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L'ÉNERGIE ATOMIQUE DU CANADA LIMITÉE

DENSITY GRADIENTS IN CERAMIC PELLETS MEASURED BY COMPUTED TOMOGRAPHY

Mesure des gradients de densité de pastilles céramiques par tomographie informatisée

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MESURE DES GRADIENTS DE DENSITÉ DE PASTILLES CÉRAMIQUES PAR TOMOGRAPHIE INFORMATISÉE

par

B.D. Sawicka et B.J.F. Palmer*

RÉSUMÉ

Les gradients de densité sont d'une importance fondamentale dans le traitement de la céramique et la Tomographie Informatisée peut fournir des mesures précises des profils de densité d'éléments en céramique frittée et non frittée. Pour démontrer cette capacité, on a mesuré par scannographie les gradients de densité d'une pastille en céramique non frittée agglomérée à partir d'une poudre d'alumine. Pour détecter des faibles gradients de densité de ce genre, il faut une bonne reconstitution de la densité par les images du scanneur et que celles-ci ne subissent pas les effets de durcissement du faisceau. On a réalisé ceci en mesurant les images de fort contraste (faible perturbation) à l'aide d'une source isotopique (Ir-192). On a corrigé le durcissement du faisceau. On examine les images consécutives en fonction de la transmission des forces à travers la masse de poudre lors du pastillage.

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ABSTRACT

Density gradients are of fundamental importance in ceramic processing and computed tomography (CT) can provide accurate measurements of density profiles in sintered and unsintered ceramic parts. As a demonstration of this potential, the density gradients in an unsintered pellet pressed from an alumina powder were measured by CT scanning. To detect such small density gradients, the CT images must have good density resolution and be free from beam-hardening effects. This was achieved by measuring high-contrast (low-noise) images with the use of an Ir-192 isotopic source. A beam-hardening correction was applied. The resulting images are discussed relative to the transmission of forces through the powder mass during the pelletizing process.

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INTRODUCTION

Projections of world-wide markets growing steadily to tens of billions of US dollars sometime in the next decade [1-4] have led to "ceramics fever". It is generally accepted that the penetration of this potential market is limited by technology. In particular, improvements in processing and nondestructive evaluation are required before ceramics will have the reproducibility and reliability required for wide-spread use [3,4]. Computed tomography (CT) [5-7] has potential for use as a NDE tool for testing ceramic products on production lines, and is currently being used to trouble-shoot and develop ceramic processes [8-10].

Density gradients are of fundamental importance to ceramic processing. They are responsible for many of the problems with "shrinkage" cracking, warping, and deviations from near-net shape that have traditionally frustrated the ceramic engineer [11]. CT offers a method of nondestructively mapping the density gradients in green (unsintered) or sintered ceramic parts without tedious sectioning, measuring and weighing.

This paper presents the application of isotopic-source (photon) CT for the quantification of density gradients in ceramic parts. A pellet was pressed from alumina powder to create a specimen containing small density gradients. High-contrast CT images were obtained at various planes in this specimen using a CT scanner equipped with an Ir-192 isotopic source. The observed density gradients are compared to those measured earlier using destructive sectioning techniques.

MEASUREMENTS

The specimen was prepared by cold pressing Alcoa[1] A16SG alumina powder into a cylindrical pellet 14 mm in diameter and 30 mm long. A tungsten-carbide-lined die was used. Pressing was done from both ends using a pressure of 100 MPa.

A first-generation (translate/rotate, single detector) transmission gamma-ray computed tomograph [12] was used. This scanner consists of an isotopic source (50 Ci of Ir-192 for this work), a CsF detector and associated electronics, a mechanical unit that allows translation and rotation of the specimen relative to the source-detector axis, and a computerized data acquisition and control system. The gamma-ray beam is collimated equally from both the source and detector sides. It is possible to change the spatial resolution of this scanner by adjusting the beam width to match the application.

Projections were sampled twice per beam width, so that the pixel (spatial element of the image) width was half of the spatial resolution, as determined by the beam width. Images were obtained using a beam width of 0.56 mm (FWHM), a pixel width of 0.28 mm, and an in-slice thickness of 0.9 mm (FWHM), using 180 projections spaced 2 degrees apart, with 64 rays per projection. Images were reconstructed in matrices of 64-by-64 pixels using the back-projection method [6] of linearly interpolated projections with a Ramachandran-Lakshminarayanan filter. The Ir-192 source is polyenergetic and the images were corrected for beam-hardening effects. With about 2.6 x 10^9 photons contributing to each image, the theoretical statistical noise from pixel to pixel was about 0.7%, which is close to the experimental value of pixel-to-pixel density variation of 0.7 to 0.9%.

RESULTS

CT images were obtained in planes perpendicular to the symmetry axis of the pellet. The height of the image plane relative to the length of the pellet is denoted by h. For example, the image obtained 15 mm from the end of the 30 mm pellet is at the pellet mid-plane where h = 0.5. Likewise, the relative axial position within an image plane is denoted by r, such that at the center point r = 0 and at the edge r = 1.

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[1]Aluminum Company of America, 1501 Alcoa Building, Chemicals Division, Pittsburgh, PA 15219.

The images obtained are presented in Figure 1 using a linear gray scale with a relatively high threshold, in order to emphasize the small density gradients present. The scale is the same for all four images and covers the density range from 2.20 g/cm^3 (dark) to 2.35 g/cm^3 (light). Assuming a density for α-alumina of 3.97 g/cm^3, this scale ranges from 55.4 to 59.2% of theoretical density (TD). An average density of 57.1% TD was determined for this specimen by measuring and weighing it.

The pellet cross sections do not have uniform densities. Near the pellet end (h = 0.10 and 0.13), the density in the center is lower than near the edge, while for the pellet mid-plane (h = 0.50) the density is higher in the center than at the edges. From the density profiles shown in Figure 1, it can be determined that the difference in density between the center and edges is less than 2% (absolute). For the plane approximately half way between the mid-plane and the end (h = 0.27), no long-range density gradient was observed, but the densities of individual pixels scatter around an average value. This pixel-to-pixel scatter is between 0.7 and 0.9% for all four images. It represents the combined effect of the statistical counting error of the CT images with any real pixel-to-pixel variations in density that might exist in the sample.

Statistical noise in the CT images sets the practical limit for the determination of density differences between pixels. For these images, the counting statistics resulted in a statistical noise of 0.7%, which means that pixel-to-pixel density differences smaller than this value cannot be detected. The fact that the actual pixel-to-pixel scatter was as high as 0.9%, indicates that there were some real pixel-to-pixel (short-range) variations in density. If it had been necessary, these short-range variations could have been better observed by increasing the spacial resolution and improving the counting statistics. However, for observing long-range variations, matching the statistical noise to the real pixel-to-pixel scatter represents an optimum in counting efficiency.

By taking advantage of the symmetry of the sample and averaging data accordingly, the statistical errors can be reduced, and the long-range variations in density can be more clearly observed. In this case, the density gradients are axially symmetric, suggesting that pixels should be averaged in concentric annuli. Average density data calculated in this manner are provided in Figure 2. Because each annulus contains a different number of pixels, the statistical error in the averages decreases from 0.5% for the innermost annulus to below 0.1% for the outermost annulus.

DISCUSSION

The density gradients that form when powders are consolidated lead to problems, such as cracking and non-uniform shrinkage during sintering. Because this represents an important problem to the ceramic, pharmaceutical, powder metallurgy and other industries, it has been the subject of a number of studies. Previous work has relied on destructive methods for measuring density gradients in pressed compacts. For example, sectioning, machining step-by-step [13], and X-ray absorption of slices [14]. Tomography offers a way of observing density gradients accurately and non-destructively, thereby facilitating the direct observation of the effect of density gradients on further processing, such as sintering.

The results shown in Figures 1 and 2 are consistent with the previous work [13 - 18] and can be rationalized as follows. As the die pushes the powder ahead of it during compaction, the powder flows into a more dense structure. The material near the center of the plunger relocates relatively easily, whereas the outer material is somewhat confined by the die wall and consequently densifies more. This explains why the CT images obtained near the pellet end (h = 0.10 and 0.13) show a higher density near the outside. The load applied to the plungers is transferred by the powder mass to the die wall. Consequently, the powder located near the mid-plane experiences lower pressures during compaction than that near the ends. This explains why the average density of the slices near the pellet ends are higher than the slices away from the ends. Due to the influence of die-wall friction on the flow pattern of the consolidating powder, the powder near the die wall and away from the ends densifies the least. Density contours obtained by Duwez and Zwell for a pellet pressed from copper powder show all of these same features [15]. A more exact discussion of the transmission of forces through confined powder masses can be found in references 13 to 18.

SUMMARY

The application of transmission computed tomography to the quantitative evaluation of small density gradients in ceramics has been demonstrated. High-contrast CT images, with a pixel-to-pixel density error in the 0.7 to 0.9% range, were obtained using a CT scanner equipped with an Ir-192 source and corrected for beam hardening. Although this demonstration was made using alumina, the photon CT scanning technique is also applicable to ceramics containing elements with higher atomic numbers.

Density gradients spanning less than a 2% range were observed in the CT images obtained of a green (unsintered) alumina pellet. By averaging data from concentric annuli of the circular cross sections, the variation of density across the pellet radius could be measured to accuracies of 0.1% (i.e. 0.002 g/cm^3 for a ceramic with a green density of 2 g/cm^3). The density gradients measured by CT are consistent with similar measurements made earlier by others using destructive techniques involving sectioning.

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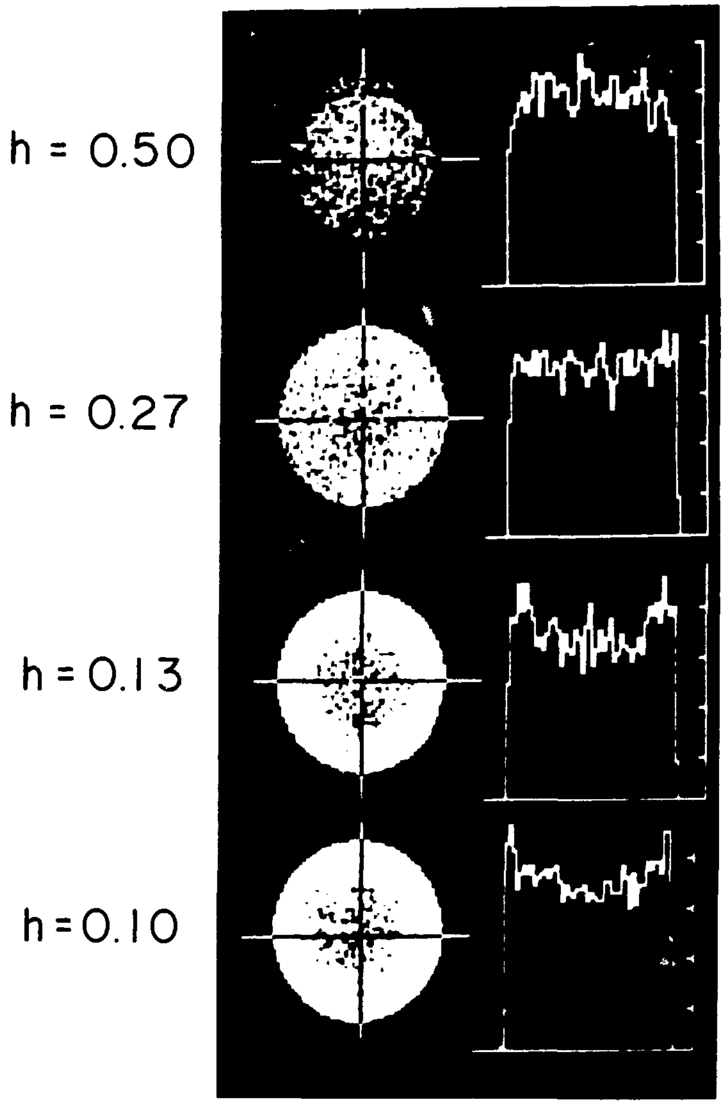

Figure 1: CT images of scans of transverse sections through a pellet pressed from alumina powder. The position of the image plane relative to the pellet length is given by h. To the right of each image is a graph that represents the density profile along the horizontal line superimposed on the image. The density scale of these graphs ranges from 90 to 100% of the maximum value observed.

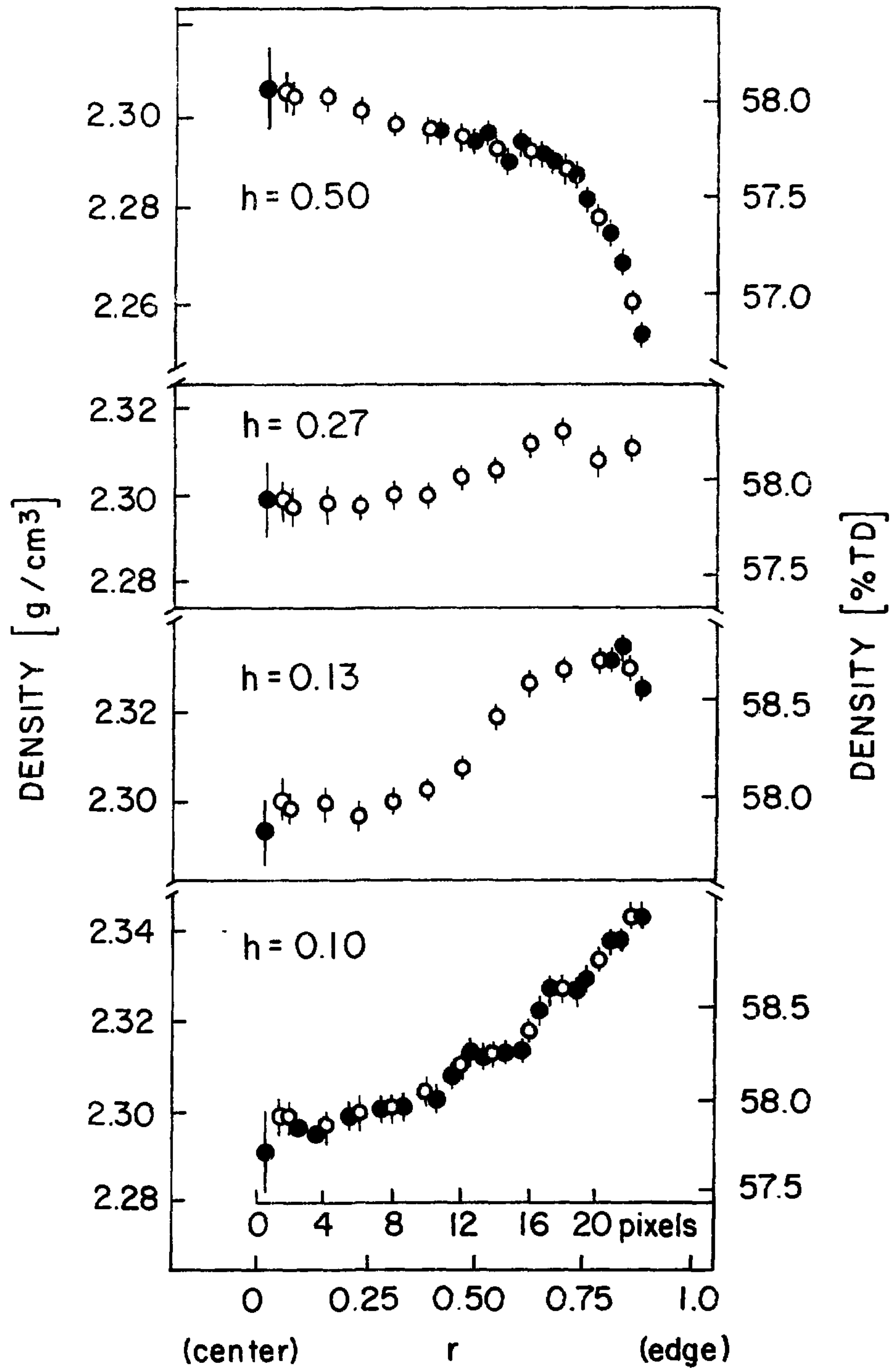

Figure 2: Density as a function of the relative distance (r) from the pellet center. The density values were obtained from the CT data by averaging pixels in concentric annuli. Averages of annuli one pixel thick (filled dots) agree well with averages obtained from annuli two pixels thick (open dots).

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