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Analysis of Carbon/Carbon Fragments From the Columbia Tragedy

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December 2005

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In Memoriam

This report is dedicated to the seven astronauts who perished in the Columbia tragedy.
To learn from their mission is to honor their memory.

Rick D. Husband, Commander
William C. McCool, Pilot
Michael P. Anderson, Mission Specialist
Kalpana Chawla (KC), Mission Specialist
David Brown, Mission Specialist
Laurel Blair Salton Clark, Mission Specialist
Ilan Ramon, Payload Specialist

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Abstract

The extensive investigation following the Space Shuttle Orbiter Columbia accident of February 1, 2003 determined that hot gases entered the wing through a breach in the protective reinforced carbon/carbon (RCC) leading edge. In the current study, the exposed edges of the recovered RCC from the vicinity of the breach are examined with scanning electron microscopy and Raman spectroscopy. Electron microscopy of the exposed edges revealed regions of pointed carbon fibers, characteristic of exposure to high temperature oxidizing gases. The Raman technique relates the observed 1350 and 1580 to 1600 cm^{-1} bands to graphitic domains and their corresponding temperatures of formation. Some of the regions showed evidence of exposure temperatures beyond 2700 °C during the accident.

Introduction

The Wing Leading Edge Thermal Protection System

Figure 1 illustrates the reinforced carbon/carbon (RCC) panels on the wing leading edge and nose cap of the Space Shuttle Orbiters. These parts are exposed to the highest temperatures on re-entry and, when intact, this RCC material is an excellent thermal shield. The RCC on the wing leading edge is composed of 22 large panels designated with numbers 1 to 22 followed by R for the right (starboard) and L for the left (port). The numbers increase moving outward from the fuselage. The hottest parts are panels 8 to 11 which may reach temperatures of nearly 3000 °F (1650 °C) for ~900 sec during a normal re-entry.

The structure of the RCC material is shown in Figure 2. It is a two-dimensional lay-up of phenolic-resin-impregnated carbon fabric that has been cured and pyrolyzed. Densification is accomplished by repeating the following process: impregnation with a liquid carbon precursor, curing, and pyrolyzation. Oxidation protection is attained with several additional steps. First a SiC conversion coating is formed via Si(g) and SiO(g) infiltration. Due to the thermal expansion mismatch between SiC and carbon/carbon, this SiC coating develops cracks and fissures on cool-down. These are plugged with a vacuum infiltration of tetra-ethyl orthosilicate, which decomposes to SiO_2(s) on heat treatment. In addition two outer layers of a Type A sealant are applied. This is a sodium silicate glass with SiC grit and SiC chopped fibers as fillers. The glass is fluid at re-entry temperatures and fills open cracks or fissures.

The attachment of the RCC panels to the wing bulkhead is illustrated in Figure 3. Various alloy parts are used for attachment of the RCC panels to the wing. These are constructed to allow thermal expansion and contraction of the large RCC panels. The pocket between the RCC attachment hardware and the RCC contains Cerachrome® (Thermal Ceramics, Augusta, GA) insulation, which has the composition: 43 w/o Al_2O_3, 54 w/o SiO_2 and 3 w/o Cr_2O_3. This is used to protect the metallic components from the expected heat experienced during a normal re-entry.

The Columbia Tragedy and Ensuing Investigation

On February 1, 2003 the Space Shuttle Orbiter Columbia tragically disintegrated on re-entry to the earth's atmosphere. After an exhaustive investigation, it was determined that a panel of the protective RCC on the wing leading edge had been damaged by foam shed from the external tank during lift-off (Refs. 1 and 2).

The investigation involved a massive effort collecting Columbia debris. In an intensive 4 month effort, personnel from several NASA Centers, other government agencies, industry, and academia cataloged and analyzed the debris. This investigation has been discussed in the reports (Refs. 1 and 2). A large amount of RCC was recovered and it was possible to reconstruct much of the wing leading edge from the fragments.

The accident investigation determined that the breach was in the underside of panel 8L, allowing the entry of the hot plasma and a subsequent series of catastrophic events. Several factors led to this conclusion. As noted, panels 8 and 9 are among the hottest panels on re-entry. Several fragments of these panels showed a sharp 'knife edge' appearance on their sides, as shown in Figure 4. When an exposed edge of RCC is exposed to a high velocity plasma stream, a 'knife edge' develops (Ref. 3). Such edges were not observed on fragments collected from other panels.

A large concentration of droplet-like deposits were found on the backside of the upper portion of panel 8L. These were termed 'slag deposits' and were composed of Cerachrome insulation and structural metal components. In many cases there was the appearance of melting and solidification as well as partial reaction. The backside of panels 8L and 9L showed a substantially larger concentration of these slag deposits than did other panels.

It was initially hoped that some information could be obtained on the conditions which existed during accident (e.g., gas partial pressures and temperatures) from an analysis of the slag deposits, which could then be correlated with a point on the re-entry flight trajectory. However, the slag contained up to four different alloys and several ceramics and most slag analyses could not provide definitive pressures and temperatures.

Nonetheless, some regions showed melted and solidified Cerachrome insulation. The center region in Figure 5 was not melted as it is not faceted and has a composition similar to the as-fabricated Cerachrome. The outer regions had melted and solidified, precipitating out alumina. The fact that the Cerachrome appeared to have melted and solidified set the temperature near the breach at 1760 °C or greater.

Figure 6 is a diagram showing the probable path of plasma entry from the breach on the underside of panel 8L. The location of the slag deposits are also shown. The upper portion of panel 8L was recovered and only a small amount of panel 9L was recovered.

This study is a deeper examination of the affected RCC on panels 8, 9, and 10L in search of further information about the conditions of the accident. Recovered samples near the breach are closely examined with both scanning electron microscopy (SEM) and Raman spectroscopy to estimate maximum exposure temperatures of carbon based on the size of the formed graphitic domains (Refs. 3 to 5).

Experimental

Specimen Location and Electron Microscopy

A three-dimensional reconstruction of the port wing RCC panels 5 to 10 is shown in Figure 7. The seven arrows point out the locations where samples were taken, roughly following the probable entry pattern of the plasma. The numbers were assigned as the Columbia wreckage was collected and catalogued.

Ideally the same regions would be examined with both electron microscopy and Raman spectroscopy. However this was not possible in this study. Therefore two ~2.5×2.5 cm specimens were cut from each region—one was analyzed with electron microscopy at the NASA Glenn Research Center in Cleveland,

Ohio and one with Raman spectroscopy at Sandia National Laboratory in Albuquerque, NM. A suffix 'A' is given to the sample for Raman spectroscopy and a suffix 'B' is given to the sample for electron microscopy.

A field emission scanning electron microscope (FE-SEM, Hitachi S4700, Schaumberg, IL) equipped with an energy dispersive spectrometer (EDS, EDAX, Mahwah, NJ) was used to examine the 'B' specimens. The FE-SEM specimens were mounted to view the carbon/carbon portion tilted several degrees from an 'end on' view. Voltages of 6 KV were used and it was not necessary to coat the specimens.

These specimens were recovered from a violent accident and subsequent impact with the ground. Analysis proved very challenging. In some instances the sample appeared to be contaminated with soil. In other instances the exposed RCC appeared to have fractured on impact with the ground. These surfaces were not formed during the break-up of Columbia on re-entry and therefore are not included in this report. As will be discussed, the SEM study gave a signature which showed clear evidence of carbon oxidized at high temperatures, and the focus of the investigation was on specimens with this signature.

Raman Spectroscopy of Carbon Chars

The Raman technique has been described in detail elsewhere (Refs. 4 to 6). The technique involves measuring the wavelength and intensity of scattered light from a sample. The wavelength of the scattered light is shifted from the incident light due to molecular vibrations. Information on composition and molecular structure can be obtained from the resultant spectra. Raman spectra were obtained in this study using a microscope optically coupled to a triple spectrometer and charge-coupled-device detector (CCD). The microscope focuses the laser beam that excites the Raman scatter to an approximately one-micrometer spot (horizontal spatial resolution), collects Raman scattered light and transfers it to the spectrometer/CCD. The CCD records spectra over a certain bandpass. Spectra must be obtained over several ranges and spliced together to cover the entire Raman region (0 to 4000 cm^{-1}). Raman spectra are plotted as the intensity of the scattered light versus the difference in frequency between the scattered light and the incident laser, or "Raman Shift" units (cm^{-1}). Peaks in the Raman spectra correspond to vibrational frequencies of the molecules scattering the laser light. Raman spectroscopy is analogous to infrared absorption spectroscopy in that both reveal the vibrational structure of molecular species. Sampling areas were typically a few square millimeters. It was not possible to sample exclusively from the fibers or the matrix—the spectra represent a mix of both.

This technique is based on the relationship of Raman shift to the size of the graphitic domains (Ref. 4), which in turn relates to the exposure temperature. The most stable configuration of elemental carbon consists of graphene rings stacked in a graphitic planar structure. This structure is generally formed by pyrolysis of organic materials. At very high temperatures (>3000 °C) graphite forms large crystals (or domains) that can be millimeters in size. At low temperatures (a few hundred degrees Celsius) it forms with domains of nanoscale dimensions. The low temperature form of graphite (a charcoal-like material) is often referred to as glassy or amorphous carbon, but it is more accurately termed "nanocrystalline" graphite. There is an amorphous form of carbon, but it is formed by deposition of high-energy carbon particles, as are created by laser ablation of a carbon target. The development of larger graphitic domains with increasing temperature is generally not reversible without the disassembly of the graphite into atomic-scale particles. The temperature determined from Raman spectra of carbon materials represents the highest temperature experienced by the carbon sampled.

The Raman spectrum of graphite-like materials consists of two bands, one peaking near 1350 cm^{-1} and one peaking between 1580 and 1600 cm^{-1}. At low formation temperatures (hundreds of degrees Celsius) the two bands are broad and of similar intensity. The 1580 to 1600 cm^{-1} band is attributed to a 'breathing-like' vibrational mode of the graphene rings and is active in the bulk of the graphitic domain. The origins of the 1350 cm^{-1} band are still controversial, but there is agreement that is related to structural disorder (Ref. 4). Its intensity depends on the 'edge to bulk' ratio of the domain and hence it decreases with larger graphitic domains. As the exposure temperature and the domain size increases, the carbon

Raman bands become narrower, and the lower frequency band decreases in relative intensity. At exposure temperatures above 3000 °C, the 1350 cm^{-1} band may completely disappear.

A set of seventeen reference spectra were obtained from carbon materials heated in a furnace to temperatures from 375 to 2700 °C. These carbon materials were formed by pyrolysis of pieces of a carbon/phenolic composite used in re-entry vehicle heat shields (FM5055G, U.S. Polymeric Corporation). This material is a phenolic resin with low temperature carbon powder. The heat treatments cause graphitization, as occurs in RCC. Although this reference material is different than the actual RCC, it should be adequate for qualitative estimates of temperature. Selected reference spectra are shown in Figure 8. Note the lower frequency band decreases in intensity with temperature, as discussed.

In this analysis reference spectra were visually compared to the actual RCC spectra. This was preferred over a statistical least-squares type analysis (Ref. 7) for two reasons: (1) While the statistical analysis is relatively robust, the references are not RCC materials and there is the possibility that the spectra from the RCC material contain factors not adequately accounted for in the calibration based on the references; and (2) Many of the RCC spectra show a 1350 cm^{-1} band less intense than that in the spectrum from the reference exposed to 2700 °C—these spectra correlated to exposure temperatures higher than the range encompassed by the references. The statistical analysis (Ref. 7) of all the pyrolyzed references (375 to 2700 °C, using different furnaces and temperature measurement techniques) combined into one data set resulted in a standard error prediction of ±250 °C. However, restricting the analysis to references heated to 1500 °C and above reduces the prediction uncertainty to about ±100 °C. Visual analysis of the Raman spectra is expected to have an uncertainty of about ±100 °C for exposure temperatures between the 500 and 1000 °C and between the 2000 and 2700 °C, ranges in which changes to the graphite structure result in relatively obvious changes in the Raman structure.

These forms of carbon are very opaque to the visible laser light (514 nm) used by the Raman probe. Penetration depths of this light are probably a few tens of nanometers. The temperatures inferred from carbon Raman spectra correspond to those experienced by the thin layer of carbon at the surface of the sample, i.e., at the carbon/atmosphere interface.

Analysis of Fragments

As-Fabricated RCC

Figure 9 is an optical micrograph of the as-fabricated carbon/carbon cloth. This is made of rayon-derived carbon fibers by Avtex (Formerly of Front Royal, VA). Raman spectra for this cloth are given in Figures 10(a) and (b) with illumination along the edge of the fiber and at the end of the fiber, respectively. Note that when illumination is at the fiber ends (versus along the length of the fibers), the 1350 cm^{-1} band is enhanced in intensity because the ends of graphene planes are sampled, where symmetry results in a larger Raman cross-section for the vibrational mode resulting in the 1350 cm^{-1} band. Therefore, care is necessary in interpreting Raman spectra from highly oriented assemblages of carbon, as in high-temperature-processed fibers.

As discussed in the introduction, the actual carbon/carbon substrate consists of the carbon cloth impregnated with a phenolic resin. The graphite fibers are exposed to a proprietary heat treat process, but probably see temperatures of 1650 to 2000 °C. After lay-up the substrate is treated three times with a liquid carbon precursor to fill porosity. The matrix material is then graphitized. The highest temperature seen during fabrication from the cloth and oxidation protection application is 1760 °C, during the SiC conversion coating process. Figure 10(c) is the Raman spectra of the as-fabricated carbon/carbon which includes both the fibers and the matrix material. Note that this gives a substantial peak 1350 cm^{-1} band. A comparison to the standards gives a highest temperature of exposure for this material of 2300 to 2400 °C. This seems high since, as noted, the fibers would have been exposed to heat treats of 1650 to 2000 °C and the matrix to a maximum temperature of ~1760 °C. The disparity is most likely due to inadvertent sampling of oriented fiber material along with matrix material. Figure 12, for example, shows carbon Raman spectra corresponding to exposure temperatures in the 1600 to 1800 °C range, as expected.

In this analysis, it is particularly important to compare the Raman bands from the recovered RCC from Columbia to the Raman bands in Figure 10(c). Particularly important is the relative height of the 1350 cm^{-1} band. If the Raman bands from the recovered RCC show a substantially smaller 1350 cm^{-1} band than the as-fabricated material, it is very likely that the RCC was exposed to a higher temperature during the accident.

Recovered RCC From the Accident

Six specimens examined were taken from the recovered fragments of panels 8L and 9L, near the breach and are listed in Table I. These leading edges had ~20 to 30 missions on them, with normal re-entries prior to the accident. The highest processing temperatures and the highest re-entry temperature that these fragments would have seen would be ~1650 °C.

As noted, these specimens showed not only the RCC constituents but oxides and metals (slag) deposit from the internal wing components, however the focus here is only on the exposed carbon/carbon portions. Some of these showed fractured surfaces with no evidence of high temperature exposure; whereas others showed microscopic features consistent with oxidation and erosion. These also showed evidence for very high temperatures in the Raman spectra. Macro-photos of each specimen will be shown. As noted, two adjacent specimens were taken from each location.

TABLE I.—SAMPLES ANALYZED
['A' samples were examined with Raman spectroscopy and
'B' samples were examined with electron microscopy]

Sample designation	Location	Comment
38223A, 38223B	Panel 9	Pointed fibers
1860A, 1860B	Panel 9	Pointed fibers
52018A, 52018B	Panel 9	Slag deposits, pointed fibers
58291A, 58291B	Panel 8	Possible flowing carbon and erosion
49619A, 49619B	Panel 8	Slag deposits, pointed fibers
2200A, 2200B	Panel 8	Extensive slag deposits

Specimen 38223B (Panel 9) had a fracture face with exposed carbon/carbon, as shown in Figure 11(a). Figure 11(b) shows a high magnification micrograph of a longitudinal fiber bundle. Note the pointed ends of the fibers, which is a morphology characteristic of high temperature oxidation (Refs. 8 and 9). This indicates that the fibers were exposed to hot oxidizing gases during the break-up of Columbia and that the fracture probably did not occur on impact to the ground. However, we cannot ascribe a temperature from this. Oxidation of carbon begins at about 500 °C and leads to this pointed morphology over a range of temperatures (Refs. 8 and 9).

The Raman spectra for this sample 38223A (Panel 9) are shown in Figure 12. Locations 1 and 2 are from the fracture surface, similar to that examined with the SEM. Locations 3 to 8 are from the SiC surface and the carbon detected may be a contaminant from the exposed, pyrolyzed phenolic or soot deposited during Columbia's break-up. The temperatures listed are estimated from comparison to the reference materials.

Sample 1860B (Panel 9) results are shown in Figures 13 and 14. The micrograph shows the pointed fibers, again indicating exposure to high temperature oxidizing gases. The Raman results are in Figure 14. Locations 1 and 2 are from the fracture surface. At location 1 the carbon Raman bands are low-temperature (400 to 1000 °C) and charcoal-like, which probably indicates deposition of pyrolysis products from after the break-up or contact with organic materials in the soil in which the part landed. However, the presence of Raman bands due to elemental silicon in the spectrum from location 1 indicates that, at some point, a much higher temperature was experienced. If the elemental silicon came from the

SiC layer of the RCC composite, the temperature at this location must have exceeded the point at which an appreciable Si(g) vapor pressure is generated (~2000 °C). Locations 3 to 7 indicate intermediate (1000 to 2000 °C) temperature carbon and silicon carbide, apparently a remnant of the SiC layer in the RCC composite. However, the Raman spectrum from location 8 indicates some very high temperature carbon, which may have deposited during the accident.

Sample 52018 (Panel 9) is shown in Figures 15 to 16. The micrographs show a bundle of pointed (oxidized) fibers along with some slag deposits. As shown in the lower macrograph in Figure 16, one of the SiC sides had spalled off, exposing a large region of carbon/carbon. The Raman spectrum from location 1 exhibits bands due to elemental silicon and carbon with an exposure temperature consistent with that required to decompose SiC (~2300 °C). Locations 4 to 6 were on the carbon/carbon and all showed high temperature carbon.

Sample 58291 (Panel 8) (Figure 17) showed some unusual microstructural features in the carbon/carbon. A large region of matrix is shown at the end of some fiber bundles in Figure 17(a). An EDS analysis of this region showed it to be all carbon. This may be a deposit of matrix material at the end of a fiber bundle; however this is an unusual morphology. Alternatively, this morphology may have resulted from the accident. It appears that the matrix has flowed, but it is unlikely that the melting point of carbon was reached (~3500 °C). The image in Figure 17(b) is a fracture surface with pulled out fibers and remaining matrix. There are no clear pointed fibers here but the microstructure is similar to that of eroded carbon/carbon (Ref. 10). In the Raman spectra of Figure 18 the bands indicate high temperatures were reached in locations 1, 5, and 6 in the exposed carbon/carbon. Note that the spectrum for location 6 shows high temperature carbon that is off the scale of the reference carbons. The lower temperature carbon in locations 2, 3 and 4 may be due to late-term deposition of pyrolysis products or soil contaminants.

The remaining two panel 8 samples showed high temperature carbon. Sample 49619B (Panel 8) had slag deposits and fibers with flat fracture surfaces. However, at the knife edge of sample 49619B (Panel 8) regions of fibers were found exposed between surrounding slag, as shown in Figure 19. The pointed morphology observed is characteristic of exposure to high-temperature oxidizing gases.

The Raman spectra for sample 49619A are given in Figure 20. Note the presence of high temperature carbon on several points of this sample. Location 3 in particular showed a carbon spectrum which was off the scale of the reference materials and estimated to have been exposed to temperatures much greater than 2700 °C. Note that the 1350 cm^{-1} band in this case is barely observable. This location was studied in more detail to determine whether these extremely high temperatures are the result of exposure that occurred during the accident or whether they reflect processing temperatures reached during manufacturing of the RCC.

Figure 21(a) shows Raman spectra from the survey of RCC sample 49619A (Panel 8), near location 3 (designated 3-1), before and after scraping. An estimated 0.5 mm was removed by scraping to search for a possible thermal gradients. Prior experience indicates that substantial thermal gradients can be found in carbon/carbon exposed to re-entry environments (Ref. 5). The Raman spectra obtained before scraping are relatively consistent in band pattern and correlate to temperatures much greater than 2700 °C from the references. The Raman spectra obtained after scraping show a greater variation in band pattern. The average spectrum from after scraping has a more intense lower frequency band (Figure 21(b)), correlating to a lower average temperature. The before/after Raman spectra are consistent with a top layer of carbon uniformly exposed to very high external temperatures over layers more diverse in carbon structures (i.e., fiber and lower temperature matrix).

Figures 22(a) (before scraping) and 22(b) (after scraping) show ten Raman spectra each from RCC sample 49619A (Panel 8), also near location 3 (designated 3-2). The spectra obtained before scraping show some variability in band pattern but all correlate to temperatures greater than 2700 °C. The spectra obtained after scraping have a consistently different carbon Raman band pattern, with a more intense lower frequency band. The spectra obtained after scraping correlate to exposure temperatures near 2400 °C, lower and closer to that seen in the as-fabricated material (Figure 10(c)). The difference between the carbon Raman band patterns before and after scraping can be seen more readily from their average spectra, plotted in Figure 22(c). These scraping experiments indicate that the carbon at location 3-2 on

RCC sample 49619A (Panel 8) experienced a thermal gradient consistent with exposure to extremely high temperatures at the carbon/atmosphere interface, consistent with re-entry heating. It appears that the extremely high temperature exposures, inferred from the carbon Raman spectra at locations 3-1 and 3-2 on RCC sample 46919A, and probably at other locations as well, occurred during Columbia's break-up rather than during manufacturing of the RCC panels.

Figure 23 is a view of sample 2200B. As with the other samples, this sample had large amounts of slag deposits. On this microscopy sample, 2200B, no pointed fibers were observed. However the Raman sample, 2200A (Fig. 2), two of the locations (3 and 4) had spectra indicating high temperature carbon. As noted, the microscopy and Raman samples were adjacent, but may not be identical. The spectra from RCC sample 2200A also include bands due to remnants of the SiC layer in the RCC.

Summary and Conclusions

This is a brief follow-on study to the Columbia accident investigation. Recovered fragments of RCC near the breach are examined with scanning electron microscopy and Raman spectroscopy. The fragments are from a tragic and violent vehicle break-up at an altitude of 122,000 m. The microstructures are complex, but clear evidence of high temperature oxidation identifies these fragments as having been exposed to high temperature oxidizing gases during Columbia's break-up.

Interpretation of the Raman bands is done via comparison to both as-fabricated RCC and carbon reference materials. Temperatures were estimated by visual comparison to the references and represent the highest temperature to which the specimen had been exposed. These estimates have a significant uncertainty, given the differences between the RCC and the references. Nonetheless, some locations exhibited bands indicating very high temperatures, higher than the processing temperatures or normal re-entry temperatures. This is strong evidence that the location had been exposed to those temperatures during the accident. In these very high temperature regions the Raman band at 1350 cm^{-1} virtually disappeared. Furthermore, some regions indicated an outer layer which had been exposed to these high temperatures and removal of that outer layer revealed a layer exposed to lower temperatures.

As noted, quantifying the highest temperatures was difficult. Certainly these temperatures were well above the highest RCC processing temperature of 2000 °C. The very small 1350 cm^{-1} peak was only seen on the reference materials exposed to the very highest temperatures (2700 °C). On basis of these observations, it is likely that during the break-up of Columbia some regions were exposed to material temperatures greater than 2700 °C. This is higher than previous estimates, but consistent with the extremely high temperatures of the plasma (~5000 °C) which entered the wing.

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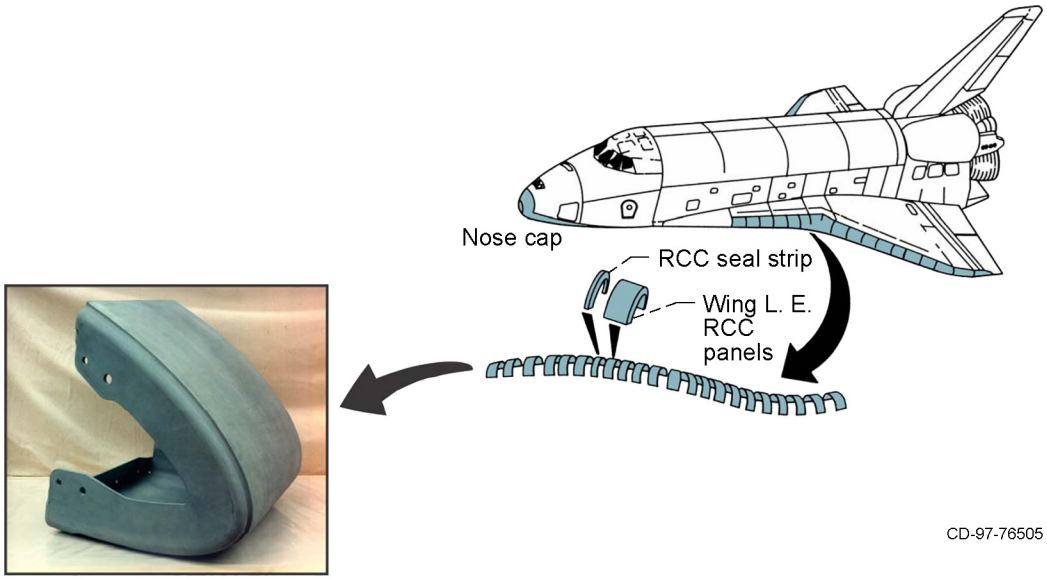


Figure 1.—Location of RCC panels on the wing leading edge of the Shuttle Orbiter.

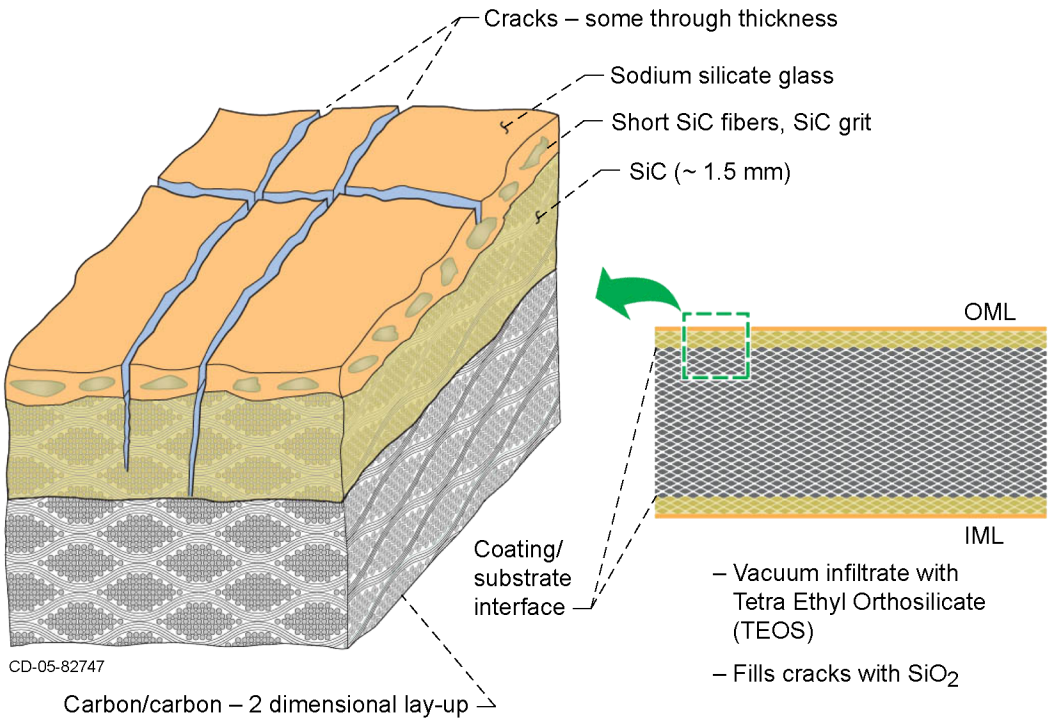


Figure 2.—Schematic of RCC structure showing carbon/carbon and oxidation protection system.

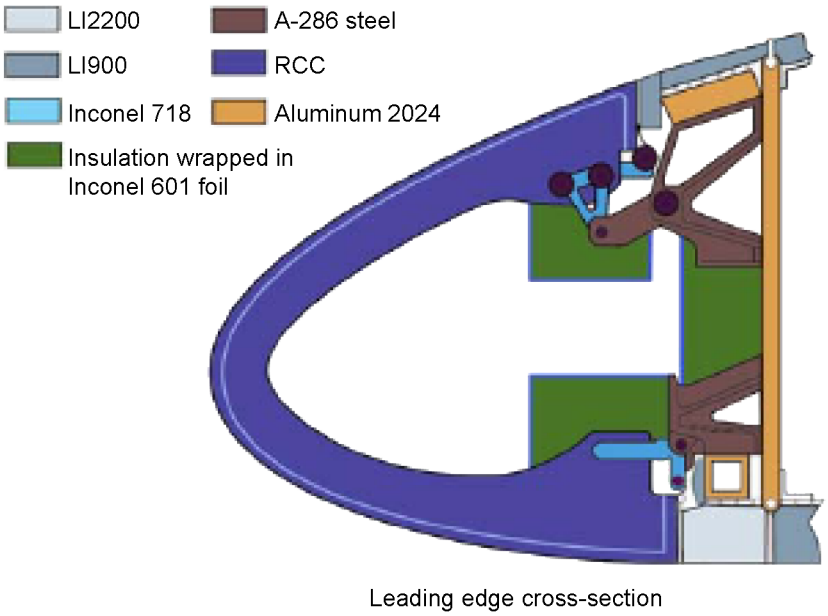


Figure 3.—Schematic showing RCC wing leading edge panel and attachment hardware to wing bulkhead.

Figure 4.—Recovered portion of panel 8L, showing knife edge.

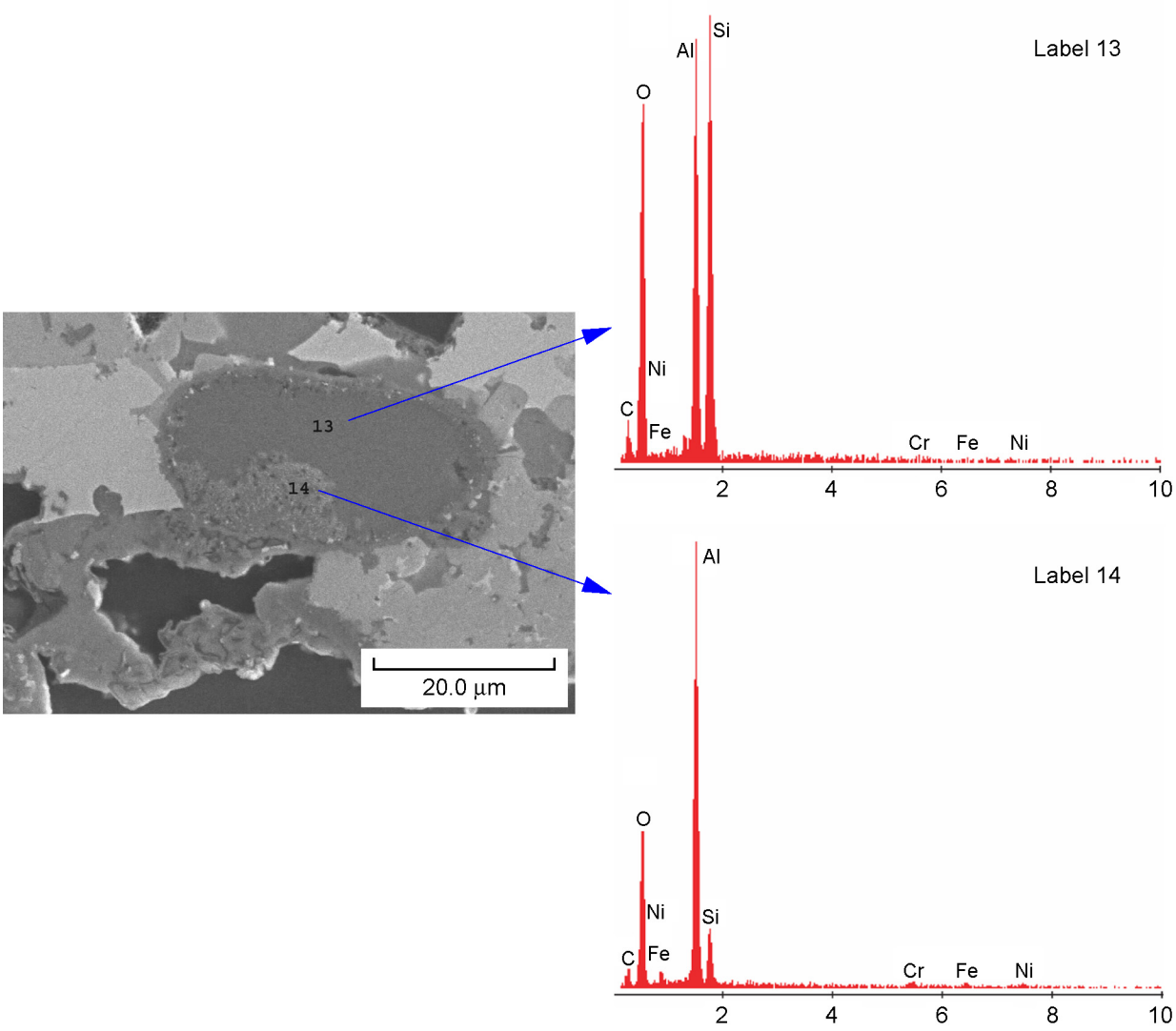


Figure 5.—Micrograph and EDS of probable melted and solidified Cerachrome® insulation.

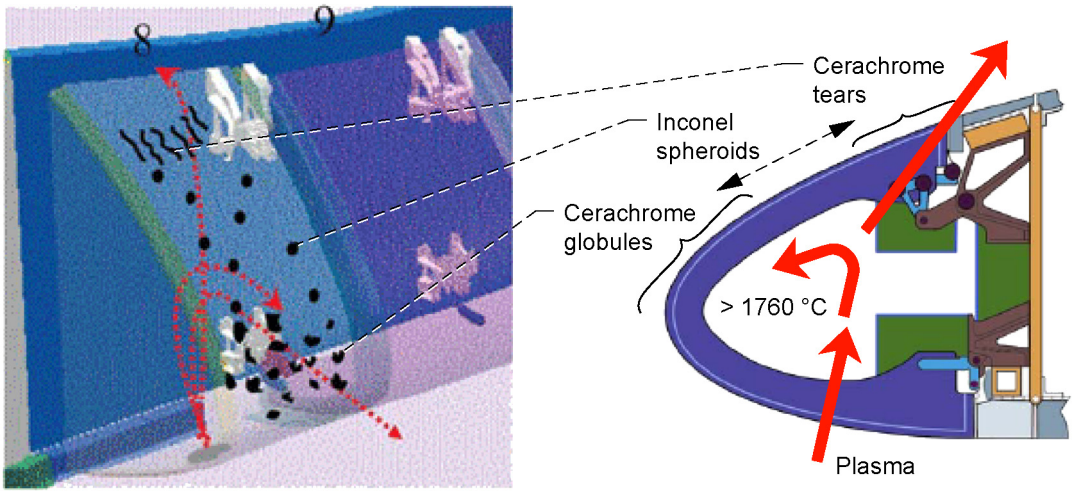


Figure 6.—Schematic showing breach and entry of hot plasma gases on the underside of panel 8L.

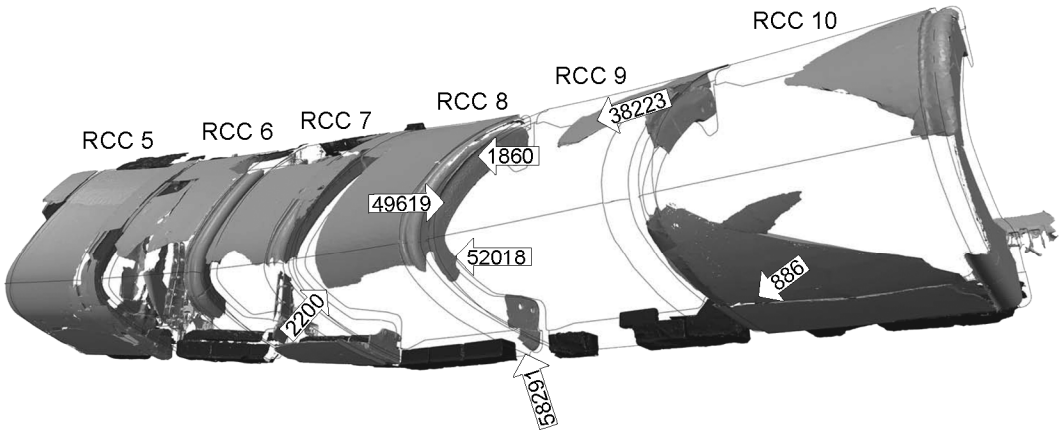


Figure 7.—Schematic showing recovered portions of port wing leading edge panels from Columbia. Samples taken for this study are marked.

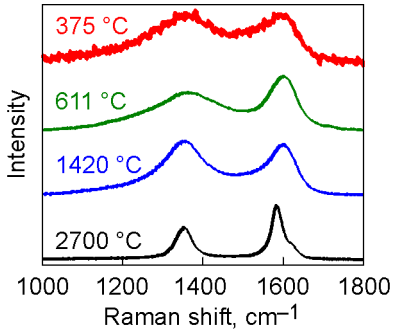


Figure 8.—Reference Raman spectra from 375-2700 °C.

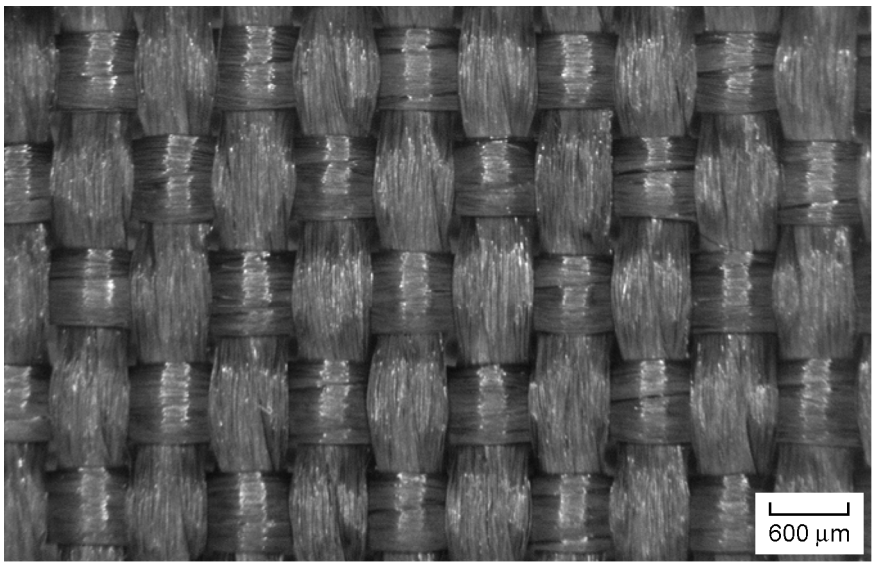


Figure 9.—Photograph of the as-fabricated RCC (Avtex) cloth used in RCC.

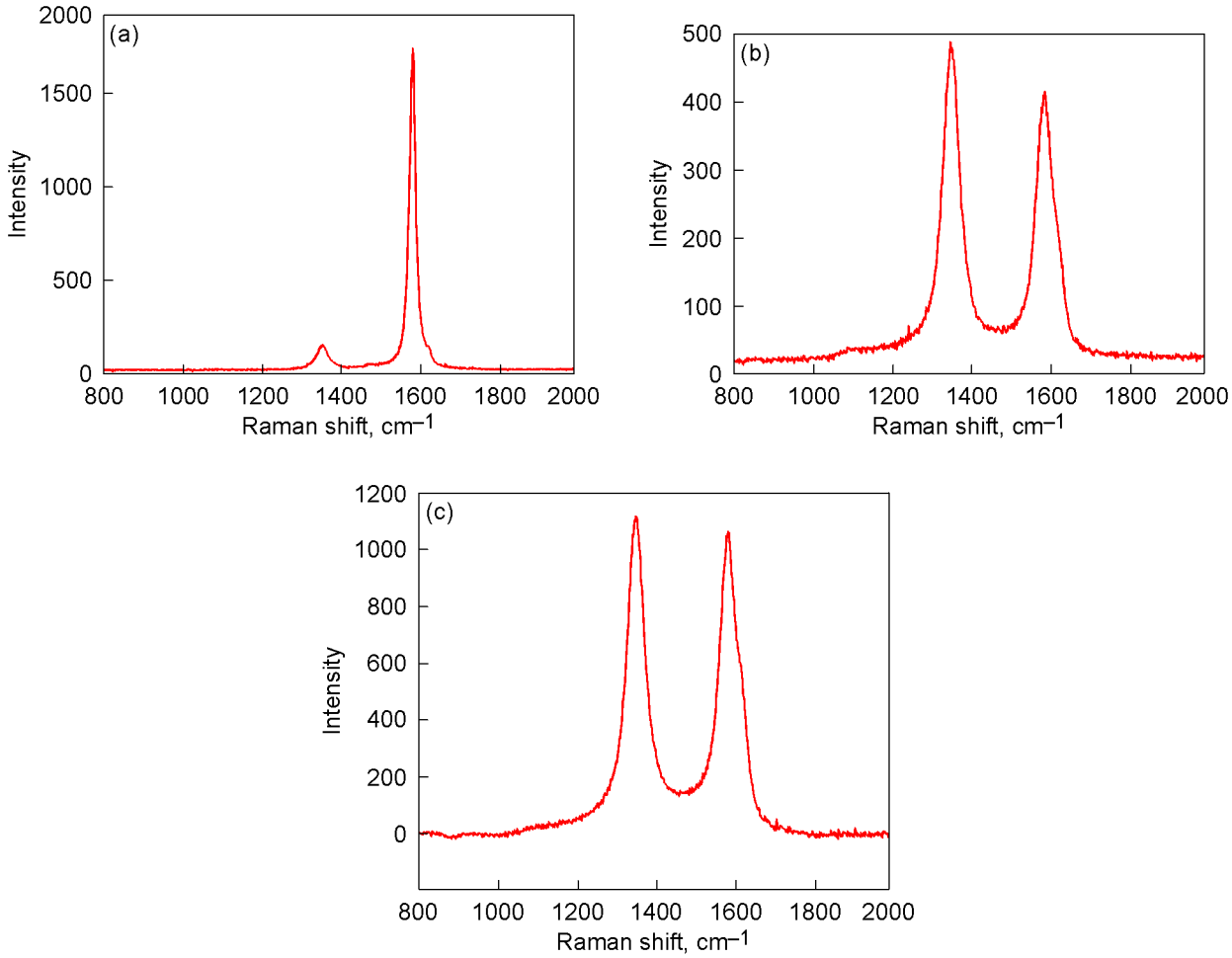


Figure 10.—Raman spectra of the as-fabricated materials. (a) Cloth illuminated along the edge of the fiber. (b) Cloth illuminated at fiber end. (c) As-fabricated RCC core (includes cloth lay-up, graphitized phenolic resin, and graphitized liquid carbon precursor).

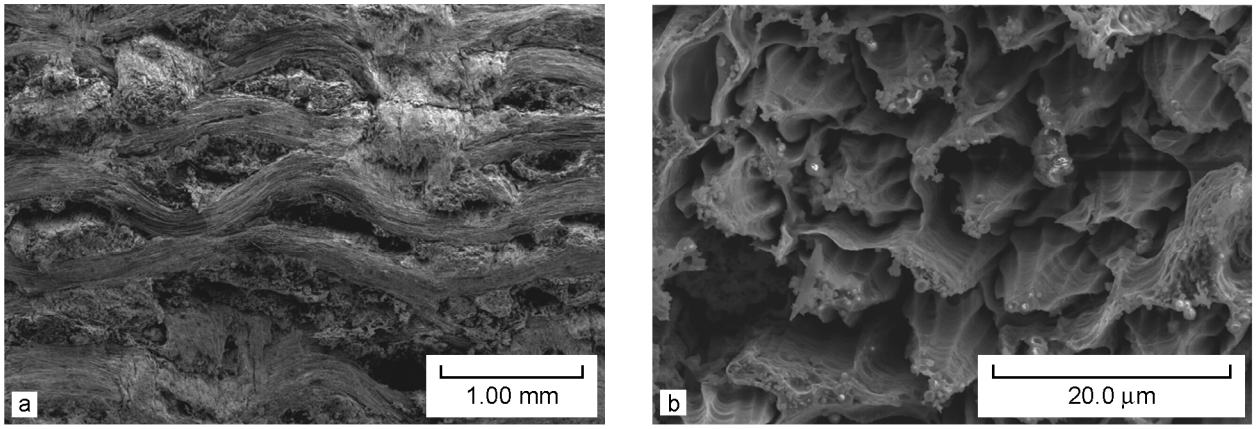
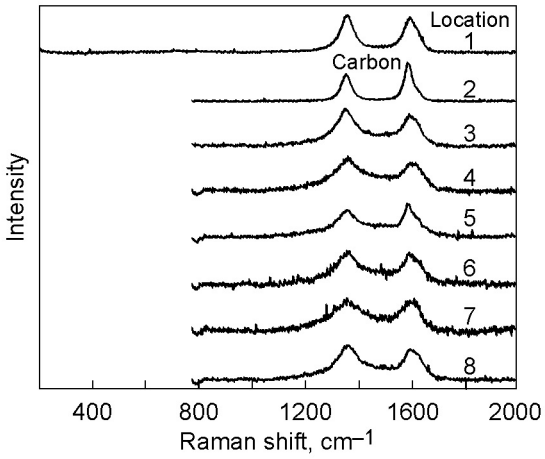


Figure 11.—Electron micrograph of sample 38223B. (a) Low magnification view showing transverse and longitudinal fibers and slag (bright areas). (b) High magnification view, showing pointed fibers.



Location	Location Description	Summary of Identified Bands
1	Slope, smooth side	2300 °C carbon
2	Slope, smooth side	2600 °C carbon
3	Flat, lower, smooth side	1700 °C carbon
4	Flat, upper, smooth side	1300 °C carbon
5	Flat, lower, smooth side	1800 °C carbon plus low temperature carbon
6	Flat, lower, rough side	1400 °C carbon
7	Flat, upper, rough side	1200 °C carbon
8	Flat, lower, rough side	1500 °C carbon

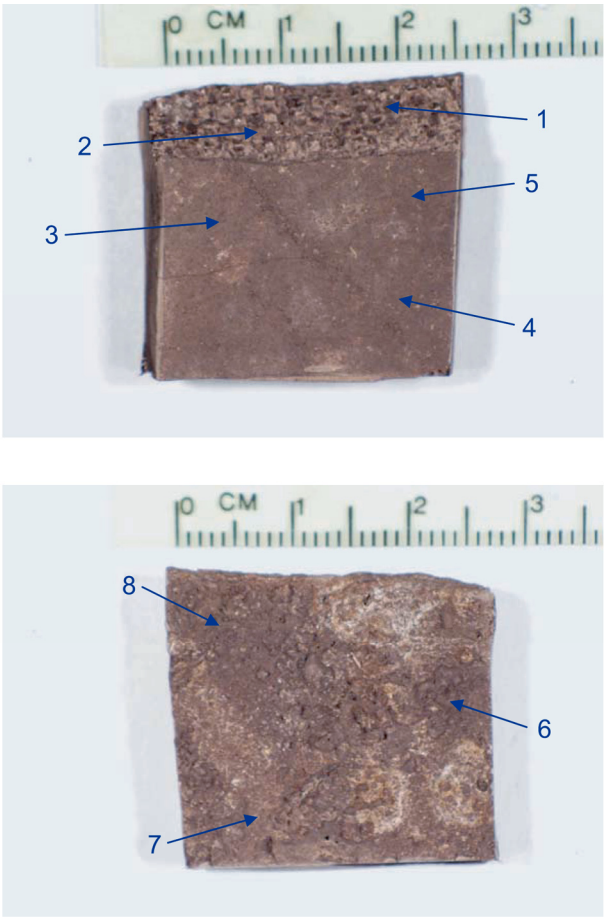


Figure 12.—Raman spectra and analyses of sample 38223A.

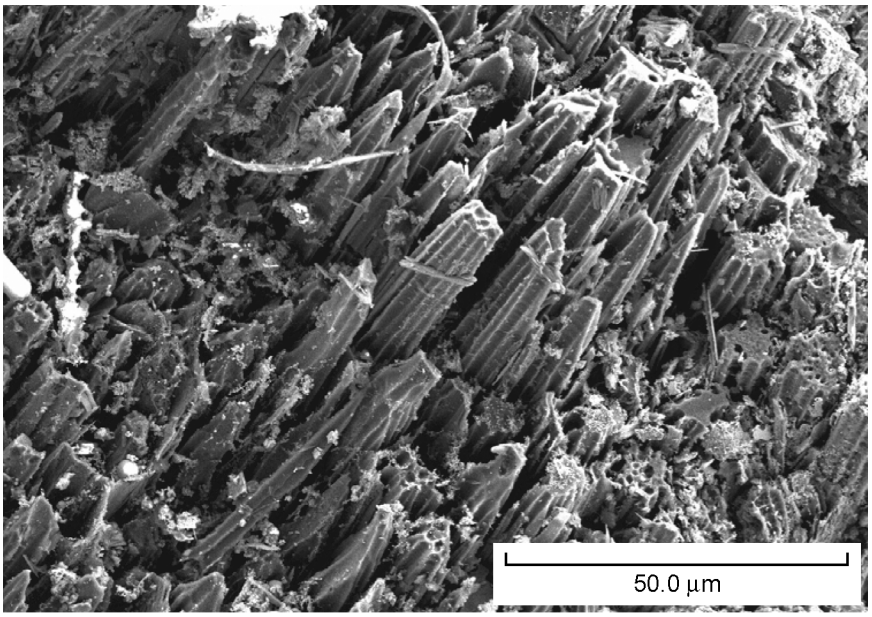
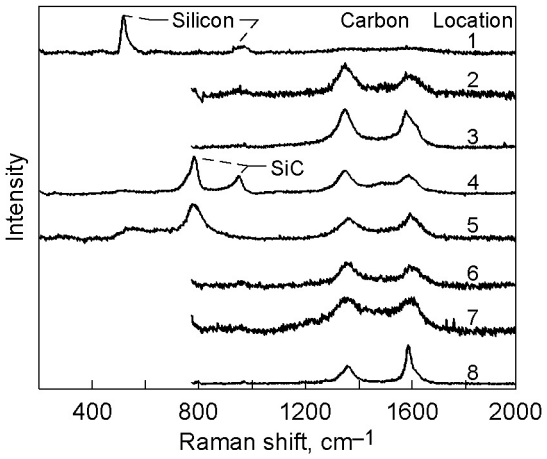
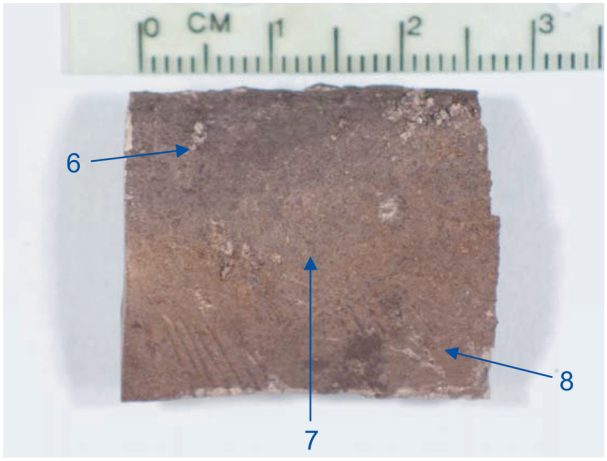
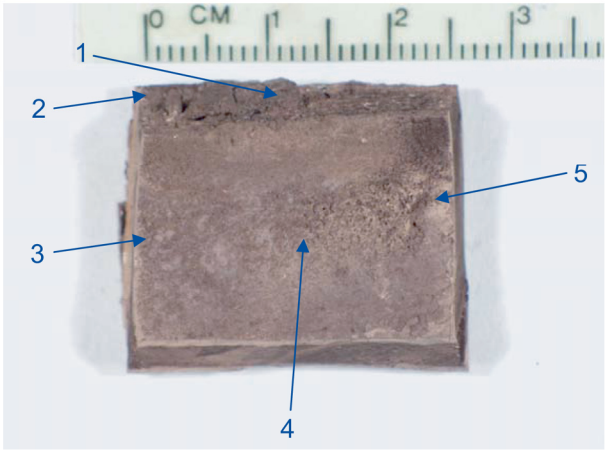


Figure 13.—Electron micrograph of exposed fibers from sample 1860B.

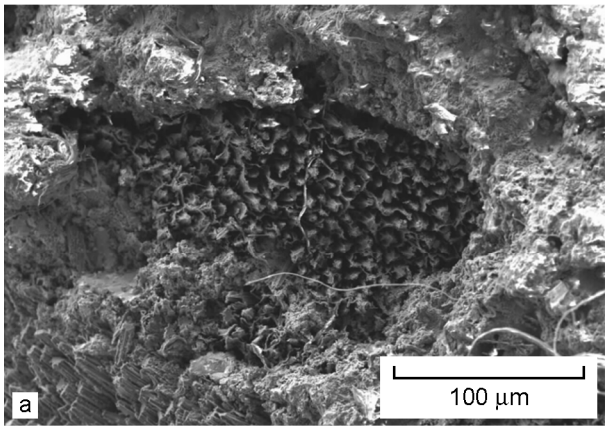






Location	Location Description	Summary of Identified Bands
1	Sloping edge, concave side	Microcrystalline silicon, 400 °C carbon
2	Sloping edge, concave side	1800–2200 °C carbon*
3	Edge, concave side	1800 °C carbon
4	Center, concave side	Silicon carbide,1800–2200 °C carbon, amorphous carbon
5	Near side, concave side	Silicon carbide, 1600 °C ? carbon
6	Lower, convex side	1600 °C carbon
7	Center, convex side	1000 °C carbon
8	Upper, convex side	Silicon carbide (minor), > 2700 °C carbon
*Relative intensities not a good match to references, peaks shifted		

Figure 14.—Raman spectra and analyses of sample 1860A.



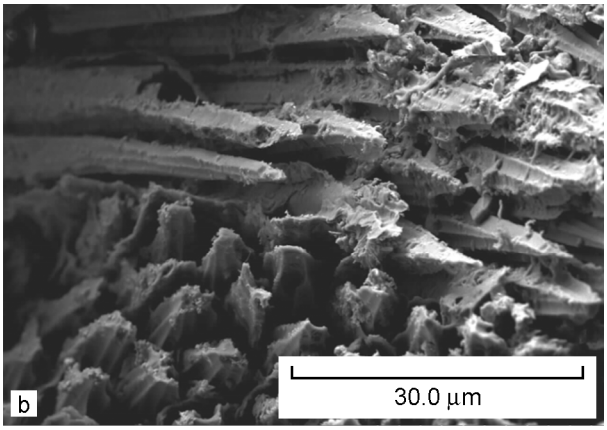
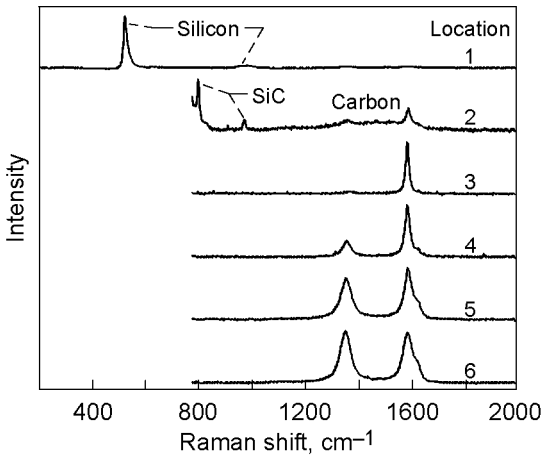
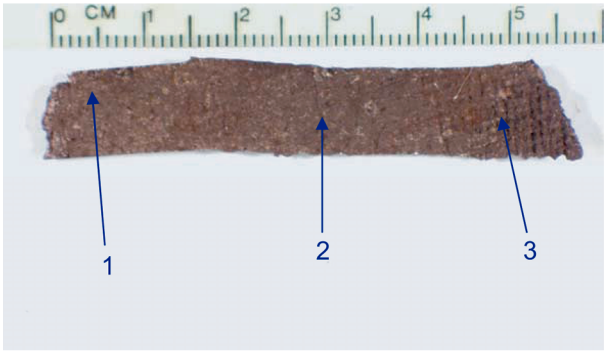
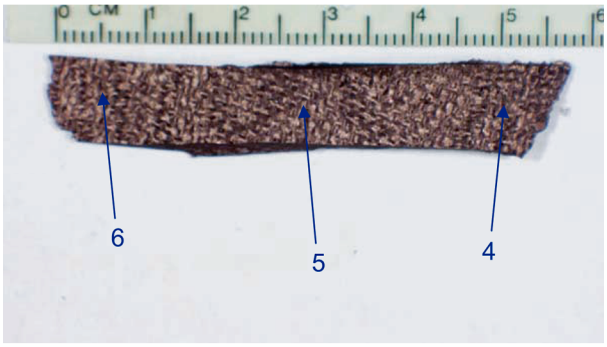


Figure 15.—Electron micrographs of sample 52018B. (a) Bundle of fibers surrounded by slag (brighter area). (b) Higher magnification view of pointed fibers.

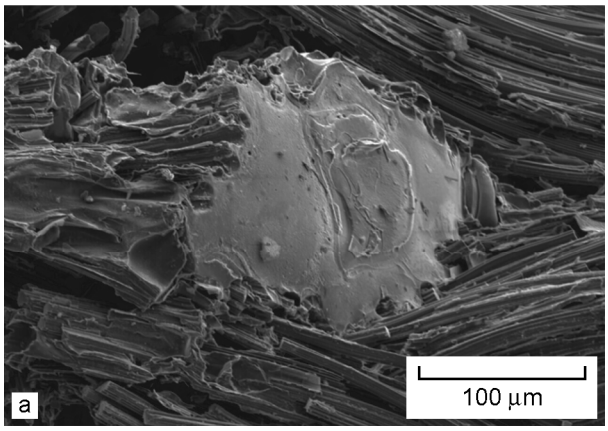






Location	Location Description	Summary of Identified Bands
1	Smooth side	Silicon plus low-intensity, 2300 °C ? carbon and low temperature carbon
2	Center, smooth side	Silicon carbide plus > 2700 °C carbon and low temperature carbon
3	Smooth side	>> 2700 °C carbon (off scale of reference carbons)
4	Rough side	> 2700 °C carbon (off scale of reference carbons)
5	Center, rough side	2500 °C carbon
6	Rough side	2300–2400 °C carbon

Figure 16.—Raman spectra and analyses of sample 52018A.



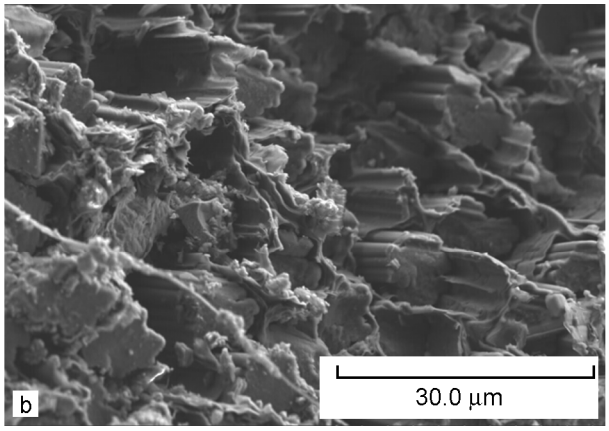
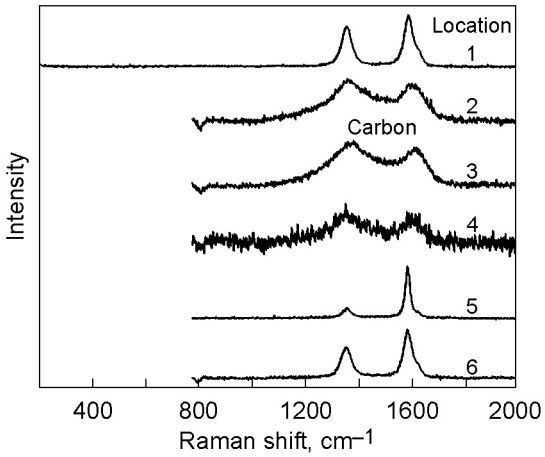


Figure 17.—Electron micrograph of unusual features in sample 58291B. (a) Large area of matrix which had possibly flowed. (b) Longitudinal fibers, showing fractured and possibly eroded surface.



Location	Location Description	Summary of Identified Bands
1	Knife edge (arrow), smooth side	2500 °C carbon
2	Knife edge, smooth side	700–1000 °C carbon
3	Erosion hole, center, smooth side	700–1000 °C carbon
4	Erosion hole, center, smooth side	700–1000 °C carbon
5	Knife edge, rough side	> 2700 °C carbon (off scale of reference carbons)
6	Knife edge, rough side	2700 °C carbon

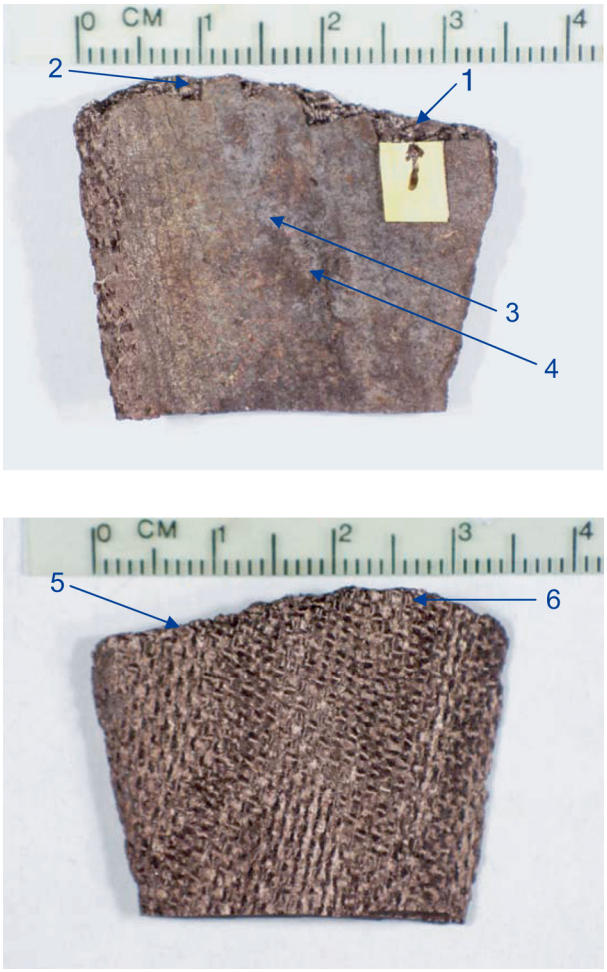


Figure 18.—Raman spectra and analyses of sample 58291A.

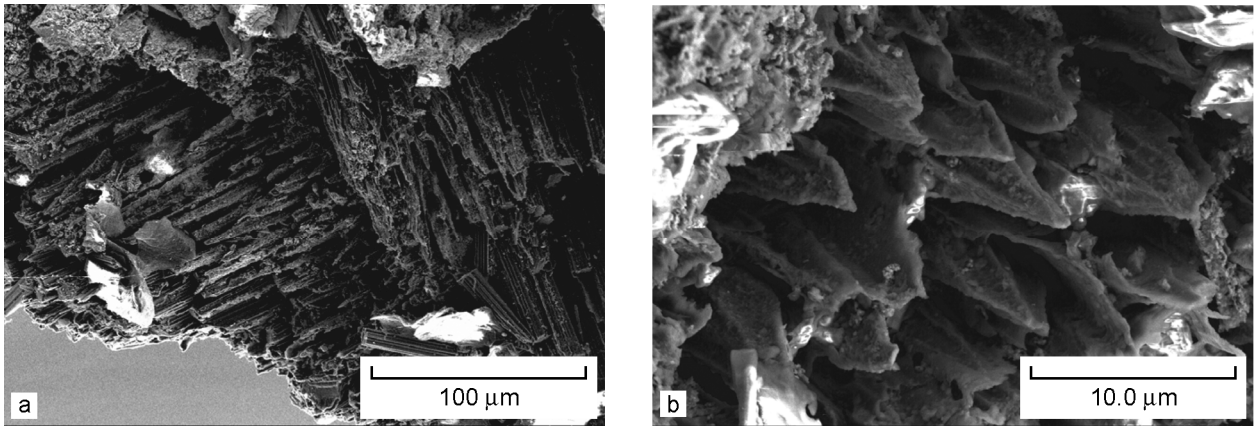
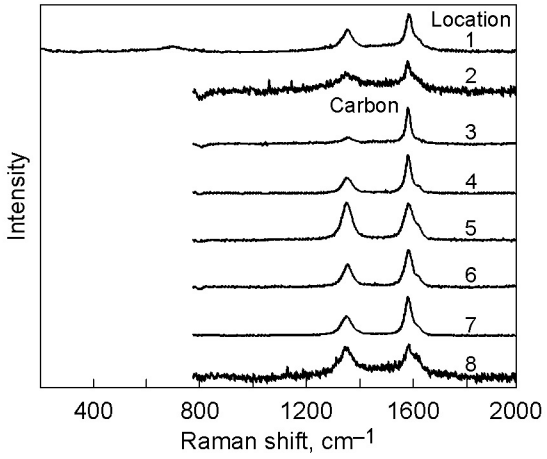
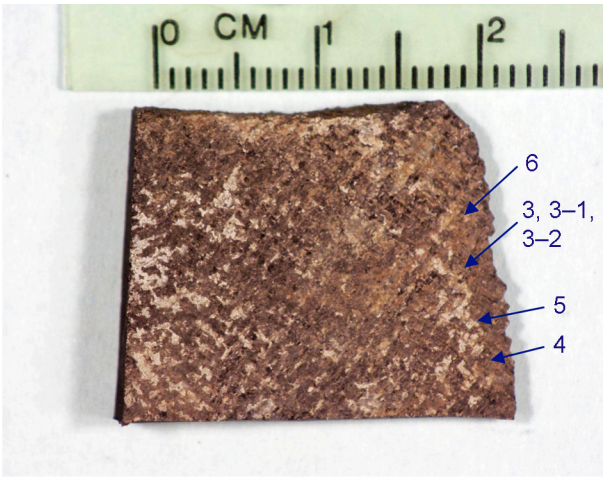


Figure 19.—Electron micrographs of sample 49619B.





Location	Location Description	Summary of Identified Bands
1	Knife edge	2700 °C carbon plus small amount of low temperature carbon
2	Knife edge	2700 °C carbon plus small amount of low temperature 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

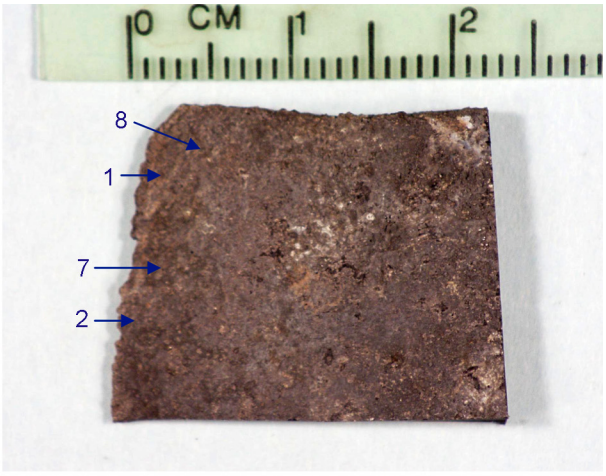
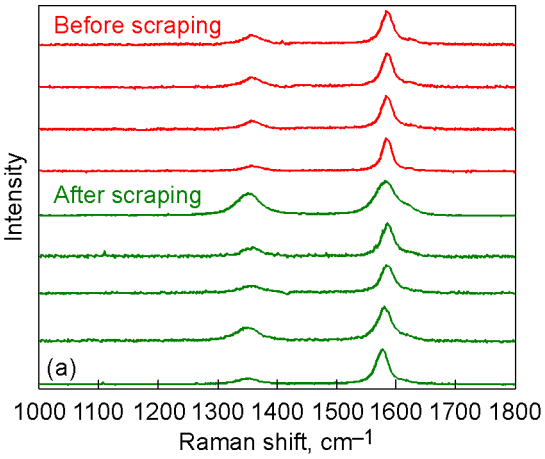


Figure 20.—Raman spectra and analyses of sample 49619A.



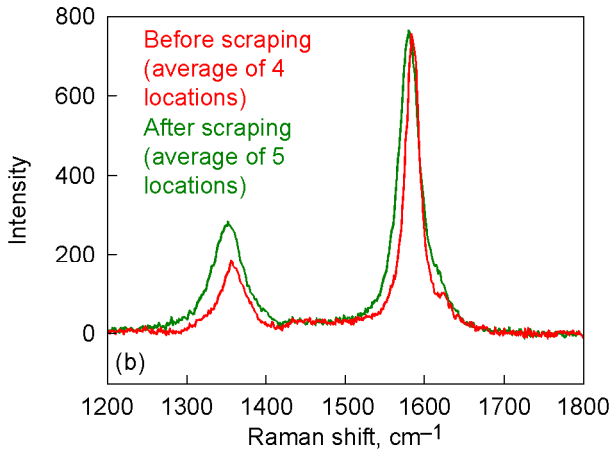


Figure 21.—Raman spectra from sample 49619A, location 3-1. (a) Spectra from multiple locations before and after scraping. (b) Average Raman spectra before and after scraping.

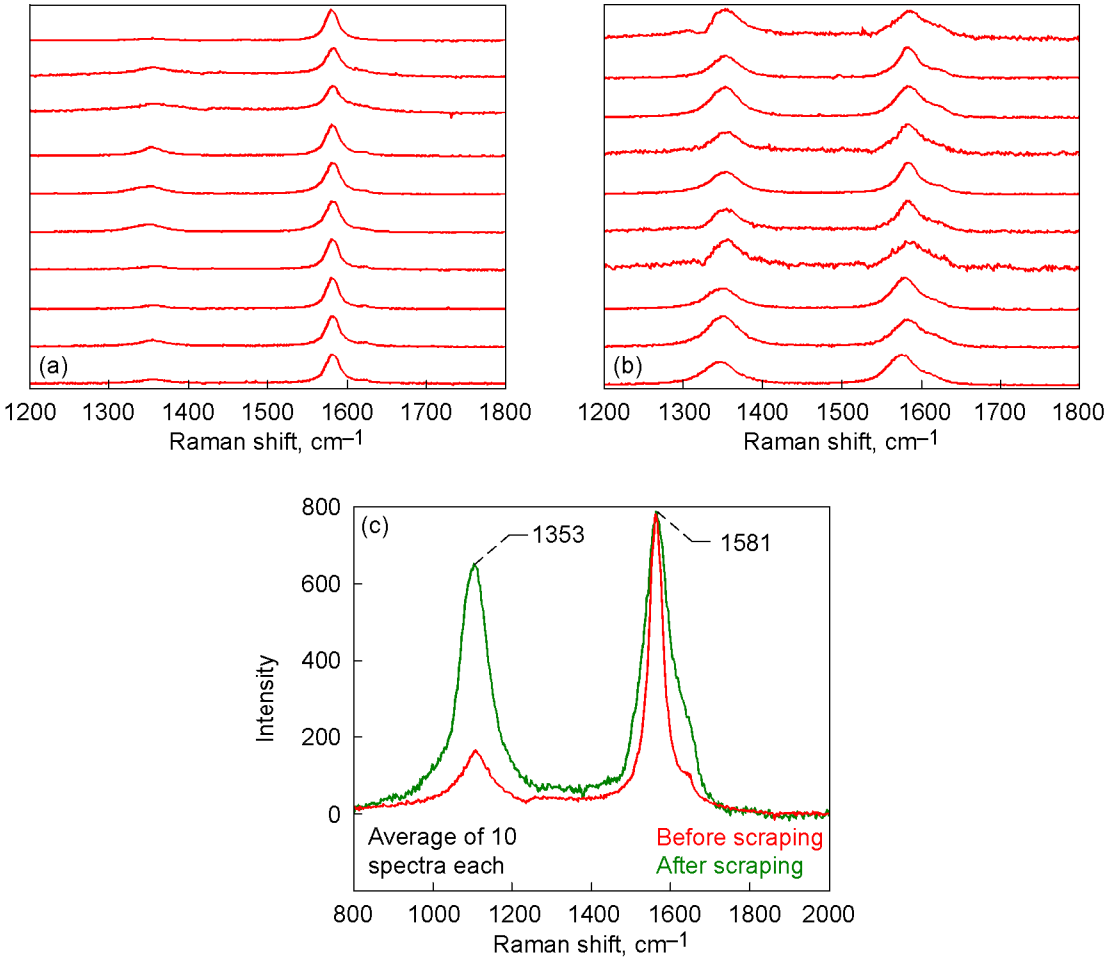


Figure 22.—Raman spectra from sample 49619A, location 3-2. (a) Spectra from multiple locations before scraping. (b) Spectra from multiple locations after scraping. (c) Average spectra before and after scraping.

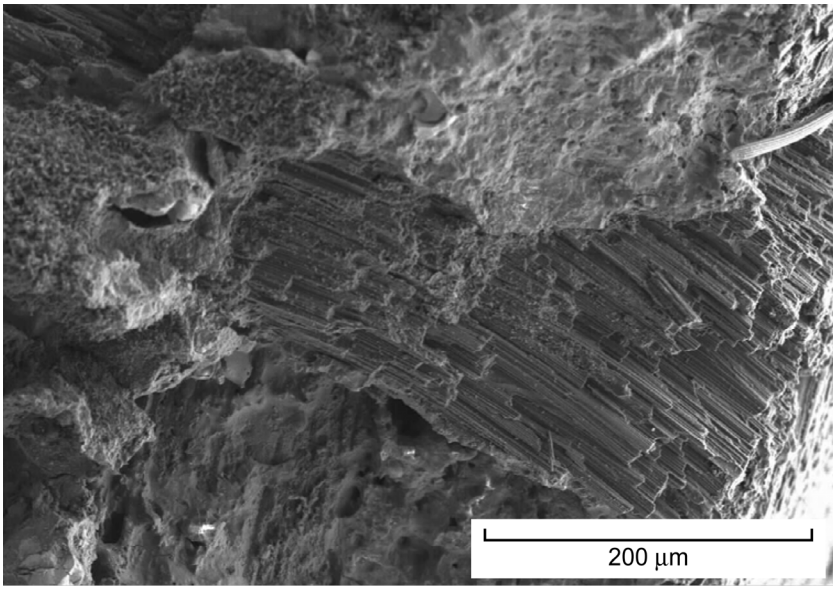
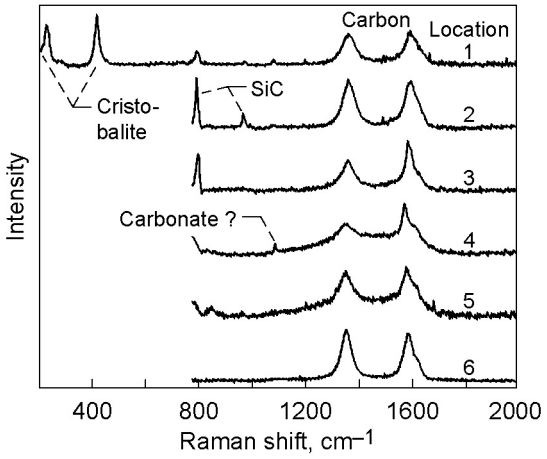


Figure 23.—Electron micrographs of sample 2200B. The portion near the top (brighter region) was covered with slag.



Location	Location Description	Summary of Identified Bands
1	Knife edge, smooth side	SiO_2 (cristobalite), silicon carbide, 2400 °C carbon
2	Knife edge, smooth side	Silicon carbide, 2400 °C carbon
3	Knife edge, rough side	Silicon carbide, 2700 °C carbon
4	Knife edge, rough side	Carbonates ?, 2700 °C carbon plus low temperature carbon ?
5	Crack, near edge, rough side	2100–2300 °C carbon plus low temperature carbon
6	Crack, near center, rough side	2300 °C carbon

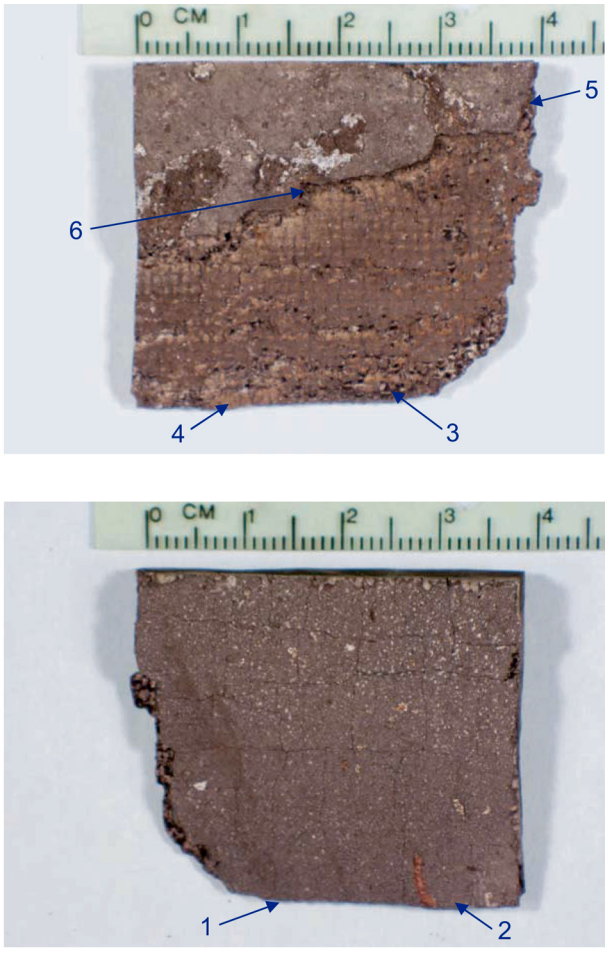


Figure 24.—Raman spectra and analyses of sample 2200A.

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13. ABSTRACT ***(Maximum 200 words)***

The extensive investigation following the Space Shuttle Orbiter Columbia accident of February 1, 2003 determined that hot gases entered the wing through a breach in the protective reinforced carbon/carbon (RCC) leading edge. In the current study, the exposed edges of the recovered RCC from the vicinity of the breach are examined with scanning electron microscopy and Raman spectroscopy. Electron microscopy of the exposed edges revealed regions of pointed carbon fibers, characteristic of exposure to high temperature oxidizing gases. The Raman technique relates the observed 1350 and 1580 to 1600 cm^{-1} bands to graphitic domains and their corresponding temperatures of formation. Some of the regions showed evidence of exposure temperatures beyond 2700 °C during the accident.

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