Oak Ridge National Laboratory

Tribological characterization of graphite in dry argon and molten salt environments

Tomas Grejtak, Jim Keiser, Jun Qu, Nidia Gallego

DOE ART GCR Review Meeting

Hybrid Meeting at INL

July 16–18, 2024

U.S. DEPARTMENT OF ENERGY

GAS-COOLED REACTOR

ADVANCED REACTOR TECHNOLOGIES PROGRAM

Initial tribological studies of graphite in dry argon and molten salt environments

Objectives:

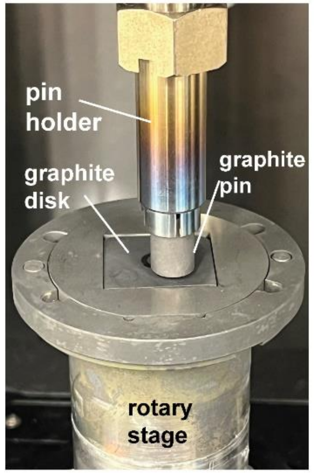
- Investigate the tribological (wear and friction) behavior of graphite pebbles in conditions similar to High Temperature Gas-cooled Reactor (HTGR) and Molten Salt Reactor (MSR) environments.

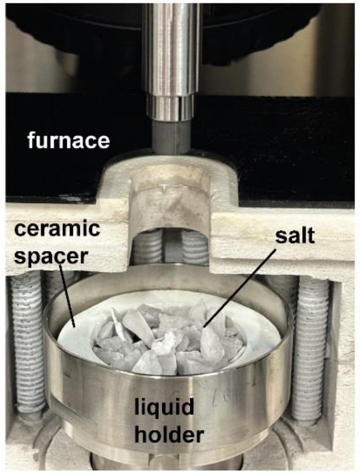
Approach:

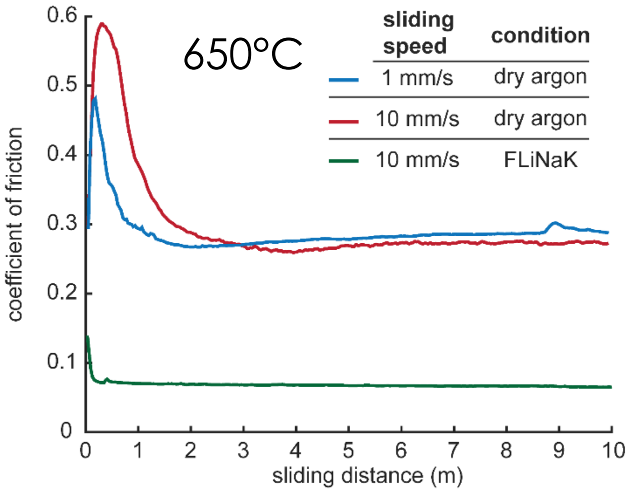
- Determine tribologically relevant conditions.
- Conduct wear and friction experiments on graphite in dry argon and molten FLiNaK salt environments.

Accomplishments:

- Determined sliding/rolling speeds and contact loads of pebbles in HTGR and MSR.
- Performed initial tribological testing of graphite in dry argan and molten FLiNaK salt environments.
- Completed Milestone Report: Initial tribological studies within molten salt environment.







ORNL/TM-2024/3253

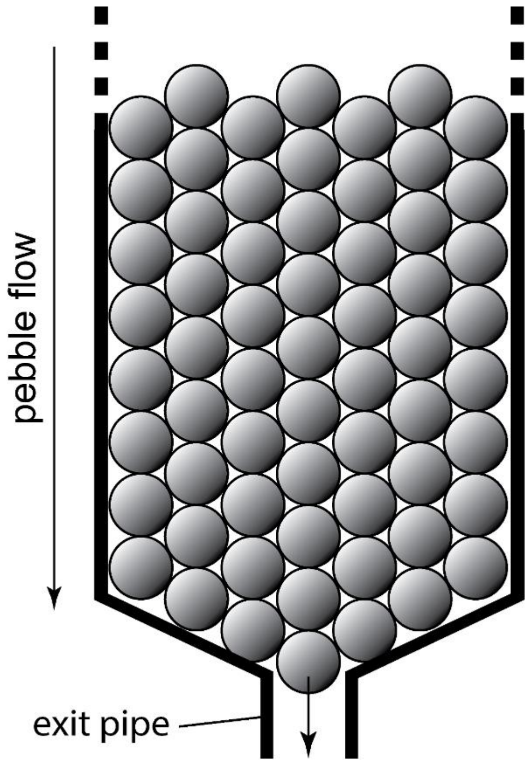
Report on Initial Tribological Studies of Graphite in Dry Argon and Molten Salt Environment

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

January 2024

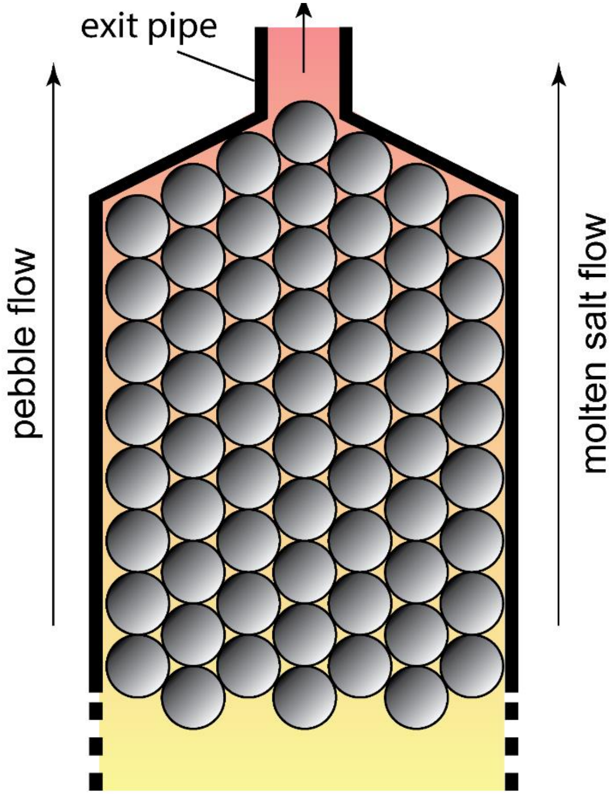
Pebble-bed high temperature reactor

Gas-cooled reactor



- Cooled with inert gas (He)
- Pebbles flow downwards.
- Operating temperatures 550 - 900°C.

Molten salt reactor



- Molten salt used as the coolant.
- Pebbles flow upwards.
- Ar gas environment.
- Operating temperatures 550 - 650°C.

Molten salts

Fluoride salts: FLiNaK, FLiBe

Advantages:

- Allow lower operating pressures
- Low neutron absorption
- Allow higher burnup rates
- High thermal conductivity
- Chemical stability
- Serve as a lubricant

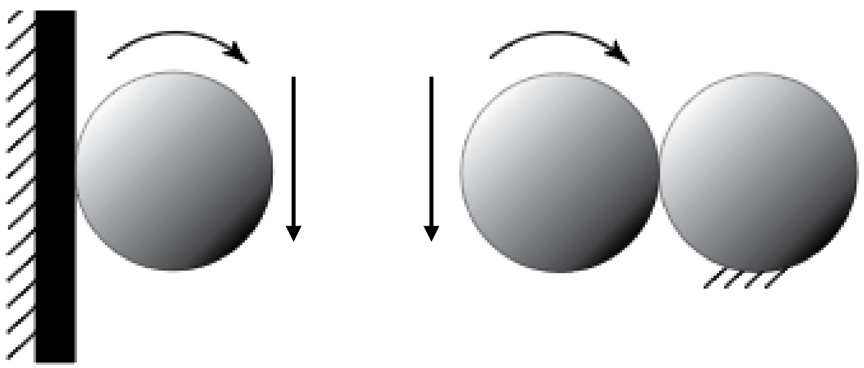
Disadvantages:

- Hazardous substance.
- Causes degradation and corrosion of graphite and other structural materials.

Why tribological characterization?

Graphite wear

Sliding & Rolling



Pebbles slide and roll against other pebbles and the graphite wall during circulation in the reactor.

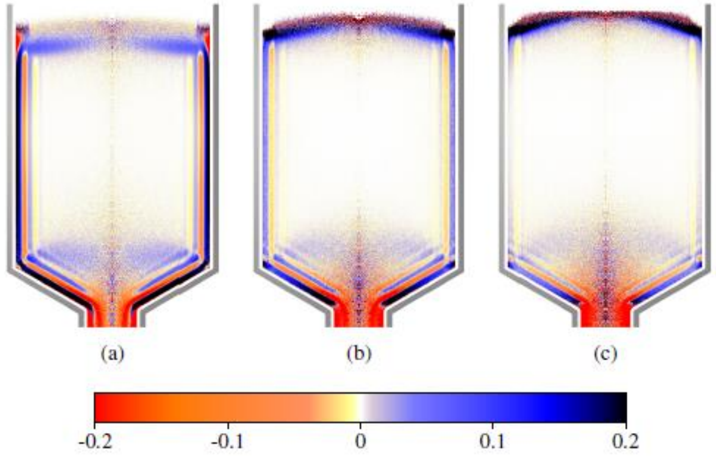
↓

abrasive wear, surface damage, generation of dust

Pebble flow

- Friction of pebbles and graphite wall affects the pebble movement in the reactor.
- The tribological behavior of pebbles is a critical parameter related to the motion of the pebbles, which affects their trajectory and accumulation.
- Understanding pebble flow dynamics is essential for reactor core design, pebble drainage cycle, and safety assessment.

pebble friction: μ=0.01 μ=0.125 μ=0.5



averaged pebble angular velocity

Y. Tang, L. Zhang, Q. Guo, B. Xia, Z. Yin, J. Cao, J. Tong, C. H. Rycroft, Analysis of the pebble burnup profile in a pebble-bed nuclear reactor. *Nucl. Eng. Des.* **345**, 233–251 (2019).

Graphite-on-graphite in dry argon environment

Contents lists available at ScienceDirect

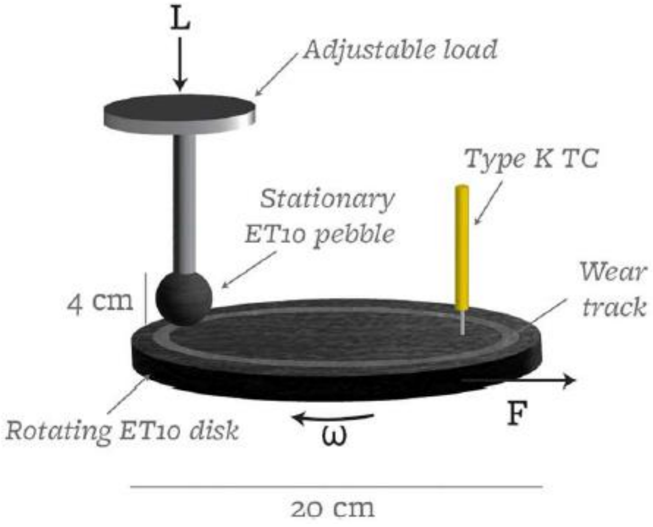
Tribology International

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

Self-lubrication of nuclear graphite in argon at high temperature

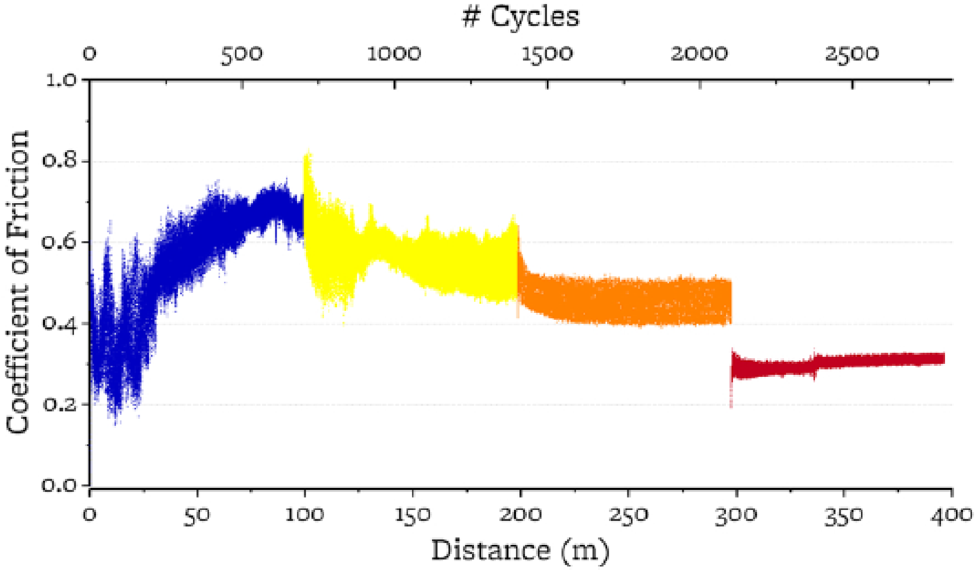
L. Vergari[a], J. Quincey[b], G. Meric de Bellefon[b], T. Merriman[c], M. Hackett[b], R.O. Scarlat[a,*]

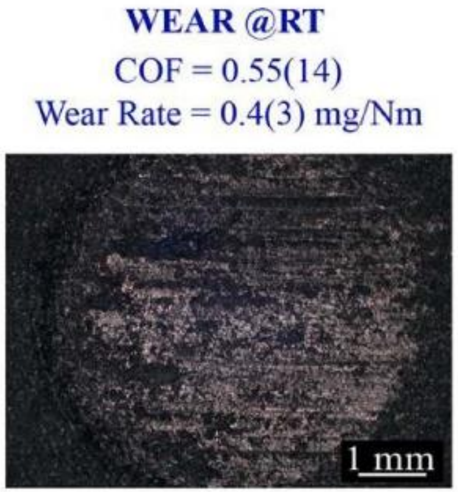
[a] Nuclear Engineering Department, University of California Berkeley, Berkeley, CA, USA
[b] Kairos Power LLC, Alameda, CA, USA
[c] Tribology Associates, Columbus, OH, USA

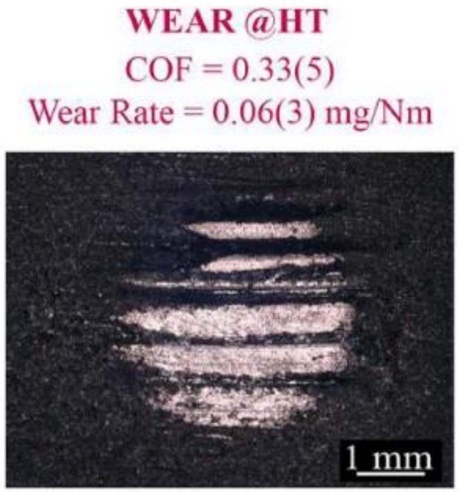


- Temperature = 20 - 600 °C
- Normal force = 50N
- Sliding speed = 0.15 m/s
- Sliding distance = 150 m
- Environment: argon

T	20 °C	200 °C	400 °C	600 °C
Avg. COF:	0.55±0.13	0.57±0.05	0.46±0.03	0.30±0.01







Graphite on 316H SS in molten FLiNaK salt.

ELSEVIER

Wear

Volume 522, 1 June 2023, 204706

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

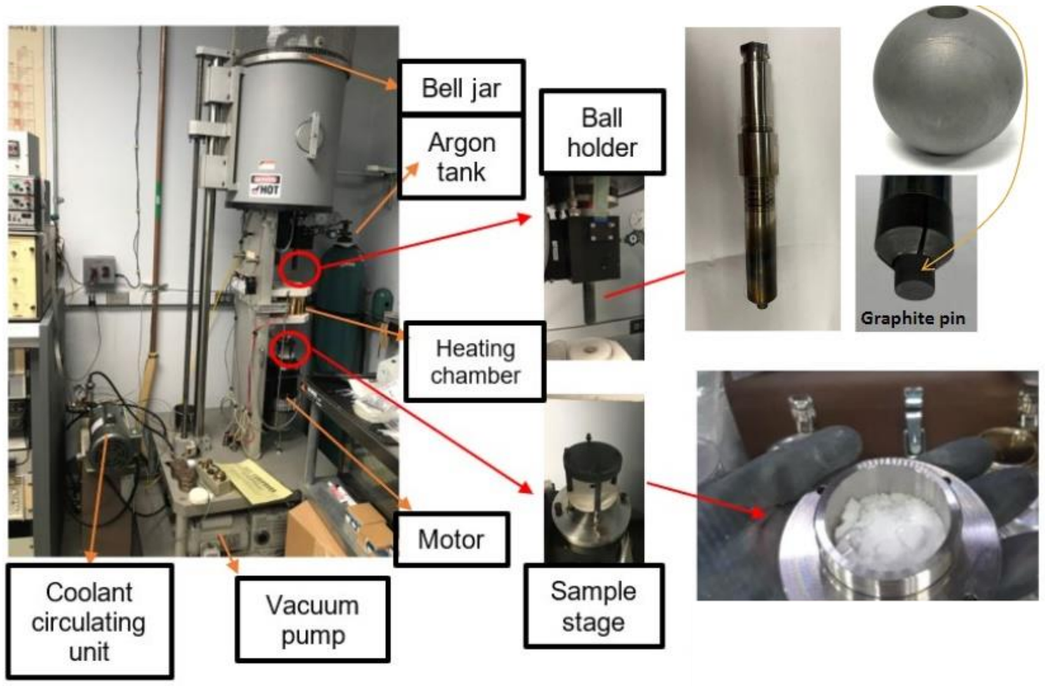
Xin He [a], Chanaka Kumara [a], Dino Sulejmanovic [a], James R. Keiser [a], Nidia Gallego [b], Jun Qu [a]

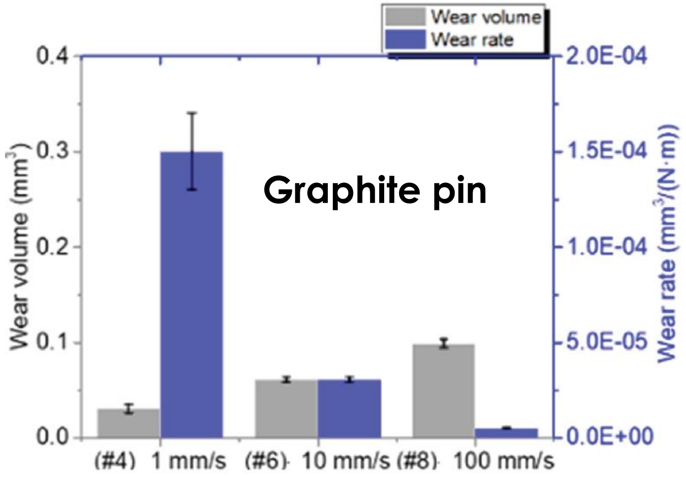
Temperature = 550 and 600 °C

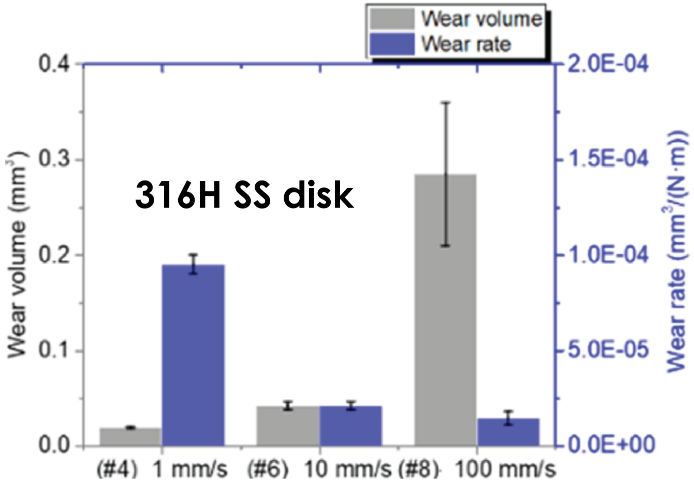
Normal force = 20N

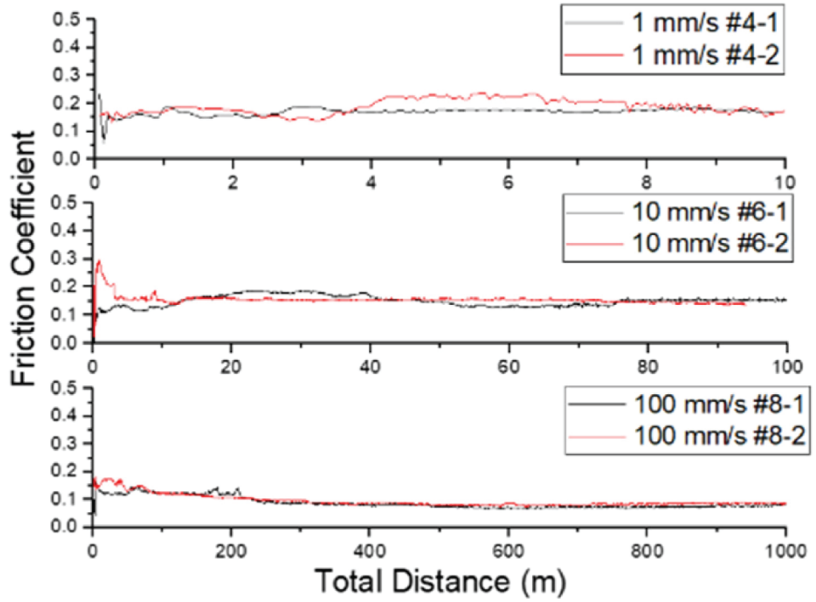
Sliding speed = 1, 10 and 100 mm/s

Environment: molten FLiNaK salt/argon









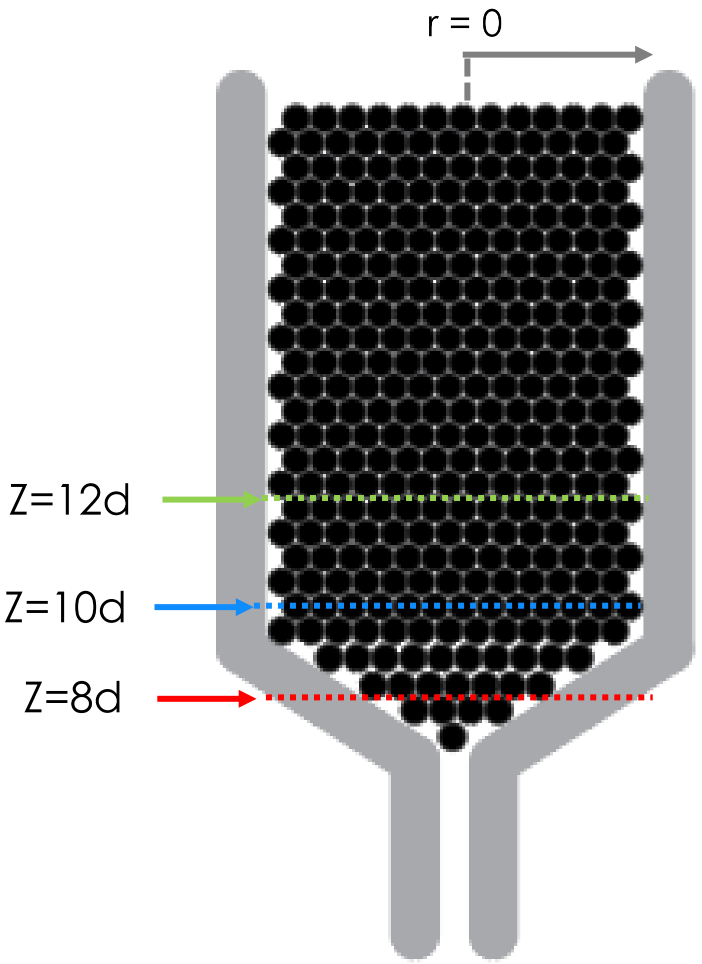
OAK RIDGE National Laboratory

Determination of tribologically relevant conditions

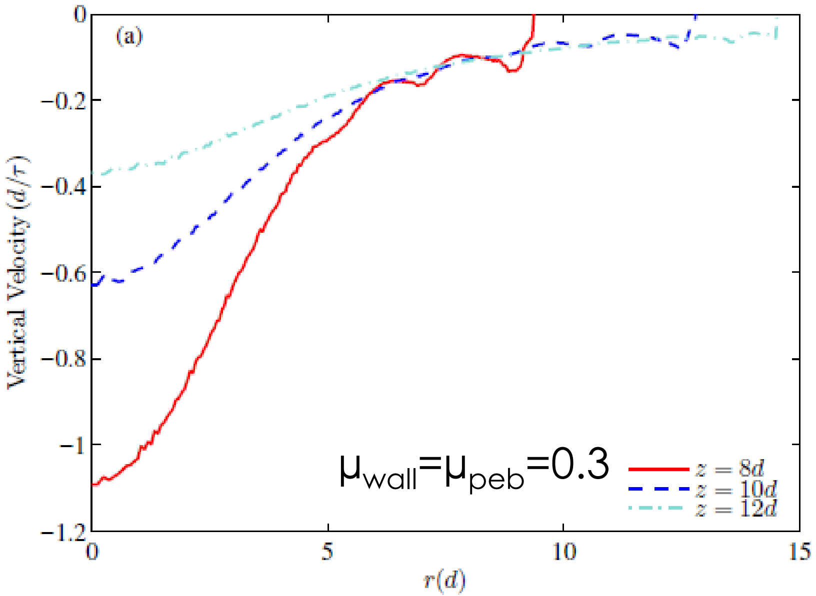
Unknown conditions/parameters:

- How do pebbles move and interact with each other in HTGR and MSR?
- Is sliding or rolling the dominant contact mode?
- What are the sliding and rolling speeds?
- What is the pebble-on-pebble and pebble-on-wall pressure/load?

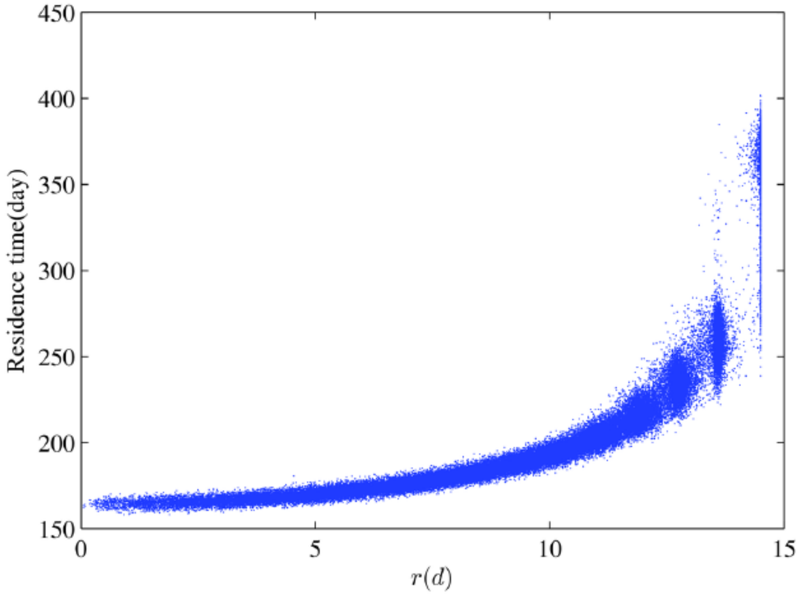
Determination of sliding speed



Pebble vertical velocity



Pebble residence time



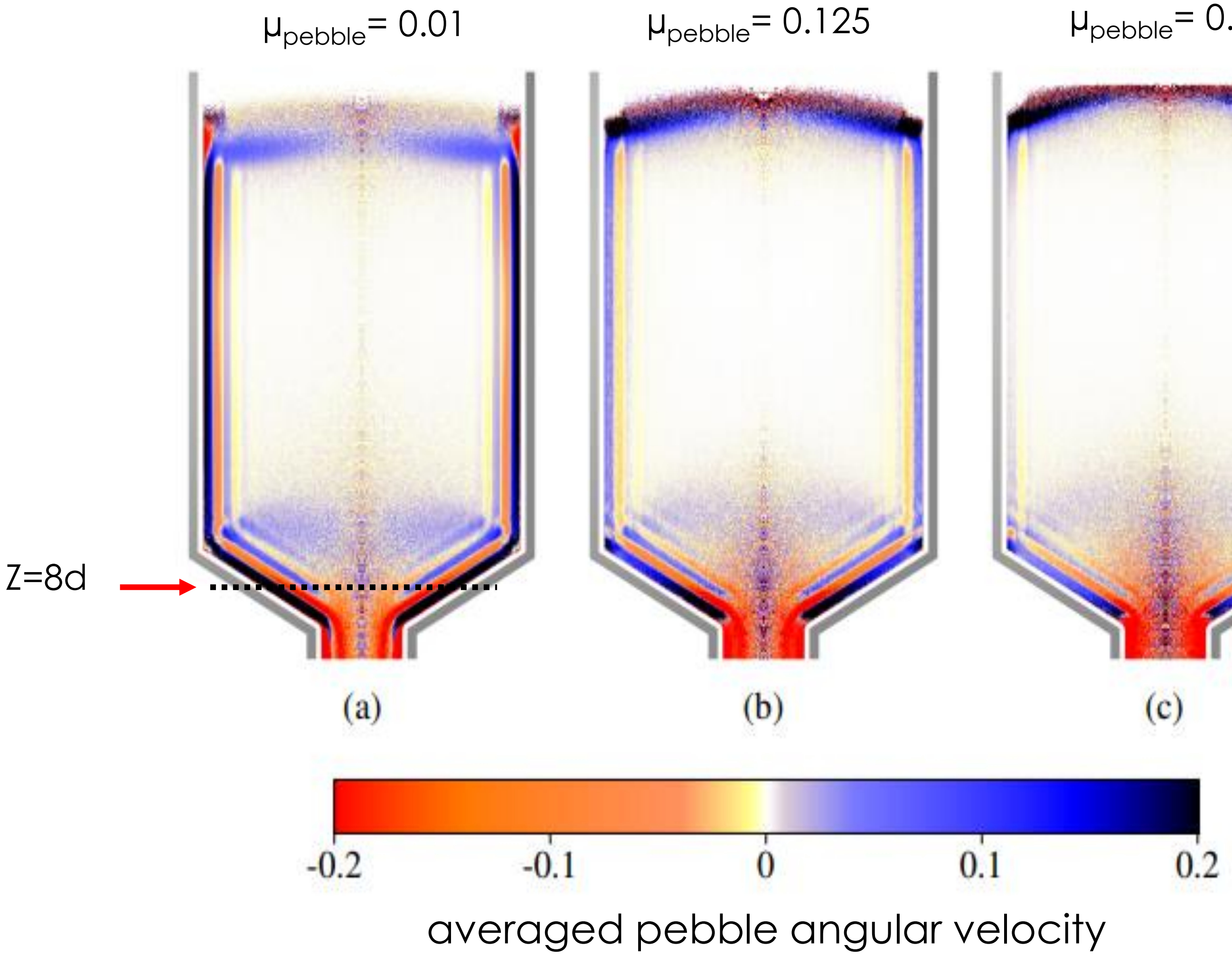
- Pebble sliding speeds are the highest at the center (r(d)=0) and bottom (z=8d) of the reactor core.
- Pebble residence time in the core is the lowest at the center of the reactor core.

Sliding speeds = 0.1 - 1 μm/s

Y. Tang, L. Zhang, Q. Guo, B. Xia, Z. Yin, J. Cao, J. Tong, C. H. Rycroft, Analysis of the pebble burnup profile in a pebble-bed nuclear reactor. *Nucl. Eng. Des.* **345**, 233–251 (2019).

Determination of rolling motion

Angular velocity



- Pebble angular velocities are the highest at the reactor's wall (for low μ values) and on the top and bottom of the reactor.
- Angular velocities decrease with increasing friction coefficient.

Rolling speeds = 0.05 – 0.1 μm/s

Y. Tang, L. Zhang, Q. Guo, B. Xia, Z. Yin, J. Cao, J. Tong, C. H. Rycroft, Analysis of the pebble burnup profile in a pebble-bed nuclear reactor. *Nucl. Eng. Des.* **345**, 233–251 (2019).

OAK RIDGE National Laboratory

Determination of contact force

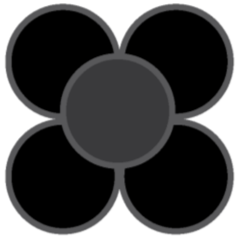
Inputs from:

Y. Tang, et al., Analysis of the pebble burnup profile in a pebble-bed nuclear reactor. *Nucl. Eng. Des.* **345**, 233–251 (2019).

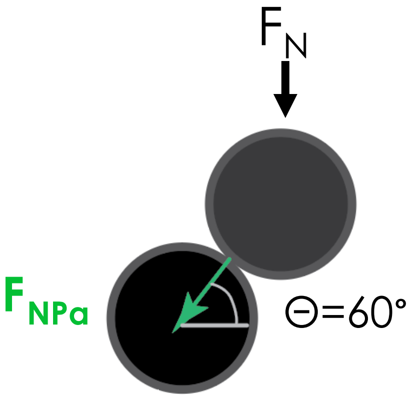
# of pebbles (N_c) = 30,000
Pebble diameter = 60 mm
Pebble mass = 210 g

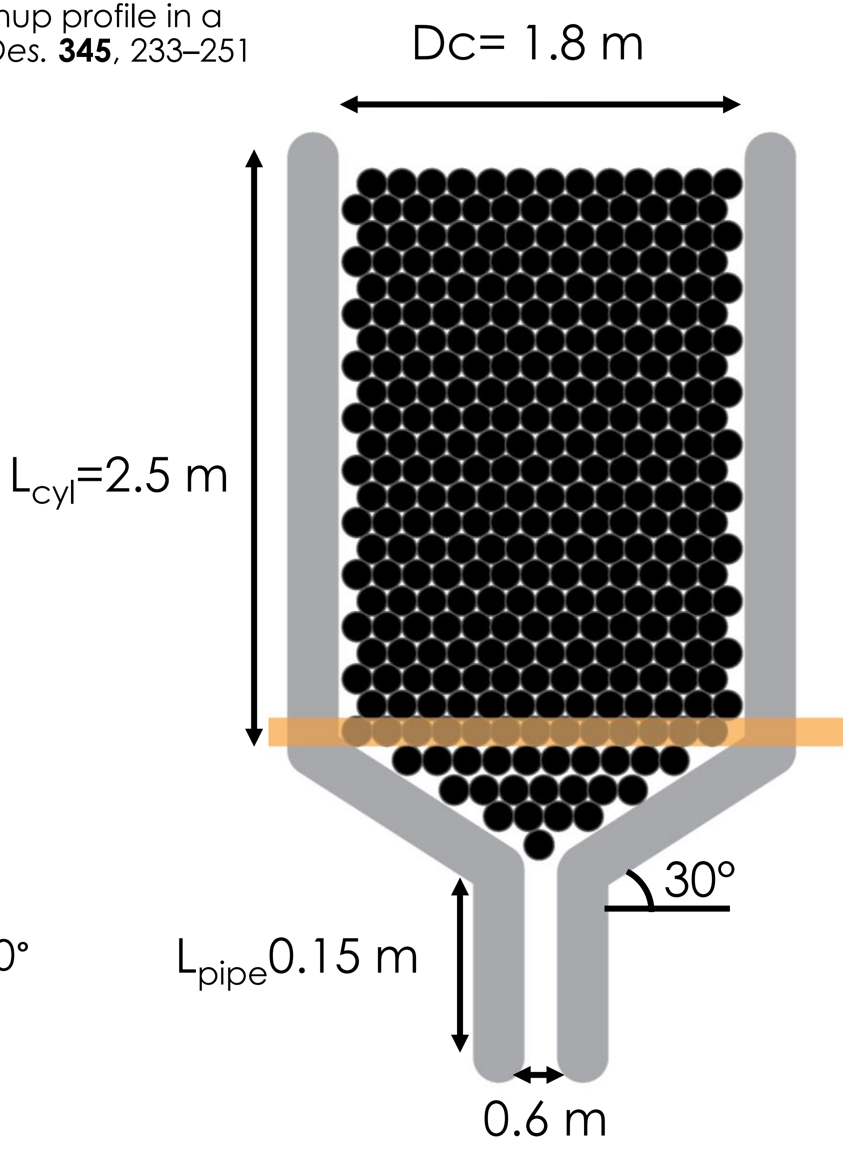
Pebble configuration

Top view



Side view





cross-sec. area of a pebble

A_v=0.0028 m^2

cross-sec. area of the core

A_c = 2.5 m^2

# of pebbles in the cross-section

$$N_{cross-sec} = \frac{A_C}{A_v} \sim 900$$

total load of the pebbles in the reactor

$$F_{NC} = N_c * mass_{peb} * 9.81 \sim 50\ kN$$

load on a pebble in the bottom of reactor

$$F_{NP} = \frac{F_{NC}}{N_{cross-sec}} \sim \mathbf{55\ N}$$

$\mathbf{F}_{NPa}$ = 1/4(F_{NP})/cos(Θ) ~ **28 N**

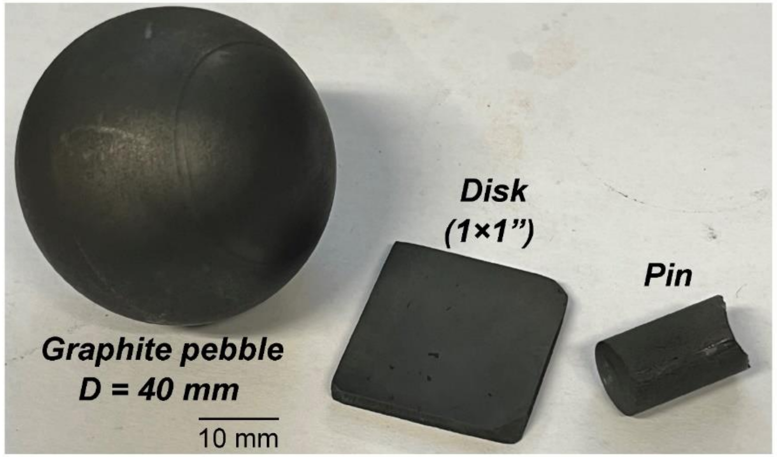
OAK RIDGE National Laboratory

Estimated conditions

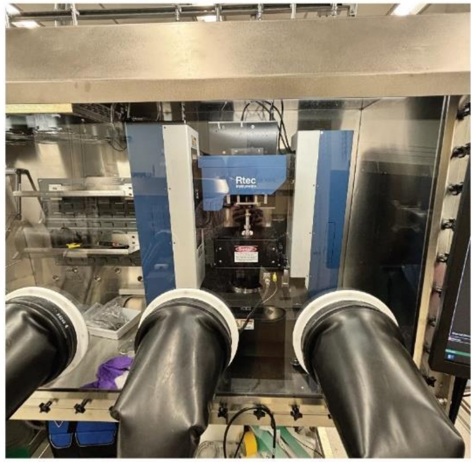
	Gas-cooled reactor (modeling data from Tang et al.)	Molten salt reactor (discussion with Kairos Power)
Contact load (N)	~28	<100
Sliding speed (mm/s)	$<1.2\times10^{-3}$	$\sim4.0\times10^{-4}$
Rolling speed (mm/s)	$<1.1\times10^{-4}$	N/A
Travel distance (m)	~10	~18
Temperature (°C)	550-900	550-650

Experimental setup

Samples (nuclear graphite ET-10)
Provided by Kairos Power



Tribometer in a glovebox



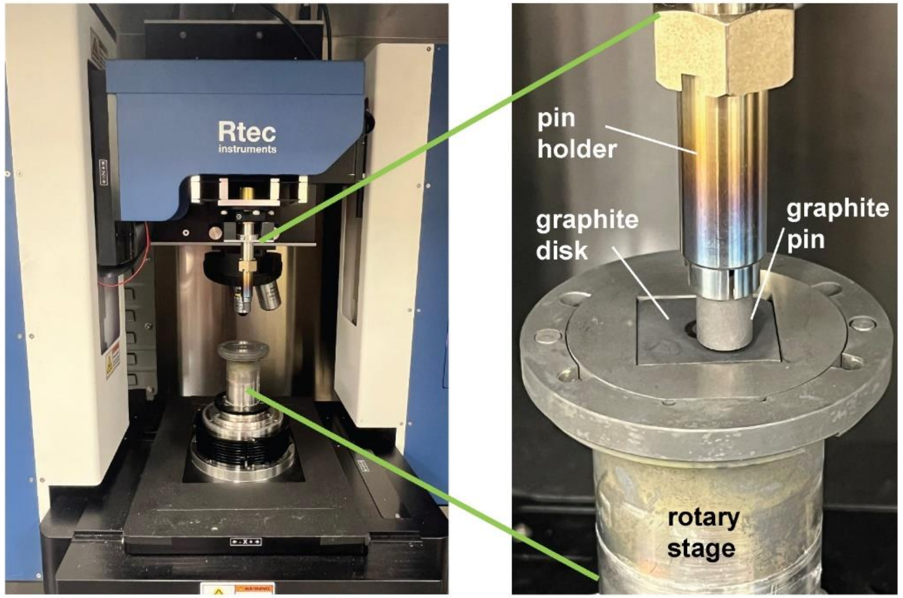
Test conditions

Gas: argon
Temperature: 650°C
Normal load: 20 N
Sliding speed: 1 & 10 mm/s
Sliding distance: 10 m

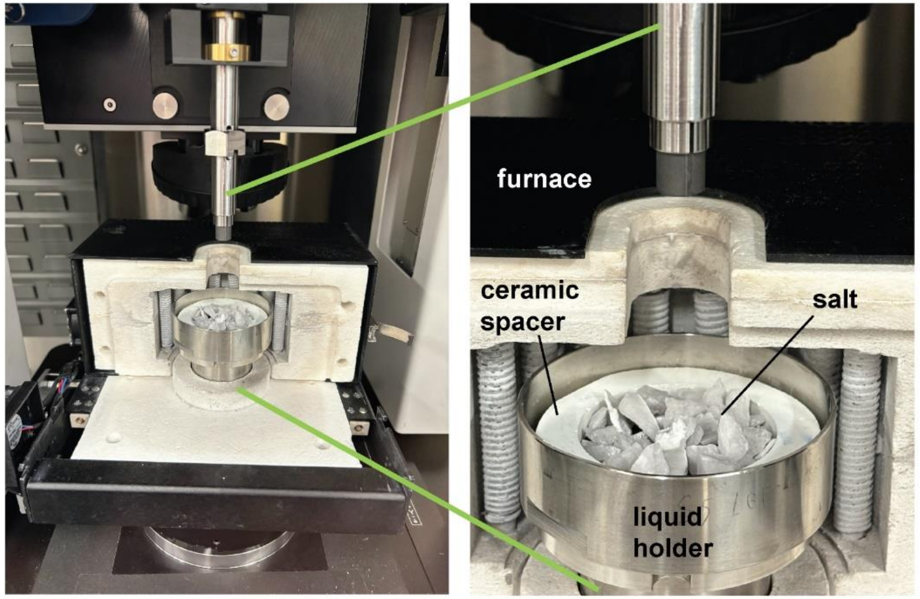
Molten salt

FLiNaK (mol %: 46.5% LiF, 11.5% NaF, 42% KF)
Melting point: 454 °C
Viscosity: 3.66 mPa·s @ 650 °C

Dry argon environment



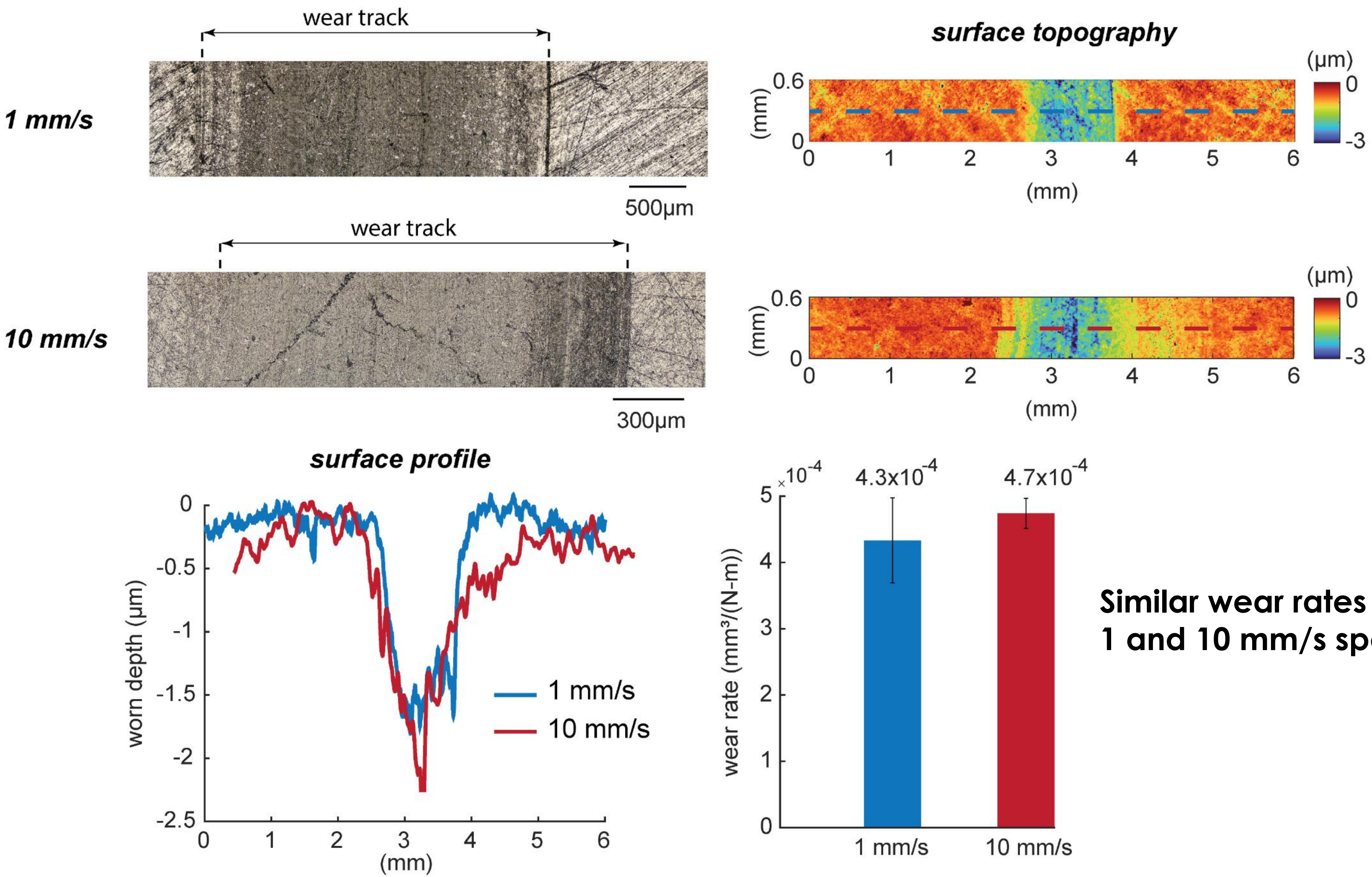
Molten (FLiNaK) salt argon environment



Furnace

Wear rate of graphite disk in dry argon

Environment: dry argon **Temperature**: 650°C **Normal load**: 20 N



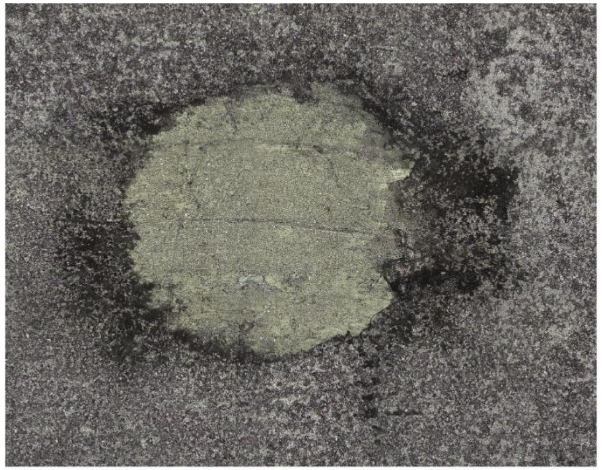
Similar wear rates at 1 and 10 mm/s speeds.

Wear rate of graphite pin in dry argon

Environment: dry argon **Temperature**: 650°C **Normal load**: 20 N

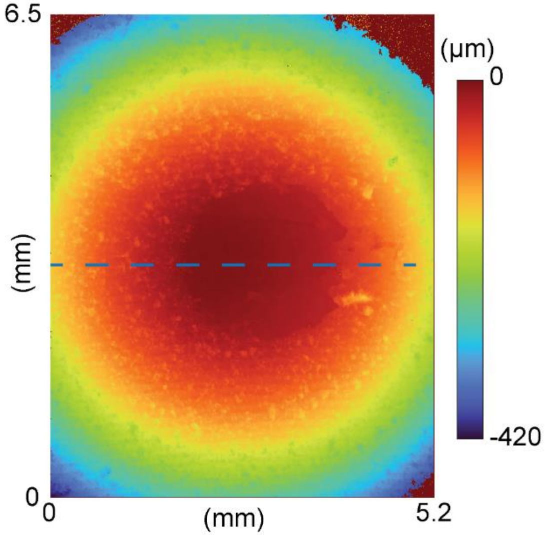
sliding direction

1 mm/s

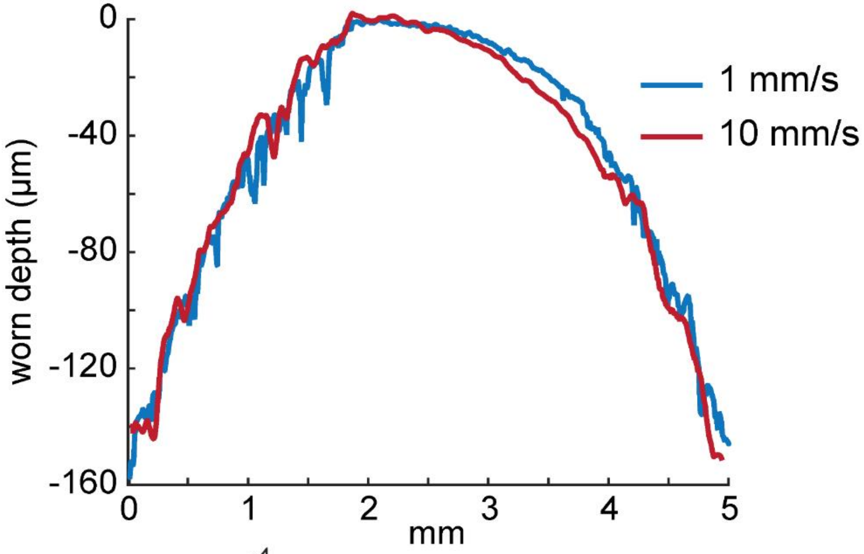


500μm

surface topography

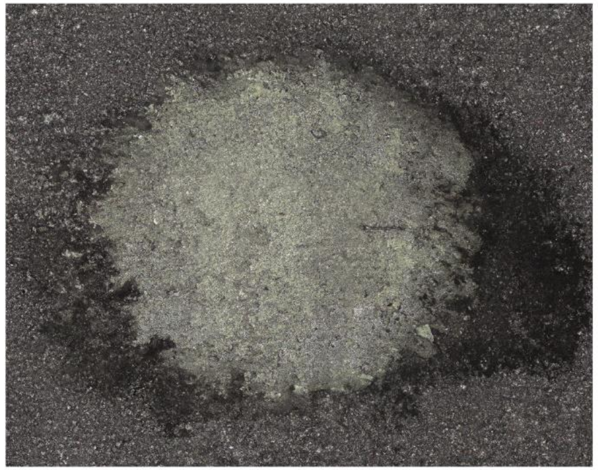


surface profile

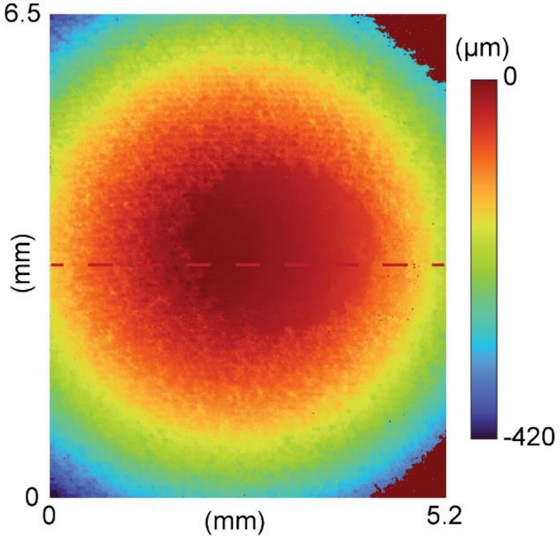


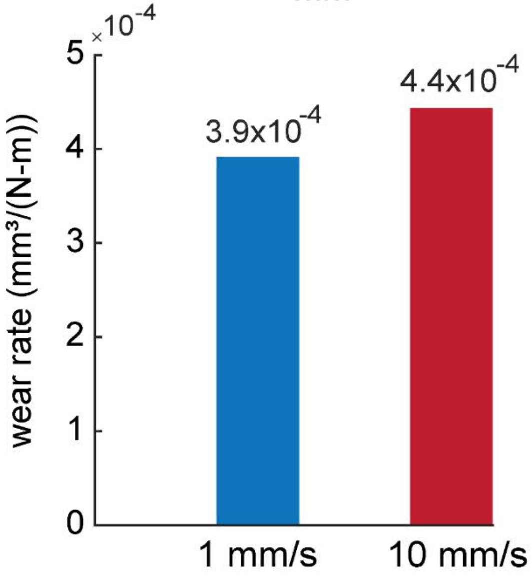
sliding direction

10 mm/s



400μm

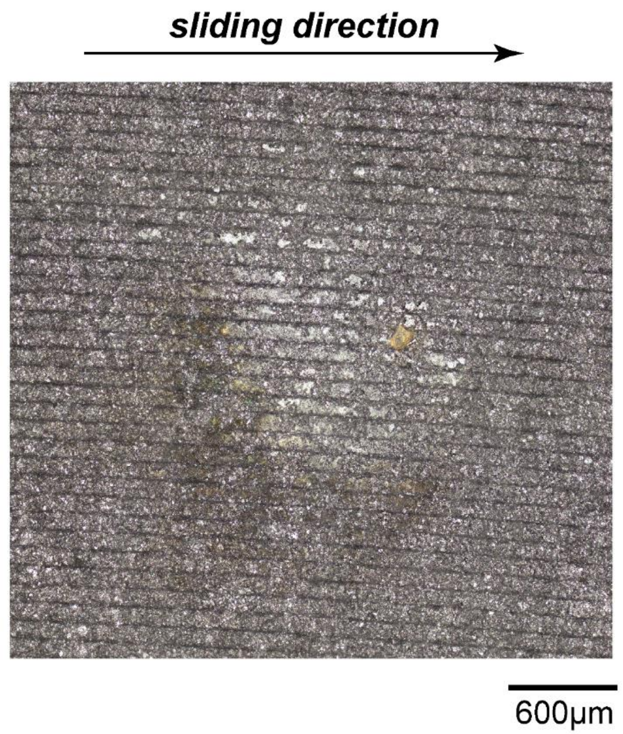


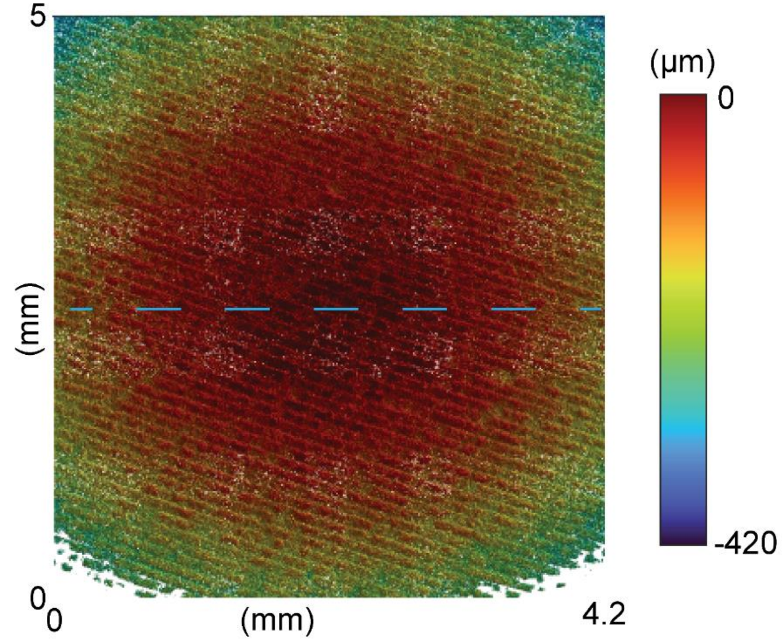


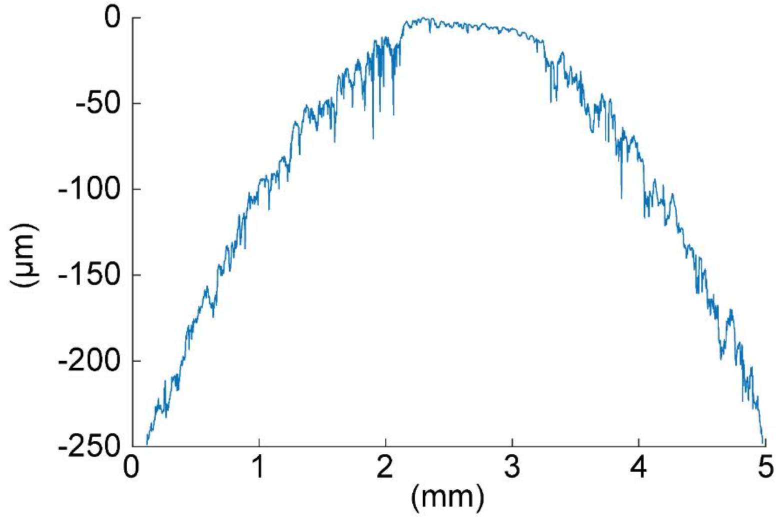
Wear rate of graphite pin & disk in molten FLiNaK salt

Environment: molten FLiNaK salt **Temperature**: 650°C **Normal load**: 20 N

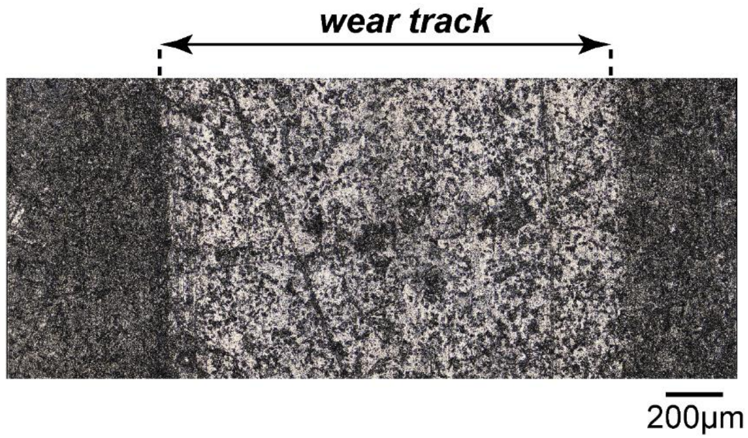
Pin

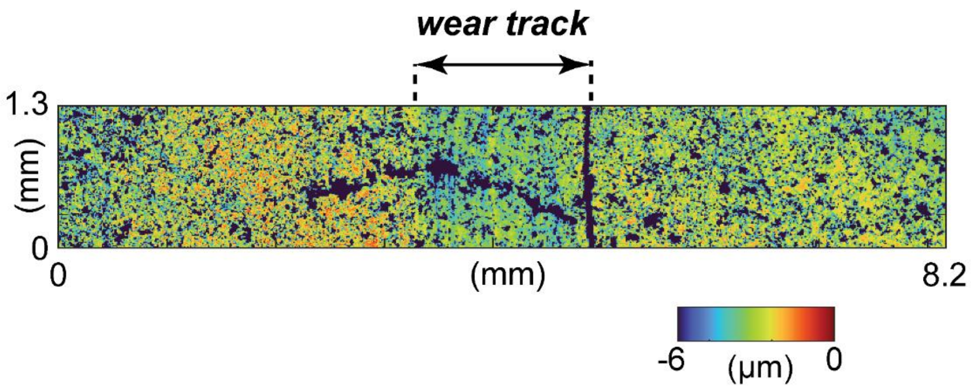


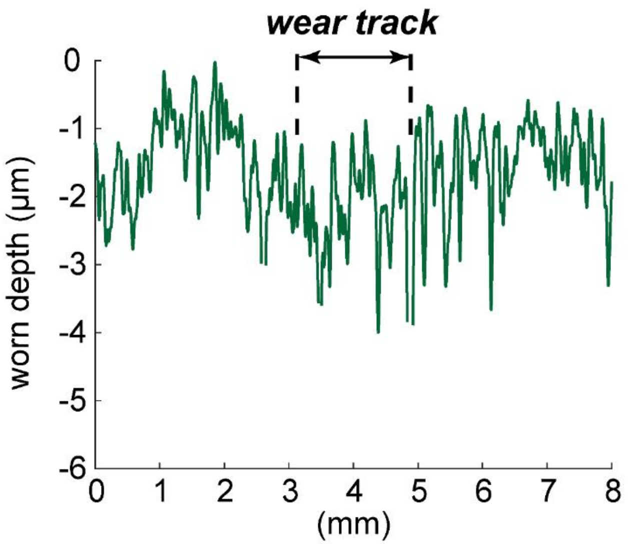




Disk

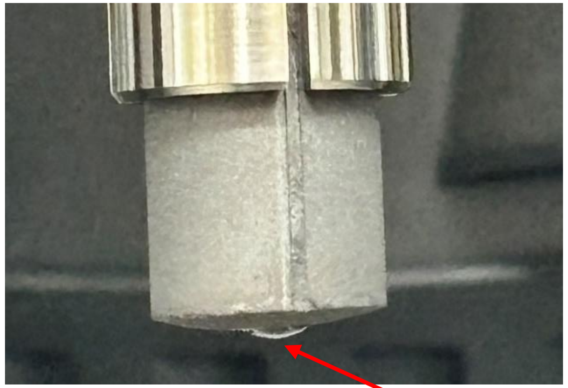


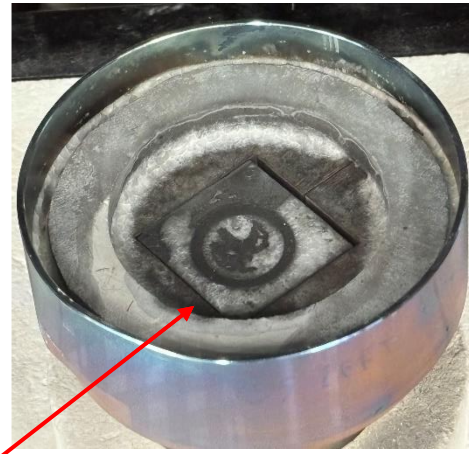




Challenges: removing salt from graphite surface

- Salt remains on the graphite surface as a residue.
- Salt needs to be dissolved by sonicating in DI water.
- Dissolving and sonicating salt from the graphite surface can prevent investigation of key morphological and chemical alterations of the sliding surface.

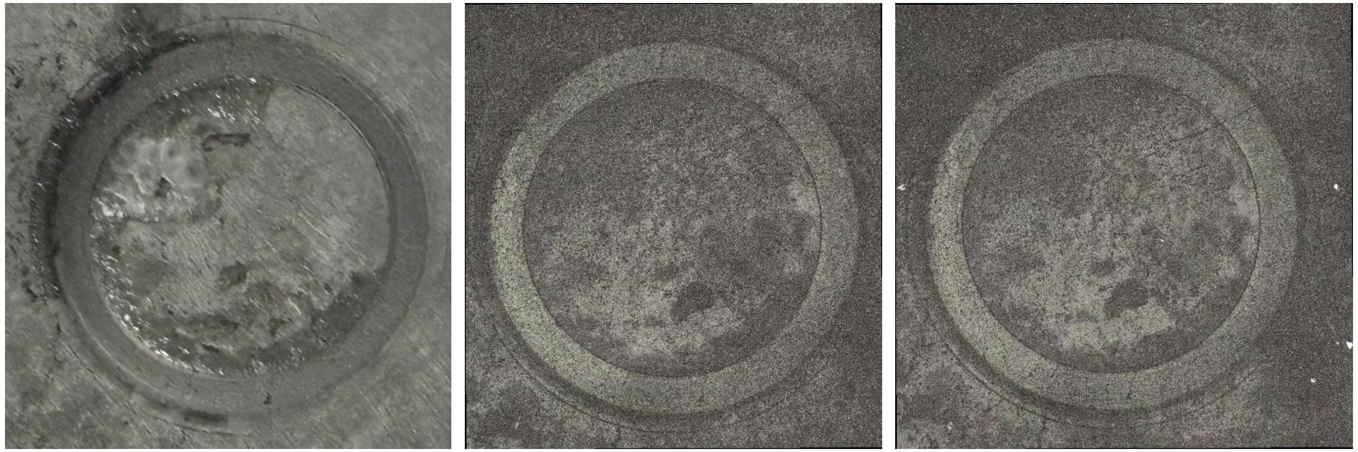


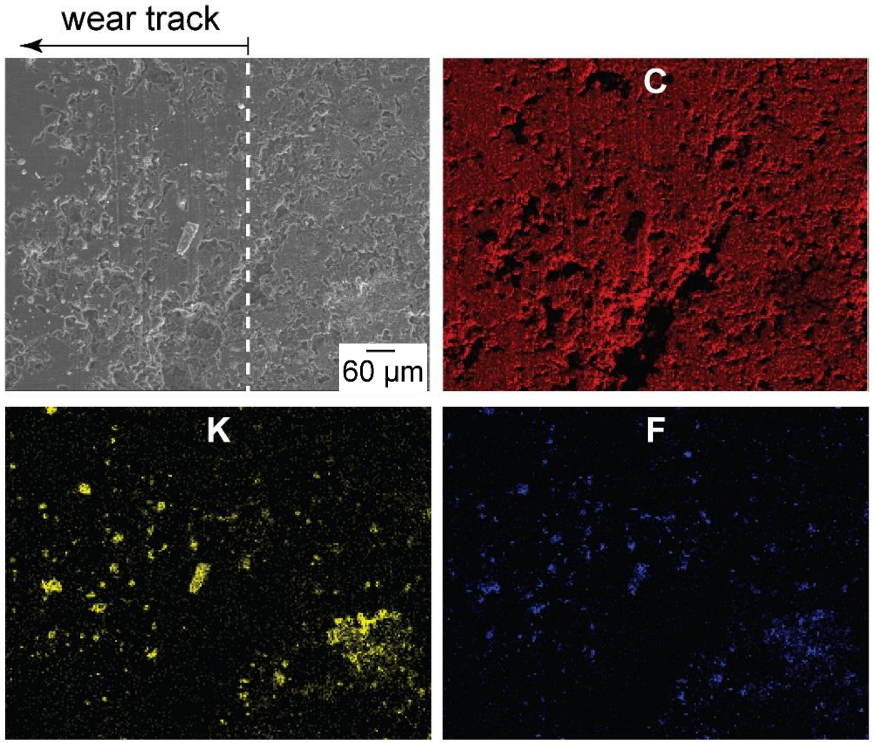


salt residue

Sonication time

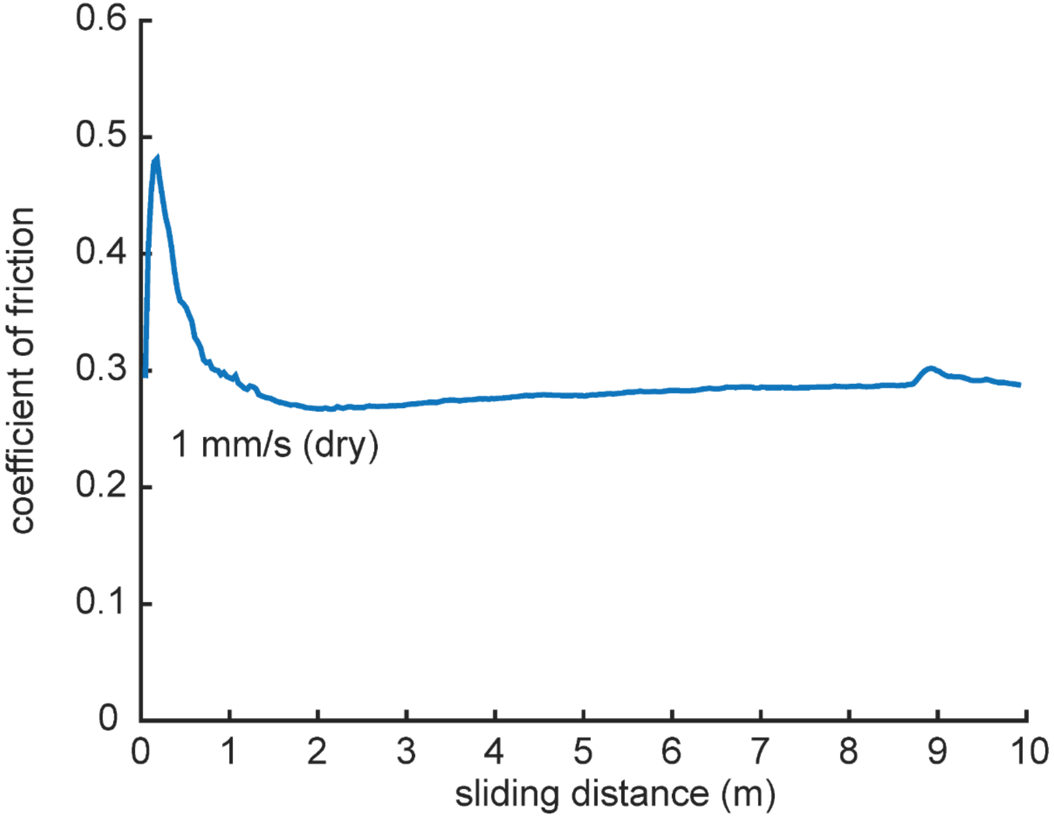
1 hour | 2 hours | 3 hours



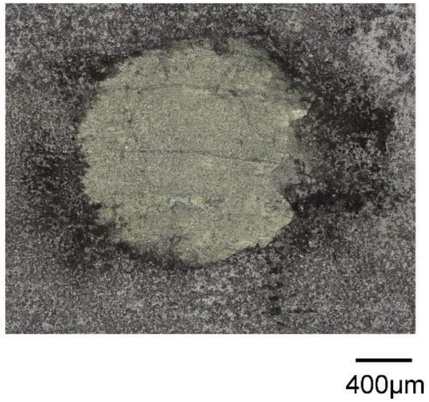


Salt residues remain on graphite surface after sonication!

Friction coefficient



1 mm/s (dry)

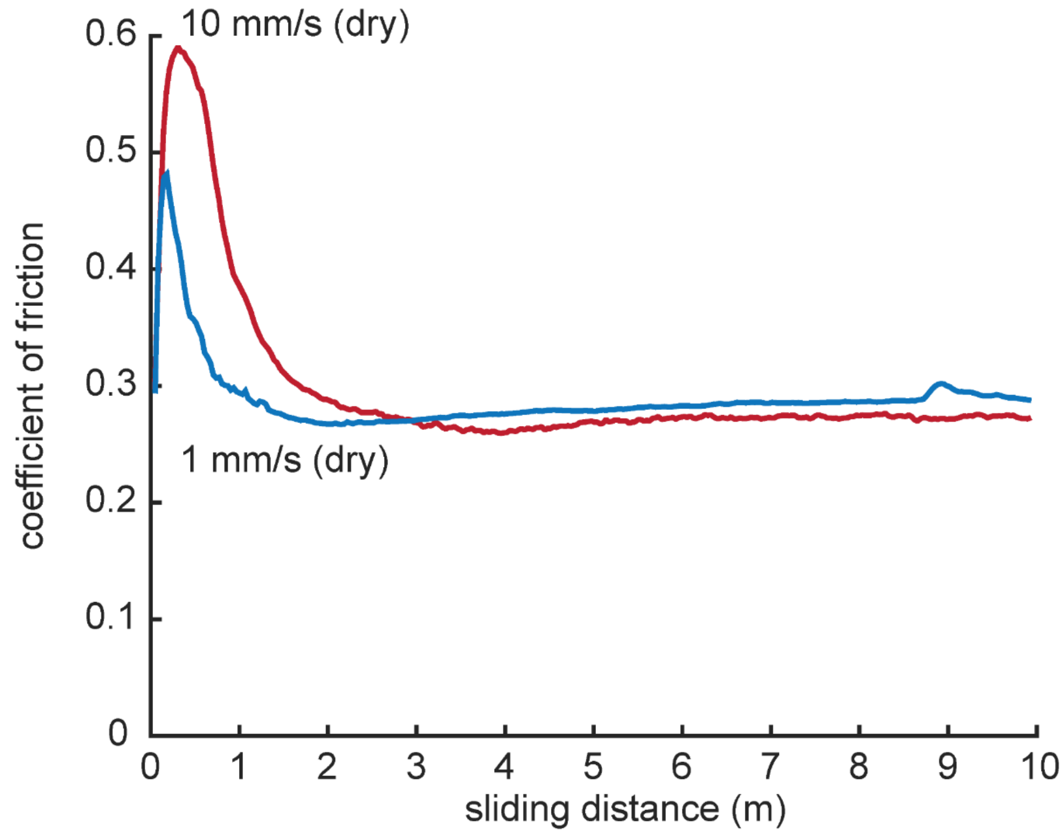


Temperature=650°C Load=20 N

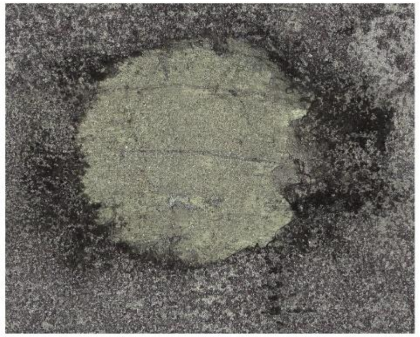
Lubrication condition	Sliding speed (mm/s)	Maximum COF	Steady state COF
Dry argon	1	**0.47**	**0.28**
Dry argon	10		
Molten FLiNaK Salt	10		

Oak Ridge National Laboratory

Friction coefficient

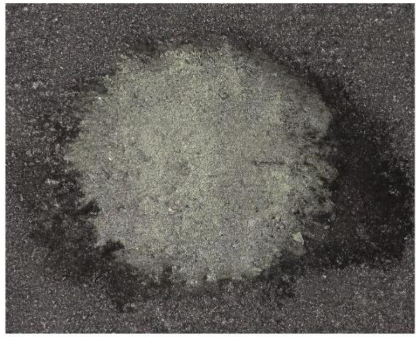


1 mm/s (dry)



400µm

10 mm/s (dry)



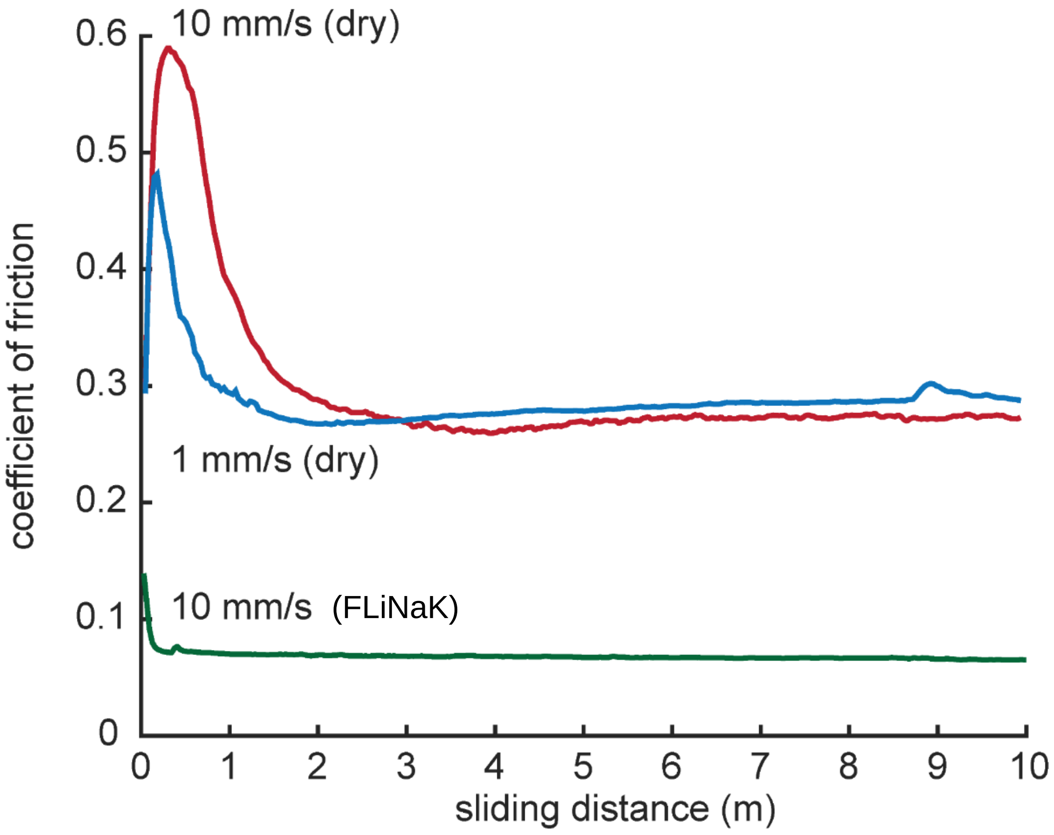
400µm

Temperature=650°C Load=20 N

Lubrication condition	Sliding speed (mm/s)	Maximum COF	Steady state COF
Dry argon	1	**0.47**	**0.28**
Dry argon	10	**0.58**	**0.27**
Molten FLiNaK Salt	10		

- Higher run-in friction at 10 mm/s.
- Similar steady-state friction.

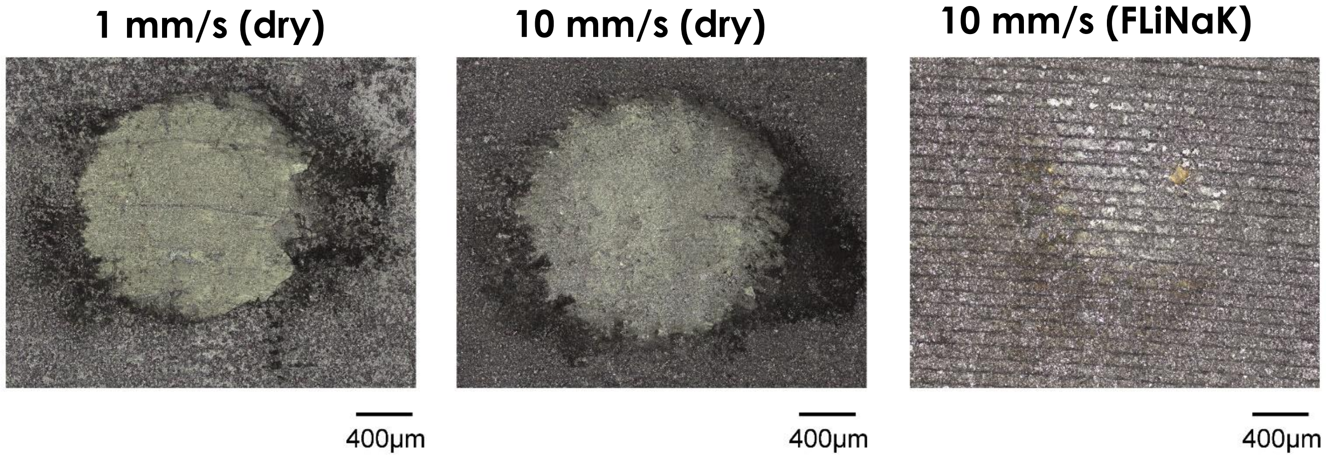
Friction coefficient



Temperature=650°C Load=20 N

Lubrication condition	Sliding speed (mm/s)	Maximum COF	Steady state COF
Dry argon	1	**0.47**	**0.28**
Dry argon	10	**0.58**	**0.27**
Molten FLiNaK Salt	10	**0.14**	**0.07**

- Higher run-in friction at 10 mm/s.
- Similar steady-state friction.
- **Molten salt significantly reduces friction.**



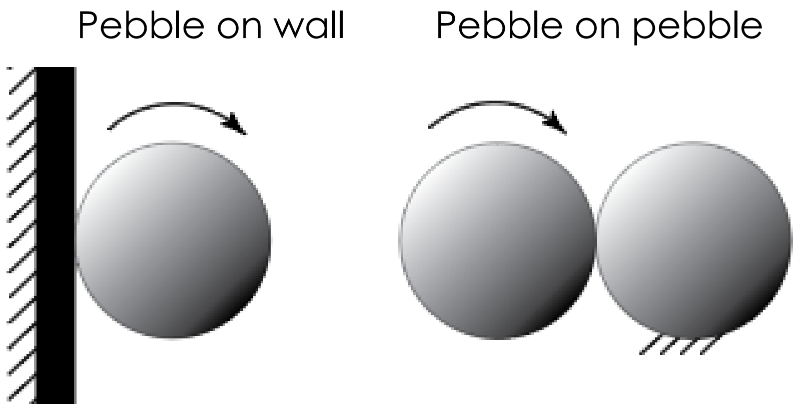
Test matrix for upcoming studies

Environment: dry argon & molten salt

Force (N)	**20**		**40**		**80**	
Sliding speed (mm/s)	1	10	1	10	1	10
Temperature (°C)	650	650	650	650	650	650
Sliding distance (m)	20	20	20	20	20	20

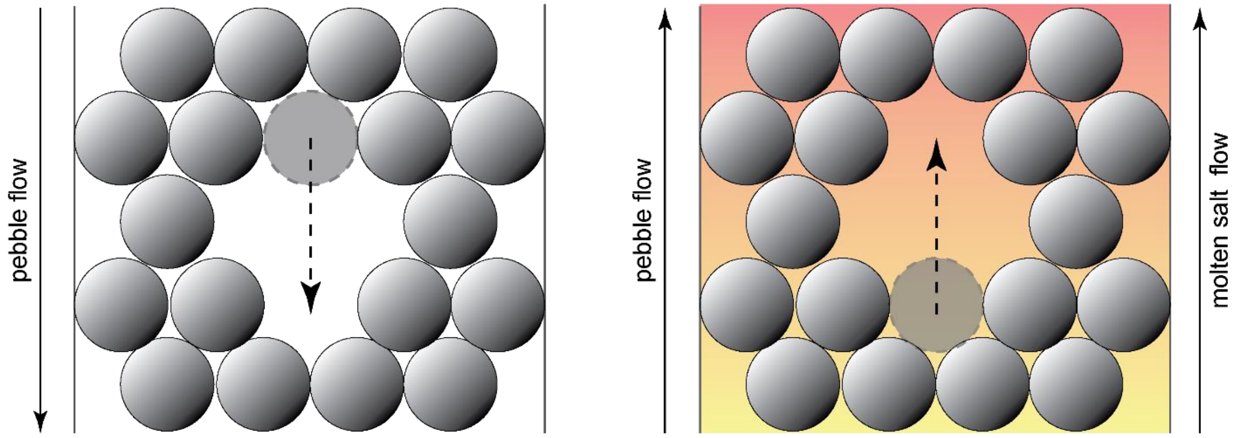
Topics of Interest (Potential Future Studies)

Rolling contact



- Pebbles roll against other pebbles and the graphite wall during circulation in the reactor.
- Designing rolling test at high temperatures could be challenging.

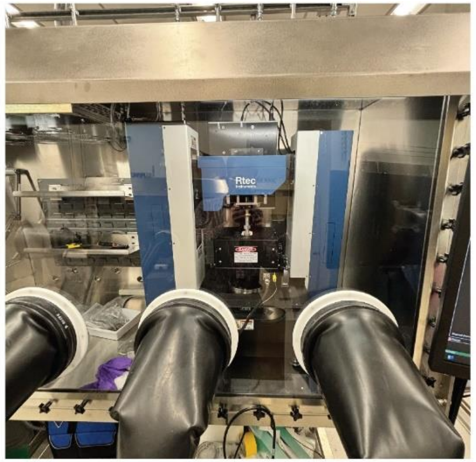
Impact contact

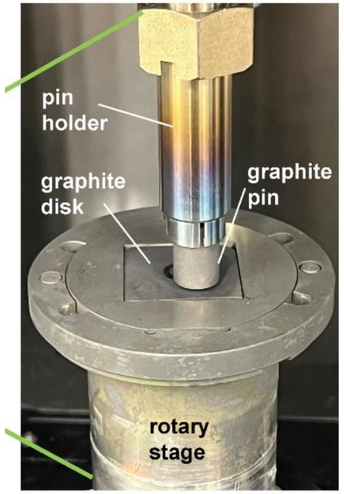


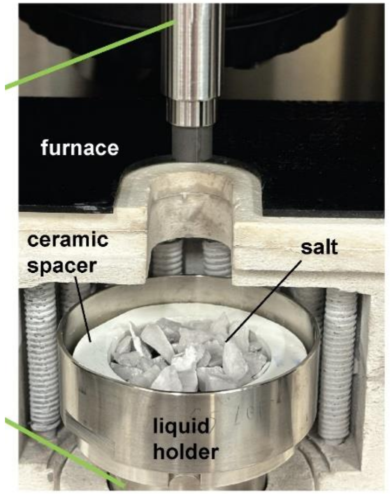
- Pebbles are not uniformly distributed and there could be voids in the pebble bed matrix.
- A pebble could move through the void and collide with an opposing pebble.
- This would cause an impact contact – higher contact loads and speeds.

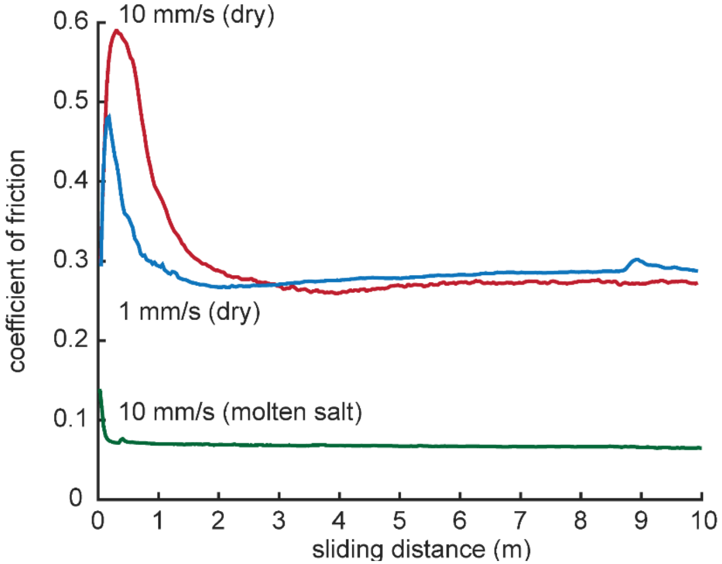
Conclusions and acknowledgements

- Developed capabilities to characterize the tribological properties of graphite in dry argon and molten salt environments at high temperatures.
- Determined the tribologically relevant conditions in gas-cooled and molten salt reactors.
- Identified challenges and limitations of tribological testing with molten salts.
- Performed initial studied tribological studies of graphite in dry argon and molten salt environment.









GAS-COOLED REACTOR
ADVANCED REACTOR TECHNOLOGIES PROGRAM

grejtakt@ornl.gov

Grejtak, T., Qu, J., Gallego, N.C., Keiser, J.R., Report on Initial Tribological Studies of Graphite in Dry Argon and Molten Salt Environment. ORNL/TM-20024/3253. Oak Ridge National Laboratory, 2024