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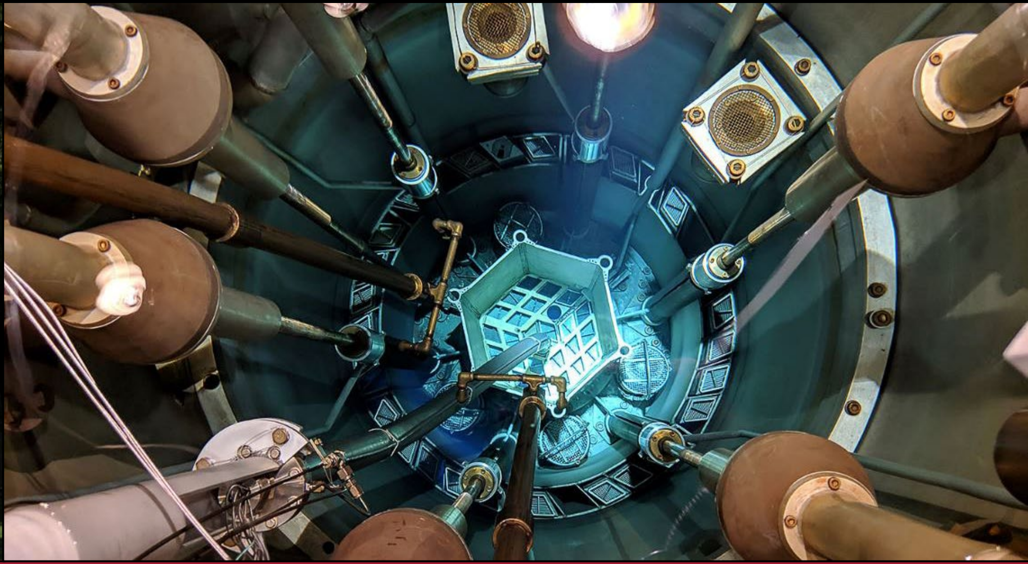


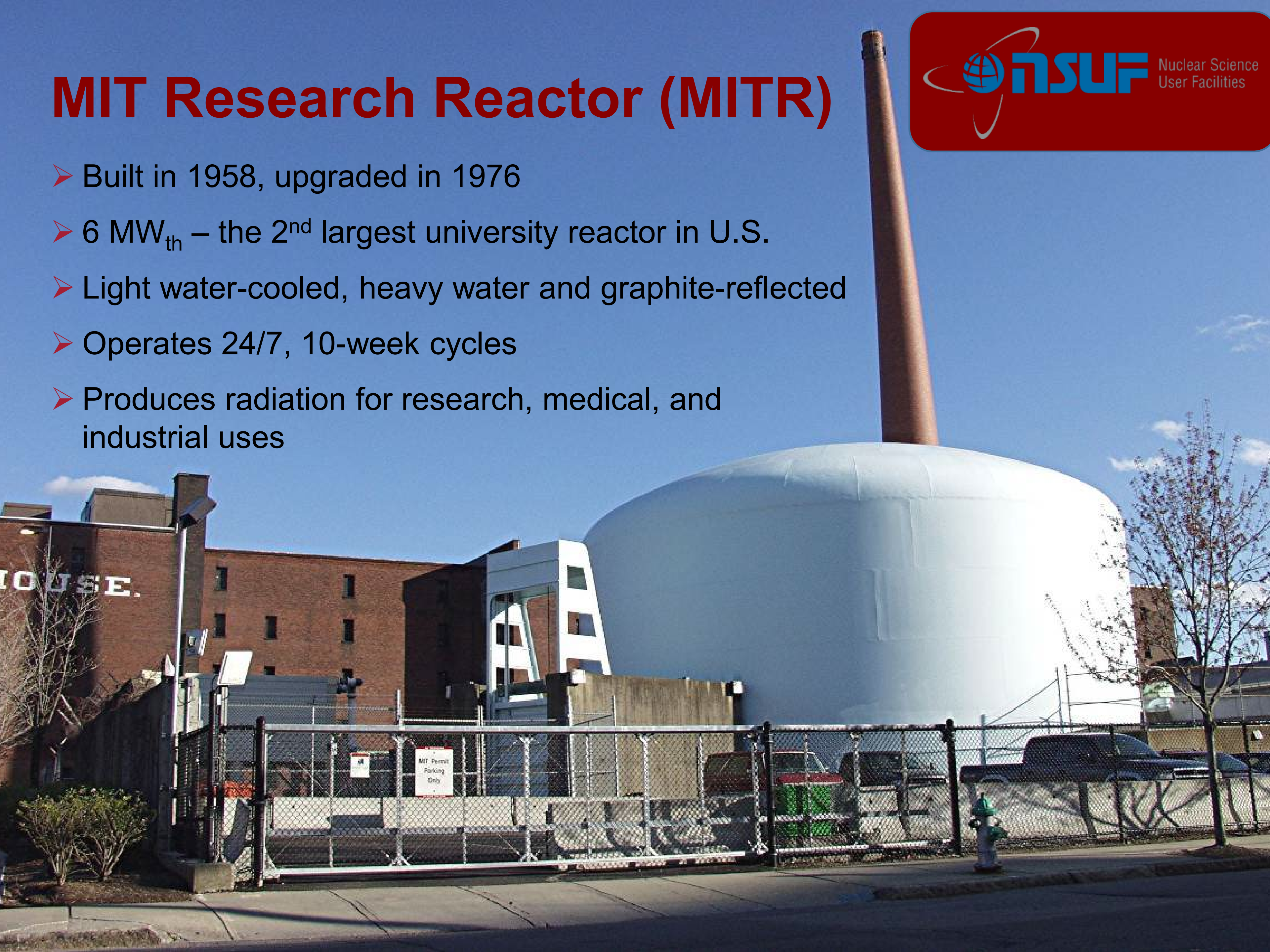
Irradiation of Graphite in Li_2BeF_4 (FLiBe) in the MIT Research Reactor

Guiqiu (Tony) Zheng and David Carpenter

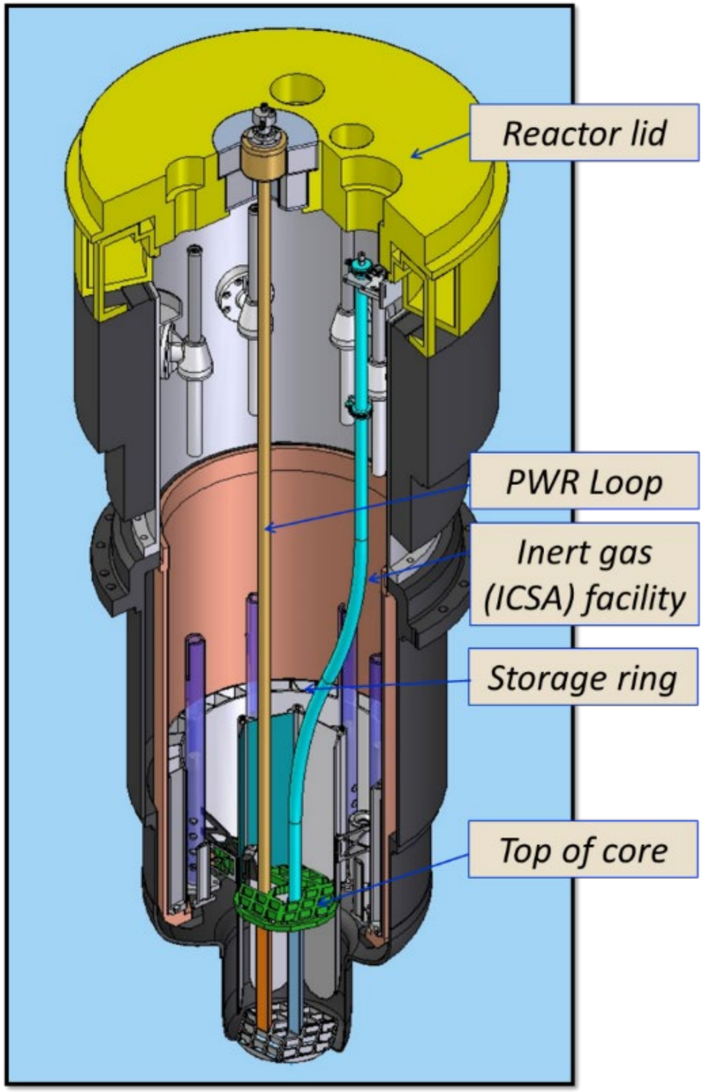
Graphite-Molten Salt Interactions Workshop, 2022-07-21

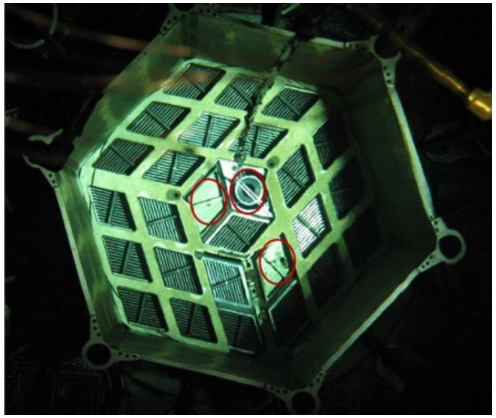
MIT Research Reactor (MITR)

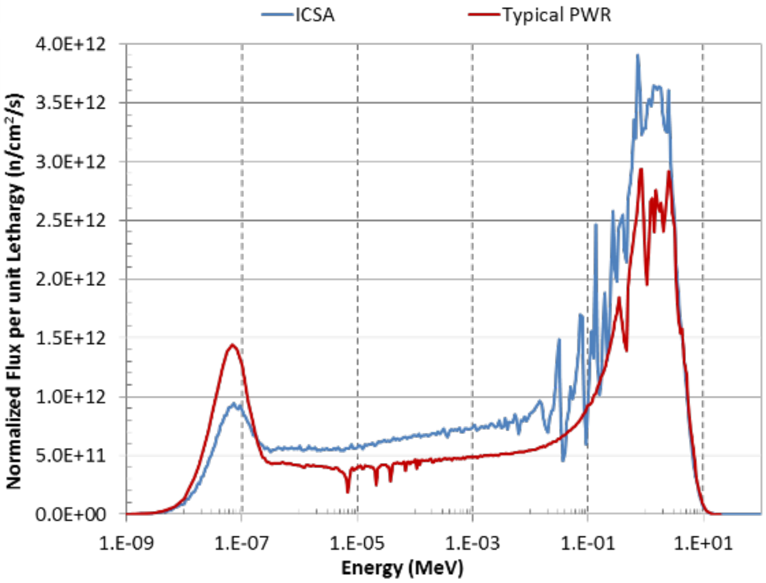
- Built in 1958, upgraded in 1976
- 6 MW_{th} – the 2^{nd} largest university reactor in U.S.
- Light water-cooled, heavy water and graphite-reflected
- Operates 24/7, 10-week cycles
- Produces radiation for research, medical, and industrial uses



MIT Research Reactor (in-core)







Thermal neutrons
3.6×10^{13} n/cm^2-s

Fast neutrons (E>0.1MeV)
1.2×10^{14} n/cm^2-s

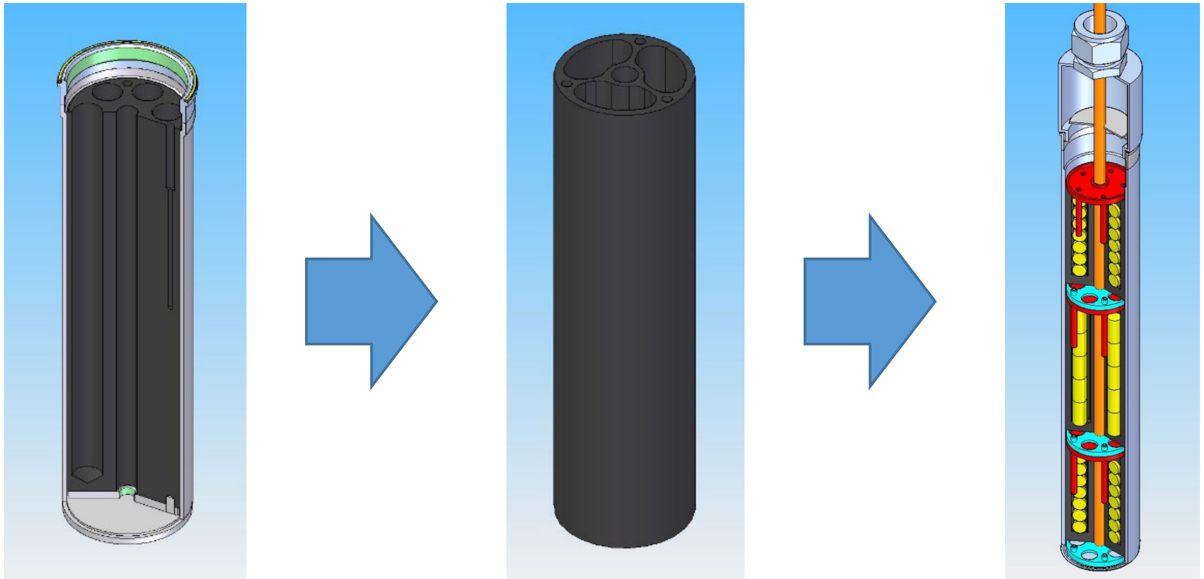
Gamma
3×10^{14} γ/cm^2-s

Radiation environment similar to LWRs

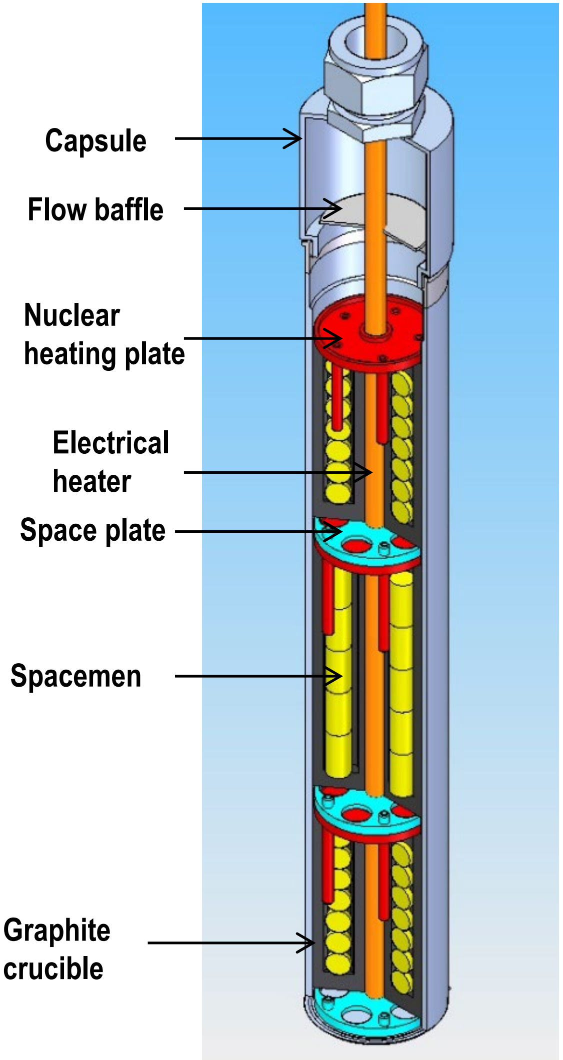
MITR In-core FLiBe Salt Irradiations

MITR Experiment	Primary Objective	Irradiation environment	Irradiation temperature and duration	Mass of total irradiated salt	Tested materials in salt
FS-1 (2013-09)	In-core corrosion, tritium transport	In MITR core	700°C, 1000 hr	121.3g of Li-7 enriched FLiBe	316ss, Hastelloy N, TRISO particles, SiC
FS-2 (2014-07)	In-core corrosion, tritium transport	In MITR core	700°C, 300 hr	326.4g of Li-7 enriched FLiBe	316ss, SiC/SiC, C/C, IG-110U, A3-3
FS-3 (2016-11)	Carbon materials compatibility with FLiBe	In MITR core	700°C, 960 hr	101.5g of Li-7 enriched FLiBe	SINAP provided graphites* and C/C, IG-110U, A3-3

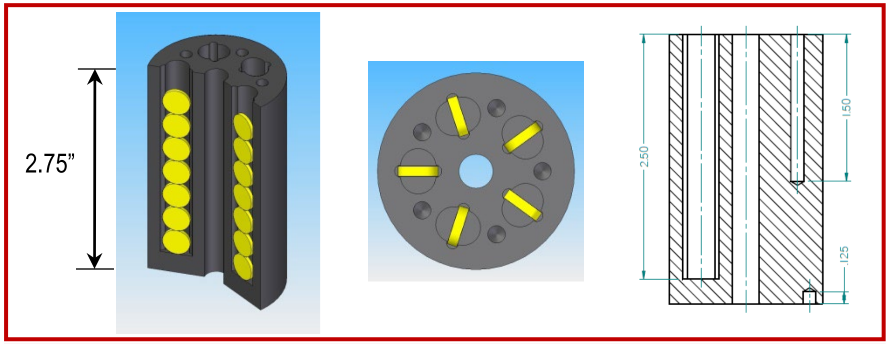
NEUP Nuclear Energy University Programs
U.S. Department of Energy



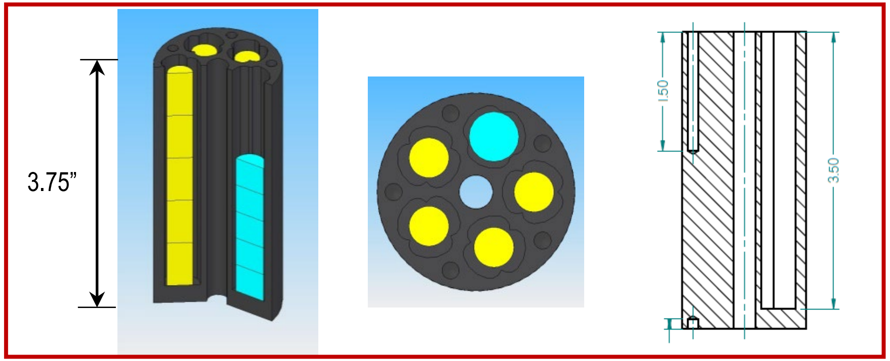
FS-3 Capsule and Crucibles



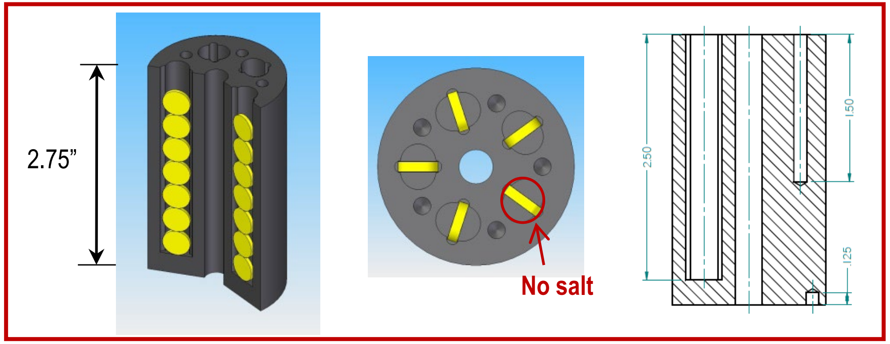
FS-S-Upper



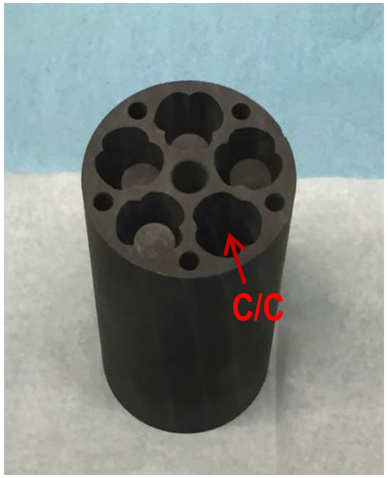
FS-S-Lower



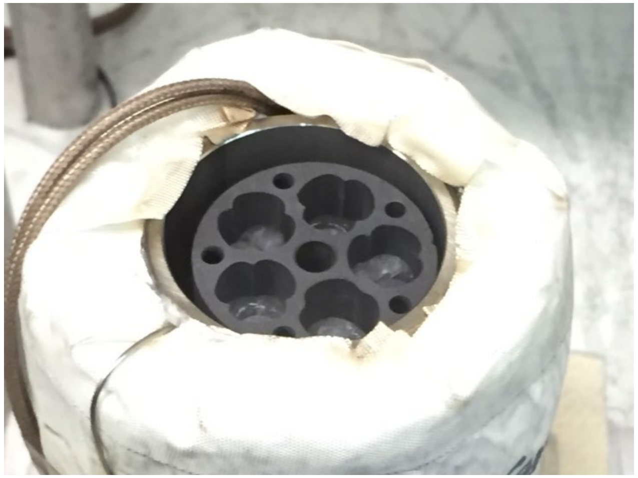
FS-3



FS-3 Samples and Salt Loading



FLiBe filled and froze

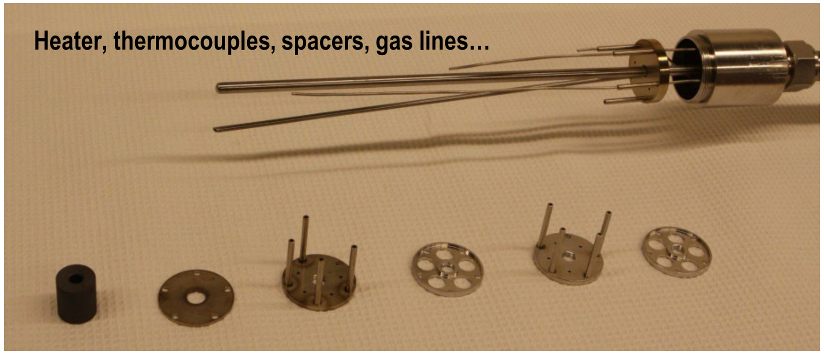


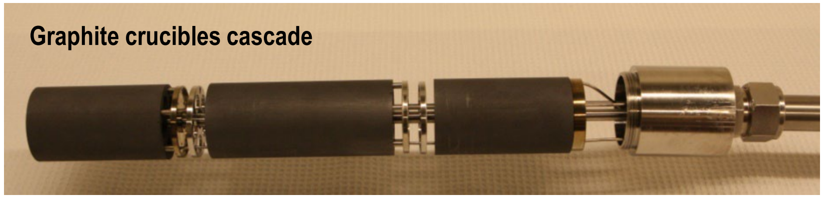
FS-S

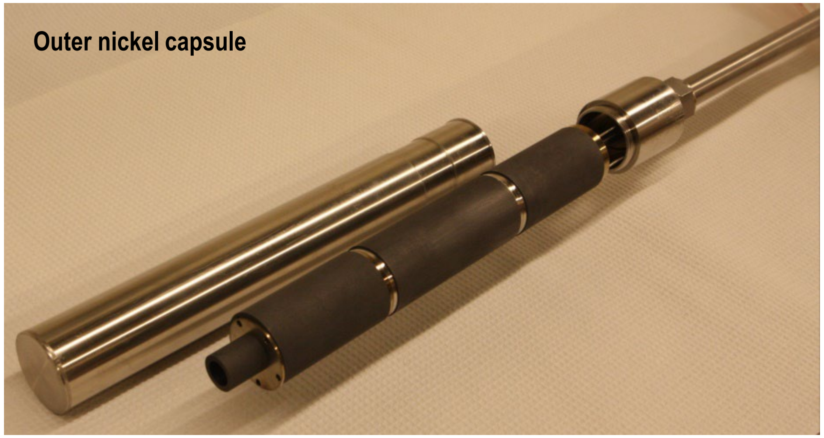
FLiBe filled and froze

FS-3

FS-3 Assembly


Heater, thermocouples, spacers, gas lines…


Graphite crucibles cascade

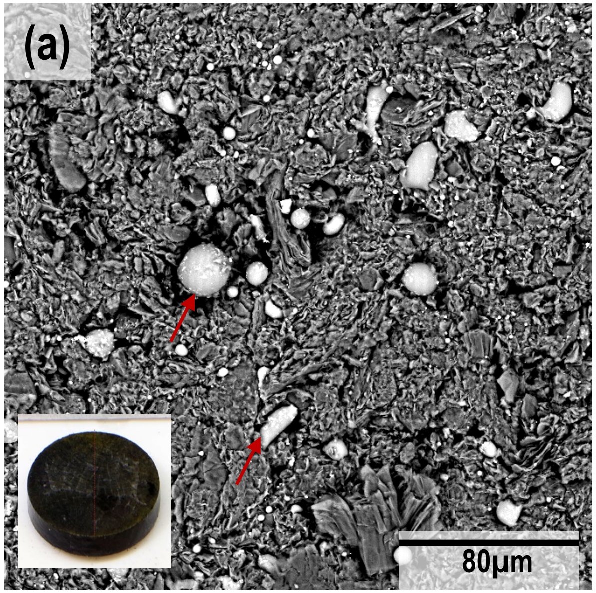

Outer nickel capsule


Tube bending and wiring

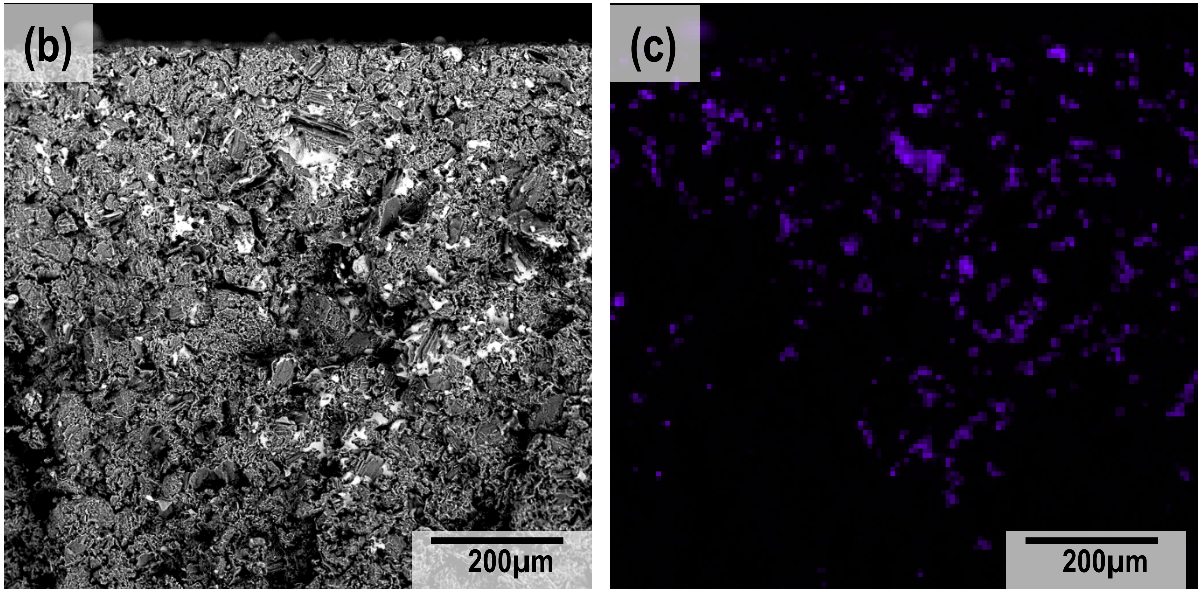
Enhancements in the FS-3 design

- **Optimizing space utilization**
- **Maximizing salt contact**
- **Minimizing specimen shift**
- **Enhancing safety and reliability**
  - Extended double-encapsulation
  - Improve ease of disassembly
  - More fluoride capture options
  - Demonstrated good integrity
  - Electrical guard heating
  - Hydrogen injection system

IG-110U Surface and Fractured Cross-section



SEM on the surface of IG-110U graphite irradiated in molten salt (different extraction approach)

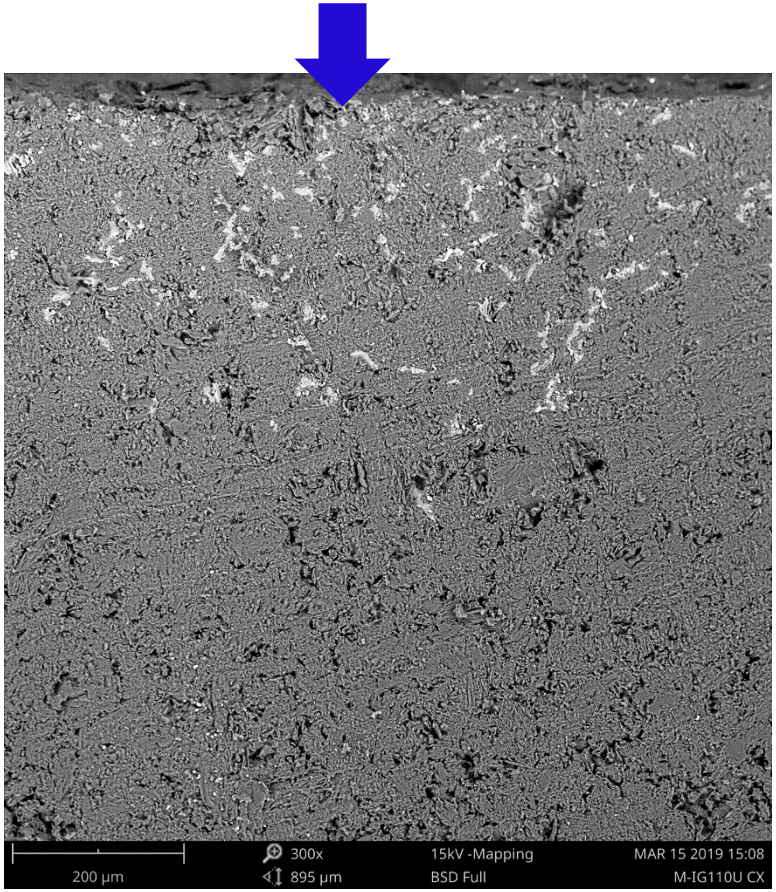


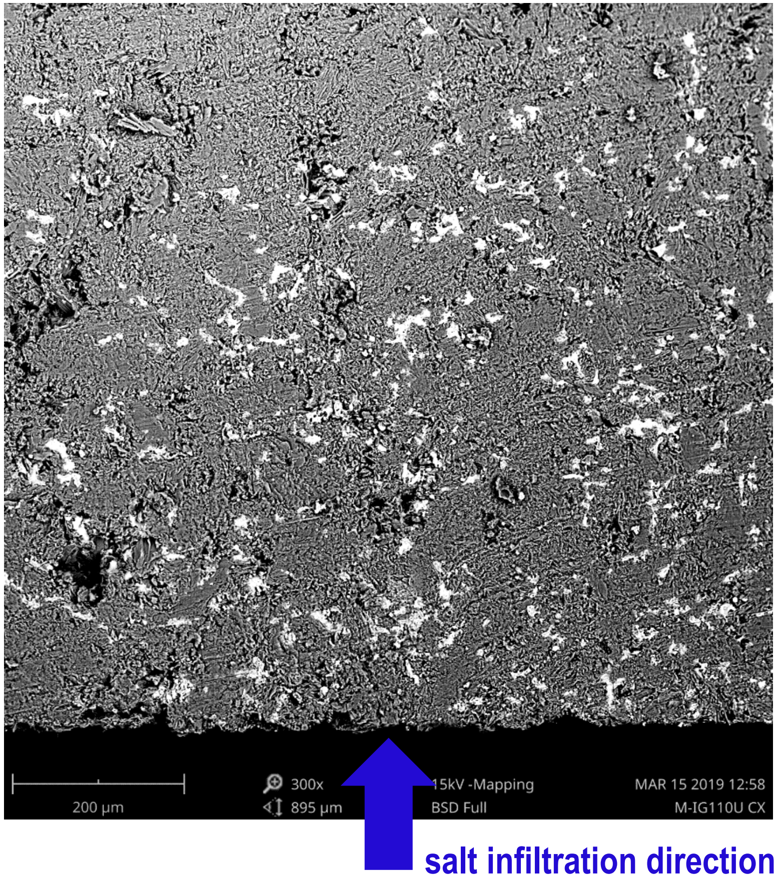
SEM and EDS fluorine mapping on the fractured cross-section of IG-110U graphite irradiated in molten salt

- ❖ **Round salt particles with various sizes on surface**
- ❖ **Significant amount of molten salt infiltrated into IG-110U**

Flattened Cross-section of IG-110U

salt infiltration direction



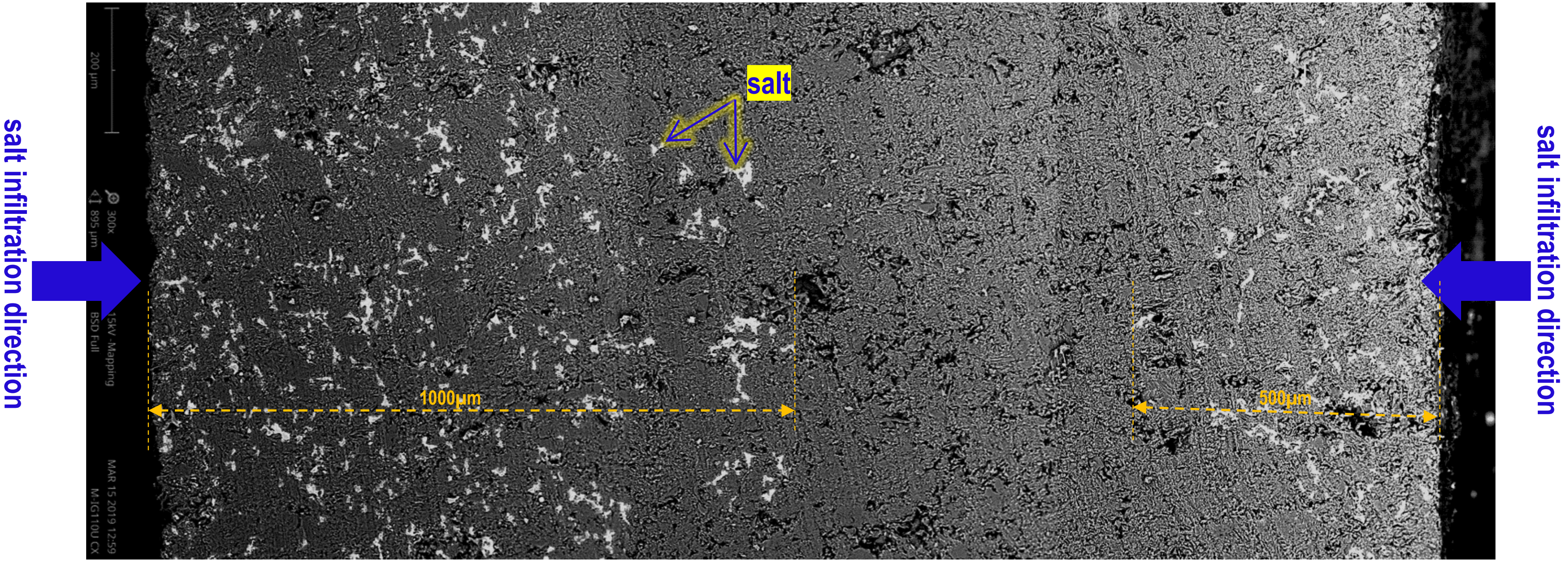


salt infiltration direction

Flattened cross-section (cut with blade)

❖ Salt infiltration depth varies by location

Depth of Salt Infiltration in IG-110U

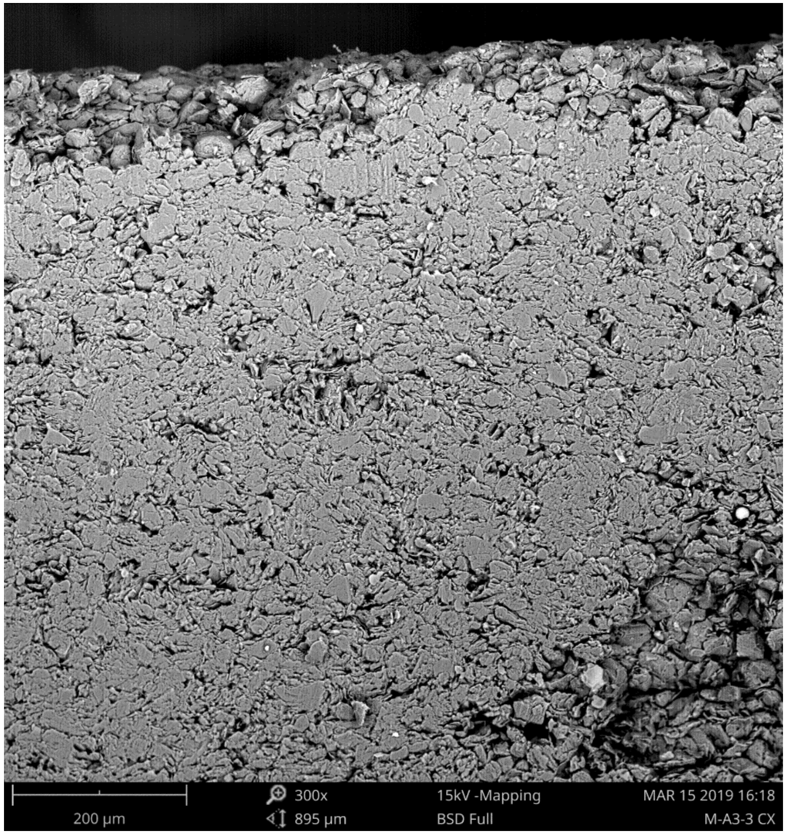


Stitched SEM images of flatted cross-section

❖ **Maximum salt infiltration depth ~1000 microns into graphite**

Cross-sections of A3-3

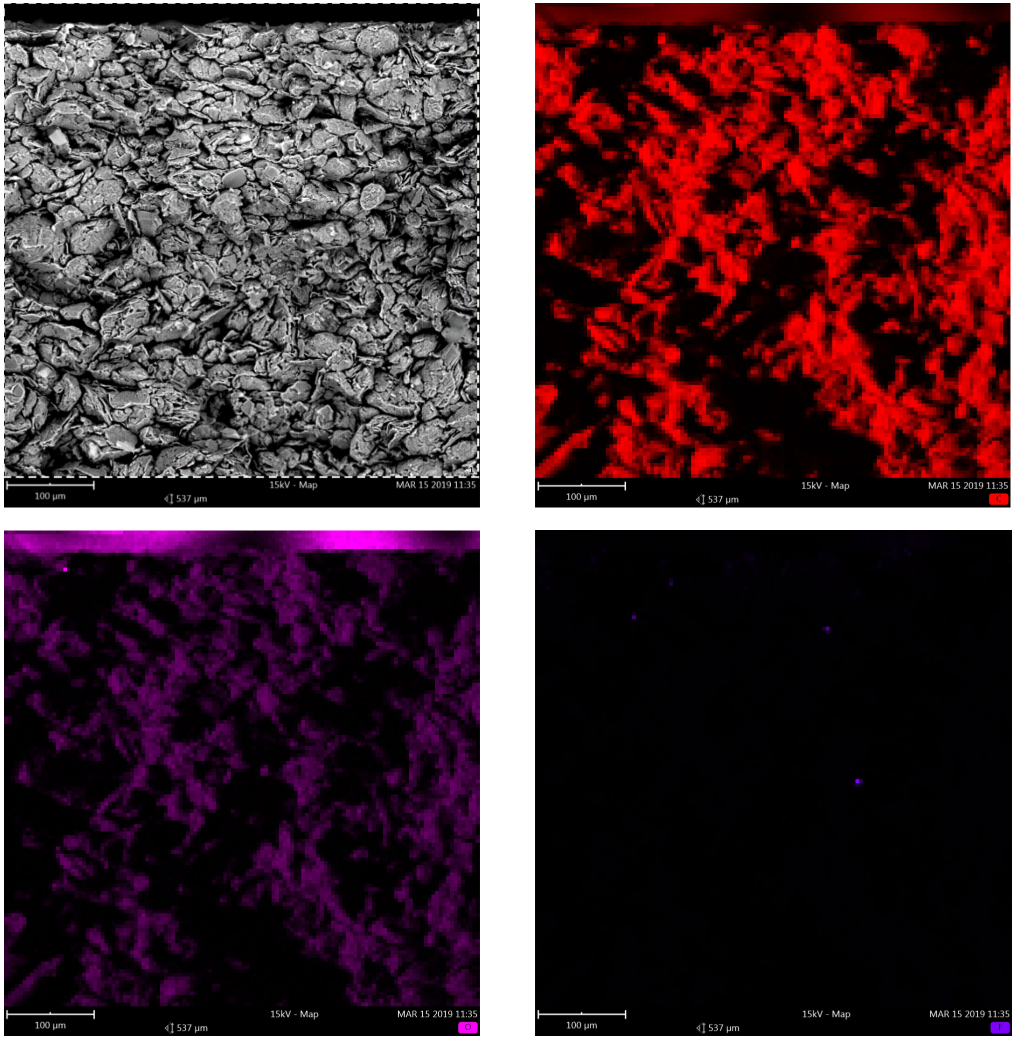
fractured cross-section



flattened cross-section

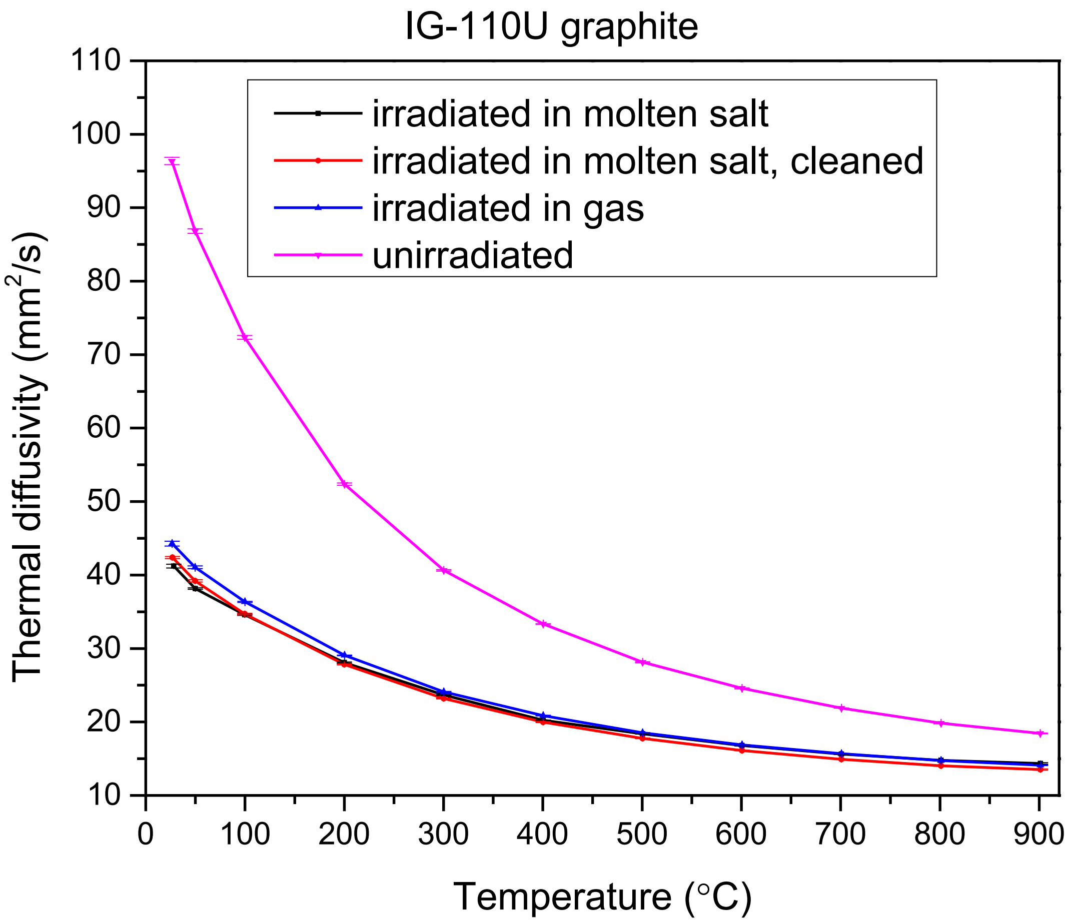
- No salt infiltration observed in in-core molten salt irradiated A3-3 graphite

EDS of Fractured A3-3 Cross-section



EDS mapping on fractured cross-section of irradiated A3-3 confirms that it is resistant to molten salt infiltration under neutron irradiation.

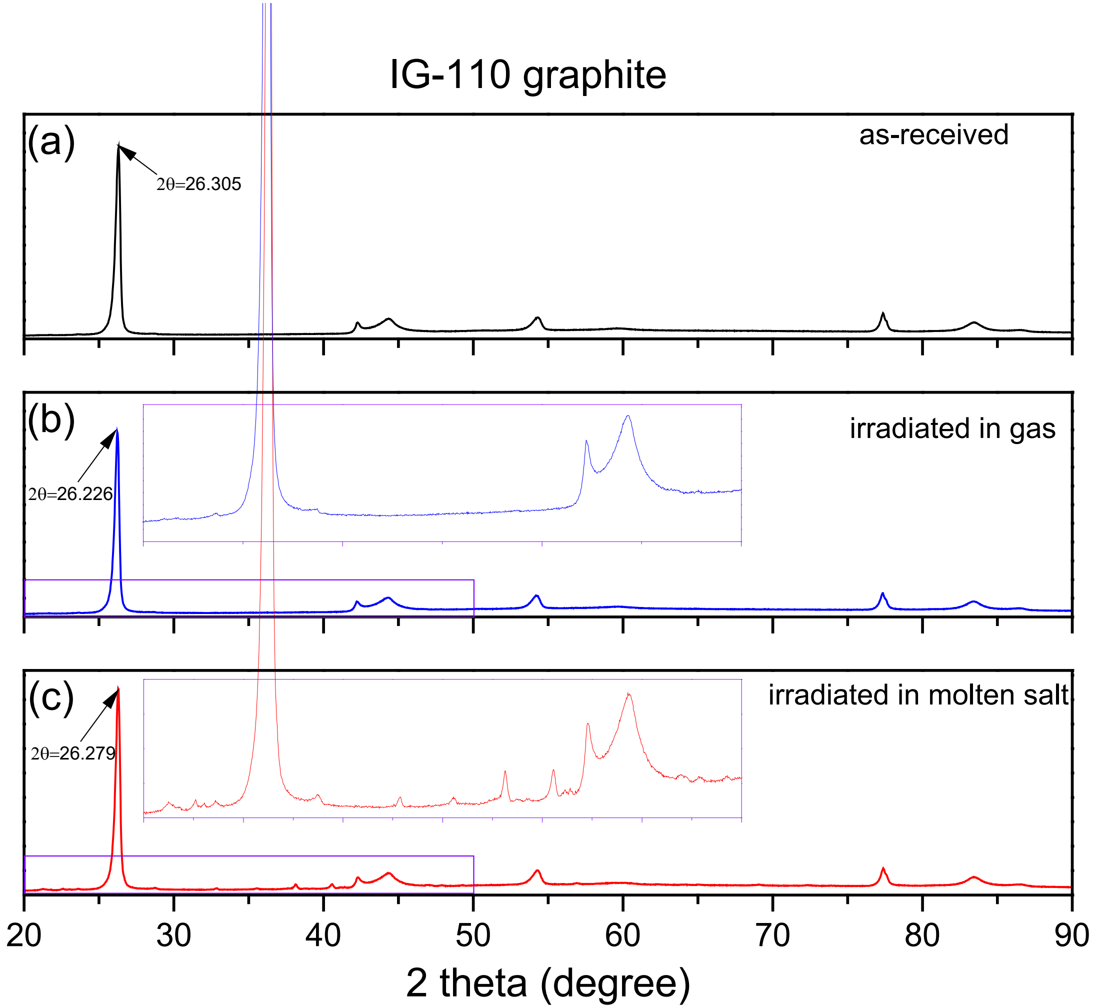
Effects on Thermal Diffusivity

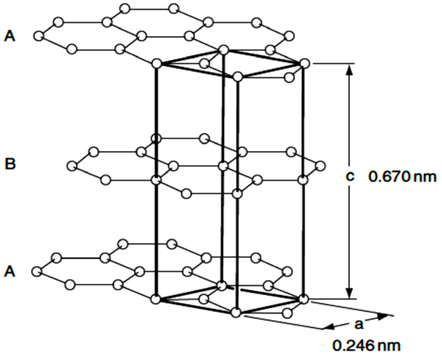


INL instrument: LFA427
temperature: RT to 900°C

1. **Neutron irradiation significantly changes the thermal diffusivity of graphite**
2. **Infiltrated salt in graphite affects thermal diffusivity: comparing with cleaned sample, lower thermal diffusivity at <~150°C, higher thermal diffusivity at >~150°C**

Quantitative Microstructure Analysis





sample	$d_{(002)}$ (nm)	c (nm)	a (nm)	$\overline{g}$ (%)
IG-110, non-irrad	0.33699	0.67397	0.24655	81.5431
IG-110, irrad. in gas	0.33706	0.67413	0.24639	80.6665
IG-110, irrad. in salt	0.33737	0.67474	0.24658	77.1118

Literature[1-3]:
High pressure molten salt infiltration decreases $d_{(002)}$-spacing; low dose neutron irradiation increases $d_{(002)}$-spacing

This study:
In-core molten salt irradiation increases lattice parameters, decreases $\overline{g}$

Z. He *et al.*, “Improvement of stacking order in graphite by molten fluoride salt infiltration,” *Carbon N. Y.*, vol. 72, pp. 304–311, 2014.
T. D. Burchell and T. R. Pavlov, “Graphite: Properties and Characteristics,” in *Comprehensive Nuclear Materials*, vol. 7, Elsevier, 2020, pp. 355–381.
H. Tang *et al.*, “Infiltration of graphite by molten 2LiF–BeF2 salt,” *J. Mater. Sci.*, vol. 52, no. 19, pp. 11346–11359, Oct. 2017

Summary

- Deep FLiBe salt infiltration was observed in IG-110U, but not in A3-3 after in-core irradiation
- Infiltration of molten salt into IG-110U graphite impacts thermal diffusivity at different temperature ranges
- Combined effects of salt exposure and irradiation change graphite lattice parameters and degree of graphitization ($\overline{g}$)
- Understanding of the mechanism of salt infiltration in graphite under neutron irradiation requires further investigation

Acknowledgements

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Research Group

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Safety and Operation Groups

1861

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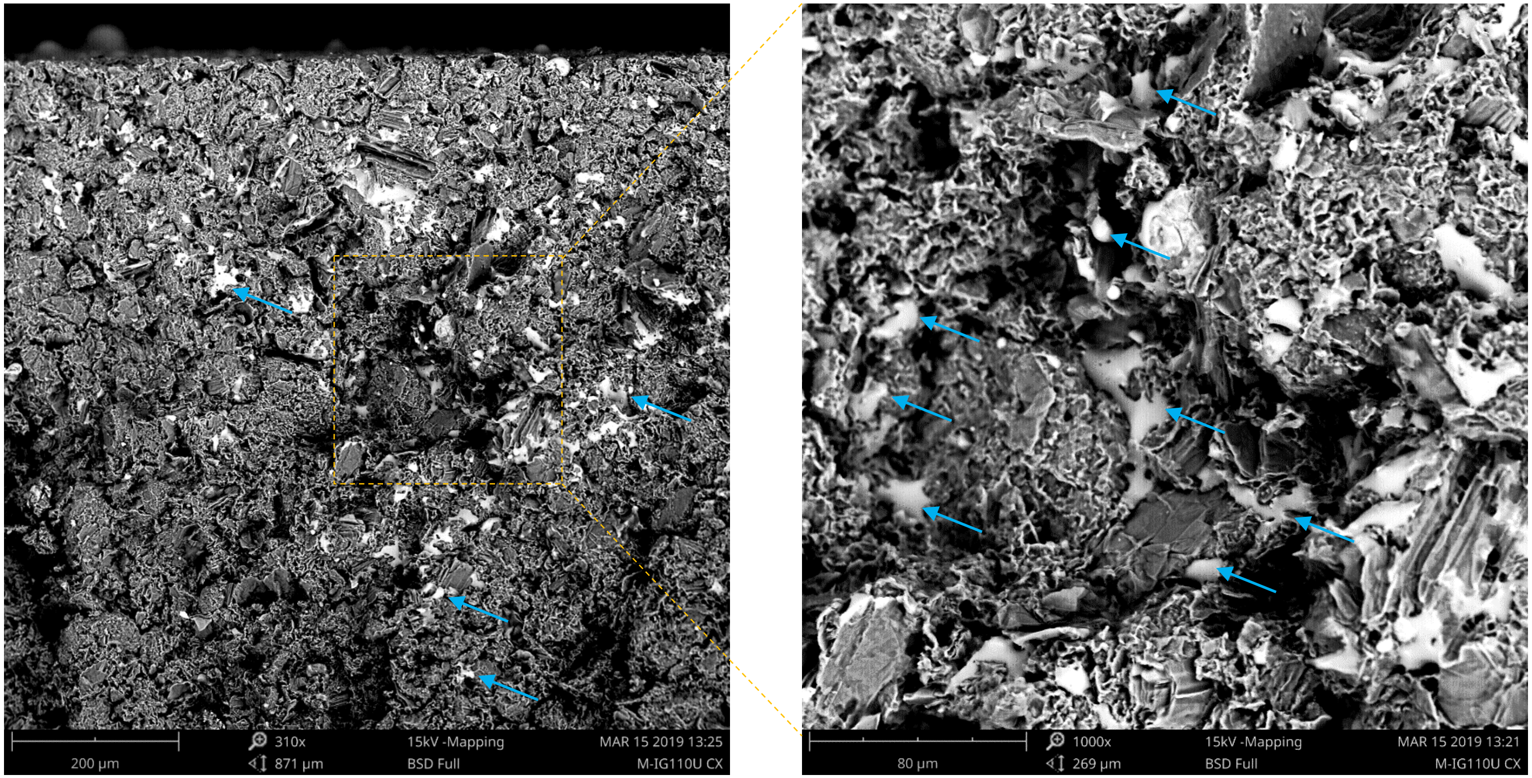
OAK RIDGE National Laboratory

Backup slides

Fractured cross-section of IG-110U



fractured cross-section

- Clearly observed significant amount of frozen salt in graphite, salt spread and tightly attached on various locations including fillers and binders