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CHARACTERIZATION OF MELT-INFILTRATED SiC/SiC COMPOSITE COMBUSTOR LINERS USING MESO- AND MICRO-NDE TECHNIQUES*

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ABSTRACT

Melt-infiltrated ceramic matrix composite SiC/SiC material systems are under development for use in combustor liners for low-emission advanced gas turbines. Uncertainty in repeatability of processing methods for these large components (33-76 cm diameter), and hence possible reduced reliability for the end user. This requires that appropriate test methods, at both meso- and micro-scale, be used to ensure that the liners are acceptable for use. Nondestructive evaluation (NDE) methods, if demonstrated to reliably detect changes caused by processing, would be of significant benefit to both manufacturer and end user. This paper describes the NDE methods and their applications in detecting a process upset in a melt-infiltrated 33 cm combustor liner and how high-resolution scanning electron microscopy was used to verify the NDE data.

1.0 INTRODUCTION

The overall engine efficiency and emissions of industrial gas turbines is limited chiefly by the mechanical and thermal properties of the hot- section materials. Solar Turbines, as well as other gas turbine engine manufacturers, is evaluating ceramic materials for use in the hot section in order to increase the turbine rotor inlet temperature (TRIT) and thus increase efficiency and reduce emissions (van Roode et al. 1997; Price et al. 1998). The Centaur 50S engine is being retrofitted with ceramic components by Solar Turbines, under support from the U.S. Department of Energy (DOE) (see Fig 1).

This engine has a SoLoNOx combustor and can produce 4350 KW_e output power. While monolithic Si_3N_4 ceramic materials are being studied for application to the first-stage blades and vanes, ceramic matrix composites (CMCs) are being studied for application as the combustor lining. The combustor is an annular unit with a 76 cm (30 in.) outer diameter and a 33 cm (13 in.) inner diameter (see Fig 2).

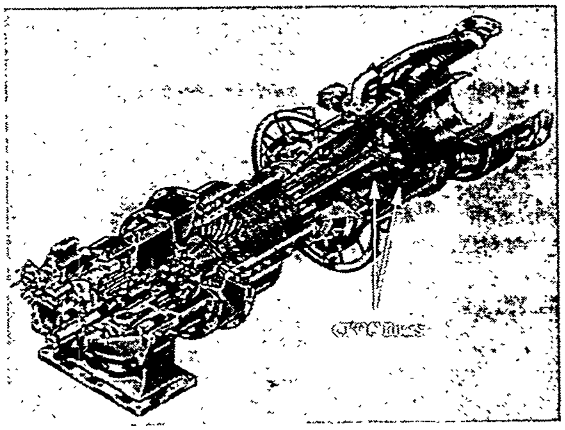


Fig. 1. Cutaway diagram of Solar Turbine Centaur 50S, showing location of ceramic matrix composite combustor liners

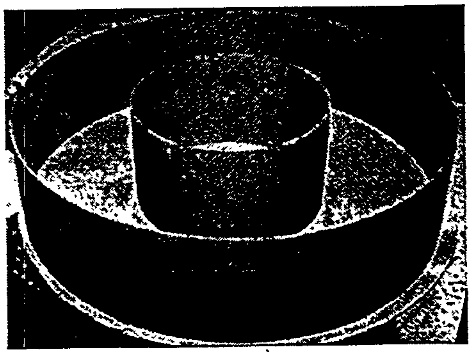


Fig. 2 Photograph of the CMC liner set for Centaur 50S

CMC liners have now been tested in operating engines at operational temperatures for >5000 h (Miriyala et al. 1998). The desired operating life of the liners is >30,000 h and up to 600 thermal cycles over a 10-year period. Thus, it is essential to have reliable nondestructive evaluation (NDE) methods to assess the status and condition of the liners before and during service. NDE is also useful for developing of processing technology aimed at reducing manufacturing costs. NDE technology is now under development for characterizing the CMC materials, and data from NDE has shown excellent correlations with destructive analysis (Sun et al. 1999). Numerous processing methods are being developed for processing CMC materials; one such method for SiC/SiC composites is melt-infiltration (MI), in which Hi-Nicalon SiC fabric is shaped into a perform and subsequently the matrix material is added via infiltration by using molten metallic silicon. While any upset during the infiltration process is undesirable, it was thought that no residual effects would result from such upsets. This has since been discovered to not be the case, and the increase in porosity that resulted from an upset was shown to be detectable by NDE methods and verified by high-resolution SEM. An MI SiC/SiC liner that had been shown by NDE to have a detectable difference in density due to a known process upset was run for 2758 h and then removed from the Centaur 50S engine and destructively analyzed.

2.0 FABRICATION OF MI SIC/SIC LINER

Melt-infiltration processing is known to yield a matrix with a higher density than does chemical vapor infiltration (CVI) (Luthra et al. 1993; Suyama et al. 1998). However, the required processing temperatures for MI are significantly higher than for CVI. To achieve infiltration, higher-temperature fibers such as Hi-Nicalon or S-200 must be used, rather than the lower-cost and lower-temperature ceramic-grade (CG) Nicalon. During processing, keeping the metallic silicon present is essential.

3.0 DESCRIPTION OF NDE TECHNIQUES

Argonne National Laboratory (ANL) has been developing several NDE techniques primarily for mesoscopic (>0.2 mm) characterization of CMC material systems (Pillai et al., 1997; Spohnholtz 1999; Sun et al. 1999; Sivers et al. 1996). These NDE techniques include infrared-based thermal imaging used to measure thermal diffusivity; air-coupled ultrasound systems that do not require water couplants; impact acoustic resonance methods for determining elastic modulus and damping factors; X-ray imaging technology (primarily tomographic imaging) for verifying internal component damage, e.g., throughwall damage on turbine engine combustor liners, transition ducts, etc.; and recently, implementation of more conven-tional water-coupled ultrasonic methods for certain CMC material systems. Each of these techniques will be briefly described, and data from application to various CMC components will be given.

3.1 Thermal Imaging

The experimental apparatus for measuring through-thickness thermal diffusivity is illustrated in Fig. 3. It includes an IR camera with a focal-plane array of 256 x 256 InSb detectors, a 200-MHz Pentium-based PC equipped with a digital frame grabber, a flash-lamp system to provide the thermal impulse, a function generator to operate the camera, and a dual-timing-trigger circuit for the camera and external trigger control (Sun et al. 1999). An analog video system is used to monitor the experiments. Processing time to measure a typical diffusivity image with 256 x 256 pixels ranges from 8 to 20 min, depending on the number of frames taken. Thermal diffusivity is calculated as described in Parker et al. (1961), which assumes that the front surface of the sample is heated instantaneously.

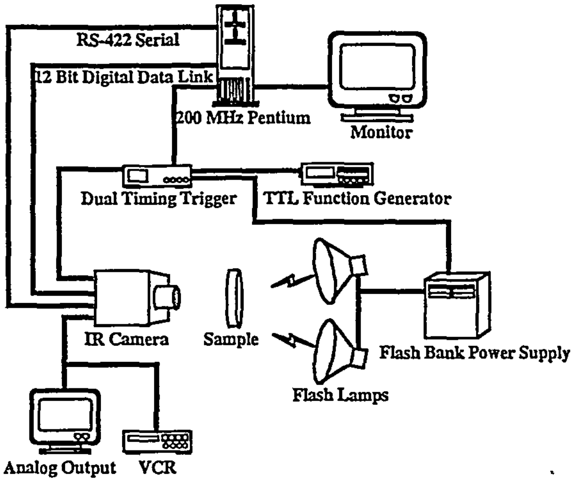


Fig. 3. Schematic diagram of experimental thermal imaging apparatus

3.2 Air-Coupled Ultrasound

The air-coupled ultrasonic system (Pillai et al. 1997) consists of a traditional computer- controlled x-y-z positioning system with two computer-controlled matched 400 kHz piezo-electric air-coupled transducers, as shown in Fig. 4. Tone bursts of 0.4-MHz acoustic energy from the emitting transducer are incident on the sample, with no use of immersion fluid or special coupling, and the transmitted energy is detected by the receiving transducer, which uses a low-noise, high-gain preamplifier. The detected signal from the preamplifier is used as input to a highly tuned amplifier and to an electronic time gate. The digital value of the peak voltage in the preset time gate is displayed and plotted in an x-y array. An "image," referred to as a C-scan image, of the component under study is built up by using nominal 800 µm step sizes in both the x and y directions. The acoustic signal is attenuated by differences in the material, e.g., a delamination, change in density, etc. Thus, areas with defects (such as pores and delaminations) cause different voltage levels.

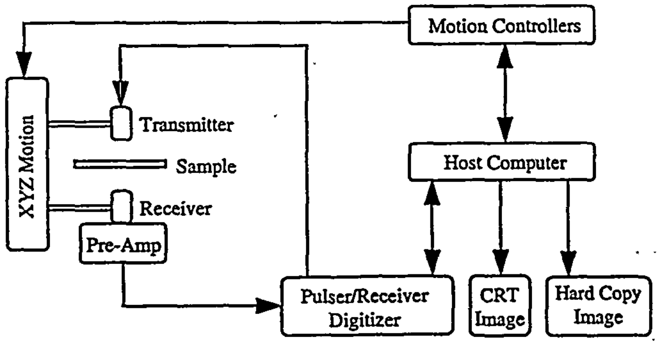


Fig. 4. Schematic diagram of air-coupled ultrasonic system.

3.3 Conventional Water-Coupled Ultrasound

This NDE arrangement was a standard commercially available SONIX system. The transducers were SIGMA 5 MHz, 12.5-mm diameter, 50-mm-focal-length immersion types. Scanning used a through-transmission arrangement similar to that shown in Fig. 4 for the air-coupled ultrasonic system.

3.4 Impact Acoustic Resonance

Impact acoustic resonant (IAR) spectroscopy, often also referred to as the "ping" test, is commonly used to manually sort "good" from "bad" parts by listening to the differences in the audible sounds emitted. For more systematic analysis, the component or specimen is excited, usually by a controlled-impact method, and the vibratory response is detected by a contact or noncontact transducer (Spohnholtz 1999). The resulting digitized detected acoustic signal is then sent to a computer containing special digital signal processing software for subsequent signal processing. Figure 5 is a schematic diagram of a typical data acquisition system utilizing a condenser microphone as a detector and impact excitation via an electromechanical shaker driven by a function generator.

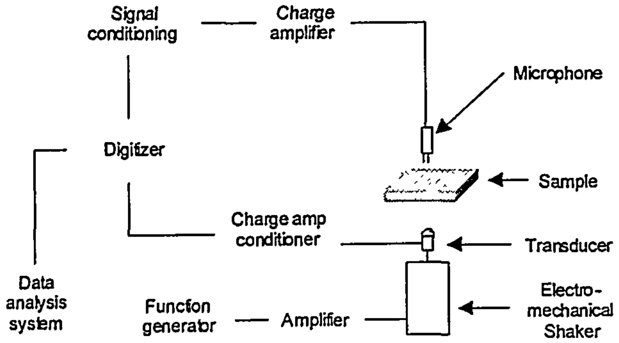


Fig. 5. Schematic diagram of typical impact acoustic system that uses a condenser microphone as the detector

3.5 X-Ray Imaging

While conventional X-ray through-transmission imaging using film or solid-state-area detectors may be useful for fairly flat specimens (providing plan-view information about location and size of large voids, or large variations in the fiber layup architecture), such an NDE approach provides no information about delaminations or density variations through the thickness. Tomographic X-ray imaging, on the other hand, is insensitive to object shape and provides direct information about delaminations and through-thickness density variations. Use of small X-ray area detectors for computerized tomography data acquisition of large components has been demonstrated, using as components 20-cm-diameter CMC combustor liners (Sivers et al. 1996). Figure 6 shows images of 3-mm-thick SiC/SiC CMC combustor liners and indicates how interior delaminations are directly detectable.

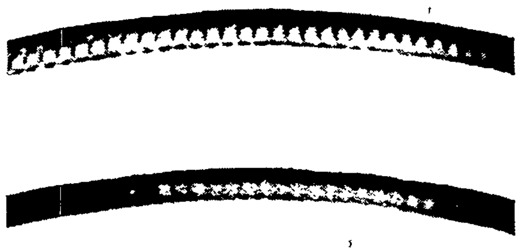


Fig. 6. X-ray tomographic images of 3-mm-thick wall sections of CMC combustor liner

4.0 NDE RESULTS

The MI liner mentioned in Section 1.0 was examined for various defects and characteristics, before and after the 2758-h test, by three NDE methods: air-coupled ultrasound with through-transmission C-scanning, water-coupled ultrasound with through-transmission C-scanning, and through-transmission thermal imaging with software to compute a thermal diffusivity map. After the liner was studied by these NDE techniques, it was cut into sections as indicated in Fig. 8. The SEM data presented earlier were acquired from segments 3 and 6 in Figure 7.

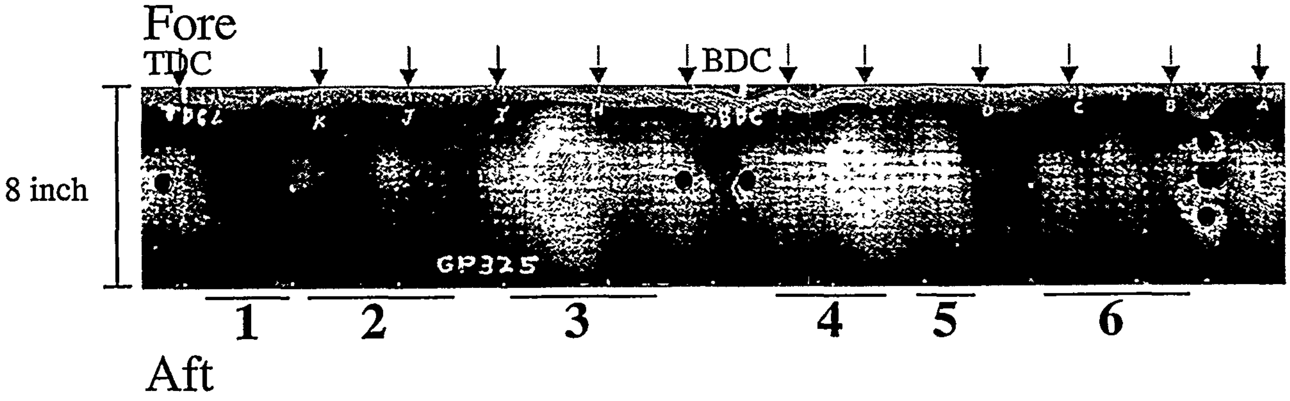


Fig. 7. Schematic diagram of SiC/SiC combustor liner, showing where segments were obtained for destructive analysis

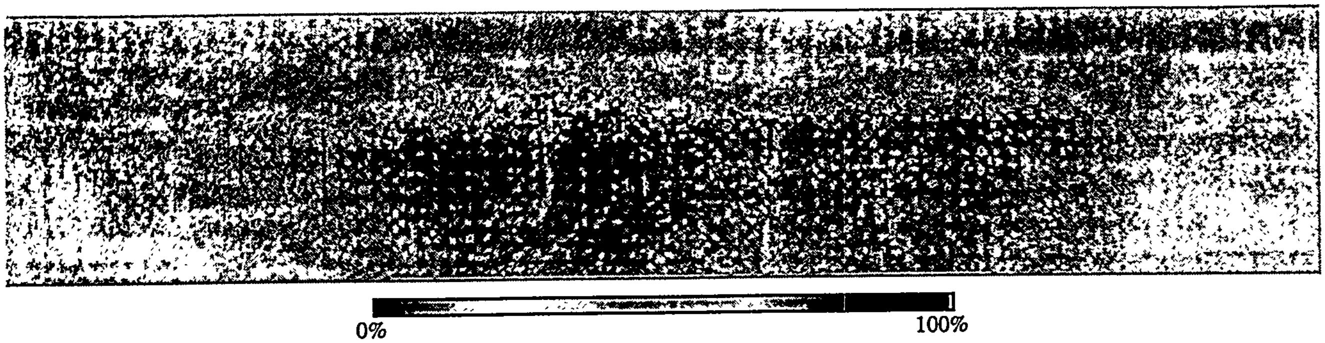


Fig. 8 **Air coupled UT C-scan before testing in engine**

The air-coupled ultrasonic data were acquired by using the 400 kHz transducers in a through- transmission arrangement. Scan speed was 4 cm/s. Data obtained before the 2758-hr run and shown in Fig. 9, and the air ultrasound data after the 2758-h run are shown in Fig. 10. Virtually no difference is seen in the data, indicating no change in the liner's condition.

The water-coupled ultrasound data were obtained with the same set-up before (Fig. 11) and after the test run. Data were not acquired after engine testing because of experimental problems.

The thermal diffusivity data were obtained with the through-transmission arrangement. Data from before and after engine testing are shown in Figs. 11 and 12. Again, little change is noted, indicating no change in the condition of the liner. This is in agreement with post-mortem tensile testing, which shows that the material tensile strength was minimally reduced after the test run.

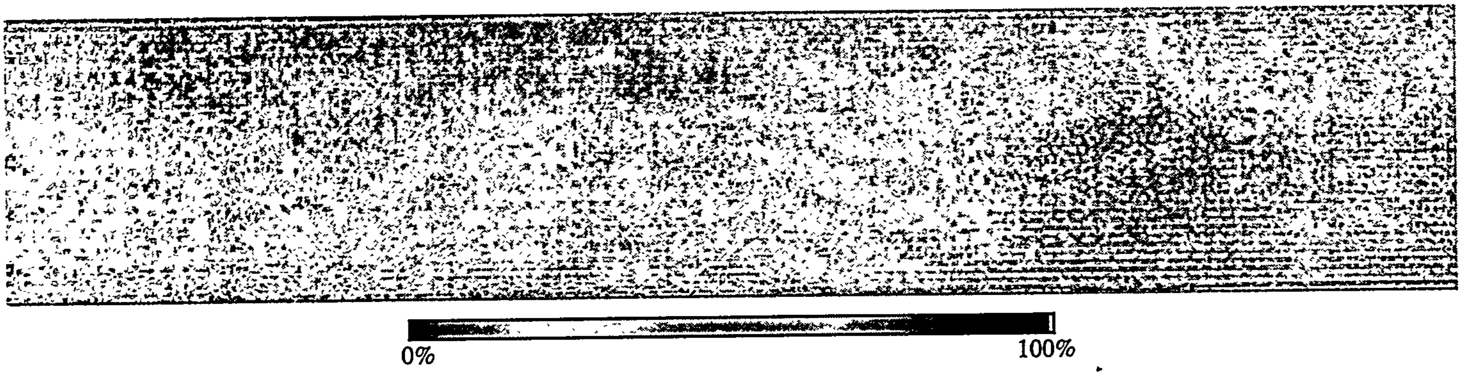


Fig. 9. **Air-coupled ultrasound C-scan after testing in engine**

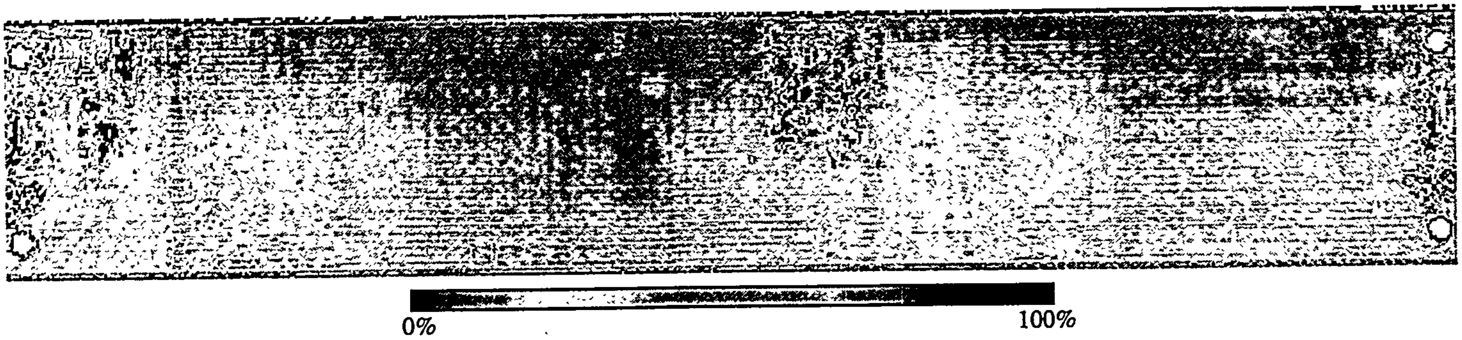
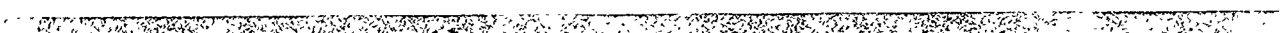


Fig. 10. **Water-coupled ultrasound C-scan before running in engine**



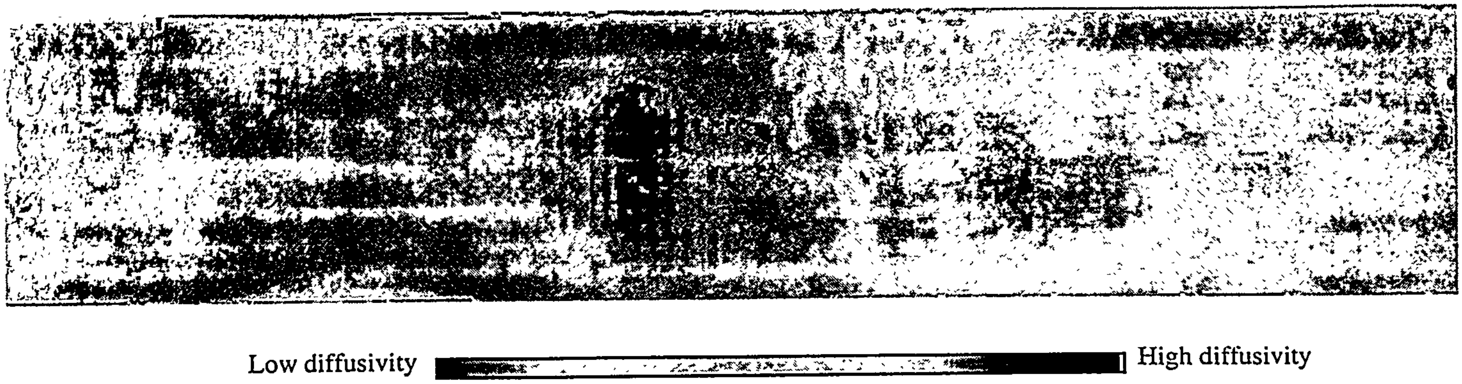


Fig. 11. Thermal diffusivity image data before engine testing

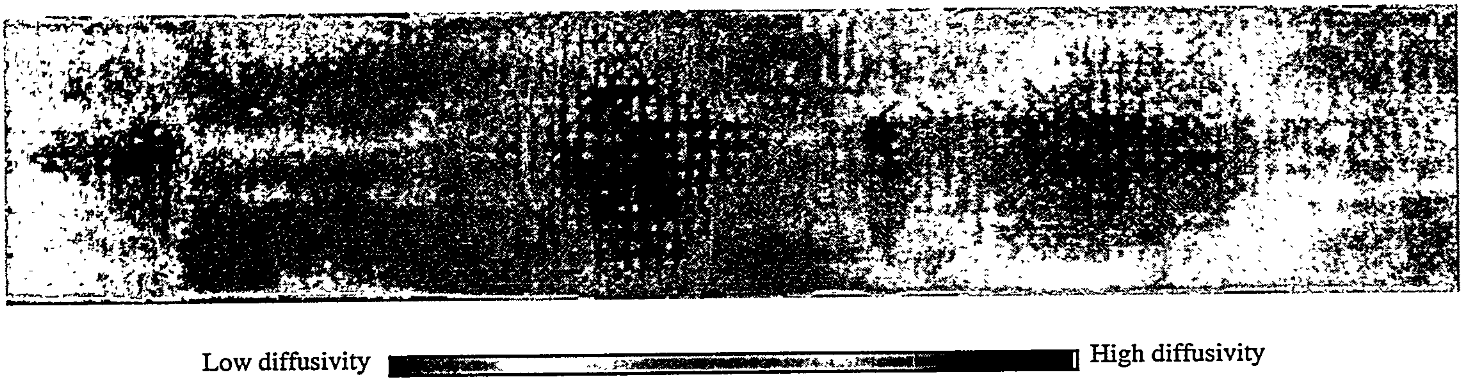


Fig. 12. Thermal diffusivity data after engine testing

5.0 DESTRUCTIVE ANALYSIS AND SEM RESULTS

Two of the sections cut from the MI inner liner change in the condition of the liner. This is in agreement with post-mortem tensile testing, which were used for cross-section evaluation by SEM. Each 20 cm (8-in.) strip was cut into eight individual 2.5-cm (1-in.) wide sections, mounted in cross section, and polished. Several SEM images were taken from each section and thus the entire cross section was viewed at high magnification, aft to fore. When all the images were viewed together, forming one complete strip in cross section, and each strip was compared to others similarly obtained, one feature was clearly evident in all the sections. The top section of the liner (aft) was consistently less dense than the bottom section (fore). In fact, the entire top half of the liner was much less dense than the bottom half. SEM images comparing a typical aft region and fore region are shown in Figs. 13(a) and 13(b), respectively. The macropores evident in Fig. 13(a) are very large (hundreds of micrometers in length) and are readily detected by the NDE techniques. Thus, the observed differences in the NDE data can be explained by the excessive amount of open porosity in the fore section of the liner. The porosity is present within the MI matrix between fiber tow regions and indicates lack of infiltration.

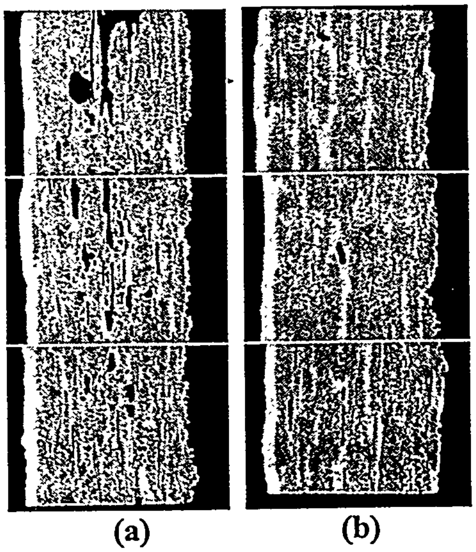

(a) (b)

Fig. 13. SEM image data from two regions of MI SiC/SiC CMC liner: (a) upper (fore) section, (b) lower (aft) section

6.0 SUMMARY

Three NDE methods were used to examine a melt-infiltrated SiC/SiC ceramic matrix composite combustor liner. All three demonstrated detection of a process upset that resulted in a porosity increase in the liner. After the liner was run in an engine for 2758 h and then subjected to the five NDE techniques, it was destructively analyzed; high-resolution SEM verified the existence of increased porosity in the upper section of the liner. Further, the posttest NDE data suggested little change in any material property. This, too, was verified because tensile strength data from the post-mortem analysis showed little strength loss.

7.0 ACKNOWLEDGEMENTS

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