



# Alternative Crucible Materials

May 2018

*Changing the World's Energy Future*

David D



#### **DISCLAIMER**

This information was prepared as an account of work sponsored by an agency of the U.S. Government. Neither the U.S. Government nor any agency thereof, nor any of their employees, makes any warranty, expressed or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness, of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. References herein to any specific commercial product, process, or service by trade name, trade mark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the U.S. Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the U.S. Government or any agency thereof.

# **Alternative Crucible Materials**

**David D**

**May 2018**

**Idaho National Laboratory  
Idaho Falls, Idaho 83415**

**<http://www.inl.gov>**

**Prepared for the  
U.S. Department of Energy  
Under DOE Idaho Operations Office  
Contract DE-AC07-05ID14517**

# ***Alternative Crucible Materials***

Presented by David D. Tolman

**16<sup>th</sup> Joint ERWG/SSWG Meeting  
Idaho National Laboratory**

15 - 17 May, 2018



**U.S. - ROK  
Joint Fuel Cycle Studies**

[www.inl.gov](http://www.inl.gov)



## ***Alternative Crucible Materials Outline***

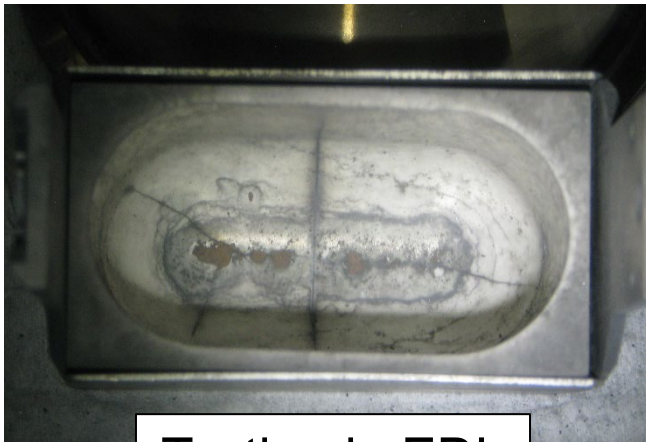
- BeO Crucible Overview
- HfN Coated Nb Crucible Development
- UO<sub>2</sub> Coating Development
- UO<sub>2</sub> Castable Refractory Development
  - Powder preparation
  - Binder selection
  - Mold development and testing – initial attempts
  - Mold development and testing – redesigned mold
  - Future work



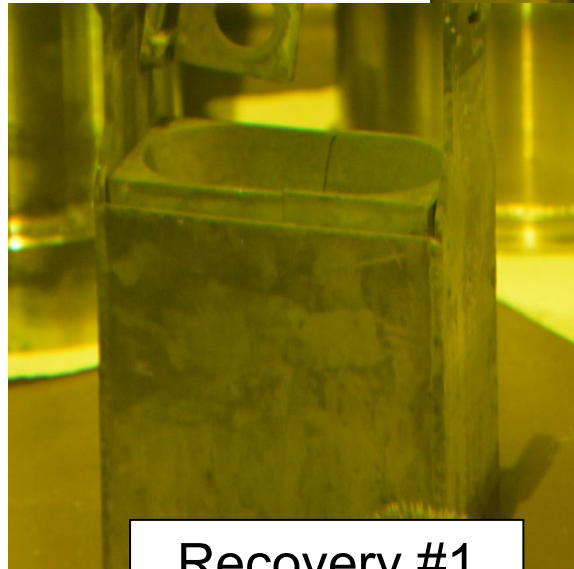
U.S. - ROK Joint Fuel Cycle Studies

# BeO Crucible Overview

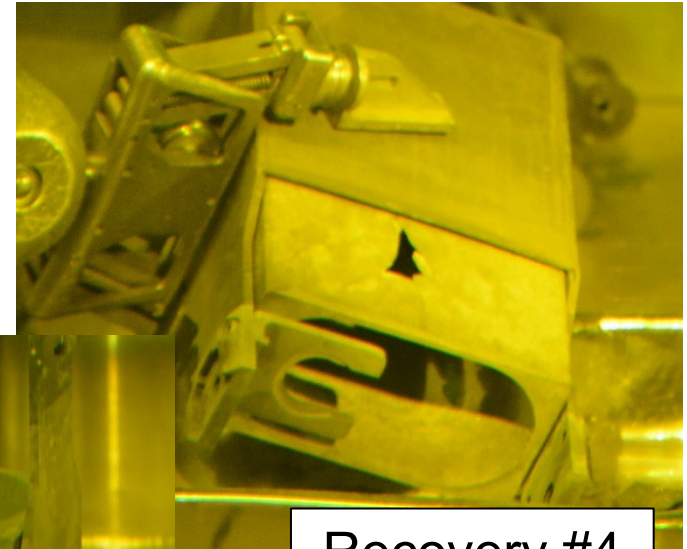
- BeO was originally chosen as the preferred crucible material
- Failure during distillation step



Testing in EDL



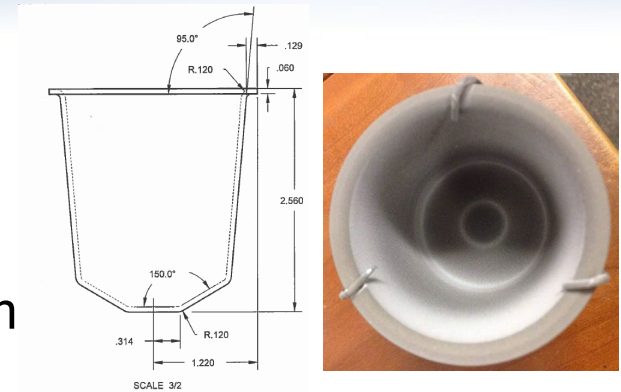
Recovery #1



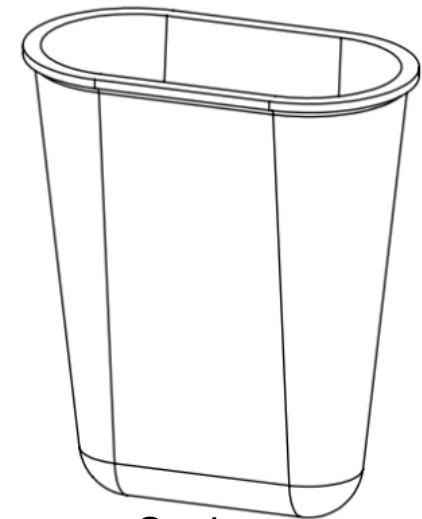
Recovery #4

# HfN Coated Nb Crucible Development

- Developed a HfN coated niobium crucible that fits inside the JFCS casting furnace graphite crucible
- Procurement of scaled up niobium crucible from Metal Technology Inc. (MTI)
  - Difficult geometry to fabricate economically
  - Expected delivery week of May 21st, 2018
- HfN coating Scale up crucible
  - The Center for Thermal Spray Research at State University of New York (SUNY) is ready to begin coating testing for this geometry once they receive crucibles
  - SUNY will develop coating using iron based (stainless steel) prior to trying on Nb



Casting Furnace



Scale up





# ***UO<sub>2</sub> Coating Development***

- Coating Method
  - UO<sub>2</sub> powder (<100 mesh)
  - 5:1 (UO<sub>2</sub> to LRC binder) ratio by weight
  - Mixture poured into graphite crucible and rotated until interior surface was entirely covered
- Preliminary Melt Testing
  - Uranium
  - U-10Zr
  - U-3RE



Coated crucible before drying  
and after drying



Crucible coating after uranium  
melt testing





# ***UO<sub>2</sub> Castable Refractory Development***

- Powder preparation
  - Feedstock: 5/8 inch (16mm) diameter pellets
  - Manual rough crush
  - Jar-milling (tumbling)
    - Sieve homogeneous sample
    - Target particle size distribution
      - Based on commercially available ZrO<sub>2</sub> castable refractory





# ***UO<sub>2</sub> Castable Refractory Development***

- **Binder selection**

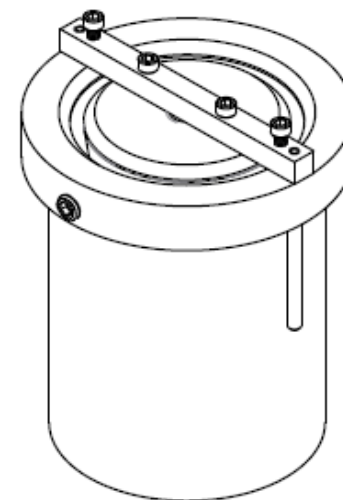
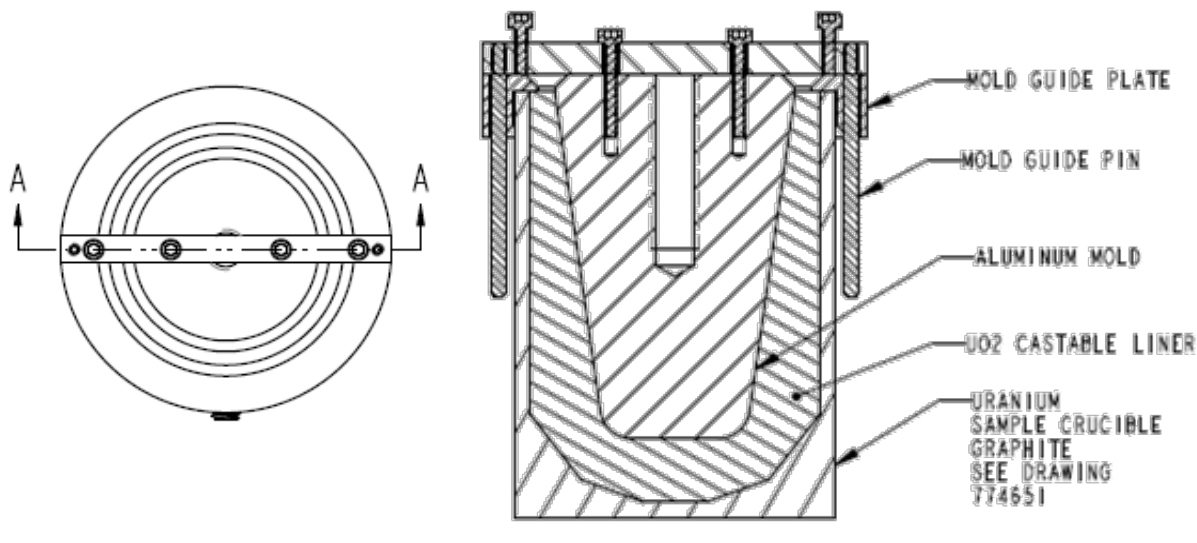
- Initial testing with two binders from ZYP Coatings
  - LRC
  - Gel-P
- UO<sub>2</sub> powder was mixed with binder in nickel crucibles and allowed to harden
- LRC binder was selected for further testing
  - Less binder required to fluidize mixture





# ***UO<sub>2</sub> Castable Refractory Development***

- Mold development
  - Molds were developed to cast UO<sub>2</sub> liners into graphite crucibles
    - Small size
      - Casting furnace crucible
    - Medium size
      - Uranium sampling crucible

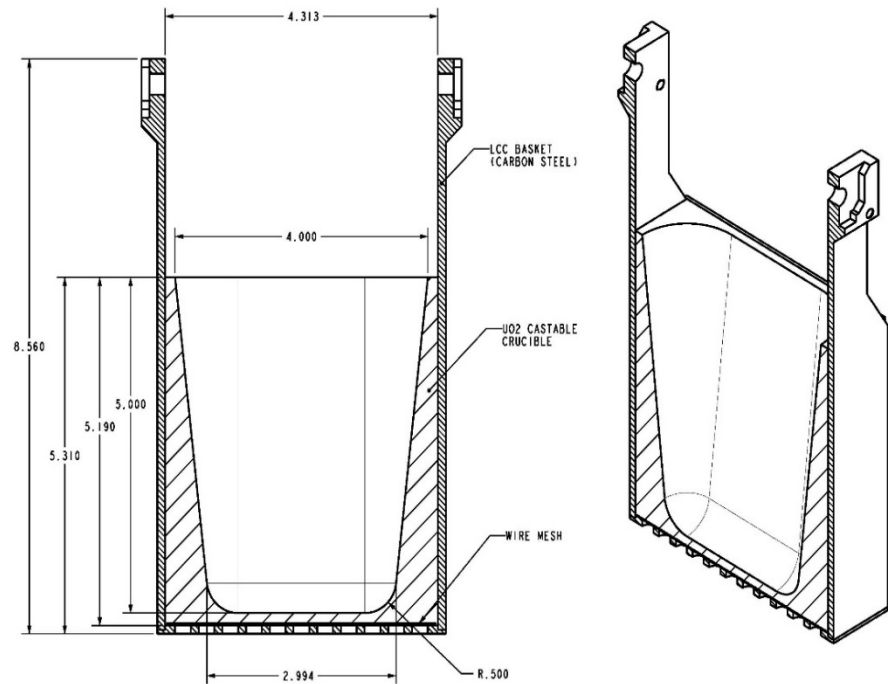
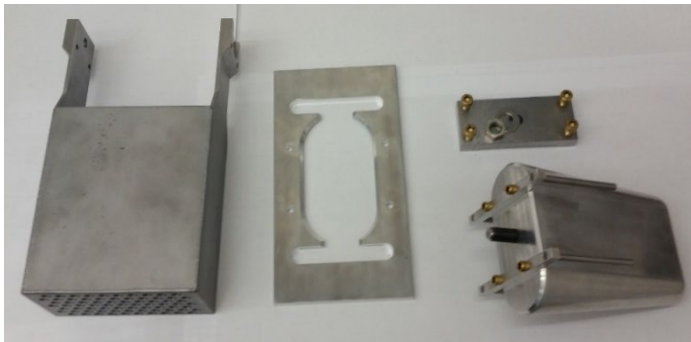
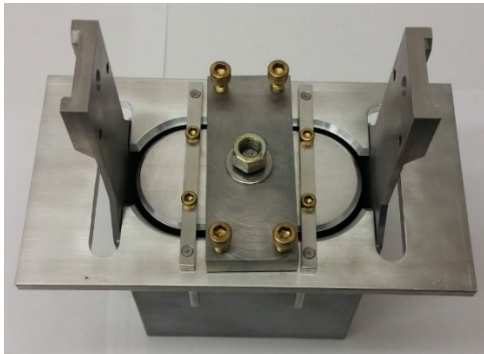




U.S. - ROK Joint Fuel Cycle Studies

# ***UO<sub>2</sub> Castable Refractory Development***

- Mold development
  - Mold developed for casting UO<sub>2</sub> liners into a steel basket



UO<sub>2</sub> LINED LCC CRUCIBLE  
TOTAL CRUCIBLE CAPACITY = 390cc

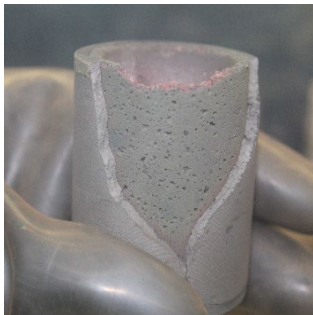




U.S. - ROK Joint Fuel Cycle Studies

# ***UO<sub>2</sub> Castable Refractory Development***

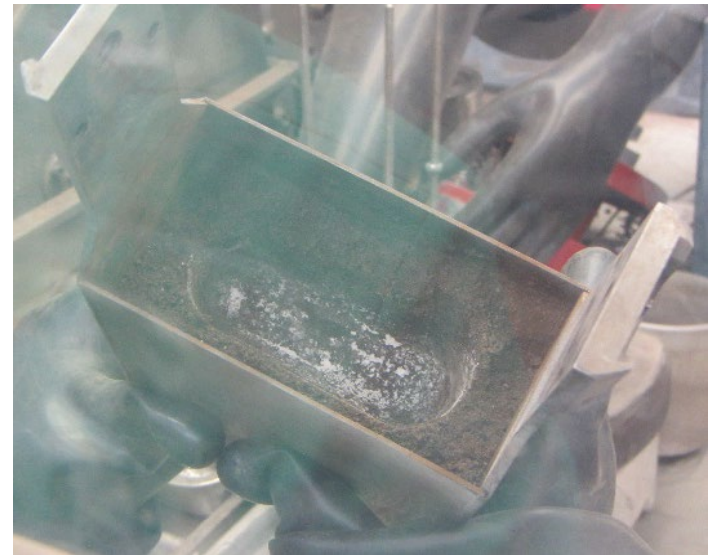
- Mold development and testing
  - Small and medium crucible liners cast using LRC binder were mostly successful
    - Cracked graphite during firing
  - Large crucible liner did not adequately dry and harden in a reasonable amount of time.





# ***UO<sub>2</sub> Castable Refractory Development***

- Issues with binder discovered in testing
  - New binder selection
    - LRC binder did not dry in a reasonable amount of time
    - Secar 71®
  - Attempts on Small and Medium crucibles were successful
    - no cracked graphite
  - Large mold was not able to be pushed sufficiently into the refractory

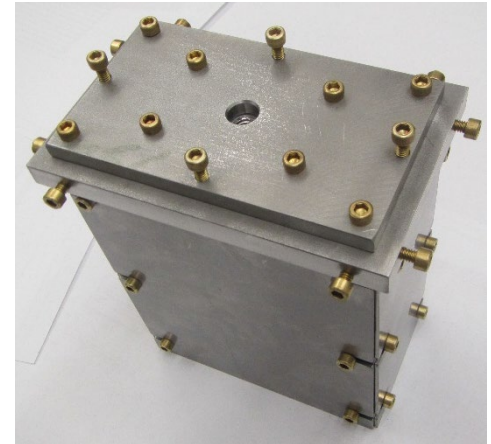
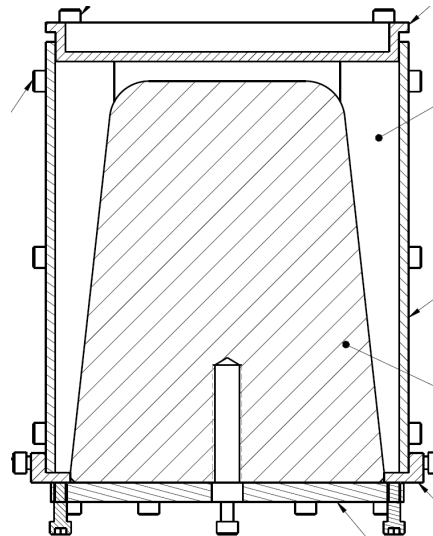






# ***UO<sub>2</sub> Castable Refractory Development***

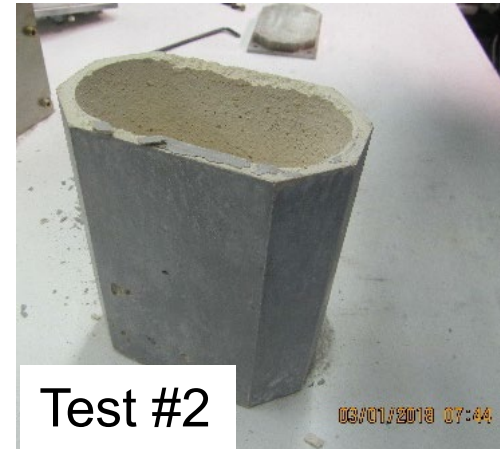
- Redesigned mold for large crucibles
  - Creates standalone crucible
  - Form crucible upside-down
  - Refractory mix is poured into the mold
    - no pushing mold into mixture
  - 48 hours cure
    - Inner mold piece is removed
  - Refractory is heated to remove excess moisture
  - Outer form pieces are removed





# ***UO<sub>2</sub> Castable Refractory Development***

- Redesigned mold testing
  - Testing with ZrO<sub>2</sub> segregate material
    - Seven tests
    - Investigated mold release agents
    - Developed methods to improve inside surface finish
    - Refined binder and water ratios
    - Developed improved methods to mix refractory in glovebox





U.S. - ROK Joint Fuel Cycle Studies

# ***UO<sub>2</sub> Castable Refractory Development***

- Redesigned mold testing
  - At this time we have formed two large crucibles
  - pending high temperature sintering/firing (1600°C)



# ***UO<sub>2</sub> Castable Refractory Development***

- Future work for UO<sub>2</sub> crucible development
  - High temperature sinter/fire large crucibles
  - Melt uranium metal in UO<sub>2</sub> crucibles to study behavior
  - Melt salts in UO<sub>2</sub> crucibles to study behavior with the refractory
  - Melt other materials (rare earth, etc.) to study interaction with the UO<sub>2</sub> refractory.
  - Deploy UO<sub>2</sub> refractory for use in IRT equipment
- Plan for future IRT material recoveries
  - Fabricate hardware for three options
    - BeO scrape crucible (without drain holes)
    - HfN coated Nb crucible
    - UO<sub>2</sub> crucible