**DEVELOPMENT OF NONDESTRUCTIVE EVALUATION METHODS
FOR STRUCTURAL CERAMICS***

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ABSTRACT

Development of nondestructive evaluation (NDE) methods for application to fossil energy systems continues in three areas: (a) mapping axial and radial density gradients in hot-gas filters, (b) characterization of the quality of continuous-fiber ceramic matrix composite (CFCC) joints and (c) characterization and detection of defects in thermal-barrier coatings. In our work, X-ray computed tomographic imaging was further developed and used to map variations in the axial and radial density of two full-length (2.3-m) hot-gas filters. The two filters differed in through-wall density because of the thickness of the coating on the continuous fibers. Differences in axial and through-wall density were clearly detected.

Through-transmission infrared imaging with a highly sensitivity focal-plane array camera was used to assess joint quality in two sets of SiC/SiC CFCC joints. High-frame-rate data-capture suggests that our infrared imaging method holds potential for the characterization of CFCC joints.

Work to develop NDE methods that can be used to evaluate electron-beam physical-vapor-deposited coatings with platinum-aluminide (Pt-Al) bonds was undertaken. Coatings of Zirconia with thicknesses of 125 μm (0.005 in.), 190 μm (0.0075 in.), and 254 μm (0.010 in.)

with a Pt-Al bond coat on René N5 Ni-based superalloy were studied by infrared imaging. Currently, it appears that thickness variation, as well as thermal properties, can be assessed by infrared technology.

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 (CFCCs) for hot-gas filters and heat exchangers, and thermal-barrier coatings (TBCs) for high gas-firing-temperature turbines.

Detection and measurement of variations in the axial and radial density of hot-gas filters are important, because change in density can be used as a process control parameter during fabrication and because density measurements can be used to study the efficiency of back-purging during cleaning.

Ceramic-to-ceramic joining is usually necessary both for shop fabrication of parts with complex shapes and for field repair. Many processes are under development to join CFCC materials. Researchers at the Idaho National Engineering Laboratory (INEL) joined one set of specimens with a lap joint. A second set, also joined with a lap joint, was produced by Dow-Corning.

While focusing on monolithic SiC and CFCC's with SiC/SiC, Rabin¹ and Rabin and Moore² of INEL clamp tape cast sheets of SiC + C precursor between parts that are to be bonded, and then infiltrate molten Si to form a reaction-bonded silicon carbide (RBSC) joint

interlayer. Regardless of the method used for joining, NDE methods must be developed to establish the completeness and quality of the joint.

Thermal-barrier coatings are key material elements that allow higher gas-firing temperatures in gas turbines with low emissions. The integrity and properties (thermal and physical) of these TBC's depends on a reliable bond coat and careful process control.

For fossil energy applications, e.g., coal-gas-fired turbines, where the turbine duty cycle is much different from a typical aircraft engine, long-term, high-temperature exposure and high-cycle fatigue can be an issue, and the likelihood of spallation is increased. Answers to questions such as; What is the status of the coating? If damaged, should the blade be repaired? replaced? become matters of concern.

Work on the NDE of TBCs is initially concentrating on two methods: a) noncontact, noninvasive time-resolved-infrared-radiometry (TRIR)^{3,4} for thermal characterization and b) elastic optical scatter⁵ for crack detection and disbonds. In the TRIR method a thermal excitation source and a highly sensitive, high-frame rate (up to 1900 frames/sec) infrared camera are placed on one side of the TBC. The thermal excitation source can be a high-energy flash lamp or a laser. Thermal images are acquired in time sequence and analyzed.

METHODS AND RESULTS

Hot-Gas Filters

The use of high-spatial-resolution X-ray computed tomographic (XCT) methods for the study of hot-gas filters and heat exchangers has been explored previously.^{3,6-8} In the work described here, two 2.3-meter-long, 3.8-mm-O.D. Nextel 611 fiber/SiC matrix hot-gas filters, supplied by 3M Corporation, were examined by XCT imaging densitometry. One of

the filters was heavily coated, whereas the other was lightly coated. Figure 1, a schematic diagram of a typical filter, shows the location of the coating.

Fig. 1. Schematic diagram of cross section of 3M hot-gas filter

The XCT images were acquired every 50 mm along the length of the filters, as shown in Fig. 2, which is a projection X-ray digital radiograph (XDR). (Note that a holder is shown at the bottom of the XDR. This holder incorporates the flanges for mounting the hot-gas filters.)

Fig. 2. X-Ray projection digital radiograph showing axial locations of X-ray tomographic images used for densitometry

The acquired XCT data clearly delineate differences between heavy and light coatings, as indicated in Fig. 3, which shows typical XCT images of the two hot-gas filters. By using digital image processing and determining the average density of each XCT image, one can plot relative density as a function of axial position. The variations in relative axial density that were obtained in this manner are shown in Fig. 4.

Fig. 3. Typical X-ray computed tomographic images of the 3M hot-gas filters;
(a) heavy SiC coating, and (b) light SiC coating

Fig. 4. Variations in relative axial density of two 3M hot-gas filters

Joints

To study the NDE of ceramic-to-ceramic joints, we used a butt-joint monolithic 15-cm-diameter SiC tube that was joined for use in a corrosion test facility at Oak Ridge National Laboratory. The tube (Fig.5) was studied by two NDE methods: XCT and impact acoustic resonance (IAR).⁹ The XCT method was used to determine if large voids existed at the joint and IAR was used to the joint establish if the joint was solid, which perhaps could be considered a function of the elastic properties.

Fig. 5. Photograph of 15-cm-diameter SiC/SiC butt-joined tube

A 450 KVp XCT system was used to obtain a set of XCT images through the joint region. The XCT "slices", which were 500 μm thick, were taken sequentially, with 300- μm off-sets. Three XCT images of the joint are shown in Fig. 6, which reveals several features,

Fig. 6. X-ray CT images through butt joint of 15cm SiC/SiC tube;

(a) open unjoined areas, (b) less dense regions, and (c) so-called drop-through or regions where the "weld" material dropped to the inside. This suggests that XCT can be used to detect regions that lack penetration and, perhaps, density variations in the joint.

In the IAR method, the specimen is struck with an instrumented hammer and the resultant acoustic signal is detected by a microphone. Both the signal and the instrumented hammer are digitized in a 100-Mhz dual-channel digitizer. Impact excitations are used at uniform distances around the tube on both sides of the buttjoint. The collected data are then analyzed for changes. Results to date correlate with the voids detected by XCT, in that poorly joined regions show significantly differing frequency peaks.

Thermal-Barrier Coatings

For the work on TBCs, nine 20-mm-diameter specimens were coated with René N5 substrate, PtAl bond coat, and ZrO₂ by electron-beam physical vapor deposition. Coatings of three thicknesses, 125 μm , 190 μm , and 254 μm , were applied. In addition, two full-scale aeroderivative turbine blades that had been coated were also studied.

An area of technical interest is uniformity of coating thickness. One-sided flash thermal imaging was used to study the three coating thicknesses and the turbine blades. Sensitivity to

thickness can be attained by obtaining time-dependent images and using digital image processing. Figure 7 shows data for each specimen and both turbine blades. These data suggest that the blades are covered with have 125- μm TBCs.

While not verified destructively, it has been suggested by the suppliers that, indeed, the typical coating thickness would be expected to be in the range of 125 μm . Thus, there is reason to believe that the thermal-flash method a reasonably be used for full field thickness measurement.

Fig. 7. Sensitivity of flash infrared imaging to EB-PVD thickness of coating applied by (Substrate is René N5 and bond coat is Pt Al).

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R. Orenstein of GE Power Generation, and B. Seth of Westinghouse Power Systems provided thermal barrier coating specimens

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