Kairos Power's Perspectives on Fluoride Molten Salt-Graphite Interactions

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JULY 20-21, 2022

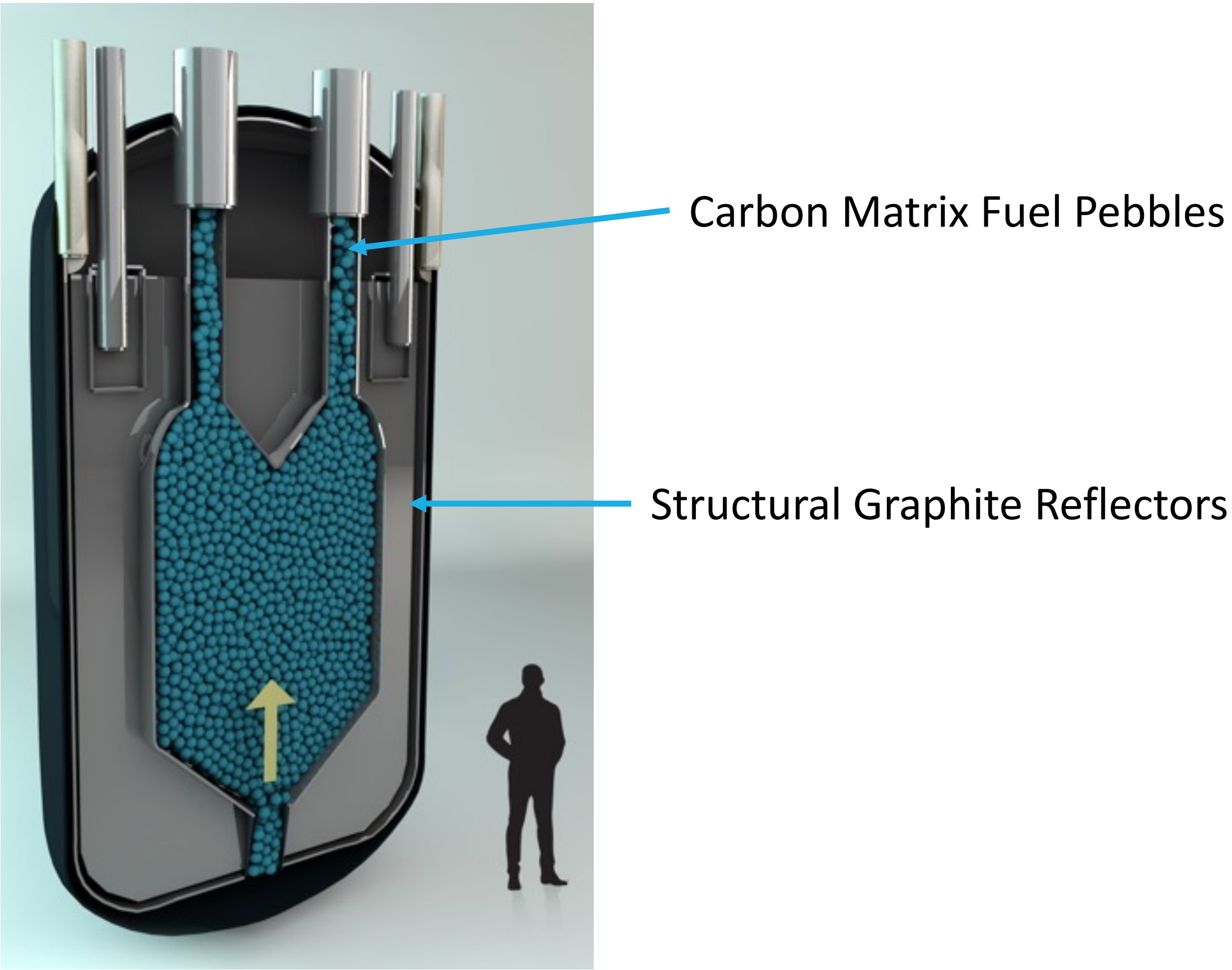
Kairos Power's mission is to enable the world's transition to clean energy, with the ultimate goal of dramatically improving people's quality of life while protecting the environment.

In order to achieve this mission, we must prioritize our efforts to focus on a clean energy technology that is *affordable* and *safe*.

Outline

- Brief introduction of Kairos Power Fluoride Salt-Cooled High Temperature Reactor
- Kairos Power Fuel/Graphite Tribology Test in Flibe Molten Salt
- Compatibility of Graphite with Flibe Molten Salt
- Practical Way to Determine the Effect of Fluoride Molten Salt on Graphite Strength

Graphite/Salt in Kairos Power Fluoride Salt-Cooled High Temperature Reactor

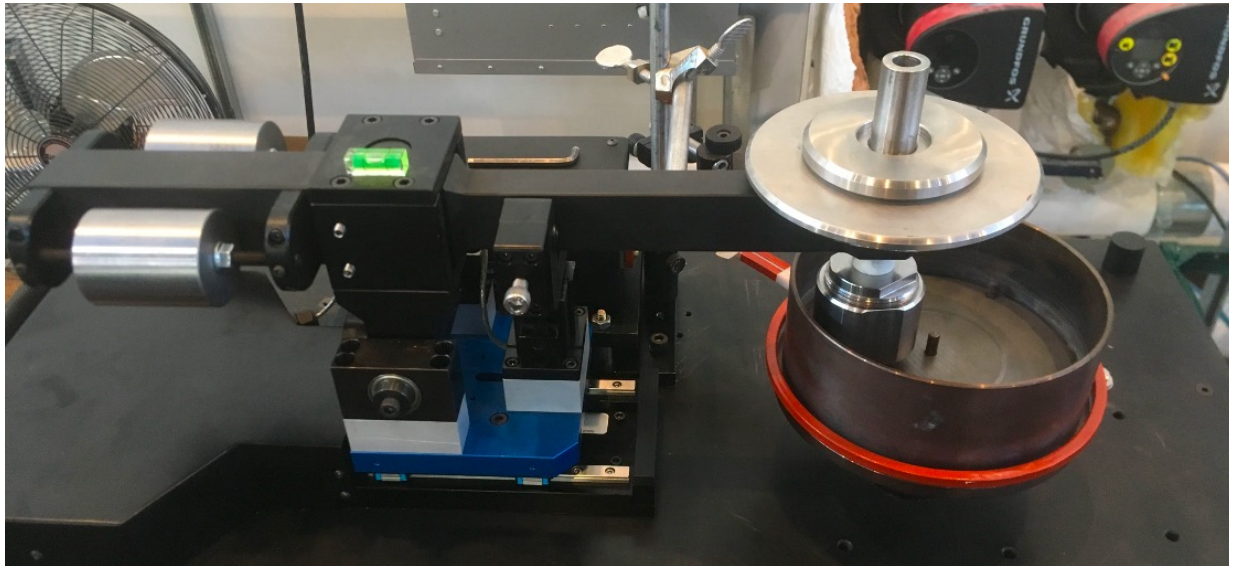


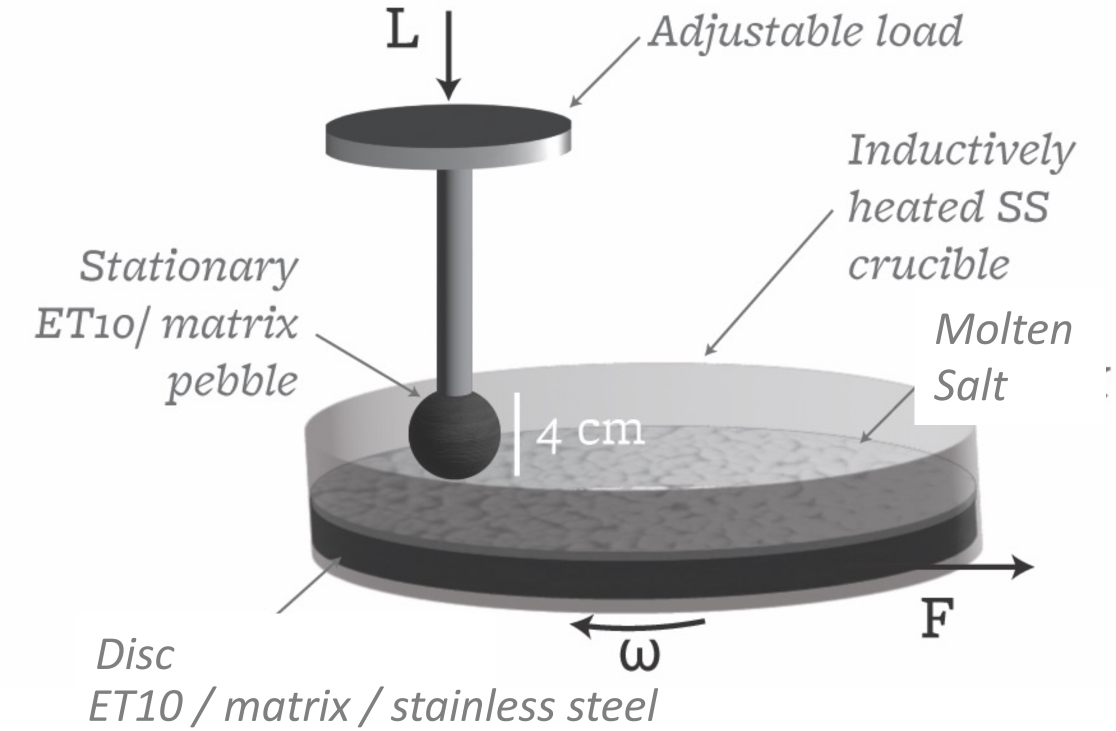
Fuel Qualification Plan, including **tribology in salt** to quantify fuel pebble wear

Graphite/Flibe Interaction Qualification Plan to demonstrate that the graphite reflector structure maintains its safety functions when exposed to Flibe

Fuel Pebble Tribology in Salt

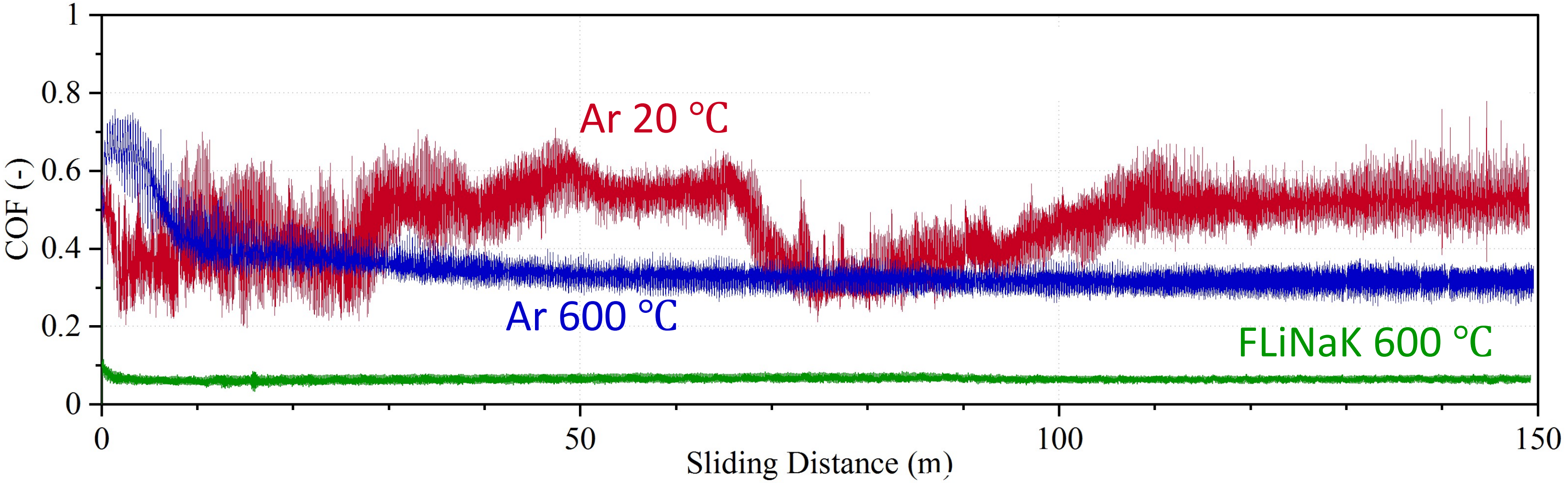
- **Setup:** Ball on disc
- **Environment**: Argon or salt, in controlled atmosphere
- **Experimental Parameters:** Representative loads and speeds
- **Observables:** Wear rate, coefficient of friction

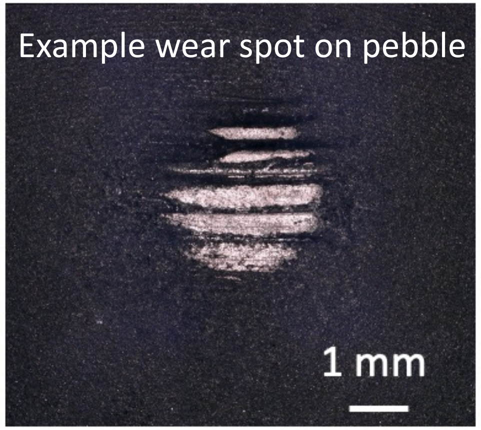


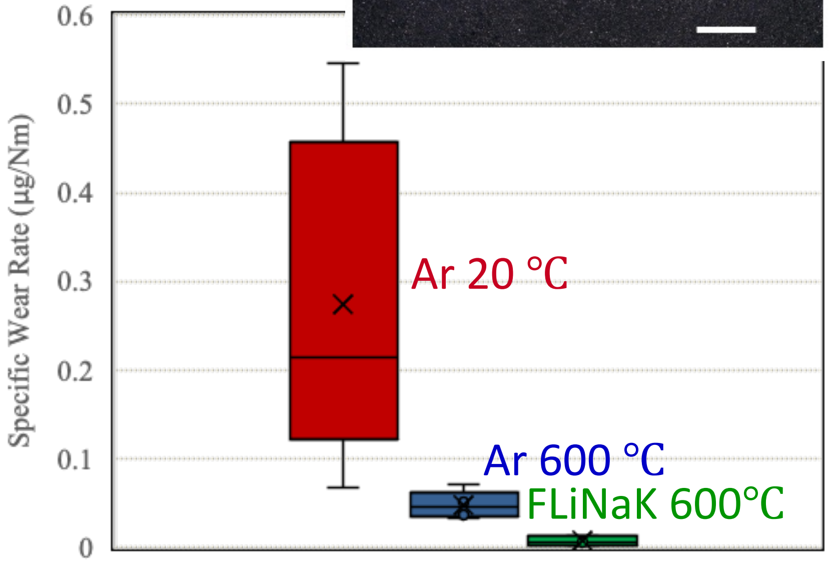


Schematic of the experimental setup

Results for ET-10 Surrogate Pebbles in Surrogate Salt (Flinak)







Next Steps: Testing being performed with test carbon matrix pebbles in Flibe

Any Chemical Reaction between Graphite and Fluoride Molten Salt?

- Graphite was considered stable in fluoride molten salt based on MSRE results.
- A recent study took a new look and observed fluorination. Was there any concern?
  - Trace level surface fluorination observed from surface analysis
  - No result indicate its impact to graphite integrity
- What kind of reactions should be a concern?
  - Intercalation, leading to exfoliation---this is a known reaction that could cause graphite structure degradation.

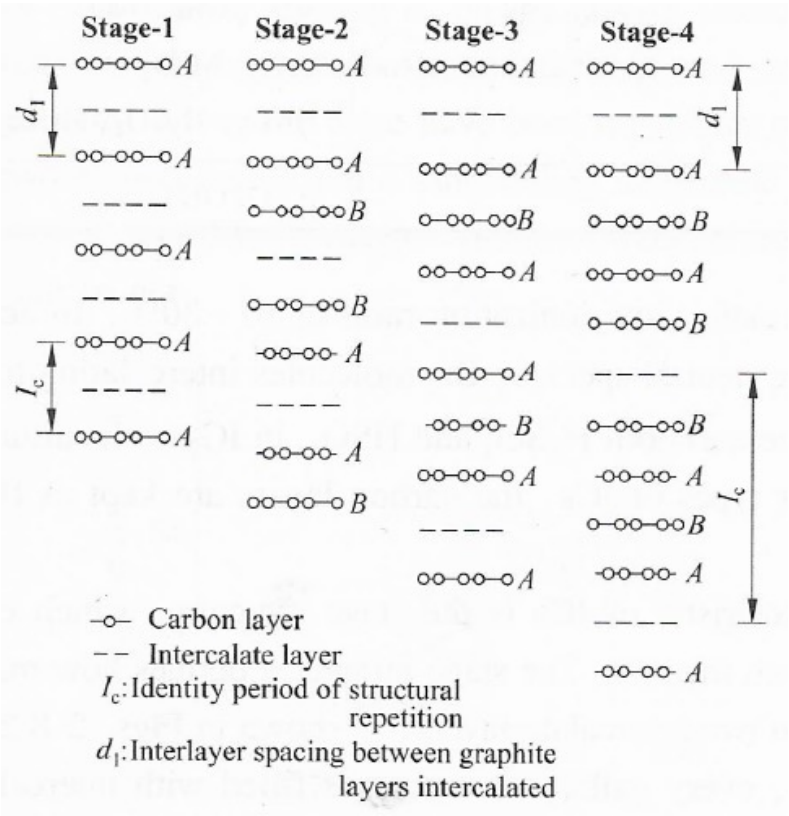
Review of MSRE Results on Graphite Compatibility in Flibe Molten Salt

- Graphite grade: CGB grade, entrance pore diameter ≤1μm
- Salt: $LiF\text{-}BeF_2\text{-}ZrF_4\text{-}UF_4$ (65-30-5-<1% mole); $LiF\text{-}BeF_2$(66%-34% mole)
- Operation condition
  - Graphite exposed to Flibe salt for 3.5years (including 2.4years with fuel salt), at 500-650°C
  - Peak fluence graphite received, ~ 1×10^{21} neutrons/cm^2 (>50keV)
- Post operation examination, 2 pieces of graphite inspected
  - One graphite core moderator elements.
    - "Visual examination revealed machining marks still plainly visible in the fuel channels..."
    - Inspection of core graphite moderator elements dimension, "all measurements were within the original tolerance for fabrication of the core block"
  - Graphite surveillance specimen
    - "…machining marks were clearly visible, and we found no evidence of chemical reaction with the fuel salt."
  - No mechanical property test performed (as no salt infiltration detected)

Ref: ORNL TM4174, 1972

Graphite Intercalation

- Graphite Intercalate Compound (GIC) can be detected by XRD
- Intercalation leading to exfoliation is a known mechanism to damage graphite structural. For example,
  - Process to produce expanded graphite,
  - Li-ion battery graphite anode failure mode
- Intercalation observed with Chloride Salt, F_2, LiF with F_2, but not with Flibe and FLiNaK
- Temperature effect: In general, intercalation is discouraged at high temperature. For example,
  - $FeCl_3$ reacts with graphite at ~300°C, but not at 750°C
  - F_2 reacts with graphite at 350-600°C, but not at > 2000°C
- Irradiated graphite tends to be less graphitic, which makes intercalation even more difficult.



Graphite Intercalate Compound stage structure

Inagaki, M., Kang, F. Carbon Materials Science and Engineering

Recent Findings on Fluorination

- Yang, X.; et al. claimed that there is large amount of H in graphite, which is attributed to the residual C-H bond after graphitization…, H atom on the edge plane is substituted by the F.
  - Comments: C-H bond will not survive at graphitization temperature (2800-3000°C), the observed H may not be from "residual" C-H bond. "large amount of H" has no evidence at bulk level. The Fluorination is possible but is at trace level at most.

- Wu, H.; et al. claimed observation of the fluorination mainly based on XPS analysis and proposed a reaction mechanism via –COOH, C=O, -H on the graphite edge plane.
  - Comments: XPS results interpretation could be tricky for those weak signals. Those functional groups in graphite, if any, are at trace level on the graphite surface. Therefore, the fluorination, if any, is also at trace level on the surface.

The literature data suggests the possible fluorination, but no convincing data for bulk level fluorination.

Key question to be addressed: Do these reactions affect the graphite's strength?

References:
Yan, X.; Feng, S.; Zhou, X. et al. Interaction between Nuclear Graphite and Molten Fluoride Salts: A Synchrotron Radiation Study of the Substitution of Graphitic Hydrogen by Fluoride Ion. J. Phys. Chem. A 2012, 116, 985
Wu, H.; Carotti, F.; Gakhar, R. et al. Fluorination of Nuclear Graphite IG-110 in Molten 2LiF-BeF_2 (FLiBe) Salt at 700°C, J. Fluorine Chem. 2018, 211, 159

Effect of Fluoride Molten Salt on Graphite Strength

- Molten salt may affect graphite strength in two possible ways
  - Salt-graphite chemical reaction leads to damage graphite structure
  - Physical effect induced from infiltrated salt, e.g., stress

- Should graphite' strength be measured with or without infiltrated salt?
  - With infiltrated salt in graphite, one measures strength of "graphite-salt composite"
  - Without infiltrated salt, one measures the strength of graphite after contacting salt.

Summary of Current Understanding

- At room temperature, compressive strength increases with infiltrated salt (FLiNaK).
  - Salt acts as reinforcement
- At high temperature (700°C), compressive strength decreases with the amount of salt infiltrated
  - Stress induced from solid salt and stress concentrator are responsible for the strength reduction.
    - Sample went through cooling-reheating cycle
    - $CTE_{\text{Graphite infiltrated with FLiNaK}} > CTE_{\text{pristine graphite}}$, at temperature below salt melting temperature
    - $CTE_{\text{Graphite infiltrated with FLiNaK}} \approx CTE_{\text{pristine graphite}}$ at temperature above salt melting temperature
    - CTE of salt (FLiNaK) is ~ x10 of graphite's CTE
- The results indicate a clear understanding of physical effect of salt, but chemical effect cannot be determined.

References

Zhang, C. The effect of molten FLiNaK salt infiltration on the strength of graphite. J. Nucl. Mat. 2018, 512, 37
Qi, W. Effects if FLiNaK infiltration on thermal expansion behavior of graphite. J. Mat. Sci. 2017, 52, 4621

Determination of Chemical Effect of Fluoride Salt on Graphite Strength

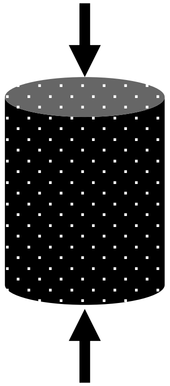
Question: Is there any Fluoride salt – graphite reaction causing reduction of graphite strength?

To address this, chemical and physical effects on strength must be separated.

- Proposed Methods
  - Approach 1: Wash off infiltrated salt (FLiNaK or Flibe), then measure the strength
  - Approach 2: Without washing off salt, but perform tensile strength test instead of compressive strength test, only at room temperature

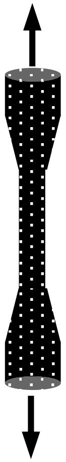
Compression mode

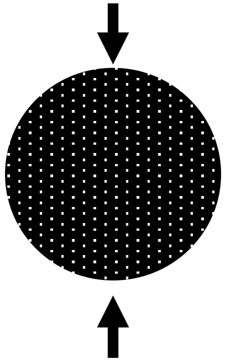
- Solid salt reinforcement cannot be avoided



Tension mode

- None or minimum solid salt reinforcement
- "Dog bone" specimen may be ideal but complicated.
- "Splitting disc" specimen is more practical but need to be validated.





Summary

- Kairos Power is developing the in-depth understanding of the effect of fluoride molten salt on graphite properties.
- Kairos Power established and is establishing in-house capability in the following areas
  - Tribology test
  - Molten salt infiltration test
  - Characterization instrument to determine the change of graphite properties
- Expand capability through external collaboration and DOE funding (NSUF) project to address the integrated effects of irradiation and Flibe molten salt on graphite properties

Thank You