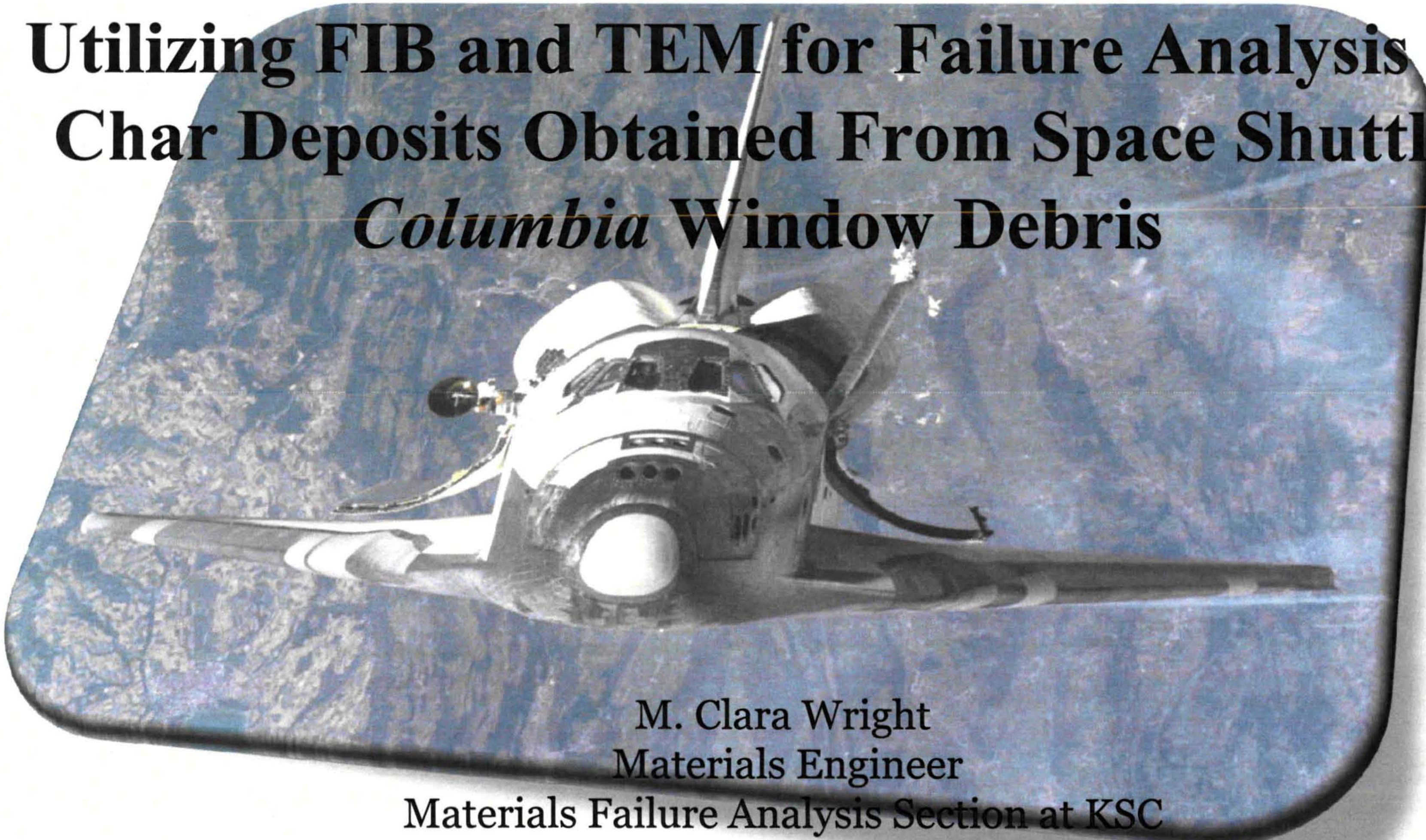
Kennedy Space Center
Engineering and Technology

Utilizing FIB and TEM for Failure Analysis of Char Deposits Obtained From Space Shuttle *Columbia* Window Debris



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Materials Failure Analysis Section at KSC

Microscopy & Microanalysis 2011
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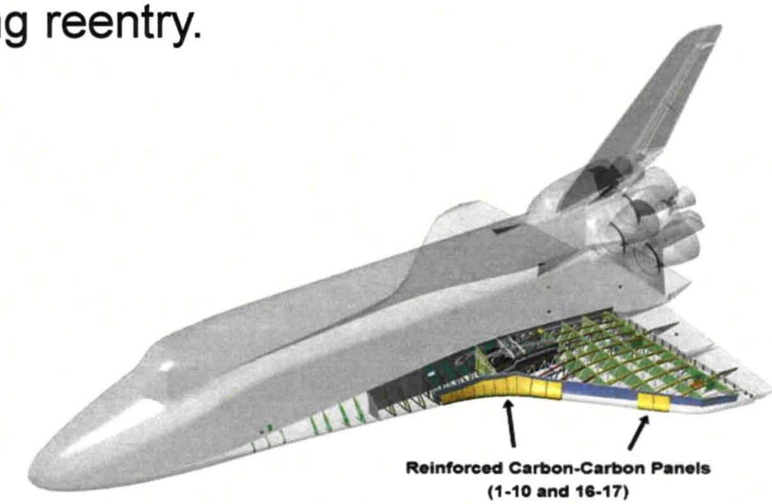
Research Collaboration

- Kennedy Space Center (KSC)
  - Materials Science Division: Clara Wright, Steve McDanels
    - ➢ Metallography, SEM, XRD, logistics
- Johnson Space Center (JSC)
  - Astronaut Office: Col. Pam Melroy, Dr. Danny Olivas
    - ➢ Survivability study
  - Materials & Processes (M&P) group
    - ➢ Metallography, SEM
  - Astromaterials Research & Exploration Science (ARES) Lab: Dr. Roy Christoffersen
    - ➢ TEM
- Langley Research Center (LaRC)
  - M&P group
    - ➢ TEM (SAD indexing)
- University of Central Florida (UCF)
  - Materials Characterization Facility (MCF): Zia Rahman*
    - ➢ In-Situ FIB Sample Prep, TEM
    - * Now at ARES lab at JSC

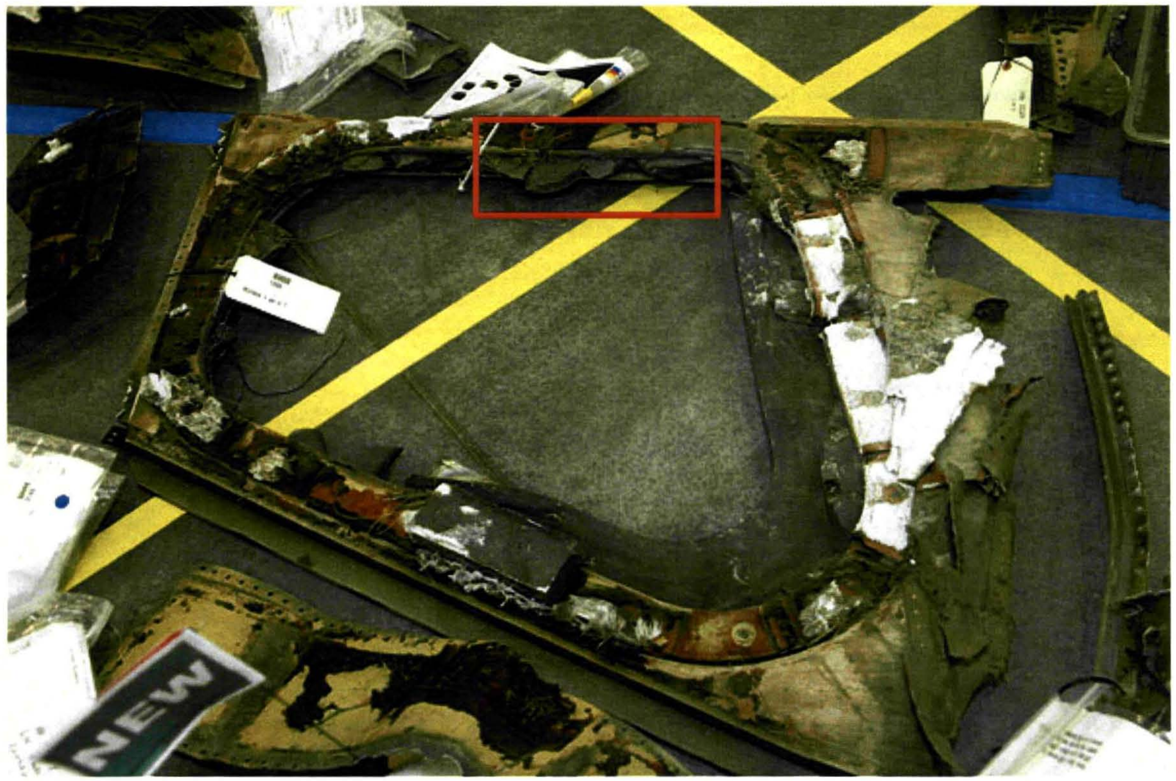
Columbia Accident

- The breakup occurred on February 1, 2003
  - Velocity was in excess of Mach 18 (13,700 mph)
  - Altitude was over 60,960 m (200,000 ft)
  - Resultant debris experienced temperatures in excess of 1750°C (3182°F).
- The *Columbia* Accident Investigation Board (CAIB) determined that foam debris impact from the external tank left bipod ramp breached the left hand wing leading edge reinforced carbon-carbon (RCC) panels 8-9 during launch, which led to a considerable thermal event during reentry.



http://caib.nasa.gov

Orbiter Window Debris

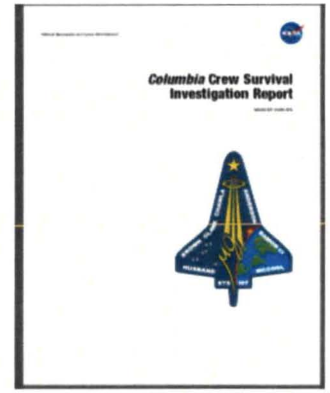


Columbia window lying exterior-side up. Photographed at the *Columbia* reconstruction hangar at KSC on March 3, 2003. Available at http://caib.nasa.gov/

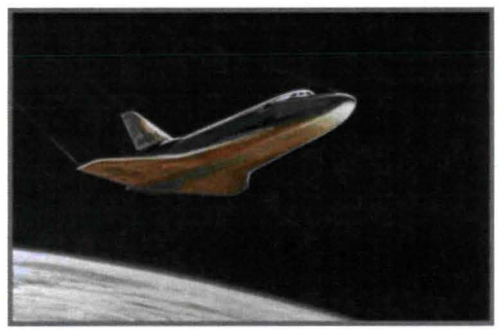
- Cockpit windows and related hardware were among the 84,000+ pieces of debris recovered after the unfortunate breakup of *Columbia.*
- Window pane fragments removed from the damaged overhead window frames were analyzed as part of the Spacecraft Crew Survival Integrated Investigation Team (SCSIIT) [1].

Crew Survivability Study

- The SCSIIT was chartered to:
  - Perform a comprehensive analysis of the accident focusing on factors and events affecting crew survival
  - Develop recommendations for improving crew survival for all future human space flight vehicles.
- A sub-team to the SCSIIT analyzed the effect of the plasma reentry environment on selected orbiter structural and sub-structural materials



http://www.nasa.gov/news/reports/index.html



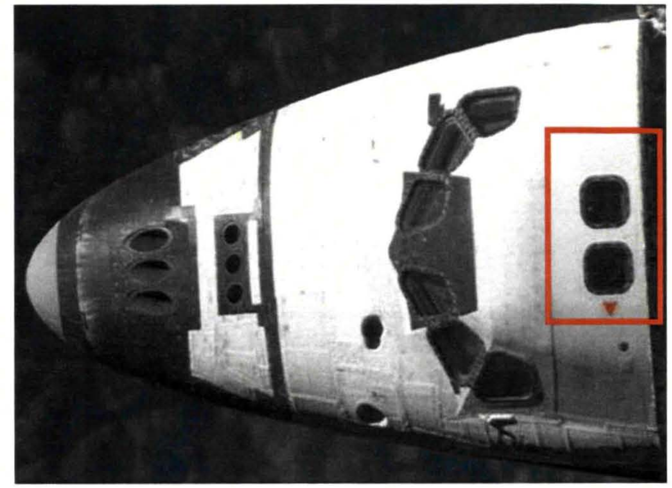
- The research presented in this talk is discussed in detail in the following article:

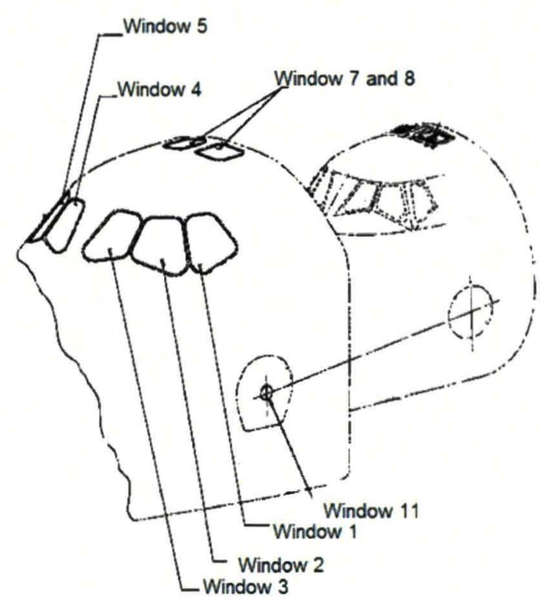
  J. D. Olivas, M. C. Wright et al, Acta Astronautica, *Crystallographic oxide phase identification of char deposits obtained from space shuttle* Columbia *window debris;* Vol. 67, Issues 5-6, September-October 2010

- This talk will focus on sample preparation and analysis

Window Fragments

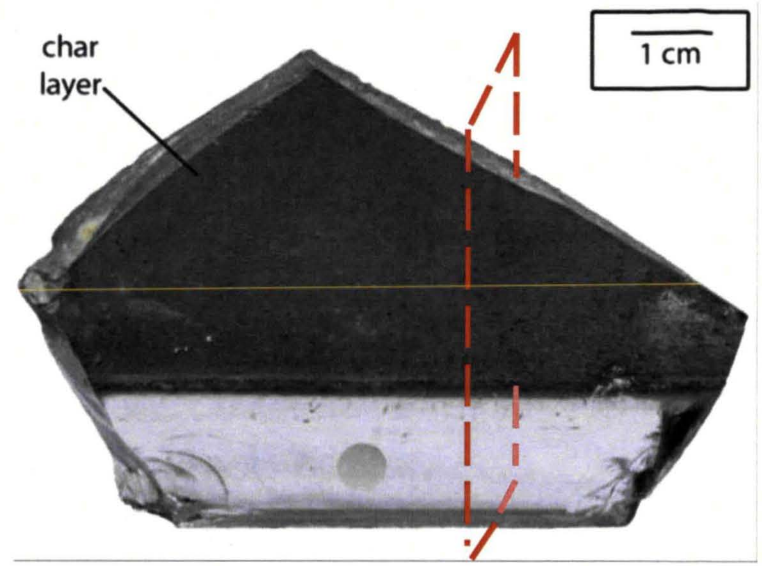
- The Orbiter windows are comprised of three panes:
  - Inner pressure pane, tempered aluminosilicate glass, 0.625” thick
  - Center pane, low-expansion fused silica for thermal shock, 1.3” thick
  - Outer thermal pane, tempered aluminosilicate glass, 0.625” thick (interior is coated, exterior is not and withstands 430°C)
- Window fragments were removed from 8 thermal panes.
  - Concentrated on deposits on Window 8 overhead thermal panes.
  - Characterization of darkened char that had deposited on overhead windows.

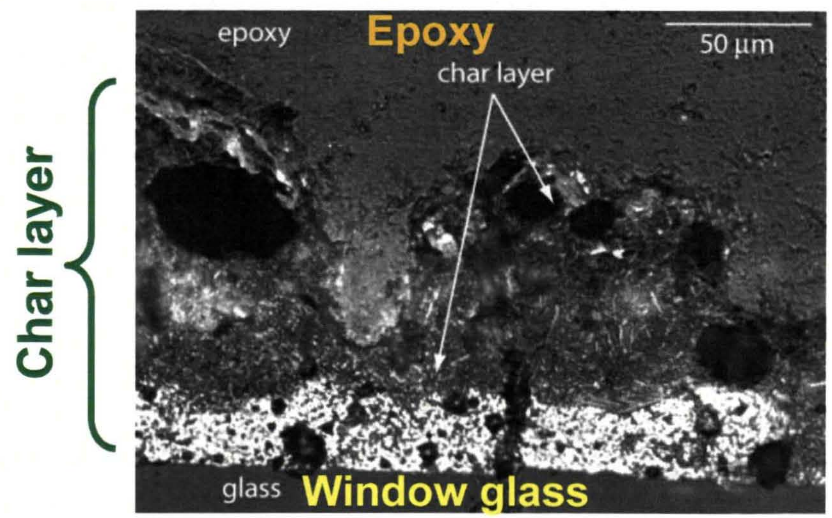




Char layer Sample Prep & OM

- A fragment of glass containing considerable amount of dark "char" layer on the surface was removed from the frame for characterization.
- Preparation included using cold mounting two-pour techniques to preserve the char layer.
  - The sample was first completely covered in epoxy and allowed to cure over night.
  - Then the mounted sample was cross-sectioned and cut to desired shape to reveal the area of interest.
  - The second sample was re-mounted using a two-part epoxy, providing a preserved cross section of the char layer
- Optical microscopy (OM) revealed stratified char layer:
  - Bright and reflective constituent near glass interface
  - Non-uniform, erratically deposited needle-like constituents in middle layer
  - Dark globular constituents throughout

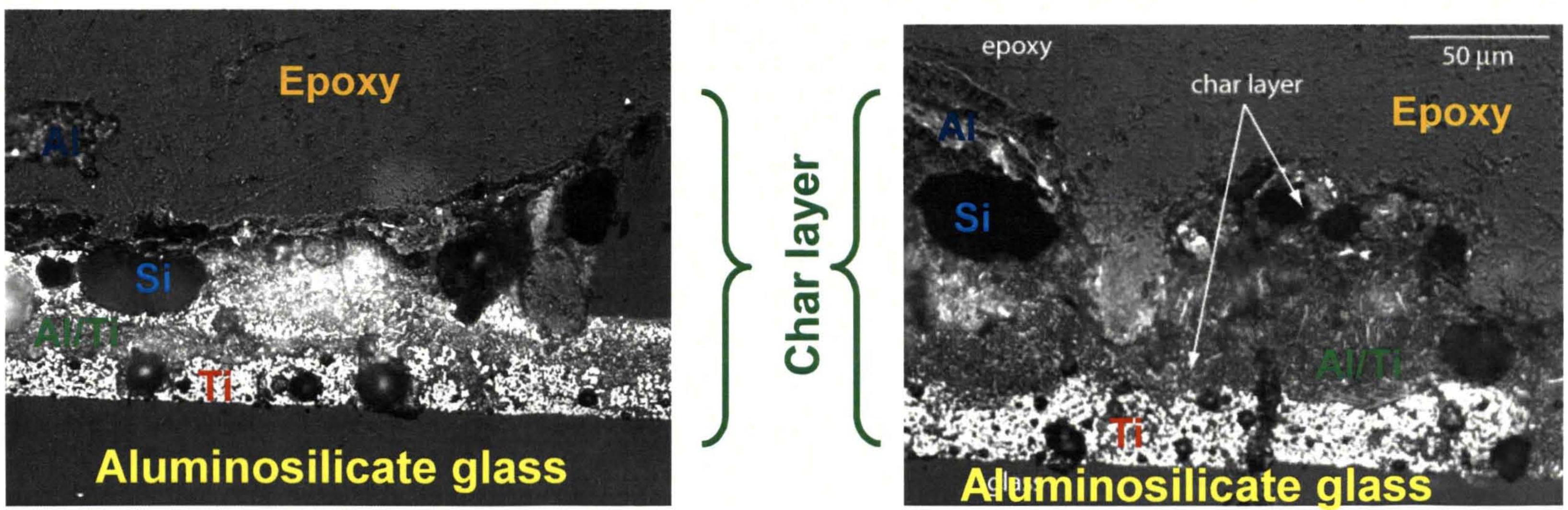




Original Magnification: 500X

Char layer SEM & EDS

- Scanning electron microscope (SEM) and Energy Dispersive Spectroscopy (EDS) work characterized layers as:
  - Ti-rich oxide material near the glass substrate, reflective layer
  - Al/Ti-rich oxide material away from glass substrate, needle-like (acicular) layer
  - Si-rich globules throughout
  - Spongy Al layer furthest from glass, likely 2000-series
- Ti deposition being first in sequence of events seemed counter-intuitive:
  - ➢ Melting point of Al = 660°C
  - ➢ Melting point of Ti = **1668°C**
  - Need further understanding of deposition event.



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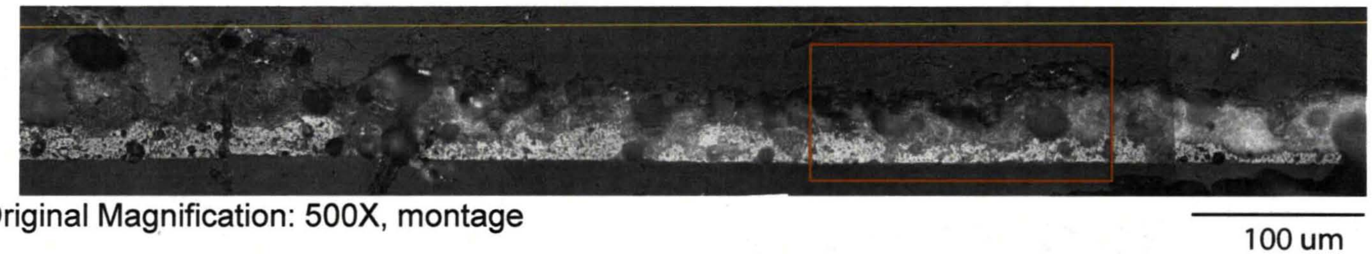
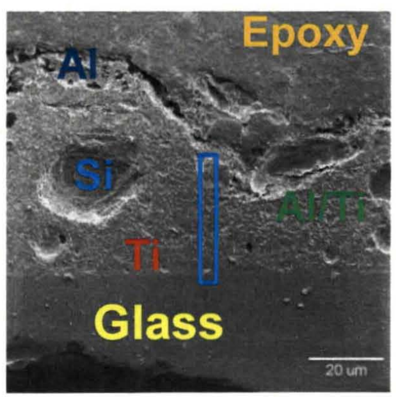
FIB Sample Preparation

- TEM analysis was needed to further characterize the phases in the char layer.
- Optical micrographs were used to determine areas best suited for the TEM:
  - Layer thickness
  - Constituents at that location (significant amounts of the Ti-rich and Al/Ti acicular layers)

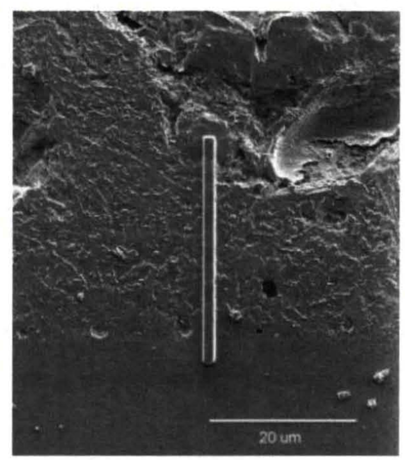


Original Magnification: 500X, montage

- Platinum was deposited over the Region of Interest (ROI) to protect the layer from ion damage during focused ion beam (FIB) sample preparation.



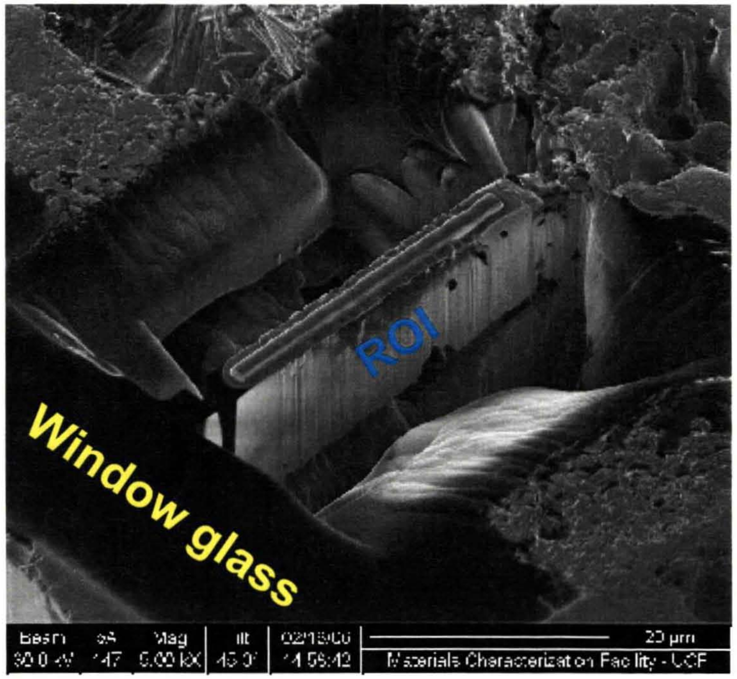
Original Magnification: 2000X

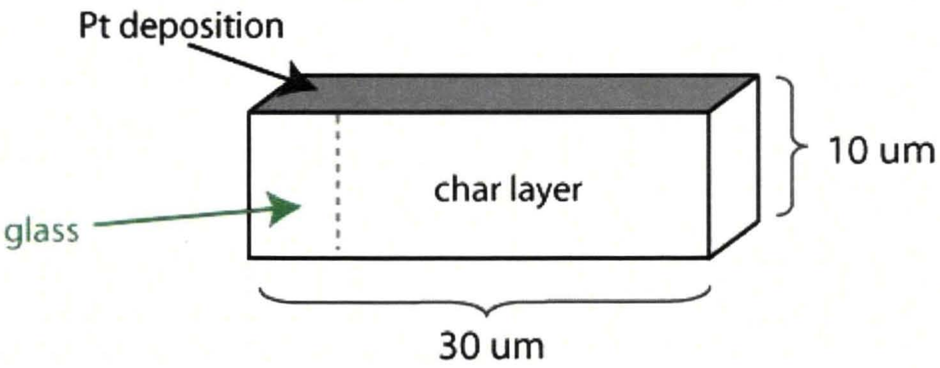


Original Magnification: 5000X

FIB In-Situ Sample Preparation

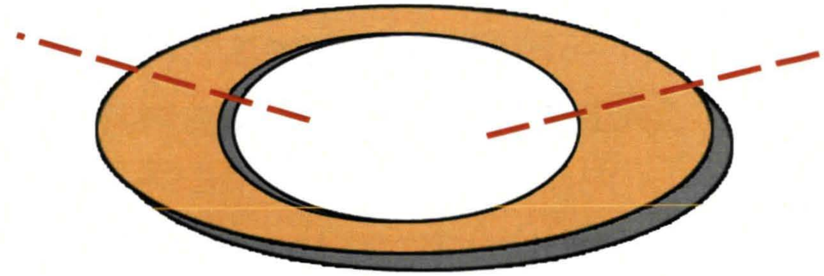
- In-situ sample removal:
  - A rectangular segment was created by milling "trenches" at the front and back of the ROI with the ion beam
  - Rectangular specimen was separated by tilting the stage 45° and using ion beam to cut all sides except one small ligament
  - Pt is used to adhere specimen to in-situ needle

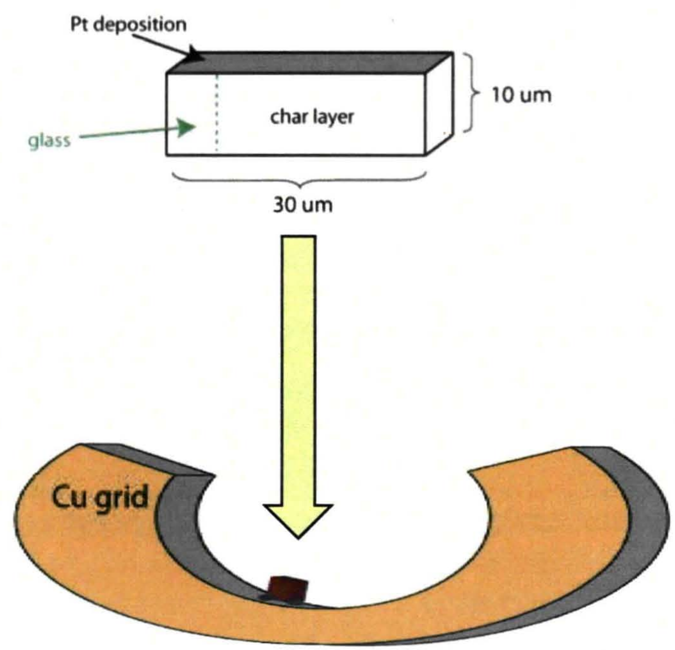




FIB In-Situ Sample Preparation

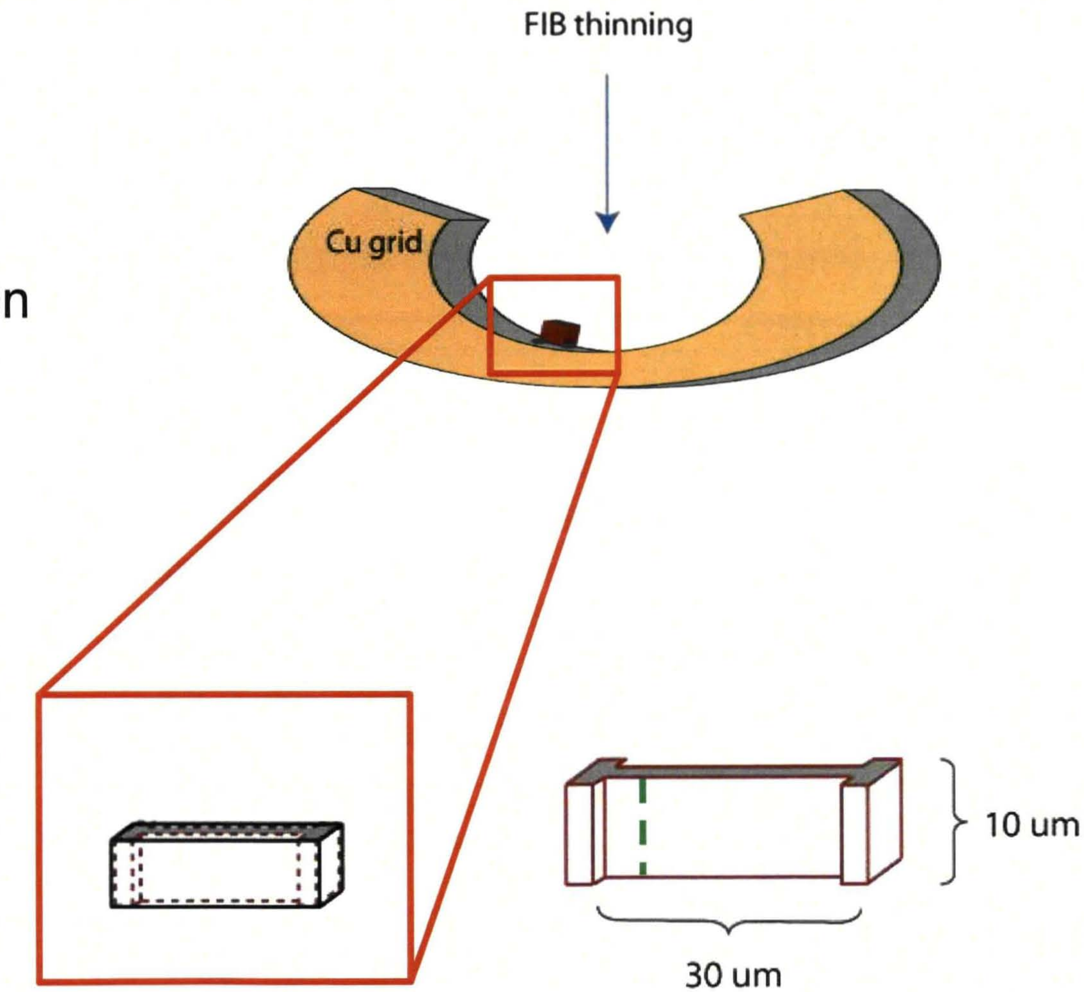
- Adhering sample to Cu grid:
  - A TEM copper grid was snipped to create a horse shoe shape
  - The FIB was used to create a ditch slightly larger than the rectangular sample on the inner radius of the Cu grid
  - Rectangular specimen was positioned inside ditch by lowering the in-situ needle and cut free
  - Pt deposition was used to "spot weld" the rectangular specimen in place





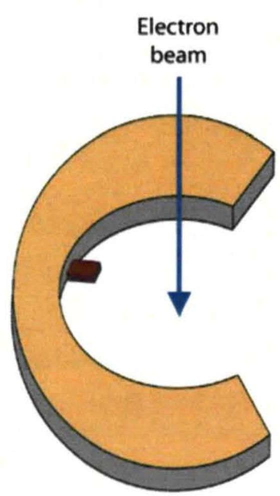
FIB In-Situ Sample Preparation

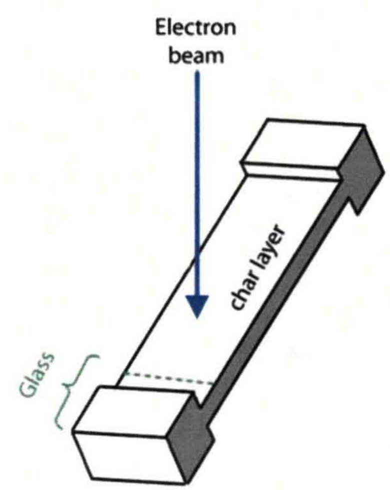
- Specimen thinning:
  - The FIB was used to thin out the rectangular specimen to thickness of <1000 Å to make it electron transparent
  - As a result of material removal, the sample took the shape of an I-beam, with the thicker regions as support "flanges"
  - Specimen was ready for TEM evaluation



TEM Evaluation

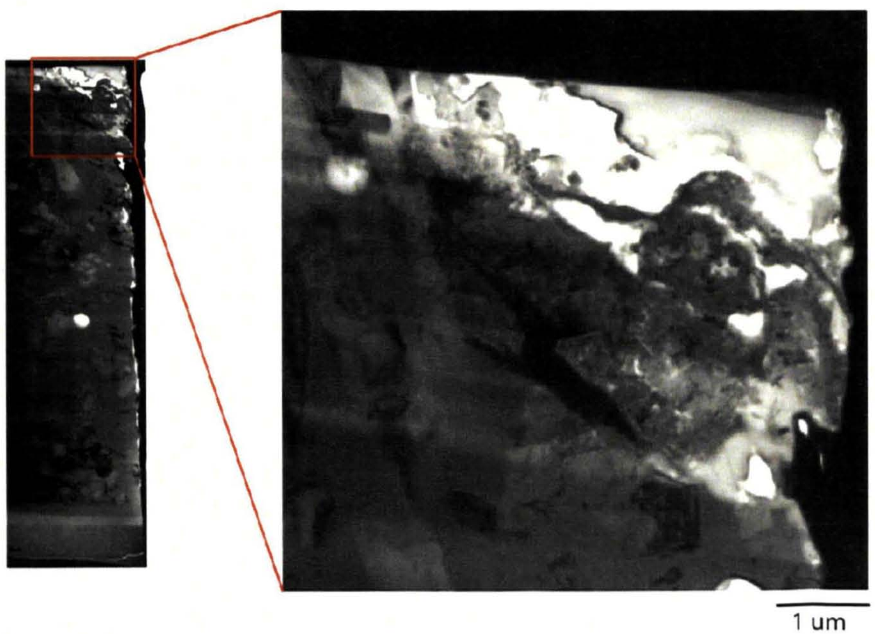
- Specimen inserted in TEM for analysis
- The glass portion of the specimen spanned the “flange” and “web” of the I-beam
  - Between the two regions, there is a transition region (varying thicknesses)
  - A distinction in contrast becomes apparent between the thicker portions and the thinner portions of the glass phase

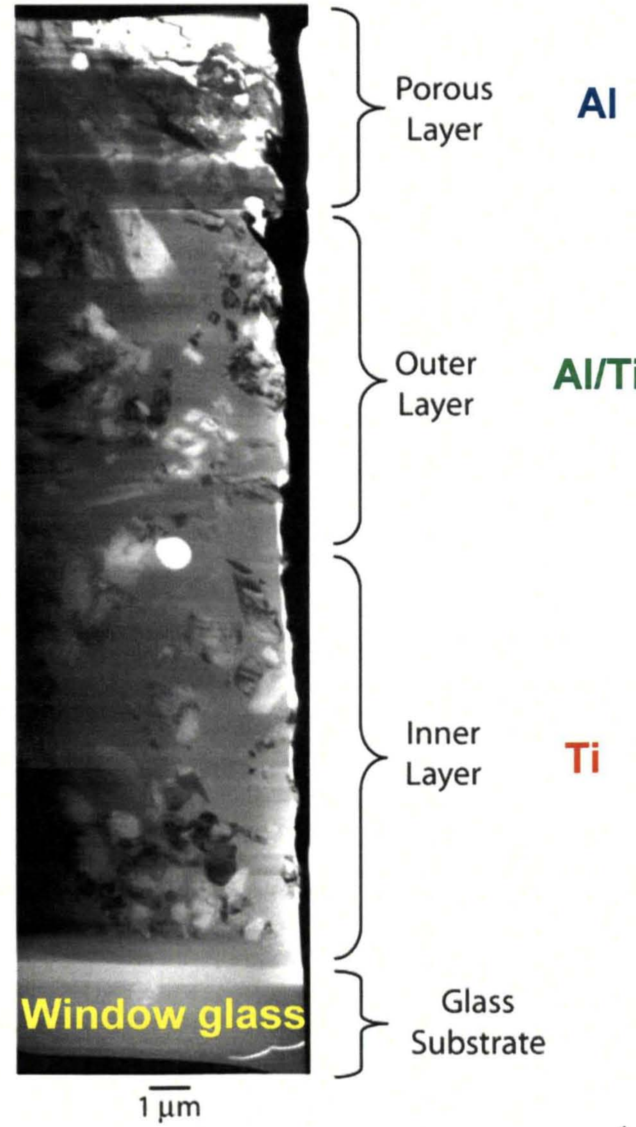




TEM of Char Layer

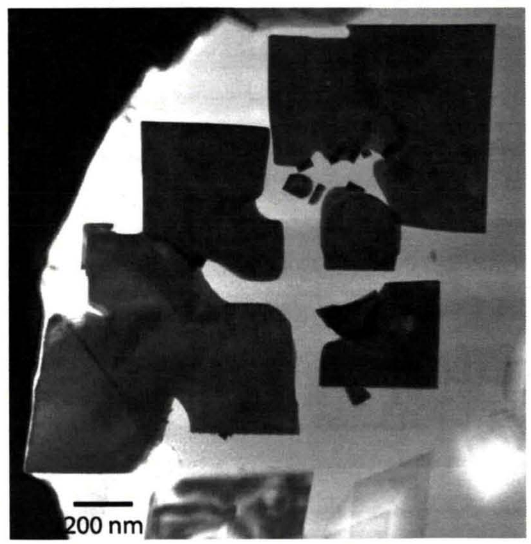
- TEM analysis gave a wealth of information
- TEM images, Selected Area Electron Diffraction (SAED) and EDS analysis revealed crystallographic oxide phases dispersed throughout a glassy matrix
- Regions were referred to as "porous layer", "outer layer" and "inner layer"





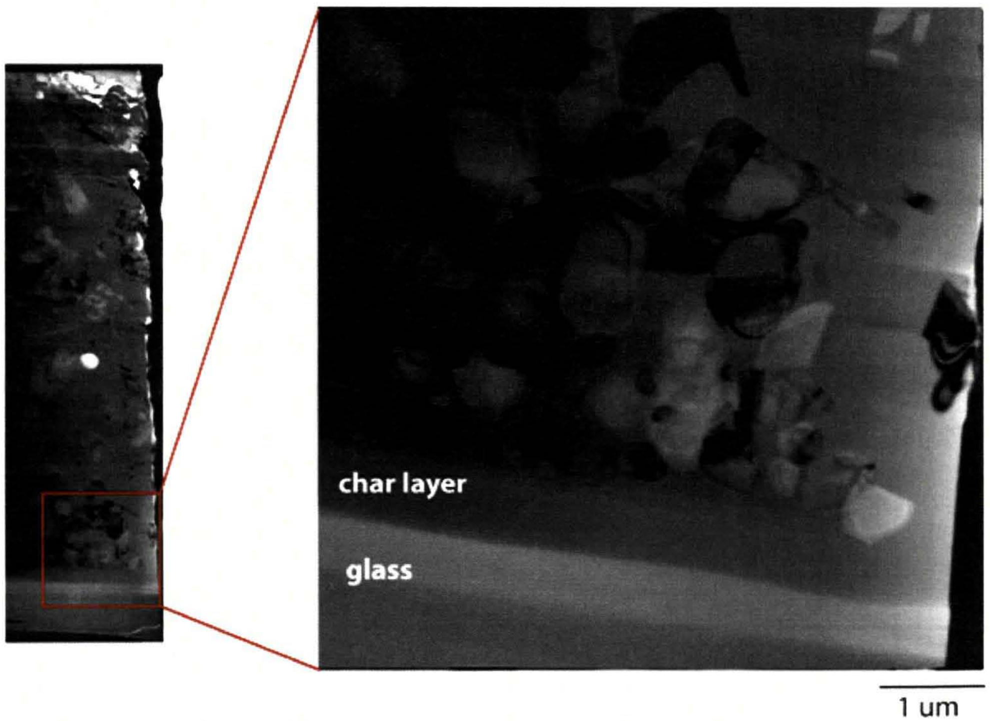
Outer Layer TEM

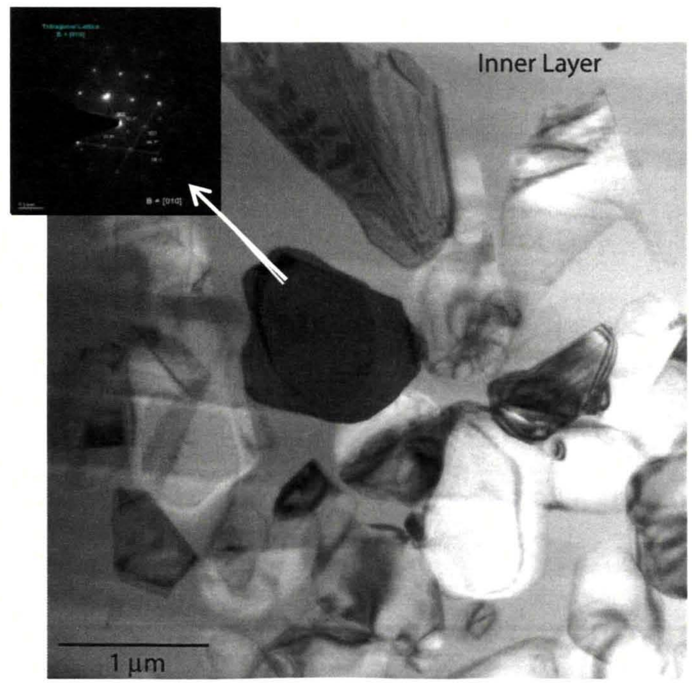
- Elongated crystals further from the window were a crystalline aluminosilicate phase called mullite ($Al_{4+2x}Si_{2-2X}O_{10-x}$).
  - 11 um from glass substrate
  - As small as 500 nm x 750 nm to 1 um x 3.5 μm
- Relatively homogeneous grains
- Some exhibited skeletal morphology due to rapid crystallization from a silicate melt



Inner Layer TEM

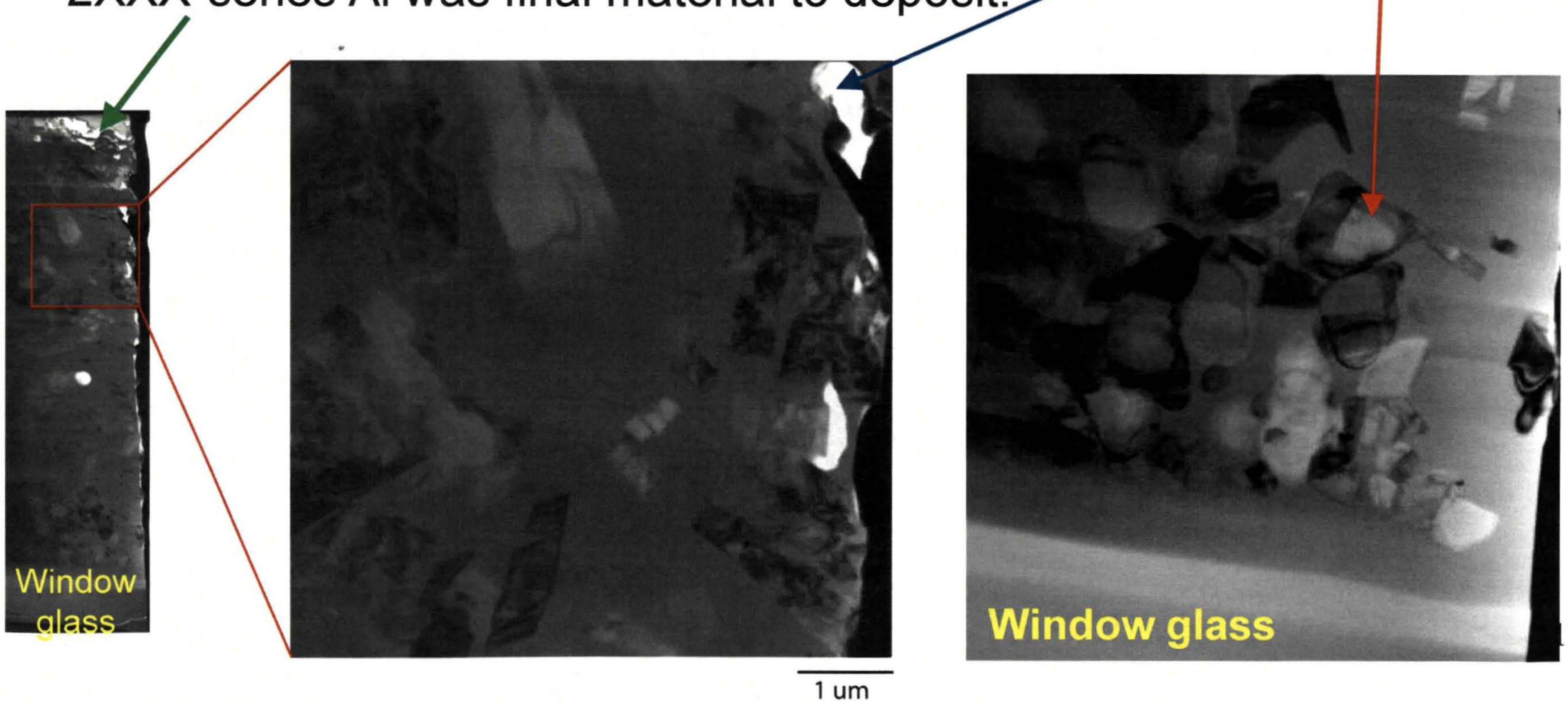
- Crystals closer to the window glass were identified as titanium dioxide (TiO_2, rutile).
  - Crystals were equidimensional near glass substrate interface and appeared in polycrystalline clusters or isolated islands
  - Morphology became more irregular further from glass interface
  - Crystal size ranged from 300 nm to 1.8 μm





Summary of Char Layer TEM Results

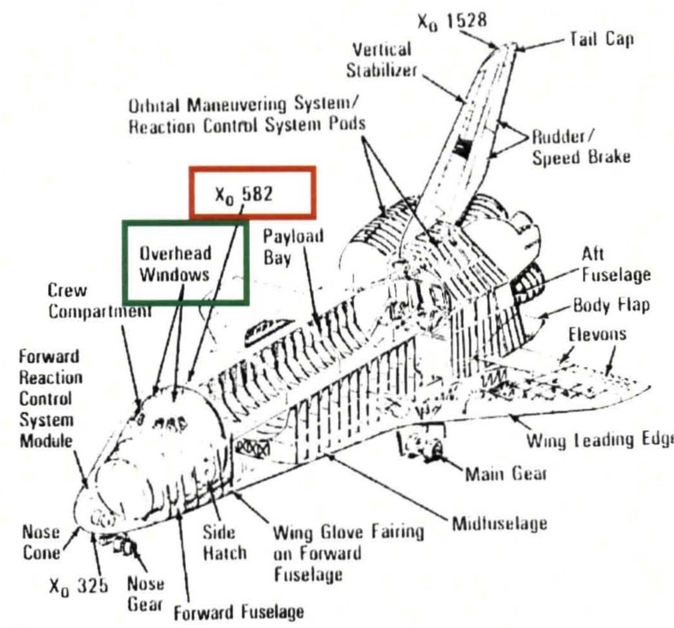
- Found that each region was comprised of crystals dispersed in a glass matrix (aluminosilicate).
- Crystals closer to the window glass were titanium dioxide (TiO_2).
- Elongated crystals further from the window were a crystalline aluminosilicate phase called mullite ($Al_{4+2x}Si_{2-2X}O_{10-x}$).
- 2XXX-series Al was final material to deposit.



Temperatures Experienced

- A gradient in glass composition throughout the char layer was used to determine the temperature at which crystallization occurred.
  - Components underwent temperatures above 1700°C prior to being deposited onto the glass substrate.

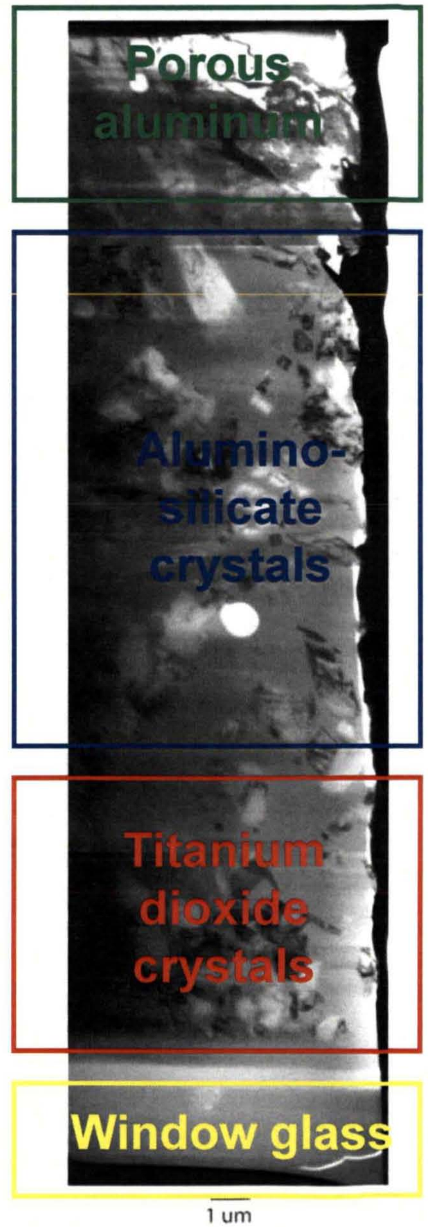
- Three-zone char layer was only observed on overhead windows 7 and 8 thermal panes
  - Majority of structural material surrounding the overhead windows is 2000-series aluminum.
  - Payload bay bulkhead latch rollers in close proximity to the overhead windows and are composed of titanium alloy Ti-6Al-4V.
  - Thermal protection system (TPS) nearby is source for silicon.



Orbiter

Investigation Conclusions

- Microscopic evaluation of a char layer deposited during re-entry revealed an unexpected sequence of deposition events.
  - Expect Al to deposit first (lower melting point).
- Deposits from a Ti source preceded deposits from other sources.
  - Thermal erosion of suspect Ti-6-4 components was discovered.
- It was hypothesized that materials deposited on windows 7 and 8 were the result of combustion of a Ti-6Al-4V source in addition to interaction with surrounding Al and Si components.
  - Exothermic conversion from metallic Ti to oxidized Ti.
  - Additional heating beyond aerothermal heating was present.
- **Metallic combustion of structural components exposed to entry environments needs to be a design consideration in future spacecraft.**
  - **Environmental reactivity of sub-structural material if there is a chance of comprised thermal protection system (TPS).**



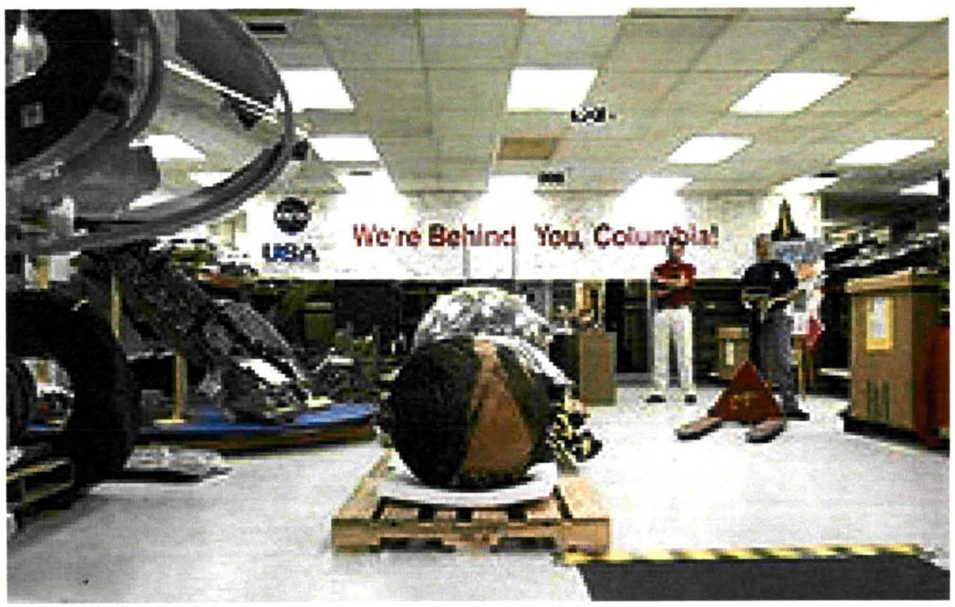
Conclusions relevant to FIB/TEM

- Astronaut Danny Olivas summed it up best:

"The many observations made on a 500 μm thick layer of char atop an inconspicuous fragment of the glass which participated in the Columbia *tragedy only serve to highlight the wealth of information that lies within her debris."*

"The nature of the incident and subsequent breakup allow engineers to learn how to not only make our existing space flight safer, but provide a greater breadth of core knowledge for the next generation of rocket designers…as human beings venture go beyond low earth orbit and on into deep space."

- *Columbia* debris was made available through the *Columbia* Debris Loan Program
- Debris is available to academic institutions by contacting Richard.W.Russell@nasa.gov



http://www.nasa.gov/missions/shuttle/*columbia*_debris.html

Equipment Used in Investigation

- FEI 200 TEM FIB
  - 30 kV Ga liquid metal ion source
- FEI Tecnai F30 TEM
  - 300 kV FE source, STEM
- JEOL 2500SE FE STEM
  - High Angle Annular Dark Field (HAADF)
  - X-ray Energy Dispersive Spectroscopy (EDS)
- Zeiss Axiovert Metallograph
- Philips XL 40 FEG SEM

References

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5. J. D. Olivas, M. C. Wright et al, Acta Astronautica, *Crystallographic oxide phase identification of char deposits obtained from space shuttle* Columbia *window debris;* Vol. 67, Issues 5-6, September-October 2010
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