National Aeronautics and
Space Administration

An Overview of CERMET Fuel Development

…at Marshall Space Flight Center

Marvin W. Barnes
NASA MSFC
10/11/2017

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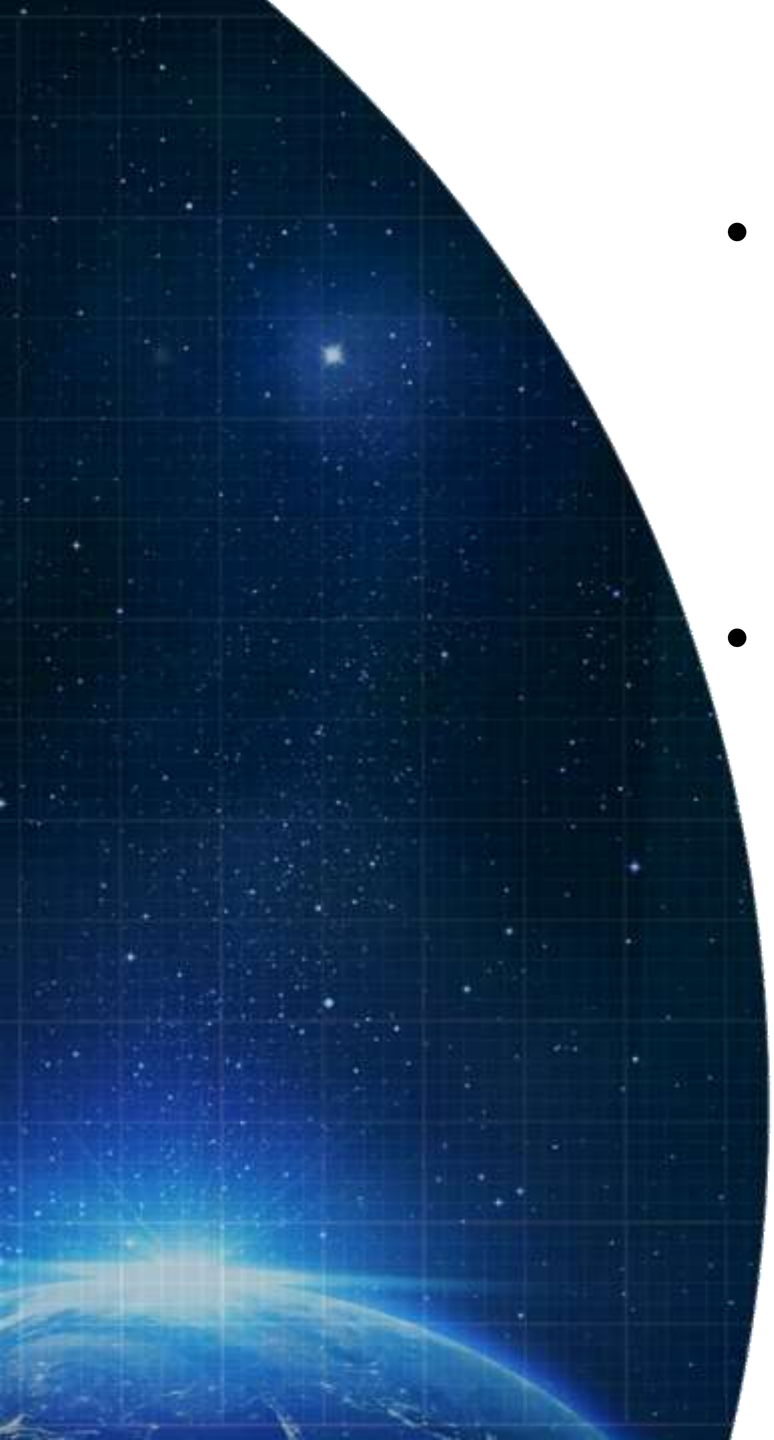
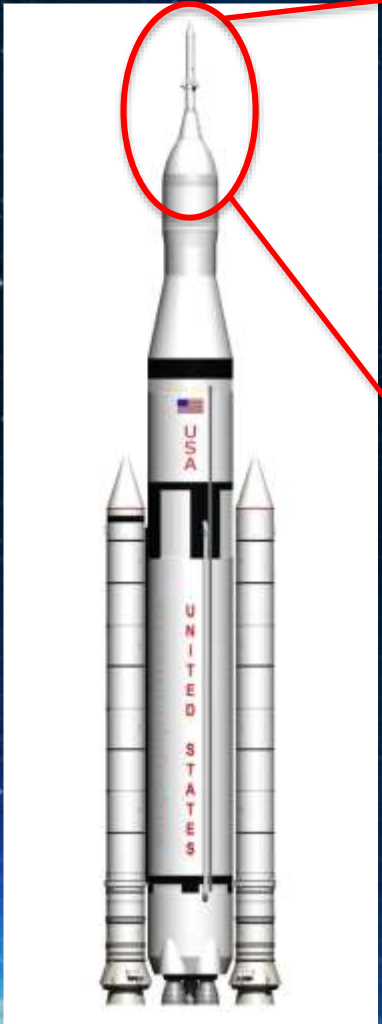


My Background

- Academic
  - BSE in Chemical and Materials Engineering
  - MS in Material Science (in progress)
- Employment
  - United Space Alliance
    - R&D Space Shuttle SRBs Thermal Protection System (TPS)
  - NASA
    - Solid Propulsion Division
      - Launch Abort System
    - Metal Joining and Processes
      - Nuclear Thermal Propulsion
      - Fuel Development

Solid Propulsion

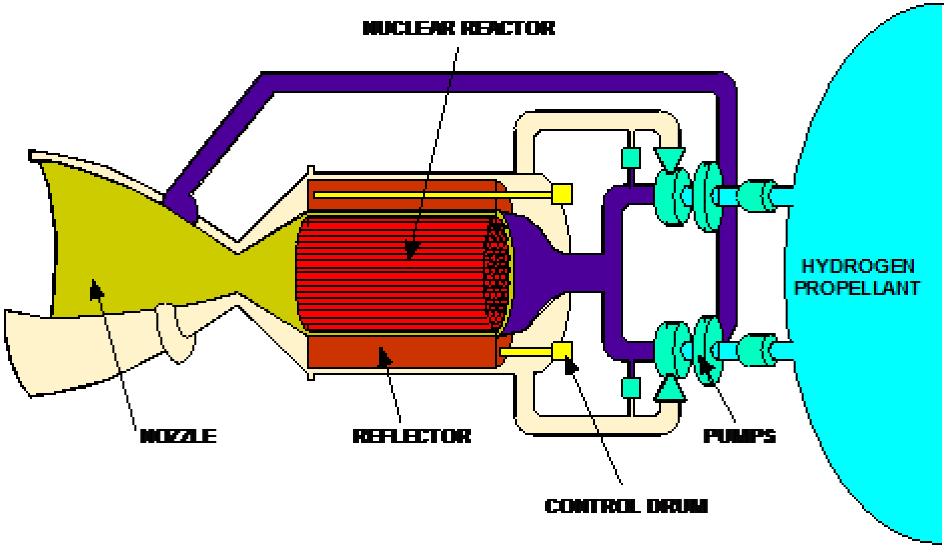


SLS

Orion and Launch Abort System

How Does Nuclear Thermal Propulsion (NTP) Work?

- Propellant heated directly by a nuclear reactor and thermally expanded/accelerated through a nozzle
- Low molecular weight propellant – typically Hydrogen
- Thrust directly related to thermal power of reactor: 100,000 N ≈ 450 MW_{th} at 900 sec
- Specific Impulse directly related to exhaust temperature: 830 - 1000 sec (2300 - 3100K)
- Specific Impulse improvement over chemical rockets due to lower molecular weight of propellant (exhaust stream of O_2/H_2 engine runs much hotter than NTP)

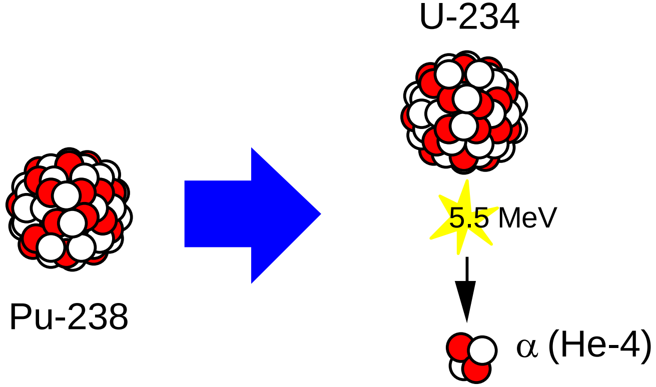


Major Elements of a Nuclear Thermal Rocket

NERVA Nuclear Thermal Rocket Prototype

Fission is Different from Previous NASA “Nuclear”

Radioisotope



Heat Energy = 0.023 MeV/nucleon (0.558 W/g Pu-238)

Natural decay rate (87.7-year half-life)

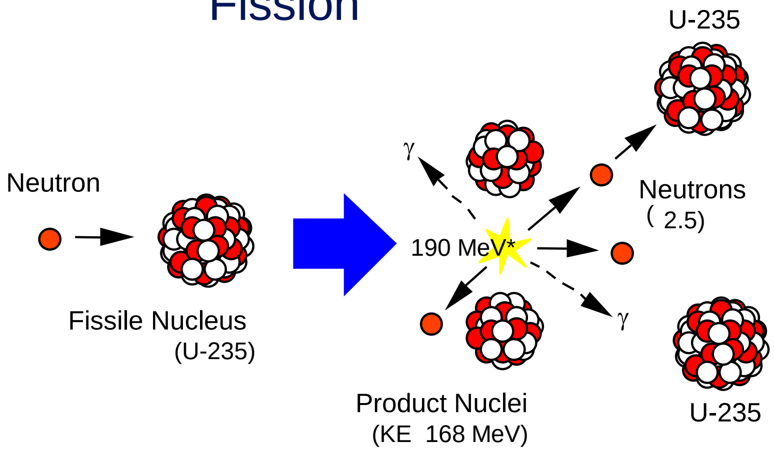
Long history of use on Apollo and space science missions

44 RTGs and hundreds of RHUs launched by U.S. during past 5 decades

Heat produced from natural alpha (a) particle decay of Plutonium (Pu-238)

Used for both thermal management and electricity production

Fission



Heat Energy = 0.851 MeV/nucleon

Controllable reaction rate (variable power levels)

Used terrestrially for over 70 years

- Fissioning 1 kg of uranium yields as much energy as burning 2,700,000 kg of coal (>20 GW-hr)

One US space reactor (SNAP-10A) flown (1965)

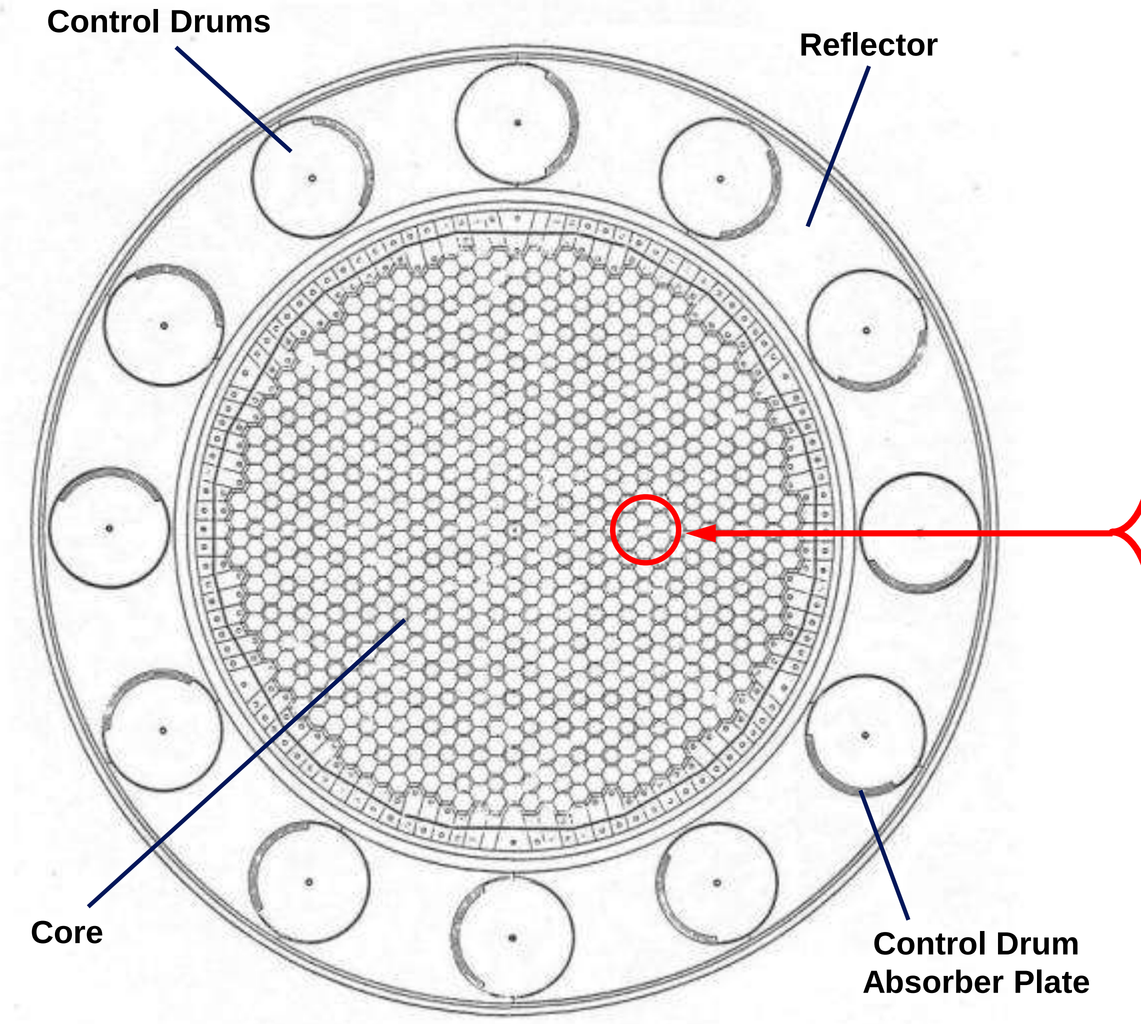
- Former U.S.S.R. flew 33 space reactors

Heat produced from neutron-induced splitting of a nucleus (e.g. U-235)

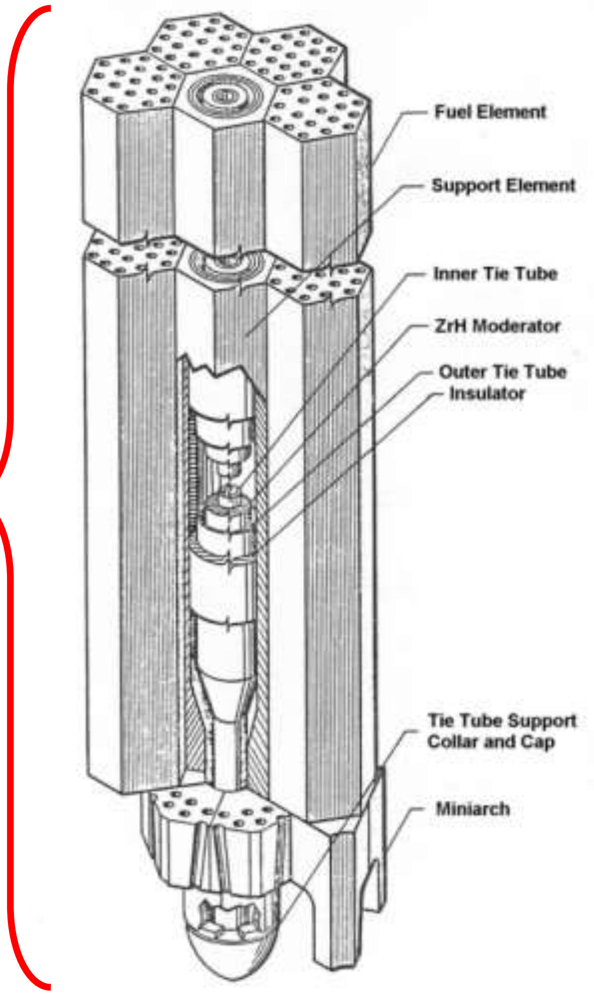
- At steady-state, 1 of the 2 to 3 neutrons released in the reaction causes a subsequent fission in a “chain reaction” process

Heat converted to electricity, or used directly to heat a propellant

Typical First Generation NTP Reactor Design



NERVA Reactor Cross Section



Fuel Segment Cluster

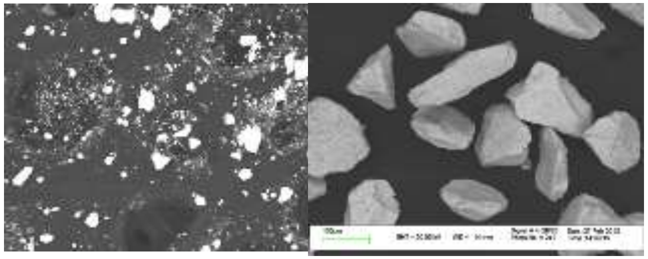
MSFC Fuels Laboratory Capabilities

- Facilities/Capabilities stand up FY12-13
  - Three new laboratories brought on line with power and exhaust system facility modifications
- All fuel fabrication laboratories are licensed by NRC for handling dU/natU
- All MSFC DU fabrication processes have been approved by the RSO and are operational
- Nuclear Regulatory Commission (NRC) performed a spot inspection of the laboratories with no findings
- MSFC is now equipped to fabricate CERMET fuels from feedstock acceptance through HIP fabrication, testing and characterization

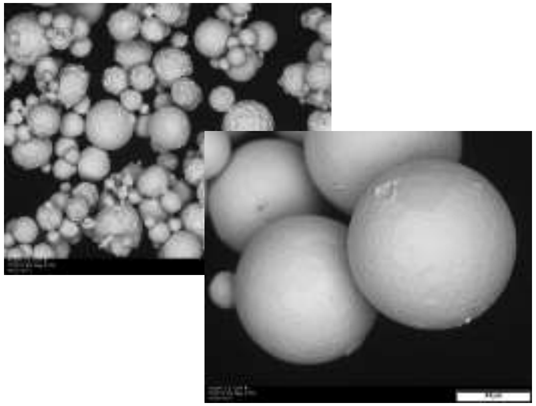
Three glove boxes are now online and operational with DU Top Right: HIP can fill GB with full length HIP can extension installed. Above Right: Powder sieving and separation GB. Bottom Right: HIP can evacuation and close out GB.

Feedstock Development

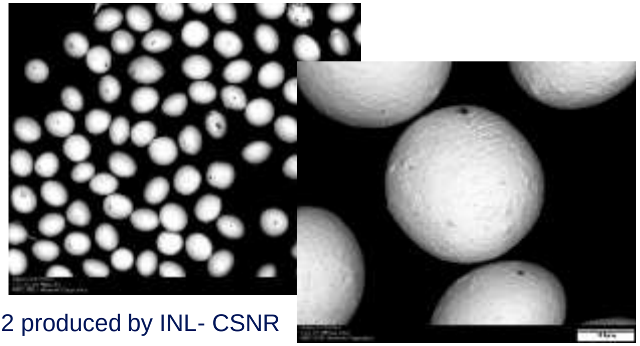
- Development of UO_2 and surrogate powders focused on particle size, shape, density and stoichiometry
- 2kg of angular UO_2 purchased from Y-12
  - Not optimal for post HIP microstructures
  - Not optimum for CVD W coating process
  - Fine particles, <5µm, clumps and does not flow well
- 3.3kg of spherical UO_2 procured from ORNL
  - Qualified Sol-Gel process for TRISO fuels
  - Required development to produce the required size, 100µm
  - Good spherocity and with a tight size distribution
- 3kg of spherical UO_2 procured from INL-CSNR
  - internal gelation being developed by INL-CSNR
  - First UO_2 powders produced for NCPS development
  - Very tight size distribution, good shape



Angular UO2 produced by Y12



Spherical UO2 produced by ORNL using Sol-gel process

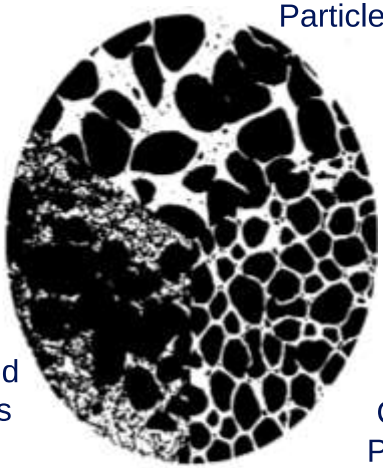


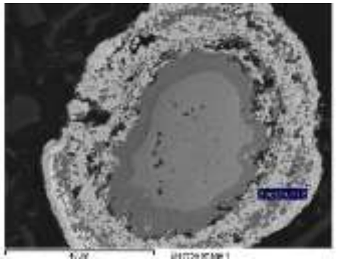
Spherical HfO2 produced by INL- CSNR gelation process

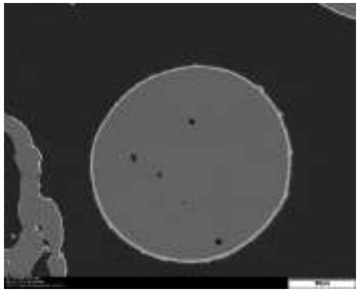
Chemical Vapor Deposition (CVD)

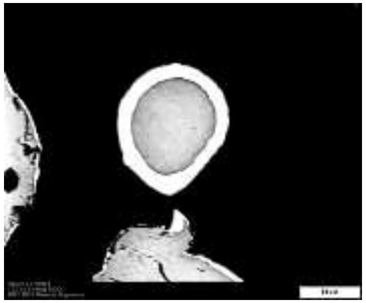
- W coated UO_2 matrix will increase the life of CERMET fuel
  - Uniform distribution of fuel throughout the matrix
  - Eliminates agglomeration and increases structural integrity of fuel
- No commercial/govt facilities doing W coated UO_2 using WCl_6
- Currently on Gen 3 of the MSFC CVD system

3rd gen MSFC CVD system

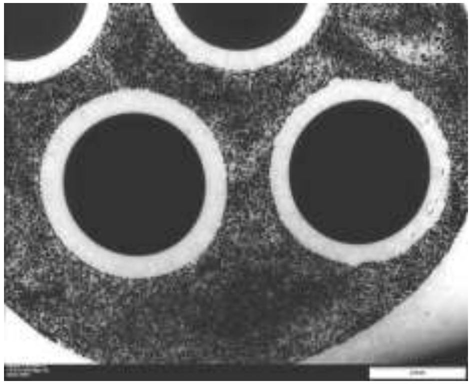








SEM image of W coated ZrO2



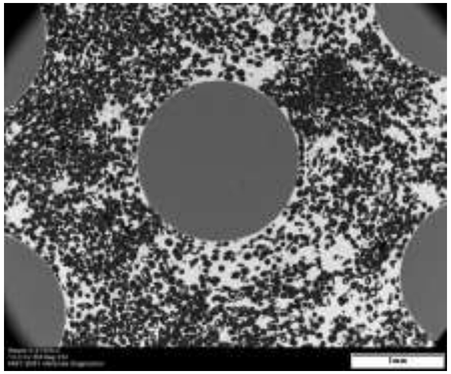
HIPed W-ZrO2 with W claddings

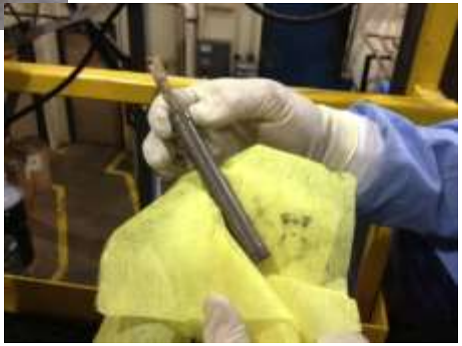
Net Shape HIP Development

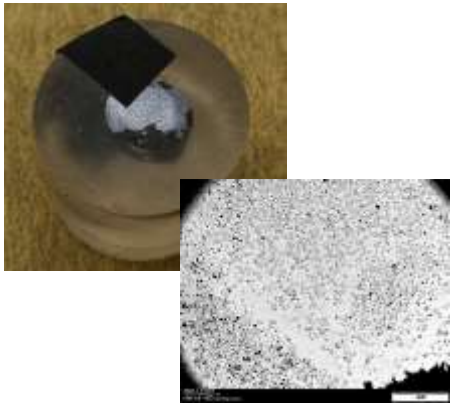
- Design and optimization of full and subscale HIP cans
- Can assembly, fill, closeout, machining and etching
- HIP cycle parameter development and HIP chamber tooling
- Equipment optimization to handle full scale HIP cans
- MSFC HIP system refurb for UO_2 HIP


HIP can Mo rod stack up prior to assy.


W-ZrO2 Post Mo etching and HIP can grinding. Sample ready for CFEET testing(Left)


SEM images of W-ZrO2 cross section


W-UO2 sample post HIP


W-UO2 CFEET sample. Agglomeration of UO2


Full scale, 61 channel HIP can failed during cycle due to embrittlement of Nb can material.

Subscale H_2 Test System- CFEET

- A system capable of testing subscale fuel elements at 3000k in flowing H_2 is required for fuel development
- Capable of multiple heating cycles per day for rapid data on fuel integrity
- Requires <100g of UO_2 versus 2.8kg for a full length (17.8” L) element- $$$$
- If the fuel cannot survive CFEET it will not survive NTREES
- Currently operating a 2nd generation CFEET system at MSFC
  - 15kw and 50kw pwr supplies available
  - Obtained 3695K with pure W sample in flowing Ar with 15kw
  - W-ZrO_2 7 channel sample reached 2338K in a 30sec shakeout test with 50kW
  - Continuing to optimize the system with 50kw and prepping for the W-UO_2 test in Feb ‘14

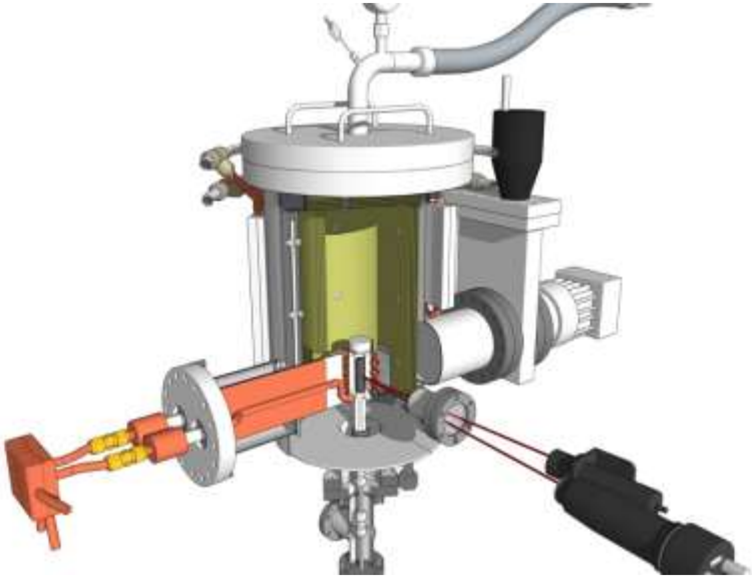
308 Stainless Steel Samples

Tungsten Rhenium Hafnium Nitride Samples

Tungsten, Graphite (L to R) tested in flowing hydrogen

CFEET

Upgraded CFEET chamber and 50 kW power supply



Cut away of CFEET Test Chamber

Above/Left: Pure W sample post shakeout run 2. Sample reached melting point (3695K) and was held in place by the BN insulator. BN insulator had to be destroyed to remove the sample

View looking down into the CFEET chamber during run 1. BN insulator and bright orange sample inside

National Aeronautics and
Space Administration

NTP CERMET Fuel Fabrication Study

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NASA Marshall Space Flight Center
[2]Center for Space Nuclear Research

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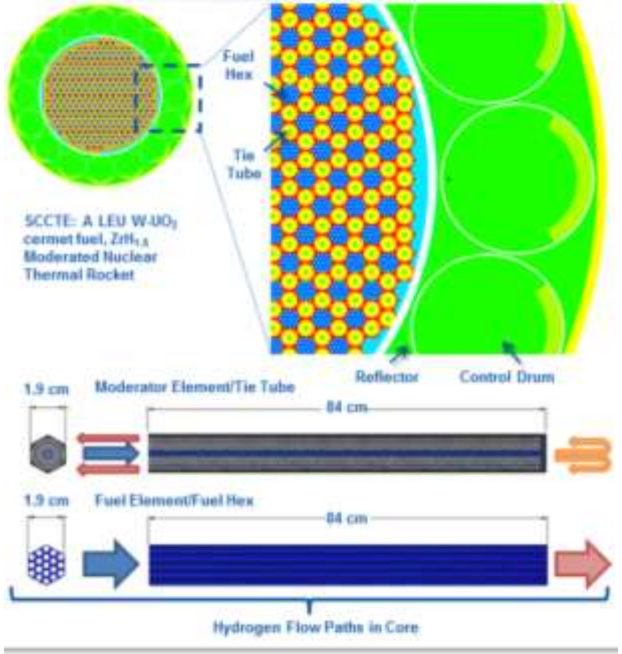
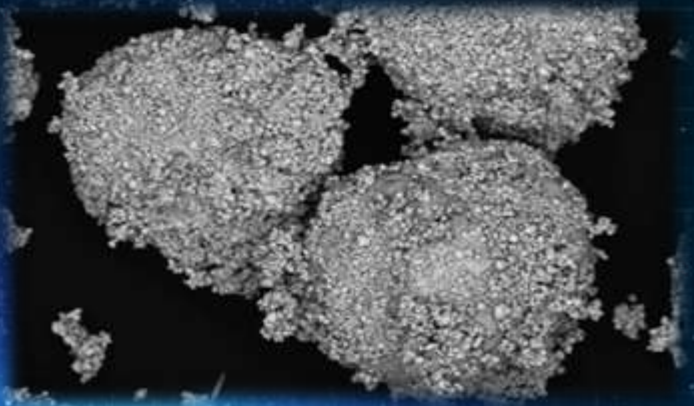
Presentation Overview

- GE710 Program
- Fuel Compact Fabrication Study
- Tungsten Powder Coating
- Spark Plasma Sintering
- Experimental Approach
- Results
- Conclusions and Future Work
- Other Fuel Development Work



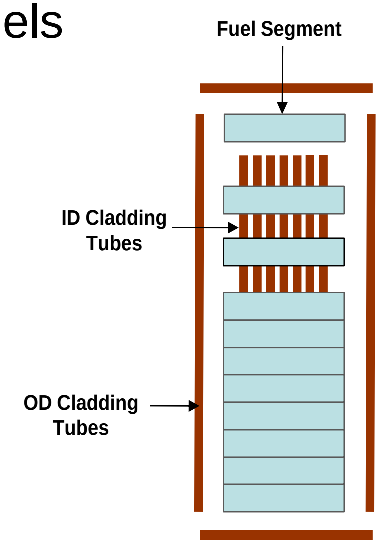
Nuclear Thermal Propulsion (FY16)

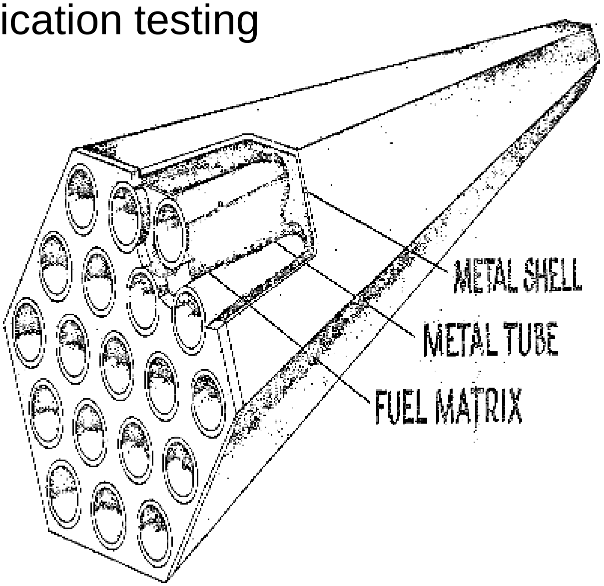
- Awarded CIF to investigate CERMET fuel development
- Innovation
  - W Powder Coating
  - Spark Plasma Sintering (SPS)


W Powder Coating

GE710 Program

- Extensive CERMET fuel development program
  - Over 15 million invested from May 1962 to Sept 1968
  - Operated fuel element fabrication line for “reactor-sized” fuel elements
  - Successfully fabricated 40+ W-60vol%UO_2 fuel elements for qual testing
    - Conducted 10 of thousands of hours of qualification testing
- 710 fabrication approach
  - Press and sinter W-UO_2 compacts
  - Machine cooling channels
  - Stack compacts
  - Weld tubes for cooling
  - Weld external cladding
- Program cancelled
  - Before qual completed

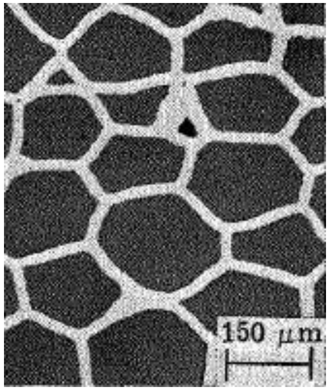


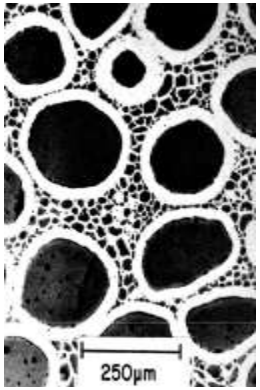


AEC Research and Development “*710 High Temperature Gas Reactor Program Summary Report*” GEMP-600; Vol I; 1969

Fuel Compact Fabrication Study

- Past efforts focused on consolidating full-length elements
  - Particle segregation/Non-uniformity of fuel particles within W matrix
- Interest in exploring 710 approach
  - Stacking and bonding fuel compacts
- Conducted compact fabrication study
  - Fabricate compacts with high density and uniformly disperse fuel particles
  - Utilizing new process and fabrication technique
    - W powder coating
    - Spark Plasma Sintering (SPS)

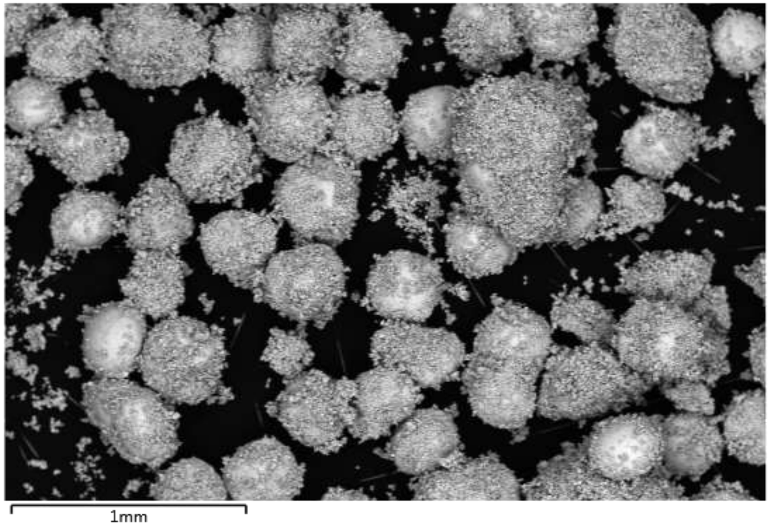


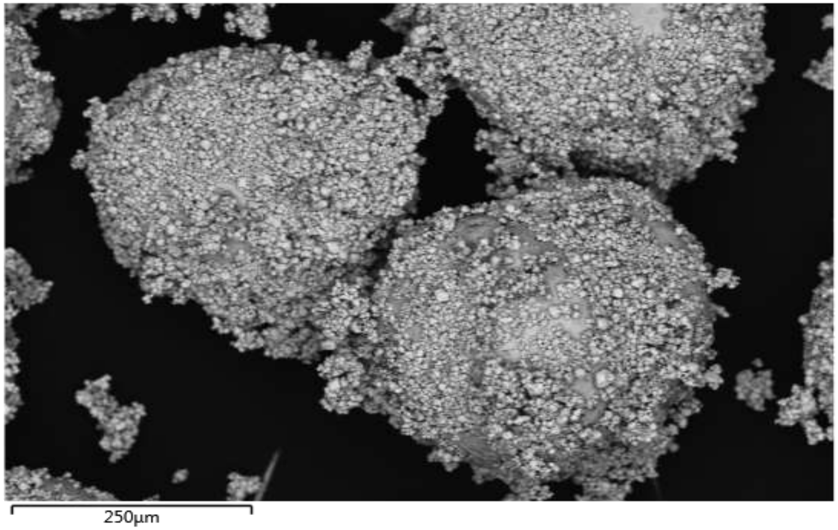


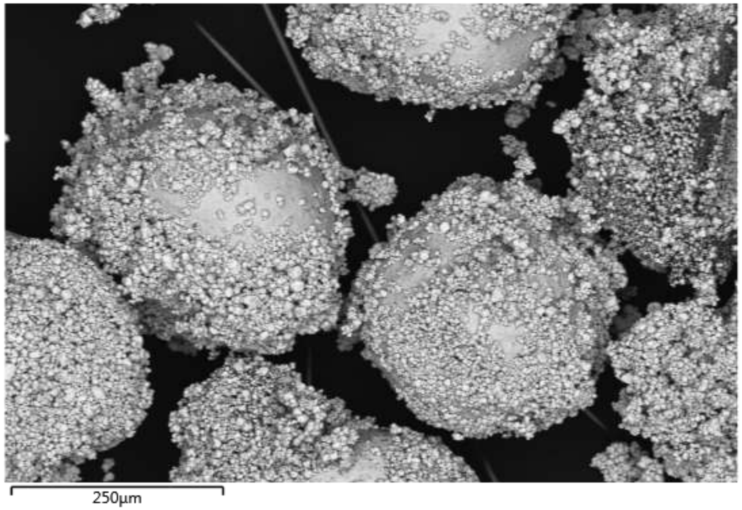
R.R. Hickman, *"Review of Past and Current W-UO2 CERMET Fuel Fabrication Development and Testing"*, National Aeronautics and Space Administration Marshall Space Flight Center, Huntsville, AL, 2014

Tungsten Powder Coating

- Straightforward approach to particle coating
- Conducted experiments with 6 different organic binders
- Coating Process
  - Blend W powder, dUO_2 particles, and binder
  - Stir mixture above binder drop point on hot plate for 5 min
- Improved fuel particle dispersion
  - Coating not as uniform as CVD coated particles



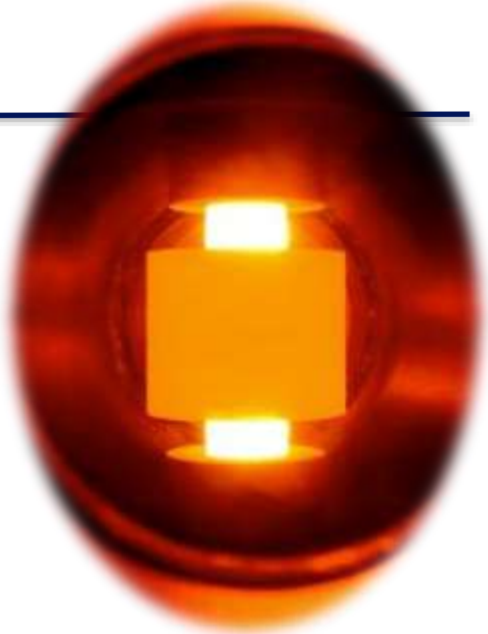


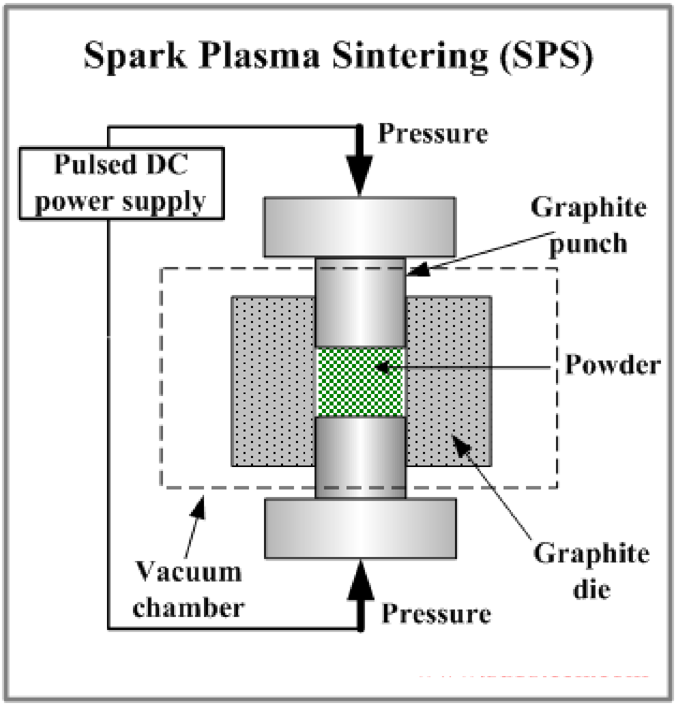


D. Tucker, A. O'Connor, R. Hickman., *"A Methodology for Producing Uniform Distribution of UO2 in a Tungsten Matrix"*, Journal of Physical Science and Application

Spark Plasma Sintering

- Simple Process
- Rapid Consolidation/Sintering
- Net-shape/Near Net- Shape Parts
- High Density Parts
- Compatible with W powder coating





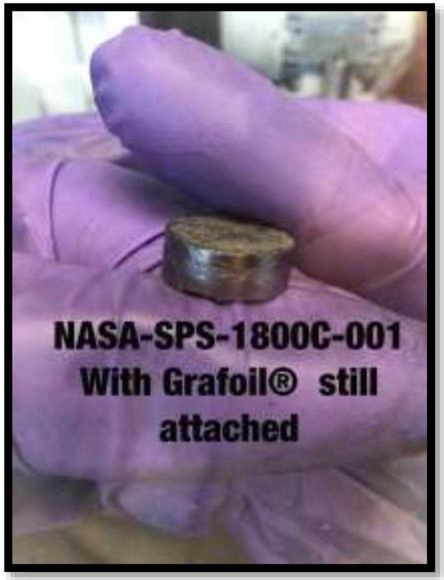
1. Pictures courtesy of UC Davis and Substech

Experimental Approach

- Utilized SPS system at CSNR to sinter W/UO_2 samples
  - Used W powder coated particles
- Sintered total of 24 samples (20 mm diameter; 6 mm thick)
  - Varied peak temperature 1600C, 1700C, 1750C, 1800C, and 1850C
  - Held constant 50Mpa axial load with varying
- 20-minute dwell time at peak temperatures
- Measured density and observed microstructure using SEM

NASA-SPS-1800C-001
Still attached to die and punches

NASA-SPS-1800C-001
With Grafoil still attached



NASA-SPS-1800C-001
With Grafoil® still attached

Results

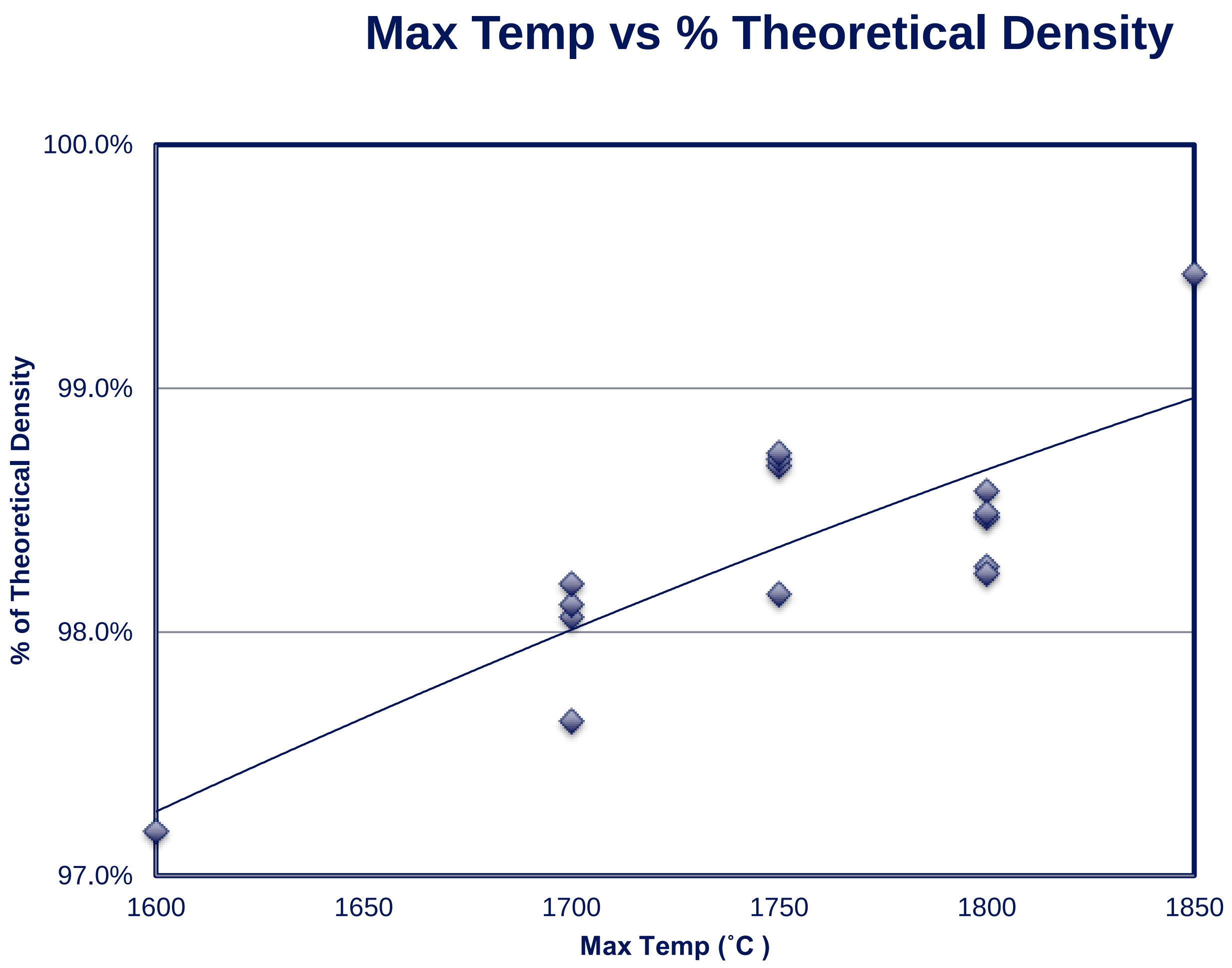
- Density
  - Increased with peak sintering temperature
  - Near theoretical density

Specimen	Thickness (mm)	Diameter (mm)	Average Density (g/cm^3)	Percent of Theoretical (%)
NASA-SPS-1850C-001	5.90	19.93	14.2	99.5
1800C-001	5.45	19.95	14.1	98.5
1800C-002	5.94	19.96	14.1	98.6
1800C-003	5.57	19.91	14.1	98.5
1800C-004	6.03	19.91	14.0	98.3
1800C-005	5.60	19.93	14.0	98.2
1750C-001	6.10	19.89	14.1	98.7
1750C-002	6.15	19.90	14.0	98.2
1750C-003	5.60	19.96	14.1	98.7
1750C-004	5.70	19.90	14.1	98.7
1700C-001	6.00	19.90	14.0	98.1
1700C-002	6.40	19.93	14.0	98.1
1700C-003	5.93	19.90	13.9	97.6
1700C-004	6.00	19.96	14.0	98.2
1600C-001	6.10	19.90	13.9	97.2

D. Tucker, M. Barnes, L. Hone, S Cook., *“High Density, Uniformly Distributed W/UO2 for use in Nuclear Thermal Propulsion“*, Journal of Nuclear Materials

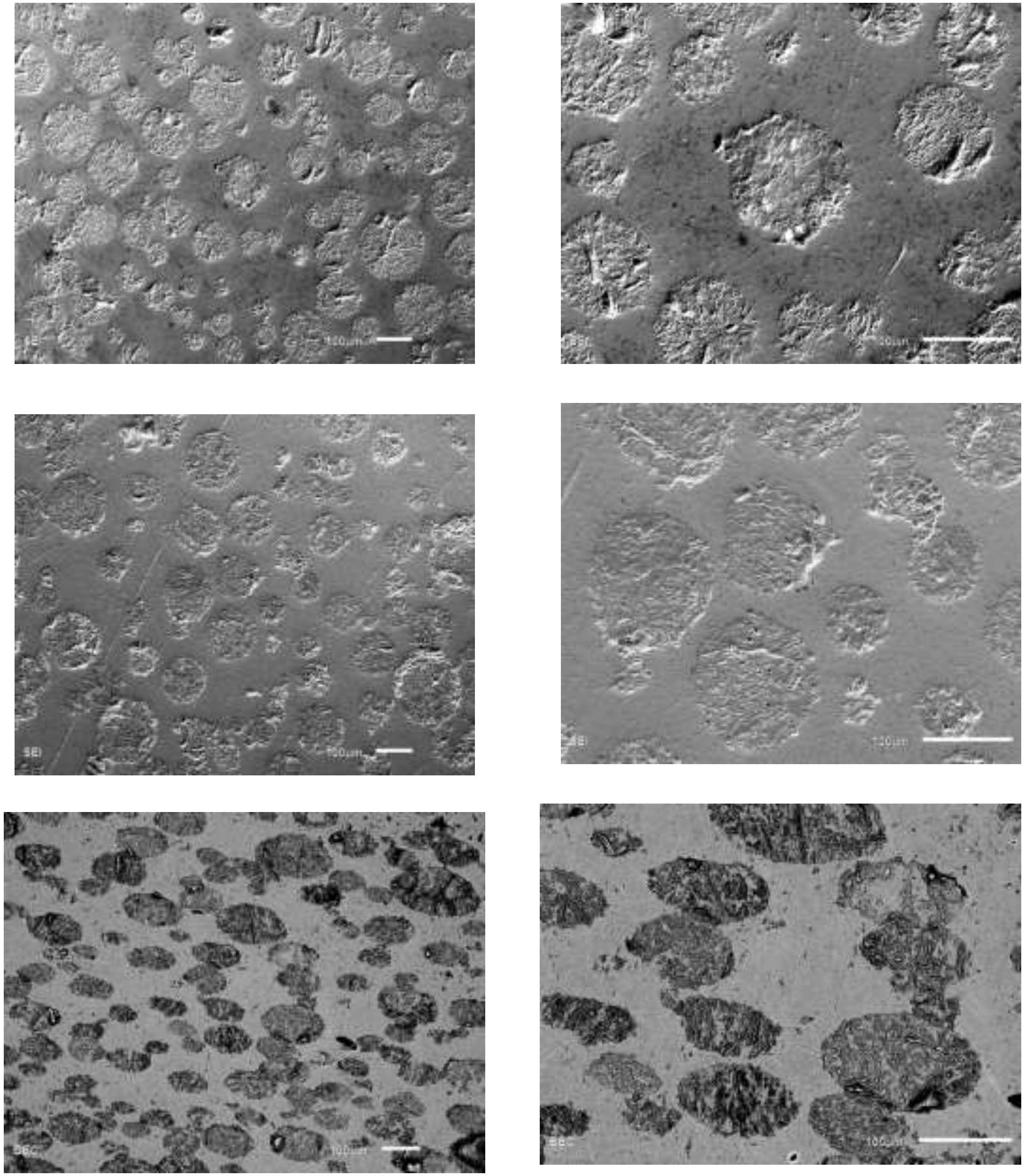
Results

- Density
  - Density can be tailored to meet material performance requirements



Results

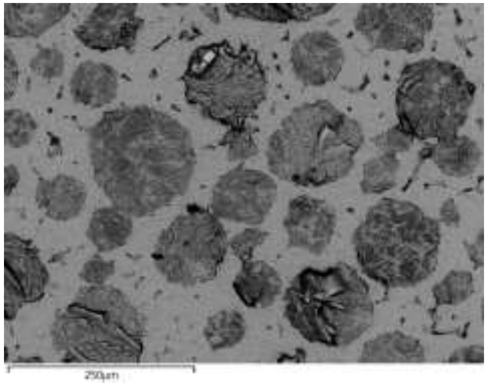
- SEM
  - Improved microstructure
  - UO_2 particles more uniformly dispersed
  - Cross-section depicts some particle elongation

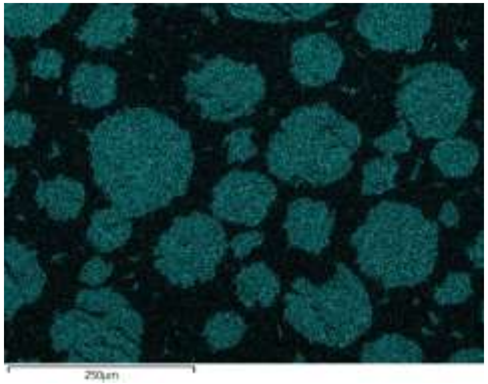


M. Barnes, D. Tucker, L. Hone, S Cook., *"Nuclear Rocket CERMET Fuel Fabrication using Tungsten Powder Coating and Spark Plasma Sintering"*, NASA Technical Paper

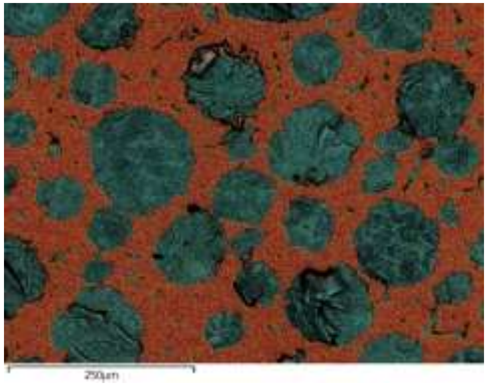
Results

- Energy-dispersive X-ray spectroscopy (EDS)
    - No unexpected phases

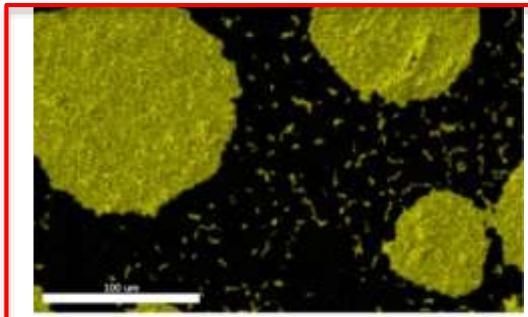




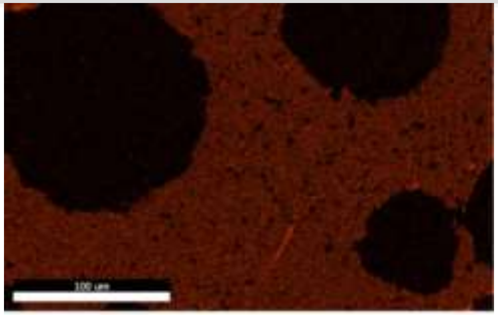
Uranium Phase



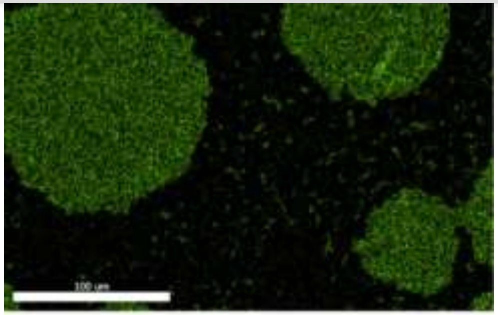
Tungsten Phase



Uranium Phase



Tungsten Phase



Oxygen Phase

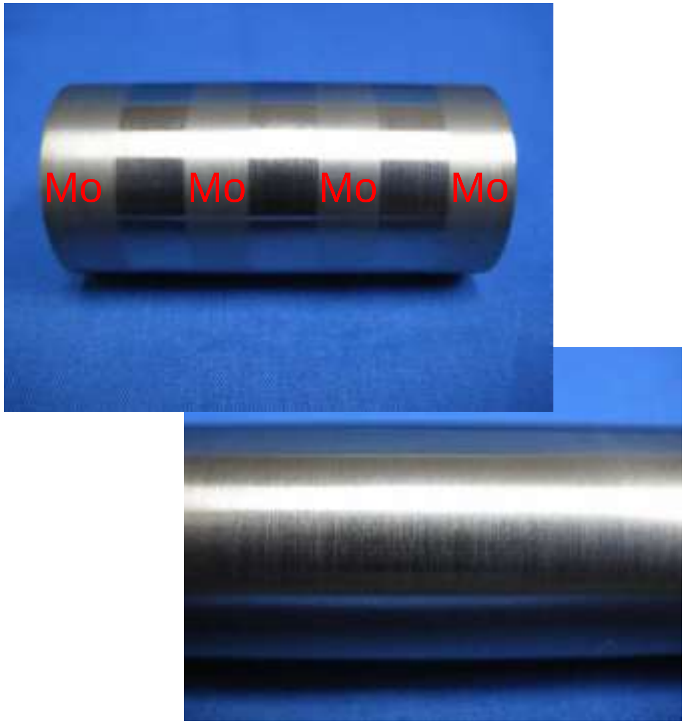
Conclusion and Future Work

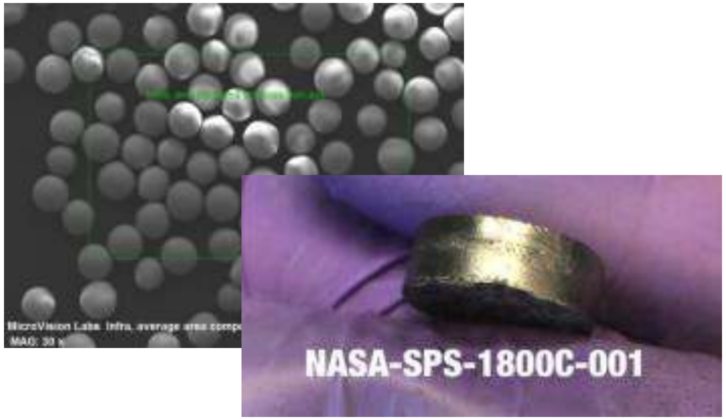
- Improved density and microstructure
- Further characterization needed and planned
  - Mechanical Properties
    - Hardness Testing
    - Tensile Testing
  - Thermal Properties
  - Analysis
    - TEM/EDS
    - Local Electrode Atom Probe (LEAP)
    - Further SE to quantify dispersion
  - Chemistry
  - CFEET testing planned

S. O'Dell "*Phase I Final Report Joining of Tungsten Cermet Nuclear Fuel*", Plasma Processes LLC, 2016

Other Fuel Development Work

- FY16 Development Efforts
  - Phase I SBIR – Bonding tungsten CERMET compacts
  - Phase I SBIR - Electrolytic method for tungsten coating





S. O'Dell *"Phase I Final Report Joining of Tungsten Cermet Nuclear Fuel"*, Plasma Processes LLC, 2016

NTP Technical Briefing and Continuation Review

Fuel Fabrication & Testing Milestone
September 26, 2017

National Aeronautics and Space Administration

Marvin W. Barnes, NASA MSFC

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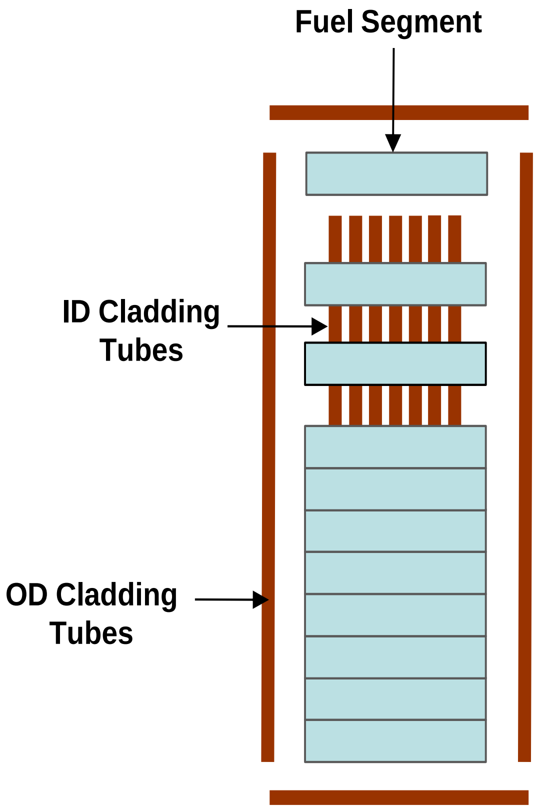
Test Specimen Fabrication Process

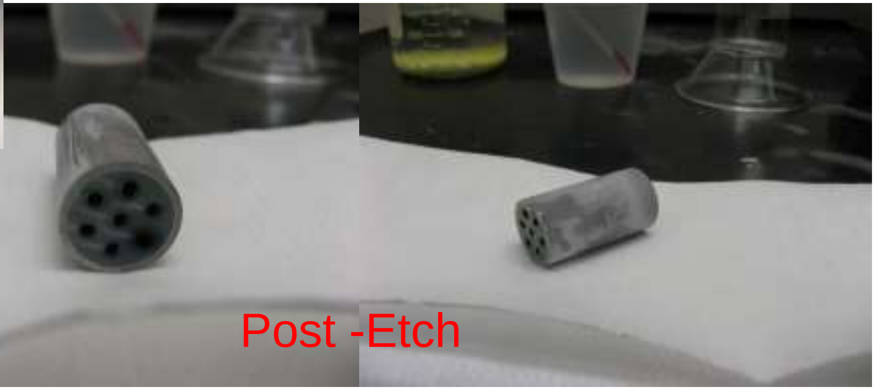
- Exploring GE710 Process
  - Machined or SPS cylindrical wafers (5/8” OD by ½” Long)
  - Machined seven 0.110” cooling channels
  - Machined cylindrical tantalum HIP enclosure
  - Stacked wafers with Mo rods & E-beam welded enclosure
  - HIP Bonded at 1800 °C and 30,000 psi
  - Chemical etch to remove Mo rods




Wafers


Post-HIP


HIP Assemblies


Post -Etch

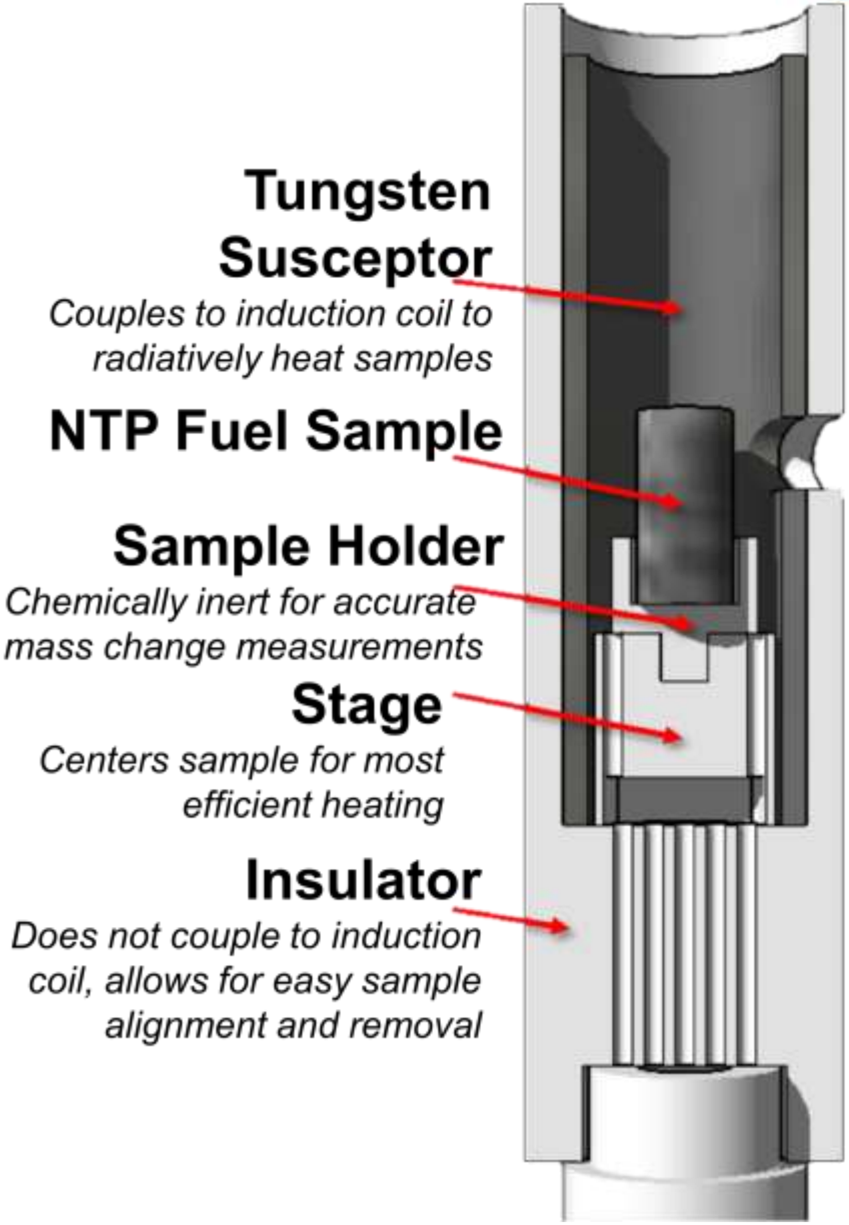
Specimen Fabrication Accomplishments

- Fabricated 12 pure tungsten wafers
- Fabricated 6 pure tungsten SPS wafers
- Fabricated 15 W/ZrO_2 SPS wafers
- Fabricated 3 stacked and HIP bond pure tungsten samples
- Developed process to form cooling channels in pure W wafers
- Identified vendor for W/ZrO_2 machining
- Developed process to form cooling channels in W/ZrO_2 wafers
- Fabricated stacked and bonded pure tungsten sample with cooling channels
- Conducted W/ZrO_2 microscopy and W powder coating optimization
- Conducted W/dUO_2 LEAP analysis at INL


NASA-SPS-1800C-001
With Grafoil® still attached

CFEET Test Preparation

- System Modifications
  - Redesigned sample loading apparatus
  - Designed, fabricated and tested tungsten susceptor
  - Optimized induction coil and power supply for operation with susceptor
  - Upgraded insulator and pedestal design



Tungsten Susceptor

Couples to induction coil to radiatively heat samples

NTP Fuel Sample

Sample Holder

Chemically inert for accurate mass change measurements

Stage

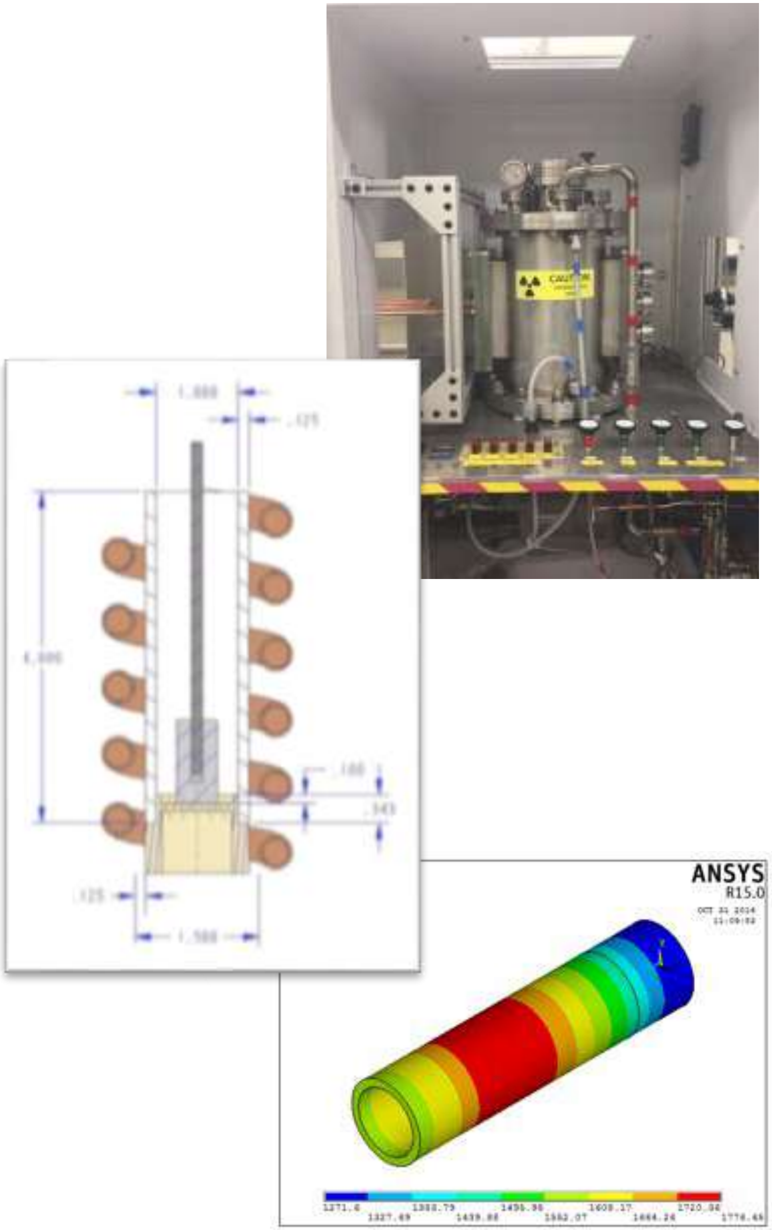
Centers sample for most efficient heating

Insulator

Does not couple to induction coil, allows for easy sample alignment and removal

CFEET Test Preparation

- Instrumentation Upgrades
  - Replaced data acquisition unit
  - Installed and verified Williamson pyrometer 650 – 3000 °C (823 – 3273 K)
  - Procured FAR pyrometer
  - Installed Basler networking camera
- Verifying Functionality
  - Developed thermal model of SiC test
  - Verified system operation above 2719 K sample temp (above 2800 K susceptor temp)
  - Mitigated chemical compatibility anomaly

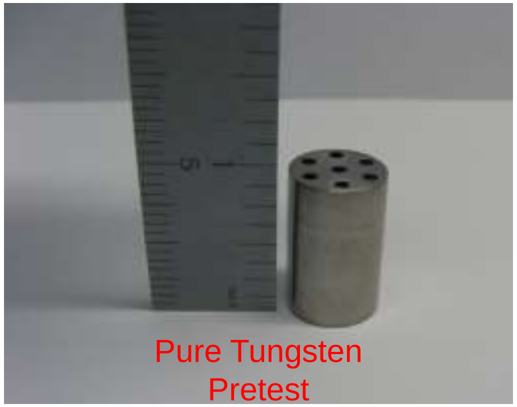


CFEET Test Preparation

- Characterized 2 induction coils
- Conducted 5 pyrometer verification tests
- Conducted 25 steady-state tests (30 minute hold at peak temp)
- Tested various materials
  - Refractory Carbides
    - SiC, ZrC, NbC, TaC, Tricarbides
  - Refractory Metals
    - W, Nb, Hf, Zr
  - Refractory Oxides
    - ZrO_2, HfO_2
- Conducted 5 W/ZrO_2 tests
- Conducted 2 tricarbide tests
- Conducted sintering trials in CFEET
- Optimized transformer ratio and capacitance
- 40+ tests conducted

Pure Tungsten (W) Specimen

- Test Date: Aug 28, 2017
- Test Description:
  - Exposed material to simulated environment (elevated temperature and pure hydrogen) in the Compact Fuel Element Environmental Test (CFEET) system using the following parameters:
    - Hold Temperature: 2500K (24% power level)
    - Hold Time: 45 minutes
    - Hydrogen Flow Rate: 5 SLPM

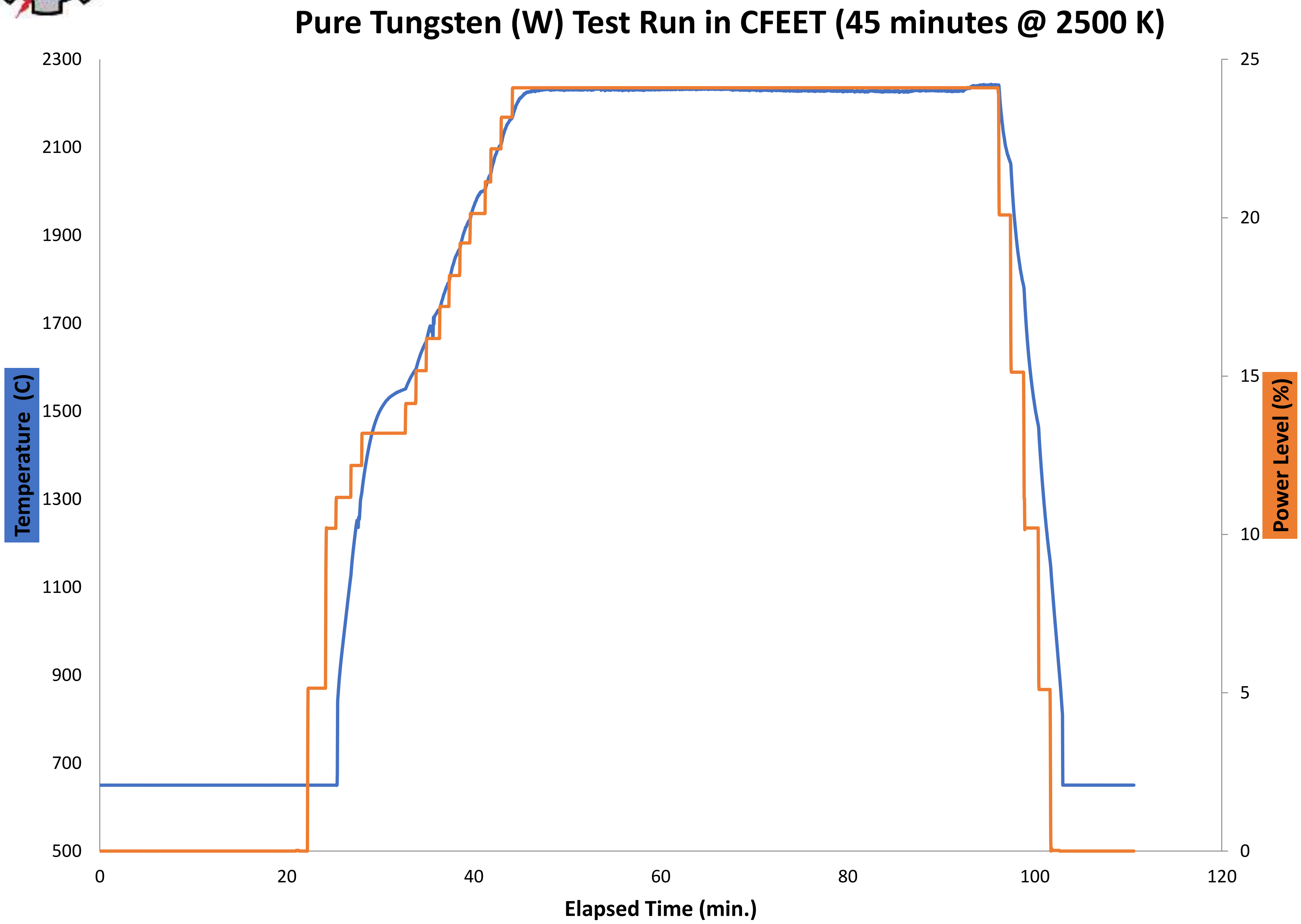


Pure Tungsten
Pretest

Pure Tungsten
During

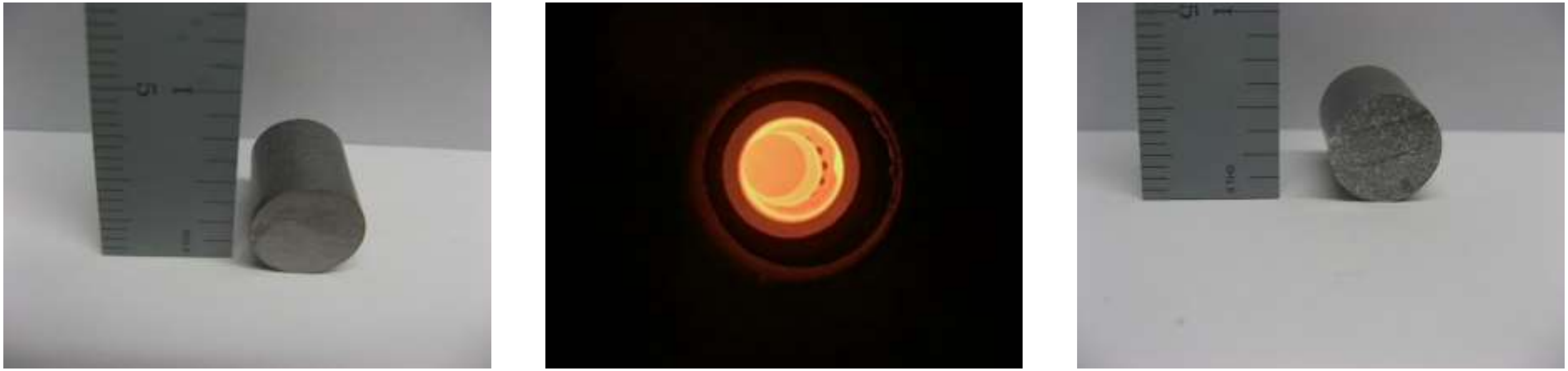
Pure Tungsten
Post-Test

Temperature and Power Profile



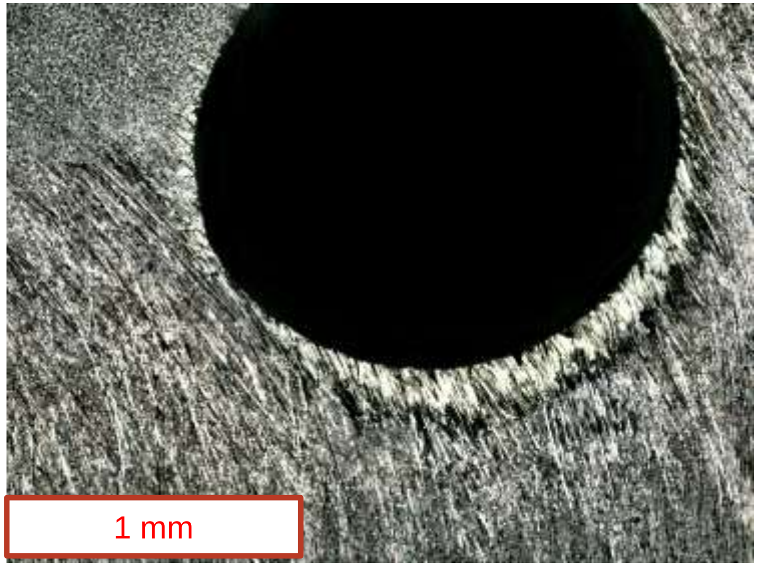
Tungsten Zirconia CERMET Surrogate Fuel Specimen

- Test Date: Aug 30, 2017
- Test Description
  - Exposed material to simulated environment (elevated temperature and pure hydrogen) in the Compact Fuel Element Environmental Test (CFEET) system using the following parameters:
    - Hold Temperature: 2500K (24% power level)
    - Hold Time: 45 minutes
    - Hydrogen Flow Rate: 5 SLPM

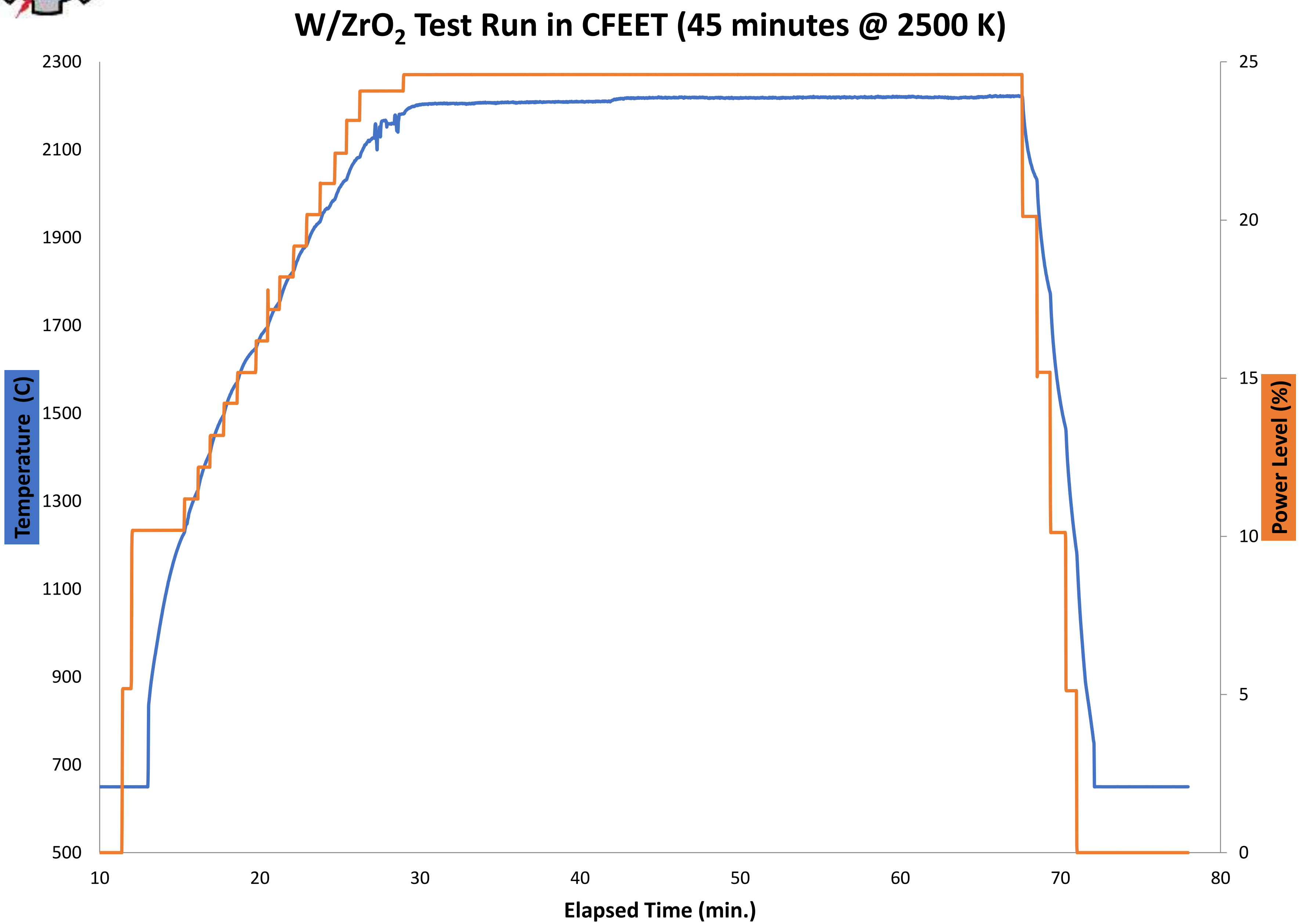


CFEET Test Results

- Results for Pure Tungsten Specimen:
  - No macroscopic or microscopic degradation noted
  - No macroscopic or microscopic wafer debonding observed
  - No significant change in mass (less than 0.04%)
  - Increase in luster/sheen of surface due to hydrogen "cleaning" effect
- Future Work for Specimen
  - Scanning Electron Microscopy (SEM)

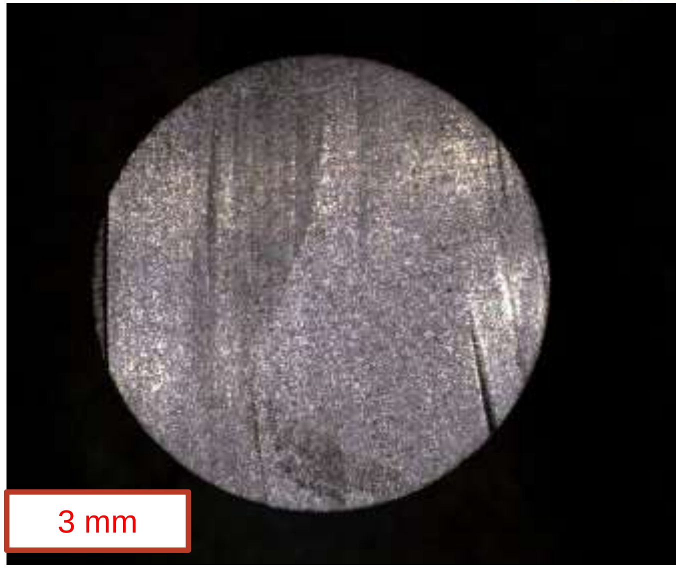


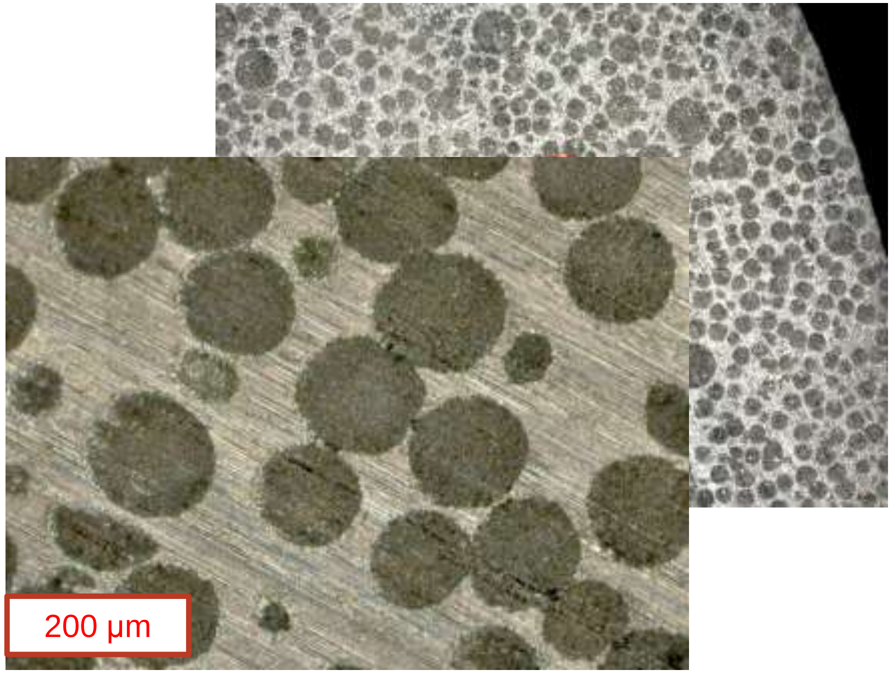
Temperature and Power Profile



CFEET Test Results

- Results for Tungsten Zirconia:
  - Negligible degradation (some reduction of zirconia may have occurred, further analysis required)
  - No macroscopic wafer debonding observed
  - Minimal mass loss (0.10 g or 0.27%)
  - Increase in luster/sheen of surface due to hydrogen “cleaning” effect
  - No significant microscopy changes
- Future Work for Specimen
  - Scanning Electron Microscopy (SEM)





Conclusions

- Materials and processes show promise, but further development is required
- Additional development needed to assess cladding formation and integrity
- Additional development work to assess bondline integrity
- Additional research needed to develop OD cladding materials
- Further testing need to fully characterize material performance with depleted uranium (microstructure, chemistry, mass loss, etc.)