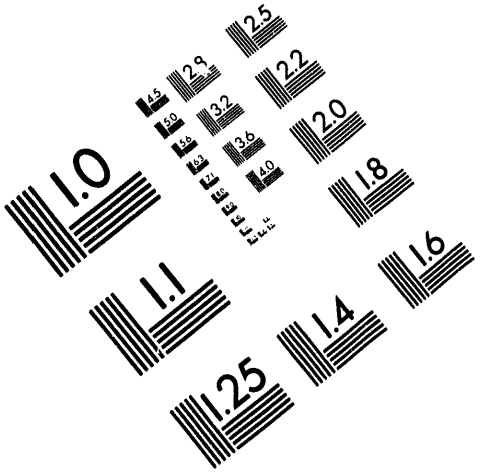

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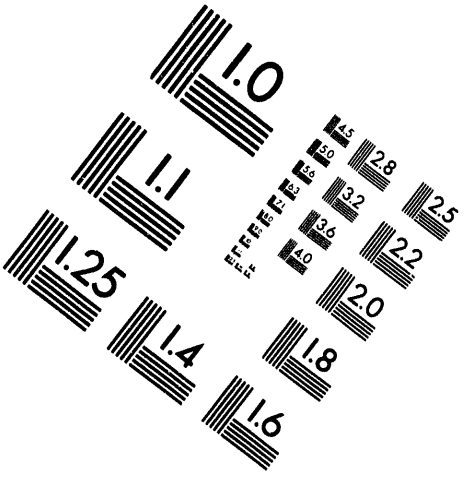

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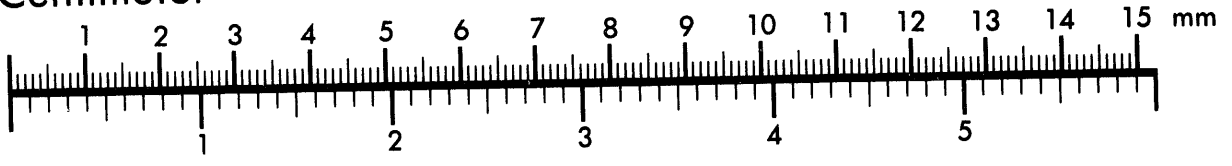

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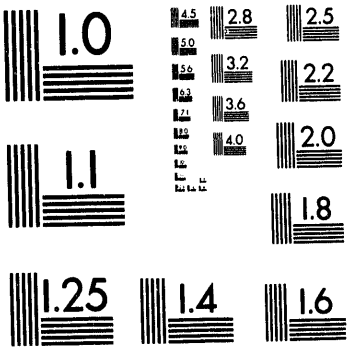

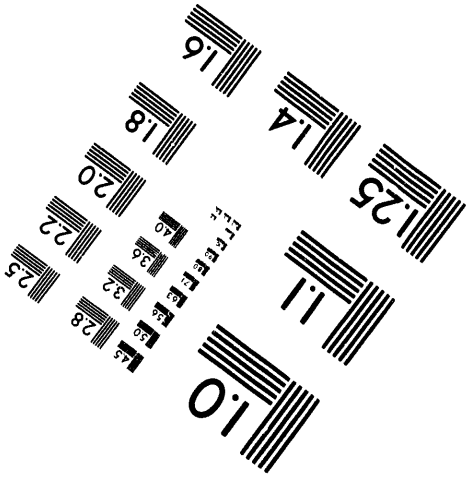

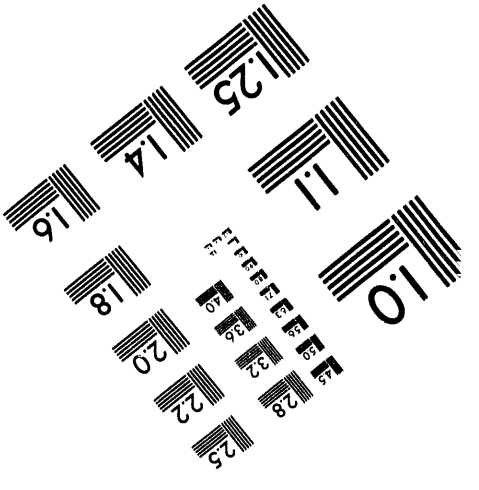

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NONDESTRUCTIVE CHARACTERIZATION OF STRUCTURAL CERAMIC COMPONENTS

W. A. Ellingson,* J. S. Steckenrider, E. A. Sivers
and J. R. Ling†
*Argonne National Laboratory, Argonne, IL USA
†Shanghai Institute of Ceramics, Chinese Academy
of Sciences, Shanghai, China

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and J. R. Ling†
*Argonne National Laboratory, Argonne, IL USA
†Shanghai Institute of Ceramics, Chinese Academy
of Sciences, Shanghai, China

ABSTRACT

Advanced structural ceramic components under development for heat–engine applications include both monolithic and continuous fiber composites (CFC). Nondestructive characterization (NDC) methods being developed differ for each material system. For monolithic materials, characterization during processing steps is important. For many CFC, only post process characterization is possible. Many different NDC systems have been designed and built. A 3D x–ray micro computed tomographic (3DXCT) imaging system has been shown to be able to map density variations to better than 3% in pressure slip cast Si_3N_4 monolithic materials. In addition, 3DXCT coupled to image processing has been shown to be able to map through–thickness fiber orientations in 2D lay–ups of 0°/45°, 0°/75°, 0°/90°, in SiC/SiC CVI CFC. Fourier optics based laser scatter systems have been shown to be able to detect surface and subsurface defects (as well as microstructural variations) in monolithic Si_3N_4 bearing balls. Infrared methods using photo–thermal excitation have been shown to be able to detect and measure thermal diffusivity differences on SiC/SiC 2D laminated CFC which have been subjected to different thermal treatments including thermal shock and oxidizing environments. These NDC methods and their applications help provide information to allow reliable usage of ceramics in advanced heat engine applications.

Introduction

Reliable and economical application of structural ceramics in advanced heat engines continues to demand: (a) improved processing, including lower cost machining, (b) higher fracture toughness materials and (c) criteria and methods for life–time prediction. Nondestructive characterization (NDC) methods which can provide data useful for process control development as well as for input to life–time prediction models become an integral part of the insertion of ceramics in these applications. Several recent developments in NDC technology for monolithic and continuous fiber ceramic matrix composites (CFC) include: three–dimensional X–ray computed tomography for mapping distributed properties such as density; elastic optical scattering for characterizing surfaces and near subsurfaces; infrared methods for measuring thermal properties, especially for CFC materials typically used at elevated temperatures.

Part A: Monolithic Ceramics

NDE for Greenbody Characterization

Monolithic structural ceramics of interest to heat–engine applications, primarily Si_3N_4 and SiC, have been under development for a number of years. However, low cost, reliable, and high volume production has usually caused process methodology to focus on slip casting or injection molding. A primary concern in slip casting (including pressure slip casting) is the measurement of density variations.

Measurement of density in pressure–slip–cast ceramic components is important for reliable performance of ceramic components and for the development of reliable production processes.[1] Correlation between density and fracture strength in different ceramic materials has been a subject of study for some time, and a nondestructive method to measure density variations would significantly enhance ceramic processing development and result in more–uniform ceramic properties. A primary reason for development of a nondestructive measurement system is the high cost and difficulty in making destructive measurements, especially on "green" or presintered specimens. X–ray computed tomography with 3–D cone–beam methods[2] offers the potential as a nondestructive, noncontact method to measure density differences in as–cast ceramic components.

Recent research at Argonne utilized a 3–D microfocus X–ray computed tomography (3DXCT) system to study density variations in pressure–slip–cast SiC(w)/Si_3N_4. The 3–D XCT system has been described elsewhere.[3] Data acquisition time for the specimens was about 1 hr for the entire 3–D data set. The reconstruction times for an entire 3–D data set are 30 min to 1 hr on a SUN SPARC 10GX.

All test specimens in this study were made with SiC whiskers and Si_3N_4 matrix. The right–circular cylinder specimens were made with a pressure–slip–casting method. The mold was plaster of paris, and dewatering was in the radial and axial directions. Two sets of specimens were produced. Set 1 consisted of specimens 25 mm in diameter and 40 mm long and contained 20, 23, 27 and 30 wt.% whiskers. Set 2 specimens were identical but only 15 mm in diameter. Table 1 lists the parameters for set 2.

Table 1. Parameters for Slip Casting

Whisker content (wt.%)	Solids content (wt.%)	Slip viscosity (cps)	Density at demolding (g/cm^3)
20	69.8	135	2.07
23	68	93	2.47
27	65	105	2.15
30	63	362	2.30

Whisker type: American Matrix, HF–Etched; Pressure level: 0.276 MPa (40 psi)

Prior to testing, the as–cast specimens were presintered at low temperature. The specimens were then examined by 3DXCT to obtain X–ray attenuation data of the apparent density variations throughout the entire volume.

To establish the reliability of the variations in X–ray attenuation (gray scales in images) relative to real density variations, destructive data were obtained at three axial locations (L/4, L/2 and 3L/4). A 2–mm–thick "slice" was removed from each of three axial locations. From each "slice," six radially spaced 2 x 2 mm squares were cut. These 2 x 2 x 2 mm cubes were then weighed and density was determined.

These destructive density data were compared to the gray scales on the XCT images. Typical results are shown in Fig. 1 for 27 wt.% whiskers for L/D ratio of 1.60. By such analysis, radial density measurements as a function of L/D ratio were made. Radial density variations for two L/D ratio billets sets, 1.6 and 2.67, showed that as expected, a larger diameter has a greater density gradient.

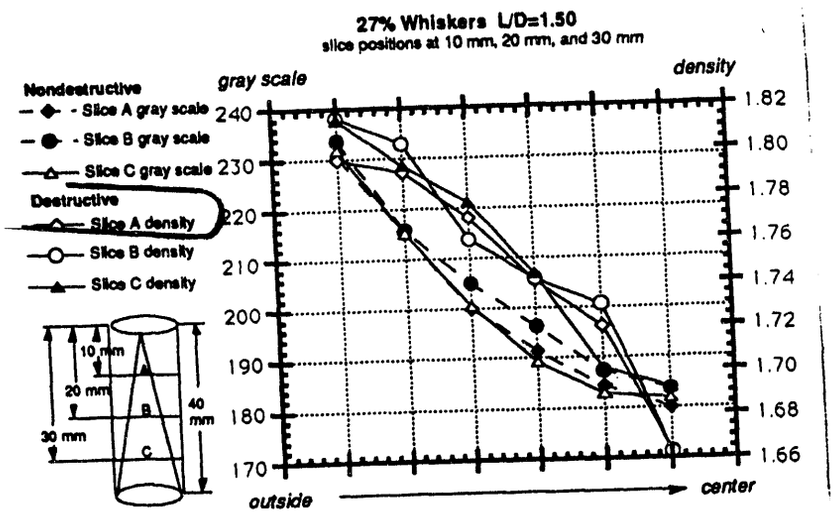

Fig. 1. Correlation of 3DXCT data with destructive analysis for 27 wt.% whiskers in 4D = 1.6 specimen.

In the case of injection molding, the distribution of organic binder materials is important and the quantification of this distribution has been explored by nuclear magnetic resonance (NMR) imaging as well as 3DXCT methods. These results have been reported elsewhere by the authors.[4]

NDE for Dense Body Characterization

Many uses of monolithic ceramics requires that some machining be conducted prior to application. Machining methodology for ceramics has been the subject of complete conferences.[5] Characterization of the surface and the near subsurface is vitally important to ceramics because of the relative sensitivity to small flaws. Recent work at Argonne[6] has explored the use of optical wave length elastic scattering to detect surface and near–surface flaws. Whereas the optical attenuation characteristics of single crystal Si_3N_4 and SiC, have been established, no data were available on optical attenuation of polycrystalline Si_3N_4 or SiC of interest to structural ceramic applications. A comparison of the optical transmission properties of single crystal Si_3N_4 vs polycrystalline Si_3N_4 has been reported.[6] The difference in attenuation among polycrystalline Si_3N_4 materials likely comes from the second phase materials used for optimizing sintered density.

An experimental optical arrangement which incorporates a low–power He–Ne laser and Fourier optic detection was established and has been discussed elsewhere.[6]. Utilizing this experimental arrangement, scanning results using polished flexure specimen bars with various defects, real and simulated, has allowed an evaluation of this method. Further application to Si_3N_4 bearing balls has

demonstrated the ability to detect subsurface microstructural variations including localized porosity. Figure 2 shows the changes in spatial frequency of the detected scatter patterns and corresponding microstructural variations which caused these scatter pattern changes. Similar results have been obtained on Si_3N_4 specimens with surface finishes up to 2 μm Ra.

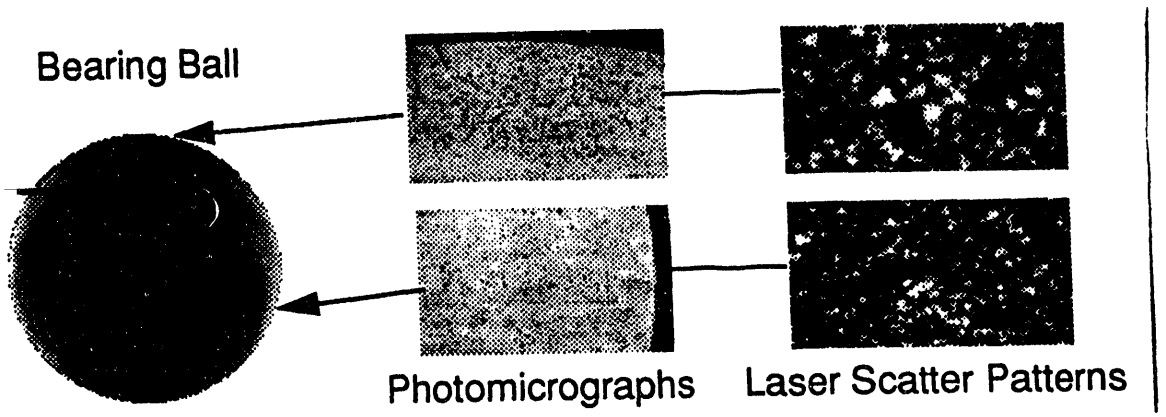

Fig. 2. Correlation between detected laser scatter patterns and microstructural variations in Si_3N_4 bearing ball.

Part B: Continuous Fiber Composite Ceramics

Continuous fiber composites (CFC), are under development to provide higher fracture toughness, and therefore less sensitivity to small flaws. Nondestructive characterization methods, as with monolithic ceramics, are being developed to provide information about process development as well as to provide information which can be used together with life–prediction models for predicting remaining component life. Many CFC process methods are under development.[7] However, generic to all CFC materials is the need to measure density variations, thermal shock damage, presence or absence of delamination and thermal properties. As opposed to monolithic ceramics, most applications of CFC materials use thinner cross–sections, e.g., <10 mm. In addition, spatially distributed density variations in any CFC process which produces a "green–body" seem likely to have relatively minor impact on flaw generation. For these reasons rather than employing 3DXCT for mapping density variations, we have examined through–transmission digital radiographic imaging methods. Initial test specimens consisted of a set of four SiC/SiC specimens made using 4 different processing times to change the final density. The 50 x 50 x 4 mm specimens had measured geometrical densities of 1.92, 2.02, 2.05 and 2.17 g/cm^3. The data were taken with the 1.92 g/cm^3 specimen always in place. Thus data were obtained using difference data via the gray scales on the resulting digital images. Thus corrections for any change in X–ray flux from the X–ray head or any change in the detector system could be made. The resulting data, normalized to the maximum gray scale detected had a correlation coefficient of 0.94. Nondestructive methods to measure thermal properties of CFC materials is also important as delaminations, oxidation at fiber/matrix interfaces and thermal shock damage will likely cause changes in thermal properties of CFC material systems. Thermal conductivity measurements require knowledge of density and thermal diffusivity. Measurement of thermal diffusivity, α, however, can be directly determined from surface

temperature versus time plots which can be obtained by use of an infrared camera/flash lamp system provided certain details are known, as

$$\alpha = \frac{L_2}{t_{1/2}}$$

where L_2 is the specimen thickness and $t_{1/2}$ is the time to reach one–half peak surface temperature.

Recent work has utilized an infrared camera, 3 KJ photo–flash, and computer based image analysis system to study thermal diffusivity of ceramic composites. A commercially available camera and flash system is coupled to a computer with a digital frame grabber board and locally written software. The system is calibrated using known diffusivity graphite of various thicknesses.

As damage to the fiber/matrix interface, e.g., via thermal shock or oxidation, could have a major impact on mechanical properties, tests are being conducted to establish the relative sensitivity of the method to thermal diffusivity changes. In one set of experiments, 10 mm x 20 mm x 4 mm 2D laminated Nextel fiber reinforced Si_3N_4 specimens, produced by chemical vapor infiltration (CVI) methods, were subjected to thermal shock by heating to 600°C and then quenching in a water bath at room temperature. After each cycle, thermal diffusivity measurements were made using the IR camera/photo–flash system. The change in measured thermal diffusivity as a function of quench cycles is shown in Fig. 3. Correlations have been established between flexure strength and quench cycles and these data correlate with measured thermal diffusivity.

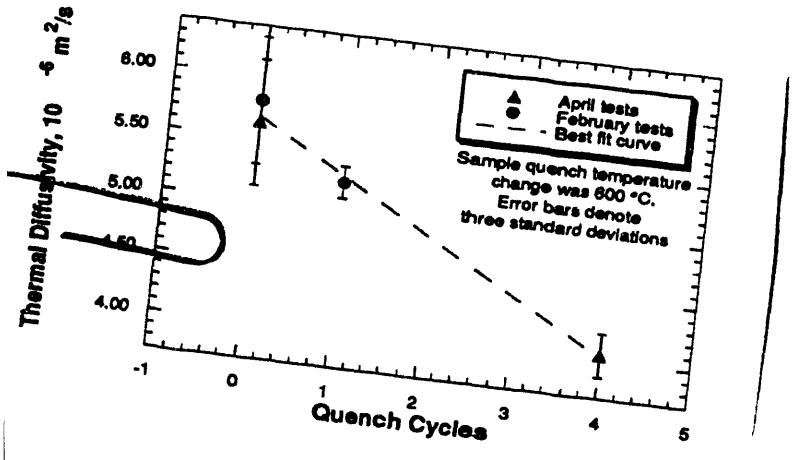

Fig. 3. Correlation between thermal shock cycles (600°C to room temperature) and measured thermal diffusivity for Al_2O_3 fiber/Si_3N_4.

Conclusions

Nondestructive characterization methods underdevelopment have been shown to be able to measure important parameters for processing as well as life prediction in both monolithic and CFC materials. Specifically, 3DXCT has been demosntrated to map and measure densities throughout green bodies, even of complex shape, to better than 2%. Elastic optical laser scattering seems likely to be able to sense and characterize surface and subsurface damage in dense monolithic materials (at depths <200

μm), if the materials have proper optical transmission characteristics.

Digital X–ray imaging seems likely, with proper corrections, to be able to map and measure density variations in reasonably thin (<10 mm) CFC materials to better than 5%. Thermal imaging methods have been demonstrated to be able to measure thermal diffusivity on CFC materials and likely can provide data about oxidation damage levels, thermal shock damage levels and can be used to perhaps measure in–situ thermal conductivity if the density values are obtained via X–ray methods.

Acknowledgments

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