Methods for Post Irradiation Examination of Tritium Producing Burnable Absorber Rods

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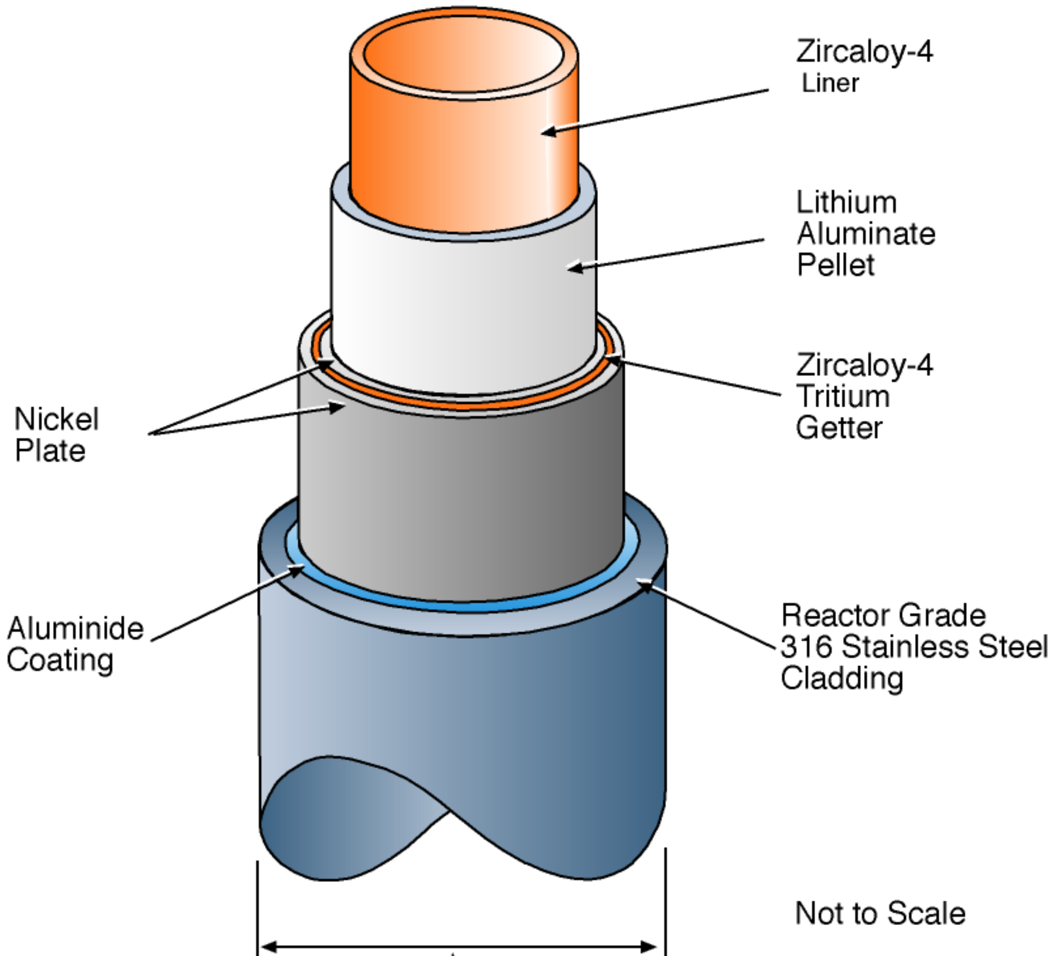
Tritium Technology Program

- US Government Requires a Tritium Stockpile
- US Department of Energy Ended Tritium Production at SRS in 1988
- Several options for Tritium Production were evaluated
  - Dedicated Reactors
  - Accelerator Production
  - Commercial Light Water Reactors
- Tritium Technology Program initiated in 1997 to produce tritium at TVA Watts Bar Nuclear Reactor using Tritium Producing Burnable Absorber Rods (TPBARs)
- In 2004, Watts Bar showed slightly elevated levels of Tritium in Cooling Water
- Post Irradiation Campaign Initiated in 2006
- Currently Irradiating the 7th core with TPBARs

TPBAR Components



Why TPBAR PIE is Necessary

- Permeation of Tritium into Reactor Cooling Water
  - During initial irradiation of TPBARs in WBN1, higher than expected levels of tritium were detected in the reactor cooling water
  - Information needed for Design Changes
- Fundamental Understanding of Transport in TPBARs
  - Transport phenomena during irradiation
  - Performance of components

Data Needs - Methods

- Location of Tritium within the TPABR
  - Protium/Tritium assay – All Components
  - Radial Gradient Analysis of tritium in Getters
  - Low Level Tritium Analysis in Cladding
- Secondary Tracking of Tritium
  - ^{4}He in Pellets – by Tritium Assay Method
  - ^{3}He in Cladding

Data Needs - Methods

- General Transport of Species
  - Visual Observations – slitting and videography
- Carbon Analysis – all components
  - Bulk Carbon Analysis
  - Auger Electron Spectroscopy/X-Ray Florescence Spectroscopy
  - Carbon analysis during Extraction/Protium Analysis
- Other Analyses
  - FTIR Spectroscopy – Liner oxidation
  - Microscopy - Optical, SEM, TEM – all components
  - Miscellaneous Measurements – all components

Protium/Tritium Assay

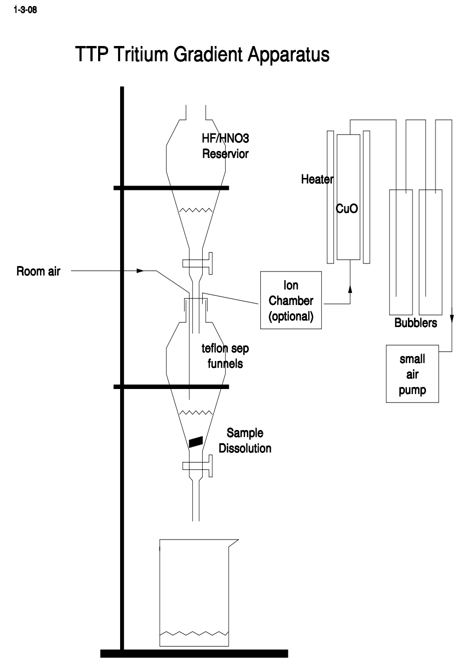
- High Temperature Furnace
- Pressure transducer to determine total gas evolved
- Samples collected for Mass Spec
- CuO bed to oxidize T_2 to T_2O
- Bubblers to collect T_2O
- Samples counted to determine Tritium

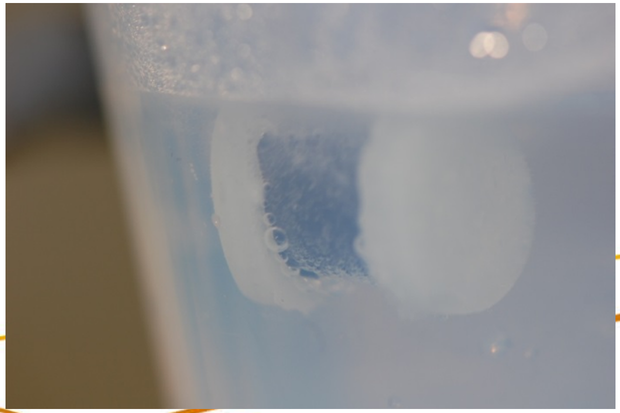
Tritium/^{4}He Assay

- Induction Furnace
- Samples collected for Gas Mass Spec
- CuO bed to oxidize all tritium to T_2O
- Liquid samples counted for tritium

Tritium Radial Gradient Analysis

- Dissolve Getter in HF/HNO_3 for specified time intervals
- Collect Off-gas in bubblers
- Analyze dissolution and bubbler solutions for Tritium
- Analyze dissolution solutions for Zirconium and Nickel by ICP-OES





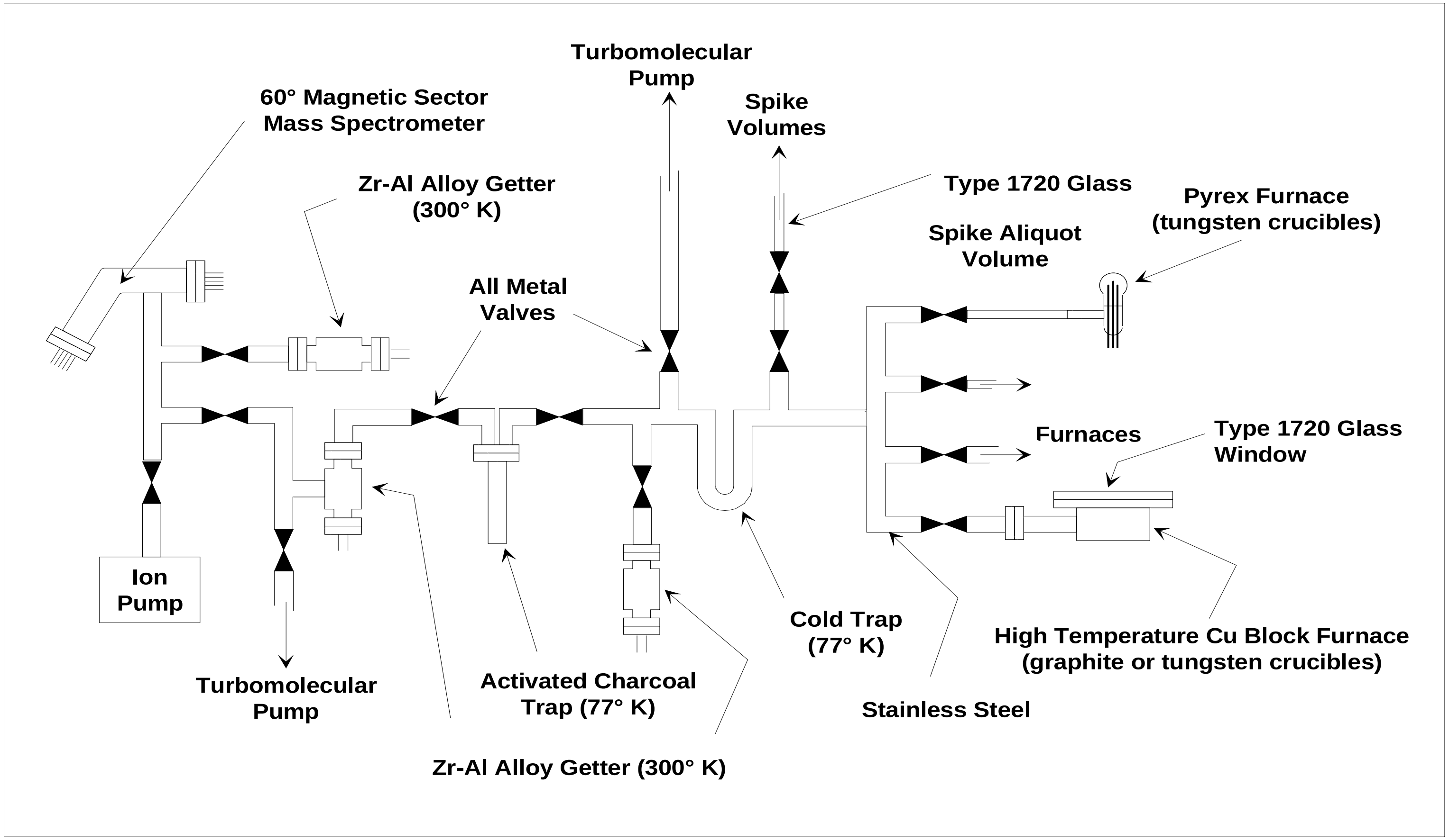
Low Level Tritium Analysis

- Dissolve Sample in HNO_3 or HF/HNO_3
- CuO bed to oxidize all tritium to T_2O
- Use bubbler to collect T_2O
- Analyze bubbler solution for tritium

Helium system description

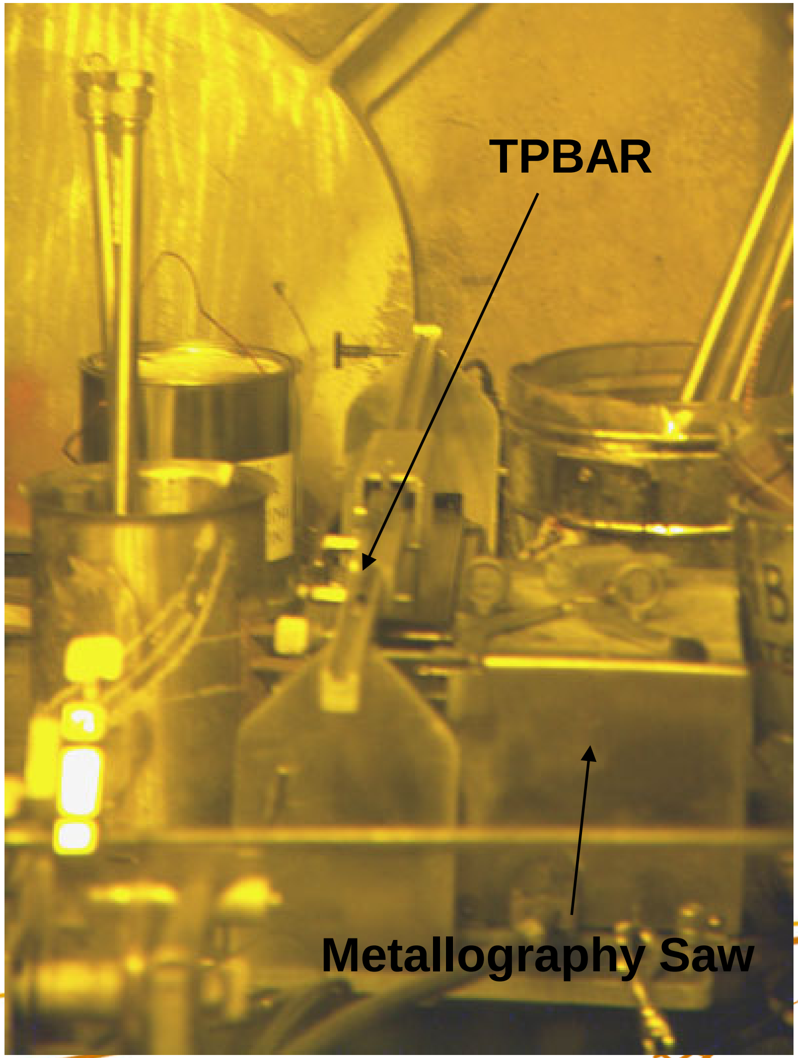
- Measurements conducted by isotope dilution gas mass spectrometry.
  - Analyzes radioactive or non-radioactive samples.
    - Activity levels up to R/hr at contact.
  - Measures from ~10^8 to ~10^{18} atoms (^{3}He and/or ^{4}He).
  - 1% absolute accuracy above ~10^{10} atoms.
  - Total He or He release with time or temperature.
  - Sample sizes from micrograms to grams.
  - Indirectly determine tritium by measurement of ^{3}He decay product.
  - Determine boron levels to wt. ppm by ^{4}He increase from ^{10}B(n,α) reaction.

Helium analysis system



Longitudinal Slitting Saw

- Section TPBAR into ~12 inch pieces
- Slit sections longitudinally
- Videograph Internal and external surfaces of each component
- Evaluate structural characteristics and unusual deposits

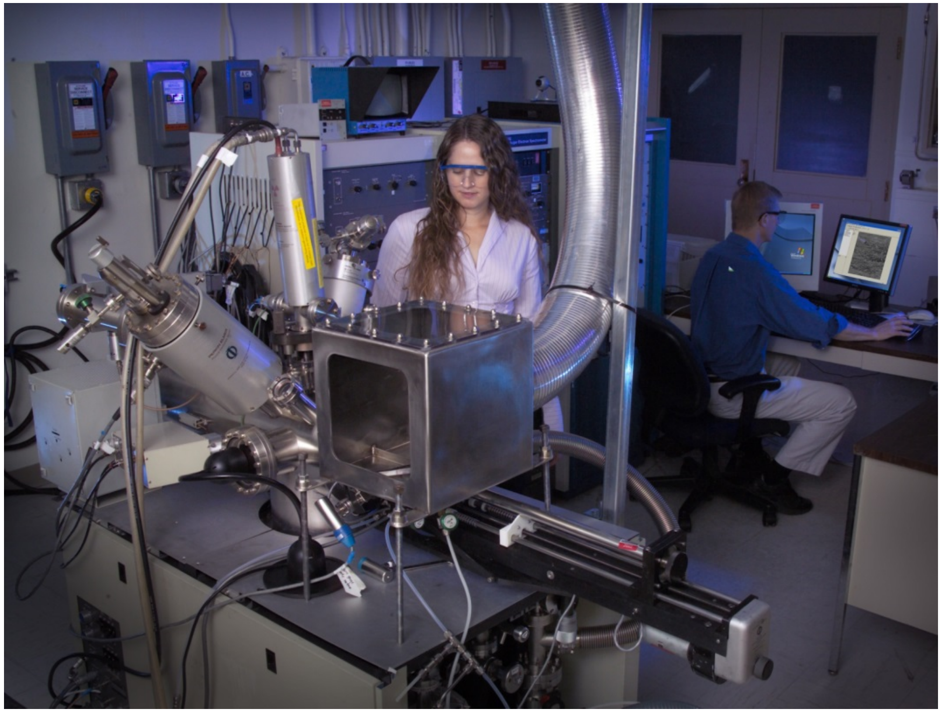


Bulk Carbon Analysis

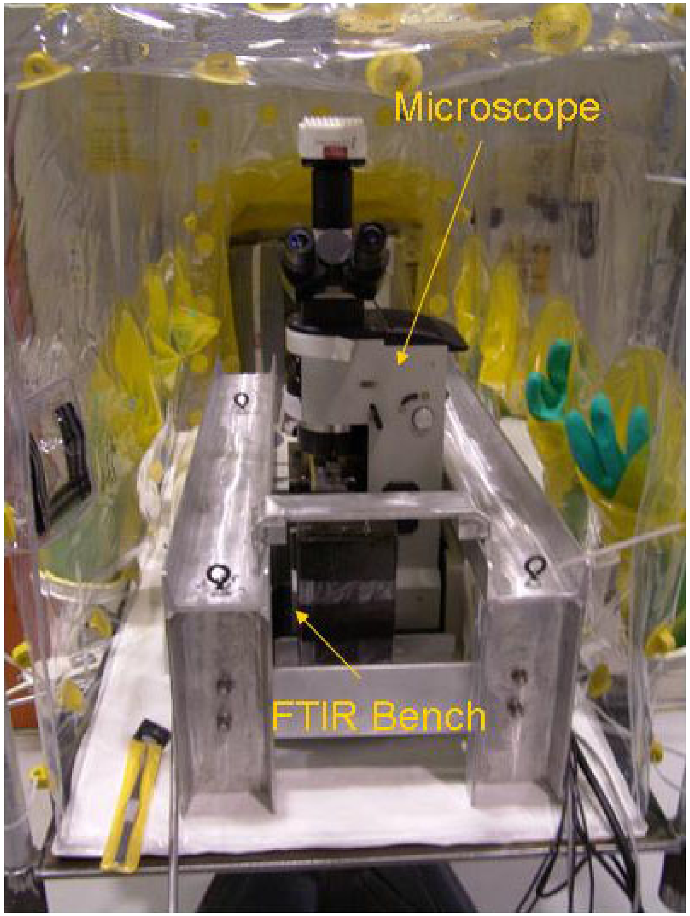
- LECO Carbon Analyzer
- Powderize pellet samples
- Metallic samples analyzed as received

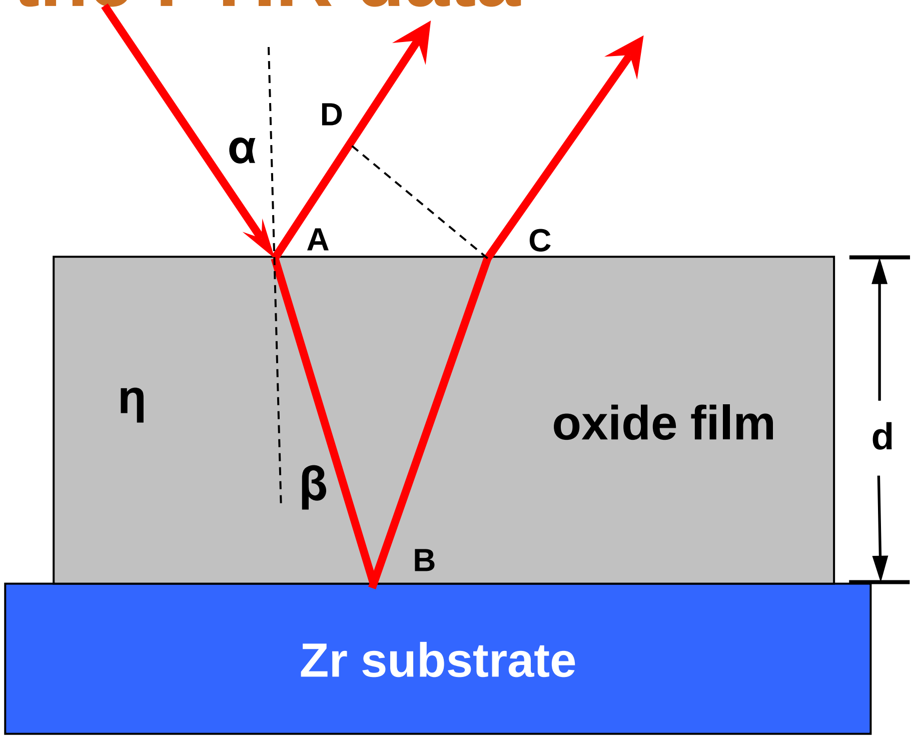
Surface Chemistry

- Auger Electron Spectroscopy
  - Metallic surfaces
- X-ray Photoelectron Spectroscopy
  - pellets
- Secondary Ion Mass Spectrometry
  - All components



Film Thickness: Optical interference pattern observed in the FTIR data



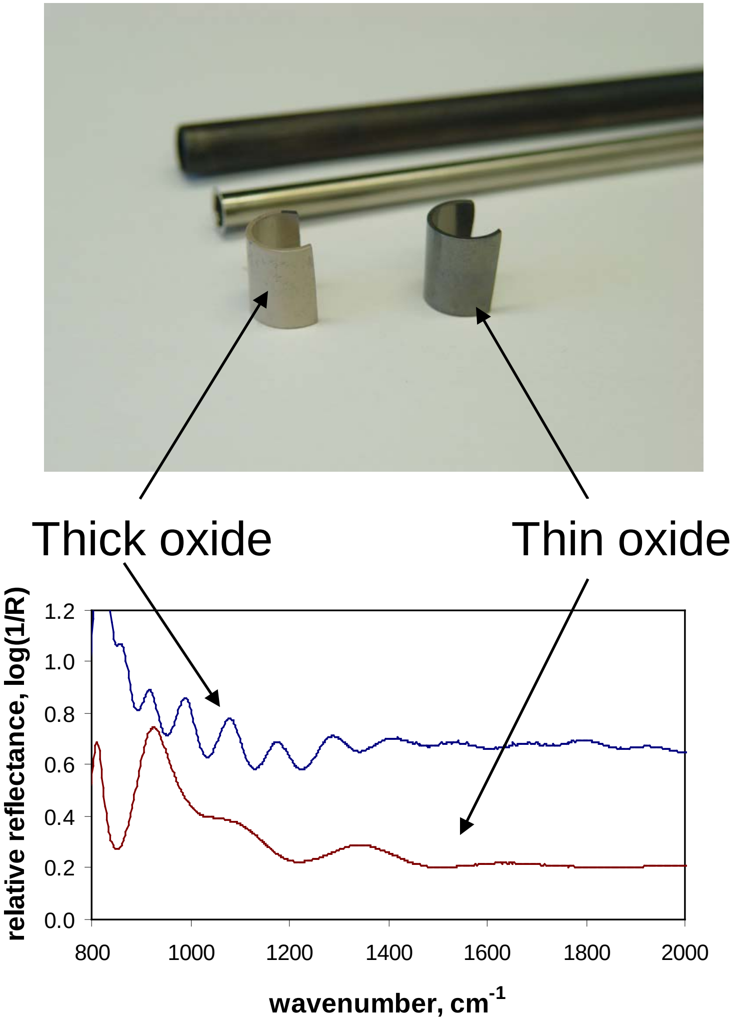


$$d = \frac{(m_2 - m_1)}{2\eta(v_2 - v_1)}$$

d = film thickness
m = order of interference peaks
v = wavenumber of interference peaks
η = index of refraction of oxide film

FTIR Analysis

- Measurement performed using Nicolet FTIR spectrometer with reflectance stage
- Measured directly on Zr oxide sample
- Bragg law analysis of interference pattern yields film thickness, $n\lambda = 2d \sin(\theta)$



17

Optical Microscopy

- Nikon Eclipse Metallurgical microscope with XRF
- Oxide thickness measurements
- Structural information of pellets
- Tritide detection in liners and gettters
- Sample selection for further analyses

Scanning Electron Microscopy

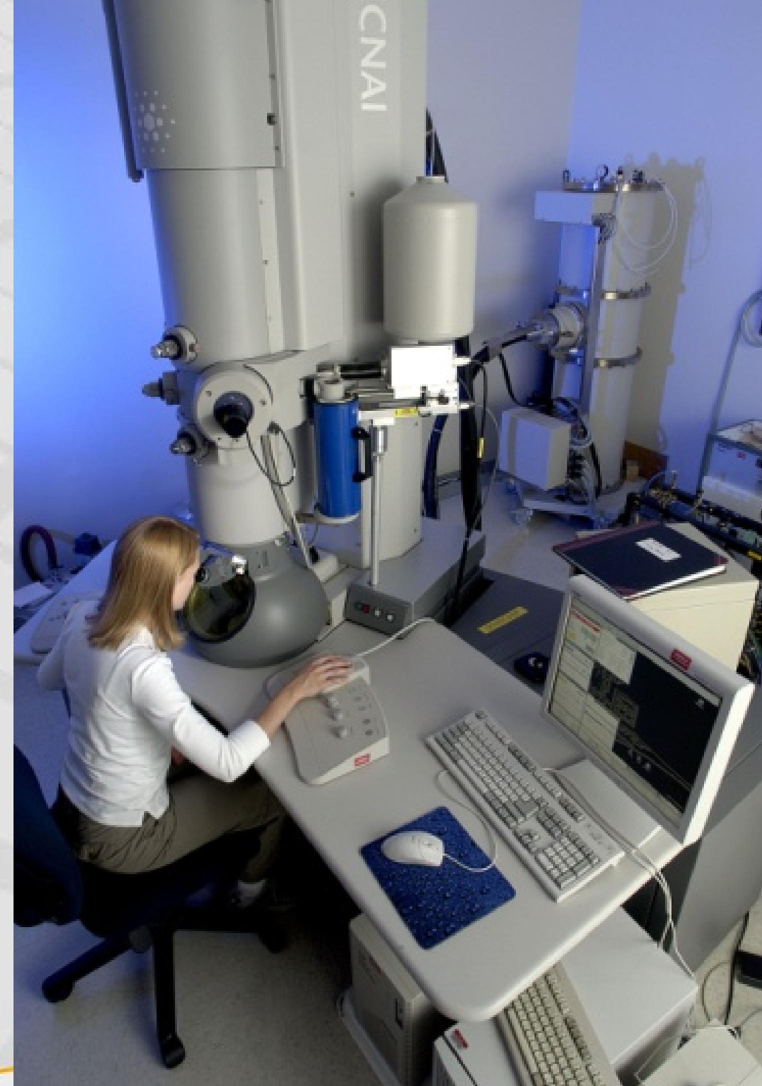
- Quanta 250 FEG with EDS, WDS and EBSD detectors
- Compositional information
- Imaging of components
- Structural characteristics

Transition Electron Microscopy

- FEI Technai T30
- Compositional information on pellets

FEI 250 Quanta FEG

FEI Tecnai 30S-Twin



Miscellaneous Measurements

- Metrology – dimensional information
- Mass change
- Component crush testing
- Pycnometry
- BET surface area