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Friction and wear of nuclear graphite exposed to molten FLiNaK salt environment



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July 2025

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Materials Science and Technology Division

**FRICITION AND WEAR OF NUCLEAR GRAPHITE EXPOSED TO MOLTEN FLINAK
SALT ENVIRONMENT**

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ABBREVIATIONS

CoF	coefficient of friction
FLiBe	LiF–BeF ₂
FLiNaK	LiF–NaF–KF
ORNL	Oak Ridge National Laboratory
PB-FHR	pebble-bed fluoride salt-cooled high-temperature reactor

ABSTRACT

This report formally documents the completion of the Advanced Reactor Technologies Level 3 Milestone (M3TG-25OR0501104), “Initiate graphite wear studies of graphite samples exposed to molten salt environment,” due August 1, 2025. The report summarizes the ongoing activities aimed at characterizing the friction and wear behavior of graphite in molten LiF–NaF–KF (FLiNaK) salt environment. The wear and friction behavior of self-mated ET-10 nuclear graphite were tested at varying temperatures (550°C and 650°C), contact loads (40 and 80 N) and sliding speeds (1 and 10 mm/s) in a controlled argon environment. The results were compared to the wear and friction behavior of the same graphite material in a dry argon environment. This report presents initial studies on the friction and wear behavior of graphite in sliding contact mode, with future work focused on investigating the graphite matrix materials, as well as rolling and impact contact modes. The outcomes of this report could provide insights into assessing the integrity of the graphitic components of pebble-bed fluoride salt-cooled high-temperature reactors (PB-FHRs) to enhance their safe operation.

1. INTRODUCTION

A PB-FHR contains thousands of spherical fuel-carrying elements known as pebbles, which continuously circulate through the reactor core several times before their final discharge (1, 2). Pebbles are typically 4–6 cm in diameter, consisting of tri-structural isotropic (TRISO) fuel particles that are dispersed in a graphite matrix (2, 3). The fuel particles are surrounded by a thin fuel-free outer matrix shell. The inlet and outlet temperature in PB-FHRs are 550°C and 650°C, respectively. Molten fluoride salts serve as the coolant, circulating from the bottom to the top of the reactor. Graphitic pebbles are buoyant because they are less dense than fluoride salts and flow in the direction of the circulating salt coolant (1).

As pebbles circulate in the reactor, they experience repeated contact interactions with the neighboring pebbles, the graphite wall, and the metallic components of the containment assembly. These interactions can result in various types of dynamic contact modes, including sliding, rolling, and impact. Repeated interactions can cause abrasion and microfracture of both the pebbles and the graphite wall components, leading to their structural degradation. Additionally, excessive wear can generate dust particles, which can pose a safety hazard and negatively affect the reactor operation (4, 5). Furthermore, friction between the pebbles and against the reactor wall significantly influences their movement, making it an important factor in the core design. As a result, it is essential to understand the tribological behavior of the pebbles to assess their integrity and service life and to ensure the safe operation of the reactor.

Several studies have attempted to characterize the friction and wear behavior of graphite in a fluoride salt environment. All reviewed studies used ET-10 nuclear graphite and argon as an inert gas. In the past, Oak Ridge National Laboratory (ORNL) investigated tribological behavior of graphite sliding against 316H stainless steel lubricated by a molten FLiNaK salt at 550°C and 650°C (6). The study showed that increased temperature led to higher wear loss due to the reduced viscosity of the molten salt and an elevated corrosion rate. Compared to stainless steel, graphite experienced higher wear loss at higher sliding speeds because it is more susceptible to microfractures triggered by vibration. ORNL also performed initial comparative tribological studies of self-mated (both contacting surfaces are the same material) ET-10 nuclear graphite in both molten FLiNaK salt and dry argon environments at 650°C (7). The study suggested that the molten FLiNaK salt provides effective boundary lubrication, which notably reduced friction compared to the dry argon conditions. The steady-state coefficient of friction (CoF) of graphite in molten FLiNaK salt was approximately 0.07, which is about four times less than the 0.28 observed in dry argon conditions. Vergari et al. (8) investigated tribological behavior of self-mated ET-10 nuclear graphite in a molten LiF–BeF₂ (FLiBe) salt at temperatures between 500°C and 600°C. The steady-state CoF was around 0.2 and was independent of temperature. Vergari et al. (9) also investigated

tribological behavior of self-mated ET-10 nuclear graphite in dry argon conditions and found that friction decreases with increasing temperature from room temperature to 600°C with an average CoF of 0.33 at high temperatures. This trend was attributed to the formation of a thicker and more stable tribofilm due to higher strength of graphite at higher temperatures. In this report, the friction and wear behavior of self-mated ET-10 nuclear graphite in molten FLiNaK salt is characterized as a function of temperature, contact load, and sliding speed. The findings are benchmarked against the results obtained under dry argon conditions.

2. FRICTION AND WEAR EXPERIMENTS

2.1 GRAPHITE AND SALT MATERIALS

ET-10 nuclear graphite material provided by Kairos Power was used to machine samples for tribological testing. Two specimen geometries were prepared: a flat measuring $25.4 \times 25.4 \times 1.6$ mm, and a pin with a cylindrical shaft of 9.42 mm diameter. The pins were machined from surrogate pebbles with a size of 40 mm using wire electrical discharge machining. The roughness, R_a , of the as-received contact surface of pins was 4.3 μm . The flats were fabricated from a rectangular block using a lathe with a final contact surface roughness of 3.4 μm . Before the tribological testing, all samples were sonicated in ethanol for 30 min, followed by drying at about 120°C for 4 h. The samples were then baked at 1,200°C for 8 h under vacuum ($P < 10^{-5}$ Torr) to remove residual moisture and oxygen. The hardness of the graphite samples was measured via Rockwell L microindentation according to the ASTM C748-20 procedure, yielding a hardness value of 94.5 HRL. Fluoride molten FLiNaK (LiF:NaF:KF;46.5:11.5:42 mol %) salt used in this work was purchased from Materion Advanced Chemicals Inc.

2.2 TRIBOLOGICAL TESTING

The tribological pin-on-disk experiments were performed using a multifunctional tribometer (MFT-2000A, Rtec Instruments, San Jose, California) as illustrated in Figure 1. The tribometer was placed in a glovebox with a controlled argon atmosphere, Figure 1b. The test conditions are summarized in Table 1. The friction and wear characterization were carried out at temperatures of 550°C and 650°C, contact loads of 40 and 80 N, and sliding speeds of 1 and 10 mm/s. The duration of the tests performed at a sliding speed of 10 mm/s was 33 min, and the 1 mm/s tests lasted 330 min. The sliding distance was 20 m in all tests. The test conditions were selected based on inputs from Kairos Power. Each test used 4 g of salt, which resulted in a molten salt thickness of approximately 3 mm. The graphite pin was first positioned near the graphite flat, as shown in Figure 1c, after which the liquid container holding the flat sample was loaded with solid salt, as shown in Figure 1d. The temperature was then increased to either 550°C or 650°C and held for 5 min. At this point, the salt had melted (melting point of FLiNaK is

454°C). The pin was then loaded against the flat to the desired contact load, and the testing was initiated. After the test was completed, the pin was retracted from contact with the flat, and the samples were allowed to cool. The salt remained as a residue on the surfaces of both the flat, as shown in Figure 1d, and the pin. The samples were removed from the sample holder and heated to 650°C to let the salt drip out. The flat samples with the holder from the 1 mm/s sliding speed tests had to be sonicated in deionized water to remove the salt.

Tribological characterization of self-mated ET-10 nuclear graphite was also performed in a dry argon environment (without FLiNaK) at the temperature of 650°C, contact load of 40 N, and sliding speed of 10 mm/s to provide a benchmark against the tests in molten FLiNaK salt. The sliding surfaces of the graphite pin and flat were characterized with a 3D laser confocal microscope (VKX-3000, Keyence, Osaka, Japan) to analyze the worn surface morphology and to determine the wear rates and wear contact modes.

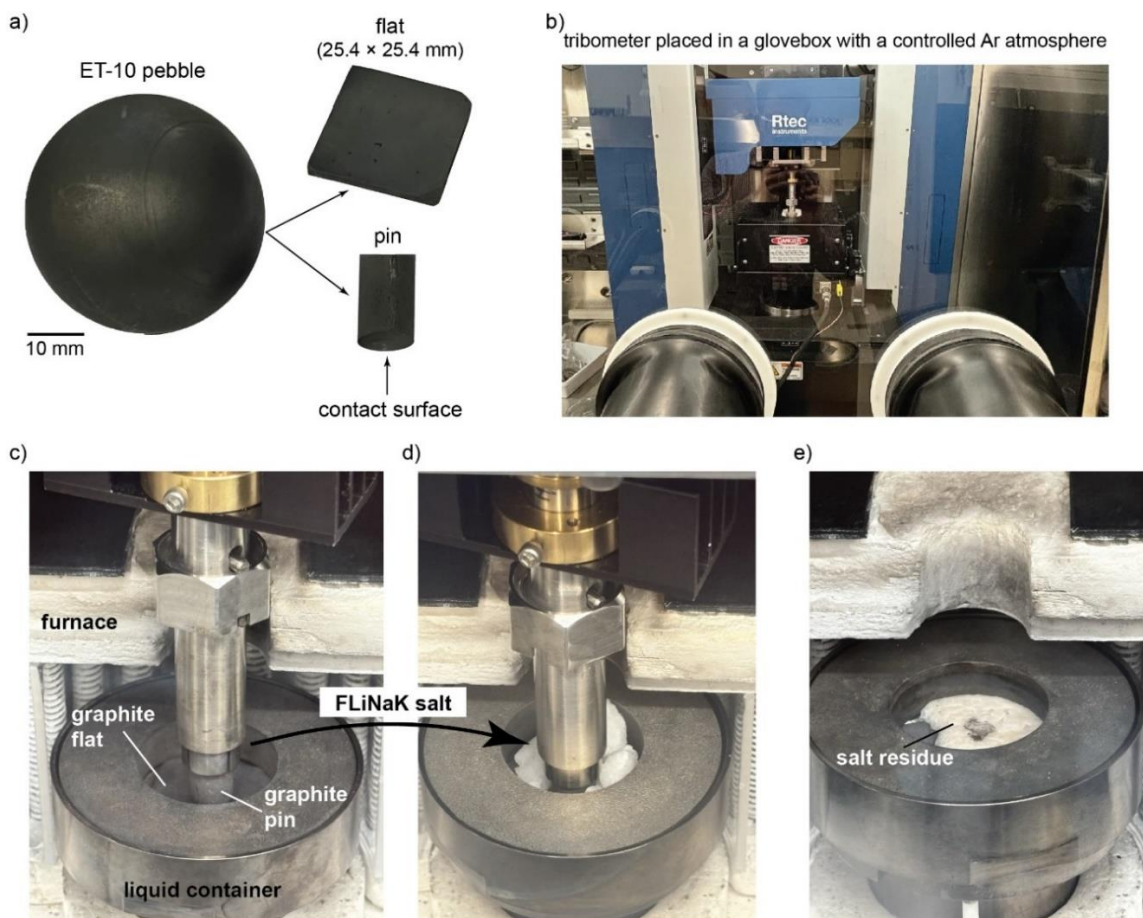


Figure 1. Test setup for tribological characterization of self-mated ET-10 nuclear graphite in molten FLiNaK salt. (a) pin and flat samples fabricated from an ET-10 nuclear graphite pebble (b) tribometer placed in a glovebox with Ar gas (c) The graphite pin is first positioned near the graphite flat and (d) the salt is then loaded into the liquid container. (e) after the test, salt residue remains on the sample surface.

Table 1. Test conditions for tribological characterization of ET-10 nuclear graphite.

Load (N)	Speed (mm/s)	Temperature (°C)	Distance (m)	Duration (min)	Environment
80	10	650	20	33	FLiNaK
80	1	650	20	330	FLiNaK
80	10	550	20	33	FLiNaK
40	10	650	20	33	FLiNaK
40	10	650	20	33	Dry argon

3. RESULTS

3.1 FRICTION BEHAVIOR

Under all test conditions, the friction behavior showed similar trends, which are illustrated in Figure 2 and Table 2. In the initial stage, the running-in friction was higher but then transitioned to a lower steady-state regime. Interestingly, the steady-state CoF remained in the range of 0.07–0.08 across all test conditions; this range is lower than the CoF range of 0.20–0.26 reported by Vergari et al. for self-mated ET-10 in FLiBe (8). The running-in friction behavior showed dependence on the test conditions. The highest CoF was observed in the test conducted at the lowest temperature of 550°C. In two repeated tests, the maximum values reached 0.40 and 0.45. The transition from running-in to a steady-state friction regime occurred after 0.61 and 0.65 m of sliding, which was the longest sliding distance among all test conditions. The effect of contact load on friction behavior was not significant: tests conducted at 40 and 80 N with the same temperature and sliding speed yielded very similar maximum CoFs of 0.33 and 0.32, respectively. However, the sliding distance necessary to reach the steady-state friction regime was slightly shorter under the lower load condition, 0.26 m compared to 0.35 m. Two repeated tests at the lowest sliding speed of 1 mm/s showed a maximum CoF of 0.31 and a similarly low running-in sliding distance, approximately 0.25 m, as observed under the 40 N test conditions. Both tests showed very good repeatability. Overall, the testing at low sliding speeds showed lower running-in friction. Dry sliding friction behavior had a similar trend compared to testing in FLiNaK but showed higher maximum CoF, 0.60 and 0.72, higher steady-state CoF, 0.30–0.36, and longer running-in sliding distance, around 3 m. The friction behavior was similar to that observed in the study by Vergari et al. (9).

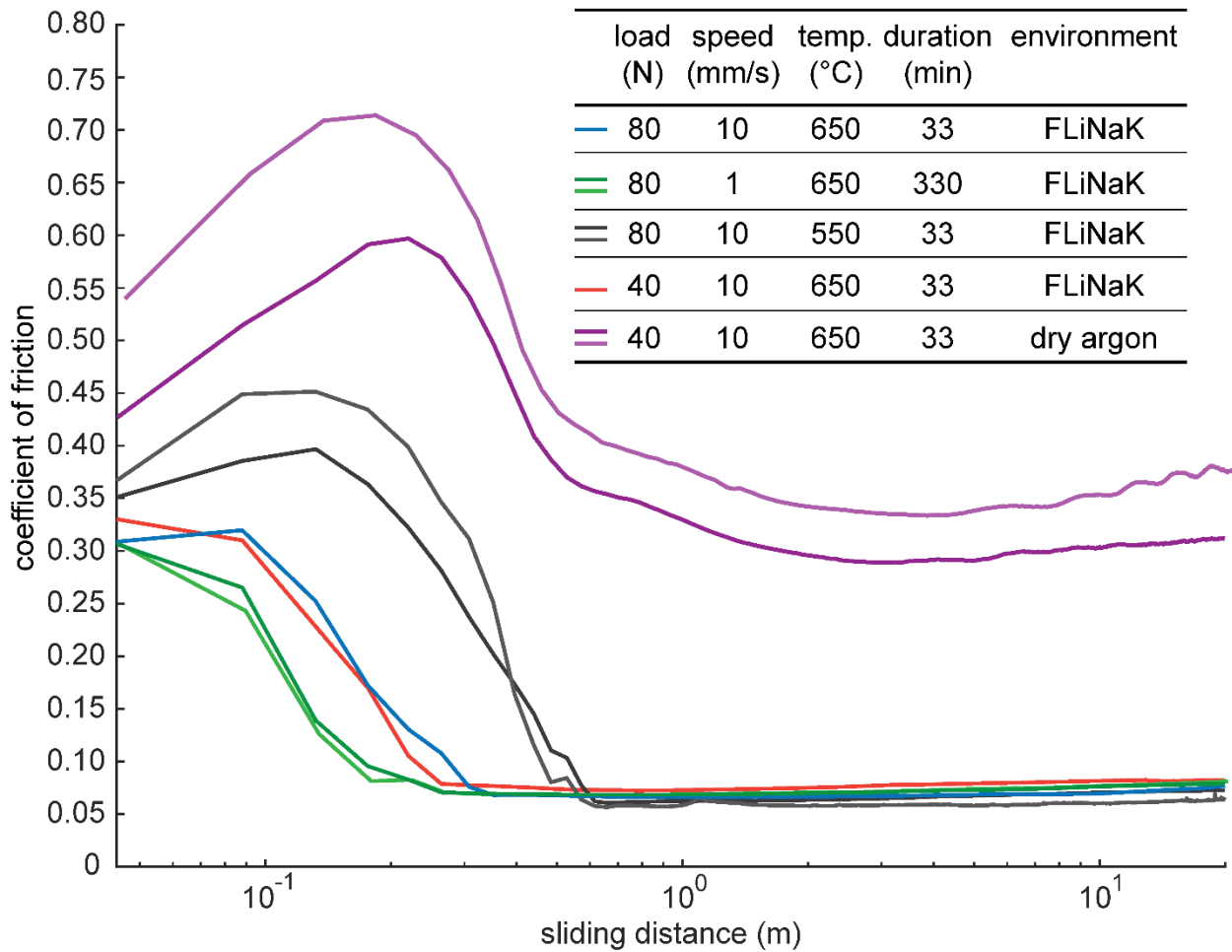


Figure 2. Friction behavior of self-mated ET-10 nuclear graphite in molten FLiNaK salt under different test conditions including dry argon.

Table 2. Summary of friction behavior of ET-10 nuclear graphite in molten FLiNaK salt under different test conditions including dry argon.

Test conditions				Friction behavior		
Load (N)	Speed (mm/s)	Temp. (°C)	Environment	Max CoF	Steady-state CoF	Running-in distance (m)
80	10	650	FLiNaK	0.32	0.07	0.35
80	1	650	FLiNaK	0.31	0.08	0.25
80	10	550	FLiNaK	0.40–0.45	0.07	0.61–0.65
40	10	650	FLiNaK	0.33	0.08	0.25
40	10	650	Dry argon	0.60–0.72	0.30–0.36	3.0

3.2 WEAR PERFORMANCE

Figure 3 and Figure 4 show the analysis of the worn surface morphology of graphite pin and flat tested in FLiNaK with the 650°C, 80 N and 10 mm/s test conditions and in dry argon conditions. The analysis consists of an optical image of the worn surface, corresponding surface topography, and line profile across the worn region. Under all test conditions in FLiNaK, the worn surface morphology of the graphite pins was very similar. A comparison of the surface line profiles of the graphite pin before and after testing indicates material loss, as shown in Figure 3b,c. The worn surface has lost its original curvature and appears smoother than the surrounding unworn regions. Residual salt deposits can also be seen on the surface. Abrasion was identified as the primary wear mechanism. Similar wear morphology was observed on the graphite pins tested in dry argon, as shown in Figure 3d,e. More wear debris deposits appear around the worn surface in the direction of sliding.

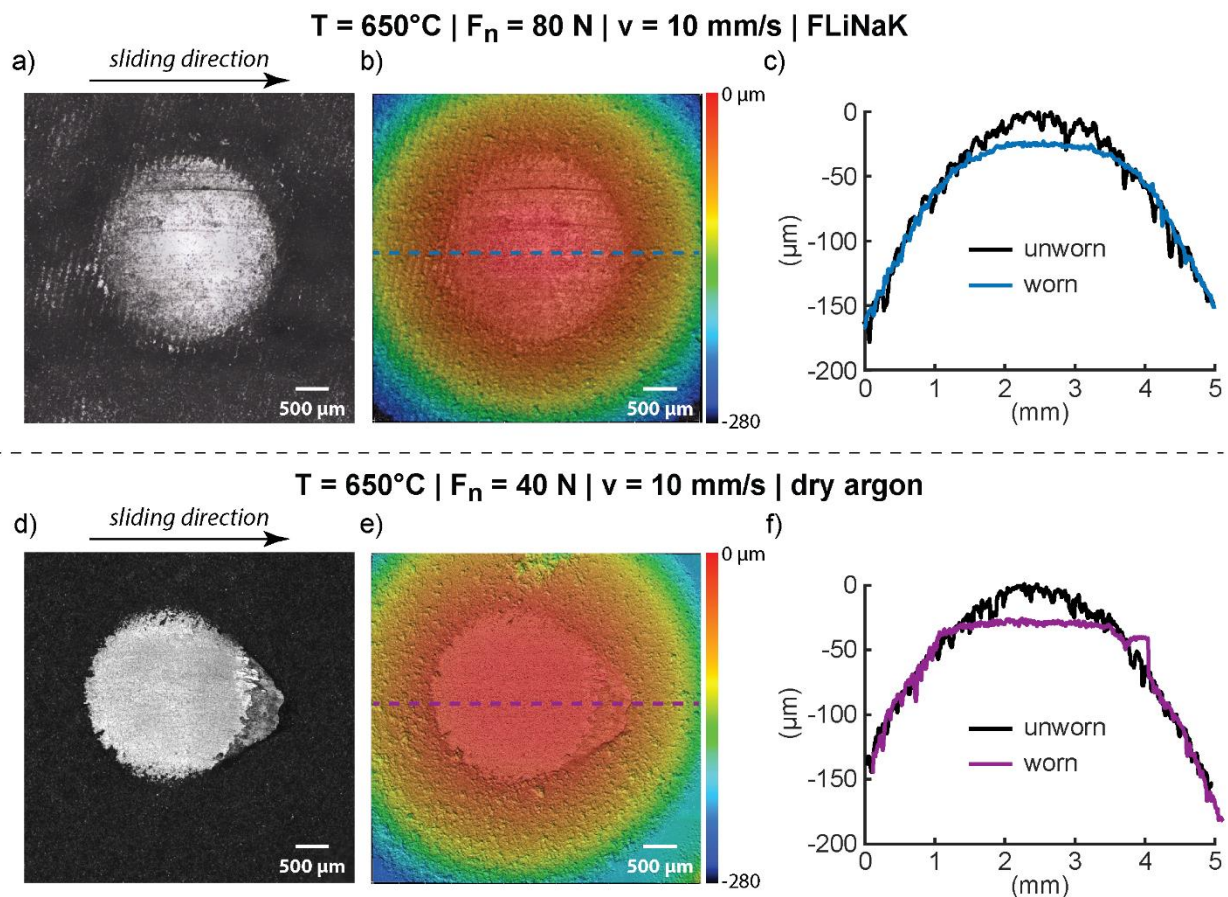


Figure 3. Analysis of the worn surfaces of ET-10 nuclear graphite pins. (top row) Molten FLiNaK salt condition: (a) optical image, (b) corresponding surface topography, and (c) line surface profiles compared before and after the test. (bottom row) Dry argon condition: (d) optical image, (e) corresponding surface topography, and (f) line surface profile compared before and after the test.

The wear volume and wear rate of the graphite pins depend on the test conditions, as shown in Figure 4 a,b. Testing at the lowest contact load of 40 N resulted in the lowest wear volume. Among the tests conducted at 80 N, those performed at the lowest sliding speed of 1 mm/s and the lowest temperature of 550°C resulted in a higher wear volume compared to the 650°C and 10 mm/s test conditions. The lowest wear rate, which is the wear volume normalized by the contact load and the sliding distance, was determined with the 650°C, 80 N and 10 mm/s test conditions and was approximately $3.4 \times 10^{-5} \text{ mm}^3\text{N}^{-1}\text{m}^{-1}$. Although testing at the lowest load of 40 N resulted in the lowest wear volume, the wear rate was $5.5 \times 10^{-5} \text{ mm}^3\text{N}^{-1}\text{m}^{-1}$ which is $1.6 \times$ higher than that of the 80 N test condition. The wear rates under the lowest sliding speeds were 5.6×10^{-5} and $6.6 \text{ mm}^3\text{N}^{-1}\text{m}^{-1}$. The highest wear rates were measured under the lowest temperature of 550°C, 6.6×10^{-5} and $7.1 \times 10^{-5} \text{ mm}^3\text{N}^{-1}\text{m}^{-1}$. The wear rate of the graphite pin tested in dry argon environment was 4.7×10^{-5} and $5.5 \times 10^{-5} \text{ mm}^3\text{N}^{-1}\text{m}^{-1}$, which is similar to the wear rate in the molten FLiNaK salt with the same contact load, speed, and distance test conditions.

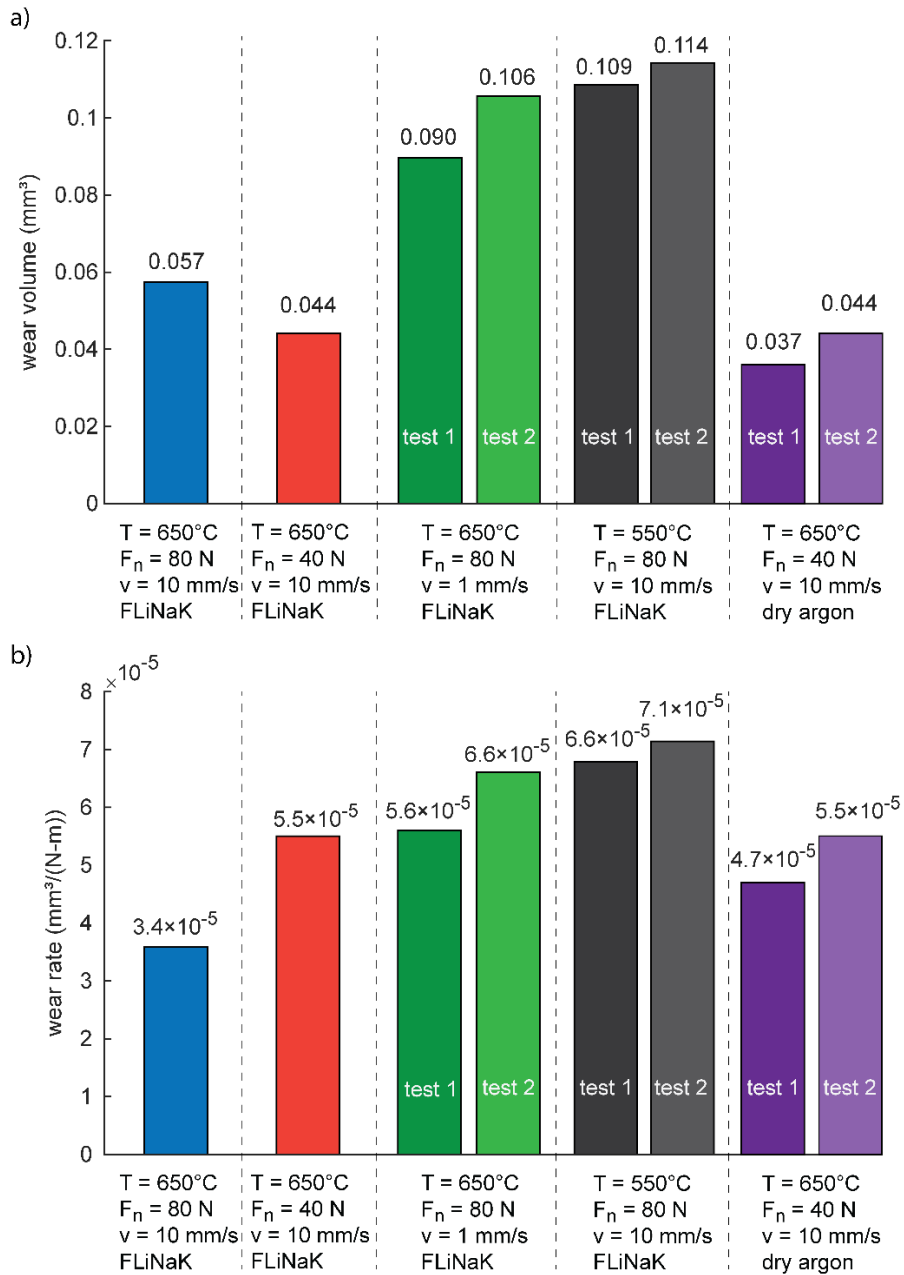


Figure 4. Wear volume and rate depend on test conditions. (a) wear volume and (b) wear rate of self-mated ET-10 nuclear graphite in molten FLiNaK salt under different test conditions including dry argon.

No measurable material loss was observed on the sliding surfaces of the graphite flats under any of the test conditions in molten FLiNaK salt. The sliding surface has lower roughness than the surrounding unworn area, suggesting that the surface asperities were smoothed by abrasion during the sliding contact with the graphite pin, as shown in Figure 5a. Traces of the residual salt were present on and around the sliding surface. The sliding surface of the flat formed in the dry argon environment had no measurable wear and appeared to be smoother than the one in the molten FLiNaK salt conditions, as shown in Figure 5b.

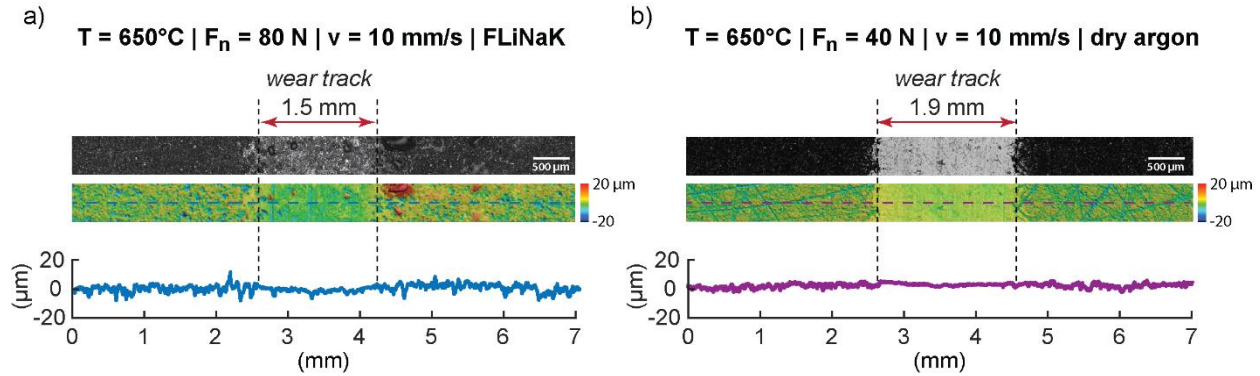


Figure 5. Analysis of the worn surfaces of ET-10 nuclear graphite flat in (a) molten FLiNaK salt and (b) dry argon conditions. The analysis includes optical images, corresponding surface photography and line surface profiles.

4. DISCUSSION

The tribological characterization of self-mated nuclear graphite ET-10 in a molten FLiNaK salt revealed that the temperature, sliding speed, contact load, and duration significantly affect the friction and wear behavior. The higher than 0.1 running-in CoF in all test conditions suggests boundary lubrication. The graphite pin experienced more material loss than the flat during the running-in period. As the sliding progressed, the CoF values decreased to <0.1. This result could be explained by the increased contact area due to the worn graphite pin tip and decreased surface roughness during the running-in. The lower and consistent CoF values after the running-in period suggest that stable boundary lubrication was established. In the pin-on-disc wear tests, material loss was observed on the pin but not on the disc surface, primarily because the pin remains in continuous contact during sliding, whereas each point on the flat experiences intermittent contact over a larger area.

Temperature had the most significant influence on the friction and wear behavior. The higher running-in CoF and wear rates were observed at a lower temperature of 550°C. This behavior could be correlated with the strength of the graphite, which increases with increasing temperatures. By contrast, the viscosity of the molten FLiNaK salt at 550°C is 6.34 mPa·s which is higher than that at 650°C, 3.66 mPa·s; therefore, the higher viscosity of the salt at the lower temperature would be expected to reduce the friction and provide a better protection against wear. However, the results indicate that the improved mechanical properties of the graphite dominated the influence of salt viscosity.

Testing at the lowest speed of 1 mm/s and the longest duration of 330 min resulted in a lower CoF during the running-in period but higher wear rate compared with the test at 10 mm/s. The molten salt lubricating film is thinner at lower sliding speeds, possibly explaining the higher wear rate. The longest test duration

of 330 min at the lowest sliding speed could have also contributed to higher wear. The 10× longer exposure to the salt could have resulted in salt intrusion into the graphite pores (10–12), causing material degradation.

Contact load had no noticeable effect on the friction behavior: testing at 40 and 80 N showed similar trends and values of CoF. The lowest wear volume was found at a contact load of 40 N; however, the lowest wear rate was achieved at 80 N. According to the Hertz theory, the contact mean pressure P scales with contact load F_n as $P \sim F_n^{1/3}$, which implies that at higher loads, the increase in contact area leads to a relatively smaller rise in contact pressure, potentially reducing the wear rate despite higher loads.

The wear mechanism of graphite in molten FLiNaK salt is fundamentally different from that in dry argon conditions. Molten FLiNaK salt acts as an effective boundary lubricant, which contributes to lower friction. In dry sliding conditions, however, the lower steady-state friction regime is attributed to the development of a tribofilm, which is a lubricious carbon layer adhered to the contact surface (9, 13). The results from this work indicate that molten FLiNaK salt provides more efficient lubrication than the tribofilm formed in dry argon conditions, as evidenced by the lower friction, although the wear performance was very similar.

5. ONGOING AND FUTURE WORK

ORNL has developed a methodology to characterize the friction and wear behavior of graphite in molten FLiNaK salt under sliding contact mode conditions. The results presented in this report represent initial studies on self-mated ET-10 nuclear graphite. However, graphite matrix material is the actual outer shell of the pebbles while nuclear graphite is the reactor wall material. Ongoing work is investigating friction and wear behavior of matrix material under dry argon conditions, with future studies planned to extend testing in FLiNaK environment. Tribological characterization will include matrix-on-matrix configurations to assess wear and friction between pebbles, as well as matrix-on-graphite configurations to simulate contact between pebbles and reactor walls. Additional investigations are planned to explore other relevant contact wear modes, including rolling and impact, which are critical for fully understanding graphite performance in reactor environments. These expanded studies will provide a more comprehensive understanding of material degradation mechanisms in molten salt systems.

6. CONCLUSIONS

This report summarizes the initial tribological characterization of self-mated ET-10 nuclear graphite in molten FLiNaK salt. The friction and wear behavior were studied using a pin-on-disk configuration for

varying temperatures (550 and 650°C), contact loads (40 and 80 N), and sliding speeds (1 and 10 mm/s) in a controlled argon environment. The tribological behavior in FLiNaK was compared to that in dry argon conditions.

- The temperature had the most significant effect on the friction and wear behavior. Testing at 550°C resulted in the highest running-in CoF and the highest wear rate among all test conditions. This result was attributed to the lower strength of graphite at lower temperatures.
- The lowest sliding speed of 1 mm/s resulted in the lowest running-in friction but a higher wear rate compared with the 10 mm/s sliding speed. Lower friction could be due to less-intense vibration during sliding, whereas higher wear could be caused by a thinner molten FLiNaK salt lubricating film at lower speeds and a longer exposure of graphite to the salt.
- Increasing the contact load from 40 to 80 N had no effect on the friction behavior. However, the wear rate decreased with increasing contact load.
- Under all test conditions, the steady-state CoF had very similar values, 0.07–0.08, suggesting that a stable boundary lubrication regime was established.
- The running-in and the steady-state CoF of graphite in dry argon conditions were higher compared with molten FLiNaK salt. The wear behavior was similar to the molten FLiNaK salt conditions.

The results from this report provide key information about the tribological behavior of graphite components in PB-FHRs and could contribute to safer operation. The future work will focus on investigating the friction and wear of graphite matrix material as well as rolling and impact contact wear modes.

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