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ORNL/LTR-2012/383

September 7, 2012

Mr. Ken Sorenson
Sandia National Laboratories
P.O. Box 5800
Albuquerque, New Mexico 87185-0747

Dear Ken:

**Completion of the ORNL Fuel Cycle Research and Development (FCR&D) Level 4 Milestone – ST R&D Investigations-ORNL FT-12OR080303, MS# M4FT-12OR0803034, "End of Year Status Report for R&D Investigations," due 9/7/2012**

This letter documents the completion of the FCR&D Level 4 milestone for the ST R&D Investigations - ORNL work package (FT-12OR080303), "End of Year Status Report for R&D Investigations" (M4FT-12OR0803034), due September 7, 2012. A letter report describing the activities of ORNL personnel on the R&D Investigations Team is attached to this memo.

If you have any questions, please contact me at (865) 241-5750, Bob Jubin at (865) 574-4934, or John Wagner at (865) 241-3570.

Sincerely,

Robert L. Howard
Reactor and Nuclear Systems Division

RLH:dw

Enclosure

c: C. V. Bates (INL)
B. D Hanson (PNNL)
R. T. Jubin
M. C. Vance
J. C. Wagner

**ORNL/LTR-2012/383**

**LETTER REPORT**

Reactor and Nuclear Systems Division

**Project Title:** ST R&D Investigations – ORNL FT-12OR080303

**Subject of Document:** FY 2012 End of Year Status Report – ORNL Activities Supporting Storage and Transportation R&D Investigations Task within the DOE-NE Fuel Cycle Technologies Used Fuel Disposition Campaign

**Type of Document:** End of Year Status Report for R&D Investigations

**Authors:** R. L. Howard

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**FY 2012 End of Year Status Report**
**ORNL Activities Supporting Storage and Transportation R&D Investigations Task within the DOE-NE Fuel Cycle Technologies Used Fuel Disposition Campaign**

## 1. INTRODUCTION

The R&D Investigations task is a multi-year effort investigating a range of issues pertaining to the development of the required data to establish the technical basis for extended long-term storage and transportation (ST) of used nuclear fuel. The FY 2012 objectives for this work included

- refining the technical data gap analysis performed in FY 2011 and developing an approach for prioritizing the gaps,
- identifying methodologies and approaches for closing the gaps, and
- coordination with the ST Engineered Materials Experimental Task area to properly allocate and implement technical data gap closure efforts.

Oak Ridge National Laboratory (ORNL) staff supported all of these efforts.

## 2. SUPPORTING THE DEVELOPMENT OF THE *USED NUCLEAR FUEL GAP PRIORITIZATION REPORT*

The first step in establishing the technical bases for extended storage and subsequent transportation of used nuclear fuel was to determine the technical data gaps that need to be addressed. The *Gap Analysis to Support Extended Storage of Used Nuclear Fuel* (DOE, 2012) was prepared to document the methodology for determining the data gaps and to assign an initial priority (low, medium, high) of importance for additional research and development to close these gaps. The analysis considered only normal conditions of extended storage. The primary purpose of the follow-on *Used Nuclear Fuel Gap Prioritization Report* (still in development) is to document the methodology and results of a more quantitative analysis used to prioritize the medium- and high-priority data gaps from the initial Gap Analysis Report (DOE 2012). One additional data gap, stress profiles, is included in the Gap Prioritization Report to address the gaps associated with transportation and the design-basis phenomena and accident conditions during extended storage.

ORNL staff met with the *Used Nuclear Fuel Gap Prioritization Report* development team on February 15, 2012, to discuss various quantitative and semiquantitative approaches to prioritizing the technical data gaps. Subsequent to this meeting, ORNL staff provided input on several iterations of the draft ranking criteria for data gap prioritization. Ultimately the ranking criteria were used in the development of the *Used Nuclear Fuel Gap Prioritization Report*. ORNL staff who are not part of the development effort participated in the peer review process of this report in April 2012.

## 3. PROVIDING RECOMMENDATIONS ON TESTS AND MODELING TO CLOSE TECHNICAL GAPS

A number of ORNL staff attended the Used Fuel Disposition Campaign Cladding Test Plan Workshop conducted in Las Vegas, Nevada, November 15–17, 2011. The purpose of the workshop was to discuss the latest in cladding data and models and develop an integrated test and modeling plan using a goal-oriented, science-based approach to predict cladding behavior over extended storage and subsequent transportation. ORNL staff gave presentations on the following topics:

- Clad integrity modeling using the AMP nuclear fuel performance code (Attachment 1),
- Expanded plug test approach for evaluating clad material properties (Attachment 2),
- Approaches to hydriding unirradiated cladding for subsequent testing (Attachment 3),
- Approach to developing surrogate clad materials using the High Flux Isotope Reactor (Attachment 4), and
- Capabilities and limitations of the High Flux Isotope Reactor and ORNL (Attachment 5).

Copies of the presentations are included as attachments to this status report.

## 4. COORDINATION WITH THE ST ENGINEERED MATERIALS EXPERIMENTAL TASK

Efforts in the ST R&D Opportunities task area are being coordinated with the ST Engineered Materials Experimental Task area in order to properly allocate and implement technical data gap closure efforts. In FY 2012, ORNL focused primarily on the development of processes to create surrogate material to simulate high-burnup Zircaloy cladding.

Three capsules containing hydrogen-charged Zircaloy-4 cladding material have been placed in the High Flux Isotope Reactor (HFIR). Irradiation of the capsules began in HFIR Cycle 440B on March 26, 2012. Two of the capsules contain three 1-in. hydrided Zircaloy-4 samples, and one capsule contains a single 6-in. (15.24 cm) hydrided Zircaloy-4 sample. The 1-in. samples included in the initial HFIR insertion have been removed after one and three HFIR cycles of irradiation and are awaiting shipment to the Irradiated Materials Laboratory for post-irradiation examination (PIE) metallography. The 6-in. cladding sample will be irradiated for approximately 11 HFIR cycles to accumulate a fast neutron fluence to match the end state neutron fluence of the reference high-burnup cladding material discharged from the H.B. Robinson reactor. The preliminary development work was documented in *Neutron Irradiation of Hydrided Cladding Material in HFIR—Summary of Initial Activities*, FCRD-UFD-2012-000080 (Howard et al., 2012) and was submitted as a Level 2 milestone on March 30, 2012.

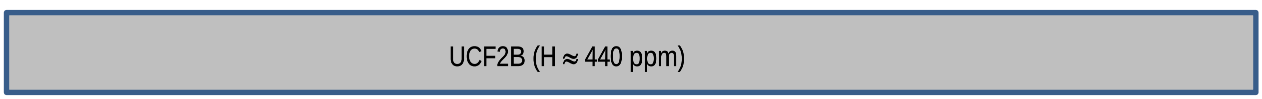
ORNL staff have been working with their colleagues at Sandia National Laboratories (SNL) and Pacific Northwest National Laboratory to identify additional material for possible irradiation. SNL staff provided ORNL with one 20-in. piece of Zircaloy-4 and one 20-in. piece of Zirlo that were excess from another project in July 2012. ORNL has begun the process of hydriding these materials and has started fabrication of additional target capsules for insertion into HFIR in October or November of 2012. Preliminary information on the hydrided cladding slated for the fourth, fifth, and sixth capsules is provided below.

## $4^{th}$ Assembly


| Sample ID | Materials | Sample Length (mm) | Sample Weight (g) | Hydrogen Content (ppm) | Comments |
|---|---|---|---|---|---|
| LRR4A7 | Zry-4 | 25.57 | 2.699 | < 20 | As-received* |
| LRR2C1C | Zry-4 | 25.80 | 2.718 | ≈ 110 | ± 40 ppm |
| LRR6F3 | Zry-4 | 26.10 | 2.750 | ≈ 280 | ±50 ppm |
| LRR6B7 | Zry-4 | 77.17 | 8.050 | ≈ 50 | ±30 ppm |

* The irradiated as-received sample LRR4A7 can serve as a baseline.

## $5^{th}$ Assembly




| Sample ID | Materials | Weight (g) | Sample Length | Hydrogen Content (ppm) | Comments |
|---|---|---|---|---|---|
| UFC2B | Zry-4 | 15.9129 | ≈ 6” | ≈440 | Hydrided to 440 ppm |

6th Assembly

| | | | |
|---|---|---|---|
| ZIRLO#1C | ZIRLO#1B3 | ZIRLO#1B5 | LRR7F7 |

| Sample ID | Materials | Sample Weight (g) | Sample Length (inch) | Hydrogen Content (ppm) | Comments |
|---|---|---|---|---|---|
| ZIRLO#1C | ZIRLO | 2.6977 | ≈1” | ≈12 | As-received* |
| ZIRLO#1B3 | ZIRLO | 2.7446 | ≈1” | ≈ 466 | ± 40 ppm |
| ZIRLO#1B5 | ZIRLO | 2.7273 | ≈1” | ≈ 450 | ±50 ppm |
| LRR7F7 | Zry-4 | 8.2510 | ≈3-1/16” | ≈ 500 | ±50 ppm |

*The irradiated as-received sample ZIRLO#1C can serve as a baseline.

In addition, ORNL staff are in the process of writing a Standard Operator Guideline, “Metallographic Preparation and Examination of Cladding Specimens Irradiated in the HFIR,” for use in the post-irradiation examination of the surrogate high-burnup material. A copy of the draft operator guideline is included as Attachment 6.

## 5. REFERENCES

DOE. 2012. *Gap Analysis to Support Extended Storage of Used Nuclear Fuel*, FCRD-USED-2011-000136 Rev. 0, PNNL-20509, prepared for the U.S. Department of Energy Used Fuel Disposition Campaign, Washington, D.C.,

R. C. Howard, R. L. Howard, J. L. McDuffee, L. J. Ott, and Y. Yan. 2012. *Neutron Irradiation of Hydrided Cladding Material in HFIR—Summary of Initial Activities*, FCRD-UFD-2012-000080, Oak Ridge National Laboratory, Oak Ridge, Tenn., March 2012.

**ORNL/LTR-2012/383**

**FY 2012 End of Year Status Report**
**ORNL Activities Supporting Storage and Transportation R&D Investigations Task within the DOE-NE Fuel Cycle Technologies Used Fuel Disposition Campaign**

## ATTACHMENTS

1. K. Clarno and G. Hansen, "Clad Integrity Modeling Using the AMP Nuclear Fuel Performance Code"

2. B. B. Bevard, R. Battiste, J. Hemrick, and W. McAfee, "Development of an Expanding Plug Test Method for Determining Hoop Stress-Strain Curves of Irradiated Nuclear Fuel Cladding"

3. Y. Yan, J. Kiggans, and C. Davisson, "Hydrogen Charging Techniques Being Developed at ORNL"

4. R. L. Howard, Y. Yan, L. J. Ott, and J. L. McDuffee, "Neutron Irradiation of Hydrided Cladding Material in the High Flux Isotope Reactor"

5. L. J. Ott and J. L. McDuffee, "Material Irradiation in HFIR and Conceptual Design for High Burnup Cladding Simulation"

6. Draft - Standard Operator Guideline, "Metallographic Preparation and Examination of Cladding Specimens Irradiated in the HFIR"

ATTACHMENT 1

U.S. DEPARTMENT OF ENERGY | Nuclear Energy

# Clad Integrity Modeling using the AMP Nuclear Fuel Performance code

***Kevin Clarno and Glen Hansen***

***clarnokt@ornl.gov; gahanse@sandia.gov***

# Why are we here?

- We are funded through NEAMS to supplement UFD
  - Engineering Analysis (EA) will leverage existing, established tools
  - NEAMS will be focused on extending new tools to address other UFD issues
    - UFD is a new focus that has just begun
  - AMP will be focused on modeling of integral effects related to VLTS and subsequent transportation
    - Extending and applying a code that NEAMS developed for fuel modeling to UFD applications
  - Other NEAMS codes may contribute in the future
    - MOOSE-Bison (last presentation), Veena Tikare (next presentation), Blas Uberuaga (yesterday)

- Our goals at this meeting:
  - Understand the experiments that will produce models we will be using
  - Understand the experiments that will be used to validate our code(s)
  - Define the questions we'll seek to assist with answering in FY12
  - Seek advice to ensure we are on the right track

- Outline
  - What is AMP and why were we invited?
  - What is our focus and what questions will we be addressing?
  - What specifically will we be doing this FY?
  - How does this meld with the experimental program?

# What is AMP?

- The AMP Nuclear Fuel Performance code is
  - An integrated nuclear fuel performance code for modeling effects beyond a single-pin
  - Parallel, fully-coupled thermo-mechanics that leverages SCALE for neutronics
- Physics modeled:
  - Neutronics
    - Depletion/decay – ORIGEN-S from SCALE
    - Radiation transport – Denovo (SCALE) radiation transport for (neutron/gamma) flux and power distribution
    - Power – From decay heat, radiation transport, or user-defined function
  - Heat transfer
    - Conduction – nonlinear FEM in 3D for fuel, clad, and structures
    - Gap conductance – radial model that replicates FRAPCON model
    - Convection – through 1D axial coolant flow
  - Mechanics
    - Elastic-plastic – nonlinear FEM in 3D for fuel and clad with contact and large deformation
    - Traditional Models – thermal expansion, stress-induced creep, damage
    - Fuel-Specific Models – relocation, densification, swelling, irradiation-induced growth
  - Single-Phase Flow
    - One-dimensional – in a fully-coupled sense
    - Three-dimensional – prototyping operator to understand coupling with CFD
  - Thermo-chemistry
    - Equilibrium solver – open source and integrated with neutronics
- Validation
  - Initial fuel validation for $UO_2$ in LWR (IFA-432); MOX and high-burnup $UO_2$ in LWR (IFA-597) in progress
  - ORIGEN-S is well validated for many reactor/fuel types
  - Radiation transport has been benchmarked on a suite of criticality safety and reactor problems

# What is the basis for the work plan?



- **FCRD UFD Gap Analysis[1] associated with VLTS**
  - **Issues associated with transportation were not included in this gap analysis**
  - **Regarding cladding creep, it concludes that:**
    - *"There are multiple mechanisms for cladding creep"*
    - *"No data or analyses on the effects of [very] long-term storage on the subsequent transportability of the fuel exist"*

- **2007 EPRI Synthesis Report[2]**
  - **Identifies the most significant in-reactor and during storage issues to transportation accidents**
    - Covers eight EPRI reports on dry storage and transport.
    - Focused on high burnup fuel with a long-term (not very long-term?) storage
  - **Relevant conclusions:**
    - "Dry storage effects on cladding physical and material conditions, including creep-related deformations, have the potential to impact fuel rod performance during transport" pp. vi
    - "*radial hydride concentration and the change in the fuel-cladding gap constitute the most important effects of dry storage on transportation*" pp. 2-9
  - **Establishes that:**
    - *"The failure frequency in the [transverse tearing mode]/[partial rod breakage mode] failure geometry… was estimated to be 50%, but no fuel reconfiguration is predicted."*
    - *"stresses at or just below the yield strength of the material are calculated. This implies that damage initiation can be expected, but progression of the damage to form a pinhole failure can be ruled out"*
    - *"The guide tubes, …, are predicted to ovalize but not fracture or totally collapse, which preserves the structural integrity of the assemblies in a non-reconfigured state."* pp. 6-2

- **This is the basis for the development of our work plan for this FY**
  - **Incorporating model for radial hydride formation and effect on mechanical state at EOS, with uncertainty estimates**
  - **Providing best estimate with uncertainty of the EOS mechanical state of cladding, for a given BOS fuel state**
  - **Evaluating the sensitivity of the EOS mechanical state of cladding due to uncertainty in the BOS fuel state**
    - **EOS = End of dry Storage; BOS = Beginning of dry Storage (after drying)**

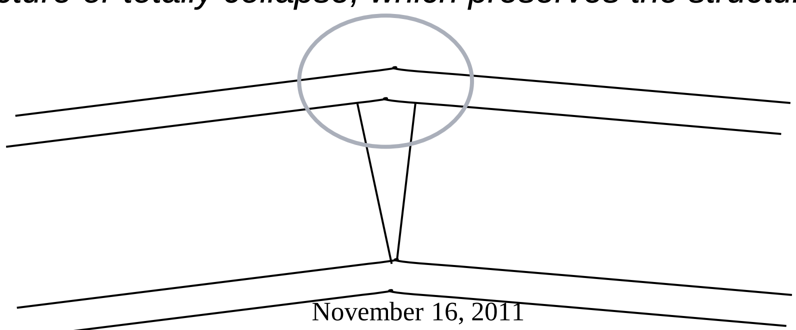
[1]*Storage – Research and Development Opportunities Work Package – Year End Report,* Fuel Cycle R&D, FCRD-Used-2010-000228 (Draft).

[2]*Spent Fuel Transportation Applications—Assessment of Cladding Performance: A Synthesis Report*. EPRI, Palo Alto, CA: 2007. 1015048.

# What questions will we focus on addressing?

- **From the list of questions for this meeting:**
  - What are the cladding properties important to high-burnup transportation and storage licensing?
  - How do we determine which of all the many variables are the important ones?
  - How do irradiation parameters affect behavior and which of these parameters and behaviors are important for high-burnup transportation and extended storage?
  - What are the effects of radiation damage in cladding on mechanical behavior (creep, hydride reorientation, strength/ductility, etc.)?
  - How do we account for multiple effects (annealing, creep, hydride reorientation, etc.) occurring simultaneously?

- **Some questions that have arisen from the EPRI report:**
  - What small-scale damage features (fretted regions, hydride lens, bowed rods/assemblies) might change the failure rates, peak pinch forces, and/or bending moments?
  - What effect might inter-pellet leverage have on the transverse tearing mode failure probabilities?
  - Will the NRC accept the conclusions in light of uncertainties?
    - *"The failure frequency… was estimated to be 50%, but no fuel reconfiguration is predicted."*
    - *"damage initiation can be expected, but progression of the damage… can be ruled out"*
    - *"ovalize but not fracture or totally collapse, which preserves the structural integrity"*



# Sensitivity analysis is required; Select uncertainties will be needed.

- **Sensitivity analysis is very useful**
  - **Sensitivity of the parameters in the EOS constitutive model -> relative significance of uncertainties from:**
    - **Post-drying state**
    - **VLTS conditions**
    - **Parameters in the constitutive models used in the VLTS simulation**
  - **Guides the program in identifying the areas of need**
    - **Uncertainty estimates make this more useful: a highly sensitive term with no uncertainty, does not need to be improved**

- **Uncertainty estimates for the EOS constitutive model will be required later**
  - **UQ in the transportation accident, requires estimates of uncertainty in the EOS state**
    - **Which needs propagation of VLTS uncertainties**
  - **But ONLY for the most sensitive inputs and parameters**

- **Uncertainty in the post-drying state**
  - **Burnup, rod internal pressure, oxide thickness, hydrogen content, radial precipitates from drying, …**
  - **Provide correlations, in value and uncertainty (co-variance), between the states**
  - **For many rods in many reactors over that past decades**
    - **Much of this probably exists, has been compiled, and may be published or simply need to be processed**

- **Uncertainty in material models used in VLTS simulation**
  - **Can this be processed from existing experiments or will this require new ones?**
  - **Can we get models and uncertainties for proprietary materials: Zirlo, M5, liners, …?**

- **Science-based improvements to the models, change what is considered sensitive**
  - **There will be more "tunable" parameters in the calibration of the models**
    - **Hopefully the sensitivity of the parameters will be significantly reduced**
  - **"Predictive science" has no "tunable" parameters**

# Planned Activities for this Fiscal Year

- **Define a problem to be solved using AMP**
  - **Model the long-term creep of single fuel pin and account for the effect of hydride reorientation**
  - **For a (set of) representative fuel rod(s) and storage condition(s) with specified post-drying state(s)**
    - **Suggestions welcome**
- **Incorporate the effects of hydrogen**
  - **Implement models for radial hydride precipitation and cladding failure**
    - **Build sensitivity analysis into the models as part of its development**
- **Demonstrate AMP with effects of hydrogen**
  - **Compute integral cladding creep during VLTS for an LWR pin with the hydrogen-aware constitutive models**
  - **Estimate the cladding mechanical properties after VLTS for use in a future transportation simulation**
- **Estimate the sensitivity of End-of-Storage (EOS) mechanical state**
  - **To hydride model parameters, post-drying condition (hydrogen, radial hydrides, burnup), and storage conditions (time)**
  - **Propagate uncertainties in the hydride model(s) to estimate uncertainty in EOS state**
- **Planning for additional work**
  - **For extension to integrated modeling of a cask**
  - **With experimenters, defining targeted experiments to characterize the uncertainties in the models**

# Proposed Models for Hydriding During Storage

- **Estimate Zircaloy radial hydride concentration at EOS[3]**
  - **Inputs:**
    - **Beginning of dry Storage (BOS) temperature, BOS hoop stress, and time**
  - **Output:**
    - **Radial hydride concentration**
  - **Validated:**
    - **With irradiated and H-charged specimens**

- **Predict Zircaloy constitutive model (including failure)[4]**
  - **Strain Energy Density (SED) is the basis for failure criteria**
  - **Accounts three-phases:**
    - **Base metal, radial hydrides, and radial-variation of circumferential hydrides**
  - **Inputs:**
    - **Hydrogen content, temperature, and radial hydride concentration**
  - **Assumes:**
    - **Shape of radial variation of circumferential hydrides**
  - **Calibrated with Critical SED (CSED) from experiments**
    - **"Hydride efficiency factor" is the parameter calibrated**
  - **Validated:**
    - **With irradiated and H-charged specimens**

[3] *Hydride Precipitation in Spent Fuel Cladding During Dry Storage,* ICEM05 1038, 2005.

[4] *Development of Metal/Hydride Mixture Model for Zircaloy Cladding with Mixed Hydride Structure,* EPRI, 1009694, 2004.

# Integration of Experiments and Modeling Meeting

- **Proposal:**
  - **We would like to host a small meeting to review and improve the hydride model and the relevancy of the demonstration problems**

- **Relevant questions:**
  - **Short-comings of the model(s) we've proposed**
  - **Identification of data/experiments currently available**
    - **To assess/validate the model(s)**
    - **To assess/validate the AMP using the models**
  - **Definition of experiments in-progress**
    - **To evaluate if AMP is capable of modeling it**
  - **Clarification our the demonstration problems to ensure relevancy**

- **Would you be interested in participating?**
  - **Please email: Glen Hansen <gahanse@sandia.gov>**
  - **Or find him at the next break**

# Possible Model Questions

- **What would be involved in propagating rod internal pressure variability, clad oxide thickness uncertainty, MWTA hydrogen content uncertainty, etc. to uncertainty in the final yield margin?**
- *Reliable failure criteria that capture relevant mechanical behavior regimes of high-burnup cladding with both circumferential and radial hydrides are presently lacking, with limited near-term prospects for developing such criteria experimentally.* (pp. 3-1)
- *Very limited data exist for fracture toughness at high hydrogen concentrations [14], and no fracture toughness data exist for Mode-III failure, which is the only mode affected by radial hydrides. Thus, new criteria and method of application of such criteria needed to be developed for high burnup cladding, as discussed in Reference [9].*
- **Is it possible to directly construct CSED curves from new or existing data?**

[9] Y. R. Rashid, M. M. Rashid and R. S. Dunham, "Failure Criteria for Zircaloy Cladding using a Damage-Based Metal/Hydride Mixture Model," EPRI Technical Report 1009693, December 2004.
[14] Y. R. Rashid, R. O. Montgomery and W. F. Lyon, "Fracture Toughness Data for Zirconium Alloys; Application to Spent Fuel Cladding in Dry Storage," EPRI Report 1001281, January 2001.

# Assembly Response Model in Report

- *The structural modeling and analysis described in the report is intended to represent the dynamic response of all the fuel rods in the cask.*
- *Instead of modeling every fuel rod in detail, which would be an impossible task, a detailed model of the Control Assembly was used as a device to bracket the dynamic response of all the rods in the cask by placing the Control Assembly in various positions in the basket structure where the deceleration is expected to vary over the expected range.*
- *The dynamic forces were calculated at thousands of points and vary over a wide range. This force system constitutes a statistically significant "database" that is appropriately suited for constructing probabilistic evaluation of the drop event consequences.*

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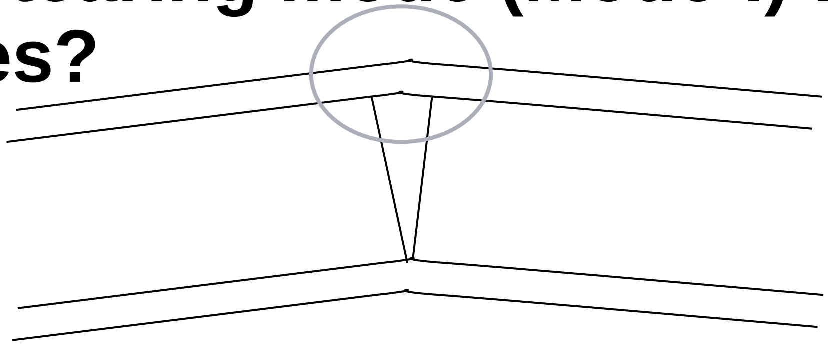
# Possible Fidelity Questions

- Are there small scale damage features (fretted regions, bowed rods, etc) that might change the peak pinch forces and bending moments?

- What effect might inter-pellet leverage have on the transverse tearing mode (Mode-I) failure probabilities?



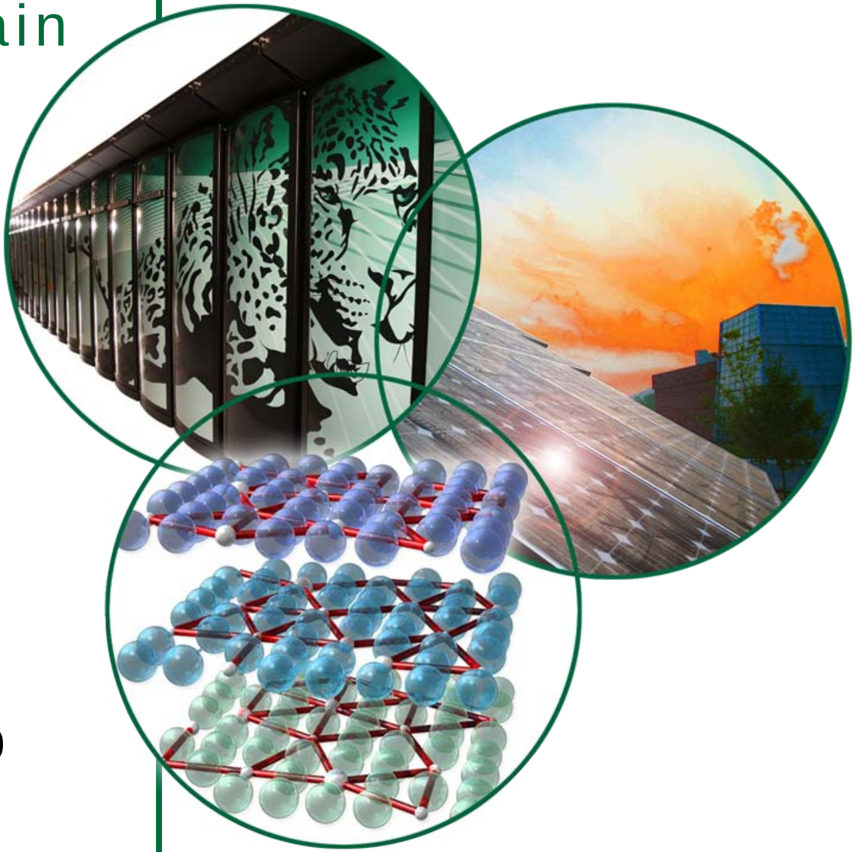
Sandia National Laboratories



Oak Ridge National Laboratory

ATTACHMENT 2

# Development of An Expanding Plug Test Method for Determining Hoop Stress-Strain Curves of Irradiated Nuclear Fuel Cladding




B. B. Bevard, Richard Battiste,
James Hemrick, and Wallace McAfee

Oak Ridge National Laboratory


Used Fuel Disposition – Cladding Workshop
Las Vegas, NV
15-17 November 2011

U.S. DEPARTMENT OF ENERGY

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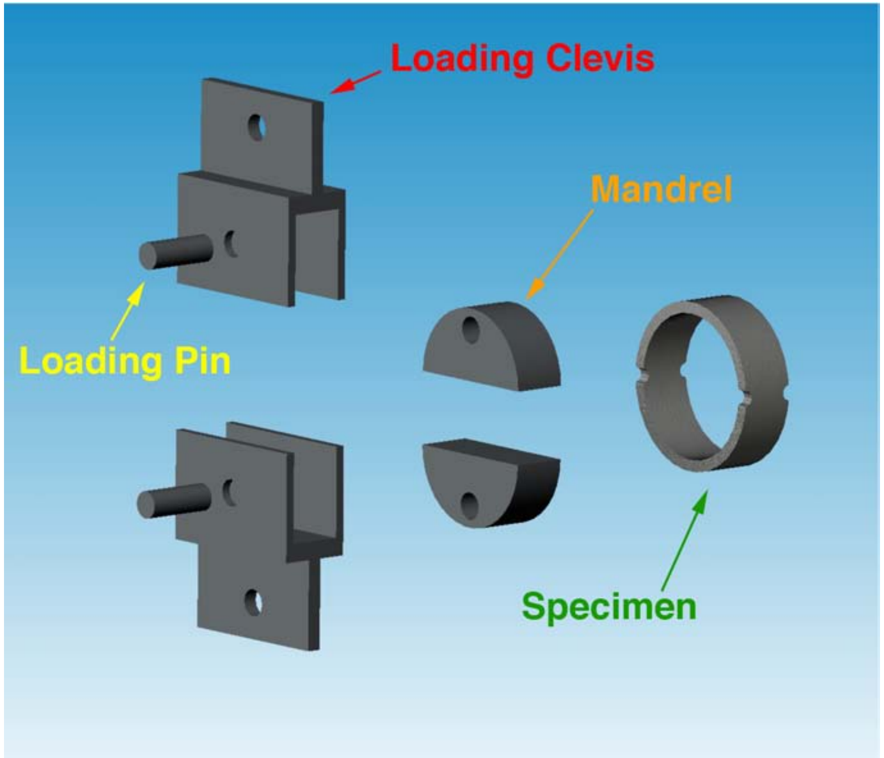
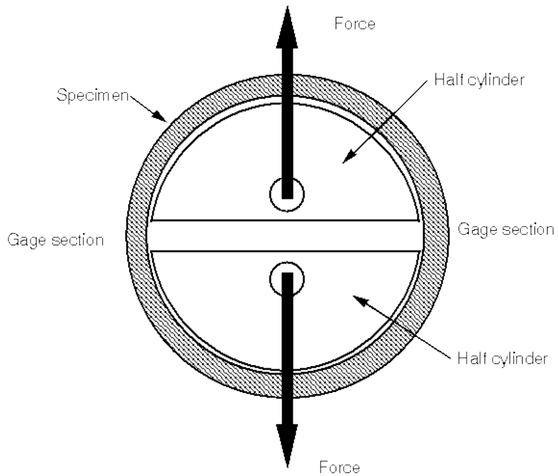
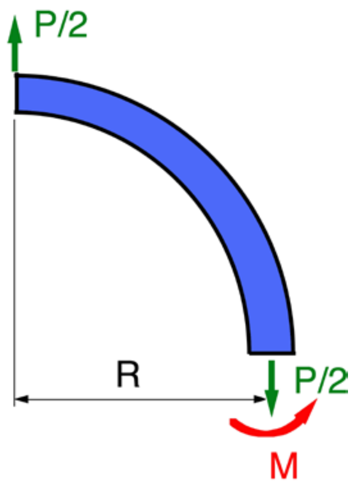
# Ring-Stretch Method Has Yielded Some Success in Developing Stress-Strain Results for Irradiated Cladding




- *Requires complex assembly in hot cell*
- *In-cell machining required if reduced gage section is used*
- *Friction develops between specimen and load mandrels*
- *Difficult to measure extension directly at gage section of specimen*

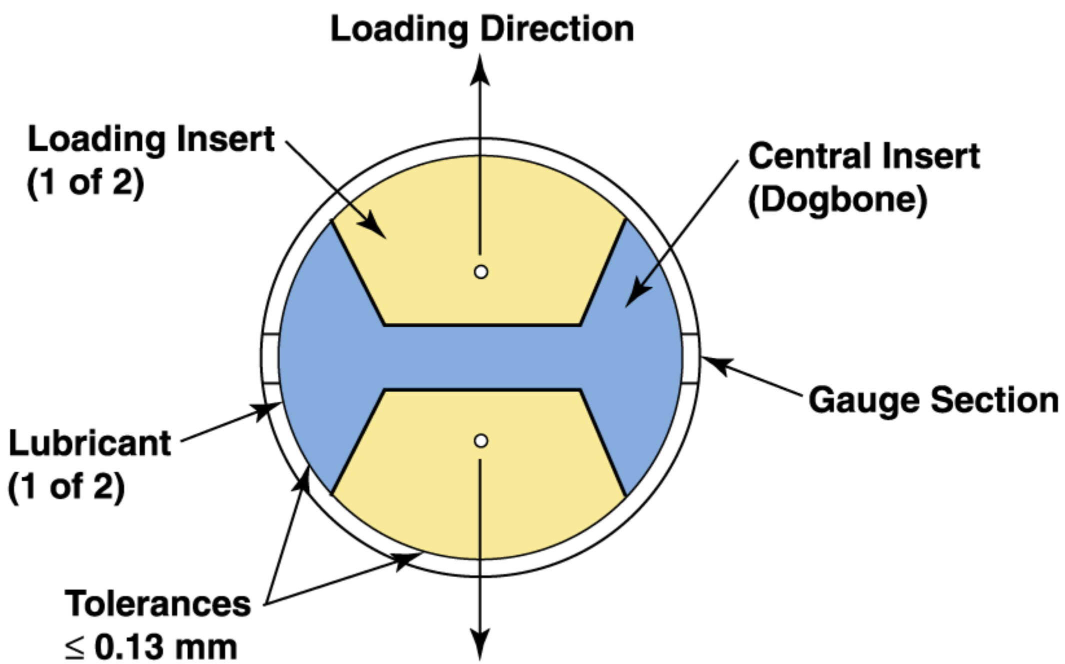
# It is Difficult to Load the Specimen Without Introducing Bending Stresses Atypical of In-Reactor Clad Loading




- ***Gage section initially subjected to high bending stress relative to axial stress***
- ***High friction between the specimen and loading mandrels***
- ***Finite element analyses required to support data reduction***
- ***For low ductility material, bending in gage section may predominate failure***
- ***Has limitations for high-burnup cladding with low ductility***

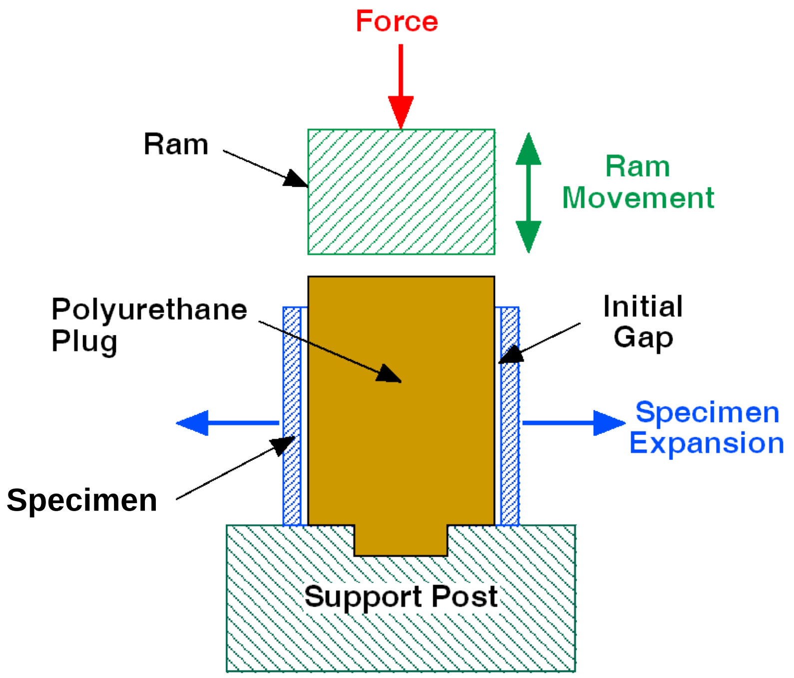
# The Bending Can be Somewhat Alleviated by Use of a Central "Dogbone" Insert and Very Tight Tolerances




- *Bending not totally alleviated*
- *In-cell assembly becomes more difficult*
- *Still requires in-cell machining*

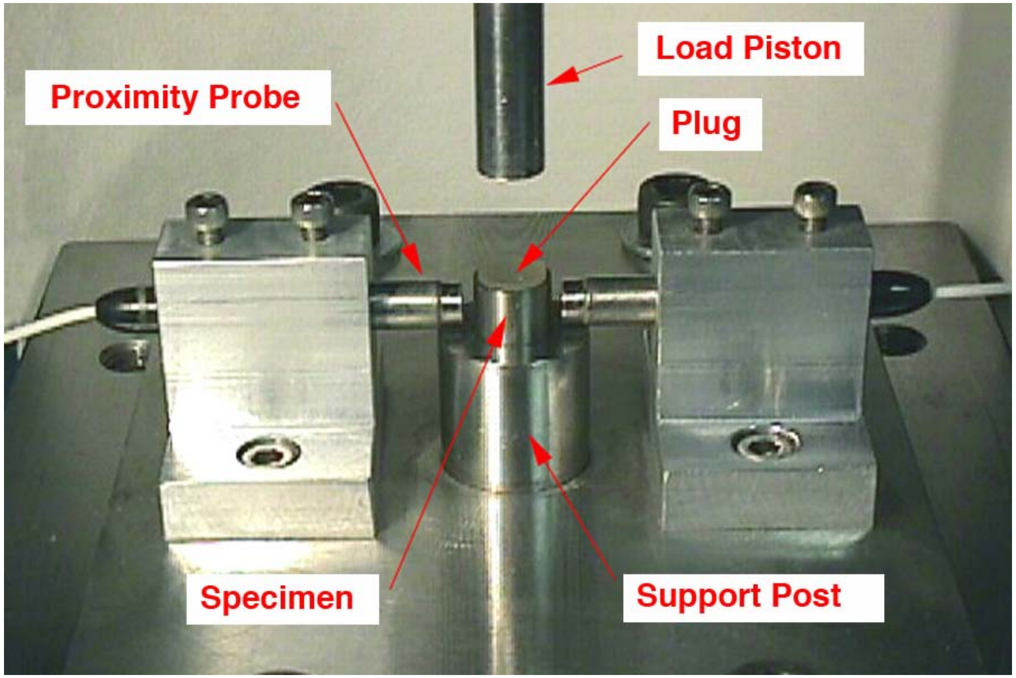
# ORNL Has Developed an Improved Experimental Technique For Measuring the Tensile Properties of Irradiated Cladding

- ***Test procedure uses compression of a polyurethane plug fitted inside a short cladding ring specimen***
  - ***Plug compression forces expansion similar to swelling of fuel or internal pressurization***
  - ***Loading leads essentially to near uniform wall stress***
- ***Procedure incorporates several specimen prep/testing simplicities***
  - ***No complex specimen machining***
  - ***Strain is uniform around clad ring***
  - ***Circumferential strain is simply the diameter increase divided by the initial diameter***



# Expanding Plug Test Technique Utilizes a Simply Set-Up That is Hot-Cell "Friendly"

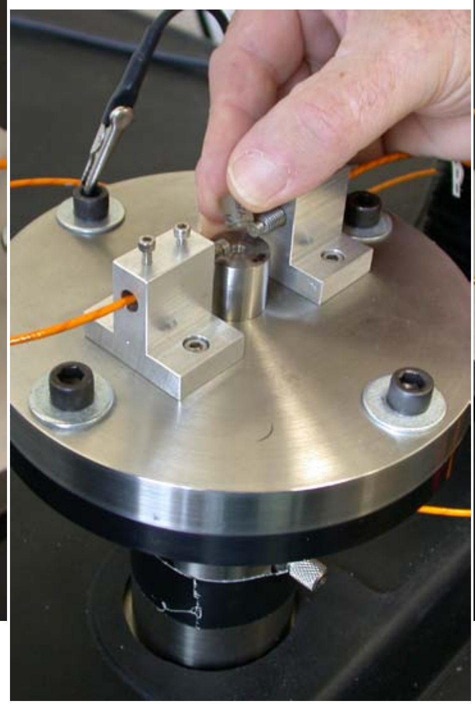



- ***Support Post and Proximity Probes are fixed***
- ***Load Piston is test machine controlled***
- ***Only Plug and Specimen require manipulator installation***

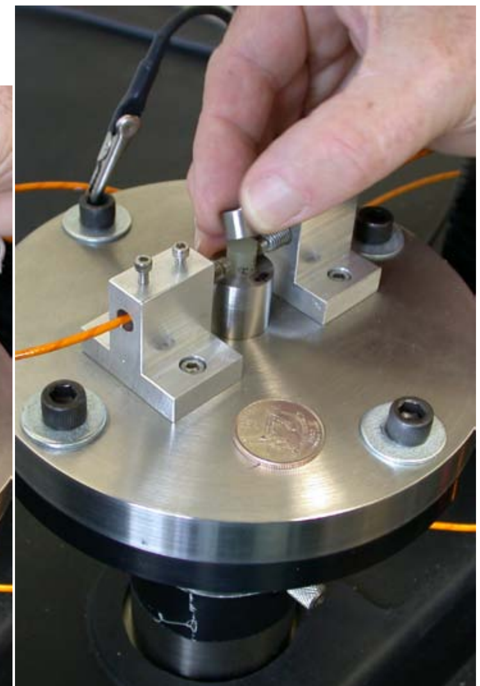
# *Illustration that Component Assembly for Specimen Testing Is Straightforward and Readily Adaptable for Use With Hot-Cell Manipulators*

**Original test fixture configuration**

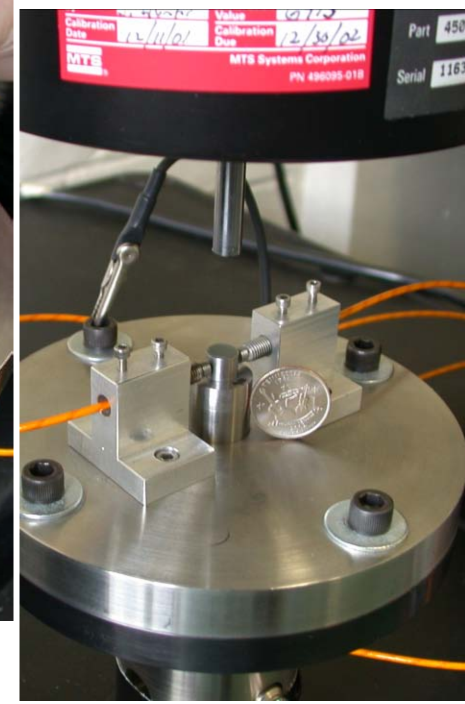
**Step 1**
**Mount plug**



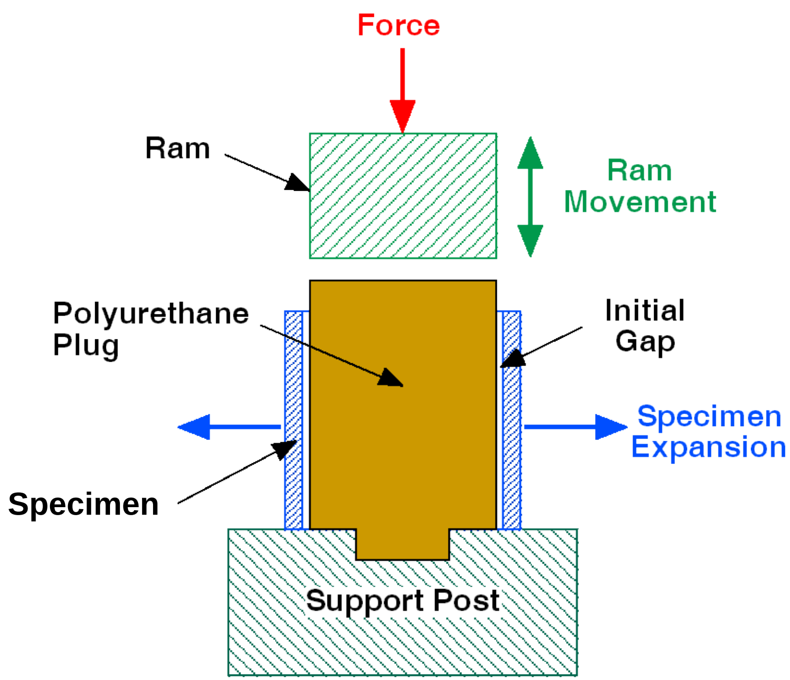
**Step 2**
**Position specimen over plug**



**Step 3**
**Specimen ready for test**



# *During Testing, Continuous Measurements of Temperature, Ram Movement, Specimen Expansion, and Load Are Made and Recorded With a Continuous Time Stamp*




**Total Load**

- **Total load is measured by in-line load cell**

**Ram Movement**

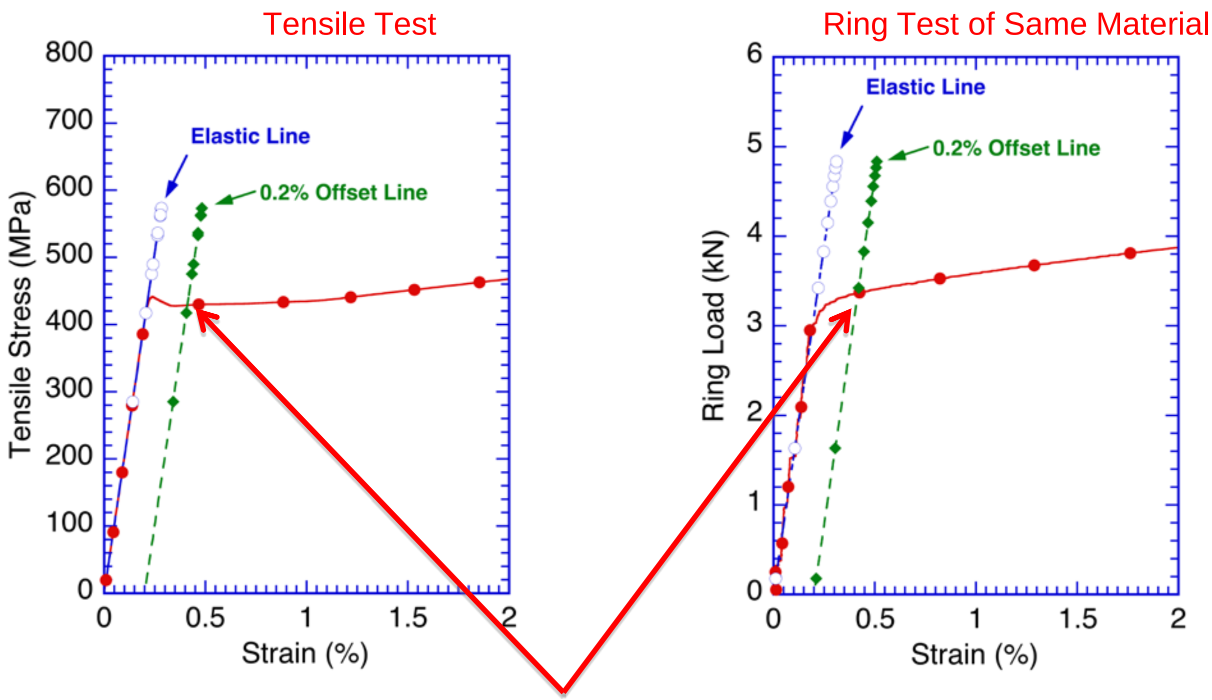
- **Movement of machine crosshead**
- **Amount the plug is compressed called "Extension"**

**Specimen Expansion**

- **Measured as reducing gap (2) between specimen surface and proximity probe**
- **Total diametrical expansion of specimen is converted directly to circumferential strain - ΔD/D**

# A Simple Methodology for Calculating Stress and Strain Has Been Developed and Extensively Verified

Based on observed general similarity in shape of Stress-Strain curve from ASTM tensile specimen and Load-Circumferential Strain data from ring test of same material




- Ring Circumferential Stress = *f*(Ring Load at Yield, Material Yield Stress)
- Function, *f*, could be constructed as a simple scaling factor, Γ-Factor
- Yield point was selected since material stress is known at that strain
- Ring specimen is treated as pressurized cylinder

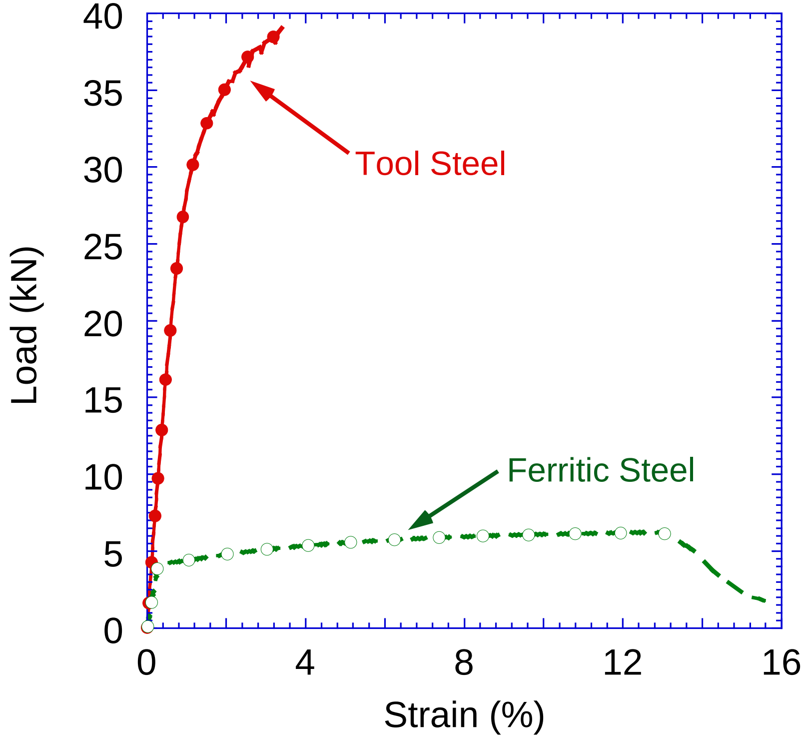
# *A Methodology for Calculating Stress and Strain Has Been Developed and Verified (cont'd)*

- ***Circumferential stress is calculated using the scaling parameter, Γ-Factor***
  - $\sigma_{Cir} = \Gamma P/tl$
  - ***Where***
    - $\Gamma = (\sigma_{Yield}/P_{yield})tl$
    - $\sigma_{Yield}$ ***= yield stress of a QA'd material determined using ASTM tensile test procedures***
    - $P_{Yield}$ ***= Load at 0.2% offset strain measured using ring specimen of above QA'd material***
    - $t$ ***= ring specimen thickness***
    - $l$ ***= ring specimen axial length***
- ***Circumferential strain is determined directly from ring expansion measurements***
  - $\varepsilon_{Cir} = \Delta D/D$
    - $\Delta D$ ***= average change in diameter from 4 proximity probe measurements***
    - $D$ ***= original specimen outside diameter***

# Comparison of the Expanding Plug and the Ring Stretch Test Method Shows That a More Direct Measurement is Possible

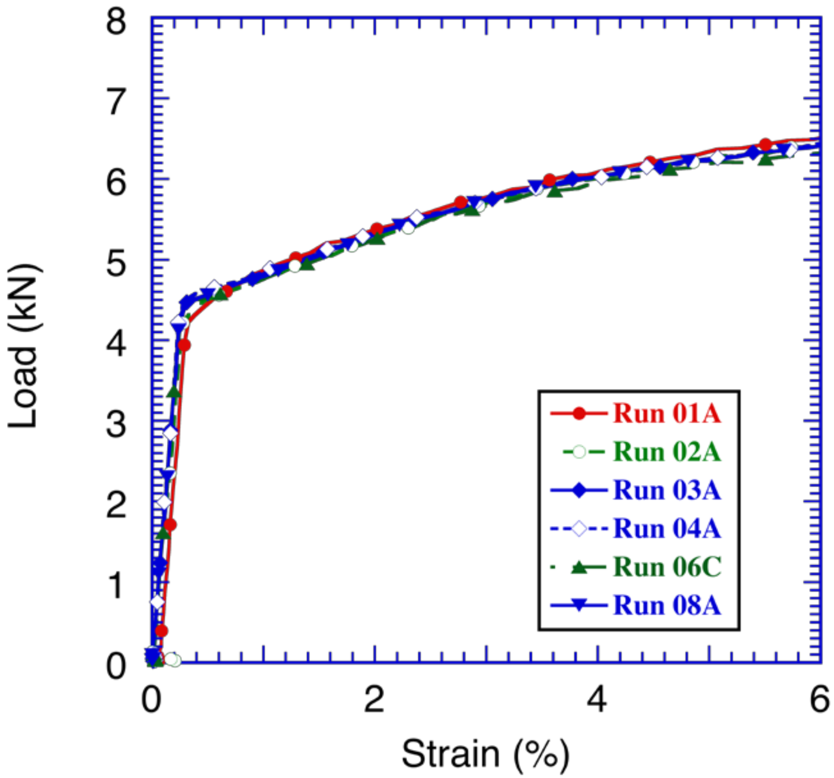
| Expanding Plug Test Data Obtained by Direct Measurement and Use of Γ-Factor | Ring Stretch Test Data Obtained Through Supporting Finite Element Analyses (FEA) |
|---|---|
| Complete stress-strain curve obtained directly | Stress-strain curve obtained through Finite Element Analysis (FEA) of test |
| Elastic Modulus | Elastic Modulus (FEA) |
| Yield Stress | Yield Stress (FEA) |
| Ultimate Stress | Ultimate Stress (FEA) |
| Uniform Elongation | Uniform Elongation (FEA estimated based on material ductility) |

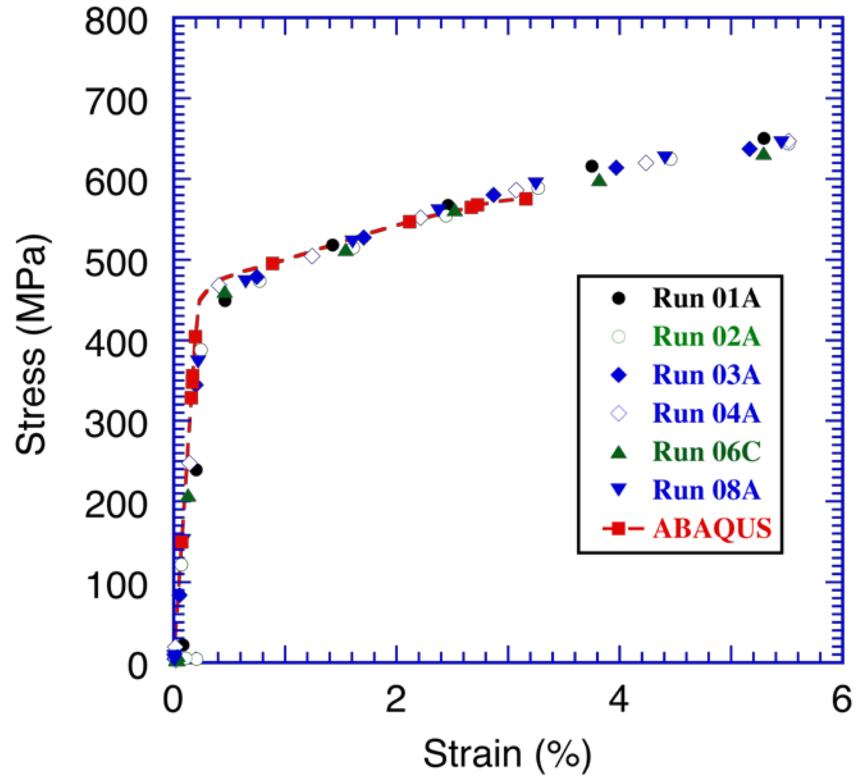
# *Validation of Test Technique Has Been Performed for a Wide Range of Material Failure Loads and Ductilities*




- **Technique permits measurement of loads and strains far in excess of those expected in highly irradiated nuclear fuel cladding**

# A Ferritic Steel (A533B) Was Used Extensively to Evaluate the Reliability and Data Quality Obtained From Expanded Plug Tests



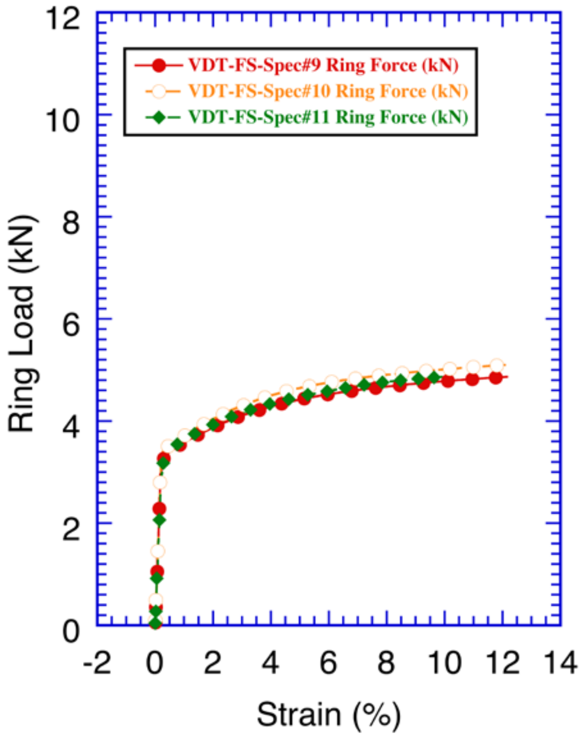




Baseline testing of 6 ferritic steel ring specimens demonstrates consistency of test data obtained using this technique

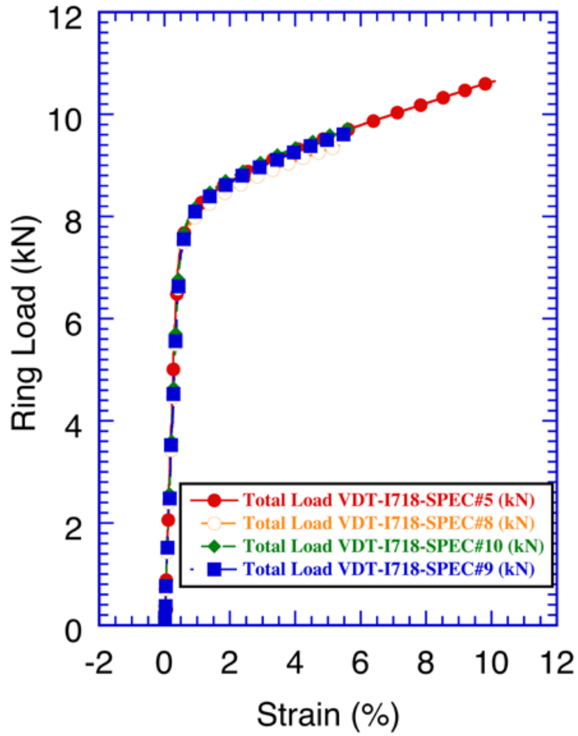
ABAQUS analyses of ferritic specimen using ASTM tensile data as input shows excellent agreement when load data are converted to stress using **Γ-factor**

# How Well Does Γ-Factor Methodology Work?

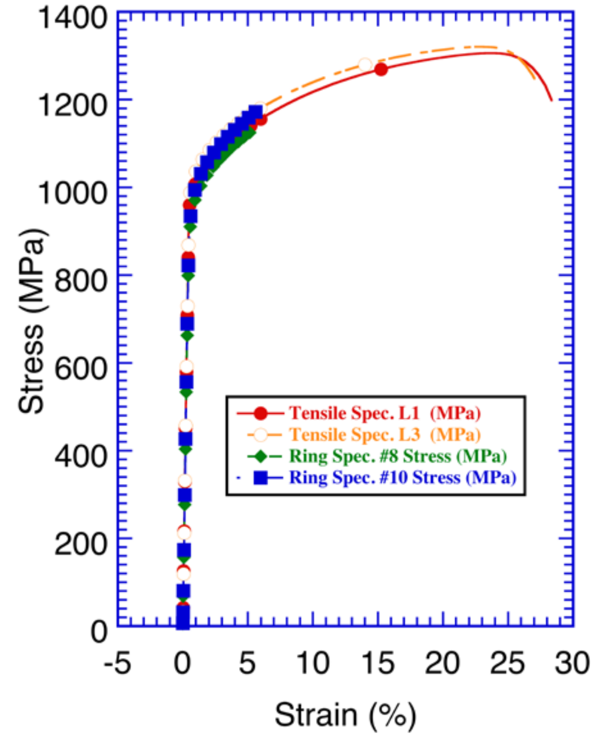
Ferritic steel ring specimens used to calculate Γ-Factor




Ring specimens of heat treated Inconel 718 used for “blind” prediction test




Comparison of calculated ring σ-ε predictions with ASTM round bar tensile test results from heat treated Inconel specimen



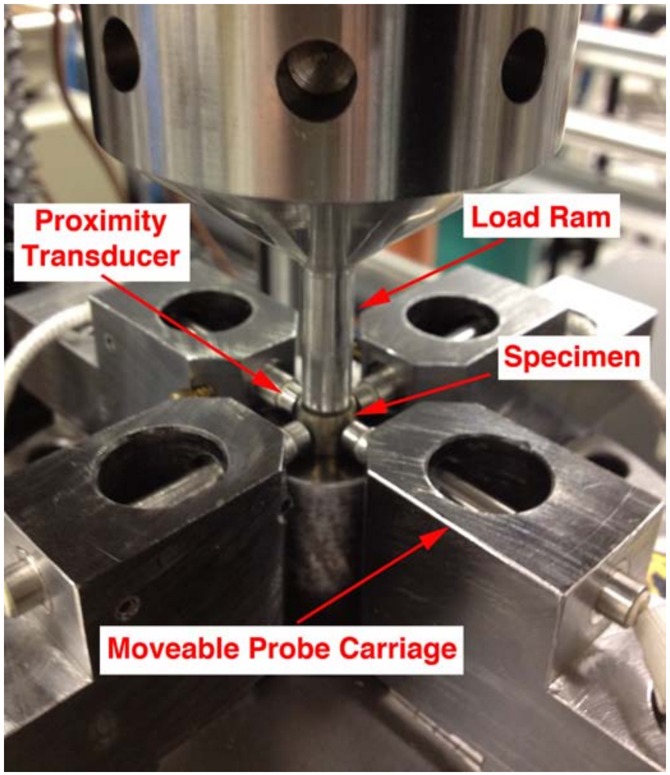

Scaling Γ-Factor calculated using properties of intermediate-strength ferritic steel makes an excellent “blind” prediction of stress-strain behavior of heat treated Inconel 718 ring specimens compared to heat treated Inconel 718 ASTM round bar tensile specimens


Managed by UT-Battelle
for the U.S. Department of Energy

# Existing Test Facility Has Been Upgraded to Improve Strain Measurement Precision and Test Temperature Range

- *Fixture uses 4 proximity transducers to measure diametrical expansion*
  - *Transducers located at 90° increments around specimen periphery*
  - *Improves precision and reliability of diameter change measurements*
- *Test temperature range expanded significantly*
  - *Uses RF heating*
  - *Tests currently have been performed over range RT to 350°C*
  - *Technique to be further extended to 800°C*

# Upgraded Fixture Emphasizes Compatibility With Hot Cell Limitations




- *Proximity probes mounted on movable carriages that retract horizontally*
  - *Clear area for specimen insertion and removal by manipulators*
  - *Clear area for RF coil insertion/removal and maintenance*
- *Carriages are synchronized to return probes to same position each time*
- *Carriages driven by simple screw mechanism easily cranked using manipulators*
- *Upgrades to test system have been verified and are fully functional*
- *An existing test program is currently underway*

# ORNL Has Developed and Validated a Simple Method for Measuring Circumferential Tensile Properties of Highly Irradiated Nuclear Fuel Cladding

- *Specimen preparation in hot cell is relatively simple and has low operator learning curve as compared to ring stretch method*
- *Specimens are small (length/diameter ≈ 1) and thus use small amounts of material*
- *Specimen test procedures can be easily implemented using existing hot cell procedures*
- *Tests and analyses have demonstrated accuracy of stress-strain determination for embrittled materials up to strains exceeding 5%*
- *Procedure has been demonstrated and verified for materials ranging from high to low ductility*

# Backup Slides

# An Evaluation of Performance and Safety of Nuclear Reactor Cores Puts Emphasis on the Need to Measure the Tensile Properties of Highly Irradiated Nuclear Fuel Cladding

**Overview of ORNL Clad Test Development Will Cover Several Areas**

- *Provide brief background on irradiated cladding tensile test techniques in current use*
- *Describe test facilities and procedures that have been developed at ORNL*
    - *Improvements over existing techniques*
    - *Limitations on application*
- *Present prototypical results*
- *Describe current status*

# *An Evaluation of Performance and Safety of Nuclear Reactor Cores Puts Emphasis on the Need to Measure the Tensile Properties of Highly Irradiated Nuclear Fuel Cladding*

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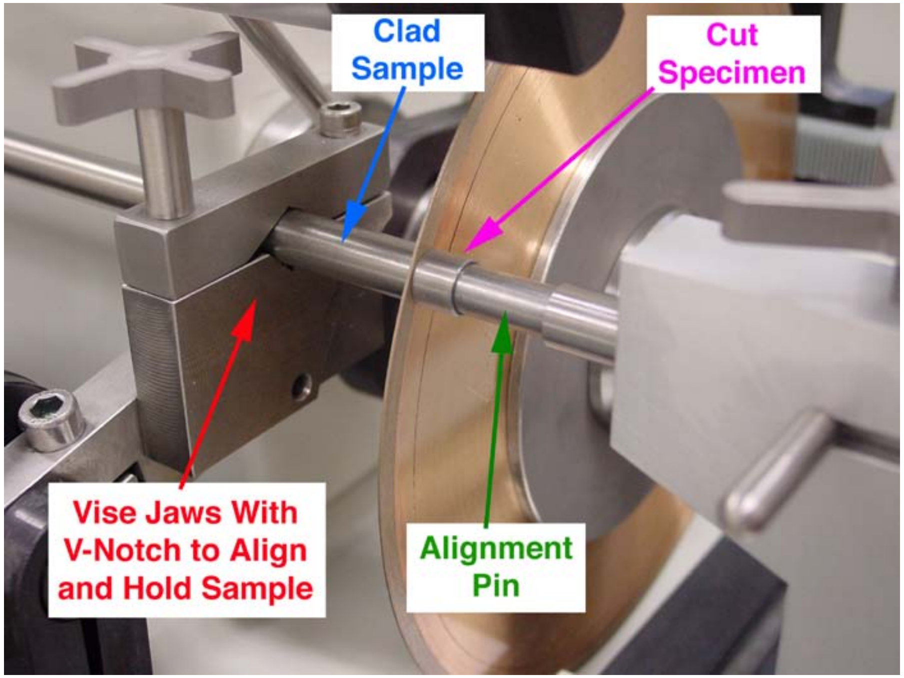
  - ***Limitations on application***
- ***Present prototypical results***
- ***Describe current status***

# Several Test Techniques Have Been Developed/Used in Attempts to Measure Post-Irradiation Tensile Properties of Fuel Cladding

- ***Ring-Stretch Method***
  - *Has been most widely used*
  - *Variations are smooth ring, notched ring, sheet tensile configuration ring*
  - *Method has been used by several institutions including ANL, CEA, JAERI, RRC_KI, and RIAR*
- ***Expanded Plug***
  - *Developed by ORNL*
  - *Significant improvement in simplicity and data quality*
- ***Other Methods Include***
  - *Tapered mandrel*
  - *Straight tension*
  - *Internal pressure*

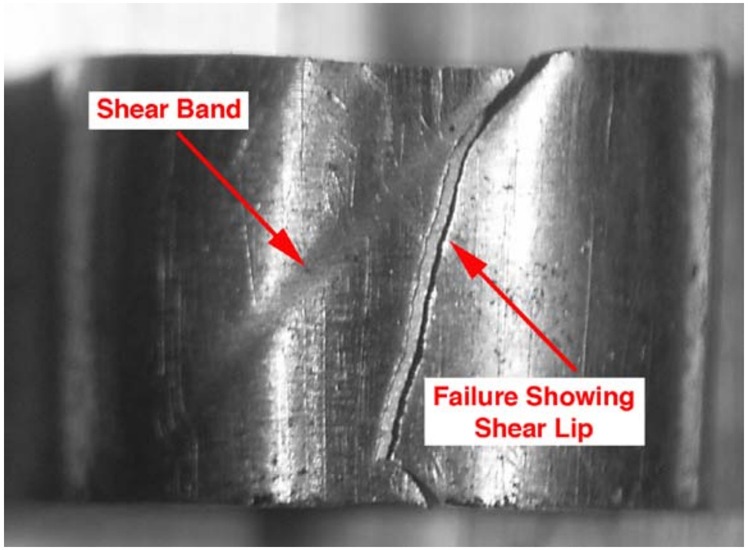
# Procedures for In-Hot-Cell Cutting and Handling of Ring Specimens from Defueled Fuel Rod Segments Have Been Developed

- Cladding defueled
- ID of cladding brushed and cleaned
- Specimens cut from cladding sample
- Specimens deburred




Note: all preparation steps are easily performed remotely in a hot cell environment. Also, there is no specimen machining or modification of outside surface of the specimen.

# The Expanding Plug Test Technique Has Been Used to Determine the Tensile Properties of a Large Matrix of Irradiated Zr-4 Fuel Cladding

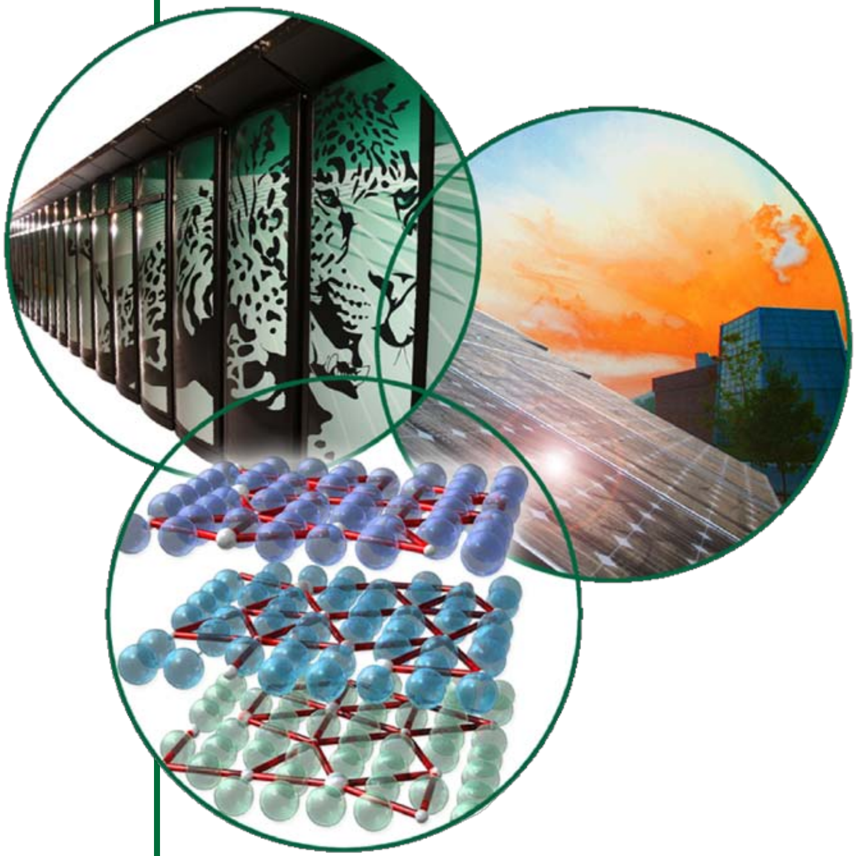



Typical room temperature failure

- ***Tests performed from unirradiated to 50 GWd/MT with no difficulty***
- ***Results at all burn-up levels were highly consistent***
- ***Yield stress varied from near 650 MPa (unirradiated) to near 950 MPa (50 GWd/MT)***
- ***Good agreement was observed with data available in the literature***
- ***Characteristics of failure tests verify uniform distribution of stress in specimen***

ATTACHMENT 3

# Hydrogen Charging Techniques Being Developed at ORNL



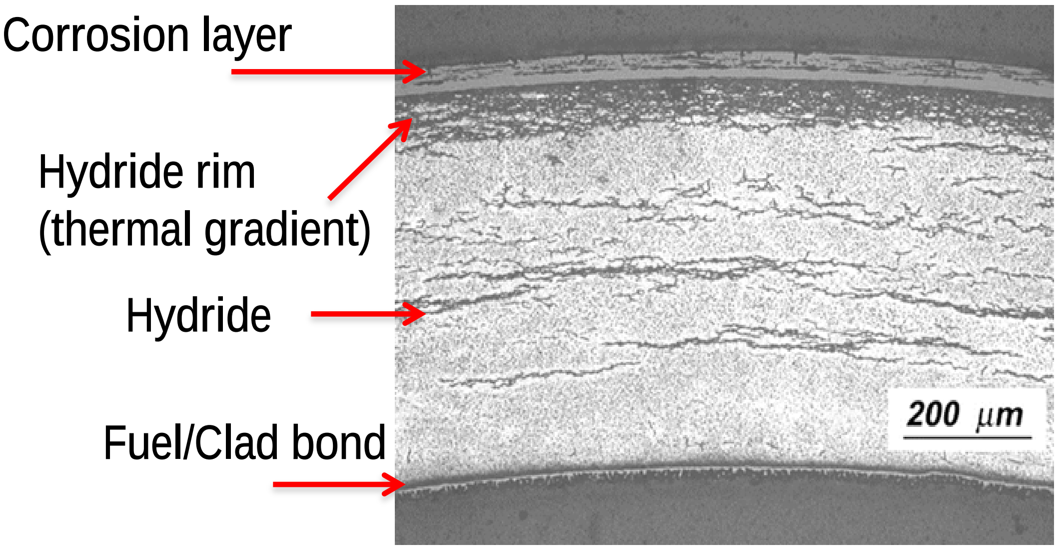
Y. Yan, J. Kiggans, and C. Davisson

DOE-UFC Clad Workshop
Las Vegas, NV
November 15-17, 2011

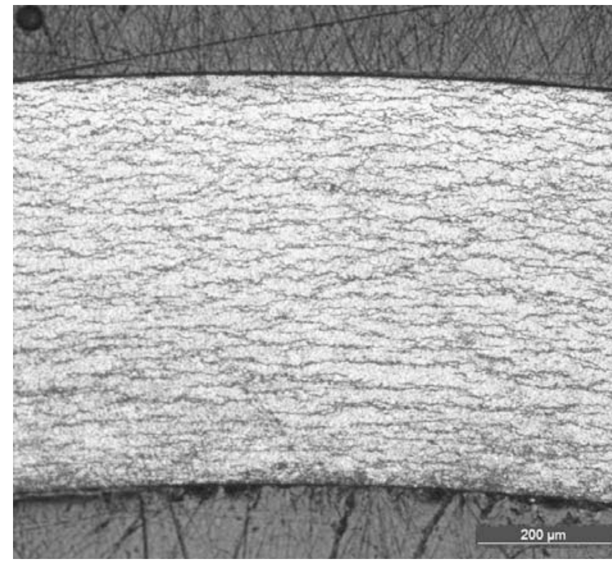
# Motivation

- Hydrogen pickup in PWR and BWR cladding increases with burnup causing decreases in cladding ductility and toughness
- Hydrogen-induced embrittlement has important implications for both higher-burnup fuel operation and used-fuel dry storage & transportation
- High-burnup used fuel samples are difficult to obtain and very expensive to handle, *but* irradiated-hydrided Zr alloys - having similar hydride morphology - can simulate the high-burnup used fuel samples

# Hydride Distribution in Used Fuel Sample and Two-sided Hydrided Materials




Hydride distribution in as-irradiated high burn-up Zr alloy sample with 660±150 wppm hydrogen.* Corrosion layer is ≈25 μm.




Hydride is uniformly distributed across the cladding wall for an unirradiated two-sided hydrided Zry-4 sample with ≈700 wppm hydrogen.

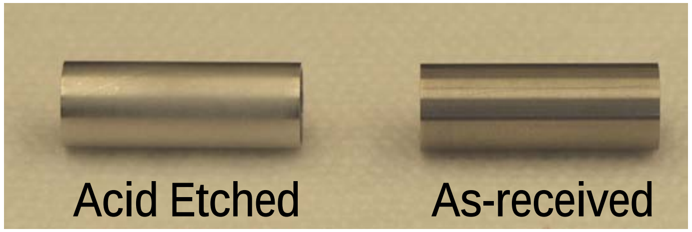
* Y. Yan, M. Billone, and T. Burtseva, “Hi Burnup Sample Characterization and Thermal Benchmarking,” NRC Program Review Meeting, ANL, October 5-6, 2007.

# Sample Preparation for Hydriding Zry-4 Cladding

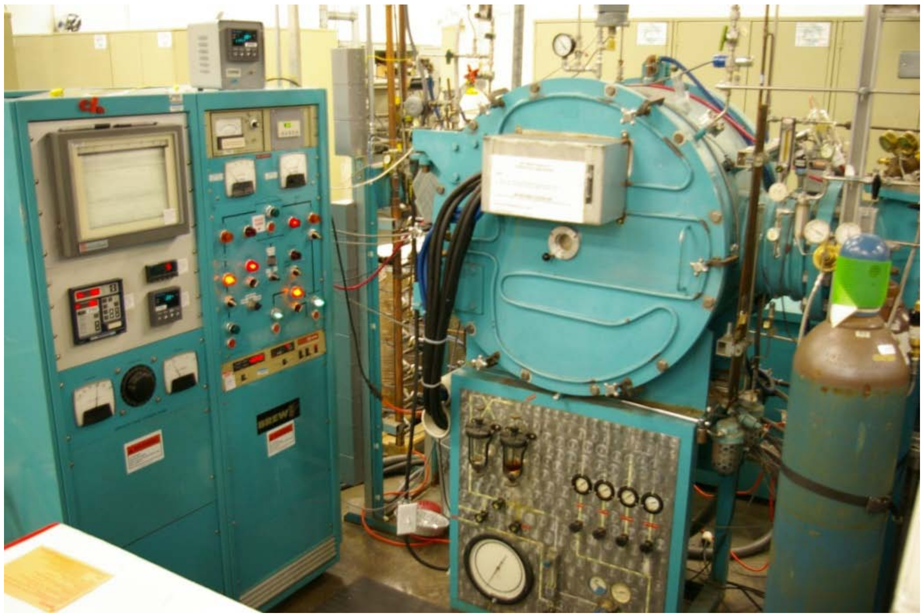
Materials: 17x17 Zry-4 (OD=9.5 mm, t=0.57 mm)

Sample Cleaning: ASTM Standard (G 2/G 2M – 06)

Surface etching: removing surface oxide layer



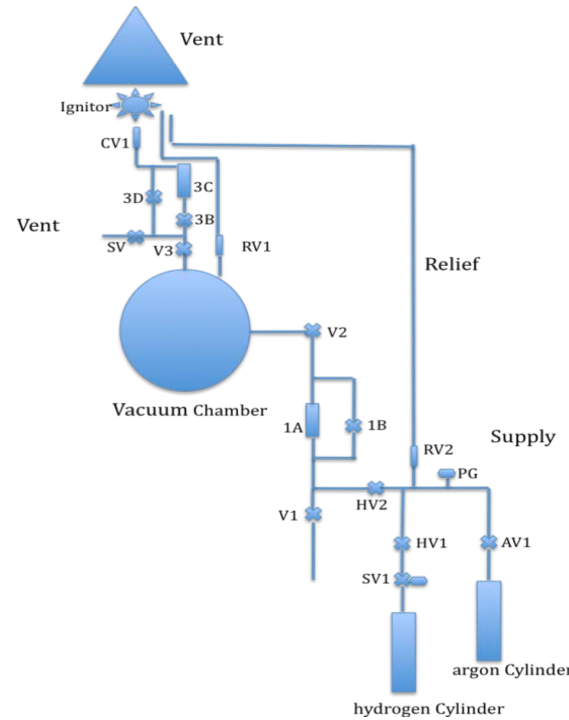

# 901 Brew Furnace for Materials Hydriding



Controller
for furnace and
vacuum system

Furnace
in vacuum
(below 50 millitorr)

$H_2$ Tank




Schematic illustration

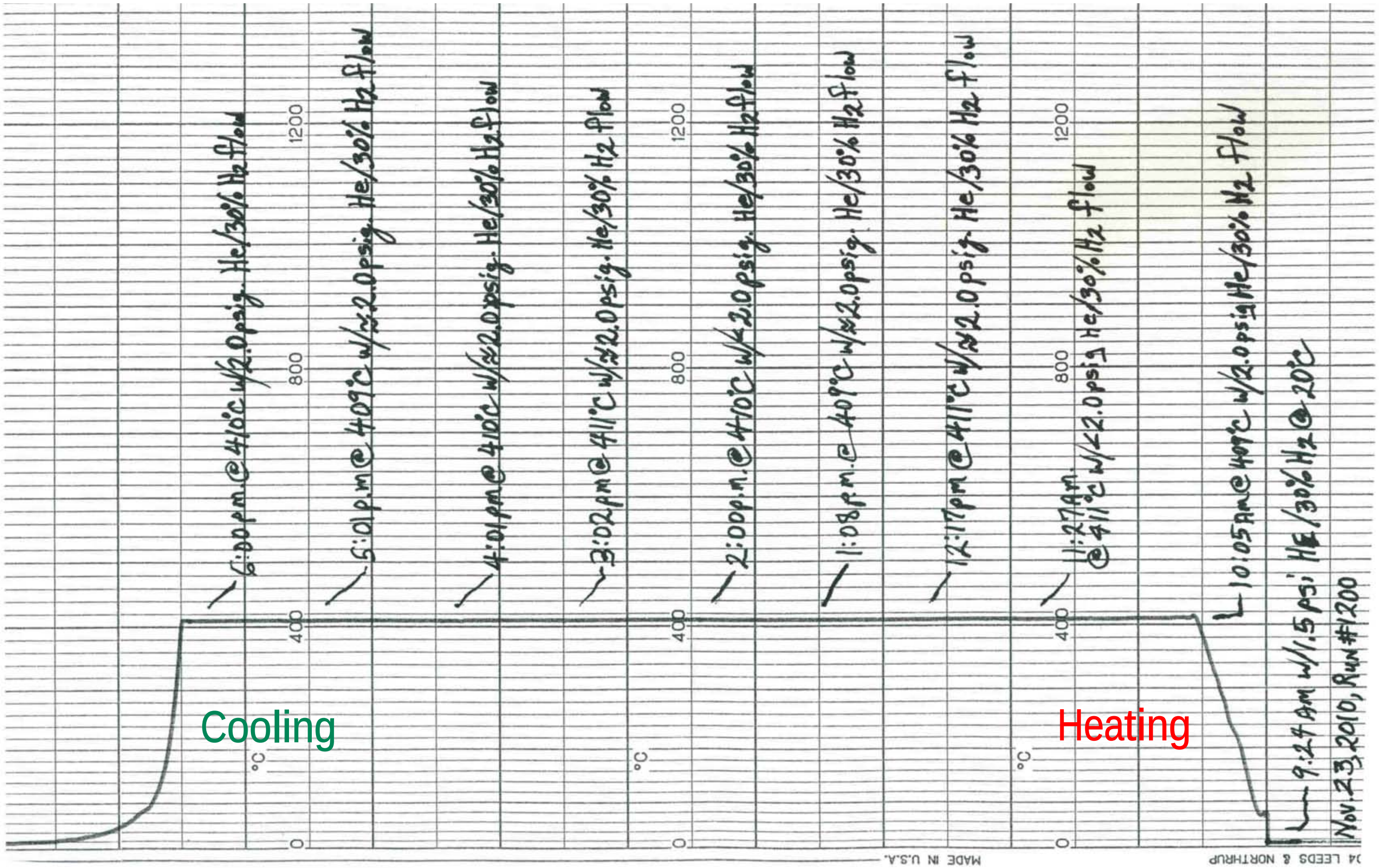
# Test Conditions for Pre-hydriding Zry-4

| | |
|---|---|
| Materials | 17x17 Zry-4 |
| Charging Gas | a) 4% $H_2$ in argon; b) 30% $H_2$ in helium |
| Cladding size | OD=9.5 mm; t=0.57 mm |
| Target hold temperature | 400°C |
| Test time | Various |
| Heating RAMP, | ≈15 °C /min. |
| Cooling rate | 15±5 °C /min. from 400 to 100 °C, followed by furnace cooling |
| Post-test H content | 200 – 1500 ppm |
| Thermal cycling | For uniform hydrogen distribution |

# Hydrogen Analysis for Hydrided Samples

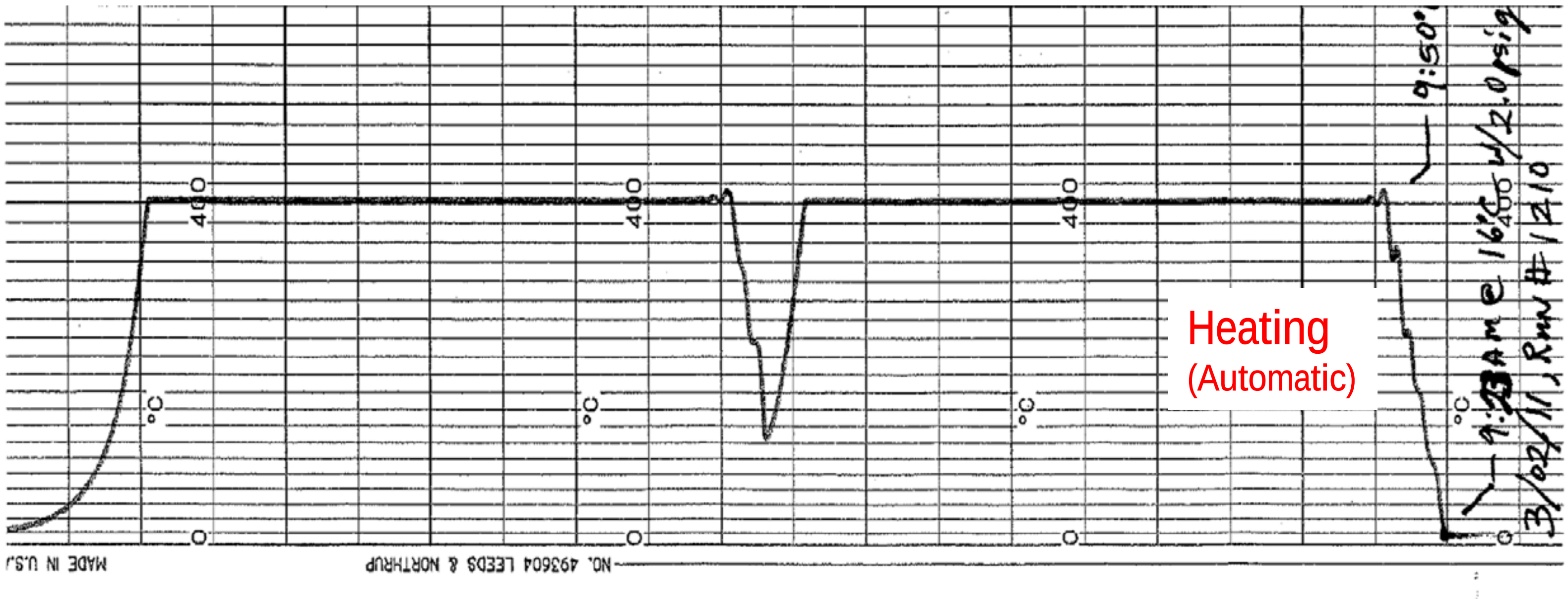

Method:  Inert Gas Fusion
Equipment: LECO RH-404 Hydrogen Analyzer
Reference: ASTM E1447-05
Calibration: Verified with standards of known hydrogen content before each run

# Temperature Profile for Hydriding Cladding Materials

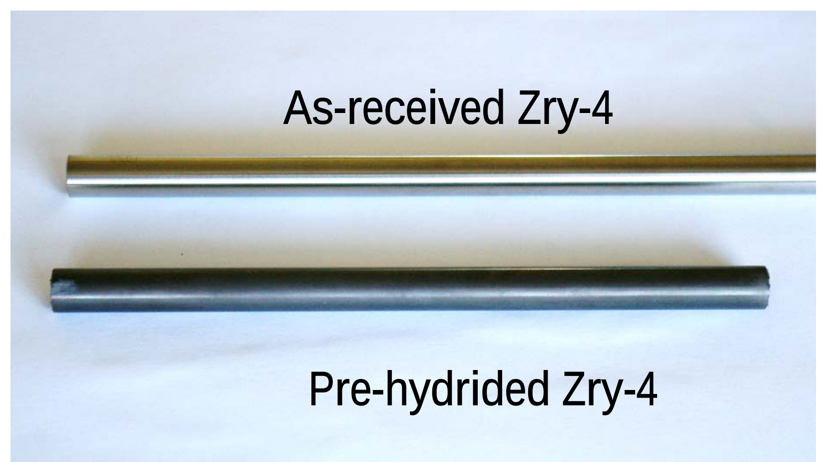



Note: RAMP was controlled manually, during which pressure must be lower than 5 psig for safety purpose.

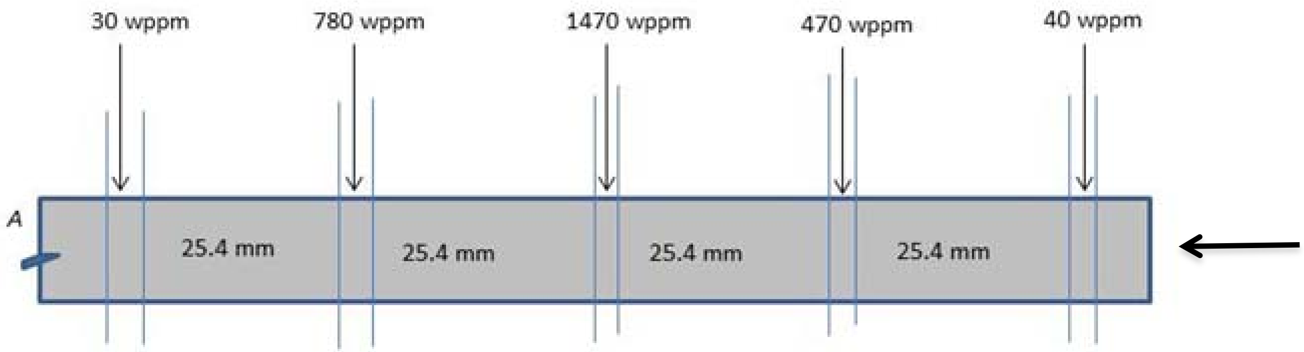
# Optimizing Test Conditions by Thermal Cycles



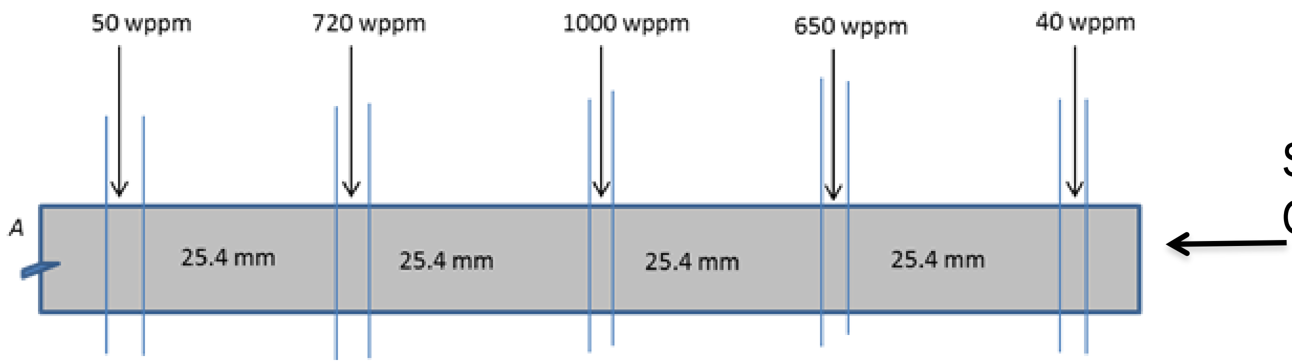

After the samples were initially heated at 400°C for 4 hours, a short thermal cycle (400°C – 120°C - 400°C) was added to obtain uniform hydrogen distribution.



# Improved Hydrogen Distribution By Thermal Cycling



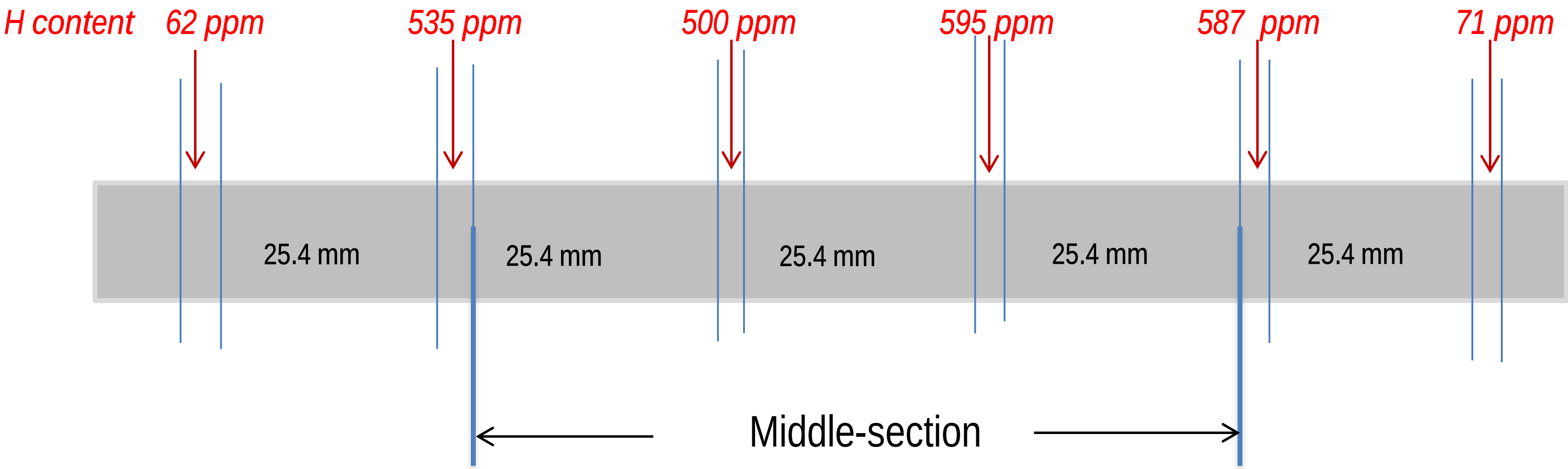

Sample LRR1A:
No cycling




Sample LRR1B:
One cycling

- Sample length ≈5-in long, OD=9.5 mm, ID=8.336 mm
- H Charged at 400°C for 8 hours with mixed gases of hydrogen and argon
- Axial gradients in hydrogen content were reduced by thermal cycling

# Uniform Hydrogen Distribution in Sample Middle-Section




Hydrogen distribution for pre-hydrided Zry-4 sample LRR1D

Sample length ≈6-in long, OD=9.5 mm, ID=8.336 mm

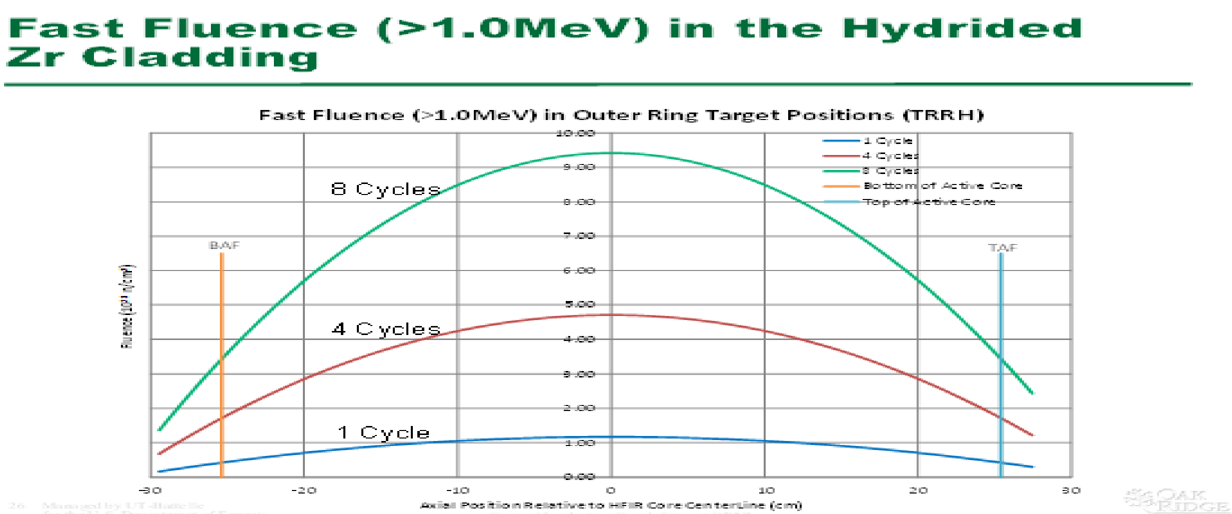
# Characteristics of used fuel rod segments in ORNL Building 3525

| Reactor name | H. B. Robinson | Limerick | Surry | TMI-1 | Calvert Cliffs | Cooper | North Anna | Catawba (MOX) |
|---|---|---|---|---|---|---|---|---|
| Reactor type | PWR | BWR | PWR | PWR | PWR | BWR | PWR | PWR |
| Enrichment, wt % | 2.90 | 3.40-3.95 | 3.1% | 4.00 | 2.45 to 3.04% | 1.33-2.93 | 4.20 | 2.4 to 5% |
| Burnup, GWd/MTU | 63-67 | 54-57 | 36 | 48-50 | 43 | 28 | 63-70 | 40-47 |
| Discharge date | 1995 | 1998 | 1981 | 1997 | 1982 | 1982 | 2004 | 2008 |
| Cladding | Zry-4 | Zr-lined Zry-2 | Zry-4 | Low-Sn Zry-4 | Zry-4 | Zry-2 | M5 | M5 |
| Nominal OD, mm | 10.76 | 11.18 | 10.72 | 10.92 | 11.18 | 14.3 | 9.50 | 9.50 |
| Initial wall thickness, mm | 0.76 | 0.71 | 0.62 | 0.69 | 0.66 | 0.94 | 0.57 | 0.57 |
| OD oxide, µm | ≤100 | ≈10 | <40 | ≤50 | Not provided | Not provided | <20 | <10** |
| Hydrogen pickup, wppm | ≤800 | 70 | <300 | ≤300 | Not provided | Not provided | <120 | <55 |
| Fueled | Yes | Yes | Yes | Yes | Yes | Yes | Yes | Yes |

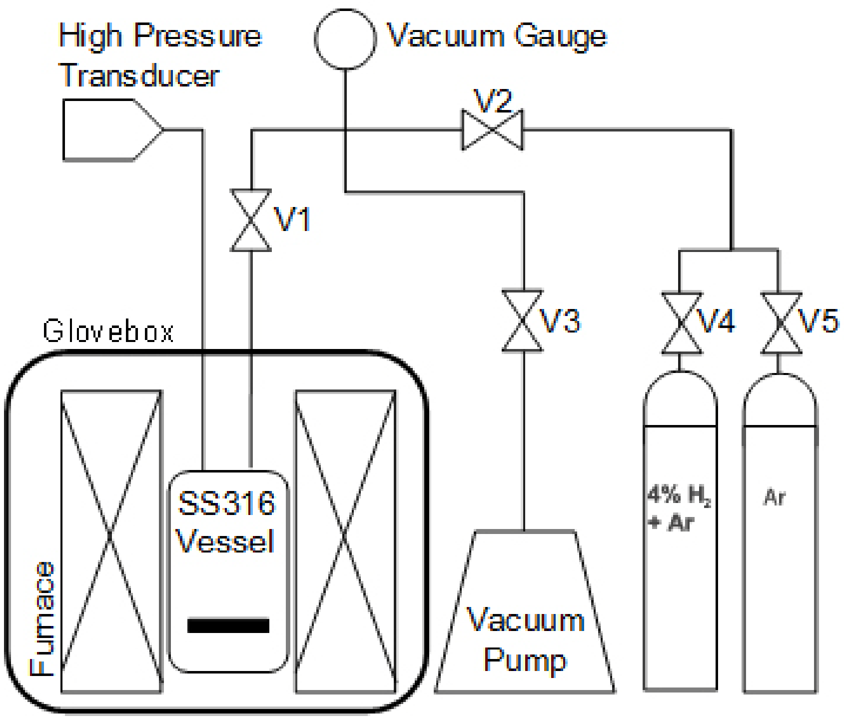
Note: burnup values are rod averaged

# Pre-hydrided Samples for 2011-2012

- Cladding Materials Received: 17x17 Zry-4 (OD=9.50 mm, Wall thickness=0.57 mm)
- Desired Hydrogen Contents:
  - 500 – 800 wppm (similar to the high burnup HBR Zry-4 Cladding)
  - 300 (similar to the high burnup TMI Zry-4 cladding)
- Proposed Sample Length: 1” - 6”-long (shorter samples are optional for small specimens, such as TEM, micro-hardness, expending plug…)
- Need inputs from other Labs/Universities, based on their needs (claddings, H contents, sample lengths, irradiation fluence…)
- Pre-irradiation characterizations and PIE data can be provided to customers



# New Hydrogen Charging System under Static Condition




The system[1] would consist of a) a furnace enclosed inside a glovebox to facilitate gas-solid reactions with tight thermodynamic controls and b) a cathodic charging apparatus to precipitate hydride layers on Zircaloy

- *Static environment (no gas flow) to produce repeatable results.*
- *Optional to be sealed in a glovebox for hydriding irradiated materials.*
- *Ability to process partially or fully hydrided cladding materials supports multiple programs and missions, such as DOE Accident Tolerant Fuel, DOE Used Nuclear Fuel, EPRI round-robin programs.*

[1]K. Terrani, et al., *J. Nucl. Mater.*, **397**, pp.61-68, (2010)

# Discussion

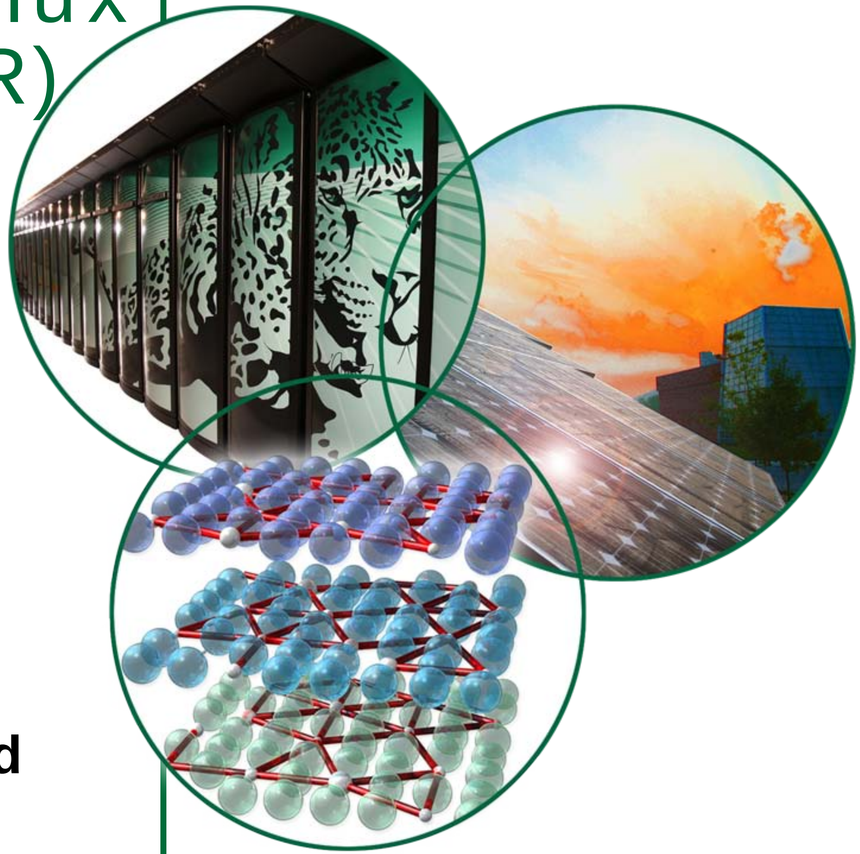
- How important is the hydride rim on waterside?
  - Ductility evaluations on both used fuel cladding (the rim on OD) and irradiated hydrided-cladding (uniformly distributed hydrides across the wall, as well as the rim on OD)
- How do we load cladding with hydrogen to get similar distribution and morphology as in used fuel cladding?
  - Hydride orientation: the same for both used fuel cladding and hydrided cladding
  - Hydride distribution: used fuel -> rim on waterside, current hydrided cladding-> uniform
    - Possible approach to produce the rim: introduce thermal gradient, one-sided hydriding…
- How do loading parameters (time, temperature, atmosphere, etc.) affect hydride formation and behavior?
  - Time: time ↑ -> hydrogen content ↑ (surface cleaning is important)
  - Temperature: ≈400°C ( below the phase transition temperature for Zr alloys and with controlled hydrogen charging rate)
  - Pressure: hydrogen charging near atmosphere
- Do we need to load hydrogen first and then irradiate, irradiate first and then hydrogen load?
  - Irradiating hydrided cladding is easier to handle, compared to hydriding irradiated cladding
  - Validations and direct comparison between hydriding the irradiated cladding and irradiating the hydrided cladding is probably needed.

# Summary

- Hydrided Zry-4 cladding has been fabricated with mixed hydrogen and argon gas flow, but the charged hydrogen content could fluctuate from test to test possibly due to the dynamic gas flow.
- The hydrogen distribution can be improved by optimizing test conditions, and uniform hydrogen distribution was achieved in middle-section of the hydrided samples.
- A new system for hydrogen charging under static conditions (no gas flow) is under consideration in ORNL to produce improved results for hydriding unirradiated and irradiated materials (within a glovebox).
- Future plan:
  - Continue to improve hydriding techniques (morphology, repeatability…).
  - Perform ductility evaluations on both used fuel cladding and HFIR irradiated hydrided cladding (the materials might be provided to others for Round-robin tests).
  - If needed, conduct direct comparison between hydriding the irradiated cladding and irradiating the hydrided cladding through ductility evaluations and microstructural examinations (Metallography, SEM, Micro-hardness, TEM…).

ATTACHMENT 4

# Neutron Irradiation of Hydrided Cladding Material in the High Flux Isotope Reactor (HFIR)




**R.L. Howard[1], Y. Yan[2], L. J. Ott[1] and J. L. McDuffee[1]**

**[1]Reactor and Nuclear Systems Division**

**[2]Fuel Cycle and Isotopes Division**


**Presented at the UFD Campaign Clad Testing Workshop**
**15-17 November 2011**
**Las Vegas, Nevada**

U.S. DEPARTMENT OF ENERGY

OAK RIDGE NATIONAL LABORATORY
MANAGED BY UT-BATTELLE FOR THE DEPARTMENT OF ENERGY

# Motivation

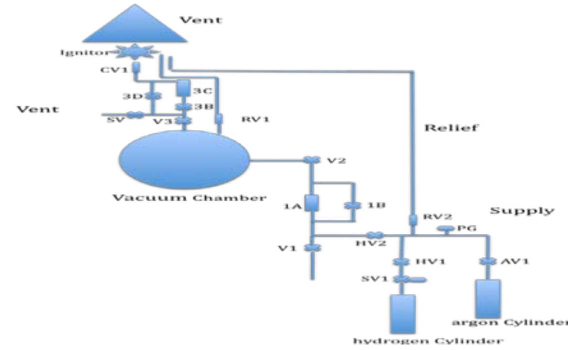
| Cladding Gaps | | |
|---|---|---|
| Degradation Mechanism/Process | Importance of R&D | Approach |
| Annealing of radiation damage | Medium | Long-term, low temperature annealing will be analyzed through advanced modeling and simulation with some *experimental* work to support the model |
| H2 effects: embrittlement and reorientation | High | A comprehensive *experimental* and modeling program to examine the factors that influence hydride reorientation will be performed with a focus on new cladding materials and high burnup fuels. Additional *experimentation* and modeling to provide the link between unirradiated and irradiated cladding performance will be initiated |
| H2 effects: Delayed Hydride Cracking | High | *Experimental* work combined with modeling will be initiated |
| Oxidation | Medium | *Experimental* work to determine the mechanism for the rapid cladding oxidation observed will be initiated |
| Creep | Medium | Long-term, low temperature , low-strain creep will be analyzed through advanced modeling and simulation with some *experimental* work to support the model |

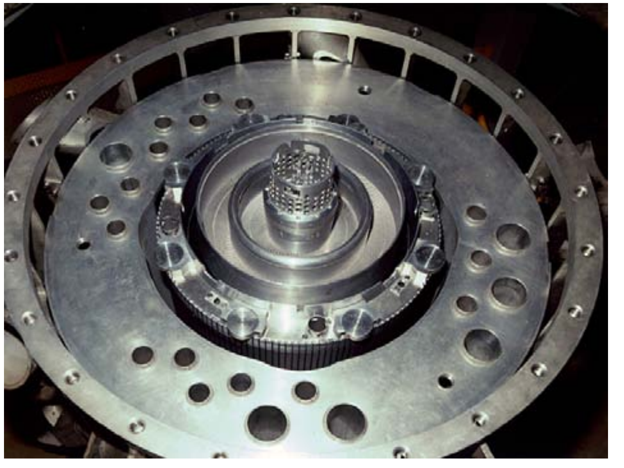
# Motivation

- Gap Closure Requires Materials for Testing
- Actual High Burn Up Clad Material  Difficult to Obtain
  - DOE Order 435.1 “Radioactive Waste Management”
  - Logistics associated with handling and transportation
- Wide Range of Used Fuel Cladding Types, Condition and Properties
- Separate Effects Testing
- Workaround: Produce Surrogate Materials

# Neutron Irradiation of Hydrided Cladding Material in HFIR

- **Pre-hydride unirradiated clad tubing samples**
- **Materials Characterization**
- **Fast Neutron irradiation of Samples in High Flux Isotope Reactor (HFIR)**
- **Samples free of Alpha Contamination Provided to Participating National Lab, University, Other Researchers**



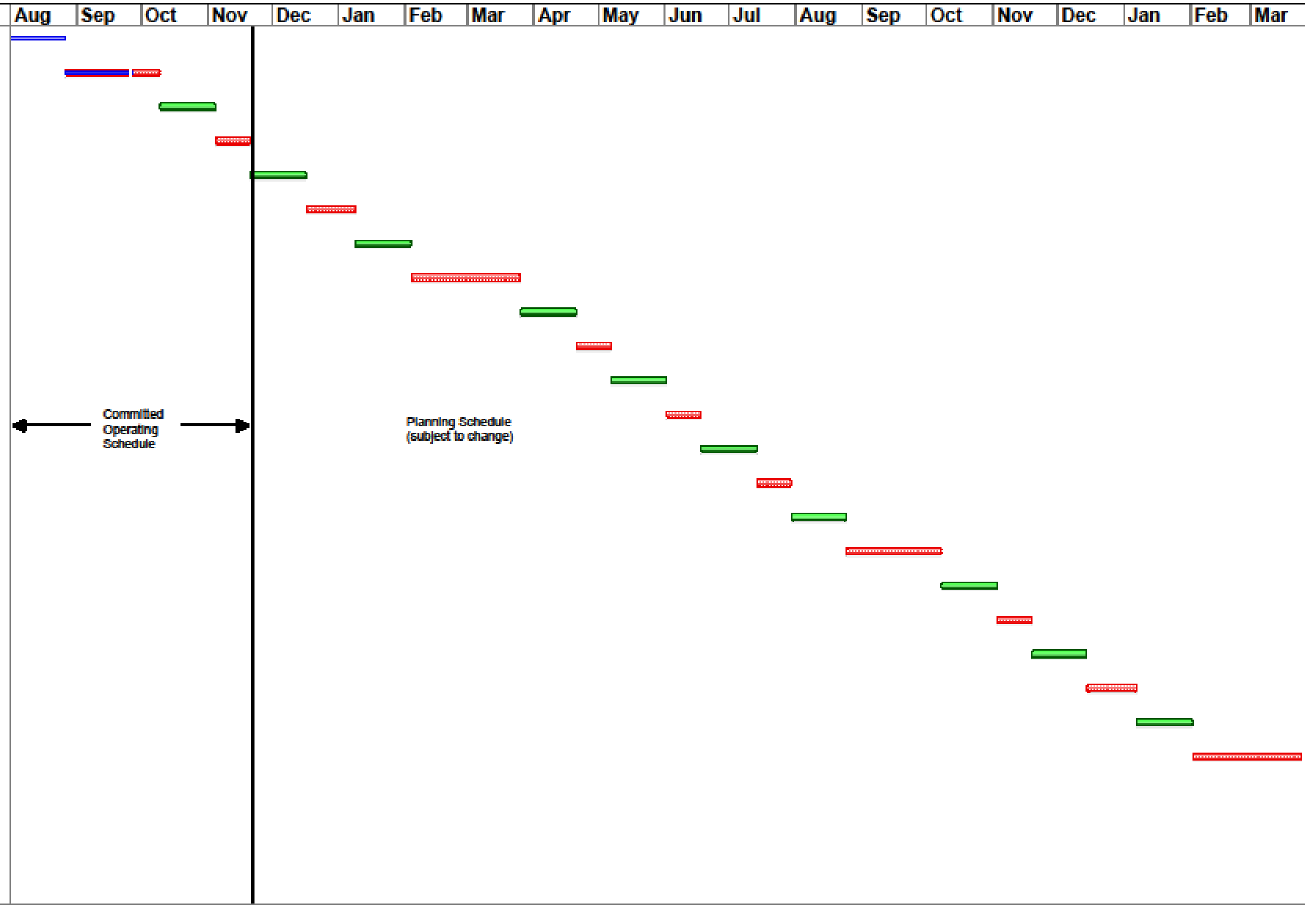



# Range of Materials and Conditions for Irradiation (Collaboration Required)

| | | |
|---|---|---|
| Irradiation temperature | 200–250°C | Service temperature of the cladding for BWR |
| | 300–350°C | Service temperature of the cladding for PWR |
| Typical sample size | Cladding OD: 9.5–10 mm | Varies with cladding type |
| | Wall-thickness: 0.60–0.76 mm | Varies with cladding type |
| | Length: 25–75 mm | Limitations apply, Researchers should articulate needs |
| Neutron fluence (>1 MeV) | Ranges from around $1.0 \times 10^{21}$ n/cm$^2$ to around $2.3 \times 10^{21}$ n/cm$^2$<br>Depends on Researcher Needs<br>Larger Fluence Will Require Longer Time in HFIR | |
| Materials | Zry-2, Zry-4, M5, and ZIRLO<br>•Zry-4 used in first batch<br>•Need additional materials | |
| Total length | Dependent on Experimental Needs | Irradiated in separated core locations; cycle start times and durations will vary. |

# Start Irradiation in March

| Cycle | Start | Finish | Duration |
|---|---|---|---|
| 437 | Mon 8/1/11 | Fri 8/26/11 | 25.74 days |
| 437 EOC | Sat 8/27/11 | Mon 10/10/11 | 44 days |
| 438 | Mon 10/10/11 | Sat 11/5/11 | 26 days |
| 438 EOC | Sat 11/5/11 | Mon 11/21/11 | 16 days |
| 439 | Mon 11/21/11 | Sat 12/17/11 | 26 days |
| 439 EOC | Sat 12/17/11 | Mon 1/9/12 | 23 days |
| 440 | Mon 1/9/12 | Sat 2/4/12 | 26 days |
| 440 EOC | Sat 2/4/12 | Mon 3/26/12 | 51 days |
| 441 | Mon 3/26/12 | Sat 4/21/12 | 26 days |
| 441 EOC | Sat 4/21/12 | Mon 5/7/12 | 16 days |
| 442 | Mon 5/7/12 | Sat 6/2/12 | 26 days |
| 442 EOC | Sat 6/2/12 | Mon 6/18/12 | 16 days |
| 443 | Mon 6/18/12 | Sat 7/14/12 | 26 days |
| 443 EOC | Sat 7/14/12 | Mon 7/30/12 | 16 days |
| 444 | Mon 7/30/12 | Sat 8/25/12 | 26 days |
| 444 EOC | Sat 8/25/12 | Mon 10/8/12 | 44 days |
| 445 | Mon 10/8/12 | Sat 11/3/12 | 26 days |
| 445 EOC | Sat 11/3/12 | Mon 11/19/12 | 16 days |
| 446 | Mon 11/19/12 | Sat 12/15/12 | 26 days |
| 446 EOC | Sat 12/15/12 | Mon 1/7/13 | 23 days |
| 447 | Mon 1/7/13 | Sat 2/2/13 | 26 days |
| 447 EOC | Sat 2/2/13 | Mon 3/25/13 | 51 days |




Operation Outage Complete Milestone

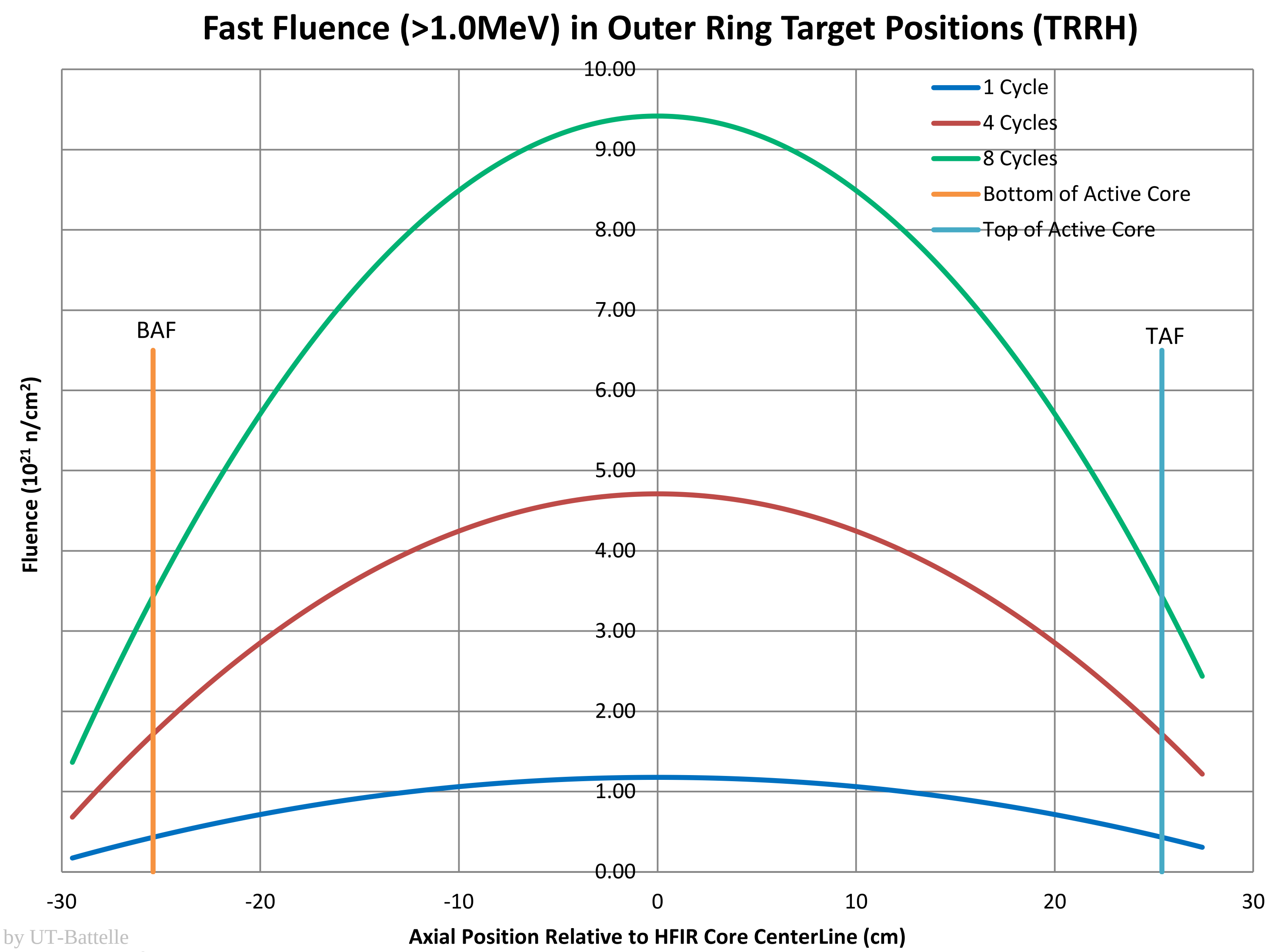
Mon 9/26/11

**HFIR Operating Forecast & Planning Schedule**

# Some Validation/ Benchmarking Choices

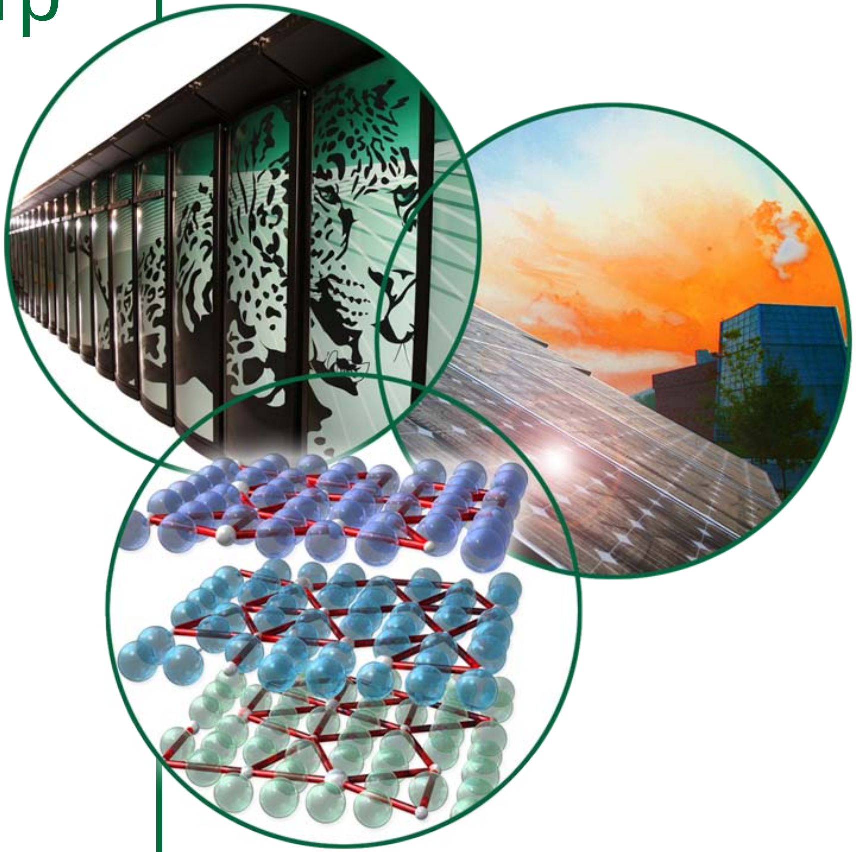
| Reactor name | H. B. Robinson | Limerick | Surry | TMI-1 | Calvert Cliffs | Cooper | North Anna | Catawba (MOX) |
|---|---|---|---|---|---|---|---|---|
| **Reactor type** | PWR | BWR | PWR | PWR | PWR | BWR | PWR | PWR |
| **Enrichment, wt %** | 2.90 | 3.40-3.95 | 3.1% | 4.00 | 2.45 to 3.04% | 1.33-2.93 | 4.20 | 2.4 to 5% |
| **Burnup, GWd/MTU** | 63-67 | 54-57 | 36 | 48-50 | 43 | 28 | 63-70 | 40-47 |
| **Discharge date** | 1995 | 1998 | 1981 | 1997 | 1982 | 1982 | 2004 | 2008 |
| **Cladding** | Zry-4 | Zr-lined Zry-2 | Zry-4 | Low-Sn Zry-4 | Zry-4 | Zry-2 | M5 | M5 |
| **Nominal OD, mm** | 10.76 | 11.18 | 10.72 | 10.92 | 11.18 | 14.3 | 9.50 | 9.50 |
| **Initial wall thickness, mm** | 0.76 | 0.71 | 0.62 | 0.69 | 0.66 | 0.94 | 0.57 | 0.57 |
| **OD oxide, µm** | ≤100 | ≈10 | <40 | ≤50 | Not provided | Not provided | <20 | <10** |
| **Hydrogen pickup, wppm** | ≤800 | 70 | <300 | ≤300 | Not provided | Not provided | <120 | <55 |
| **Fueled** | Yes | Yes | Yes | Yes | Yes | Yes | Yes | Yes |

# Fast Fluence (>1.0MeV) in the Hydrided Zr Cladding



ATTACHMENT 5

# Material Irradiation in HFIR and Conceptual Design for High Burnup Cladding Simulation




L. J. Ott and J. L. McDuffee

Thermal Hydraulics & Irradiation Engineering Group
Reactor and Nuclear Systems Division
Oak Ridge National Laboratory


**Presented at the UFD Campaign Clad Testing Workshop**
**15-17 November 2011**
**Las Vegas, Nevada**

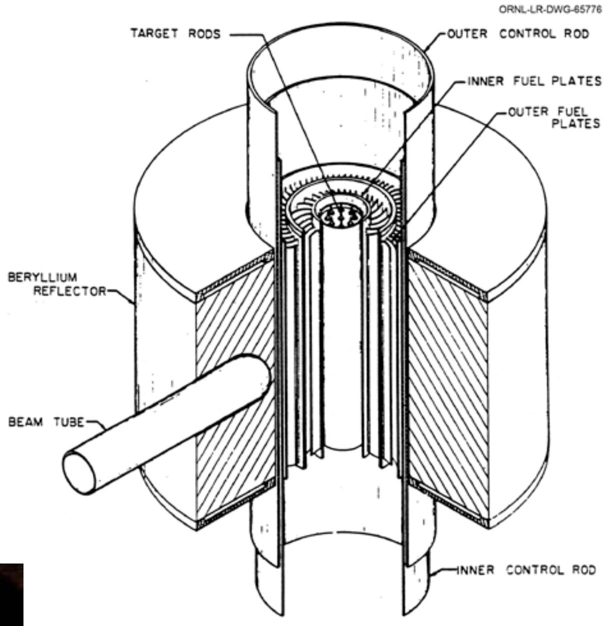
# The High Flux Isotope Reactor

- **Versatile 85 MW reactor**
- **Highest Thermal Flux in the Western World**
  - **$2.5x10^{15}$ n/cm$^2$-s Thermal**
  - **$1.2x10^{15}$ n/cm$^2$-s >0.1MeV**
- **Operation**
  - **Nominally 7-8 cycles/year**
  - **23-26 day cycle length**
  - **Typical EOC outage ~21 days**

# The U.S. Department of Energy's High Flux Isotope Reactor Is Located at The Oak Ridge National Laboratory

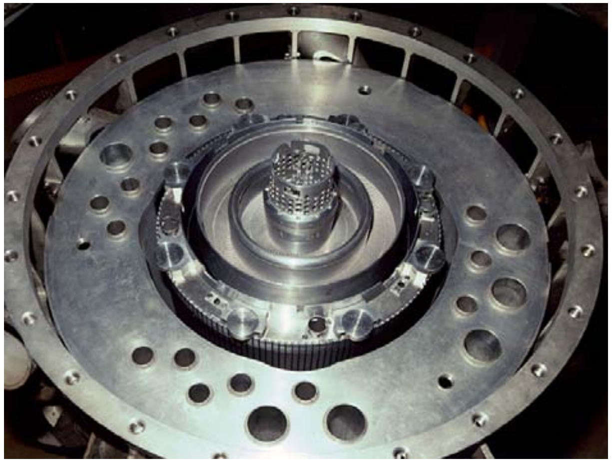
### Purpose/Missions

- **Production of medical isotopes and transuranic isotopes**
- **Materials test irradiation experiments**
- **Neutron-scattering experiments (includes cold-source, low-temperature neutron capabilities)**
- **Neutron activation analyses**
- **Fuel/cladding irradiation experiments**




### HFIR

- **Beryllium reflected**
- **Pressurized**
- **Pressurized, light water cooled and moderated**
- **Center flux trap**
- **Nominal power of 85 MWt**



### Core

- **Two concentric annular regions**
- **Aluminum-clad involute fuel plates**
- **HEU fuel - $U_3O_8$ dispersed in aluminum**
- **Active height of ~61 cm**
- **Flux Trap, ~12.7 cm in diameter**
- **Outer fueled region, ~43.5 cm in diameter**
- **Active core height of 50.8 cm**

# Top View of Reactor and Irradiation Sites

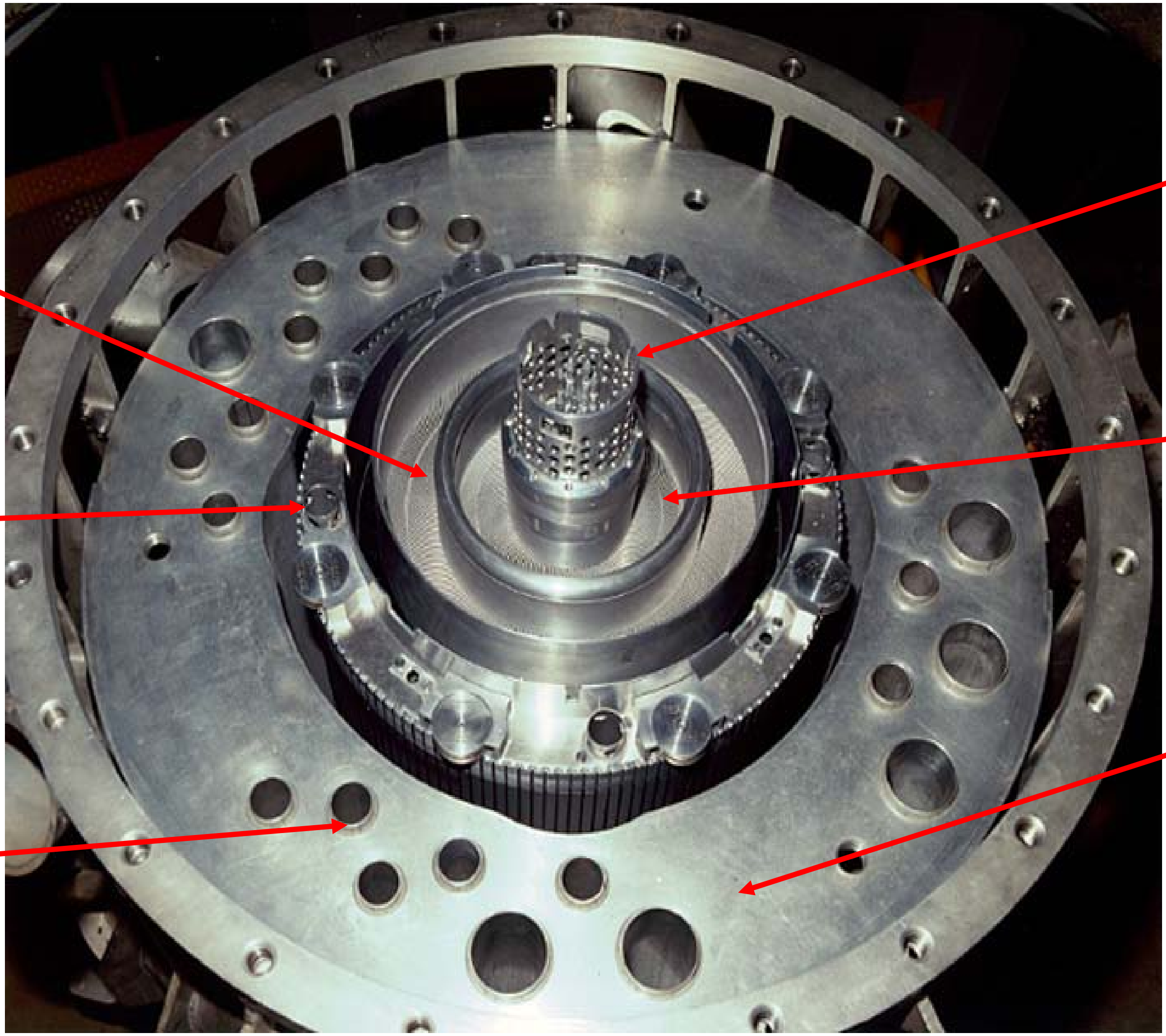
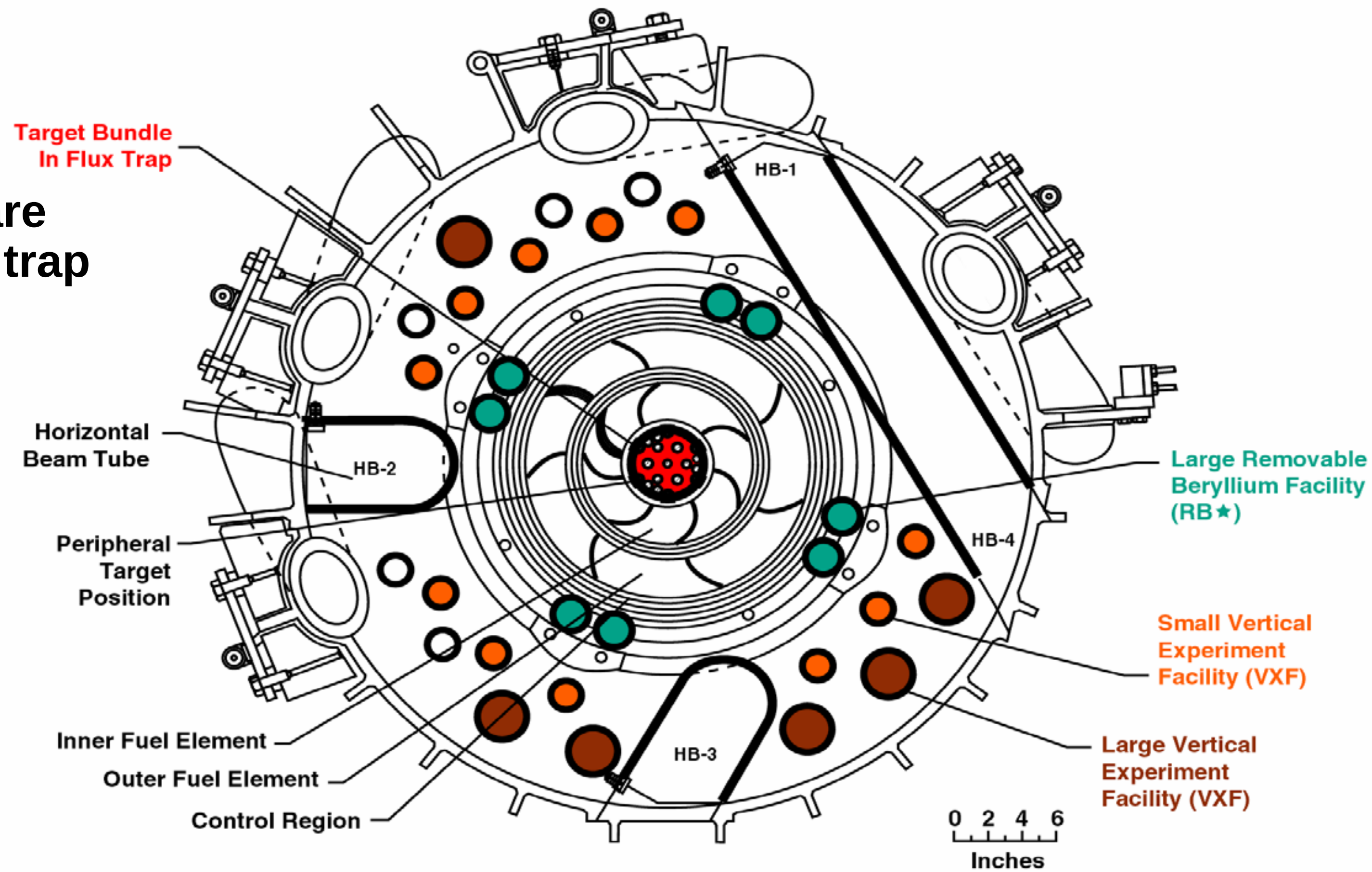



Photo of mockup with old reflector

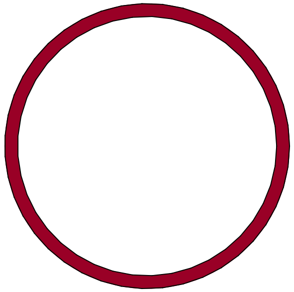
# HFIR Experimental Locations

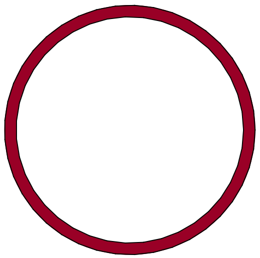
- **28-30 target pins are located in the flux trap (~1.27 cm OD)**
- **8 RB* positions (~5.07 cm OD)**
- **16 small VXF sites (~4.02 cm OD)**
- **6 large VXF sites (~7.2 cm OD)**




For description of HFIR Experimental Facilities, visit http://neutrons.ornl.gov/hfir/hfir_experiment.shtml

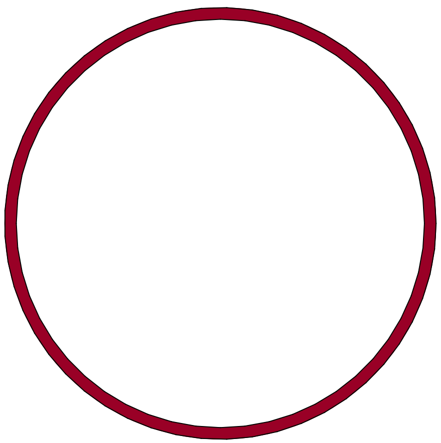
# Cross Section Through HFIR Midplane



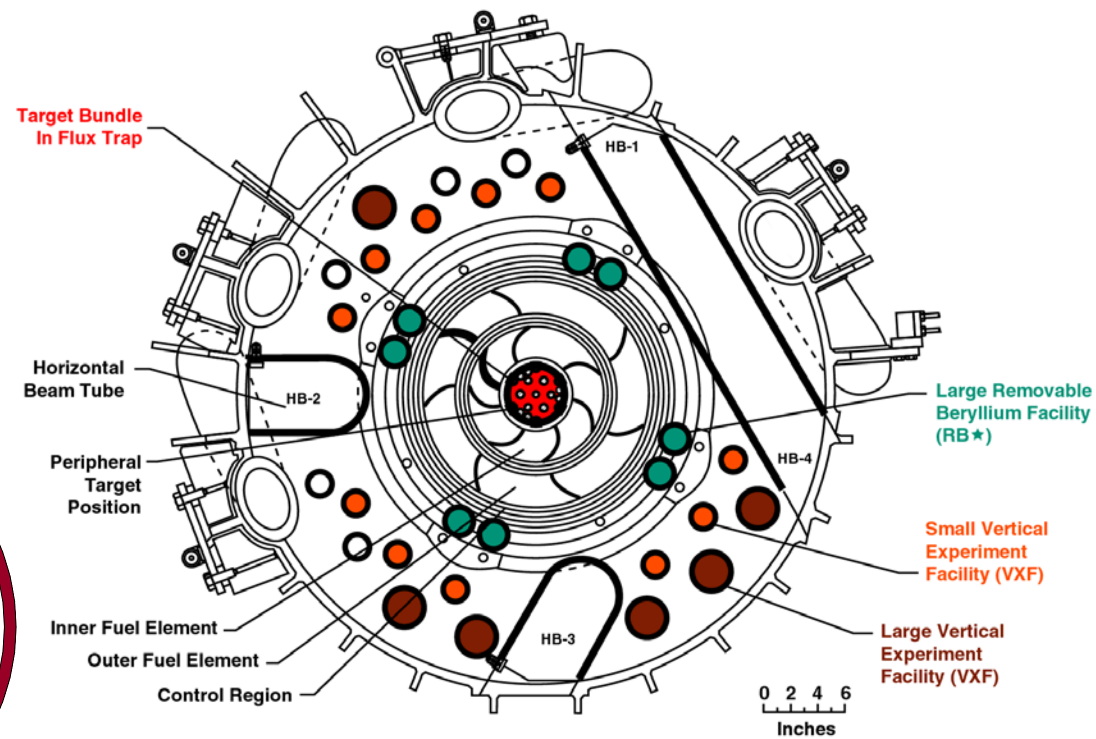


RB* Position
(1.812" id)

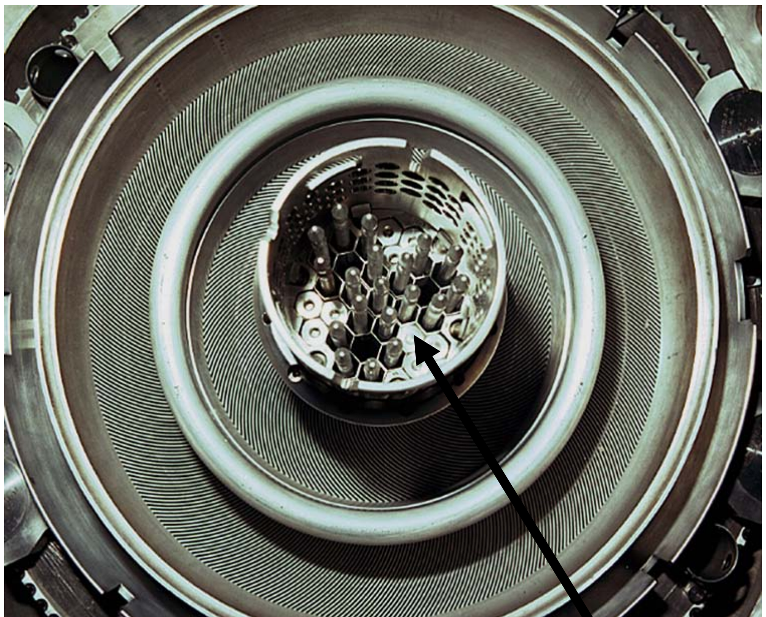
Small VXF
(1.583" id)

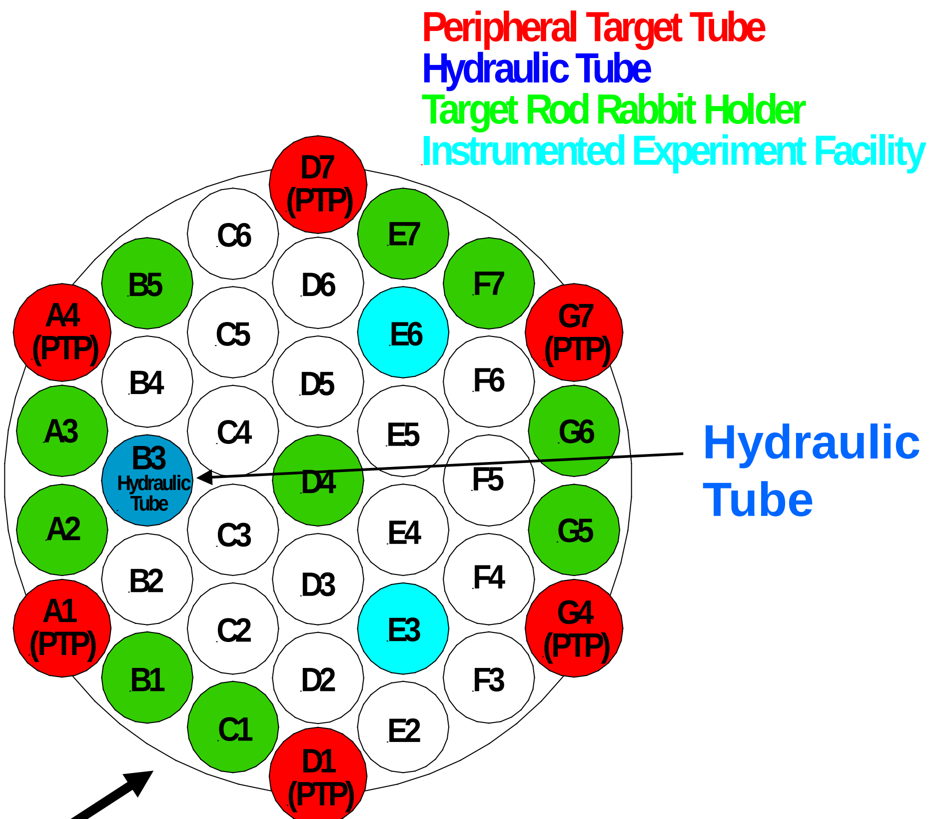


Large VXF
(2.833" id)



# Flux Trap Rabbit Capsule Locations





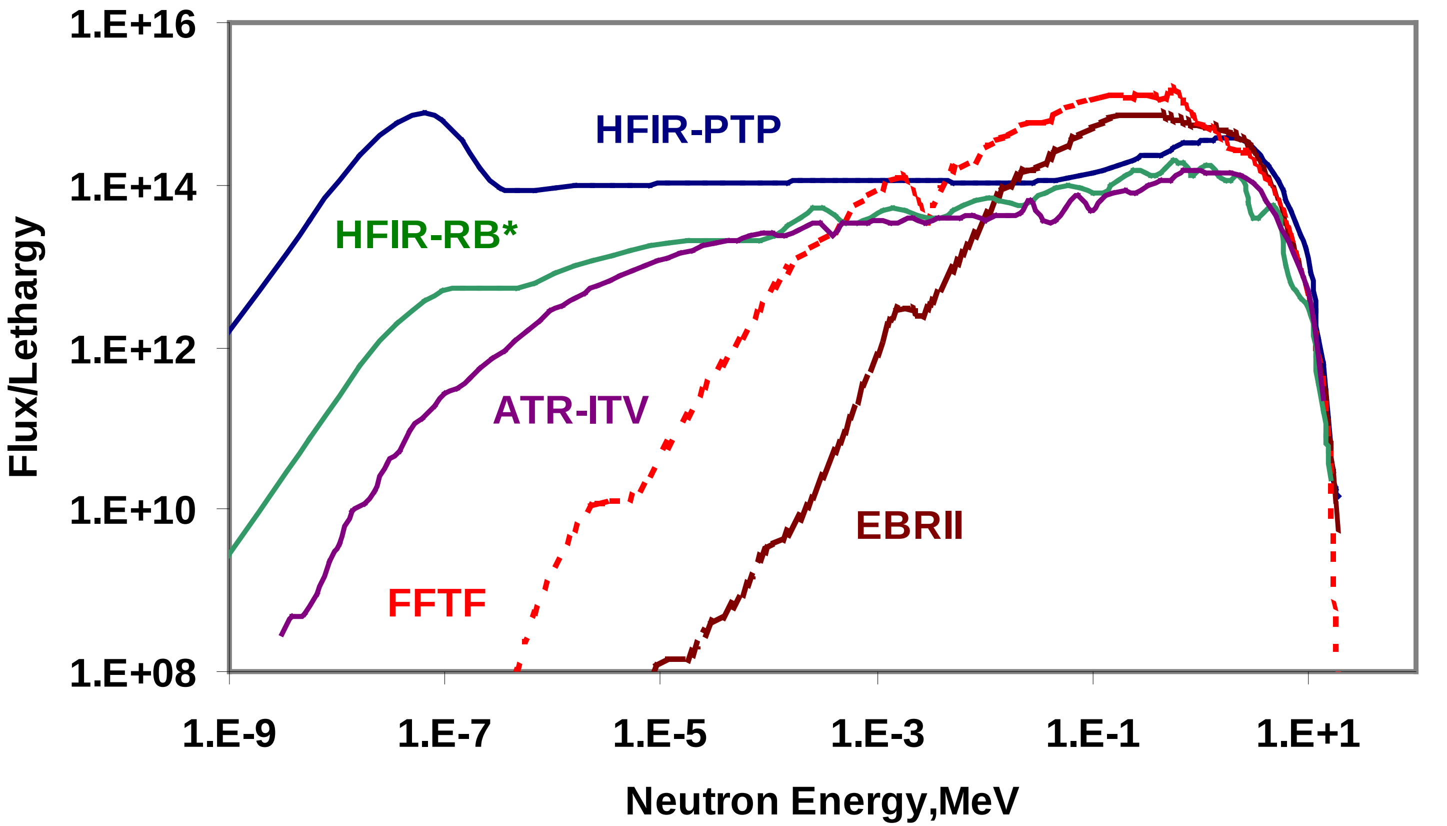

***Flux Trap***

- Hydraulic tube allows very low doses
- Fast Flux ($10^{14}$ n/cm$^2$·s)
  - PTP: 4.5 — 11
  - TRRH: 6.5 — 11
  - HT: 3.6 — 8.9

# Characteristics of Major Irradiation Facilities in HFIR

| | Target | RB* | Small VXF | Large VXF |
|---|---|---|---|---|
| Fast flux, E > 0.1 MeV ($10^{18}$ n/m$^2$-s; $10^{14}$ n/cm$^2$-s) | 11 | 5.3 | 0.51 | 0.13 |
| Peak displacements per atom (dpa) per calendar year (stainless steel) | 12.6 | 4.7 | 0.45 | 0.12 |
| Thermal flux ($10^{18}$ n/m$^2$-s; $10^{14}$ n/cm$^2$-s) | 20 | 9.7 | 7.5 | 4.3 |
| Gamma heating (W/g SS) | 46 | 16 | 3.3 | 1.7 |
| Typical capsule diameter (mm) | 13 | 43 | 37 | 69 |
| Number of available positions | 36 | 8 | 16 | 6 |

# Neutron Spectra Comparisons

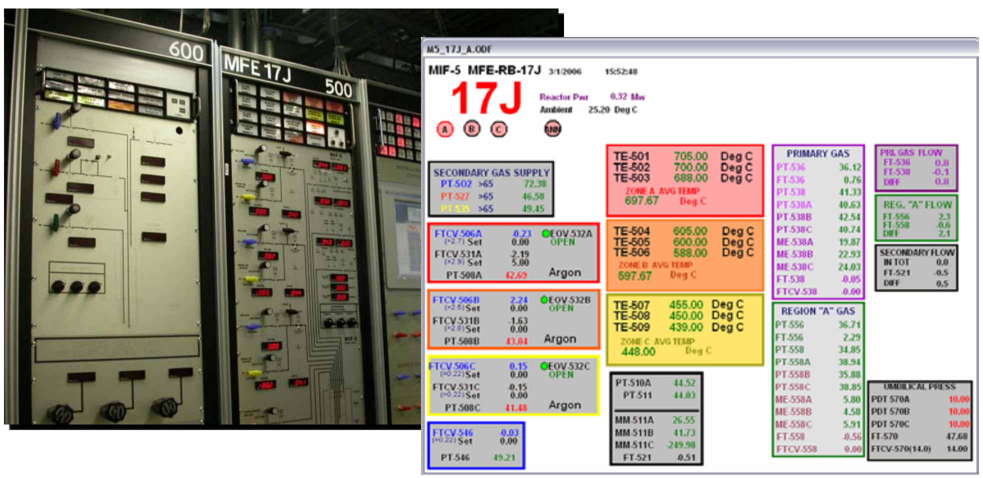



- HFIR RB* and ATR-ITV thermal flux time dependent

From L. R. Greenwood, PNNL

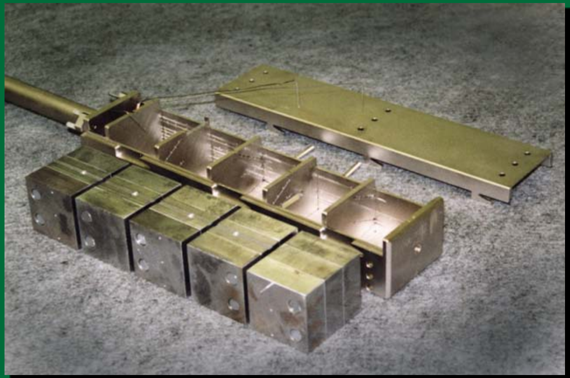
# Two Major Facilities Support Irradiation Experiment Construction and Operation

- **Materials Irradiation Facility**
  - **Monitor and control of experiment conditions in real time during irradiation at ORNL High Flux Isotope Reactor**
  - **Web-based interface allows PIs to view progress of the experiment**
- **Capsule Assembly Laboratory**
  - **Facilities for assembling and performing acceptance testing of irradiation experiments**
  - **Integrated welding facility**
  - **Vacuum test equipment**
  - **Hydrotest equipment**
  - **Test pit for acceptance testing of long experiments**




# Multiple Experimental Formats Are Used

## Reusable experiment designs



## Rabbits

- Small scale and “inexpensive”
- Located in the HFIR flux trap

## Target capsules

- Larger scale either instrumented or uninstrumented
- Located in the HFIR flux trap

## RB capsules

- Large-scale instrumented capsules
- Located in the HFIR removable reflector region

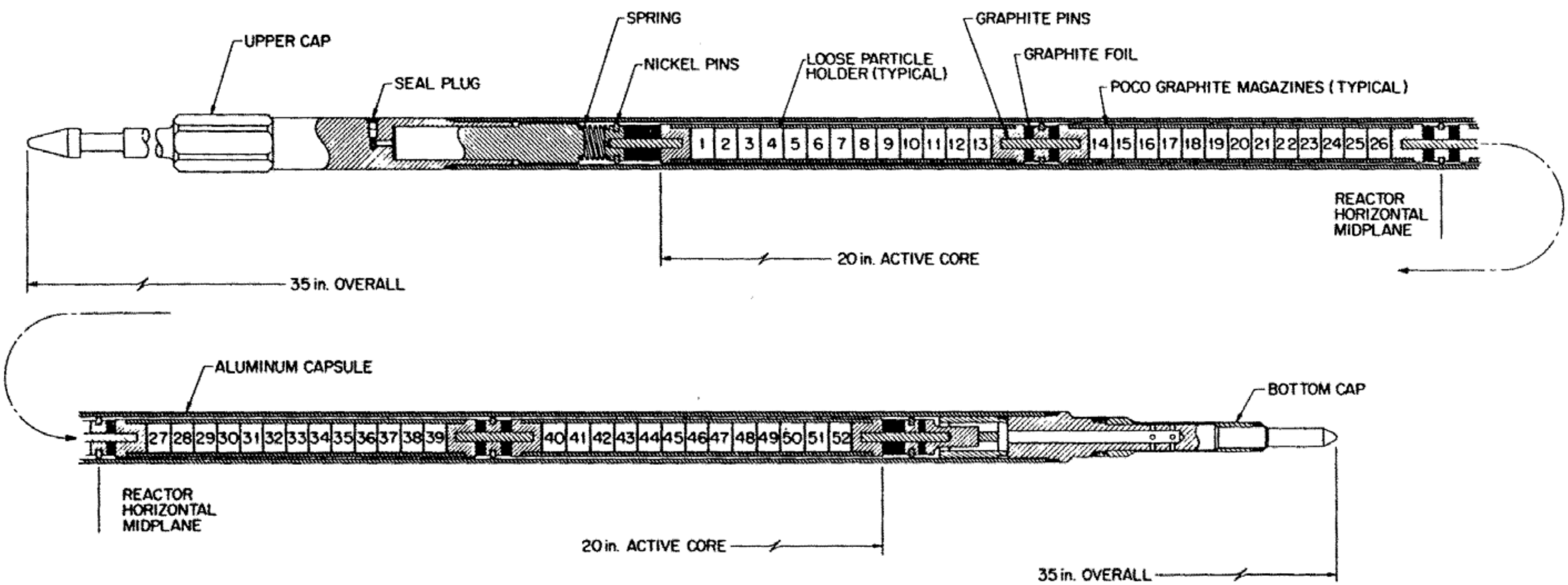
## Fuel irradiation

# Rabbit Capsules Offer Low Cost, High Flexibility

- **Small  (0.375-in. ID by 2.3 in. long)**
- **Relatively inexpensive**
- **Irradiation times from seconds to years**
- **Temperatures from 60  C to 1500  C**
- **Highest flux position in HFIR**

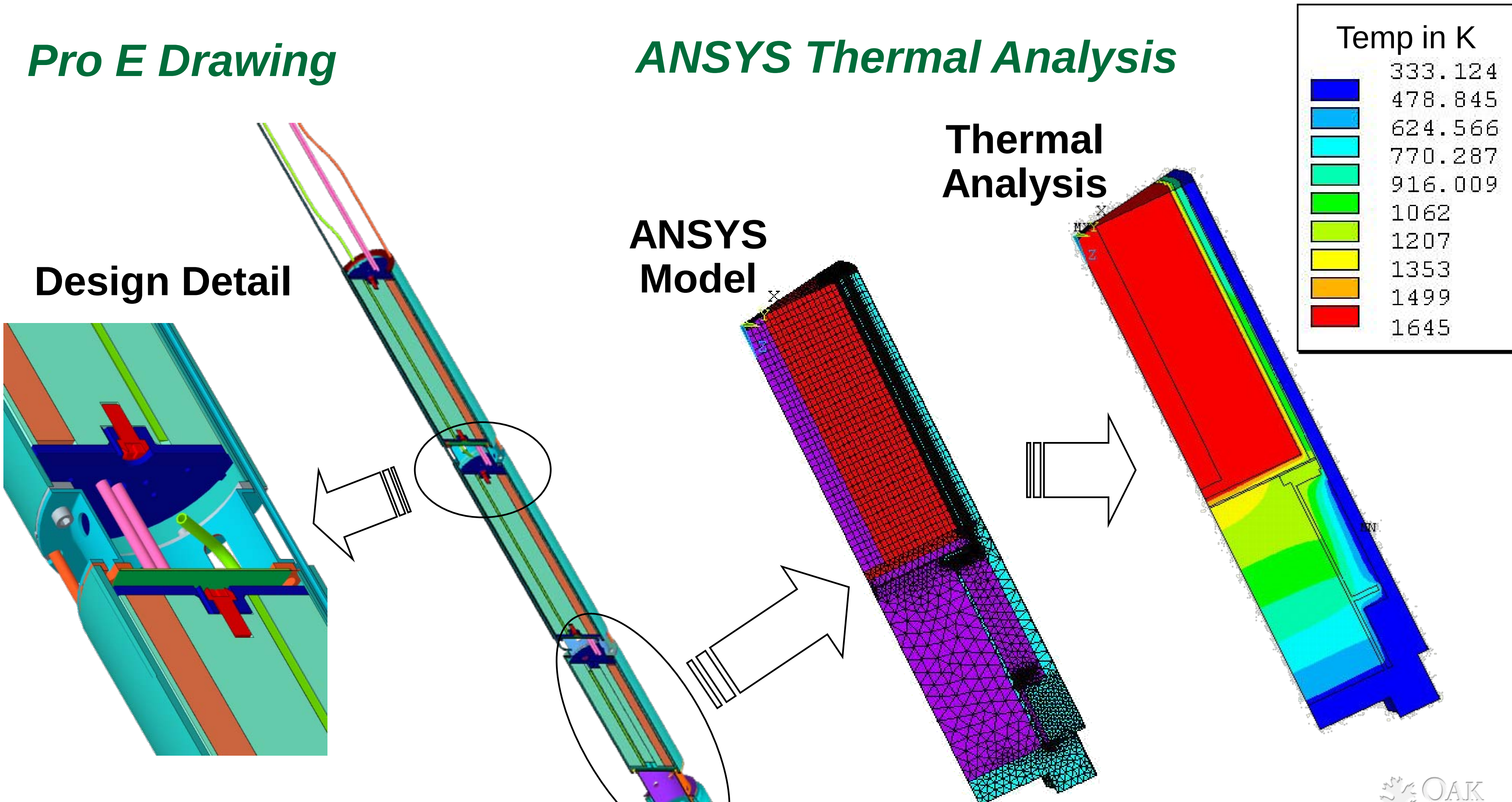
# Uninstrumented Target Capsules Offer Larger Volumes With the Same High Flux of Rabbit Capsules

- Two standard designs – 0.444 in. or 0.531 in. ID by 20 in. long
- Irradiation temperatures from 200  C to 1500  C



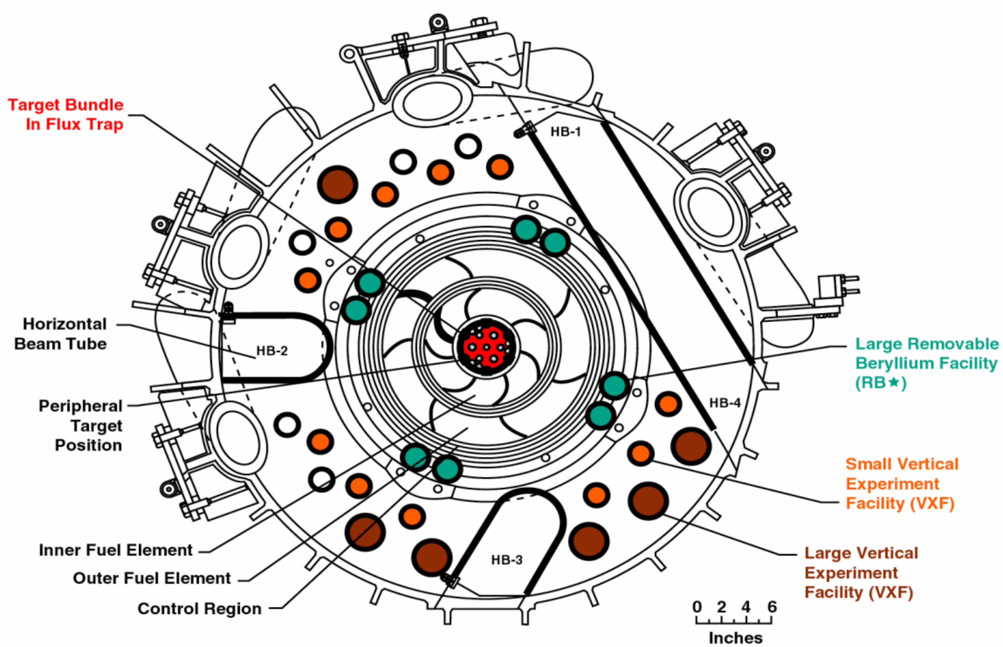
# Advanced CAD and Thermal Modeling Tools Used for Experiment Design

- Pro/Engineering CAD software used to develop 3-D models
- ANSYS multi-physics analysis code used for thermal design

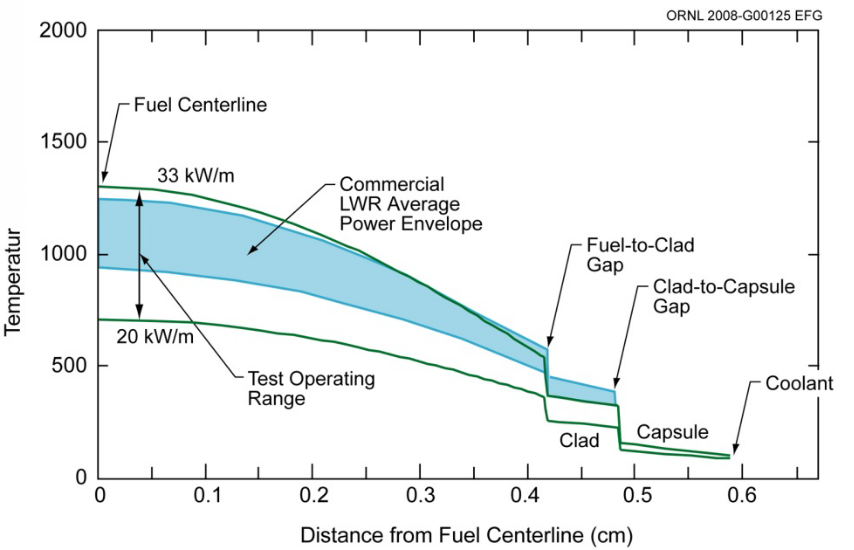


# Examples of Fuel / Cladding Experiments Currently Being Irradiated in HFIR

# 1) The HFIR LWR Fuel Irradiation Facility Provides Prototypic LWR Conditions (LHGRs, fuel/cladding temperatures, clad fluence)



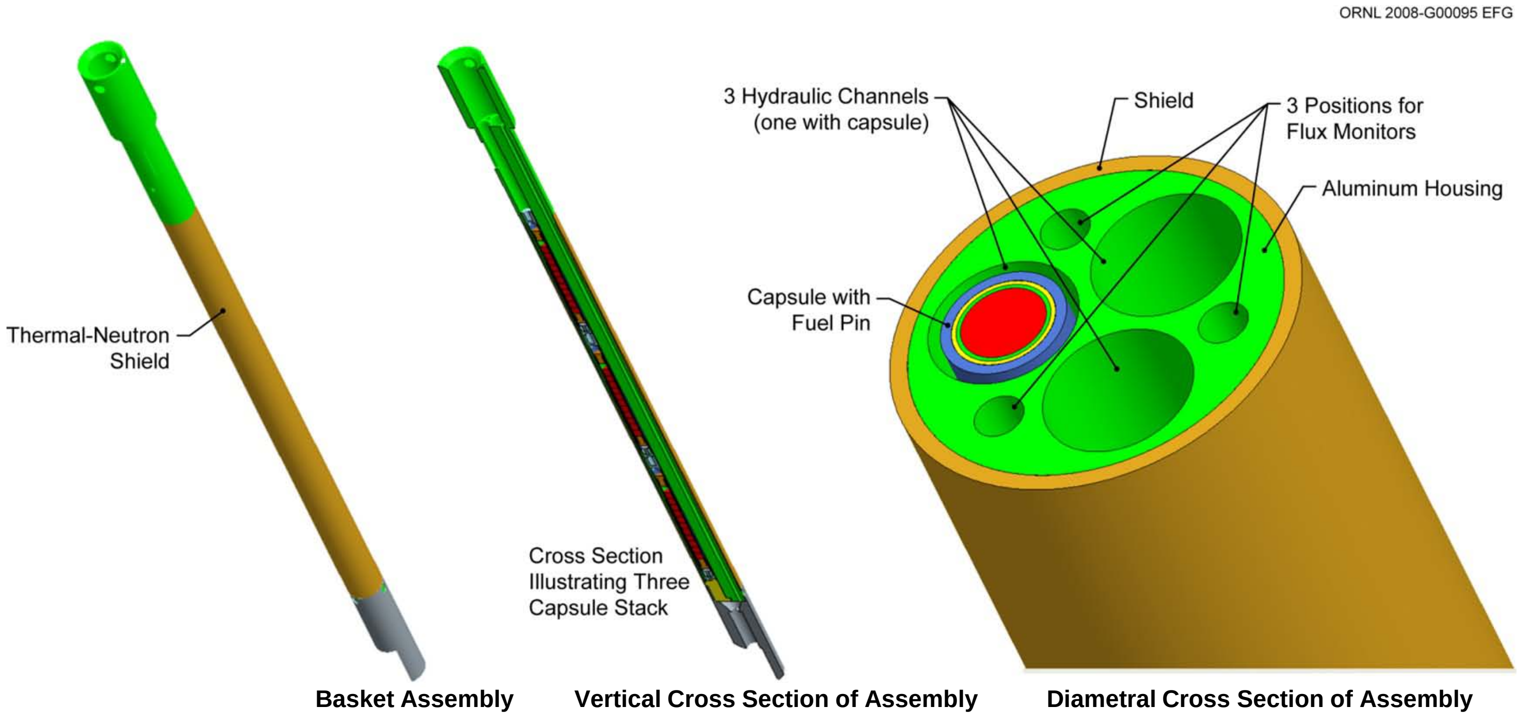

- Small vertical VXF positions
- U(CN) fuel clad in SiC,
  7 capsules, started irradiation
  in Summer 2010
- UO2 fuel clad in SiC,
  9 capsules, started irradiation
  in Fall 2010





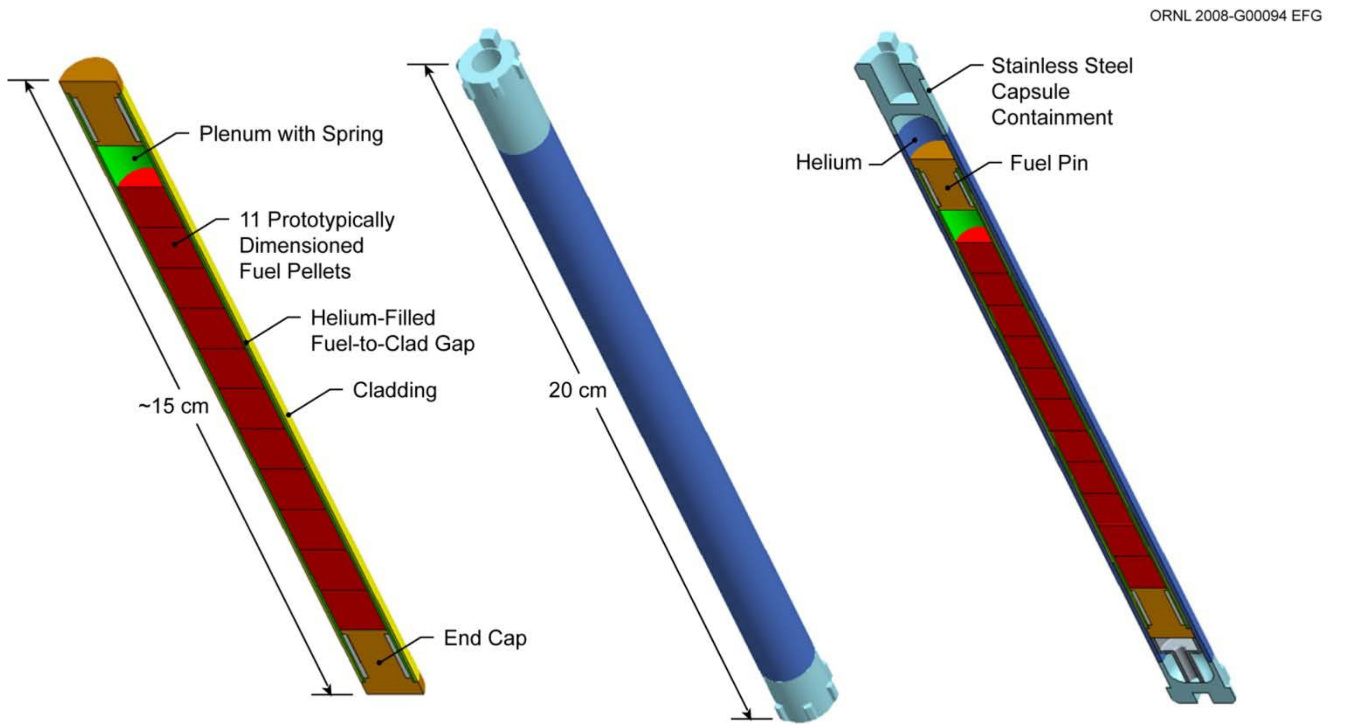

ORNL 2008-G00113_15 EFG

# 1) LWR Experimental Facility Design for the Small Vertical VXF Position




- **Allows simultaneous irradiation of nine capsules**
  - **Three hydraulic channels**
  - **Three capsules (stacked) per channel**
- **Incorporates a thermal-neutron attenuation shield**
- **Assembly easily removed/reinserted in HFIR**
- **Allows removal, replacement, and shuffling of capsules**

# 1) LWR Experimental Facility Design for the Small Vertical VXF Position (continued)



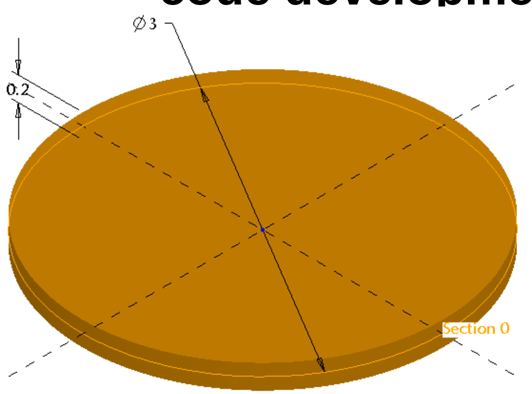

**Fuel Pin** | **Stainless Steel Capsule Containment** | **Vertical Cross Section of Capsule/Fuel Pin**

- **Fuel pin**
  - **Prototypic diametral dimensions (17 x 17 PWR fuel assembly)**
  - **Supplied by commercial team members in this ORNL project**
  - **Encapsulated in stainless steel containment**
    - **Required by HFIR technical specifications**
    - **Appropriately designed cladding-to-containment gas gap yields prototypic cladding surface temperatures**

# 2) Low Fluence Irradiation Experiments Using the HFIR Hydraulic Rabbit Facility

- **Scope: Perform irradiation experiments using the High Flux Isotope Reactor (HFIR) Hydraulic Rabbit Facility to support fuel, target, and materials development for the Advanced Fuels Campaign.**

- **Objectives: Perform irradiation experiments using HFIR Hydraulic Rabbit Facility suitable to**
  - **Investigate the early stages of the microstructural evolution of metal fuel materials as a function of elemental composition, temperature and fluence**
  - **Secure fundamental data to support fuel modeling and simulation code development**

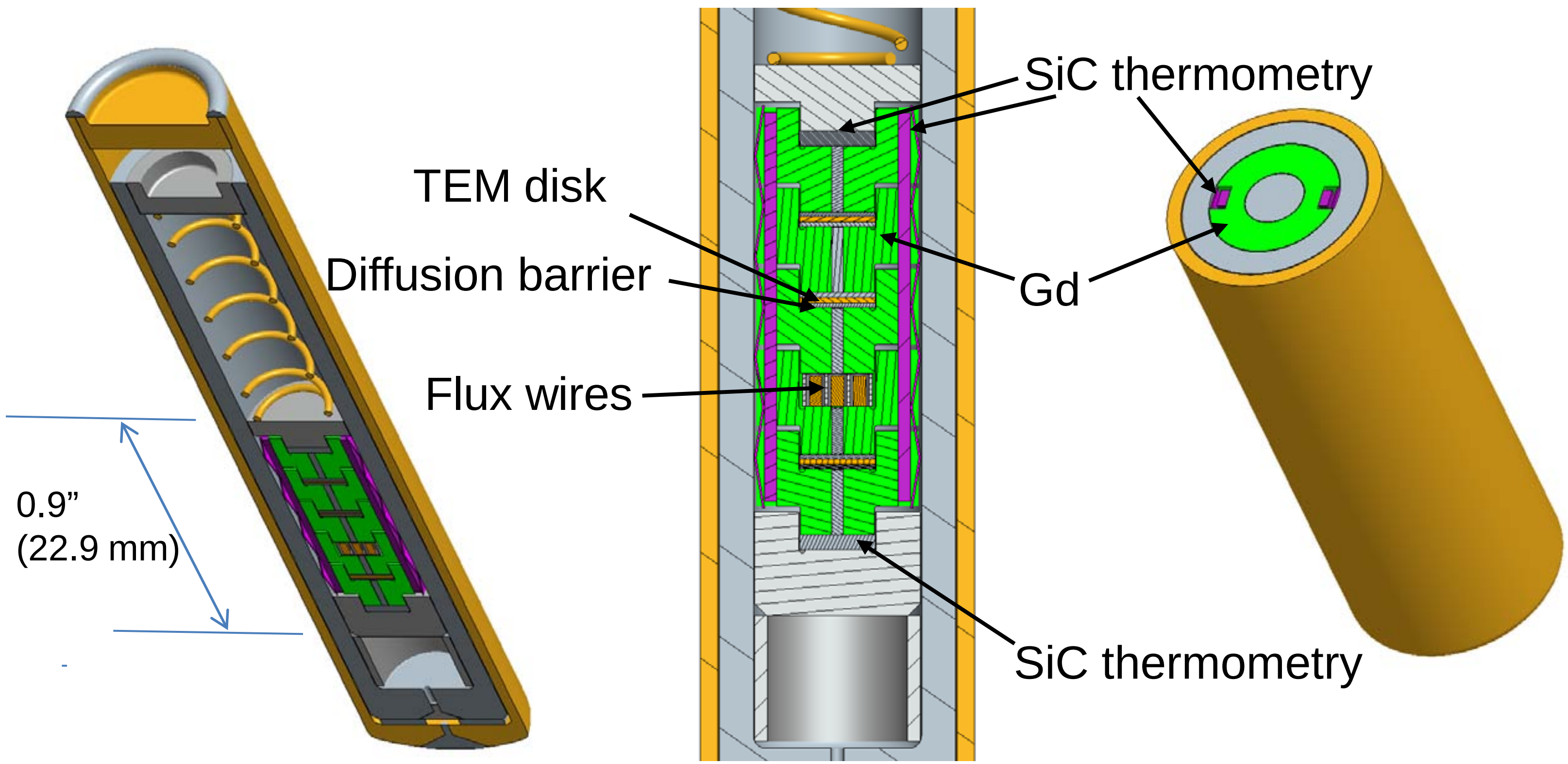



| | | | | |
|---|---|---|---|---|
| ***U*** | ***U-10Zr*** | ***U-20Pu*** | ***U-Pu-Zr*** | ***U-Pu-Zr-Am*** |
| ***Pu*** | ***U-20Zr*** | ***U-30Pu*** | ***U-Pu-Mo*** | ***U-Pu-Zr-Np*** |
| ***Am*** | ***U-30Zr*** | ***U-10Mo*** | ***U-Am*** | ***EBR II fuel*** |
| ***Np*** | ***U-40Zr*** | ***U-20Mo*** | ***Pu-Am*** | |
| ***Zr*** | | ***U-30Mo*** | ***U-Np*** | |
| ***Mo*** | | | ***Pu-Np*** | |

# 2) HFIR Rabbit Capsule Design

- **0.9" of sample length (shielded)**
- **TEM samples: 3mm diameter by 200 microns thick**
- **3 TEM disks, 4 SiC temperature monitors, flux wires**

**First Irradiation:**
**Cycle 438, October 2011**



# 2) Future Low Fluence Irradiation Experiments using the HFIR Hydraulic Rabbit Facility

- **Explore radiation enhanced diffusion (RED) and fuel cladding chemical interaction (FCCI)**
  - **Diffusion couples**
- **Research fuel constituent redistribution**
  - **Small fuel slugs**


***Irradiation Conditions***

***550-750°C***

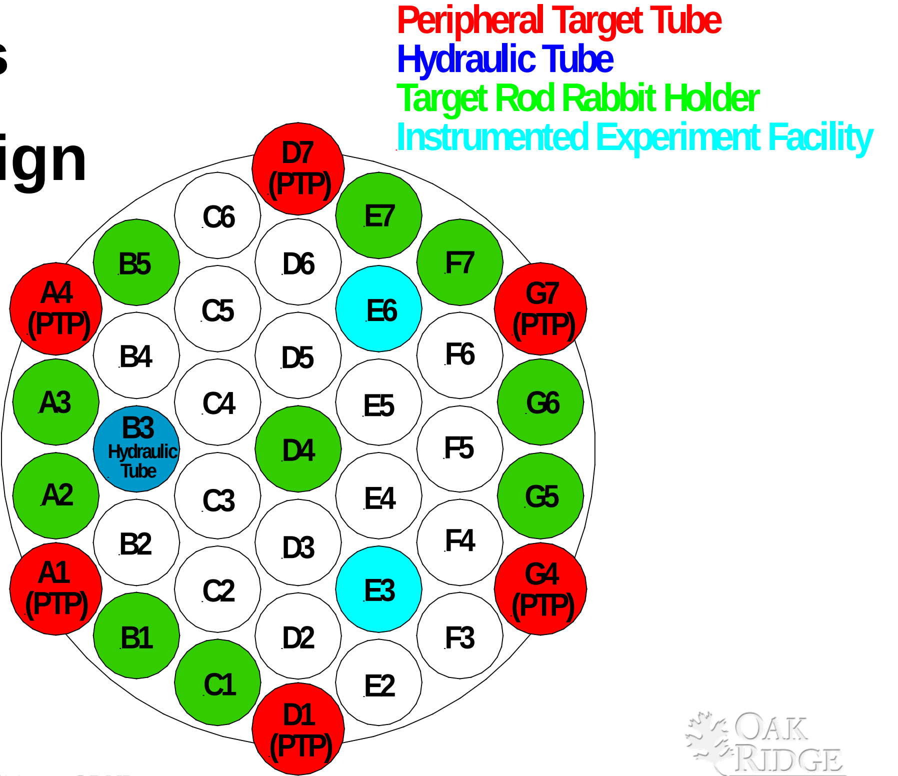
***$1x10^{-5}$ to 5 dpa***

***Linear Heat Generation Rates ~ 300-350 W/cm***

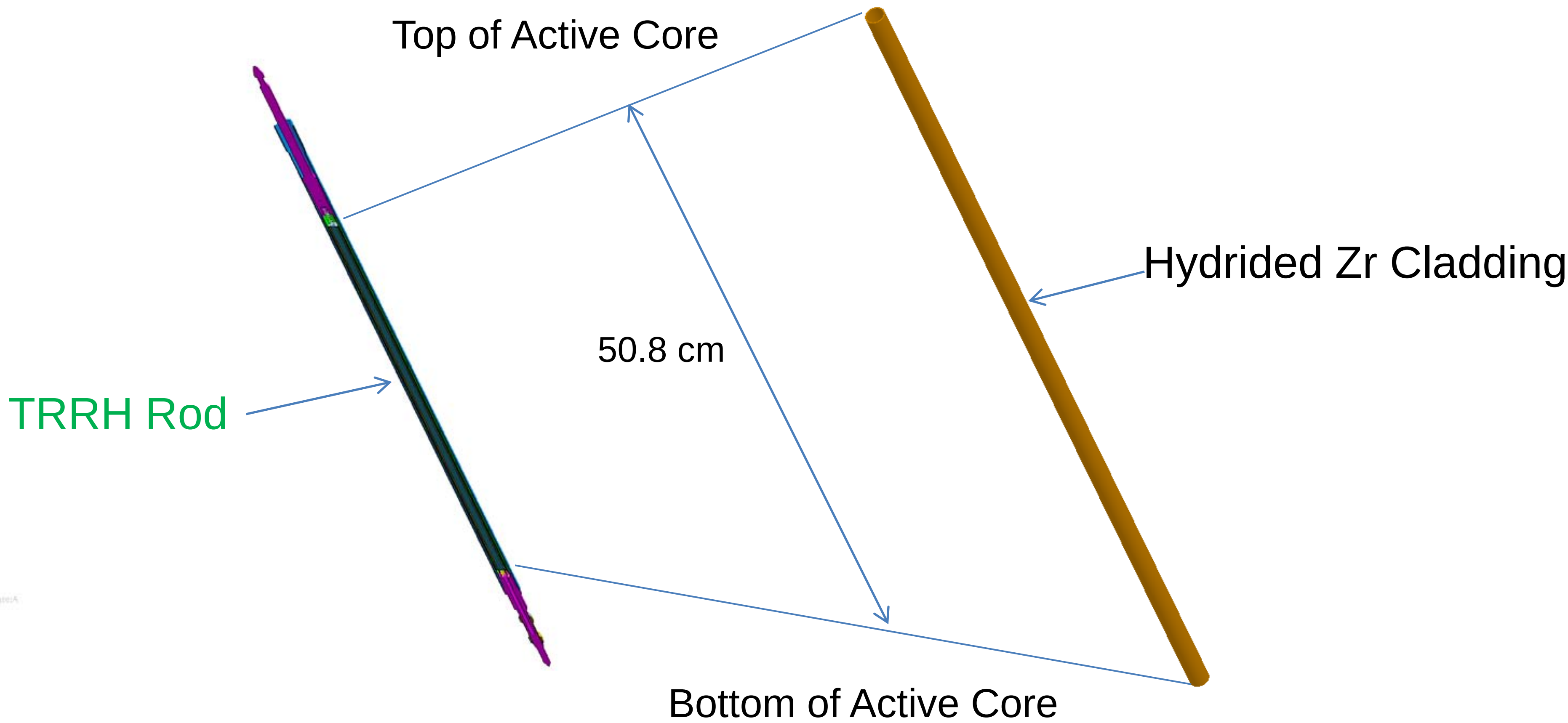
***Fast/Thermal flux ~350-400***

# Conceptual Design of HFIR Experiment to Irradiate Hydrided Cladding

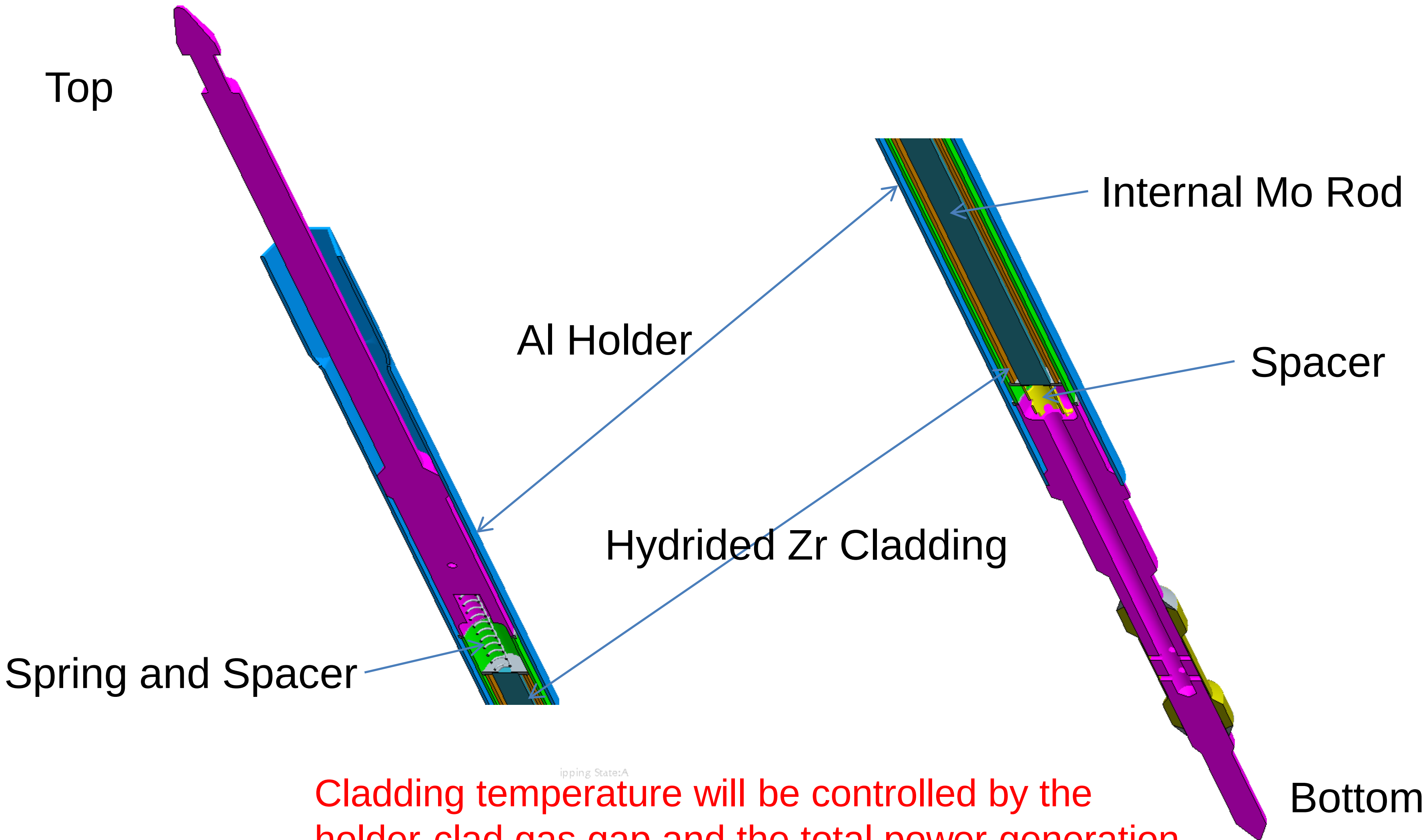
- **Position for fastest accumulation of fast fluence is the flux trap**
  - **Multiple Target Rod Rabbit Holder (green) positions available**
  - **Fast flux: 6.5-11 $10^{14}$ n/cm$^2$·s**
- **Simple, uninstrumented design**



# Conceptual Design (continued)

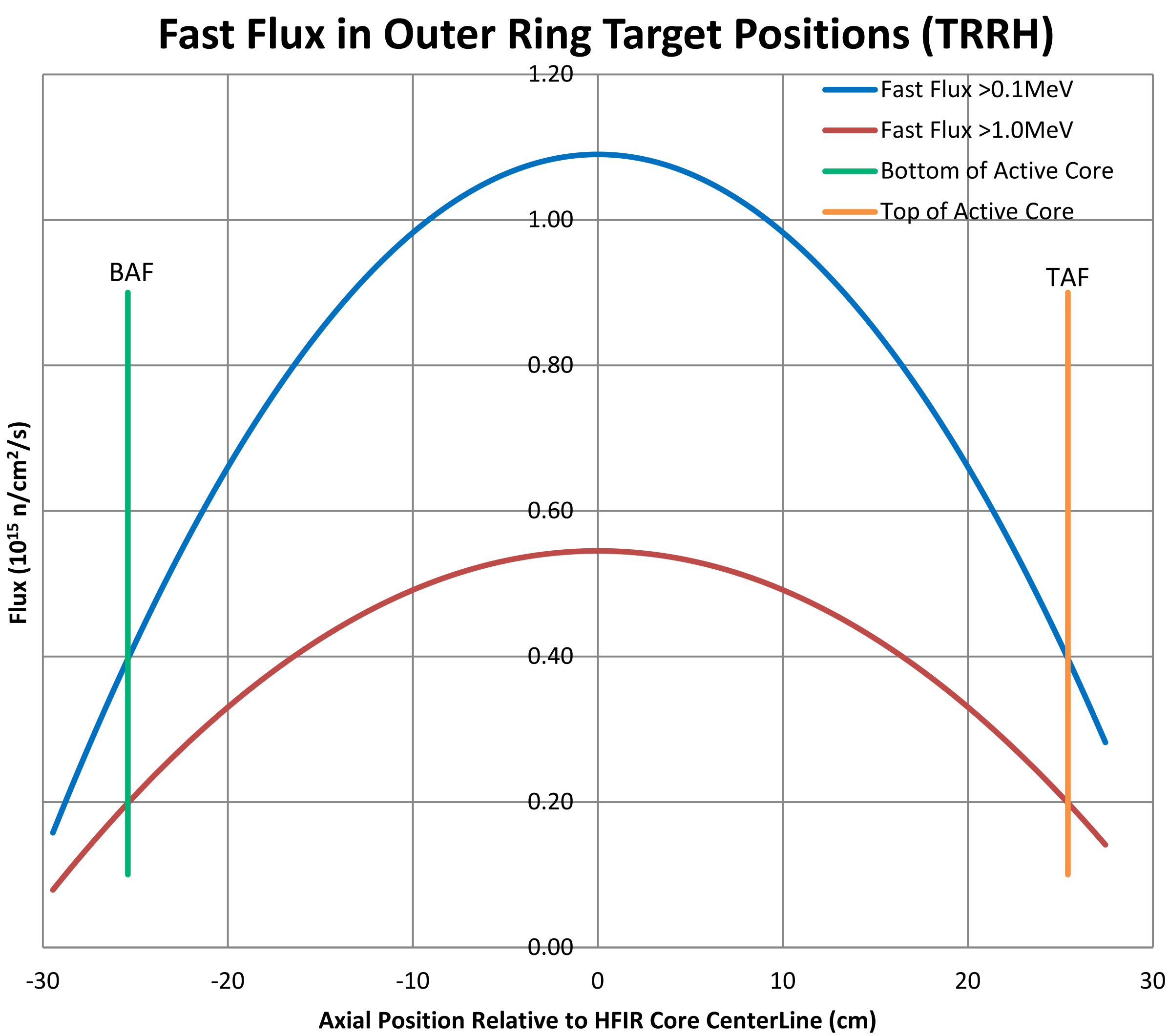


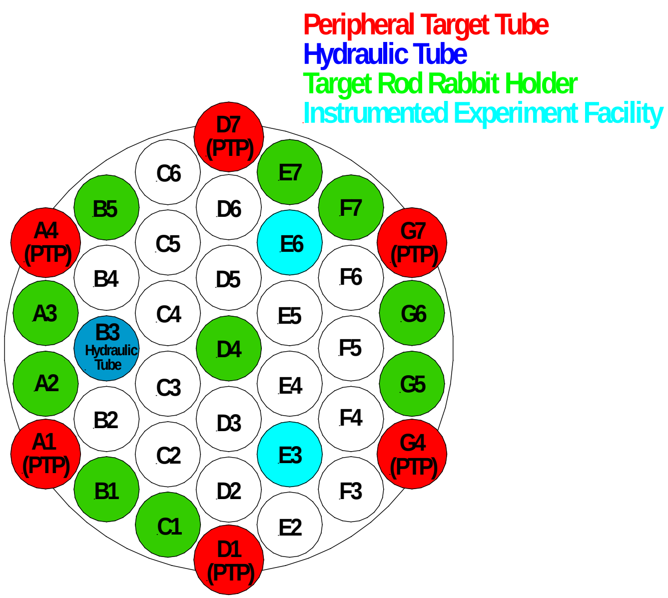
# Conceptual Design (continued)




Cladding temperature will be controlled by the holder-clad gas gap and the total power generation within the cladding and the internal Mo rod

# Fast Flux in the TRRH Positions



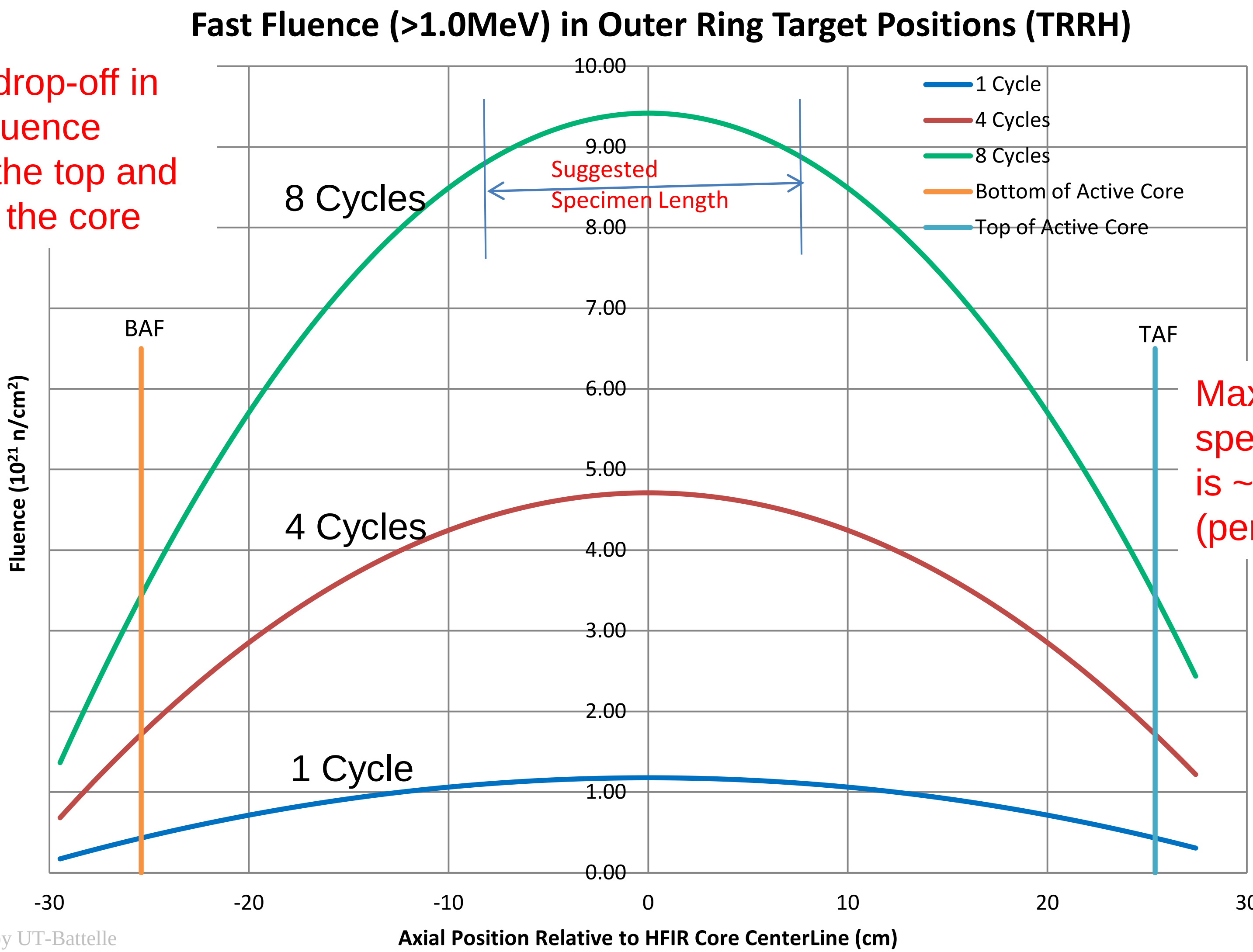






Oak Ridge National Laboratory

# Fast Fluence (>1.0MeV) in the Hydrided Zr Cladding

Note the drop-off in the clad fluence towards the top and bottom of the core




Maximum specimen length is ~15.2 cm (per Y.Yong)

# Conceptual Design Conclusions

- **Use hydrided-clad specimen length of ~6” (15.24 cm) centered about the HFIR core axial centerline**
  - **Approximately the maximum length that can be hydrided in the ORNL furnace (Y.Yong)**
  - **Yields low axial variance in clad fluence**
  - **Allows better experiment control on desired clad temperature**
- **Using the results from the PIE of rods (M5® [AREVA]) recently examined at ORNL:**
  - **At ~26 GWd/MTHM fuel burnup, clad fast fluence ~0.51e22 n/sq.cm (this would require 4-5 HFIR cycles)**
  - **At ~47 GWd/MTHM fuel burnup, clad fast fluence ~1.02e22 n/sq.cm (this would require 8-9 HFIR cycles)**

# Back-up Slides

# HFIR Reliability & Availability

- **Last major upgrade was for the cold source addition, finished in May 2007**
- **Since then, HFIR has operated for 32 cycles**

| Year | Target Operating Cycles | Actual Operating Cycles |
|---|---|---|
| 2007 | 5 | 5 |
| 2008 | 6 | 6 |
| 2009 | 7 | 7 |
| 2010 | 7 | 7 |
| 2011 | 7 | 7 |

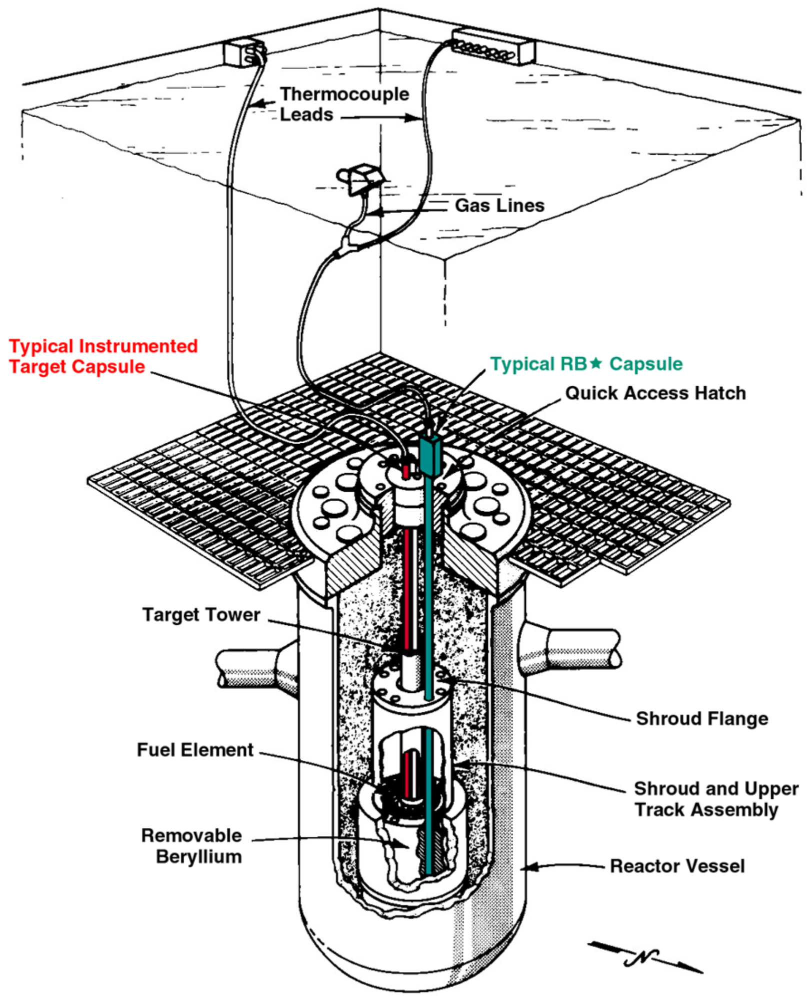
- **Only 1 cycle out of 32 total was delayed from their posted schedule**
  - **~ 48 hour delay**

# Spectrum Tailoring Enhances Versatility of RB★ Facilities

| Characteristics | RB★ Unshielded | RB★ Hf Shield | RB★ $Eu_2O_3$ Shield |
|---|---|---|---|
| Fast flux, E > 0.1 MeV ($10^{14}$ n/cm$^2$-sec) | 5.3 | 4.6 | 4.9 |
| Thermal flux ($10^{14}$ n/cm$^2$-sec) | 9.7 | 1.0 | 0.19 |
| Fast/thermal flux ratio | 0.55 | 4.6 | 26 |
| Peak dpa/cycle (SS) | 0.67 | 0.56 | 0.58 |
| Gamma heating (W/g SS) | 16 | 9 | 13 |
| Typical capsule diameter (mm) | 43 | 38 | 38 |

- ORNL has developed Hf and $Eu_2O_3$ thermal neutron shields with excellent absorption and longevity
- ORNL's SCALE software allows for accurate prediction of both shield effectiveness and fuel fission density over time

# Instrumented Experiments Are Routinely Installed in HFIR



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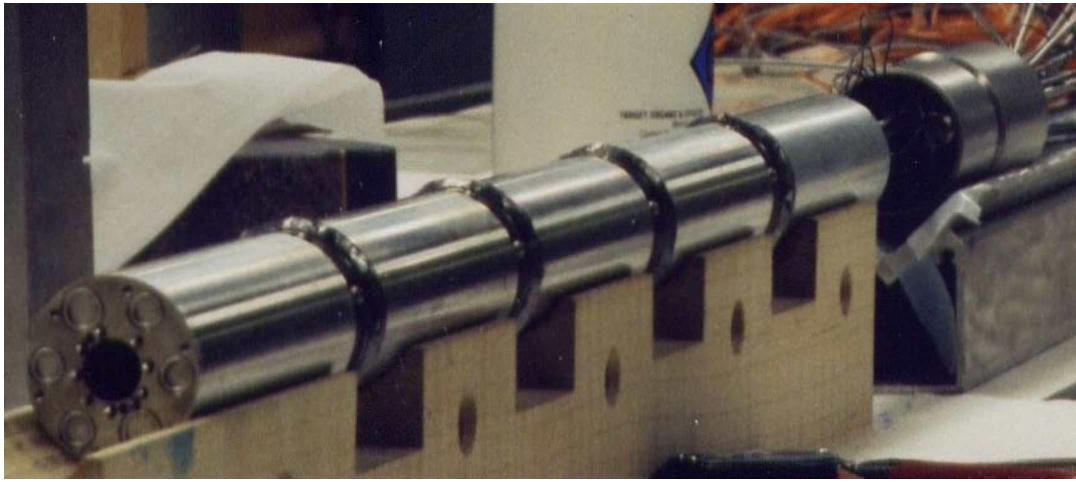
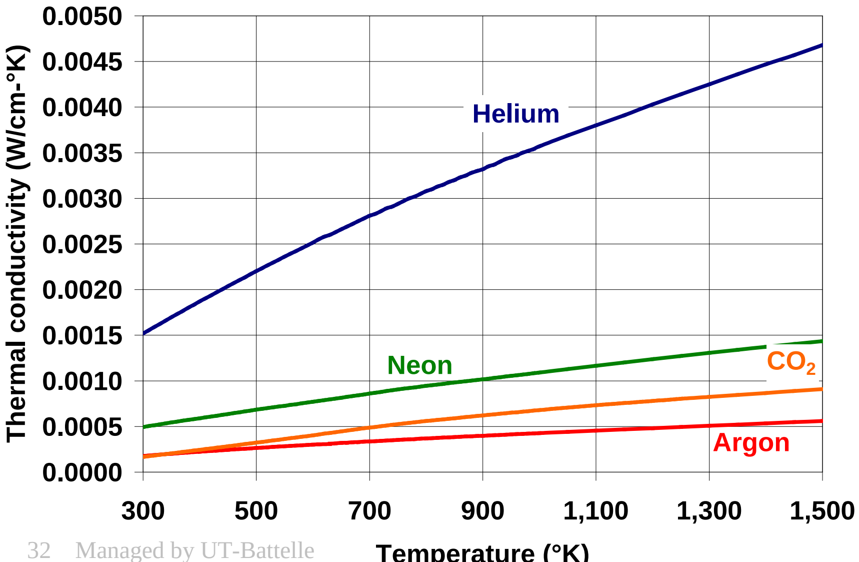
ORNL 2008-G00113_08 (2001-1288C) EFG

# Instrumented Experiments Offer Precise Temperature Control

- Precise temperature control independent of reactor power
  - Through internal heaters that run through the length of the experiment



VTX (RB-13J) Experiment

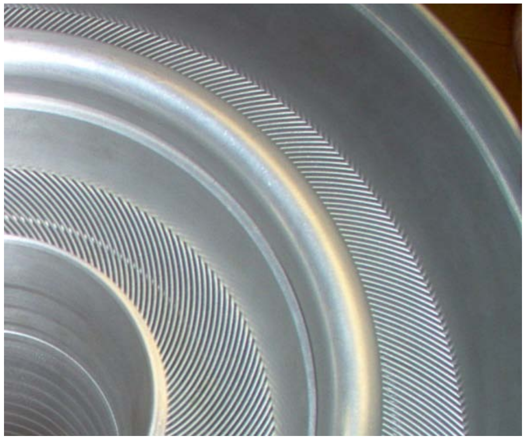


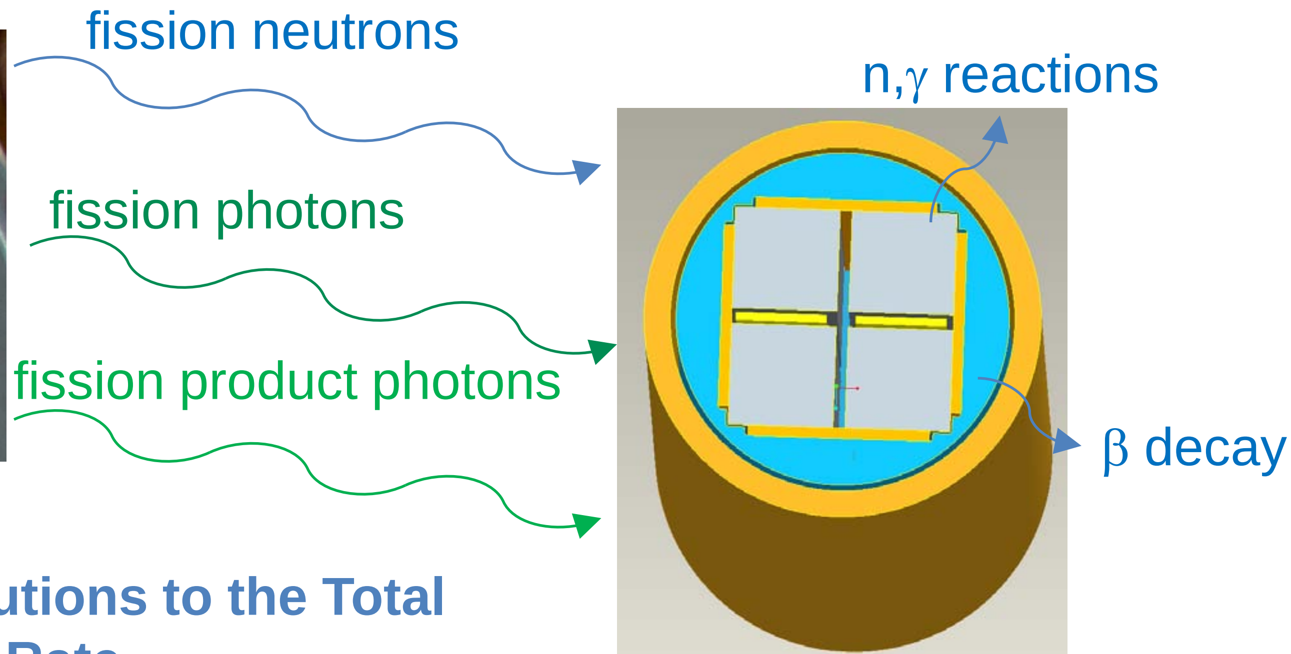

- or by active control of the gas mixture in a gap
  - Helium
  - Neon
  - Carbon dioxide
  - Argon

# Materials Irradiation Experiments Can Be Designed for a Variety of Mediums

- **Lithium-filled subcapsules can accommodate multiple specimen geometries, and minimize temperature gradients**
- **Sodium also permissible**

# Modeling & Simulation — Heat Generation Rates

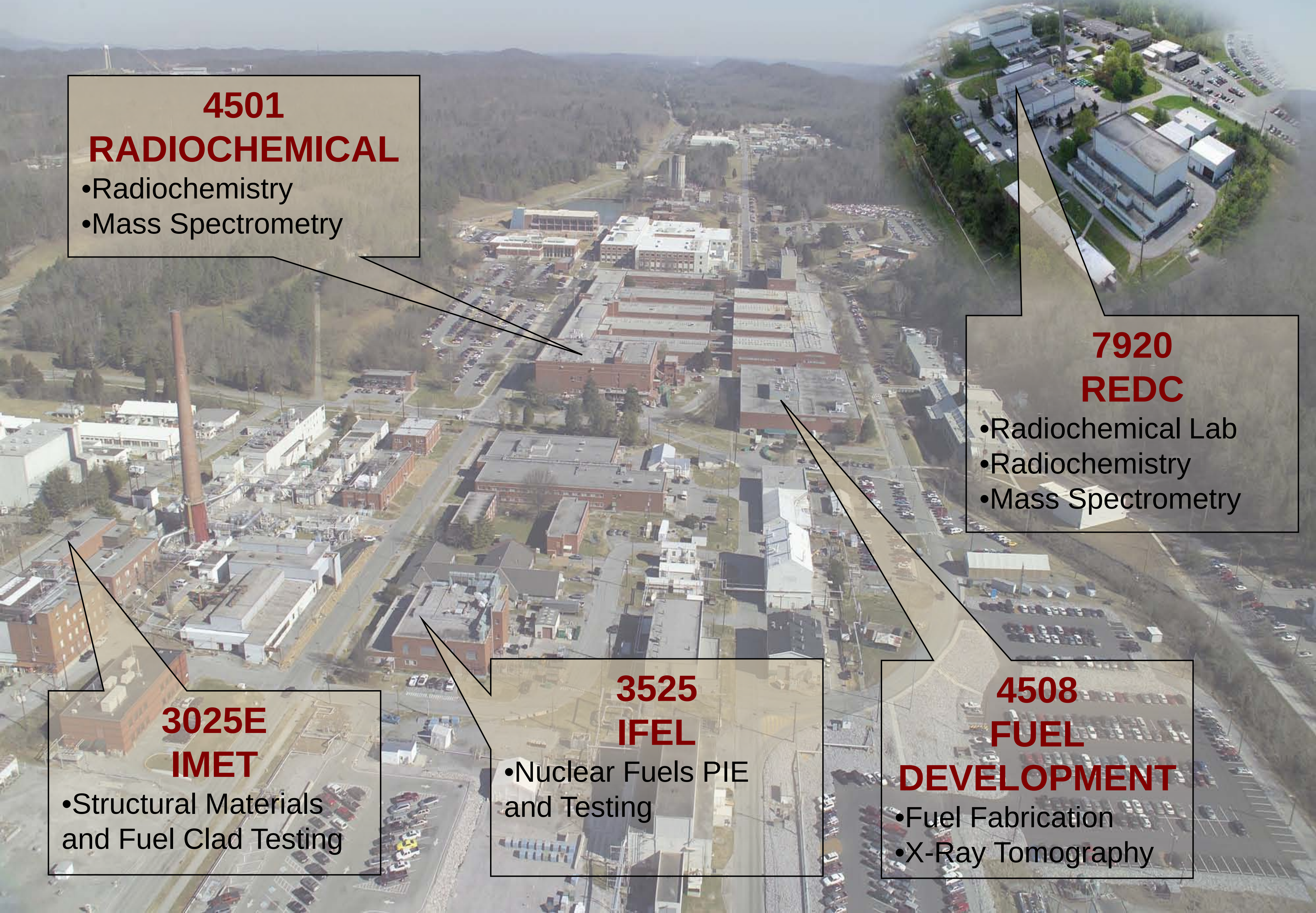





## Relative Contributions to the Total Heat Generation Rate

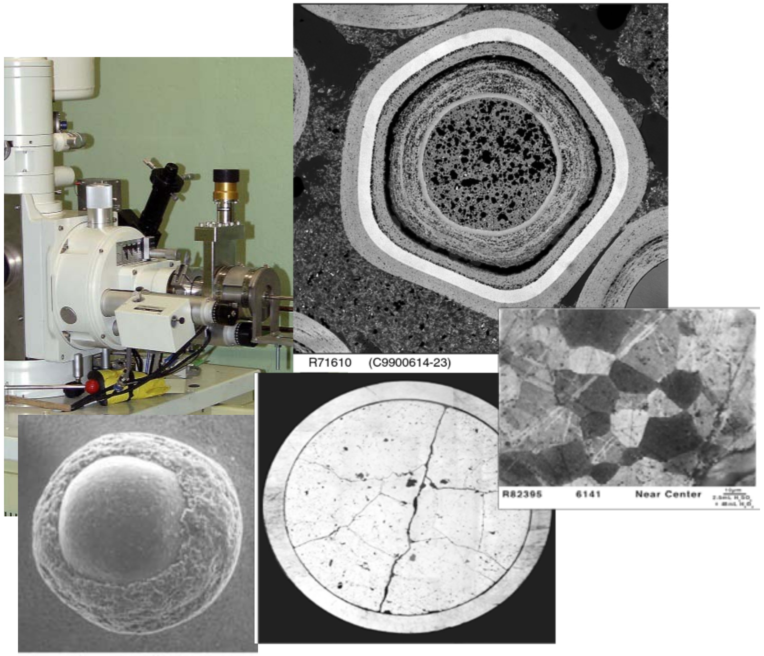
| | Vanadium | Graphite |
|---|---|---|
| Core fission neutrons | <1% | 11% |
| Core fission photons | 47% | 56% |
| Core fission product photons | 29% | 33% |
| Local β decay | 23% | — |

# ORNL NUCLEAR FUELS COMPLEX

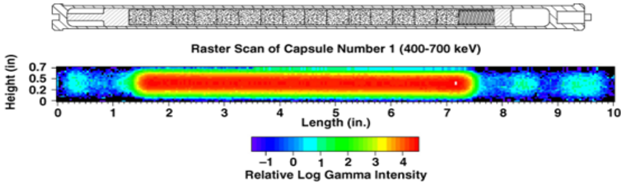


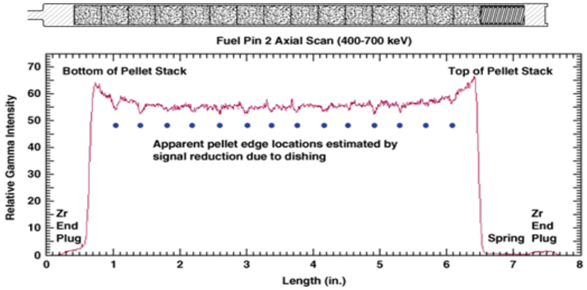
# ORNL's Irradiated Fuels Examination Laboratory Conducts Comprehensive Nuclear Fuel PIE

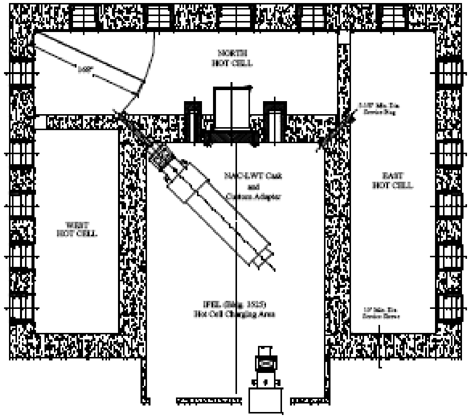
**Historically, the IFEL has played a major role in PIE & Testing of Reactor Fuels for High Temperature Gas Reactors and LWR's**




German Overcoated TRISO Fuel Particle
from the HFR-K3 Experiment



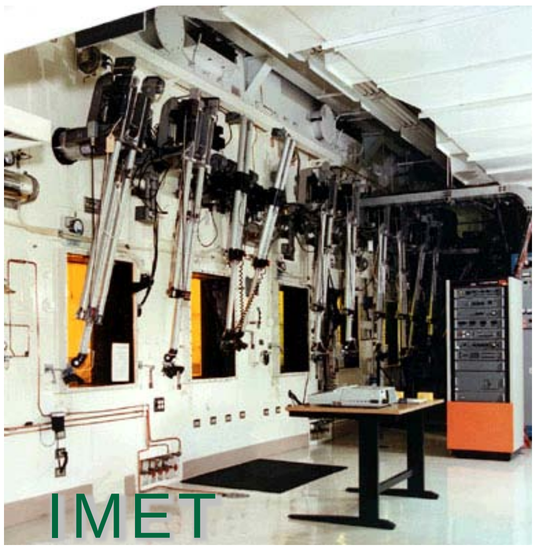

**Recent renovations enable IFEL to handle full-length LWR fuel rods.**

***IFEL provides key analysis and science for advanced nuclear fuel design.***

# The Irradiated Materials Examination Lab and Low Activation Materials Design and Analysis Lab are unique, world-class facilities for irradiated materials science

The IMET hot cells house a comprehensive suite of testing, analysis, and fabrication equipment for all classes of non-fuel materials.

The LAMDA Laboratories allow for the examination of low activation materials without the need for remote manipulation allowing handling of smaller specimens and more delicate analysis.




Samples are routinely moved between facilities to use the “right tools” and efficient research.

- Both facilities have an extensive suite of mechanical, thermal, and physical analysis available and are capable of the highest QA levels (tailored to sponsor needs).
- External collaborators can participate in hands-on research in both facilities.
- Additional capabilities are available at other Office of Science User Facilities at ORNL.


***Combined, IMET and LAMDA represent one of the most extensive capabilities for fundamental analysis as well as basic or applied research for a variety of sponsors and programs.***

ATTACHMENT 6

STANDARD OPERATOR GUIDELINES

# Metallographic Preparation and Examination of Cladding Specimens Irradiated in the HFIR

Prepared by: Yong Yan, FC&I Division Date 8/29/2012

Reviewed by: Rob Howard, Reactor and Nuclear Systems Division, Program Manager Date 04 SEPT 2012

Approved by: Gary Bell, FC&I Division Nuclear Fuels Group Leader Date August 29, 2012

Approved by: D. A. Caquelin, Bldg. 3525 Facility Manager Date

DSO Approval: Mark Baldwin, FC&I Division Safety Officer Date

QA Approval: M. C. Vance, QS & S Division Quality Assurance Specialist Date 8/29/12

STANDARD OPERATOR GUIDELINES

1. Purpose

The DOE Used Fuel Disposition Campaign (UFDC) has tasked ORNL to investigate the behavior of light-water-reactor fuel cladding material performance related to extended storage and transportation of used fuel. Fast neutron irradiation of pre-hydrided zirconium-alloy cladding in the High Flux Isotope Reactor (HFIR) at elevated temperatures has been used to simulate the effects of high burnup on used fuel cladding for use in understanding the material properties relevant to extended storage periods and subsequent transportation. The irradiated pre-hydrided metallic materials will generate data to benchmark hot-cell testing of high-burnup used fuel cladding.

In order to accomplish this research, the Nuclear Fuel Materials Group needs to perform metallographic preparation and examination of cladding specimens irradiated in the HFIR. These guidelines outline steps that are followed in remote metallographic preparation and the examination of irradiated specimens.

2. Scope

The tasks described in this document are all conducted remotely at the Irradiated Fuels Examination Laboratory (IFEL), Bldg. 3525.

3. Environmental, Safety and Health Concerns

All supplies are introduced into or removed from the small freestanding hot cells and the main Hot Cell bank by members of the IFEL operating group (who similarly handle the disposal of waste materials). All such operations are performed in accordance with established IFEL facility operating procedures and SBMS ORNL Radiological Protection procedures.

Verification of compliance status is a prerequisite to conduct of operations in the IFEL. Facility operating personnel must verify that limiting conditions have been met prior to start of work. No work with radioactive materials is to be conducted unless approved by the IFEL Facility Manager.

ES&H hazards and controls are listed in the Research Safety Summary, RSS 515.5, for activities occurring in Building 3525. Each person performing work is required to read and follow the RSS and be cognizant of specific requirements such as radiation permits. Each individual is also required to read and adhere to the limits of work specified in the Facility Use Agreement for Building 3525.

Each person performing task work is responsible to ensure that their training is up to date as listed in their FCID Division training baseline. If deficiencies exist, do not perform this work until your training is brought up to date.

The Group Leader, with help from the project manager, is accountable for developing specific training needed for this activity, including special hazards associated with vendor supplied equipment (see equipment documentation as relevant). Only individuals supervised or otherwise specified by the test engineer or project manager may perform the task work or operate equipment specified in this procedure. Modifications to task equipment and this procedure require the review and approval as specified in the Responsibilities Section.

4. Training and Qualification of Personnel

Appropriate training consists of self-study of this procedure, hands-on training, and demonstrated proficiency with the apparatus. The training/personnel qualification record in Appendix A will be completed as documentation of this training and of personnel qualification. It is the responsibility of the trainee, and of qualified personnel, to be familiar with the current revision of this document. Current revisions of these documents are posted in the ORNL Integrated Document Management System. Refresher training and requalification shall be required every three years.

5. Responsibilities

All of the activities associated with this procedure will be conducted in the IFEL and require the prior approval of the IFEL Facility Manager. The cognizant Laboratory Space Manager (LSM) must be notified in advance of the work to evaluate the impact of the activity on other work taking place in the laboratory. Work will be conducted under the direction of the Principal Investigator and/or the relevant Project Manager.

All personnel involved with operations associated with this procedure will perform their tasks in a manner that ensures that the concept of ALARA is complied with. All changes to this procedure require the approval of the Group Leader and the IFEL Facility Manager. A working, marked-up copy of this procedure will be kept for recording and dating any changes as they occur.

6. Radiation Protection

Before proceeding with the task at hand, consult with building radiation protection personnel and arrange for radiation protection monitoring and permitting as required. If the cell window reads more than 5 mR/hr stop work and consult with Radiation Protection and the Facility Manger as to the proper course of action. As the radiation and contamination levels will be job specific, the degree of radiological hazard and required monitoring will be determined on a case-by-case basis.

7. Radioactive Material Movement

All movements and/or transfers of radioactive material in 3525 will be performed in accordance with applicable SOPs. All uses of polishing, rinsing, and etching liquids must comply with the cell limits.

8. Selection of Specimens and Mount Design

The largest specimen that can be contained in the standard mount (1¼ inch outside diameter by 1 inch high) for remote metallography is limited to planar dimensions of about 0.75 by 0.75 inch. A specimen height of up to 0.9 inch can be accommodated, but the preferred height is 0.75 inch or less. Mounts are usually made from hollowed out cylindrical phenolic material, but other prepared mount styles may be used for positioning and centering small specimens. Procedures from out-of-cell MET work may be used as guide for mount design. The mounts are generally filled with epoxy resin in the hot cell just after the specimens are put in them. This can be a clumsy process, so some preplanning may be necessary such as the use of jigs and holders.

Specimen identification is usually maintained by placing a 7/8 in. diam. steel disk (stamped or engraved with a metallographic specimen number) on the bottom of the specimen mount in a recess and securing it in place with epoxy, typically before putting the mount in the hot cell. Other methods may be used, but the identifier must be fairly rugged. An in-cell poured mount may require that the disk be attached in the cell when the epoxy is poured. The side of the mount may be painted with identification numbers as well. If the metallograph in the West Cell is to be used, the steel disk must be in place because of the magnetic hold-down device on this unit. The disk is not needed if the metallograph in the SEM Cell is to be used. However, it is recommended to use the disk as it offers the greatest flexibility and a durable marking surface for identification.

9. Specimen Mounting

9.1 After sectioning the specimen to a geometry that is acceptable to the mount in use, the specimen is placed within the mount. The available volume within the mount around and above the specimen is filled with a catalyzed epoxy resin prepared per manufacturer's directions, and the assembly is immediately placed in a vacuum degasser for at least 2 minutes to remove air bubbles; the vacuum is then removed to allow air pressure to foster epoxy impregnation. In some cases, the location of the mount or the nature of the specimen may not allow the use of the vacuum degasser. In that case care should be taken to minimize the introduction of bubbles into the poured resin. Curing of the epoxy may be at cell temperature or carried out in an oven.

9.1.1 One suggested epoxy mixture is Araldite GY-502 resin and Hysol HD-3416 hardener mixed 11:1 (resin to hardener) for a slightly extended set-up time.

9.1.2 Another suitable product is Buehler EPO-THIN low-viscosity epoxy, mixed at either its standard 2:1 resin/hardener ratio or 3:1 for extended setting time in the hot cell.

9.1.3 Other resins may be used depending on the application need, but be aware of radiation issues both in hardening time (greatly shortened) and life of the mount.

9.2 Record the preparation of the specimen in a logbook and/or program database. This information must be available to the hot cell staff for material accounting purposes.

10. Grinding

NOTE: The grinding step is used prior to vibratory (Syntron) polishing; it may be incorporated within the Struers Rotopol method as well. The goal is to remove damage done by sectioning and to obtain a flat specimen surface prior to polishing. The use of water (or solutions) shall comply with the hot cell limits (check with the Facility Manager prior to the start of work).

10.1 After the epoxy has cured (normally 8 to 10 hrs), the specimen is ground with successive grades (e.g., 320, 600, 800 grit) of silicon carbide abrasive papers or coarse diamond (e.g., 30 μm) disks using water, sparingly applied, as the vehicle.

10.2 A thin layer of epoxy may be placed on the ground surface to fill in macroporosity. Curing of the epoxy may be at cell temperature or carried out in an oven.

10.3 After the epoxy resin surface layer has cured, the specimen is reground (using generally 600 or 800 grit SiC) until a flat plane for polishing has been established. The re-impregnation or regrind sequence is sometimes repeated, depending upon the type and condition of the specimen. Great care may be required for friable materials leading to a tedious preparation process.

11. Polishing

NOTE: Polishing of irradiated materials can be accomplished generally by wheel-type polishing using the Struers Rotopol apparatus or, occasionally, by vibratory polishing using a Syntron polisher. The choice is dependent on the material being prepared and is up to the judgment of the metallographer. The use of water (or solutions) shall comply with the hot cell limits (check with the Facility Manager prior to the start of work)

11.1 Struers Rotopol Method

11.1.1 The polishing technique and sequence must be determined by the metallographer, varying according to the type of specimen material. Polishing may be conducted using magnetic-backed cloths charged with diamond suspensions or by Mylar film disks impregnated with varying grades of diamond abrasive.

11.1.2 Diamond suspensions frequently include decreasing grit sizes from 9 μm down to 1 μm, possibly concluding with 0.05 μm colloidal silica. Diamond-impregnated Mylar film disks (8" diameter) are available from 30 μm down to 0.5 μm. Install on the unit and polish in the desired manner.

11.1.3 Rinse the specimen with water and allow to air dry or use a blow dryer.

11.2 Vibratory ("Syntron") Polishing

11.2.1 The specimen to be ground is secured in a weighted holder and placed on a vibratory polisher using an appropriate cloth charged with diamond paste and water (or an ethylene glycol/water mixture) as the vehicle. A suggested sequence is: 15 μm, 6 μm, 3 μm and 1 μm diamond grits.

11.2.2 The final polish may be achieved by vibratory polishing using a nylon cloth charged with 0.5 μm diamond or alumina paste and water (or an ethylene glycol/water mixture) as vehicle.

NOTE: When adding or removing specimen from the Syntron polisher, take care not to scrape the polishing cloth with the manipulator.

11.2.3 Remove the specimen from Syntron and wash the specimen thoroughly using a water filled squeeze-bottle and/or dipping bowls. Remove the specimen from the holder and wash the surface again using a cotton ball to gently wipe the wet surface. Leave a bubble of water on the surface of the specimen and place it under blow dryer to dry.

12. Etching

NOTE: Before using etching solutions, review the chemical hazards that may be involved in their preparation and handling. The use of water (or solutions) shall comply with the hot cell limits (check with the Facility Manager prior to the start of work)

12.1 Specimens may be etched by swabbing the polished surface with an etching solution, waiting for a short period of time, and then rinsing and drying the specimen.

12.1.1 There are a large number of possible etchants, methods of application, and etching times. The metallographer should consult with the principal investigator as to the desired outcome and the etchant to be used. The etchant may be introduced into the hot cell via a small bottle (10-50 ml) of solution. A cotton ball may be used as an applicator. Make sure rinse water is easily available as you will have little time to perform the task.

12.1.2 The etching process is difficult to control and several tries, including specimen re-polishing, may be required. Out-of-cell trials should be considered to save time and resources.

13. Examination and Photomicrography

12.1 The specimen can be transferred to either the Shielded Research Metallograph in the West Cell or transported to the Leica Metallograph in the SEM Cell via the cell elevator. Calibrate and photograph the specimens using the metallograph per the manufacturer's instructions and any other program documentation or procedures. Save the photos in a format and manner as dictated by the program. Log the photos numbers in either a logbook or a specified electronic medium.

14. Disposition and Records

This procedure does not provide for the logging or disposition of records. Consult with the principal investigator for the desired disposition of the photos, logbooks, and other records. Note that MET mounts deteriorate over time due to radiation and have a working time of only a few weeks, thus they cannot be depended on to be available at a future time.

**END OF PROCEDURE**

## APPENDIX A. Training and Qualification Record

The following is a record that the trainee has completed training and is qualified to perform this procedure. For each trainee, complete form and post a copy in the equipment logbook.

**Trainee** (Sign and Date):

__________________ Reviewed this procedure and all referenced documents.

__________________ Received hands on training from an approved operator.

**Trainer** (Sign and Date):

__________________ Provided hands on training to trainee.

__________________ Trainee demonstrated proficiency with method, completed the described operations and processes in compliance to the procedural requirements, and obtained satisfactory results.

**Qualifier** (Sign and Date):

__________________ Trainee is qualified to perform this procedure based on satisfactory completion of the described operations and processes in compliance to the procedural requirements and the demonstrated ability to obtain satisfactory results.

**Qualification Expiration Date** (max 3 years): __________________