an end mill and polished



Gas: argon ($O_2 < 1\text{ ppm}$; $H_2O < 1\text{ ppm}$)

FLiNaK: (mol %: 46.5% LiF, 11.5% NaF, 42% KF)

Preliminary Results - Molten Salt Significantly Reduces Friction



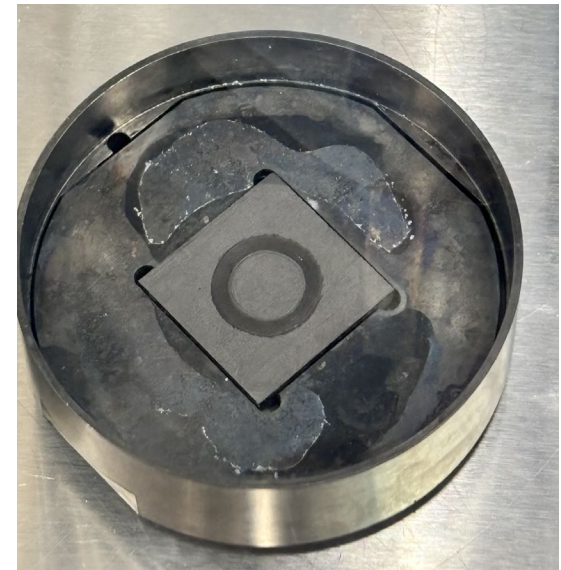
Future work: understanding friction and wear behavior in the FLiNaK

- Effect of temperature, sliding speed and contact load is not fully understood
- Technical challenges:
 - Materials compatibility with salt
 - Salt coverage of the graphite surface
- Testing under more realistic conditions:
 - Graphite infiltrated with salt
 - Testing in FLiBe
 - Matrix material

Fully submerged

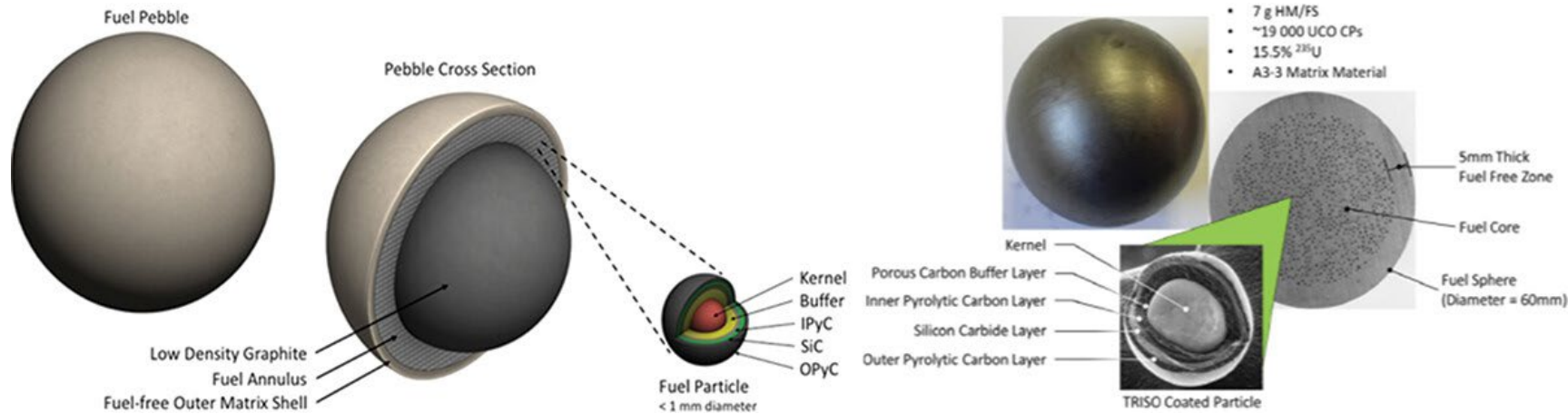


Starved



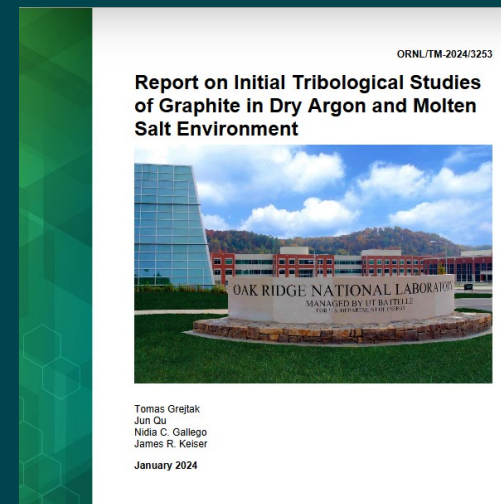
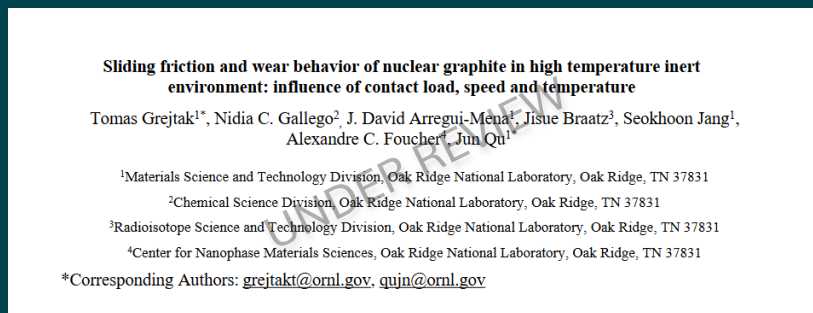
Friction and wear characterization of graphite matrix material

- Outer shell of a pebble is made of graphite matrix
- Preliminary tribological testing in dry argon using graphite matrix provided by **TRISO-X**
- Pin and flat samples machined from a pebble (50 mm diameter)



<https://www.kairospower.com/technology>

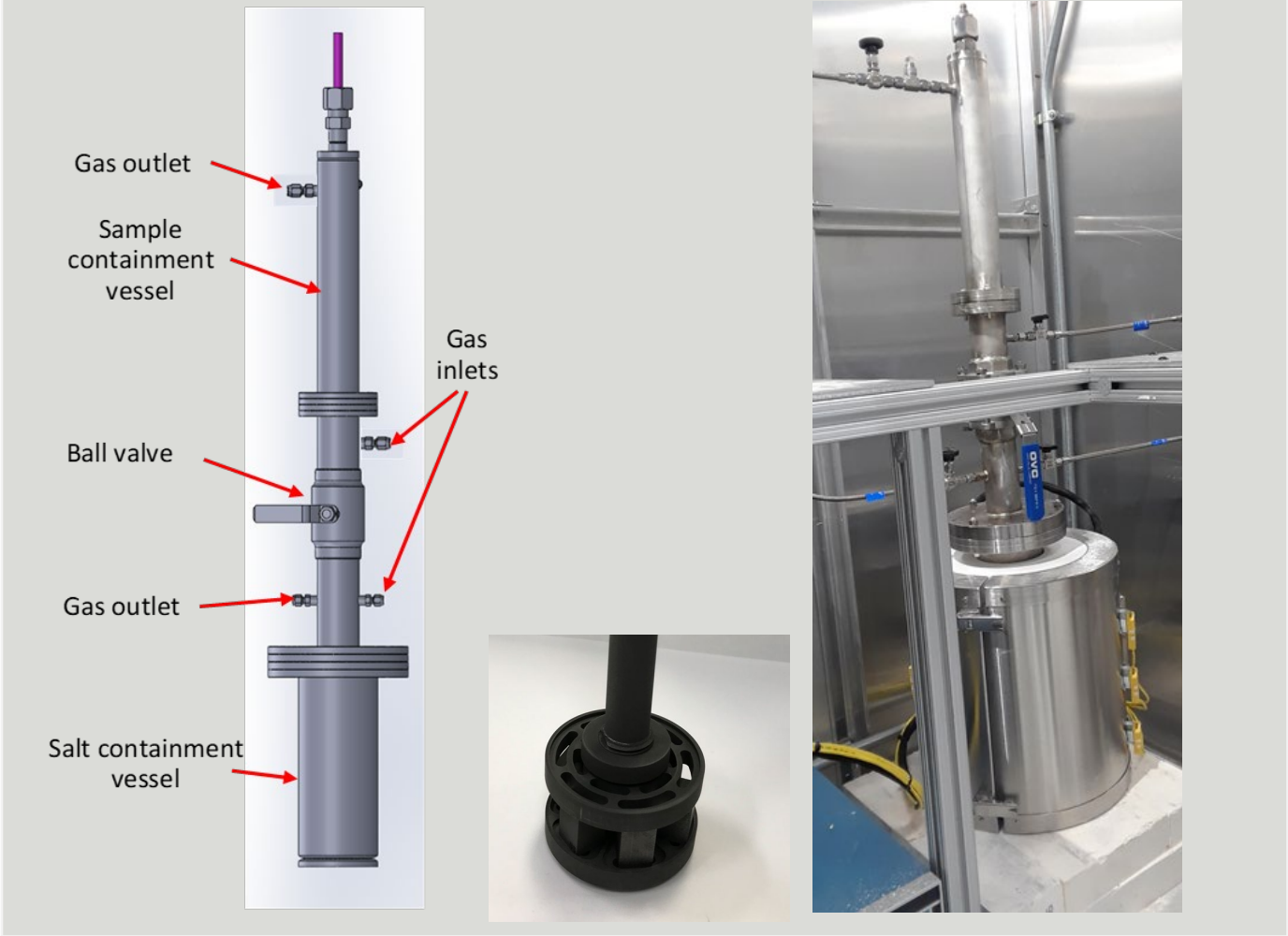
<https://x-energy.com/xe-100/>



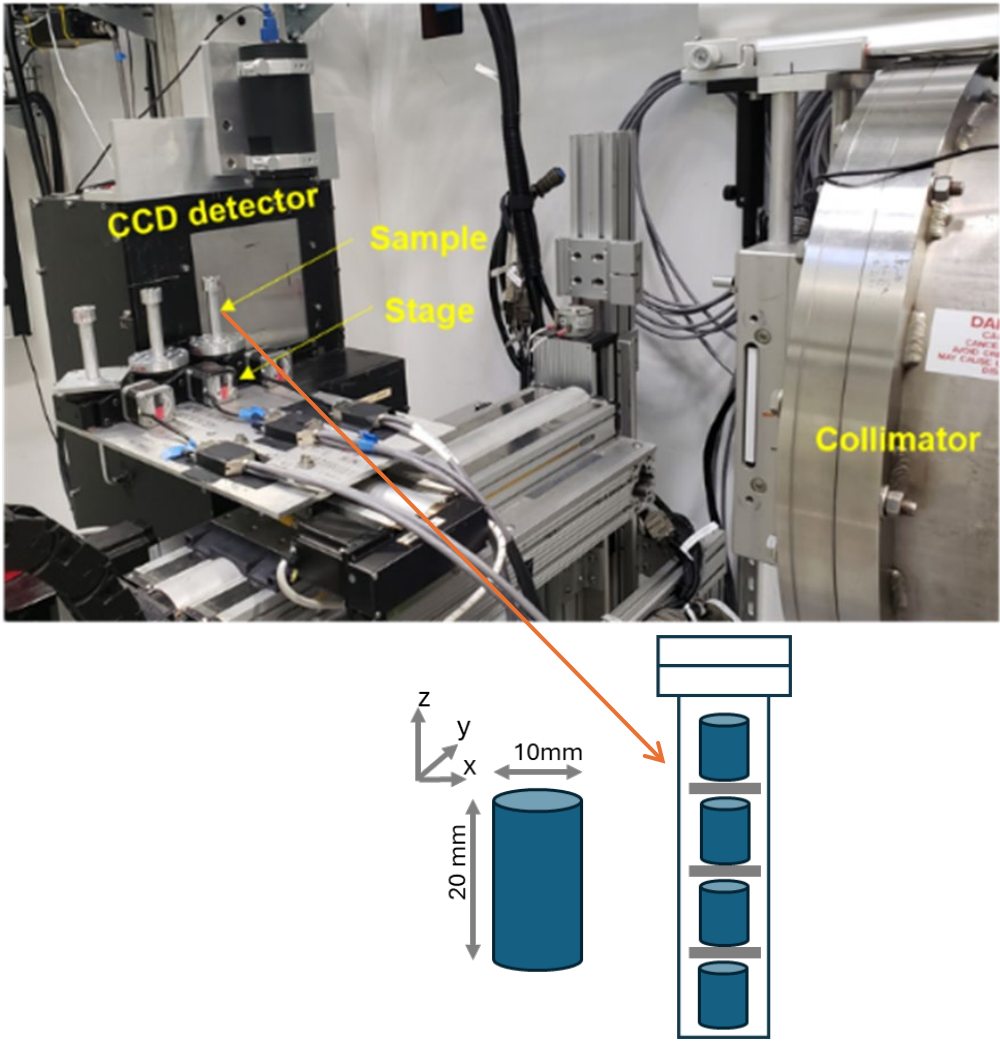
Understanding salt intrusion and salt wetting behavior on graphite surfaces

Capabilities for FLiNaK Salt Intrusion and Use of nCT for Analysis

Setup for high-temperature, high-pressure salt intrusion experiments



Neutron tomography (MARS) beamline at the HFIR, ORNL



Understanding the Effect of Exposure Time on Salt Intrusion

Graphite grade	Grain size (μm)	Grain category
IG-110	<50	superfine
ETU-10	<50	superfine
2114	<50	superfine
NBG-25	<100	fine
PCEA	<1000	medium-fine
NGB-18	<4000	medium-coarse

Workflow:

- 1) nCT scans of the pristine samples
- 2) Salt intrusion experiments
- 3) nCT scans of the salt-exposed samples

Intrusion Conditions

T: 750°C

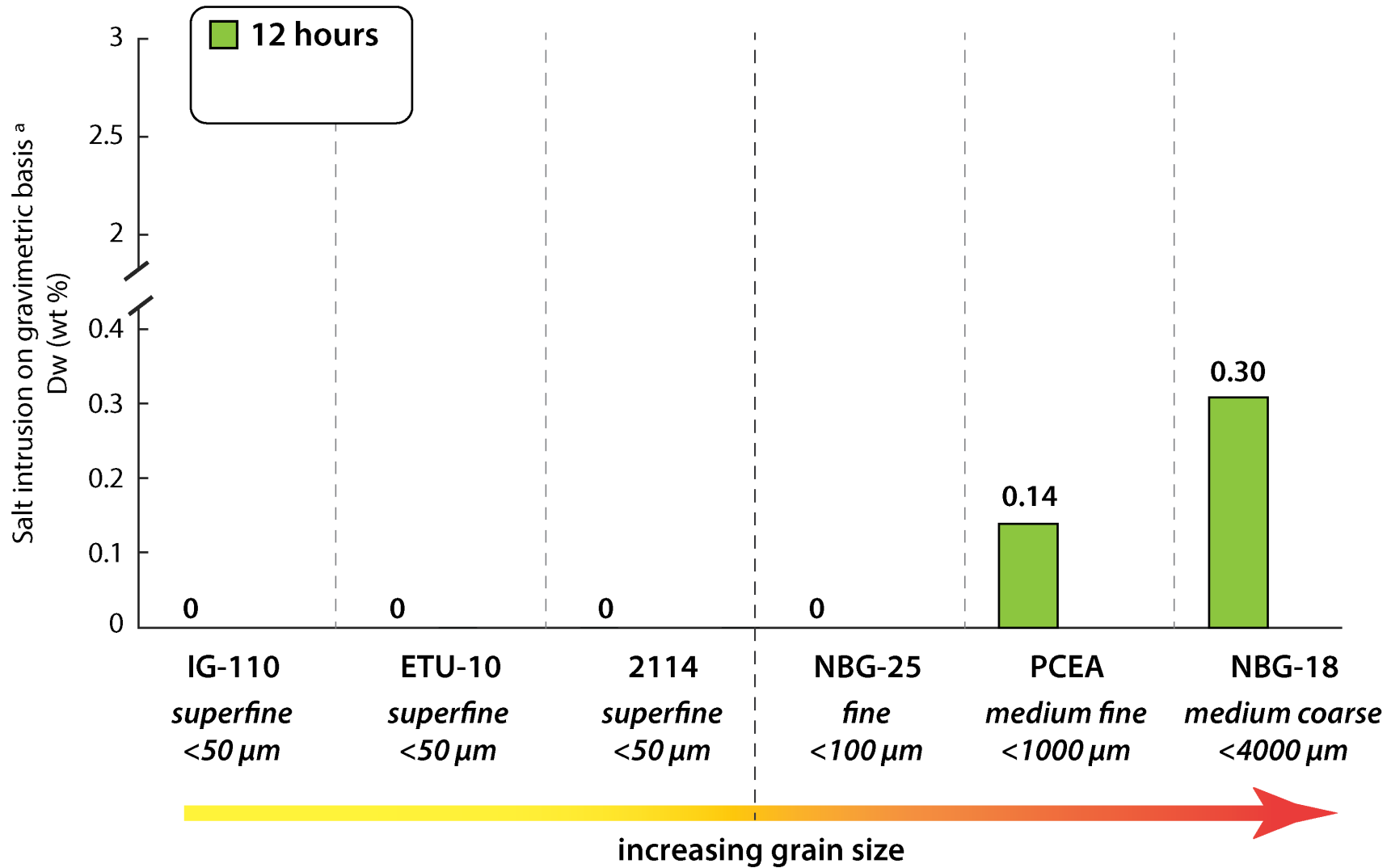
P: 3 bar

Salt: FLiNaK

Time: 12 and 336 hours

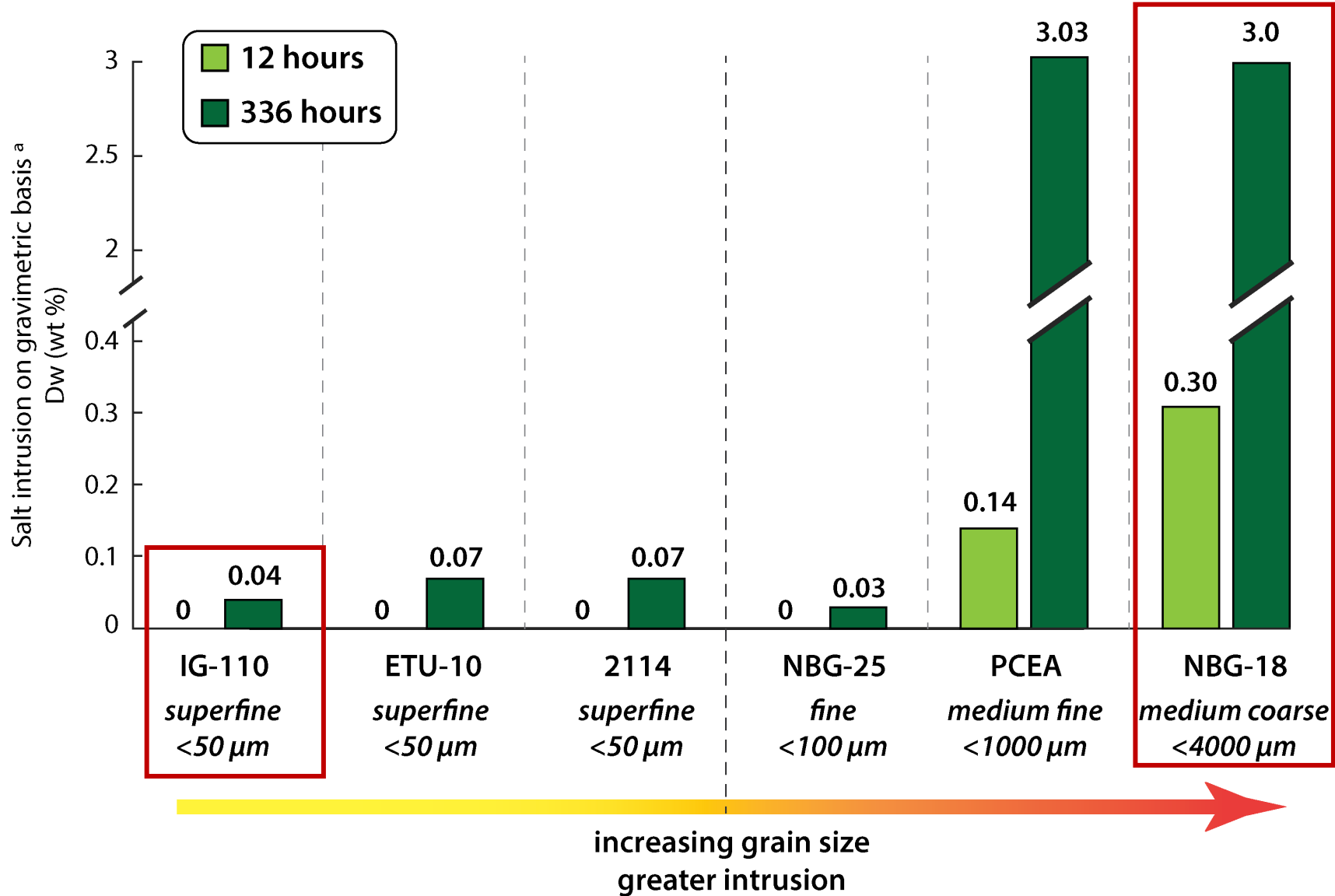
Gravimetric results indicated significant differences on weigh uptake as a function of time

Intrusion Conditions
T: 750°C
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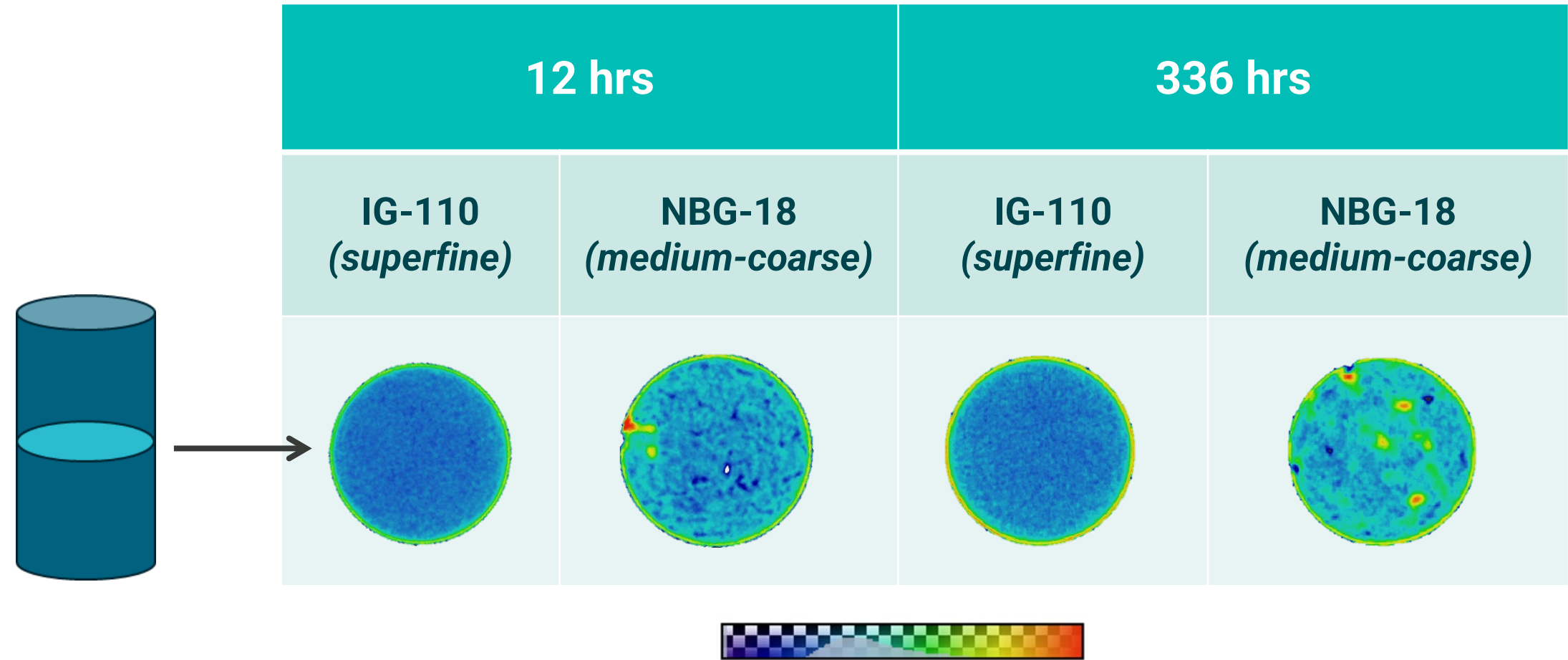


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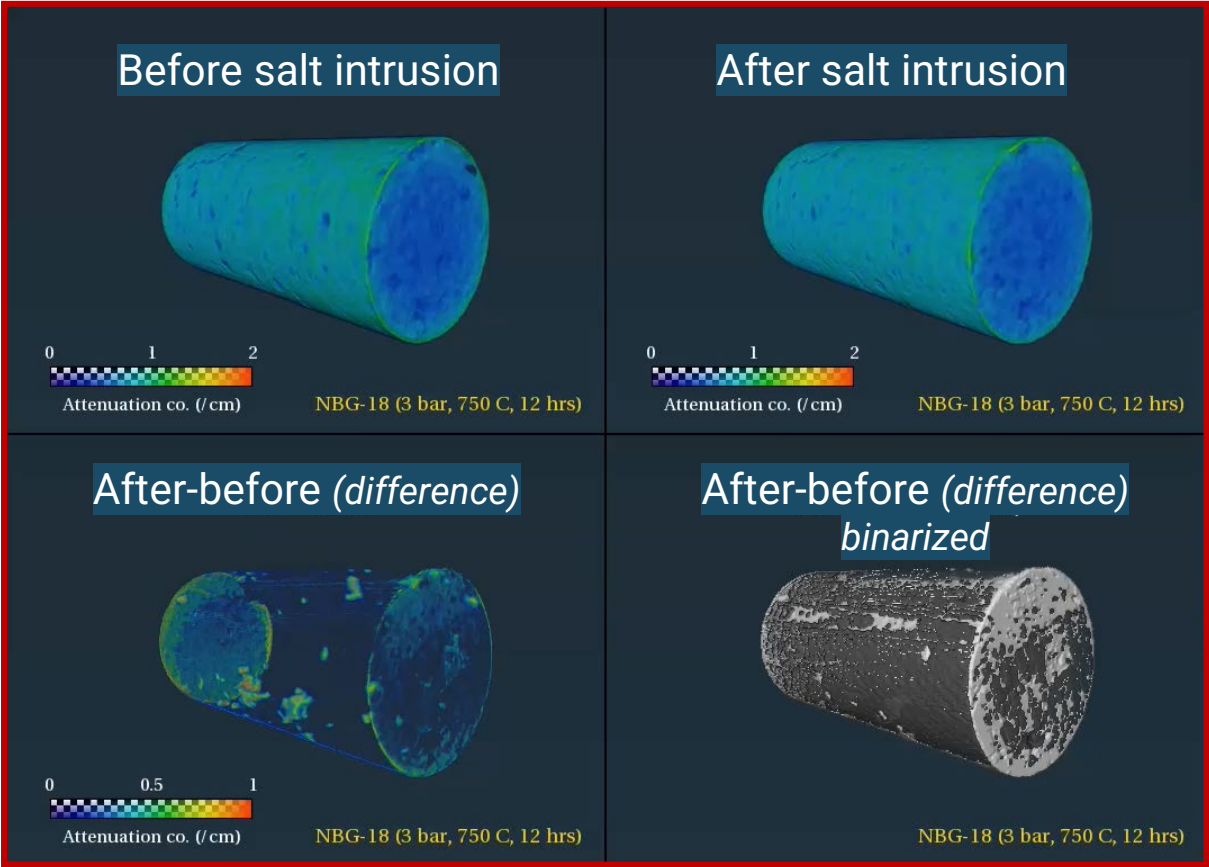


Preliminary Results had Indicated Different Behavior Between Fine and Medium Grain Graphites

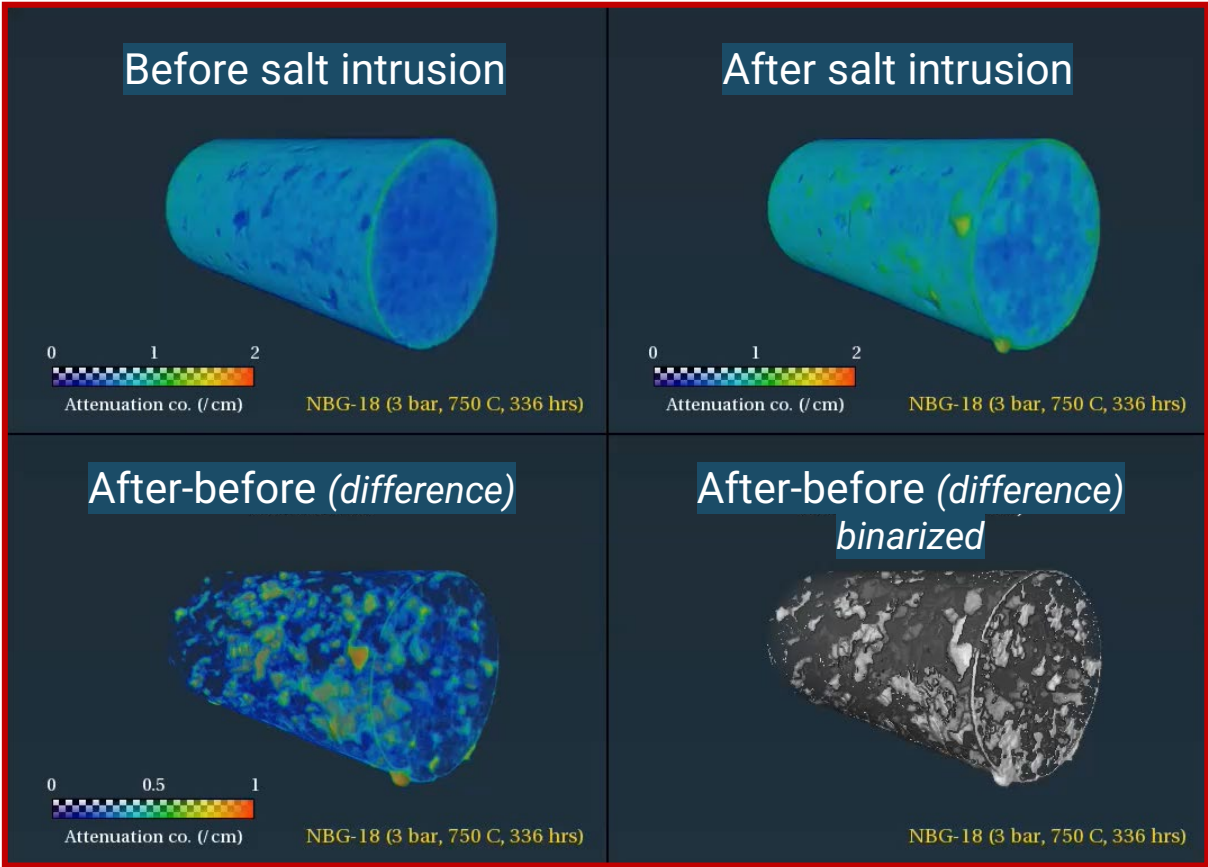


Significant differences for MEDIUM grain graphite grade NBG-18: 12 v 336 hrs

12 hrs



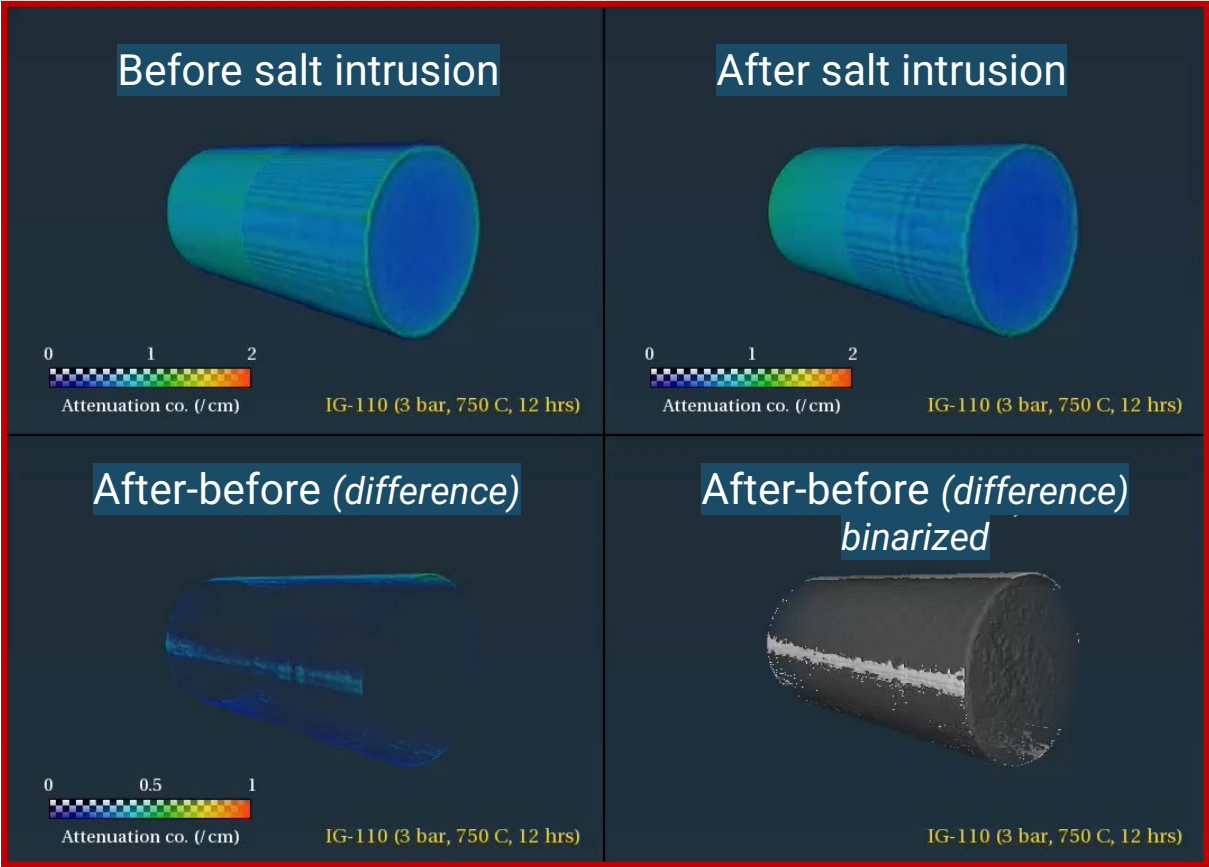
336 hrs



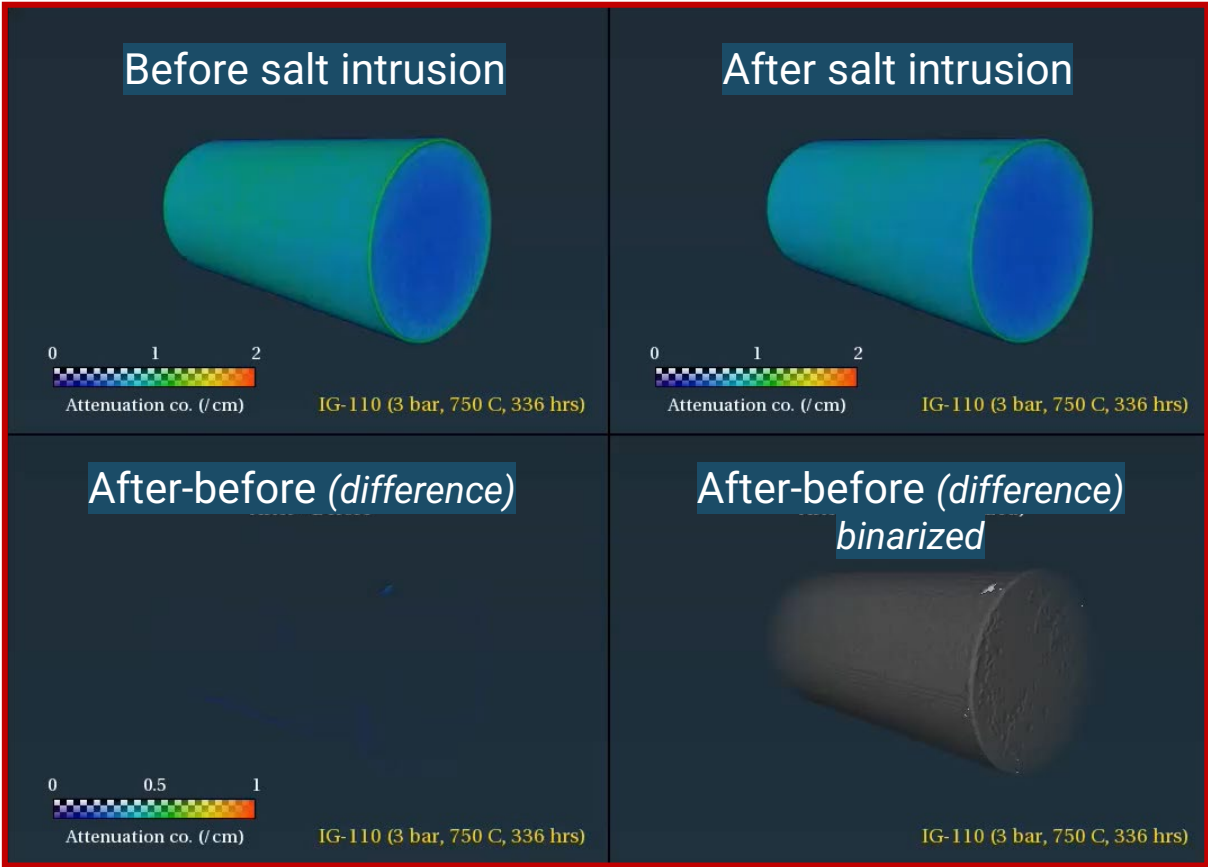
No major differences for FINE-grain graphite grade

IG-110 : 12 v 336 hrs

12 hrs

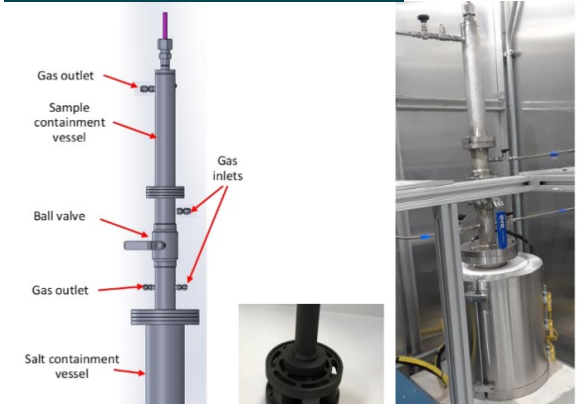


336 hrs

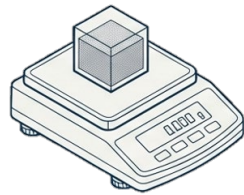
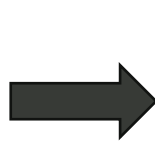


Coupling salt intrusion, nCT and wetting behavior

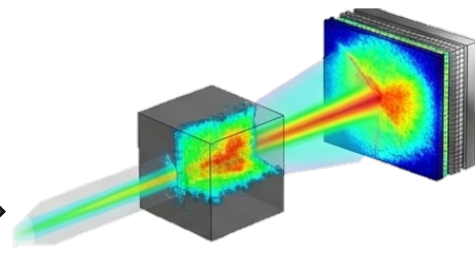
Infiltration study



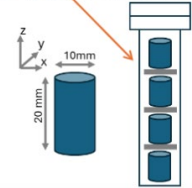
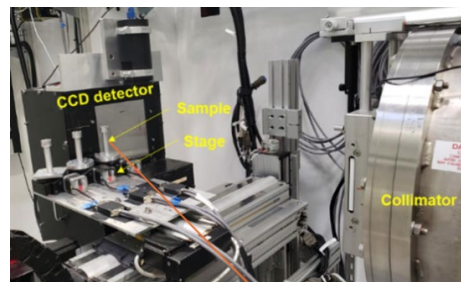
Set up for high temperature, pressure salt intrusion experiment



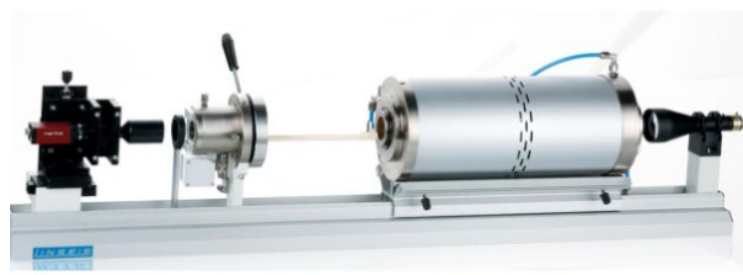
Gravimetric Uptake



HFIR-MARS neutron tomography
Spatially Resolved 3D imaging

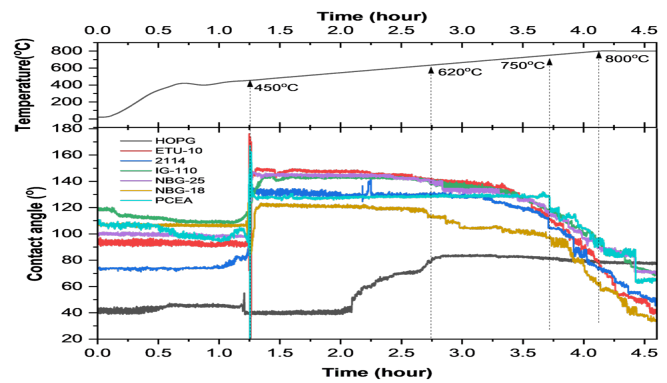


Wettability study



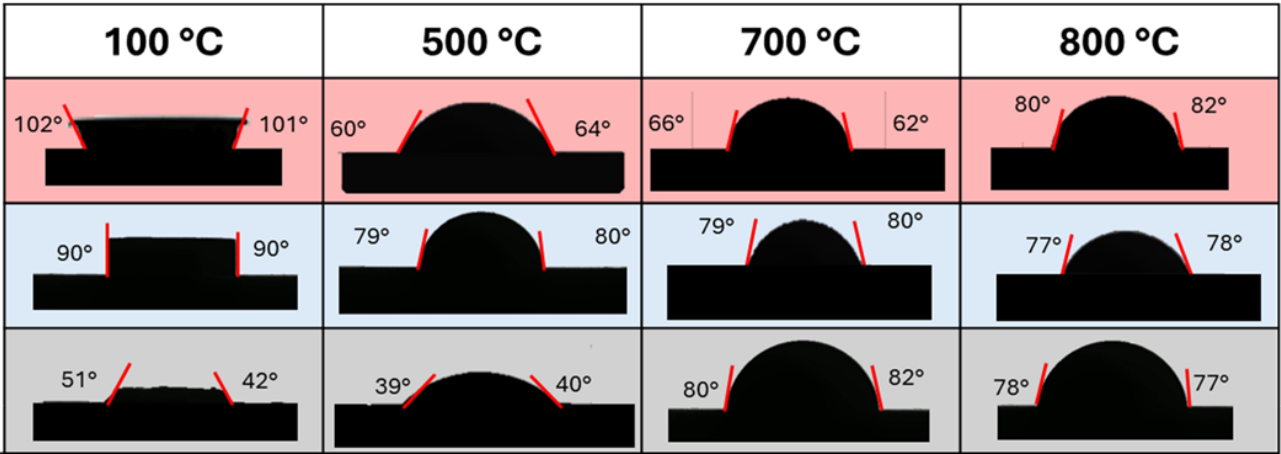
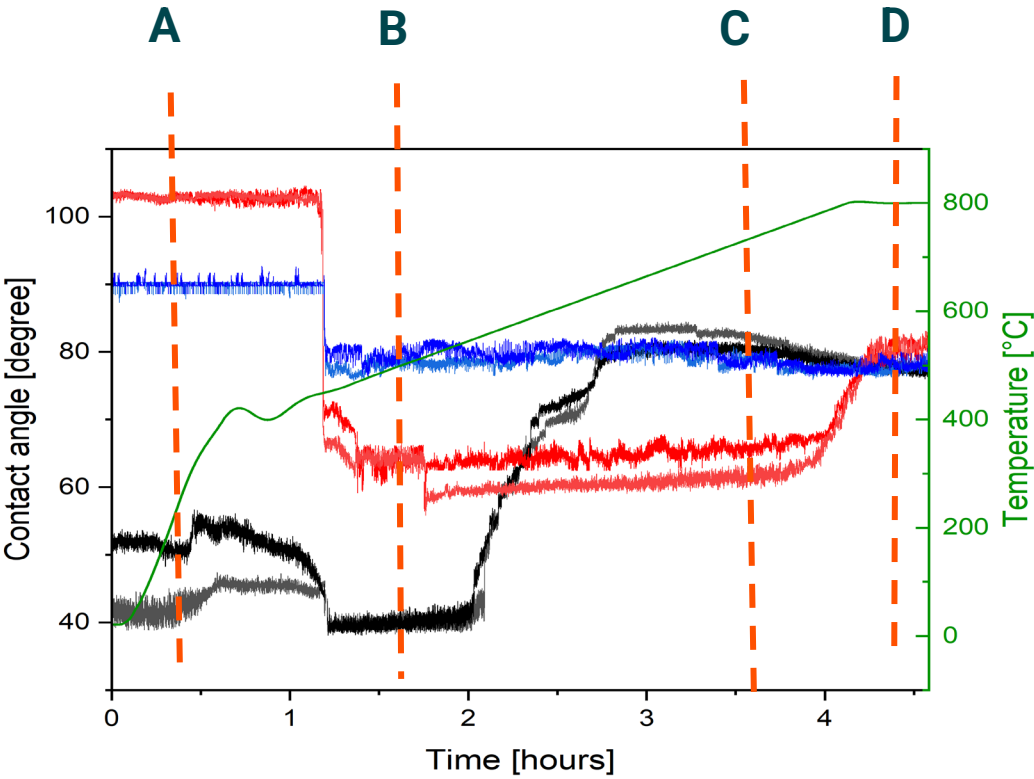
Measuring contact angle of FLiNaK using high temperature optical dilatometer

Temperature (°C)	500 °C	600 °C	700 °C	800 °C
IG-110	 141° 142°	 143° 142°	 135° 134°	 96° 98°
HOPG	 41° 42°	 72° 73°	 81° 82°	 79° 80°



Observed a lot of variability and inconsistencies in measurements

Use HOPG as a reference surface to understand the evolution of FLiNaK contact angle



$\Delta \sim 39^\circ$ $\Delta \sim 14^\circ$ $\Delta \sim 3^\circ$

Initial salt pellet geometry, significantly affects measured contact angle

High-temperature surface energy and interfacial
energy of molten FLiNaK–highly oriented pyrolytic
graphite (HOPG) system from selected contact angle
measurement

Jisue M. Braatz^{1}, Gang Seob Jung², Nidia C. Gallego³*

More recent activities using FLiBe

First FLiBe infiltration study: revealing spatial heterogeneity via n-CT

First demonstration of FLiBe infiltration in nuclear graphite under controlled temperature and pressure conditions. Neutron CT enables direct visualization of spatial infiltration, revealing pronounced heterogeneity in ET-10 despite similar bulk uptake.

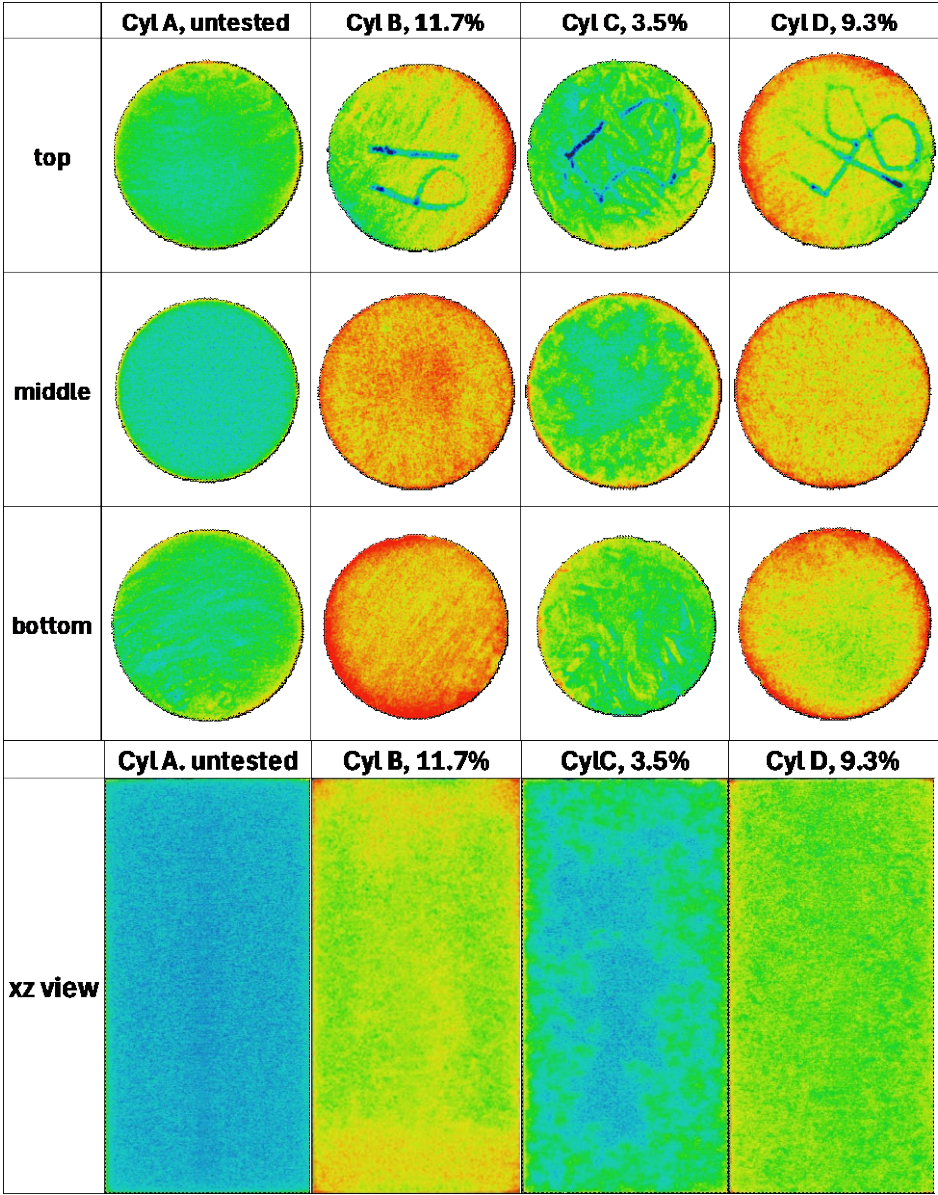
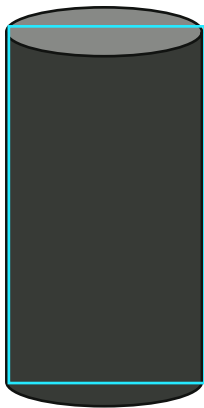
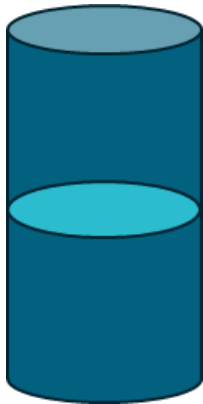
Graphite: ET-10

Infiltration result

Cyl A: untested

Cyl B: 11.7%

Cyl C: 3.5%



Ongoing work: Validation and reactor-relevant testing

Reproducibility and data validation

- Replicate key conditions(P,T, duration)
- Quantify variability across graphite grades
- Focus on anomalous/high infiltration cases
- Establish confidence bounds for infiltration measurement

Ensuring observed trends are robust and not condition-specific

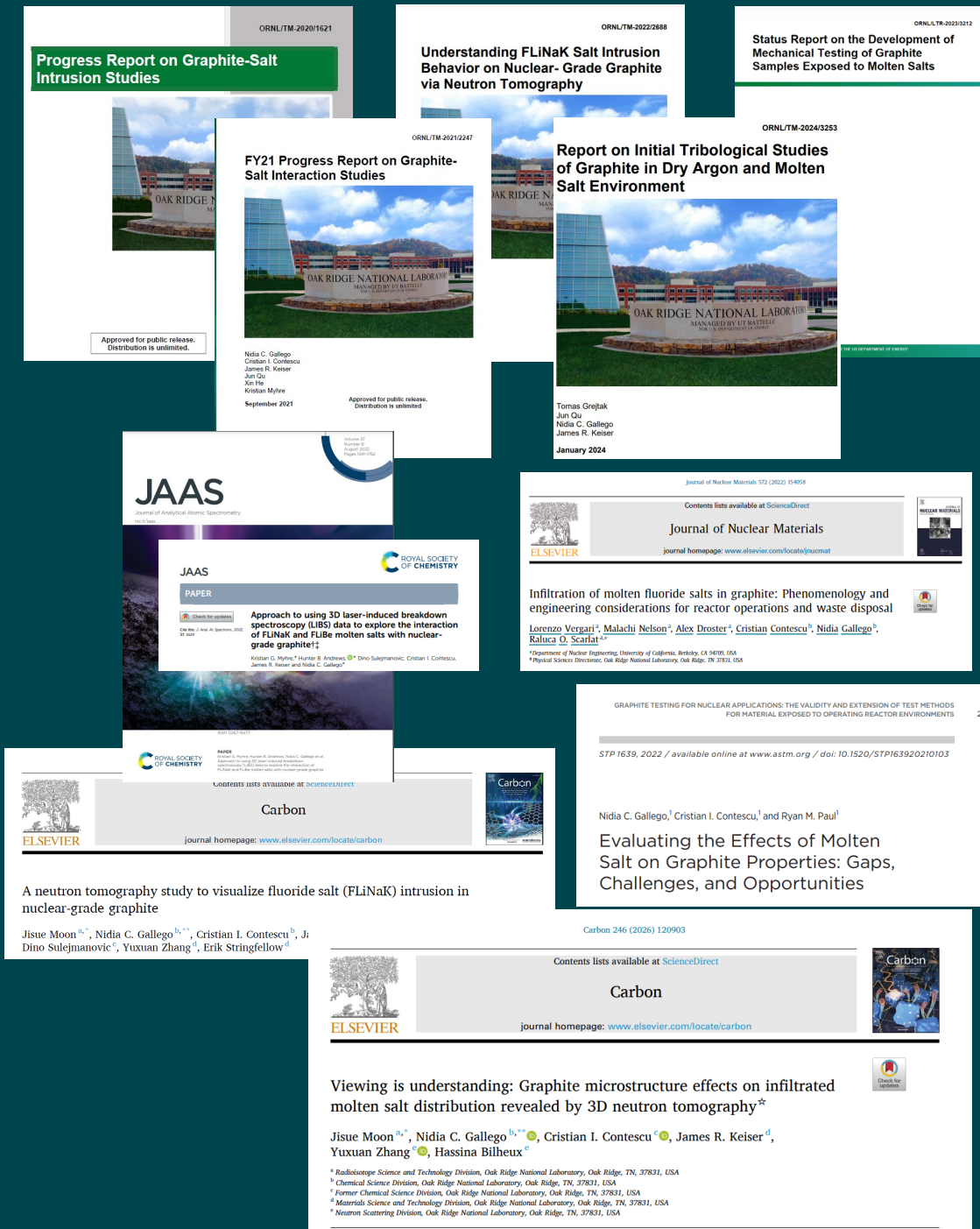
Capsule testing (FLiBe & FLiNaK)

- Static capsule experiment at elevated temperature
- Thermal cycling conditions(realistic operation scenarios)
- Compare:
 - FLiNaK(model salt)
 - FLiBe(reactor salt)
- Evaluate:
 - Infiltration evolution over time
 - Stability under cycling

Bridging lab-scale measurement to reactor-relevant environments

Summary - Intrusion Activities

- Salt intrusion happens but it is highly dependent on temperature, pressure, time and graphite grade
- Different graphite grades evolve toward equilibrium at significantly different rates.
- Data analysis of samples exposed to other conditions of Temperature and Pressure continues.
- Accurate contact angle measurements are critical for the development of predictive models
- Static capsules for cyclic exposures



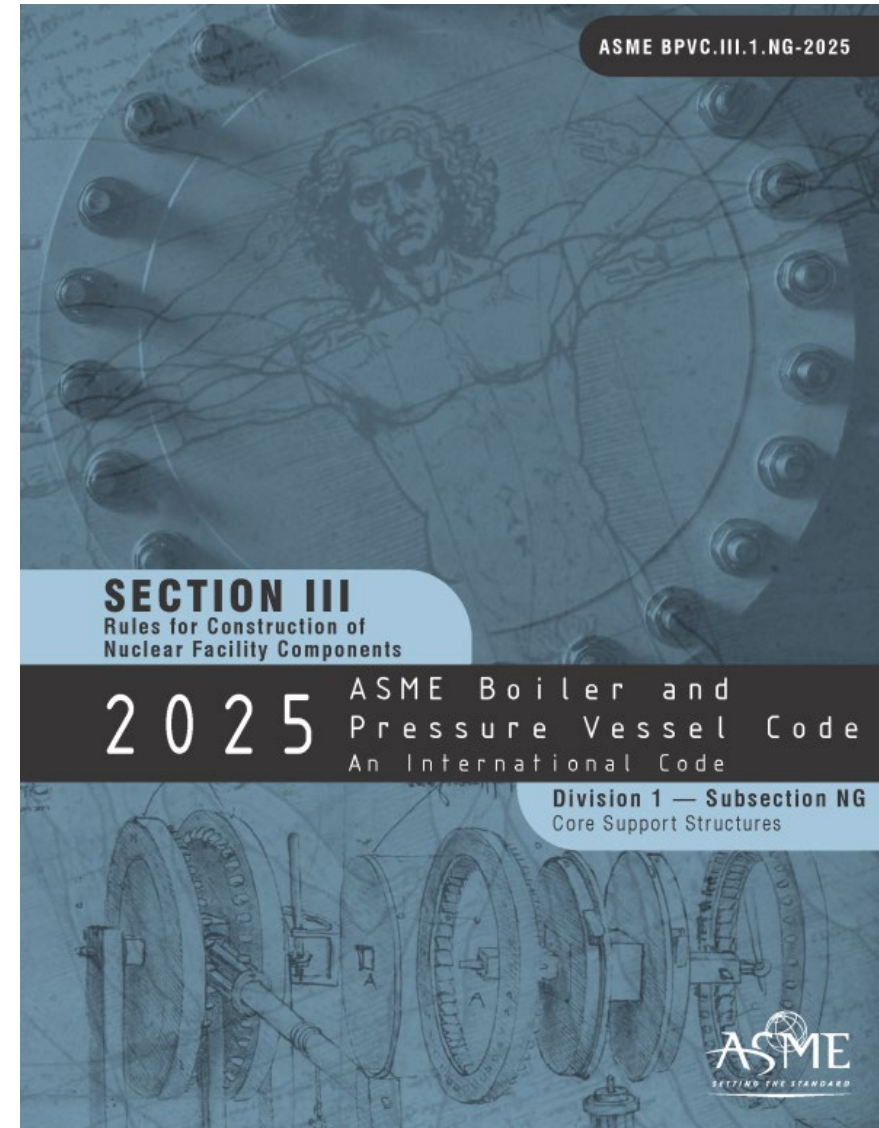
Participation in Codes and Standards Committees

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.

ASTM Standards:
D02.F0 on Manufactured
Carbon and Graphite Products



Upcoming FLiBe capabilities

GOAL

Establish capabilities for handling Be-containing fluoride salts [and fueled salts] and conduct research activities in support of the graphite program and the development of MSR/FHRs

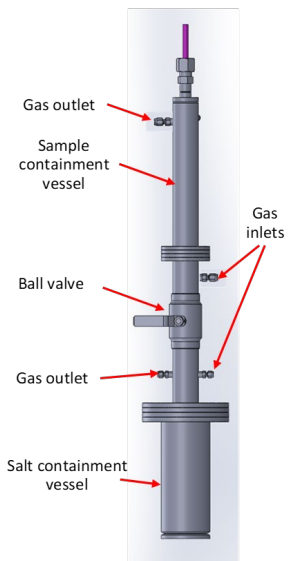
Lab space 4500S – D151 has been assigned for this work

Lab Space Renovation

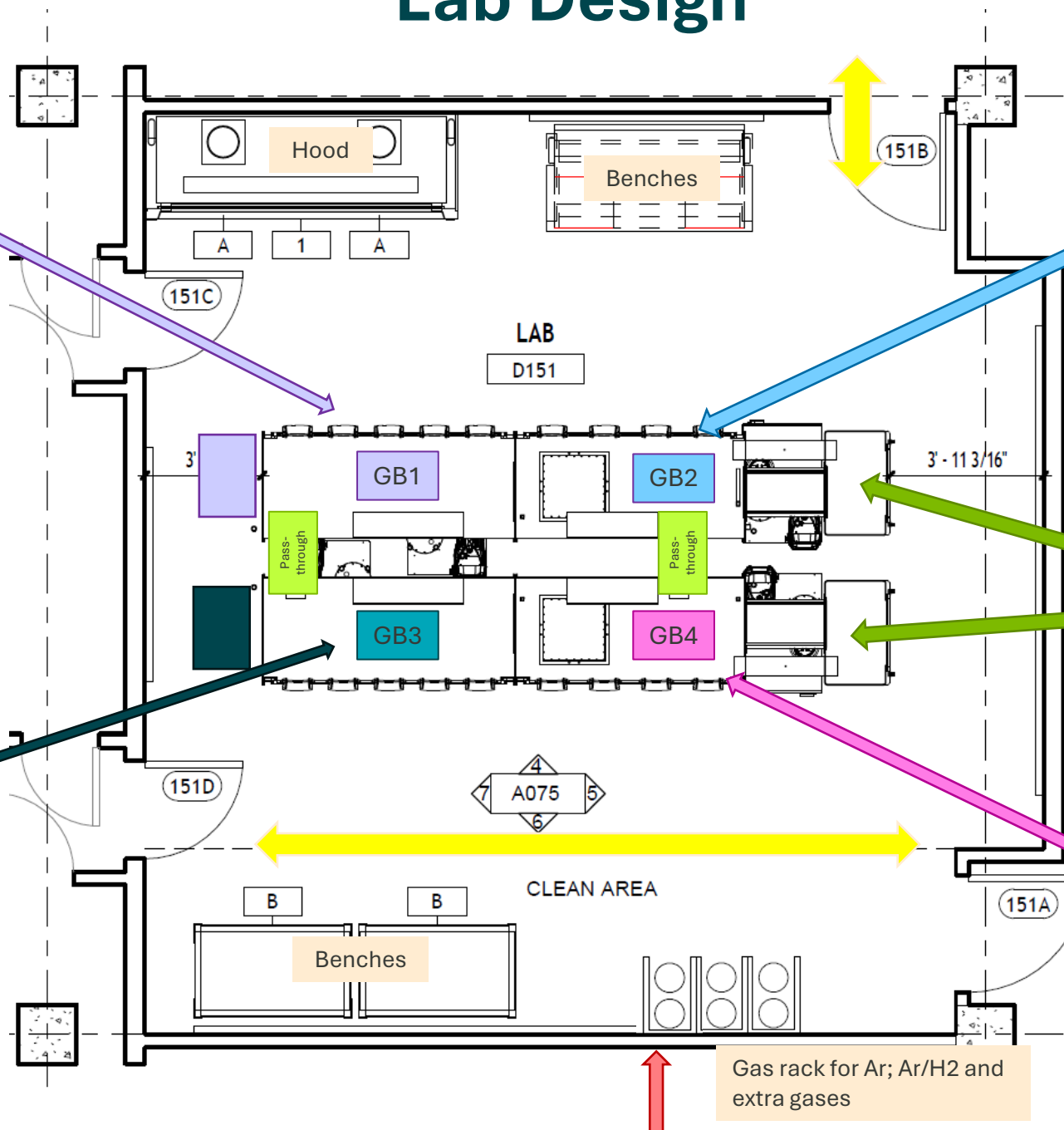
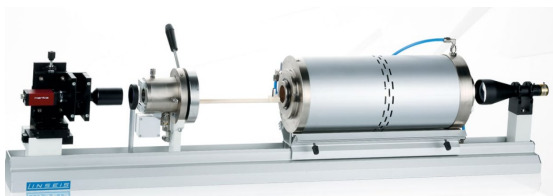


Lab Design

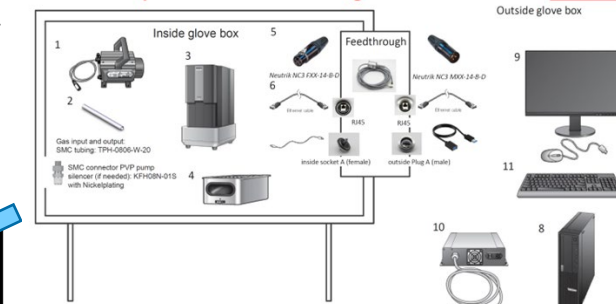
GB1 will have the salt intrusion system (furnace with high pressure, high temperature vessel)



GB4 will have the contact angle system



Technical requirements - PVP Inside glove box



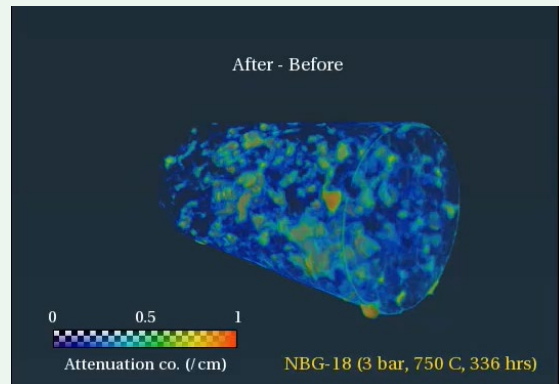
GB2 will have the tabletop SEM.

Two small 'hoods' for staging, providing additional operator safety.

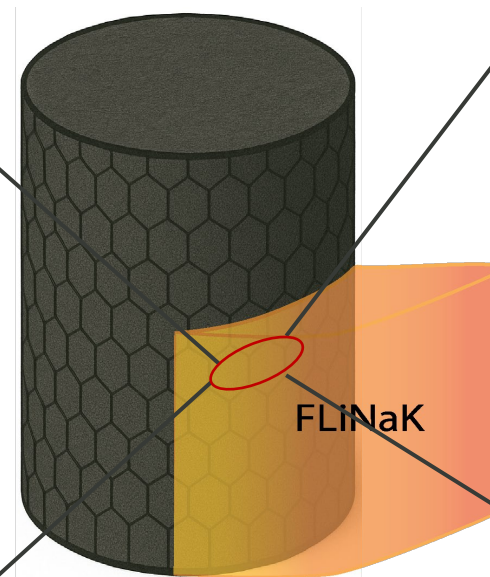


GB2 general purpose: sample preparation, storage, outgassing etc; balance, press, vacuum oven, tools, containers, etc

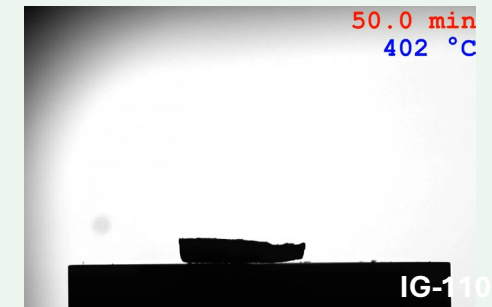
**Salt intrusion visualization:
3D neutron tomography**



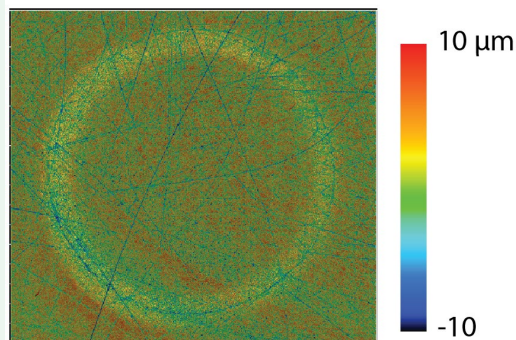
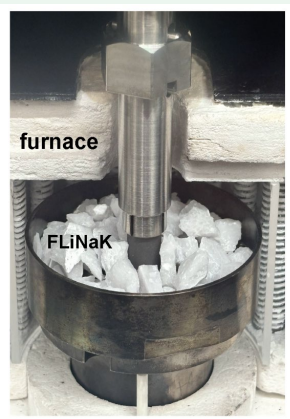
Understanding graphite-salt interactions via a multidisciplinary approach



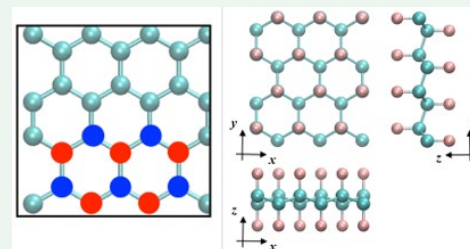
**Salt intrusion prediction:
contact angle measurements**



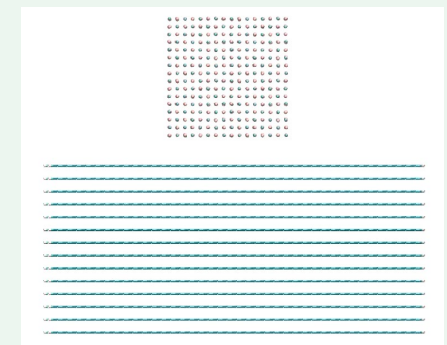
**Wear and friction behavior of
graphite in molten salts:
tribological characterization**



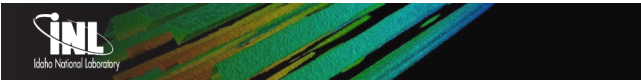
**Modeling chemical
interactions - Fluorination**



**Modeling the wetting behavior:
molecular dynamics simulations**



Want additional information on Graphite?



DOE-ART | NEUP Website | NEUP Proj Files

NDMAS

DOE-ART Graphite

The DOE-ART Graphite R&D program is the primary nuclear graphite research program for the USA. This program focuses on research and development activities necessary to qualify and license graphite components for use within nuclear applications, specifically within advanced reactor designs such as High Temperature Reactor designs. The data generated within the ART Graphite program is intended to be used in conjunction with other publicly available nuclear graphite data such as is contained within the [IAEA Nuclear Graphite Knowledge Base](#). The ART Graphite program is divided into 5 primary research areas providing a combination of data, analysis reports, and pertinent references to describe and explain the trends within the data.

- ▶ **Unirradiated (Baseline):** Establish as-manufactured (Baseline) values for unirradiated material properties that can be used to determine the quantitative changes during irradiation and degradation during nuclear applications.
- ▶ **Irradiation (AGC Experiment):** Establish evolution of material property changes due to irradiation dose and temperature. The AGC Experiment is an irradiation creep experiment which provides creep data for selected graphite grades.
- ▶ **Degradation:** Establish effects of irradiation, oxidation, and molten salt interaction on graphite behavior.
- ▶ **Behavior models:** Predictive and degradation models for graphite behavior.
- ▶ **Licensing and code:** Papers and data supporting ASME code development and NRC license assessment.



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Welcome to the IAEA Nuclear Graphite Knowledge Base

Mission Statement

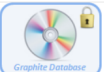
The aim of this knowledge base is to support the preservation and sharing of expert knowledge and experience, across the international Graphite Community.

The knowledge base contains two levels of knowledge:

- General information on the subject of Nuclear Graphite
- Specialist knowledge, secured for members of the international project

Please click on the below images to link to the desired section of the knowledge base

Databases



SAVE-TH-DATE

Joint EPRI Nuclear Graphite Workshop & DOE-NE ART Gas-Cooled Reactor Program Review

JULY 07-09, 2026

Join us at Idaho National Laboratory in Idaho Falls, ID.

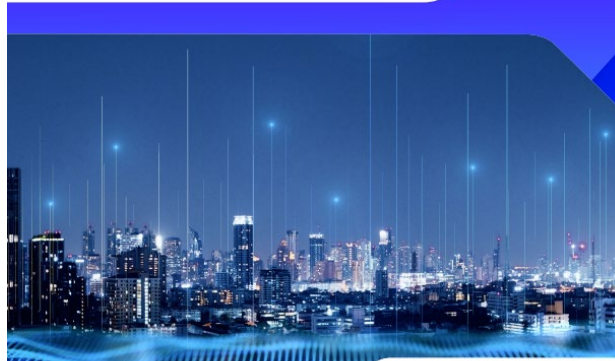
This joint event brings together industry, national laboratories, reactor developers, and technical experts to assess nuclear graphite supply challenges and opportunities, as well as qualification and performance for advanced gas-cooled reactors. Discussions will highlight graphite manufacturing capabilities, qualification progress, and key challenges impacting future deployment.

The program features an EPRI-led, industry-focused workshop and technical sessions addressing graphite supply chain readiness, high-temperature materials qualification, and HTGR core and system modeling capabilities, along with ongoing efforts supporting gas-cooled reactor deployment. The EPRI workshop (July 7) is in-person only, while the ART-GCR Program Review (July 8–9) will also offer a virtual option. A technical tour of in-town graphite and metals testing and qualification laboratories will be offered on July 8.

Register Here

JULY 07 EPRI Nuclear Graphite Workshop	JULY 08 ART-GCR: Graphite	JULY 09 ART-GCR: Methods and Metals
This workshop convenes graphite stakeholders to address challenges in nuclear-grade graphite.	Technical updates on graphite qualification activities supporting the DOE-NE ART program:	Program updates on materials and modeling supporting gas-cooled reactor deployment:
✓ Graphite supply chain readiness, manufacturing, & qualification ✓ Engineering design considerations ✓ Operational performance and maintenance ✓ Regulatory engagement and standards development	✓ Irradiation testing and materials characterization ✓ Graphite degradation (oxidation, molten salt, wear, fracture) ✓ Qualification, modeling, & standards (ASME/ASTM) ✓ Integration with fuel & core design	✓ Structural alloy qualification progress ✓ Environmental effects and materials code development ✓ Advanced core simulation tools and validation ✓ International benchmarking activities

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2024 TECHNICAL REPORT

Graphite for Advanced Nuclear Reactors

Deployment Readiness Review



Shaping the Future of Nuclear Energy
Together with Graphite

Schedule: October 18 (Sun) – 23 (Fri), 2026
Venue: Grand Front Osaka

<https://ingsm26.toyotanso.co.jp/>

Questions?

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