Microtomographic Imaging of Ceramic Membranes and Other Structural Ceramics

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Introduction

The work for the project this year was to explore the potential of high-spatial-resolution microtomography for analyzing a variety of engineering ceramic materials. These included SiC/SiC ceramic matrix composites as well as various materials being developed for inorganic high-temperature gas-separation membranes. Inorganic gas-separation membranes are being developed for producing O_2 and H_2. One primary area of interest related to membrane development is the establishment of well-defined, leaktight joints. Another area is the definition of processing parameters for the deposition of materials (in this case, palladium). The joining of membranes is of primary importance, since leaking joints can poison gas. In these first membrane studies, done in conjunction with both Los Alamos National Laboratory and Argonne National Laboratory, the materials used were (1) a high-density mixed oxide Sr-Fe-Co-O system and (2) a porous oxide substrate with a palladium coating about 20-µm thick.

Methods and Materials

All the work was done by using the specialized microtomographic station established by M. Rivers. Various energy levels between 30 and 40 keV were used, with less than 0.1% spread. The microtomographic work station used a CsI scintillator screen and a charge-coupled device (CCD) array detector. The materials used were of two types. The first type was SiC/SiC melt-infiltrated ceramic matrix composites [1, 2] prepared by the National Aeronautics and Space Administration (NASA), Glenn Research Center, Cleveland, Ohio. Of interest in these experiments was the ability to detect various crack densities when the specimens had been subjected to various schedules of tension-tension cyclic fatigue. Second was a set of specimens containing two types of inorganic membranes used for high-temperature gas separation [3-5]. One set consisted of tubes about 1 cm in diameter made of the Sr-Fe-Co-O mixed oxide system [6]. The other was a porous alumina substrate coated with palladium about 20-µm thick.

Results

The microtomographic imaging tests on the inorganic membranes demonstrated that the high resolution from the micro-imaging technique will indeed be viable for studying the quality of as-joined materials. Figure 1 shows an example of x-ray microtomographic images of a nonleaking joint, and Fig. 2 shows the same images of a leaking joint. Both Figs. 1 and 2 show a cross section through the joint and an axial section that covers the length of the joint. By visually comparing the two images, one can easily see that the joint interface has a larger gap and hence was the location of the leak. Figure 3 is a cross-sectional image of the porous alumina tube with the palladium coating. While this example is of a uniform thickness, some of the cross sections displayed a more nonuniform thickness.

This study is of interest with regard to the plating process for palladium. It is important to note that the exploratory work on joining has opened up the possibility of studying the wetting characteristics of the base material and analyzing the flow characteristics in near real time, which would allow for new advances in joining technology. The micro-imaging tests run by using the SiC/SiC composites demonstrated that one can indeed discern variations in the crack spacings of specimens that have undergone different tension-tension cyclic fatigue schedules. This kind of data allows for advances in the understanding of cyclic fatigue failure of these materials.

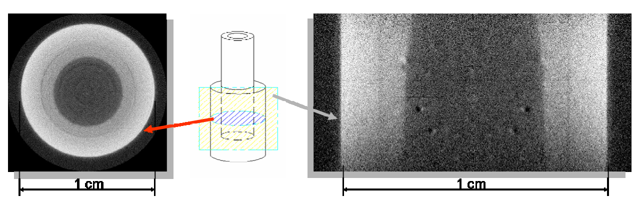

FIG. 1. Microtomographic image of a nonleaking joint.

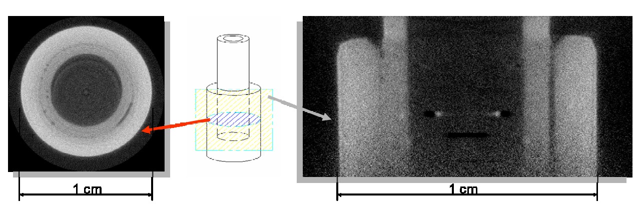

FIG. 2. Microtomographic image of a leaking joint.

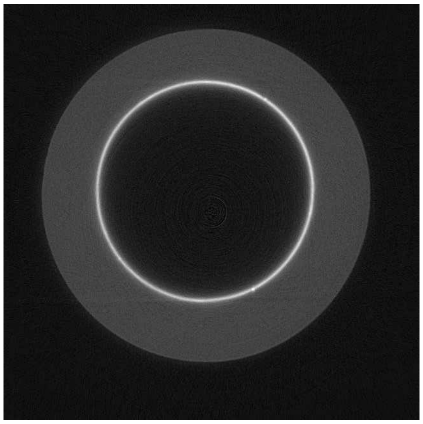

FIG. 3. Microtomographic cross-sectional image showing the uniformity of the palladium distribution on the inside of the porous inorganic membrane support.

Discussion

On the basis of the results obtained over this year, plans have been made to run more extensive tests next year, especially for the inorganic membrane studies. In this case, the effects of thermal cycling on the palladium-coated porous substrates will be studied in a cooperative effort with Los Alamos National Laboratory. Moreover, a request has been made to the U.S. Department of Energy (DOE) offices covering fossil energy and advanced materials to begin a study of the joining of inorganic membranes used for high-temperature gas separations. The current plan is to use the available beamtime over the next few years.

Acknowledgments

Use of the APS was supported by the DOE Office of Science, Office of Basic Energy Sciences, under Contract No. W-31-109-ENG-38. The authors also acknowledge the support received from the GeoSoilEnviro Consortium for Advanced Radiation Studies (GSECARS) Collaborative Access Team (CAT).

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