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Nondestructive Evaluation Development for Process Control

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DISCLAIMER

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ABSTRACT

A joint project between Garrett Ceramic Components (GCC) of Allied Signal Aerospace Corporation and Argonne National Laboratory (ANL) is ongoing to evaluate nondestructive characterization (NDC) methods to detect and measure process-induced variations in ceramic materials. The process methods of current focus are slip-casting and injection molding and the NDC methods being evaluated are microfocus X-ray computed tomography (XCT) and nuclear magnetic resonance computed tomography (MRCT). As part of this work, SiC whisker reinforced Si3N4 (GCC's GN-10 material) has been pressure slip-cast at two casting pressures, 15 and 40 psi; and at length/diameter ratios of 1.5, 2.5 and 3.0 with whisker contents of 20, 23, 27 and 30 wt.%. Three-dimensional microfocus XCT has been used to study density variations in billets produced by different process conditions. Destructive measurement of density variation has been compared to the XCT measurements and correlations established. XCT has been shown to be able to detect <5% variations in as-cast density and these were destructively verified.

INTRODUCTION

Measurement of density in pressure-slip-cast ceramic components is important relative to reliable performance of ceramic components as well as to help in reliable process development.[1–3] Correlation between density and fracture strength for different ceramic materials has been a subject of study for some time and method is to measure density variations nondestructively would significantly impact ceramic processing development in order to achieve more uniform ceramic properties. One primary reason for development of a nondestructive measurement system is the high cost and difficulty making destructive measurements especially on "green" or pre-sintered specimens. X-ray computed tomography[4] using 3D cone beam methods[5–6] offers the potential as a nondestructive, noncontact method to measure density differences in as-cast ceramic components.

TEST FACILITIES

In this research project, a 3D microfocus X-ray computed tomography system was used to study density variations in pressure slip-cast SiC(w)/Si_3N_4. The 3D XCT system has been described elsewhere[7] and a schematic diagram is shown in Fig. 1. The data acquisition time for the specimens in this research was about 1 hour for the entire 3D data set. The reconstructive times for the entire 3D data set run from 30 minutes to 1 hour on a SUN SPARC 2/GS workstation with 64 Mega bytes of RAM.

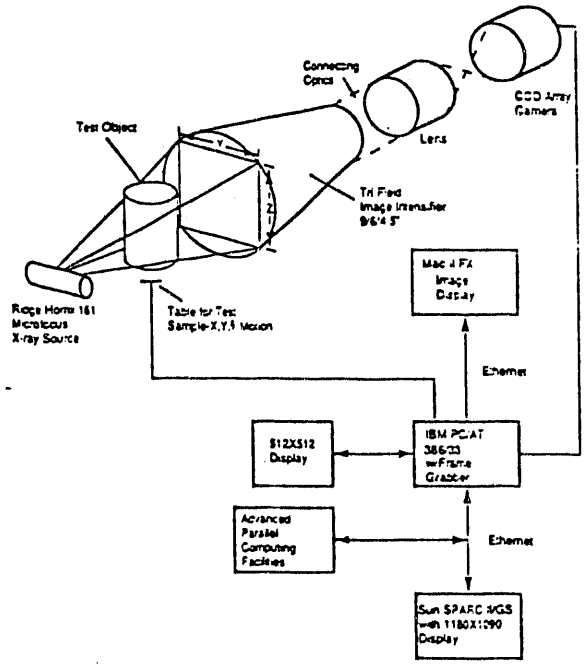

Fig. 1. Block diagram of 3D cone beam X-ray microtomography system.

TEST SPECIMENS

All test specimens used in this study were made with HF etched American Matrix or Tateho SiC whiskers and GCC's GN-10 Si_3N_4 matrix. The right circular cylinder specimens were made using a pressure slip casting method as shown schematically in Fig. 2. The mold was a plaster of paris mold and dewatering occurred in radial and axial directions. Two sets of specimens were produced. The first set consisted of two specimens 25 mm in diameter and 75 mm long and contained 20 and 30 wt.% whiskers. Each was cast at 15 psi casting pressure. The second set of specimens were also 25 mm in diameter but were 40 mm long. In this set, the casting pressure was increased to 40 psi. Table 1 lists the casting parameters and as-cast parameters.

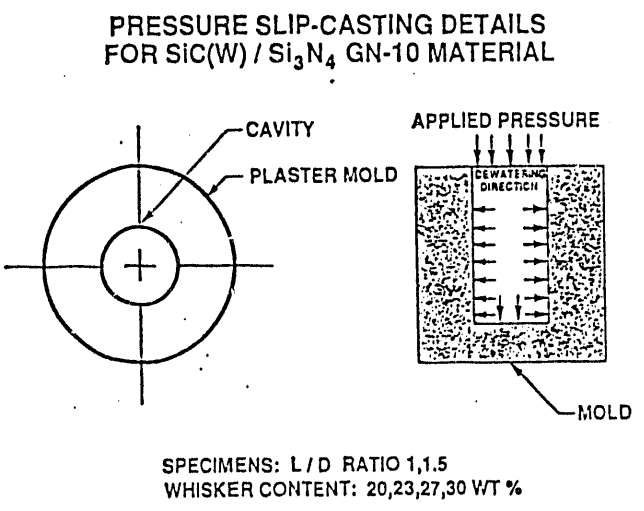

Fig. 2. Schematic diagram of pressure slip-casting.

Table 1. Casting Parameters

Billet identi-fication	Whisker content wt.%	Slip solids content wt.%	Slip viscosity cps	Bulk density at demolding
91A-013	20	69.8	135	2.07
S-910002	23	68	93	2.47
S-910362	27	65	105	2.15
S-910373	30	63	362	2.30

Mold type:	Plaster
Casting time:	50 minutes
Whisker type:	American Matrix, HF Etched
Pressure level:	40 psi

Table 2 shows a comparison between the casting parameters for sample set one and sample set two at the two whisker loadings.

Table 2. Casting Parameters for Process Development

	$SiC(w)Si_3N_4$			
Billet	T–8	91A–013	T–12	S910373
Whisker Content	20 wt.%	20 wt.%	30 wt.%	30 wt.%
Size	25mm dia 75mm long	25mm dia 40mm long	25mm dia 75mm long	25mm dia 40 mm long
L/D	3.0	1.5	3.0	1.5
Whisker	Tateho	Am. Matrix	Tateho	Am.Matrix
Slip visc.	482 cps	135 cps	274 cps	362 cps
Casting pressure	15 psi	40 psi	15 psi	40 psi
Solids content	54.6%	69.8%	53.3%	66.2%

EXPERIMENTAL PROCEDURES AND RESULTS

The as-cast specimens were either allowed to air-dry or were pre-sintered at low temperature. After air drying or pre-sintering the specimens were examined by XCT to obtain X-ray images of the apparent density variations throughout the entire volume. Relative density variations throughout the part volume can be obtained by volumetric XCT provided that proper corrections are taken. An example of the apparent difference in axial density variation between sample set number 1 with 15 psi casting pressure and set number 2 with 40 psi casting pressure for 20 wt.% whiskers is shown in Fig. 3. Figure 3 shows single axial cross-sections obtained by XCT. The lighter gray regions in the 15 psi casting pressure image is the lower density and darker gray represents higher density. In the 40 psi casting pressure image, the gray scales are reversed.

In order to establish the reliability of the variations in gray scales in the XCT images relative to real density variations, two specimens from sample set two were destructively analyzed and density measured.

The specimens were pre-sintered and destructive data were obtained at three axial location's L/4, L/2 and 3L/4. At each axial location, a 2 mm thick "slice" was cut out. On each "slice", maintaining the relative azimuthal position, six 2 mm X2mm squares were cut. These 2 mm x 2 mm x 2 mm cubes were then weighed and density determined.

These destructive density data were compared to the gray scales on the XCT images. Since data could be easily measured on the computer with XCT images, 4 azimuthal positions, 90° apart, at each axial location were measured. Figure 4 shows one example of the azimuthal variation in gray scale (relative density) measured from XCT images.

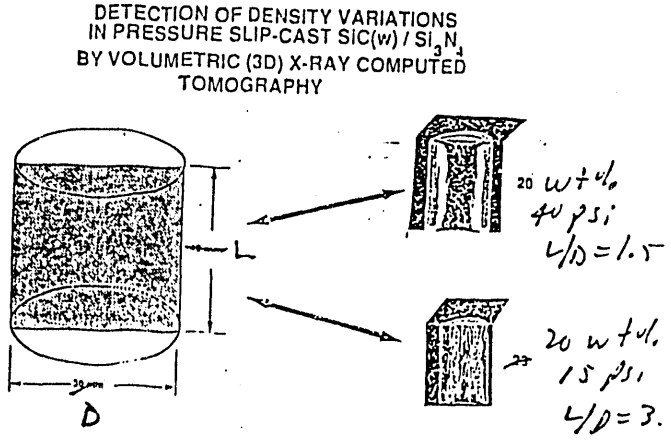

Fig. 3. Diagram and typical axial XCT images of 20 wt.% whiskers for two casting pressures: 15 and 40 psi for two L/D ratios.

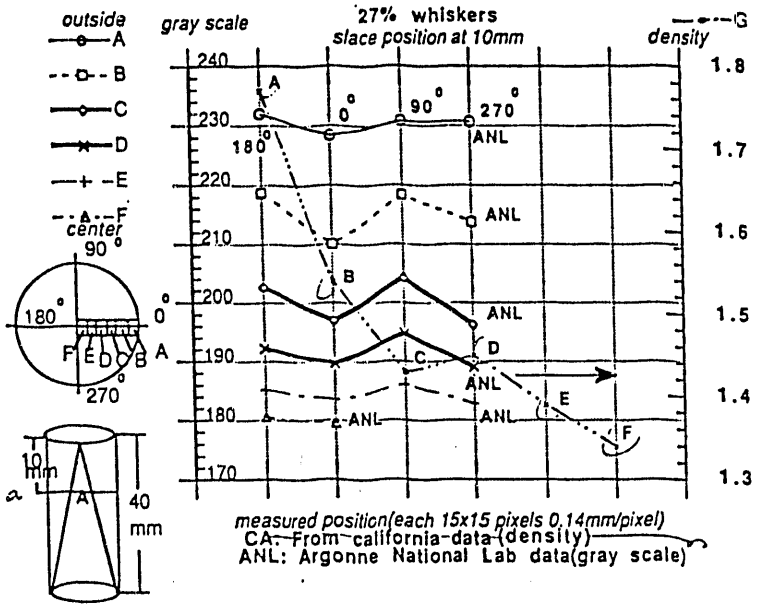

Fig. 4. Example of azimuthal variation in gray scale as detected by XCT image using 2 mm x 2 mm x 2 mm voxels. These data are for the L/4 position on the 27 wt.% whisker specimen.

The data of Fig. 1 are typical results from sample set number 2. Note that there are 2 values at the center. The two values result from a slight shift in the digital image data. The small diagram on the plot shows where the destructive data were obtained, i.e., at azimuthal position, 0°. The apparent maximum azimuthal variation is less than 5% at any radial location. Comparisons between the actual measured densities versus the XCT nondestructive data are presented in Figs. 5 and 6 for 27 and 30 wt.% whiskers respectively.

There are several ob servations to be made from the comparisons. The first observation is difference between the well correlated data for the 27 wt.% whisker data and the poor correlation on slices b and c of the 30 wt.% whisker data. The image gray scale data plotted are the averages for the four azimuthal positions as were shown in Fig. 4.

The second observation is that with the exception of slice "b" for 30 wt.% whiskers, the XCT measured density gradient, total gray scale change from center to outside, is directly proportional to the destructively measured total density gradient.

The third observation is that the total XCT gray scale change of approximately 25% from center of the specimen to the outside, agrees with the measured density change of 25%. For the 30 wt.% whisker specimen, the XCT gray scale change of 12–13%, which is the same for each axial location, is difficult to compare to the measured density because of the wide scatter. This further shows the necessity of nondestructive measurements for process variation analysis.

CONCLUSIONS

XCT image data has been shown to be able to determine in a nondestructive manner, density variations as a function of differences in pressure slip-cast process conditions including casting pressure and whisker content. Further, there is reasonable reliability in the volumetric, nondestructively measured, density variations as determined by destructive measurements. More destructive data, to account for likely measurement errors, are needed to better determine correlations. Each material condition, that is whisker content in this case, must be compared relative to that condition, unless a calibration method is established which accounts for the different material condition.

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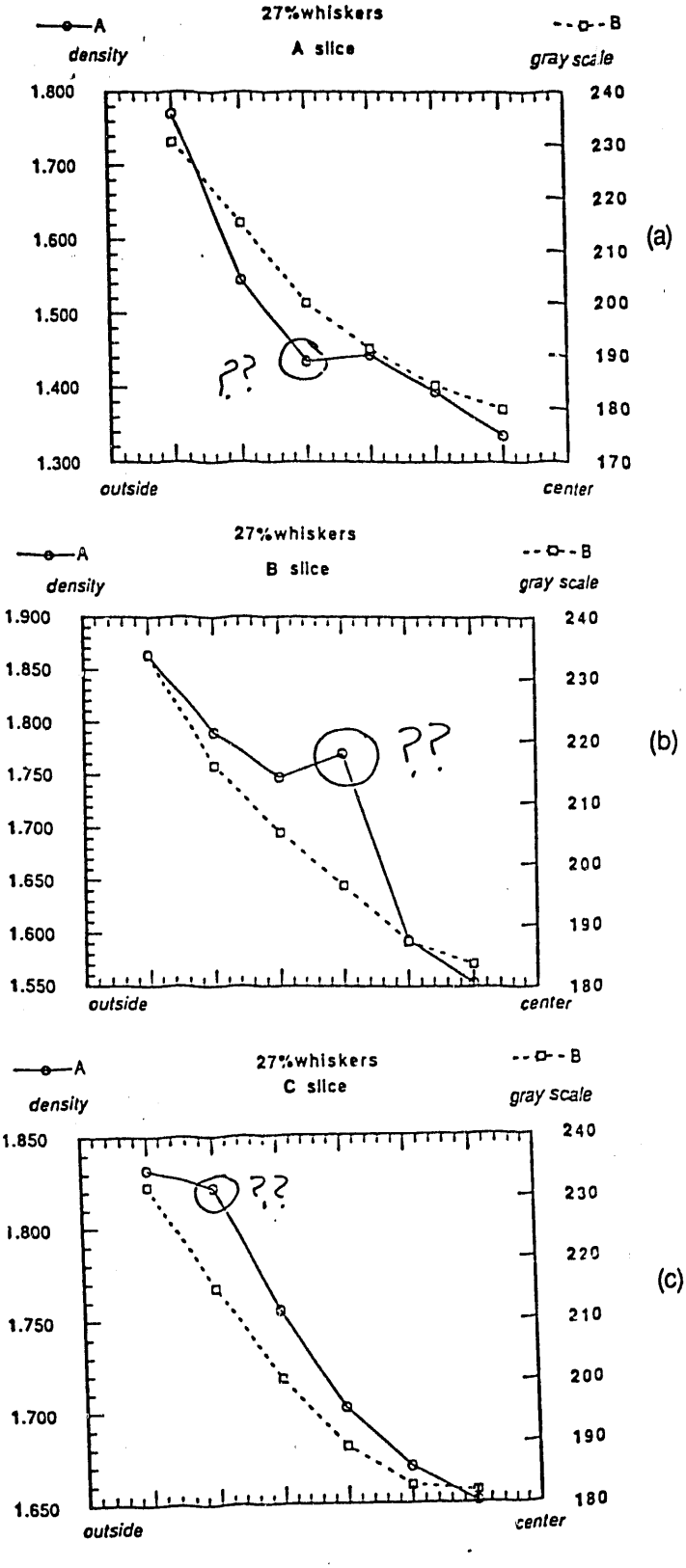

Fig. 5. Comparison between XCT gray scale image data and destructively measured density for 27 wt.% whiskers at three axial locations: (1) L/4, (b) L/2, (c) 3L/4.

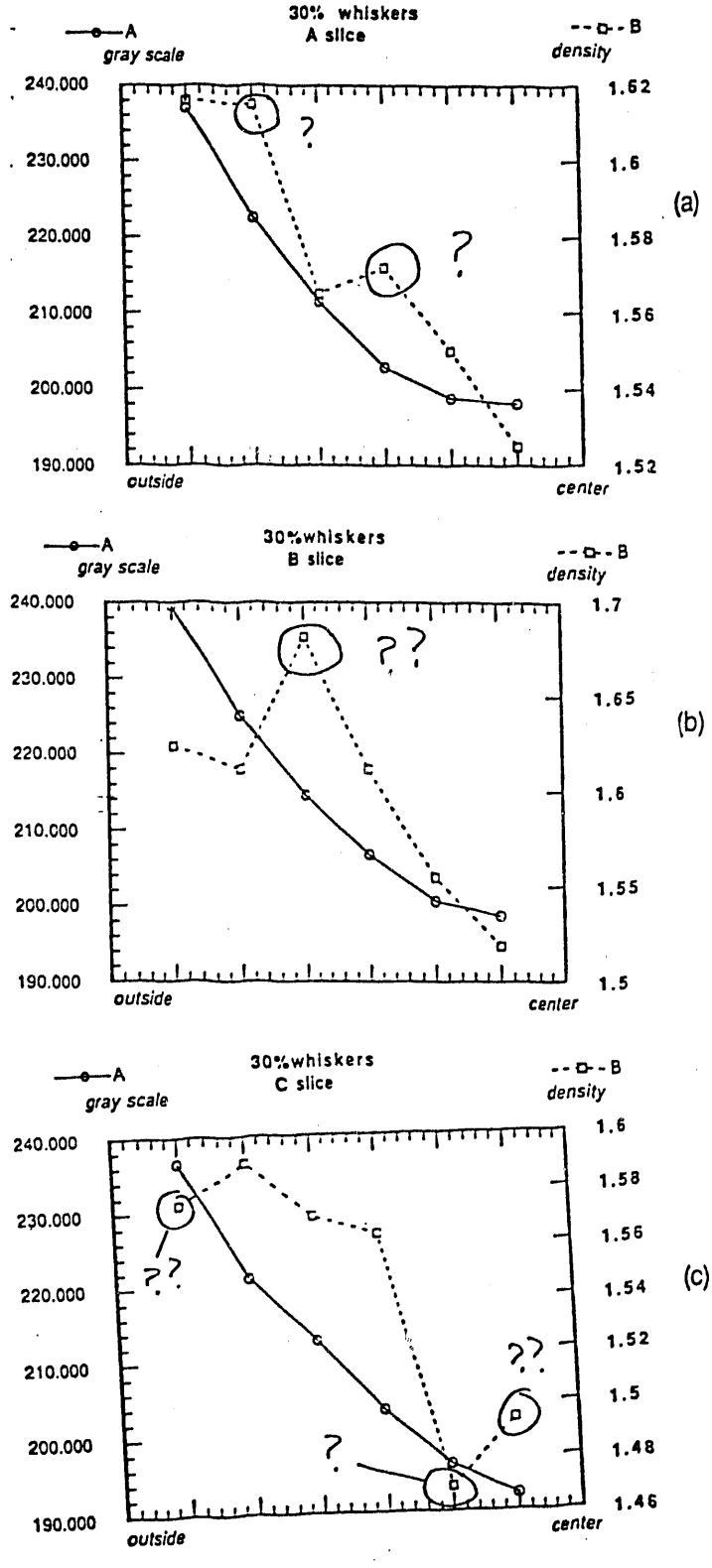

Fig. 6. Comparison between XCT gray scale image data and destructively measured density for 30 wt.% whiskers at three axial location: (a) L/4, (b) L/2, (c) 3L/4.

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