Ultrasonic imaging of silicon nitride ceramic before and after firing

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The production of ceramic components by methods which necessitate little or no post-firing treatment is of crucial economic importance to industry. Several techniques exist, including injection moulding, hot isostatic pressing (HIP) and slip casting. The latter technique is probably the simplest for both large-scale production and prototype development, because of the low cost of forming materials, and the versatility of the technique.

The processes involved in slip casting have been studied extensively [1–4]. The casting process involves the formation of a stable suspension of powder in a suitable liquid (the slip), which is then cast in an absorbent mould. Liquid is drawn from the slip by the suction pressure of the mould, leaving a cast layer which is removed once excess liquid has been poured off. The result is a ceramic in its green state, which requires drying and heat treatment to form the finished product.

One well-known method for the investigation of ceramic material properties is ultrasonic testing. This is performed conventionally in a C-scan arrangement, where an ultrasonic transducer is scanned over the area of interest, with the sample and transducer usually immersed in a liquid coupling medium. The reflected signal from the sample is analysed to form an image of internal changes in structure, either in terms of the amplitude of reflection [5] or as changes in acoustic velocity [6]. This latter technique complements conventional amplitude and defect C-scans, as ultrasonic velocities may be linked with elastic moduli and other material properties such as density. Ceramics such as silicon nitride [7] in their green state are notoriously difficult to test using conventional ultrasonic C-scanning techniques, as these require the use of water as a coupling medium which would contaminate the porous ceramic. Previous work has used methods of dry-coupling this water to a green state ceramic by protecting the sample with a vacuum sealed polymer membrane [8] or an adhesive tape [9]. Non-contact and dry-contact C-scans have been performed using wheel-contact transducers and oblique airborne waves [10], but these techniques were not used on fragile specimens, or could not use longitudinal waves. Other methods include those which are completely laser based [11], using a pulsed laser for ultrasonic generation and an interferometer for detection. However, the surface quality of a green state ceramic is often too poor to allow effective interferometry.

The present research was initiated for the specific case of silicon nitride to study the parameters affecting slip-cast ceramics, at various stages during the manufacturing cycle. This paper describes techniques whereby the dried green state ceramic could be inspected using ultrasound, without contaminating the sample. It also describes the formation of ultrasonic images of the ceramic before and after firing, so that changes in acoustic properties could be observed. A novel C-scanning system has been developed which uses longitudinal ultrasonic waves without the use of a water couplant. This uses an amorphous silicone based gel which is acoustically similar and viscous enough to retain its shape and remain non-wetting. This has an additional advantage of requiring less cumbersome scanning equipment than conventional water-based systems.

The C-scan technique can be used to find the variation in velocity through a sample, simply by measuring the time difference between multiple echoes within the sample. Alternatively, a time window can be used to detect echoes reflected from within the material, which was the method employed here. The scanning apparatus used is shown in Fig. 1. A 5 MHz immersion probe in pulse-echo configuration was connected to a Tektronix 2430A digital oscilloscope via a Panametrics pulser receiver model 5052PRX. The transducer was coupled to the ceramic sample by a 20 mm thick block of silicone gel, with a thin layer of fluid couplant between transducer and gel to permit a sliding contact during the meander scan. Waveforms captured on the oscilloscope were transferred for signal processing to an IBM PS/2 Model 30 286 PC via an IEEE/488 interface, which was also used to activate the stage motors via a Minicam motor controller.

Each scan was 40 mm by 40 mm square, with a waveform being recorded every 1 mm, resulting in 1681 separate waveforms for each scan. The entire digitized waveform was recorded each time, which allowed a variety of data processing techniques to be performed at a later date, thus reducing the scanning time to approximately 8 h.

Due to the large volume of data produced by each scan, special processing software was written to process the waveforms. The program enabled a window to be chosen at the required time in each

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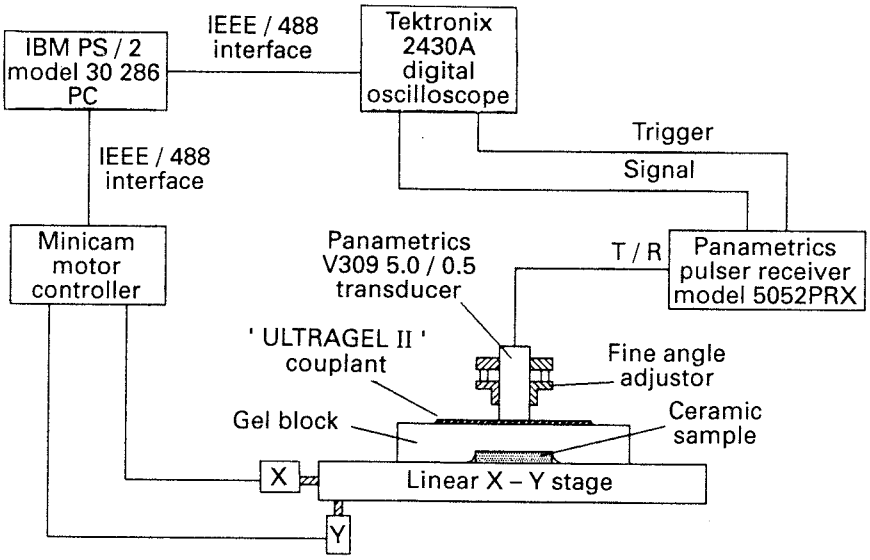

Figure 1 Schematic diagram of scanning apparatus.

waveform, and either a minimum or maximum threshold signal level specified. The first point in the window at which the signal amplitude was greater than the maximum threshold level (or less than the minimum threshold level) was recorded for each transducer position. This was done for two successive reflections within the ceramic, which could then be subtracted, producing a third grid of data which, when multiplied by the time interval used, represented the transit time within the ceramic at each point in the scan. The thickness of the sample was also measured at each point, to produce a fourth data file, which was finally divided by the transit time data to produce an image representing the local variation in velocity within the ceramic.

Three samples were made available for testing, manufactured by slip casting using 98.0 g Syalon powder in 77.0 g water with 56 wt % solid loading. The pH of each sample (numbered 1 to 3) was measured at 8.25, 8.87 and 9.10, respectively. A micrometer was used to measure the variation in thickness of each sample at 5 mm intervals and interpolated to 1 mm intervals using data processing software. It was found that all three samples appeared to be of greater thickness on one side. It is assumed that this was caused by the plaster of Paris mould not being level during the slip casting process.

After scanning with the apparatus of Fig. 1, the three green state samples were then sintered in an electric furnace at 1750 °C for 5 h, reducing their volume by approximately 25%. These fired specimens were then scanned using the same apparatus, with an identical scan size and resolution, so that direct comparisons could be made.

Signals typical of those acquired during the scans are shown in Fig. 2a and 2b for green state and fired ceramic, respectively. The first reflection R1 corresponds to the gel/ceramic interface, and the second and subsequent reflections R2, R3 correspond to echoes from the back surface of the ceramic.

Images illustrating the variation in ultrasonic longitudinal wave velocity in each of the green state samples 1, 2 and 3, respectively, are shown in Fig. 3. These were produced by dividing the thickness variation values obtained via micrometry by the time of flight values at each position. The edges of the ceramic samples in the three images are not clearly defined, partly because the edges of the samples themselves are of poor quality and this will have an effect on the method of data processing used—the threshold technique employed relies on the amplitude of the reflection in the selected window to determine the time of arrival, and at the edge of a specimen the signal amplitude will decrease due to scattering, and a reduction in the amount of sample directly beneath the transducer. The latter effect is amplified by the large size (0.5″ diameter) of the transducers used. For a homogenous sample, the velocity image should be a uniform grey, and aside from edge variations this appears to be the case for all three samples. An exception is the detection of a crack present in sample 3, starting in the top right-hand corner, and an unexpected arc-shaped abnormality in sample 2. It is suspected that this latter effect was caused by an area of the plaster of Paris mould which had non-uniform suction properties.

From the images obtained, it would appear that the technique described works well for samples of green state Si_3N_4, highlighting local variations in velocity across a cast, as well as other possible inhomogeneities that may have otherwise remained undetected by an amplitude C-scan or visual inspection.

The fired ceramic samples were measured in the same way as for the green state specimens, producing the thickness and time of flight data from which images of ultrasonic velocity could be formed. All three samples were seen to have shrunk uniformly during the sintering process, and in fact the crack present in the green state sample 3 was observed to have extended across the sample. The longitudinal velocity images for sintered samples 1, 2 and 3 are

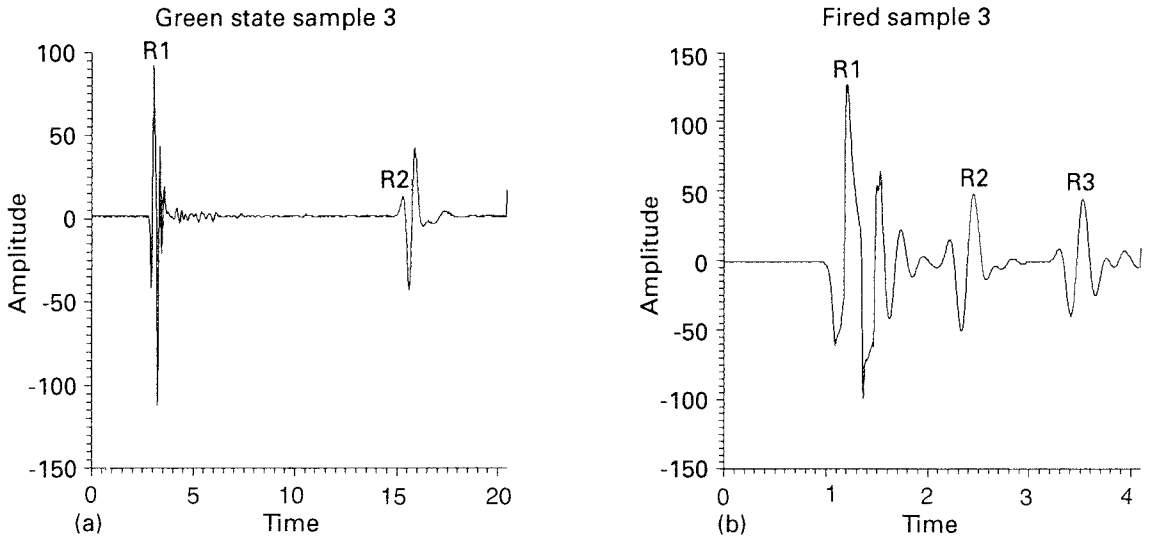

Figure 2 Signals obtained from (a) green state sample 3 and (b) fired sample 3 during scanning. Time is in microseconds.

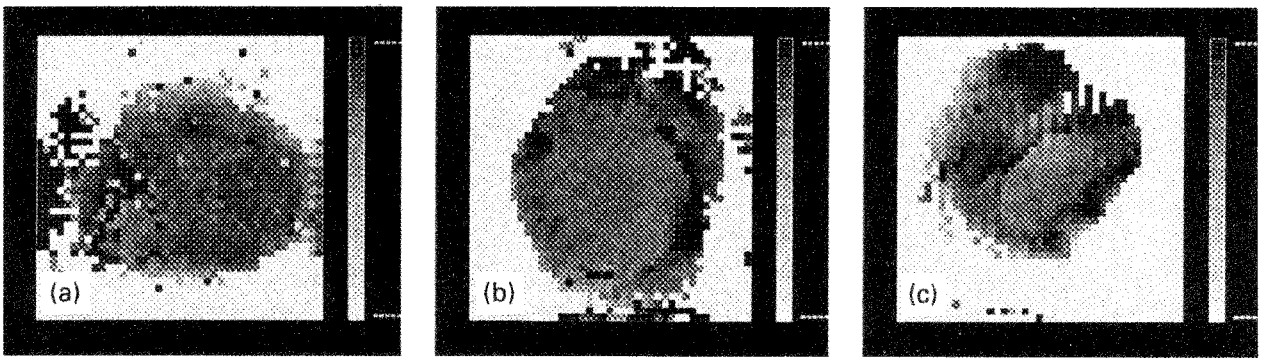

Figure 3 Variation in green state longitudinal velocity for (a) sample 1 (grey scale limits 1100–1200 ms^{-1}), (b) sample 2 (grey scale limits 1150–1250 ms^{-1}) and (c) sample 3 (grey scale limits 1200–1250 ms^{-1}).

shown in Fig. 4a to 4c, respectively. Samples 1 and 3 had abnormalities, caused by several small cracks which appeared during the firing process. Because the velocity in the fired ceramic is so fast ($\approx$10 000 ms^{-1}), the extra reflections caused by these cracks often appeared within the window limits specified for the main signal, thus producing a distorted image, particularly in the case of sample 3. It is interesting to note that the arc shaped abnormality in the image for green state sample 2 has disappeared during sintering.

The images of Figs 3 and 4 serve to demonstrate that the technique is applicable to the ceramic in both its green and sintered forms. Use of the silicone gel coupling layer allowed velocity variations to be determined across the samples, for both types of material, and demonstrated that defects within the material could be detected, without contamination of the sample by coupling fluid. It is anticipated that this technique could have wide application to the detection of anomalies within a wide range of ceramic and other materials, where a liquid couplant could cause a problem.

Acknowledgements

This work was funded via a grant from the Science and Engineering Research Council (SERC).

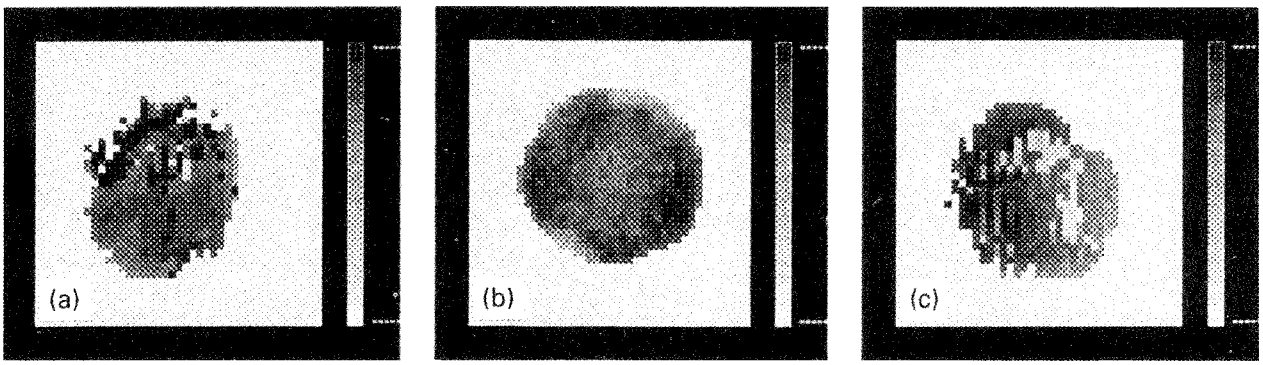

Figure 4 Images of variation in sintered longitudinal velocity for (a) sample 1 (grey scale limits 9000–11 000 ms^{-1}), (b) sample 2 (grey scale limits 10 000–11 000 ms^{-1}) and (c) sample 3 (grey scale limits 8000–11 500 ms^{-1}).

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Received 25 February
and accepted 17 May 1994