ULTRASONIC CHARACTERIZATION OF NONUNIFORM POROSITY DISTRIBUTIONS IN SiC CERAMIC

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ABSTRACT

During fabrication of monolithic ceramic silicon carbide very localized regions of high porosity can be produced. This porosity often consists of a very large density of small pores. Even at ultrasonic wavelengths considerably larger than the pore size, significant effects can be observed in ultrasonic wave propagation through these materials. These effects include attenuation, scattering, and changes in wave velocity. This paper describes the characterization of such a porosity distribution in SiC utilizing these ultrasonic techniques and their correlation with x-ray and optical microscopy measurements. Significant effects were observed due to the nonuniformity of the porosity, which resulted in enhancement of signal amplitudes greatly exceeding attenuation effects due to scattering. This unexpected result proved to be most sensitive to the boundaries of the porosity distribution and provided one of the best techniques for delineating its extent.

INTRODUCTION

Ultrasonic scattering in materials is the mainstay of nondestructive evaluation of critical microstructural features in materials, particularly metals; these techniques are currently being extended to ceramics. The primary measurement usually takes the form of backscatter or reflection of the ultrasonic wave by heterogeneities in the bulk material. These heterogeneities can be interfaces, pores, inclusions, grain boundaries, or compositional variations. Experimentally measured quantities are the backscattered ultrasonic signal or characteristics of the forward scattered wave, such as propagation velocity or attenuation. Much work has been done recently, with some success, to correlate these measurement results with known microstructural features [1,2,3,4,5]. The work described in this paper continues this correlation by comparing the results from several ultrasonic signal acquisition techniques with conventional radiography and destructive microscopic optical examination for the purpose of characterizing a known porosity distribution in a hot pressed silicon carbide plate. The results presented permit direct comparison of the information provided by each technique and its ability to either quantitatively or qualitatively characterize the distribution of porosity in the ceramic.
The sample used throughout this investigation is one half of a hot pressed silicon carbide plate (6.6 x 3.3 x 1.4 cm) intentionally processed to obtain incomplete densification (97% dense). In the unsectioned plate the pores were distributed in the shape of a disk, centrally located in the sample. The plate was sectioned transversely through the center leaving a semicircular region of porosity in the sample. Fig. 1 is a summary of microscopic optical examination of the porosity along a 31-step line scan made across the polished surface 0.5 cm from the edge formed by sectioning. Included are radiographic film density measurements made along the same line scan. The results show a unique porosity distribution in which the outer edge of the semicircular region contains an increased number of pores. The mean pore diameter remained relatively constant at about 2 μm along the entire length of the scan. On occasion, significantly larger pores of up to 8 μm were encountered. The region covered by the line scan used during the microscopic optical examination is nearly the same as that used for the ultrasonic measurements and will be referred to as the common line scan.

Backscatter radiation from the porosity distribution was recorded for this sample by windowing a slice through the material with time gating. The results of the backscatter signal energy versus position clearly delineated the overall porous region and resembled the distribution along the common line scan of Fig. 1. However, the rise in the porosity near the semicircular boundary was not well resolved.

Longitudinal velocity measurements were recorded over the sample surface by measuring time of flight from echoes off the polished surfaces. Fig. 2 shows the velocity results along the common line scan. Clearly, the effects of the porosity distribution were well resolved and the results look much like the inverse of the measured porosity and radiography data. The degree to which the velocity and radiography data are proportional is shown in Fig. 3. This is to be expected if the velocity changes are due solely to scattering off the porosity [4,5]. Following the multiple scattering formalism [6,7], the propagation of an ultrasonic wave through a density (n) of spherical scatterers of radius (r) and scattering amplitude (f) can be described by an effective wavevector (k'), which is related to the unaltered wavevector (k) for the host medium by 

\[ k' = k \left(1 + \frac{4\pi nf(0)}{k^2}\right)^{1/2} \]

This leads to:

\[ \frac{v'}{v} = \frac{1}{1 + \frac{2\pi nf(0)}{k^2}} \]

for the velocity shift with porosity. Also, the net effect of this scattering would be an attenuation of

\[ a = \frac{\gamma}{2} \]

where \( \gamma = \frac{4\pi k}{2} \text{Im}f(0) \). For the ceramic material considered here, the relevant scatterers are pores and the net fractional velocity shift with porosity becomes [6,7]:

\[ \frac{v' - v}{v} = -0.51c \]

for the values of 12.6 and 7.7 km/s longitudinal and transverse velocities appropriate for this SiC sample and (c = \( \sqrt{\frac{n^4}{3\pi r^3}} \)). Fig. 4 shows a comparison of the porosities obtained by the velocity and radiography data, assuming the linear dependence but with a coefficient of 0.77 rather than 0.51, along with the optical surface measurements. Clearly, there is reasonable agreement in the overall distribution; however, with the larger coefficient (0.77) chosen, the porosity peak at position -17mm roughly agrees in all three cases. Choosing the theoretical value (0.51) would result in better agreement for the porosity values at the edges of the sample. This difference in porosity, estimated from the velocity and radiography data, and that measured by surface optical methods could also
Fig. 1. Microscopic optical examination of the silicon carbide sample with the measured porosity and radiographic density plotted for positions along a common line scan across the surface.
Fig. 2. Longitudinal velocity recorded along the common line scan of the SiC sample.

Fig. 3. Comparison of the measured longitudinal velocities and radiographic density along the common line scan of the SiC sample.
Fig. 4. Comparison of the porosity along the common line scan of the SiC sample from the optical measurements and the velocity and radiographic density calculations.

indicate that the sample is not homogeneous throughout its thickness. In any case, the overall agreement is reasonably good, considering that the pores are not spherical, not of uniform size, and the density is very large. The small pore size maintains the product \( \frac{n_0}{k^2} < 1 \), so that even for this high porosity sample, the "weak scattering density" approximation is valid.

**REFLECTED ECHO AMPLITUDES**

The good agreement between the multiple scattering theory result and the measured velocity shifts suggests that the porosity distribution would also be evident in the attenuation values for the ultrasonic waves. However, this was not the case because strongly modulated echo decay patterns were observed for the reflections from the sample surfaces. Fig. 5 shows some of these echo decay patterns recorded at particular positions along the common line scan of the sample, designated as A - E in Fig. 2. The figure shows that normal echo decay patterns (approximately exponential) were found only for those positions on the sample where the velocity exhibited zero slope with respect to position. At all the other points, where the velocity exhibits a gradient with respect to position, the echo patterns were severely modulated. This type of modulation is similar to that expected for a sample with nonparallel sides [7], which results in phase cancellation due to unequal propagation lengths through the sample. Here, this same effect is evident except that the phase shift across the wavefront is caused by the velocity gradient in the sample. The results would be a modulation pattern following the form:

\[
(2J_1(q)/q), \quad q = 2\pi a L/v(v/dx),
\]

where \( a \) the transducer radius, \( L \) the sample thickness, \( p \) the echo number, and \( v/dx \) the velocity gradient across the wavefront. The patterns shown for positions B&D exhibit this type of behavior, although a reasonable fit of the Bessel
form to the observed pattern was found only when a larger value for the velocity gradient was assumed than was measured. This discrepancy is possibly due to the sample not being homogeneous throughout its thickness, as noted earlier. The pattern recorded for position A, where the velocity gradient is the largest, exhibits a very rapid decay, which at first suggests a very large attenuation. However, at later times in the decay pattern, an additional echo or two was always observed, which is inconsistent with this conclusion. Rather, phase cancellation is severe in this region, with the result that constructive interference can occur after many passages through the sample. The two regions of zero velocity gradient (C&E) show nearly exponential decay patterns, which is expected when the phase cancellations are not present. However, the region of largest porosity exhibits the largest echo amplitudes contrary to the expected increase in attenuation due to scattering from the large porosity at this position.

This unexpected result is likely due to a "channeling" of the radiation through refraction, similar to that observed in underwater channels, where a velocity minimum can occur with depth due to temperature gradients [8]. The net result of this velocity channel is to bend the diverging ultrasonic rays coming from the transducer, reducing or eliminating the divergence. Aerial scans [C-scans] of the echo energies show this channel exceedingly well, in comparison to the outlying porosity distribution (Fig. 6). The figure shows a comparison of the energies of the first three echoes normalized so that the observed energy at a central reference point is the same between the three plots. In this way, the normal decay of echo energy with depth (as measured at the central reference point) is masked, and only the relative shift is displayed. This brings out the channeling effect as the echo energy is seen to grow as the echo number increases. Extending this type of analysis to the fourth through sixth echo shows little further increase in relative echo amplitude and only a small broadening of the channel width.
Fig. 6. Normalized plots of the first three echo energies for the SiC sample. The energies are normalized so that all echoes at the reference point (10mm directly below the sample center) have the same value.
CONCLUSIONS

The ultrasonic examinations presented characterize the porosity distribution in this SiC sample in several ways. The velocity mapping was found to give the most quantitative and accurate results, even with the phase cancellation effects produced by the porosity nonuniformity. In addition, these results could be explained by the net effect of Rayleigh scattering from a large density of very small pores, which maintained the results in the weak scattering density regime. Attenuation mapping for porosity determination proved to be impossible due to severe echo modulation effects caused by phase cancellation across the wavefront due to the observed velocity gradients in the sample. Several unique properties of the porosity distribution were resolved, such as the gradual increase in porosity from the edges to the center and the local maximum forming a semicircular ring around the sample center. This latter effect produced a dramatic channeling of the radiation precisely at the porosity maximum, precluding any attenuation measurement for porosity determination.

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REFERENCES


