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# DEVELOPMENT OF NONDESTRUCTIVE EVALUATION METHODS FOR STRUCTURAL CERAMICS*

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# DEVELOPMENT OF NONDESTRUCTIVE EVALUATION METHODS FOR STRUCTURAL CERAMICS

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## ABSTRACT

Nondestructive evaluation (NDE) methods using three-dimensional microfocus X-ray computed tomographic imaging (3DXCT) were employed to map axial and radial density variations in hot-gas filters and heat exchanger tubes. 3D XCT analysis was conducted on (a) two 38-mm-OD, 6.5-mm wall, SiC/SiC heat exchanger tubes infiltrated by CVI; (b) eight 10 cm diam. oxide/oxide heat exchanger tubes; and (c) one 26-cm-long Nextel fiber/SiC matrix hot-gas filter. The results show that radial and axial density uniformity as well as porosity, can be assessed by 3D XCT. NDE methods are also under development to assess thermal barrier coatings which are under development as methods to protect gas-turbine first-stage hot section metallic substrates. Further, because both shop and field joining of CFCC materials will be necessary, work is now beginning on development of NDE methods for joining.

## INTRODUCTION

Nondestructive evaluation (NDE) technology is being developed to advance the reliable application of ceramic materials to fossil energy systems for improved efficiency and better environmental control. Advanced materials systems under development for fossil energy applications include continuous fiber ceramic matrix composites for hot-gas filters and heat-exchangers and thermal barrier coatings for high gas-firing temperature turbines.

# DISCUSSION OF CURRENT ACTIVITIES

## Hot Gas Filters and Heat Exchangers

### (a) Hot Gas Filters

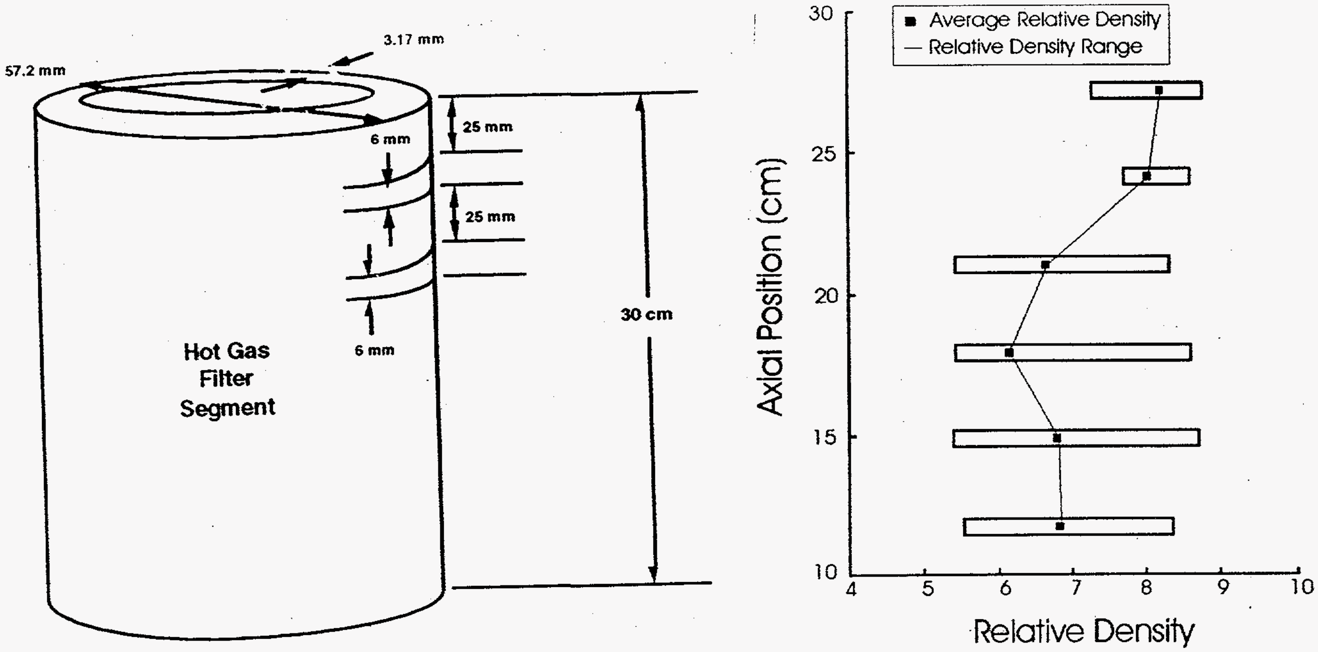
For hot-gas filter and heat exchanger studies, high spatial resolution 3DXCT methods have been explored.(1-3)

One 38 mm O.D. nextel fiber/SiC matrix hot-gas filter supplied by 3M, was examined at 25 mm intervals along the 26 cm length with data acquired using the ANL 3DCT scanner(2). The CT images were reconstructed in a 687 x 687 matrix, using 1053 projections with a pixel size of 85 µm x 85 µm, and six rows were averaged or a slice thickness of .51 mm. At each 25 mm section 14 slices were reconstructed. Image reconstructions were done using a newly installed 133 MHz dual pentium computer which has allowed faster reconstruction times. The 32-bit reconstruction files are rescaled to 8-bit scaled data for input to a 3D image display software, IDL, from Research Systems, Inc. This runs on Windows 95 in a separate 150 MHz pentium. However, a software package was written which speeds the input to the IDL software so that density related information can be determined.

Using these methods, hot gas filters have been analyzed for axial density variations as shown in Fig. 1. These data were obtained by using an in-house written software package which allows the average grayscale for each CT slice to be computed. A typical individual CT cross-section is shown in Fig. 2.

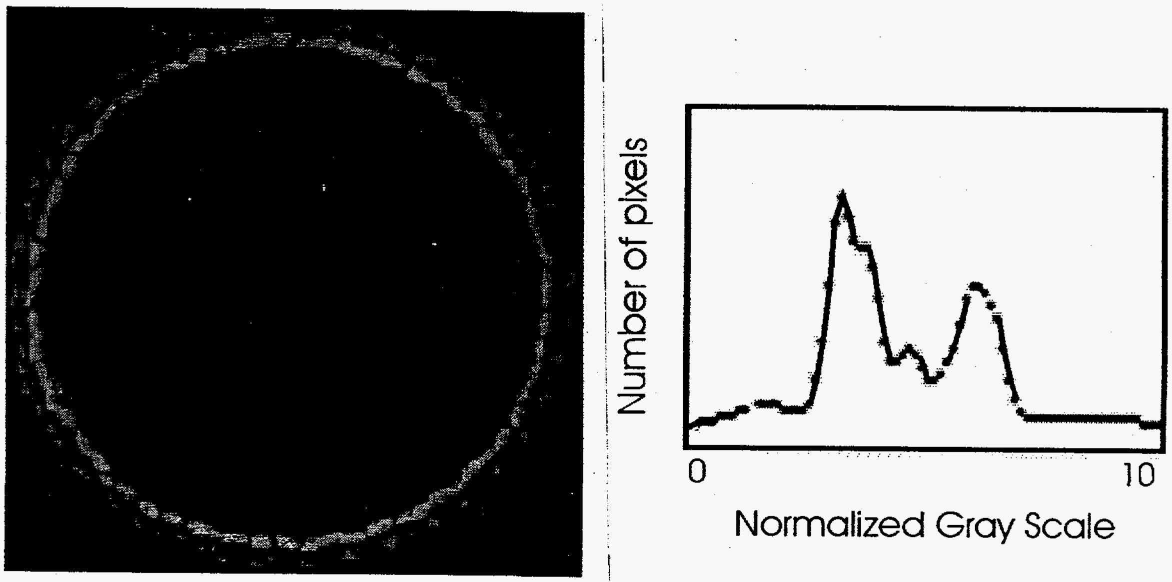
Once the 3D data sets (image and histograms) are obtained, the grayscale threshold can be set to allow any particular part of the filter tube viewed in 3D. This could allow quantification of 3D density variations by looking at before and after operation data. Examples are shown in Fig. 3. Figure 3 shows: a) the image of the outer mesh of the 3M hot gas filter; b) the internal wall (refer to Fig. 2a; white inner ring).

By such analysis, volumetric analysis of trapped material in hot gas filters may be accomplished.



(a) (b)

Fig. 1. Axial density variations for 3M hot-gas filter. (a) Schematic showing data locations. (b) Axial density profile



(a) (b)

Fig. 2. Typical X-ray CT cross section through the 3M hot-gas filter and corresponding gray-scale histogram. Note the high-density inner fabric. (a) CT image. (b) Histogram.

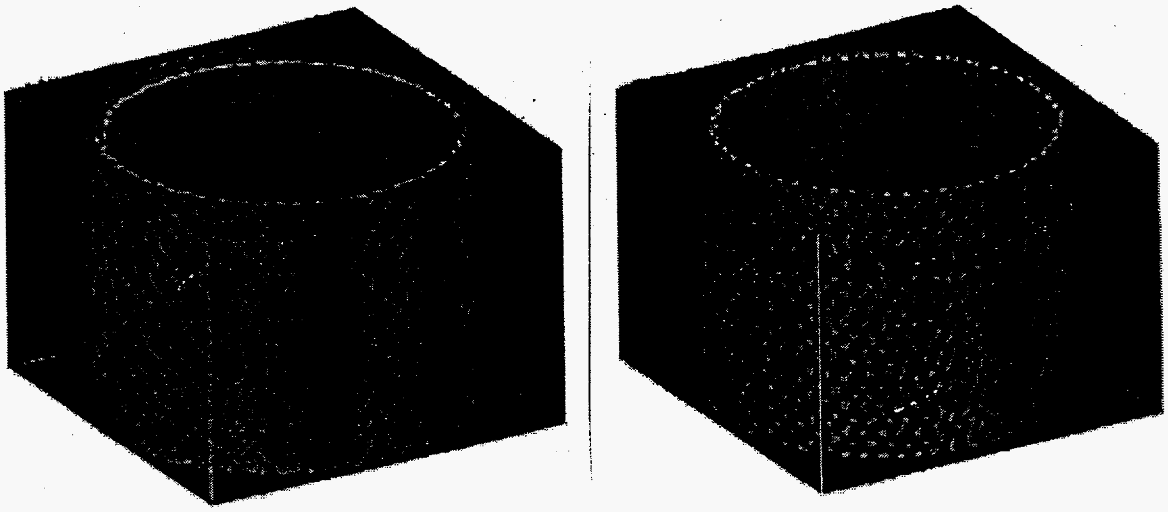


Fig. 3. Volumetric 3D X-ray CT analysis of 3M hot gas filter. a) Verification of detection of outer mesh. b) Inner surface.

b) Heat Exchangers

For the heat exchanger studies, 3DXCT methods were again explored. Two sets of specimens were available: set 1 consisted of eight 10 cm diam. by 10 cm long specimen provided by Oak Ridge National Laboratory (ORNL). These heat exchanger tubes were provided to ORNL by Babcock & Wilcox and had been exposed to conditions used in the E. I. DuPont hazardous waste incinerator. The specimens for NDE had been cut from 1.5 m long tubes. These specimens are identified in Table 1. Note that specimen AR is an as-received (unexposed) specimen. Set two (see Table 2) consisted of 2 CVI SiC/SiC specimens provided by Virginia Polytechnic Institute (VPI) with considerably different fiber architecture. Tube I appears to be a 3D weave. Tube II appears to be a 2D layup with 45° rotations between plys.

While the NDE work conducted so far consisted of analysis using X-ray CT data acquisition, future work will evaluate air-coupled ultrasonic methods and thermal imaging.

Throughwall Density Variations Including Delamination Detection

3DXCT data were acquired at various locations along the axial length of each of the 10 cm long sections from Set 1. At various azimuthal locations at any axial position, through-wall "line plots" were taken to establish the extent of throughwall density variations.

Table 1. Ceramic Heat Exchanger Tubes Used in NDE Study

| Tube ID | Diam (in) | Wall Thickness (mm) | Exposure Position | Visual Observations | Materials | Manu-facturer | Exposure Period |
|---|---|---|---|---|---|---|---|
| 1 | 4 | 3.2 | Right side Front row | Helically wound fibers | Nextel 610 fibers zirconia matrix | B&W | 8 weeks |
| 3 | " | 6.35 | Center of Center row | Hoop wound fibers on surfaces, helically wound internal fibers, thermocouples on tube | Almax fibers zirconia matrix | B&W | 27 weeks |
| 4 | " | " | Center of Rear row | Hoop wound fibers on surfaces, helically wound internal fibers, some unbonded layers visible | PRD166 fibers zirconia matrix | B&W | 27 weeks |
| 5 | " | " | Left side Rear row | Hoop wound fibers on surfaces, helically wound internal fibers, some unbonded layers visible | PRD166 fibers zirconia matrix | B&W | 27 weeks |
| 6 | " | " | Right side Center row | No evidence of fibers | SiC particles alumina matrix | DLC | 27 weeks |
| 7 | " | " | Right side Rear row | Appears to be helically wound fibers, ~3/8" repeat | Type B mixed oxides | DLC | 8 weeks |
| 9 | " | " | Left side Center row | Appears to be helically wound fibers, ~3/8" repeat | Type B mixed oxides | DLC | 8 weeks |
| AR | " | " | Unexposed | Hoop wound fibers, some unbonded layers | PRD166 fibers zirconia matrix | B&W | None |

DLC = DuPont Lanxide Composites

B&W = Babcock & Wilcox

This includes detection of delaminations. A delamination, clearly visible by inspection, was detectable. 3DXCT analysis, see Fig. 4, shows (see position b) that the visible crack is clearly detected whereas "c" position show only a slight density variation.

Table 2. CVI SiC/SiC Heat Exchanger Tubes for NDE

| Tube ID (ANL) | Diam (mm) | Wall Thickness (mm) | Length (cm) | Remarks |
|---|---|---|---|---|
| I | 38 | 6.35 | 18.9 | Appears to be a 3D braided fiber architech |
| II | 38 | 6.35 | 20.3 | Appears to be 2D layup<br>45° interlayer rotation |

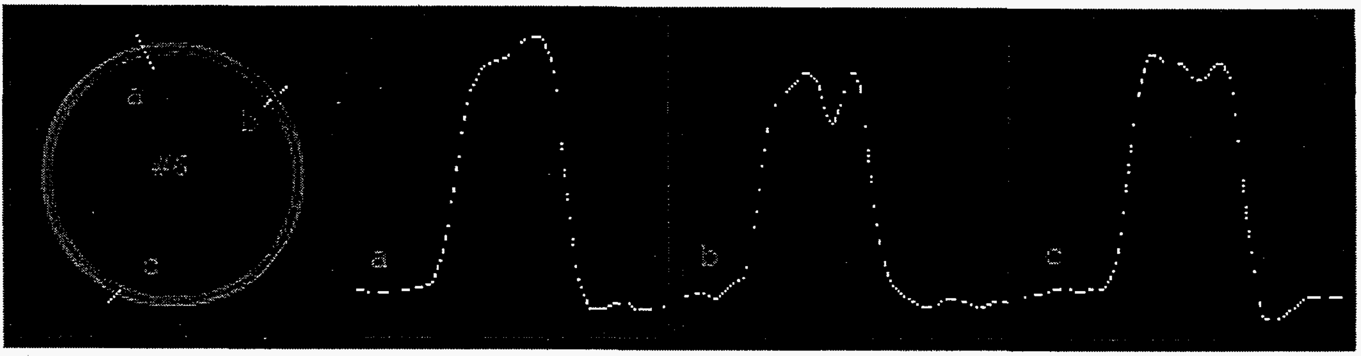


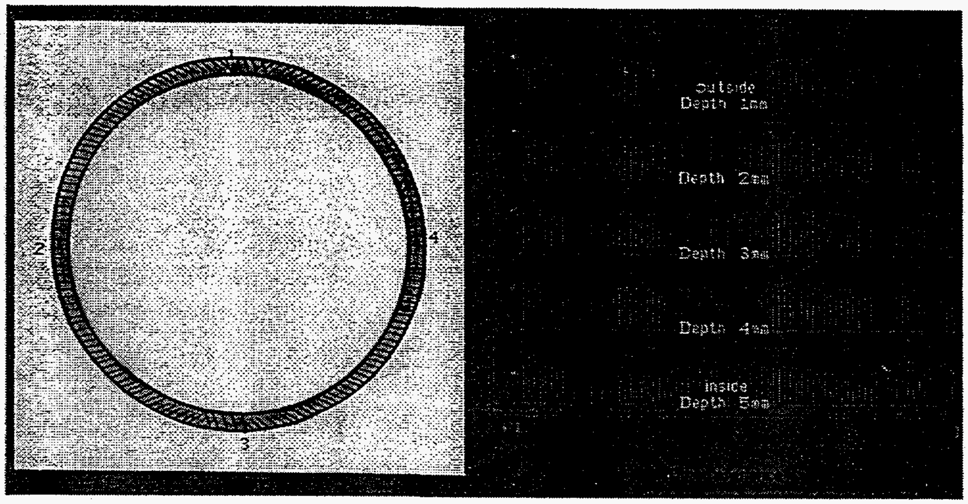
Fig. 4. X-ray CT analysis of heat exchanger tube 6, showing throughwall density profiles at 3 azimuthal positions. Note the delamination detection at location "b".

We conducted similar analysis of the as-received PRD66 hoopwound fiber specimen and again detected delaminations in the wall.

A software package was also developed which allows study of the density variation at any location through the wall using CT axial cross-sections. An example is shown in Fig. 5 for tube 1. Note that at each radial position a 360° density map is determined. The density variations are very small for this particular example.

Axial Density Variations

To establish axial density variations along the tubes, the new software package was used which allows total average density for any CT cross section as a function of axial



(a) (b)

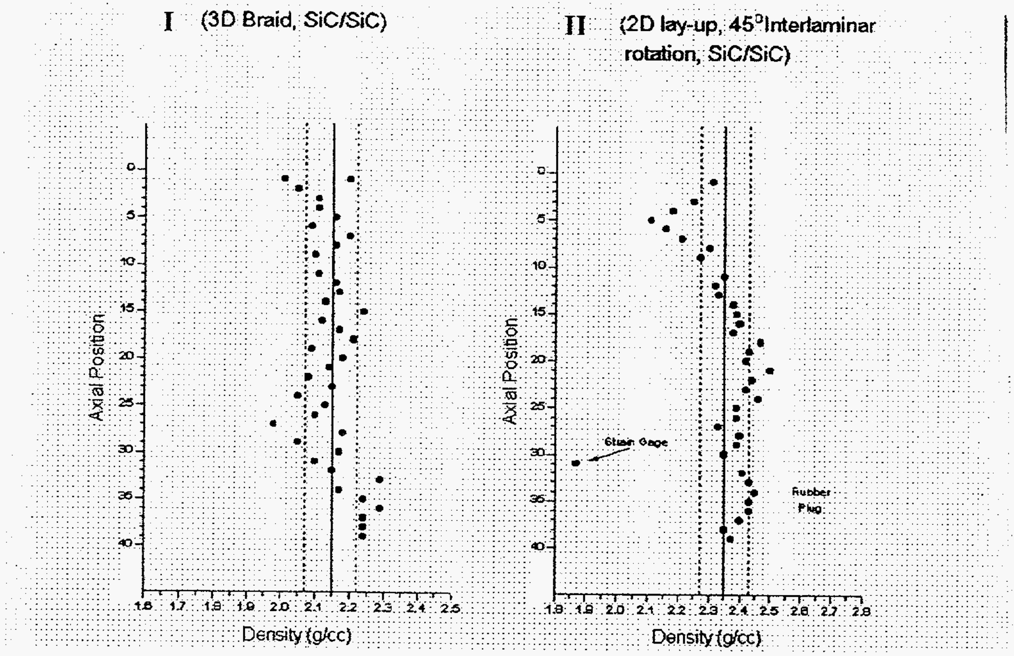
Fig. 5. X-ray CT sectional image of tube 1 and corresponding 360° density profiles at any position through the wall thickness. a) X-ray CT cross-sectional image. b) five 360° density plots at 0.95, 1.90, 2.85, 3.80 and 4.75 mm from the outer wall.

position to be obtained. Thus density as a function of axial position can be plotted for any CT data set obtained. The axial density analysis is shown in Fig. 6 for the two CFCC specimens of Set 2. The density range for the 3D braid is 1.98 to 2.29 g/cc with a mean of 2.15 g/cc while the 2D laminate is 1.87 to 2.50 with a mean of 2.35 g/cc.

Argonne has a new CT facility which will allow 2-2.5 m long tubes to be studied by CT. In addition, a new air coupled ultrasonic system is now being studied for detection sensitivity for CFCC materials part of the CFCC program. This NDE method will also be examined for applicability to hot-gas filter and heat exchanger tubes.

## Thermal Barrier Coatings

Thermal barrier coatings (TBCs) are a key materials element to allow upgrading of turbine gas-firing temperatures and subsequent hot-stage sections for evolutionary turbine materials such as superalloys. The mechanical integrity of these TBC's depends on careful control of the microstructure as well as the bond coat which is used to join the TBC to the alloy substrate.



(a) (b)

Fig. 6. Axial density profiles for two CVI SiC/SiC CFCC heat exchanger tubes. (a) 3D weave. (b) 2D laminate.

For fossil energy applications, e.g., coal-gas-fired turbines, high temperatures and long-term exposures increases the likelihood of spallation of the TBC and subsequent turbine blade failure. Key development issues for TBCs include: a) reliability of bond coat interface, b) thermal expansion characteristics of the TBC and c) thermal conductivity across the TBC thickness.

For the thermal barrier coating studies, this NDE work initially concentrates on two methods: a) a non-contact, non-invasive method called Time-Resolved-Infrared-Radiometry (TRIR).(4,5) and b) elastic optical scatter.(6) In the TRIR method (see Fig. 7), a thermal excitation source and a high sensitivity, high frame rate infrared camera are located on one side of the thermal barrier coating. The thermal excitation source can be a high energy flash lamp (we have a 1.6 Kj lamp) or a scanning laser. The thermal pulse applies a heat source which penetrates the TBC and reflects off the bond-coat (may reflect off bond coat-substrate interface depending on wave-length of excitation). The reflected thermal "pulse" is then time-dependent recovered by the infrared camera and the individual full-field thermal frames are

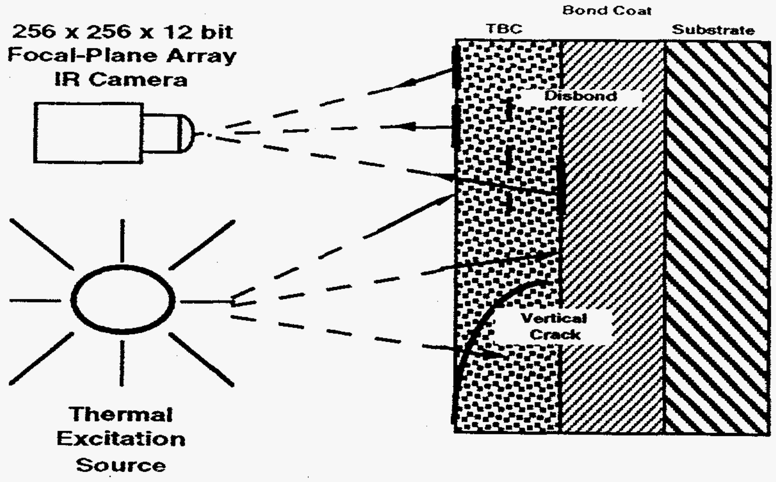


Fig. 7. Schematic diagram of one-sided time-resolved-infrared radiometry method. Note: Schematic shows two defect types: a) disbond, b) vertical crack.

stored in a digital computer. For a disbond of the coating, the temperature rise detected by the camera is much higher as the input heat pulse is not absorbed in the substrate.

## Joining

Ceramic-to-ceramic joining methods are usually necessary both for shop fabrication of complex shaped parts and for field repair. There are several potential joint architectures as shown in Fig. 8. These include lap joints, socket flange, straight socket and conical joints.(7)

Rabin et al.(8,9) of Idaho National Engineering Laboratory (INEL), have been developing joining methods as part of this program. They have been primarily focusing on tape casting sheets of SiC + C precursor, clamping the tape between the parts to be bonded, and then infiltrating with molten Si to form a reaction bonded silicon carbide (RBSC) joint interlayer.

Regardless of the method used for joining, NDE methods are necessary to establish the completeness and quality of the joint. The development of NDE methods to study joint quality is the focus of this part of the NDE work.

For the SiC/SiC NDE joining studies, efforts are concentrating on CFCC materials and this work is being conducted in cooperation with the CFCC program. Cooperative efforts have been established with DuPont Lanxide Composites (DLC) as well as Dow-Corning and with INEL.

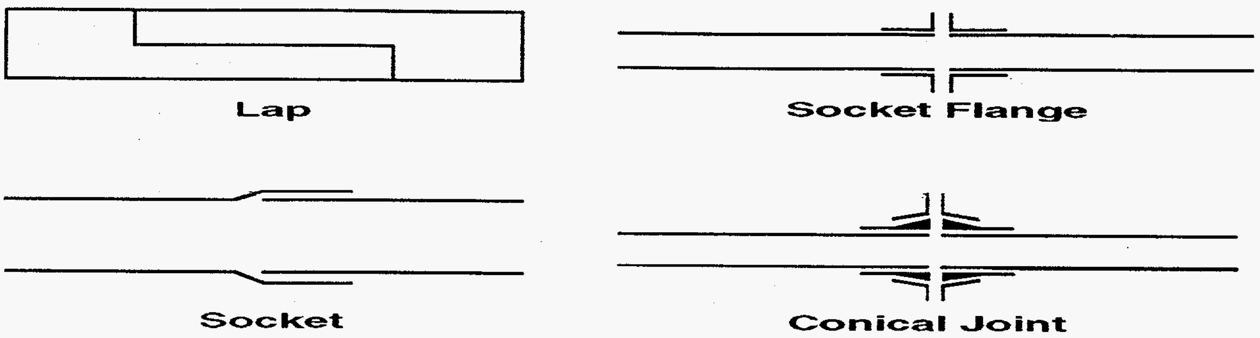


Fig. 8. Schematic diagrams of several possible CFCC joint configurations.

Lap-joint specimens have been received from DLC as noted in Table 3 below.

Table 3. Lap joint CVI CFCC SiC/SiC specimens for NDE

| Specimen Number | Specimen Length | Specimen Thickness | Comments |
|---|---|---|---|
| 4U-1B-1 | 41 mm | 3 mm | Lap joint at midpoint length Lap joint = 15 mm long |
| 3C-1B-1 | 43 mm | 3 mm | Center lap joint<br>Lap joint ≈ 14 mm long |
| 3C-2A-1 | 42 mm | 3 mm | Center lap joint unknown<br>Lap joint length: estimate 13-14 mm |
| 2A-C | 42 mm | 3 mm | Center lap joint unknown<br>Lap joint length:estimate 13-14 mm |
| 3U-2A-5 | 42 mm | 3 mm | Center lap joint<br>Lap joint ≈13 mm long |

Metallic Si flashing on the specimens was removed by a diamond grit grinding wheel. Several NDE studies will be conducted on these and subsequently mechanical property data will be obtained. NDE studies will include, thermal infrared studies, air-coupled through-transmission ultrasonic studies, pulsed multi-frequency eddy-current and microfocus through-transmission X-ray imaging. As field repairs will be part of joining, NDE methods are being developed which have field application potential.

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