Advanced Post Irradiation Techniques

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Introduction

• In last 15-20 years great strides have been made to applying advanced techniques to wide variety of material science problems
• Delayed application to the radiological field due to expense and difficulties of working with radiological samples
• New characterization techniques allows for revolutionary data to be obtained from previously irradiated samples, at a fraction of cost of performing new irradiations
Focused Ion Beam

- The “gateway” instrument
- Allows for preparation of small, site specific samples that were previously impossible
- Extremely small size of prepared samples, allows for easier transport and analysis on down stream equipment
Coating Thickness

- Can use FIB to measure thin coatings that are difficult to retain during normal sample preparation.
- Very quick (20-30 min), no need to polish or mount sample.
FIB Sample Transfer

• Use of holder that can be transferred between FIB, TEM, and atom probe without handling of grid
• Decreases odds of sample damage/loss
• Hummingbird Scientific is tips/end effectors we use
TEM Sample Preparation

- Site specific samples, from large range of materials, (irradiated U-Mo, (U,Pu)O2, triso kernels, triso layers and interfaces, zircaloy, fuel metal interfaces)
- Due to extremely small size, ~3 million times smaller by mass than traditional TEM disk, the calculated dose of single samples is ~3.5E-10 Sv/hr compared to 1.1E-2 Sv/hr for traditional sample
Cube Preparation

- ~25μm cubes
- 300 nm slices cut from cubes (can be adjusted)
- Volume studied ~25 μm x 25 μm x 6 μm
- Have prepared blocks as large as 70 μm x 60 μm x 40 μm
Large Block
EBSD/EDS Data Collection

- Step size 200 nm used for EBSD/EDS scans
- Beam energy 20 keV
- Pu, U, O, Mo, Ru, Rh, Cs, Ba, Tc, and Sr scans

Orientation During EBSD/EDS Scans

SEM FIB
Grid Holder
45°C tilt holder

Orientation During Ion Milling

SEM FIB
180° Rotation

Sample Cubes on Grid
EBSD

EBSD Scan
EDS Scan (red is Pu, Green is Mo/Ru)
EBSD Phase Maps

Cubic (U,Pu)O₂
Hexagonal Mo-Ru-Rh-Pd-Tc
3D Reconstructions

Cube 1
- Avizo Fire used to reconstruct EBSD/EDS scans into 3D volumes
- Allows for study of real microstructures, without having to make assumptions on 2D to 3D volumes
- Finite element meshes can be generated to perform 3D modeling

Cube 3
Atom probe provides unique positional, high resolution chemical (and isotopic) information
Zr – Blue
Pu – Red
U - Green

-Oxide metal interfaces are different distances from the tip surface.
*A similar linescan profile was performed, but with the ROI oriented perpendicularly, as shown on the right. The results were very similar to these.
There is a concentration of doubly oxygenated actinides, particularly PuO$_2$. This is suspected to be a grain/sub grain boundary.
Si clustering observed in Zr rich area in as cast fuel

APT of U-20Pu-15Zr
Conclusions

- FIB has enabled analysis of a wide variety of new materials and techniques, previously impossible (or very difficult)
- FIB effective method for preparation of site specific TEM samples, even from challenging interfaces/materials
- Ga damage issue with all FIB prepared samples, can be minimized with low kV cleans
- FIB polishing for EBSD effective for variety of materials (Zircaloy, SiC, MOX)
- Atom Probe
  - APT successfully performed on transuranic fuels
  - APT provides unique high yield, high resolution mass spectrum data on samples
  - No finding of cross contamination between clean and radiological samples in APT
  - Oxidation of metallic samples during transportation from FIB to TEM/APT needs to be addressed

FEA mesh generated from reconstruction of cube from near central void of FO-2 A
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