High Temperature Chemistry in the Columbia Accident Investigation

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Initial estimates on the temperature and conditions of the breach in Columbia's wing focused on analyses of the slag deposits. These deposits are complex mixtures of the reinforced carbon/carbon (RCC) constituents, insulation material, and wing structural materials. However it was possible to clearly discern melted/solidified Cerachrome® insulation, indicating the temperatures had exceeded 1760°C. Current research focuses on the carbon/carbon in the path from the breach. Carbon morphology indicates heavy oxidation and erosion. Raman spectroscopy yielded further temperature estimates. A technique developed at Sandia National Laboratories is based on crystallite size in carbon chars. Lower temperatures yield nanocrystalline graphite; whereas higher temperatures yield larger graphite crystals. By comparison to standards the temperatures on the recovered RCC fragments were estimated to have been greater than 2700°C.



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- ACCIDENT HISTORY, WHAT CAUSED BREACH
- Objectives: reinforced Carbon/Carbon Examine RCC)po - Examine RCC near breach and hot-gas-path
  - Understand response of RCC to extreme conditions
  - Estimate wing leading edge material temperatures near the breach <u>\*</u>compare to estimates from slag studies and
- Techniques
  - Electron microscropy (Glenn)
  - Raman spectroscopy (Sandia)

### **Identification of Breach**

- Multi-NASA Center Team Effort—JSC, MSFC, LaRC, GRC
- · Key features of recovered leading edge RCC
  - Large deposits of 'slag'—solidified Cerachrome insulation, aluminum alloys, Fe alloys, Ni alloys Conclusion delle
  - 'Knife edge' appearance of RCC (reinforced carbon/carbon)—known to occur when an exposed edge of RCC is heavily oxidized



#### **Previous Temperature Estimates**

- Cerachrome® mp 1760°C—95% aluminosilicate; 3% chromia
- Mullite forms at ~1100°C

10

 Examination slag deposits from upper portion of Panel 8 identified mullite and melted Cerachrome ⇒ 1760°C or greater



### Slag Deposits—Complex Mixtures of Wing Constituents



13.0mm

Region 12 – portion of carbon fiber

5

12 A3 15.0kV 13.0mm x1.00k SE(L) 04/15/200 50 0um

#### Solidified metals and ceramics

#### Region of Melted Cerachrome Only well-understood Feature for Temperature Determination



#### Summary: 'Slag' Deposits on back Side of Wing Leading Edge

- Complex mixture of solidified ceramics, aluminum alloys, iron alloys, and nickel alloys
- Only solidified cerachrome gave temperature clues
- Focus on fractured and oxidized carbon/carbon



# **Location of Carbon Samples**



recovered?

## Typical Appearance of RCC Cross Section Fragment



Inner mold line

Slag—solidified insulation (Cerachrome) and metals

Fractured/oxidized carbon fibers and matrix—Focus of this study

Outer mold line

Characteristic Pointed Fibers from High Temperature Oxidation



#### Extensive Oxidation and Erosion of Fibers and Matrix



Panel 8—This layered, plate-like structure is characteristic of heavy erosion in Carbon

#### Unusual Features in Carbon Matrix Flowing Carbon? Melting Point of Carbon is 3500 K



EDS-F EDS-E 58291-B 6.0kV 24.3mm ×15.0k SE(L) 5/5/2004

#### **Overall View**

Close-up of veins—all Carbon

OFCIC? OFC?

Raman Standards—Characteristic Peak Heights, Change with Temperature





#### Raman Analyses of Portion of Upper Panel 8, near Breach

Item #49619A







Location	Location Description	Summary of Identified Bands
1	Knife edge	2700 C carbon plus small amount of low temp carbon
2	Knife edge	2700 C carbon plus small amount of low temp carbon
3	Knife edge	>> 2700 C carbon (off scale of reference carbons)
4	Knife edge	> 2700 C carbon (off scale of reference carbons)
5	Up slope	2400 C carbon
6	Up slope	2700 C carbon
7	Up slope	> 2700 C carbon (off scale of reference carbons)
8	Up slope	2400 C carbon



## Conclusions

- Preliminary results suggest temperatures may have been higher than previously derived from melting point of Cerachrome (1760°C).
- Raman spectroscopy ⇒ >> 2700°C
  Flowing carbon ⇒ 3500°C ??
- Extensive oxidation and erosion
  - Pointed fiber morphology
  - Exposed layers of carbon—erosion

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# In Memoriam...

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