



Kennedy Space Center
Engineering and Technology

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



M. Clara Wright
Materials Engineer
Materials Failure Analysis Section at KSC

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Kennedy Space Center Engineering and Technology

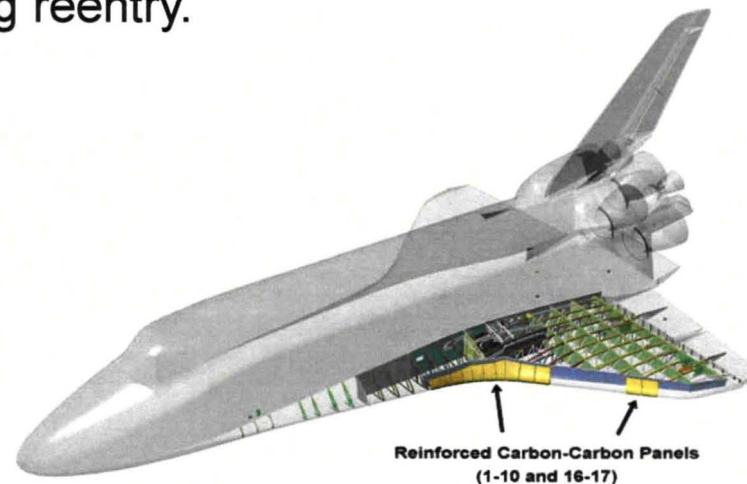
Research Collaboration

- Kennedy Space Center (KSC)
 - Materials Science Division: Clara Wright, Steve McDanel
 - 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





- 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.



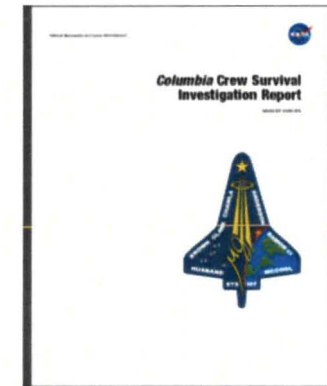
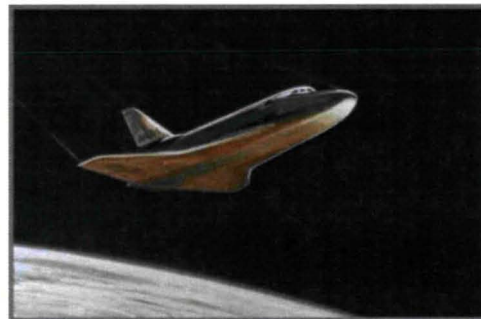


Columbia window lying exterior-side up. Photographed at the *Columbia* reconstruction hangar at KSC on March 3, 2003. Available at <http://caib.nasa.gov/>

- 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].



- 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>

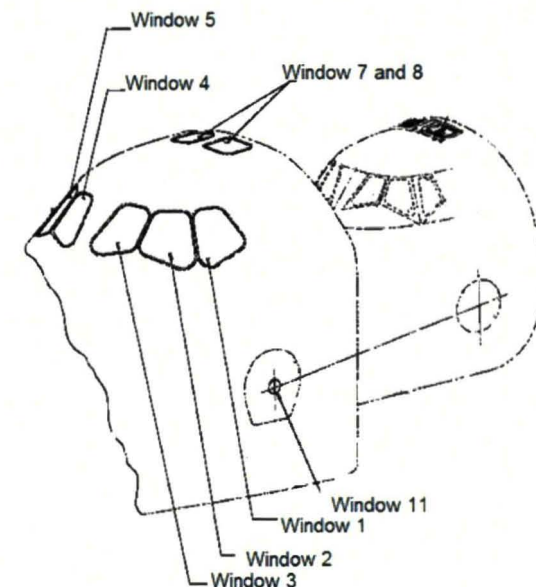
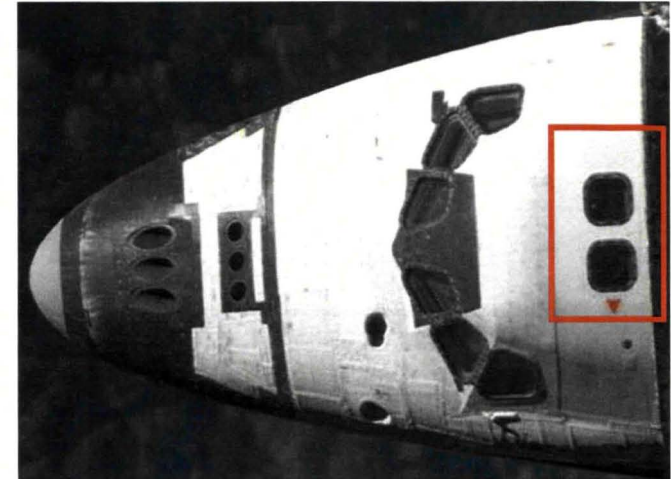
- The research presented in this talk is discussed in detail in the following article:

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

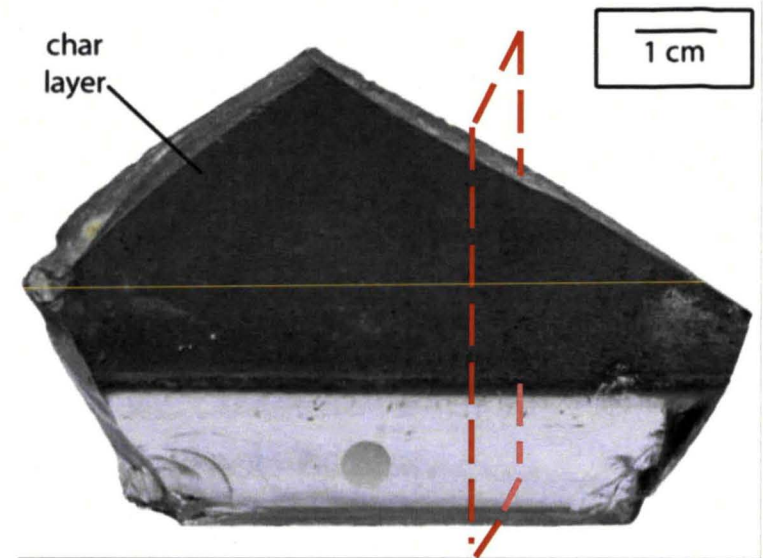
- The Orbiter windows are comprised of three panes:
 - Inner pressure pane, tempered aluminosilicate glass, 0.625" thick
 - Center pane, low-expansion fused silica for thermal shock, 1.3" thick
 - Outer thermal pane, tempered aluminosilicate glass, 0.625" thick (interior is coated, exterior is not and 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.



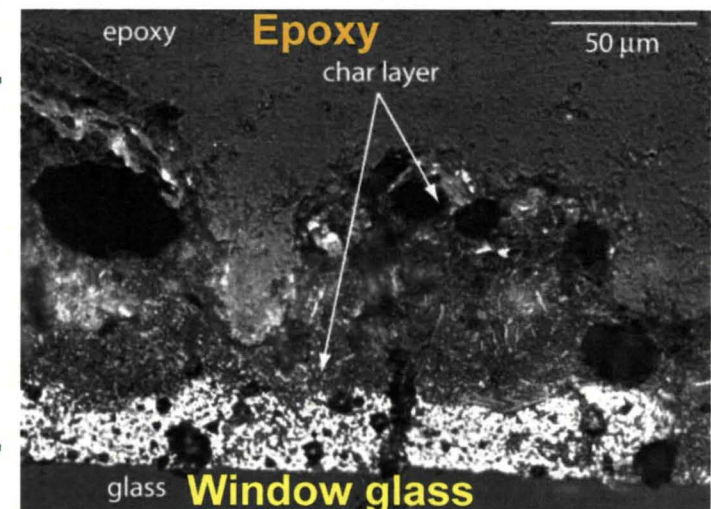


Char layer Sample Prep & OM

- 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



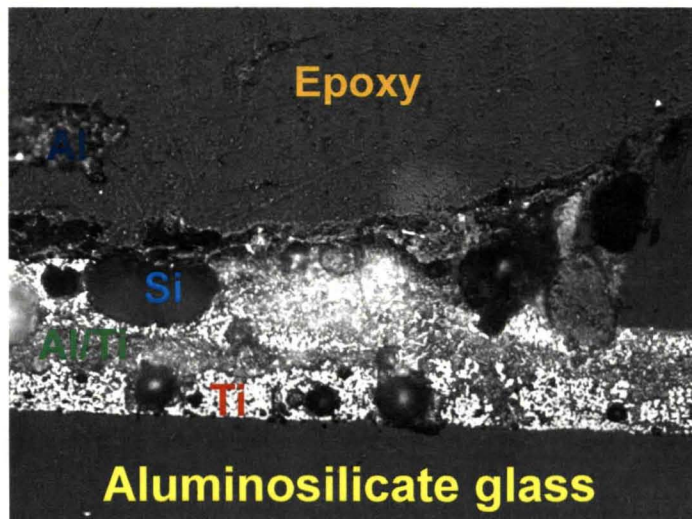
Char layer



Original Magnification: 500X

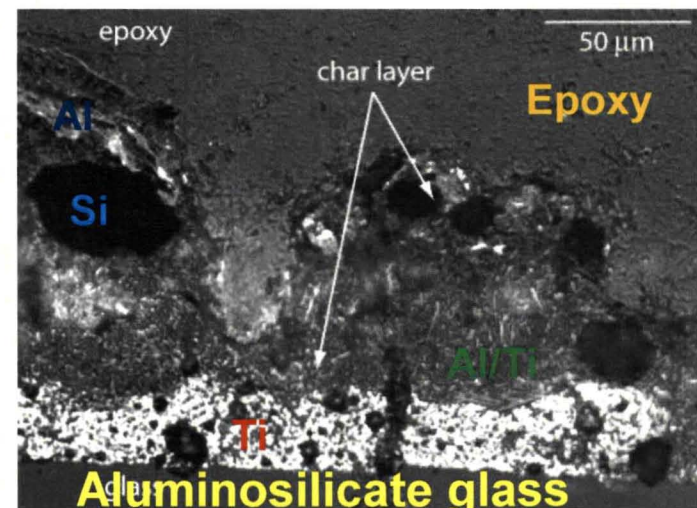


- Scanning electron microscope (SEM) and Energy Dispersive Spectroscopy (EDS) work characterized layers as:
 - Ti-rich oxide material near the glass substrate, reflective layer
 - Al/Ti-rich oxide material away from glass substrate, needle-like (acicular) layer
 - Si-rich globules throughout
 - Spongy 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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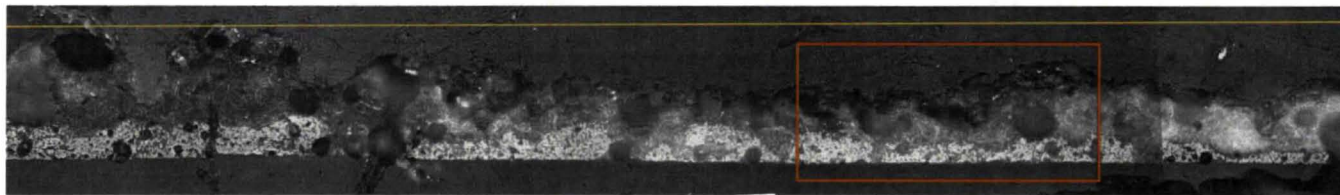
Char layer



Original Magnification: 500X



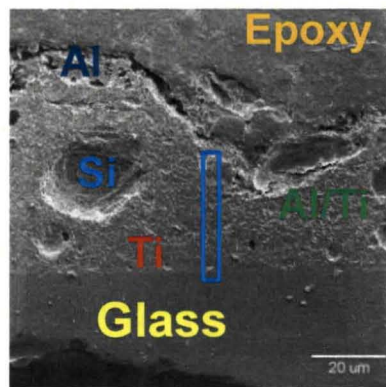
- TEM analysis was needed to further characterize the phases in the char layer.
- Optical micrographs were used to determine areas best suited for the TEM:
 - Layer thickness
 - Constituents at that location (significant amounts of the Ti-rich and Al/Ti acicular layers)



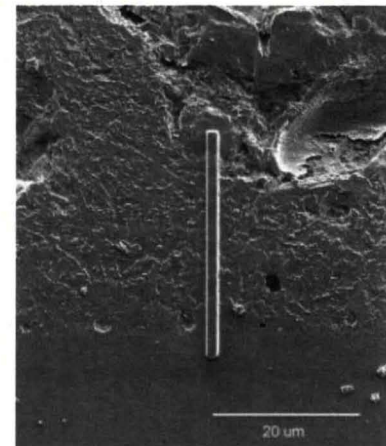
Original Magnification: 500X, montage

100 um

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



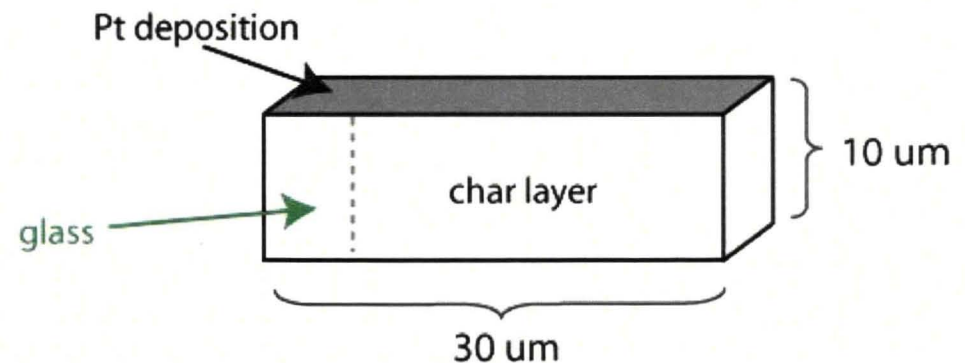
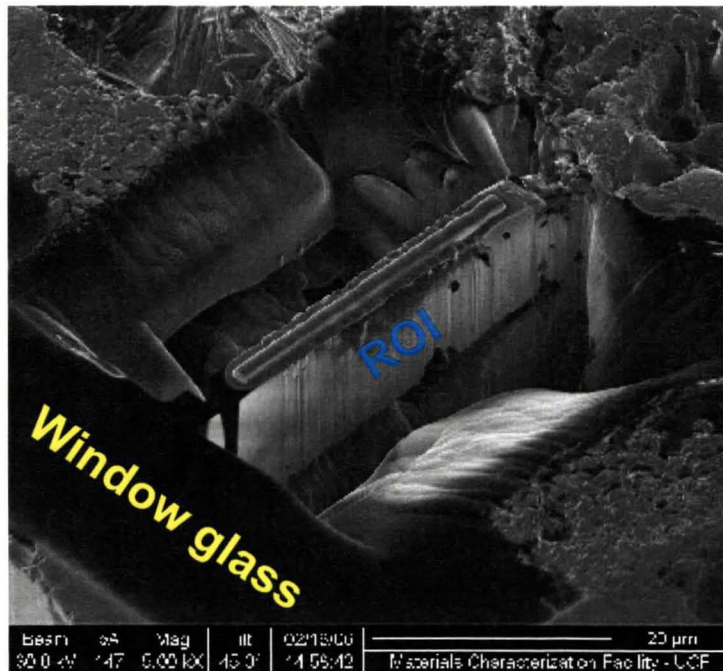
Original Magnification: 2000X



Original Magnification: 5000X

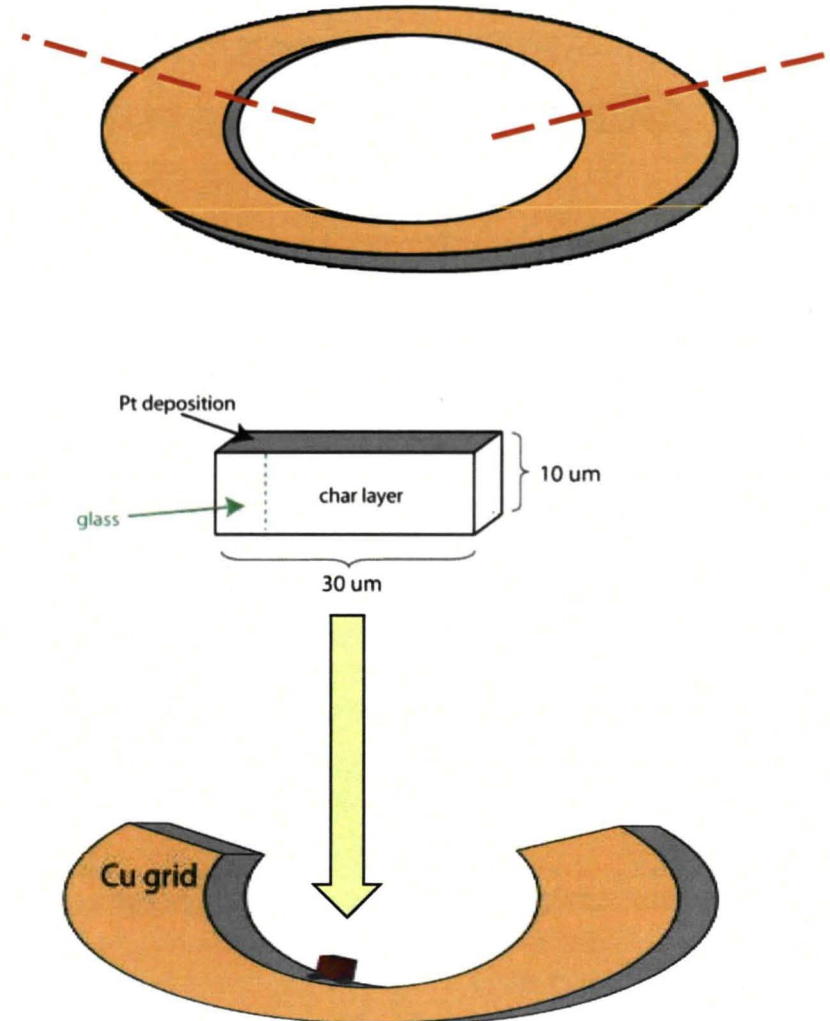


- In-situ sample removal:
 - A rectangular segment was created by milling “trenches” at the front and back of the ROI with the ion beam
 - Rectangular specimen was separated by tilting the stage 45° and using ion beam to cut all sides except one small ligament
 - Pt is used to adhere specimen to in-situ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



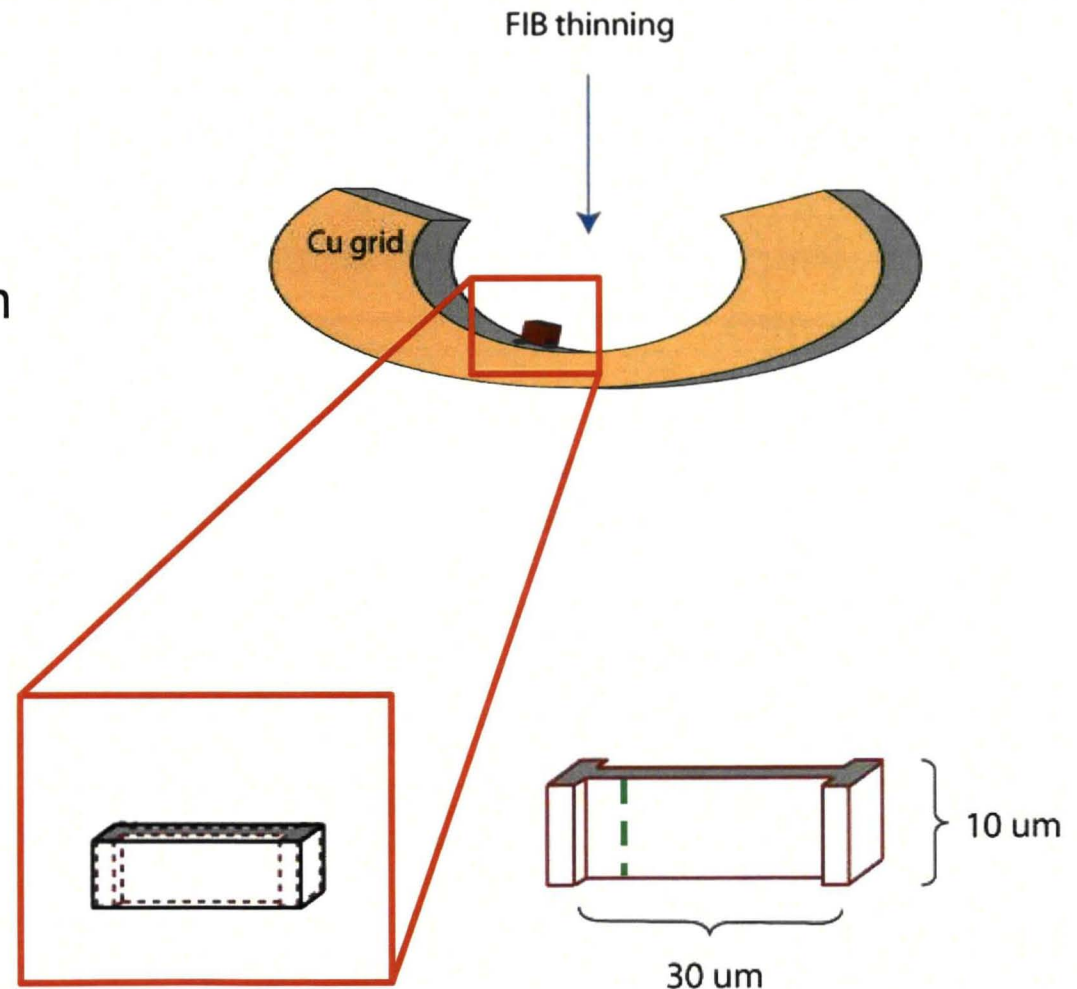


- Adhering sample to Cu grid:
 - A TEM copper grid was snipped to create a horse shoe shape
 - The FIB was used to create a ditch slightly larger than the rectangular sample on the inner radius of the Cu grid
 - Rectangular specimen was positioned inside ditch by lowering the in-situ needle and cut free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



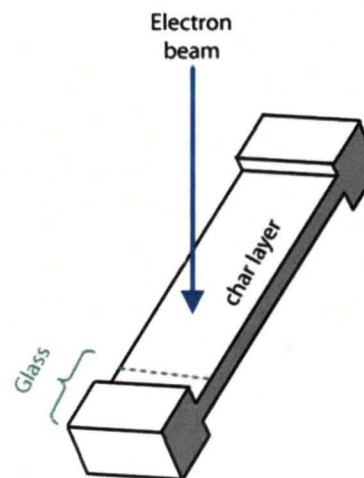
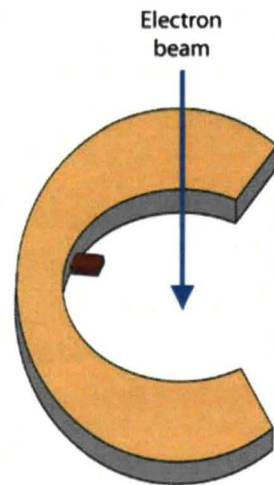


- Specimen thinning:
 - The FIB was used to thin out the rectangular specimen to thickness of $<1000 \text{ \AA}$ to make it electron transparent
 - As a result of material removal, the sample took the shape of an I-beam, with the thicker regions as support “flanges”
 - Specimen was ready for TEM evaluation





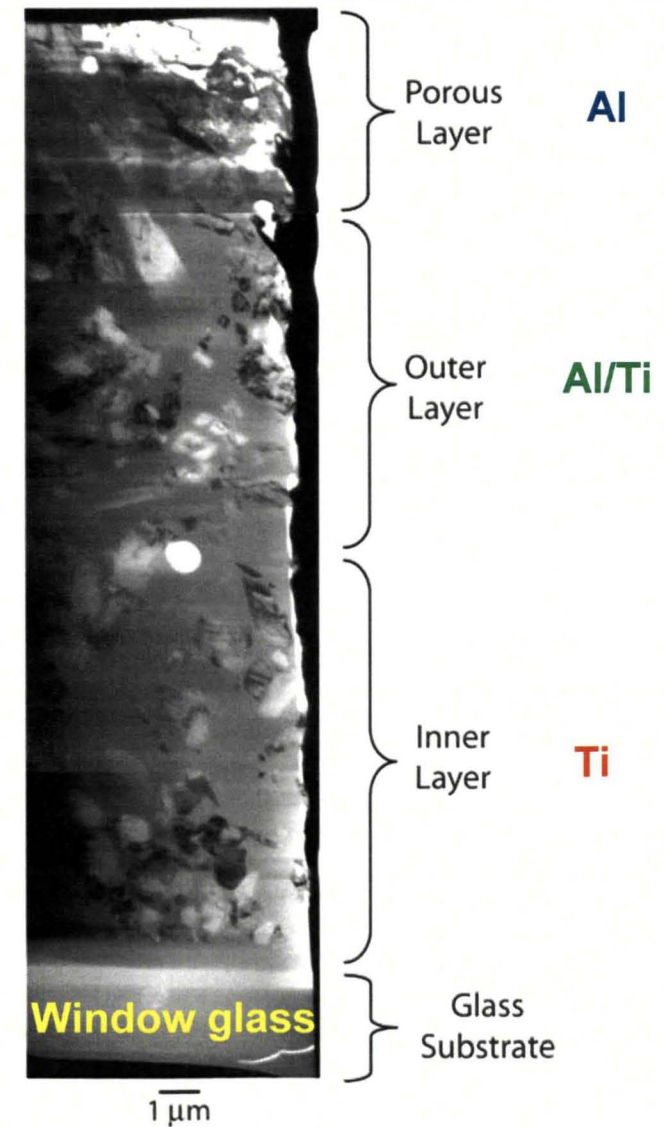
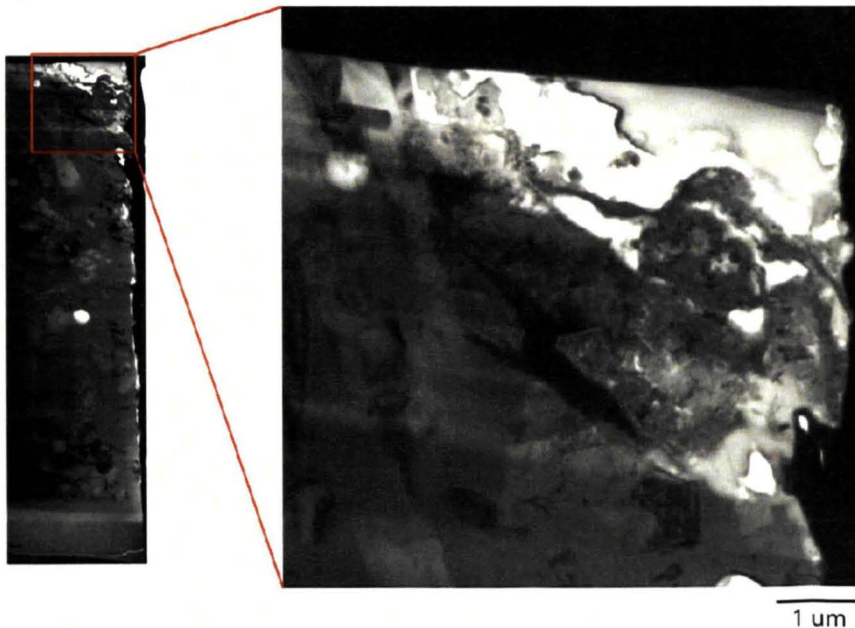
- Specimen inserted in TEM for analysis
- The glass portion of the specimen spanned the “flange” and “web” of the I-beam
 - Between the two regions, there is a transition region (varying thicknesses)
 - A distinction in contrast becomes apparent between the thicker portions and the thinner portions of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





TEM of Char Layer

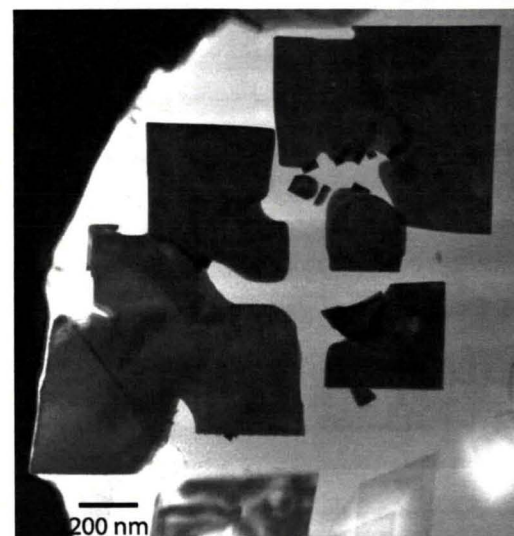
- TEM analysis gave a wealth of information
- TEM images, Selected Area Electron Diffraction (SAED) and EDS analysis revealed crystallographic oxide phases dispersed throughout a glassy matrix
- Regions were referred to as “porous layer”, “outer layer” and “inner layer”





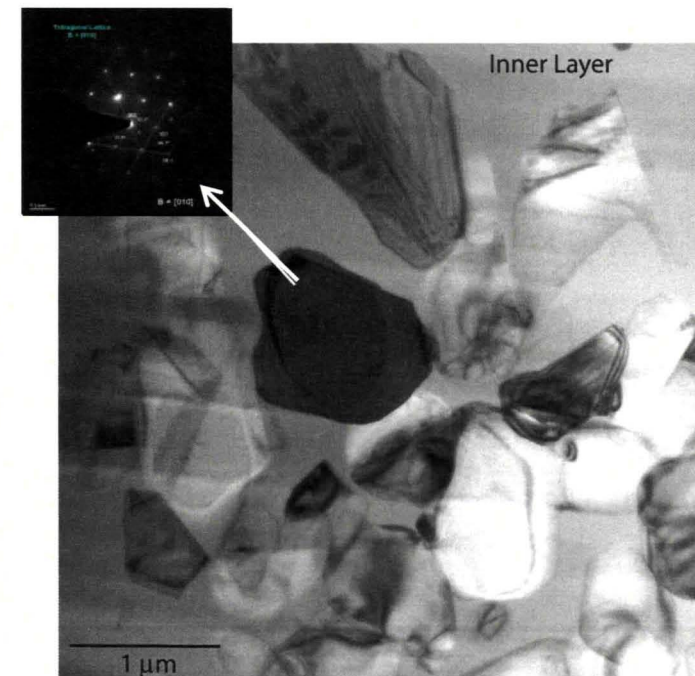
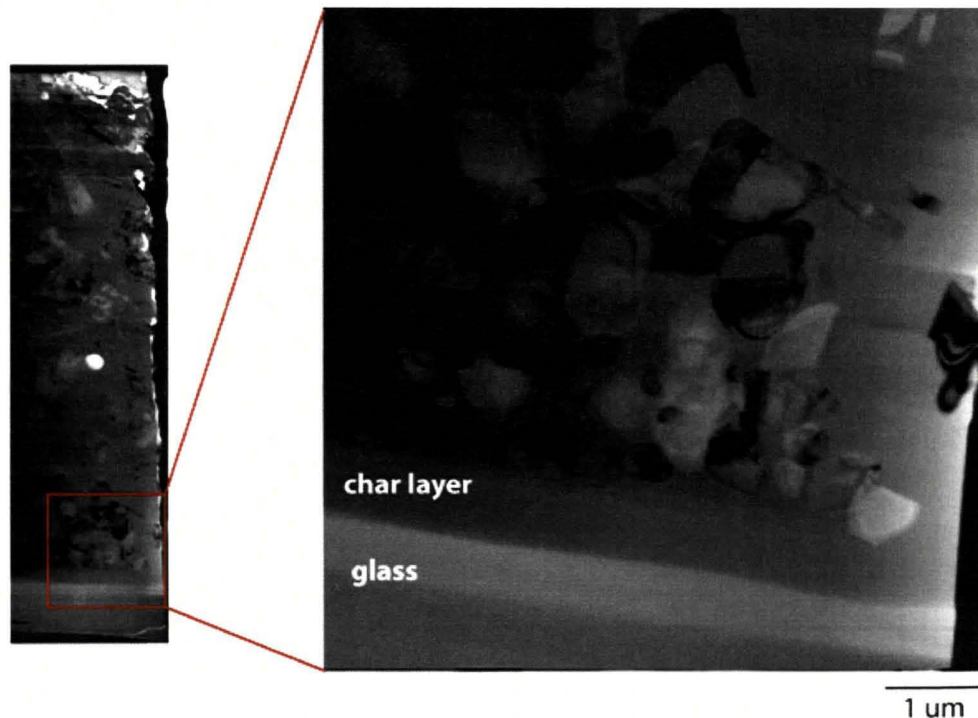
Outer Layer TEM

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



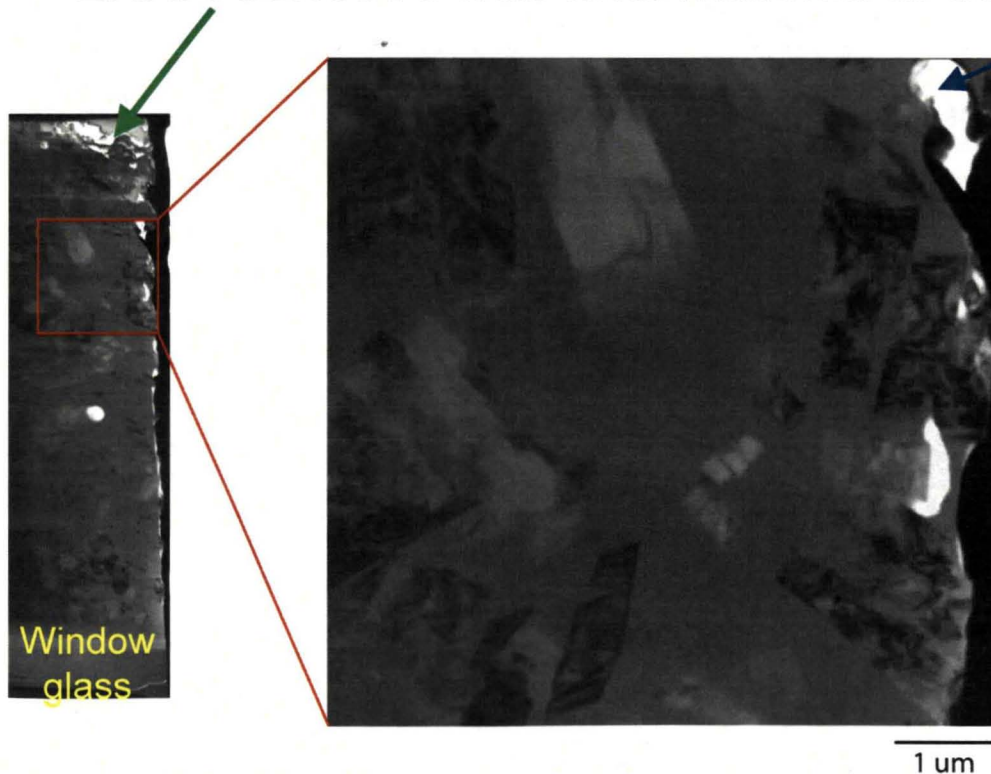


- Crystals closer to the window glass were identified as titanium dioxide (TiO_2 , rutile).
 - Crystals were equidimensional near glass substrate interface and appeared in polycrystalline clusters or isolated islands
 - Morphology became more irregular further from glass interface
 - Crystal size ranged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



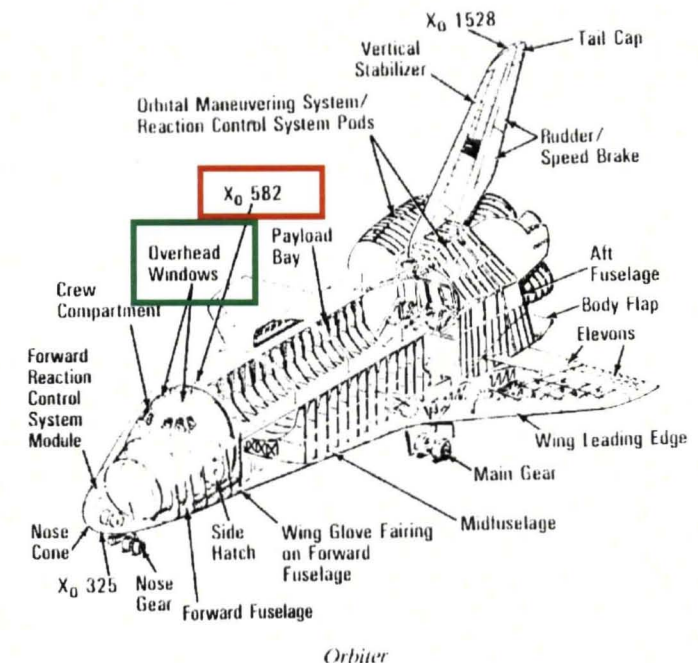


- Found that each region was comprised of crystals dispersed in a glass matrix (aluminosilicate).
- Crystals closer to the window glass were titanium dioxide (TiO_2).
- Elongated crystals further from the window were a crystalline aluminosilicate phase called mullite ($\text{Al}_{4+2x}\text{Si}_{2-2x}\text{O}_{10-x}$).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



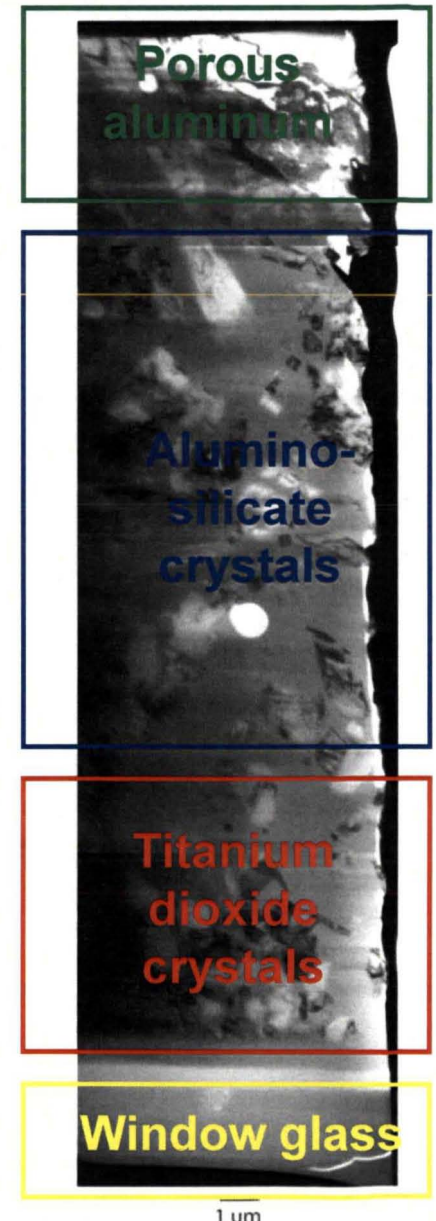


- A gradient in glass composition throughout the char layer was used to determine the temperature at which crystallization occurred.
 - Components underwent temperatures above 1700°C prior to being deposited onto the glass substrate.
- Three-zone char layer was only observed on overhead windows 7 and 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.





- 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).





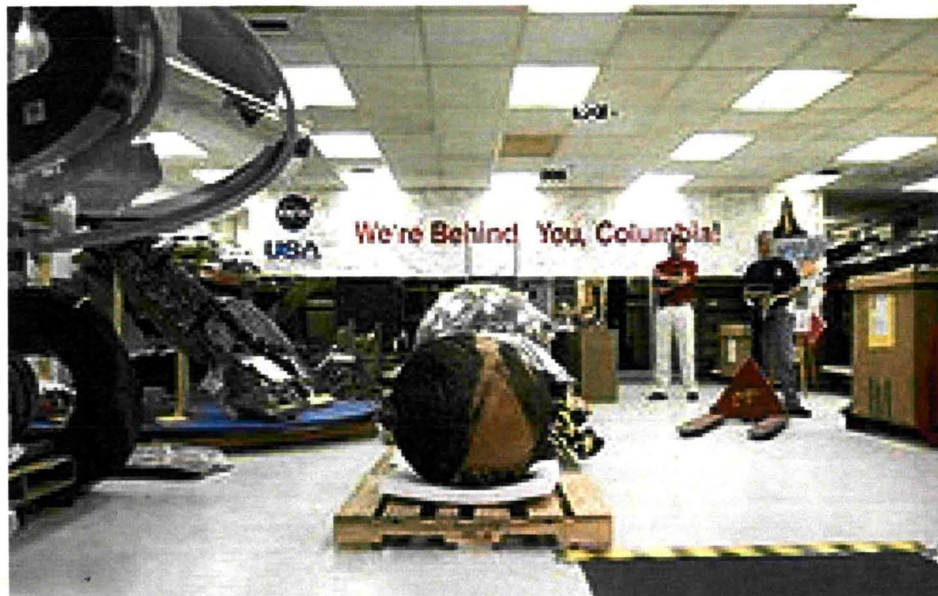
- Astronaut Danny Olivas summed it up best:

“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





- **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**



1. *Columbia* Crew Survival Investigation Report, National Aeronautics and Space Administration, SP-2008-565, Washington, DC, December 2008.
2. *Columbia* Accident Investigation Board (CAIB), Report Volume 1, National Aeronautics and Space Administration, Washington, DC, August 2003
3. Mayeaux B, McDanel S, et al, Materials analysis: a key to unlocking the mystery of the *Columbia* tragedy", Journal of Materials, February 2004
4. Olivas JD, Melroy P, et al, OV-102 thermal pane window debris analysis, part of Spacecraft Crew Survival Integration Investigation Report, NASA
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
6. Z. Rahman, et al, Tutorial: In-Situ Site-Selective FIB for High Resolution TEM Sample Preparation, Microscopy and Analysis, May 2006
7. <http://spaceflight.nasa.gov/shuttle/archives/sts-107/investigation/leftwing/index.html>
8. <http://caib.nasa.gov/>
9. http://science.ksc.nasa.gov/shuttle/technology/sts-newsref/sts_coord.html