JOURNAL OF MATERIALS SCIENCE 28 (1993) 5679–5683

Real-time monitoring of cake thickness during slip casting

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A test system was developed to measure cake growth for an alumina slip using a uniaxial, planar slip, casting assembly. This technique utilizes transducers configured in a pulse-echo mode to monitor cake thickness as a function of casting time. Studies were performed to determine the effect of varying slip properties on the attenuation of ultrasound. The ultrasonic velocity was independent of solid loading, $\phi_s = 0.27$–0.43, and slip viscosity, $\eta = 85$–300 mPa s. In addition, transducers with frequencies varying from 0.5 to 5 MHz were studied to determine the best frequency for monitoring the casting kinetics. A transducer frequency of 1 MHz was found to give the best peak resolution, and thus was best for monitoring cake thickness.

1. Introduction

Slip casting can be thought of as a filtration process, in which filtration occurs through the cake and the mould. The driving force for this process is typically capillary suction by the mould, which draws the filtrate from the slip and the ceramic filter cake which is being deposited. The flow of liquid through porous media can be described by the following relationship arrived at by Darcy [1]

$$\frac{dP}{dx} = \frac{\eta}{K}\frac{dV}{dt} \tag{1}$$

where dP/dx is the pressure gradient across the filter, η is the filtrate viscosity, dV/dt is the volumetric flow rate of the filtrate, and K is the filter permeability.

Slip-casting processes have been addressed using modified versions of Darcy's relation. Adcock and McDowall [2] described casting as a filtration process whereby the particulate suspension or slip is dewatered through a filter or mould. They offered the following expression for the growth of the cake

$$L = \frac{KA}{\eta}\frac{\Delta P}{dV/dt} \tag{2}$$

where L is the cake thickness, K the cake permeability, A the cake area, ΔP the pressure differential across the cake, η the filtrate viscosity, and dV/dt the volumetric flow rate of filtrate from the cake. This derivation assumes that the specific resistance of the filter medium is negligible. In their study, monitoring of cast kinetics was accomplished by applying head pressure to a slip and dewatering it through a thin filter while measuring the filtration rate. Determination of cake thickness was made from visual observations of cake build-up. The filtration rate and cake thickness were then used to model the process.

In addition, studies have been performed by Aksay and Schilling [3] on systems in which the specific resistance of the mould was not negligible. Aksay and Schilling improved the model developed by Adcock and McDowall by incorporating the specific resistance of the mould. Tiller and Tsai [4] further modified Adcock and McDowall's model by considering the compressibility of the consolidated cake.

There have been numerous attempts to monitor cake thickness as a function of time using techniques such as gamma-ray absorption [5], nuclear magnetic resonance (NMR) [6], and ultrasound imaging [7, 8]. Deacon and Mishkin reported [5] that thickness could be resolved to within ± 0.05 mm using gamma-ray absorption. Hayashi *et al.* [6] showed that NMR could be used to measure cast-wall thickness. Measurements were made at approximately 10 min intervals using cross-sectional NMR images to determine cake thickness. A resolution was not reported. In the case of both gamma-ray transmission and NMR imaging the prohibitive cost of the detection system has limited its use.

The study made by Gesing *et al.* [7] on ultrasound imaging showed that this is an effective method for measuring thickness while an A-16 slip-cast cake formed. However, this work did not examine different frequency transducers or a range of slip properties.

The work presented here expands on the work by Gesing *et al.* The effect of frequency on the attenuation of ultrasound by particles in the slip was studied to determine a frequency at which a maximum signal-to-noise ratio and peak resolution could be obtained. In addition, the casting behaviour of alumina slips having different rheological properties was examined.

2. Experimental procedure

2.1. Apparatus for monitoring cake thickness

Cake thickness measurements were conducted on an aqueous alumina slip which had an average primary particle size of 0.6 μm as determined by an X-ray sedigraph (Micrometritics, Norcross, GA). The transducer frequency was varied from 0.5 to 5 MHz.

The test assembly for monitoring cake thickness is shown in Fig. 1. It consists of an adjustable mechanical fixture in which a longitudinal-wave piezoelectric transducer is supported. The signal from the transducer is sent to a digital data-acquisition system (Ultran, Incorporated). Initially, a 1 MHz transducer was selected based on a series of trials that matched the frequency of the transducer to the acoustical impedance of the slip. The use of lower- and higher-frequency transducers will be discussed later. In the configuration shown in Fig. 1, the transducer is fixed within the mould cavity and focused downward onto the mould surface. Casting is uniaxial with dewatering taking place only at the mould surface. When the slip is poured into the mould, three distinct pulses become apparent (see Fig. 2). The pulse on the left is caused by the acoustic impedance mismatch between the slip and the cake. The acoustic impedance is given by the relation

$$Z = \rho V \tag{3}$$

where Z is the acoustic impedance, ρ is the medium density, and V is the velocity of sound in the medium. As the cake grows, this pulse moves steadily to the left, indicating that the time required for the sound wave to reflect off the growing cake is decreasing. The other pulse shown in Fig. 2 is caused by the acoustic impedance mismatch between the cake and the mould. This pulse position essentially remains constant, but it does move slightly to the left because the velocity of ultrasound in the growing cake is slightly higher than the velocity in the slip.

By first measuring the velocity of sound in the slip and then monitoring the time of flight (TOF) of the pulse from the slip–cake interface, the cake thickness can be measured directly. The TOF is the time it takes for an ultrasonic pulse to leave the transducer, reflect off the interface, and return to the transducer. The distance to the cake surface, X_c, is defined by the following algorithm:

$$X_c = \tfrac{1}{2} v_{slip}(\mathrm{TOF}) \tag{4}$$

The cake thickness is determined by monitoring the TOF at time zero and any subsequent time, t. The thickness of the cake, $L(t)$, is then defined as

$$L(t) = \tfrac{1}{2} v_{slip}(\mathrm{TOF}(0) - \mathrm{TOF}(t)) \tag{5}$$

This algorithm for determining cake thickness is different than that used by Gesing *et al.* The configuration used by Gesing requires that either the exact position of the transducer or the velocity of sound through the cake be known, while this configuration requires only that the velocity of ultrasound in the slip (which can be easily measured) be known. In addition, the transducer position does not have to be known. Although there is some uncertainty associated with this simplified method because some finite casting is taking place while the slip is filling to the transducer height, this was shown to be negligible. To verify this, the TOF of the first cake pulse was measured and found to be 27.063 μs. Then the TOF of the mould was measured as soon as there was a discernible difference between the cake pulse and the mould pulse. After 90 s, it was found that the TOF of the mould pulse was 26.875 μs, approximately 0.2 μs less than the first cake pulse. The lower value was due to the greater ultrasonic velocity in the growing cake than in the slip. If the TOF of the mould pulse had been the same as or greater than the TOF of the first cake pulse, the uncertainty associated with the configuration of this apparatus would have presented a problem.

As stated earlier, the use of this detection system requires prior knowledge of certain key parameters such as the ultrasonic velocity through the slip. The following section describes a series of tests performed on an alumina slip which was prepared in such a way as to vary the viscosity and to vary the specific gravity. The tests were conducted to determine the effect of these variables on the propagation velocity of the ultrasound wave.

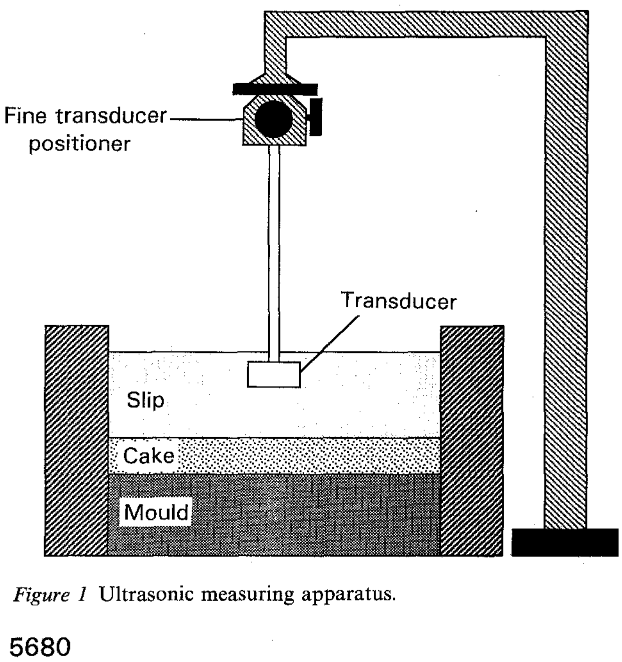

Figure 1 Ultrasonic measuring apparatus.

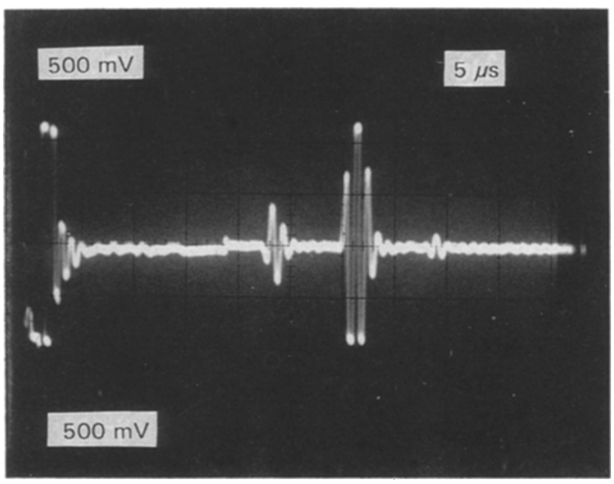

Figure 2 Oscilloscope photograph of signals generated during casting from a 1 MHz transducer submerged in an A-16SG alumina slip. The pulse on the right is from the acoustic impedance mismatch between the cake and mould. The pulse immediately to the left is from the acoustic impedance mismatch between the slip and cake. The pulse on the far right is an initial pulse from the transducer.

2.2. Velocity of sound in alumina slips

Slips were prepared by dispersing the A-16SG alumina in distilled, deionized water using tetrasodium pyrophosphate (TSPP). Each slip was ball milled for 24 h. The specific gravity was adjusted by varying the water content. Measurements of ultrasound velocities through the slips were made on samples which varied from 27 to 43 vol % solids.

To measure the velocity of the ultrasound propagating through the alumina slip, a 1 MHz transducer was set at a height between 10 and 20 mm above the bottom of a flat-bottomed container. The transducer was then manually levelled and positioned perpendicular to the bottom of the container. Water was poured into the container until the transducer face was covered. Since the velocity of ultrasound through water is known to be 1500 m s^{-1}, the distance between the transducer and the bottom of the container could be found by measuring the TOF. The container was then filled with the slip, with the transducer set at the same height. Since the slip contained alumina particles that attenuated the ultrasound signal, the signal had to be adjusted to closely approximate the signal generated in water. In this way, any variations in the TOF of the reflected ultrasound pulse due to pulse shape were minimized. An average velocity of 1330 ± 18 m s^{-1} was found for A-16 alumina slips using a 1 MHz transducer.

Kupperman and Karplus reported that the velocity of ultrasound increases with increasing density and remains constant in relation to the degree of aggregation for green ceramics [9]. In this study, however, it was found that the variation of velocity with specific gravity was minimal (Fig. 3). However, the minimal variation of ultrasound velocity with the degree of aggregation found for the alumina slip (Fig. 4) was in agreement with the studies of ultrasound in green ceramics by Kupperman and Karplus. The degree of slip aggregation or flocculation was determined by Brookfield viscosity measurements (Brookfield Engineering, Stoughton, MA), in which a higher viscosity corresponded to a higher degree of aggregation.

2.3. Frequency study

In addition to a frequency of 1 MHz, frequencies of 0.5, 2, and 5 MHz were evaluated. The corresponding signals are shown in Figs 5, 6, and 7, respectively. It was found that the signal-to-noise ratio at the 5 MHz transducer was too low to allow consistent monitoring of the cake thickness. Assuming the velocity of the ultrasound through the A-16 slip to be 1330 m s^{-1}, the calculated wavelength at the 5 MHz transducer was 266 μm.

It is probable that the larger flocs or agglomerates present in the slip act similarly to aggregates in green ceramics, severely attenuating the ultrasonic waves and making it difficult to use a frequency of 5 MHz for cake-thickness measurements [9]. Gesing *et al.* used a 5 MHz transducer for their study. In that study it was stated that the transducer must be positioned precisely in order to obtain a pulse of sufficient magnitude to make cake-thickness measurements. At a frequency of 1 MHz, precise positioning was not necessary to obtain a strong pulse.

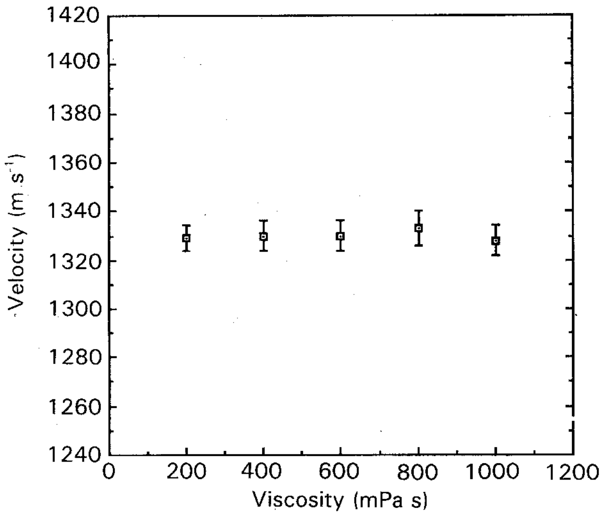

Figure 4 Velocity of ultrasound as a function of slip viscosity.

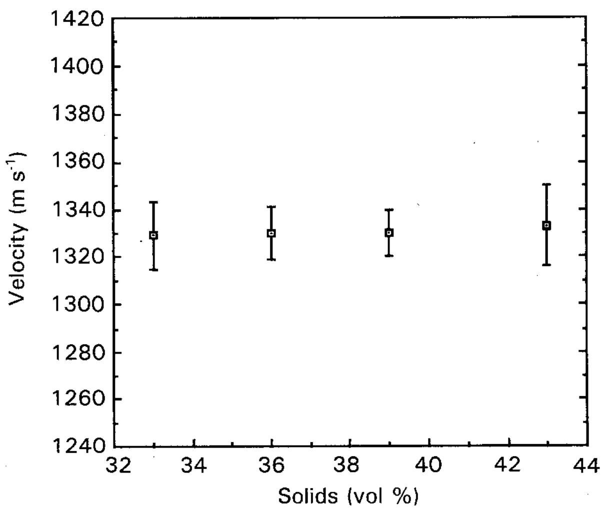

Figure 3 Velocity of ultrasound as a function of slip solid loading.

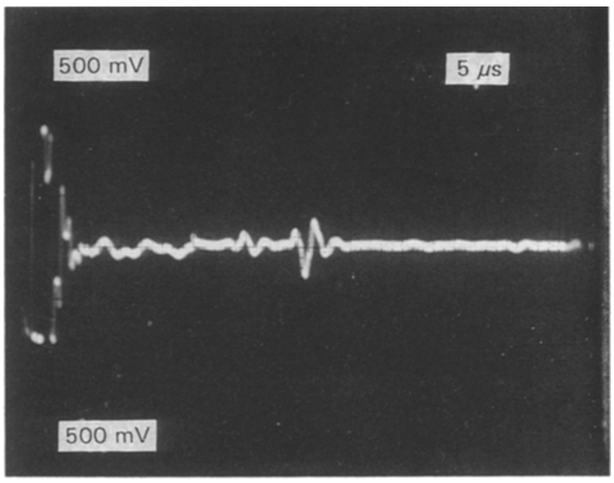

Figure 5 Oscilloscope photograph of signals generated during casting from a 0.5 MHz transducer submerged in an A-16SG alumina slip.

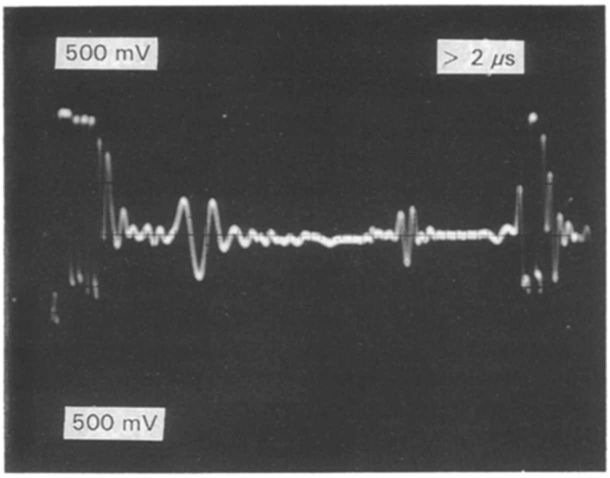

Figure 6 Oscilloscope photograph of signals generated during casting from a 2 MHz transducer submerged in an A-16SG alumina slip.

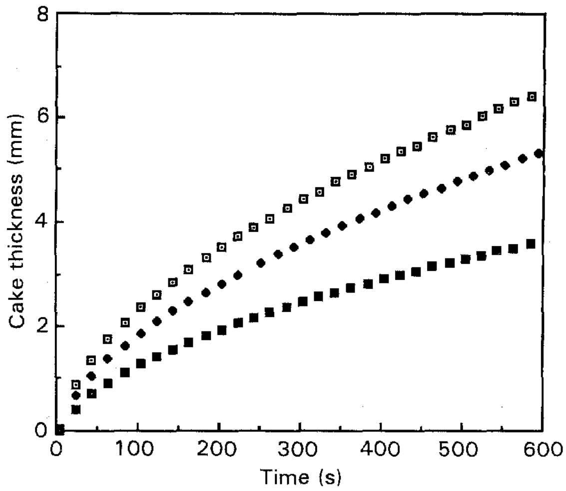

Figure 8 Casting rate of A-16SG alumina as a function of viscosity: (◘) 320 mPa s, (◆) 200 mPa s, and (■) 80 mPa s. All slips are 36 vol%.

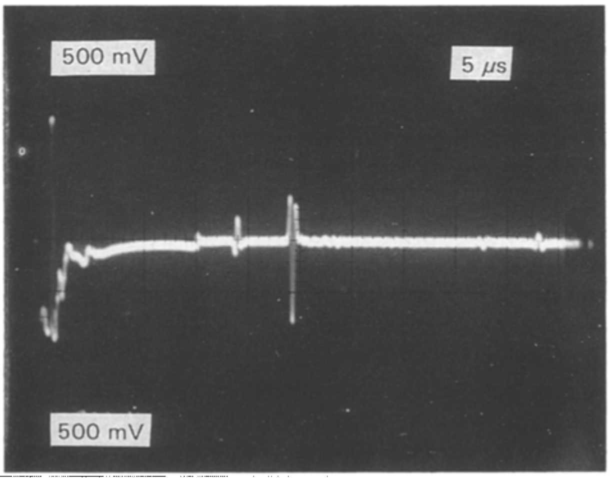

Figure 7 Oscilloscope photograph of signals generated during casting from a 5 MHz transducer submerged in an A-16SG alumina slip.

A frequency 0.5 MHz was also studied to determine its effectiveness for monitoring cake thickness. Minimal scattering by alumina flocs was expected at this frequency since the generated wavelength was expected to be relatively long compared to frequencies of 1 and 5 MHz. However, as seen from Fig. 6, it was found that the signal-to-noise ratio of the pulses was smaller at 0.5 MHz than at 1 MHz. In addition, the values obtained for cake thickness were found to be quite variable. It is possible that the generated wavelength at 0.5 MHz was not sufficiently scattered to allow sensitive cake-thickness measurements [9].

A frequency of 2 MHz was also studied, with the pulses obtained at this frequency shown in Fig. 7. It was found that the pulses obtained at 2 MHz were somewhat stronger than those at 1 MHz. The velocity of ultrasound through the alumina slip at 2 MHz was found to be identical to that measured at 1 MHz. Although a frequency of 2 MHz can be used to measure cake thickness for alumina-casting systems, 1 MHz was used primarily because the signal generated at a frequency of 1 MHz had less noise.

2.4. Measurement of the casting rate

The precision of the ultrasound apparatus was evaluated by casting a 36 vol % A-16 slip which was deflocculated to a viscosity of 130 mPa s. Five casting runs were made and the cake thickness after 10 min was determined. Five new 80-consistency plaster moulds were used for the evaluation. Consistency is defined by the plaster-to-water ratio used in preparing the mould. The typical convention states consistency to equal the parts by weight of water per 100 parts by weight of plaster. The moulds were oven dried at 40 °C for seven days. After the five casts, an average cake thickness of 3.69 mm with a standard deviation of $\pm$ 0.12 mm was found. This resolution compares well to the $\pm$ 0.05 mm found by using gamma-ray absorption. The $\pm$ 0.12 mm variation was probably due to a combination of the ultrasonic apparatus and variations associated with the casting process itself. However, the percentage of the variation due to each of these factors could not be determined from the data.

Cake thickness measurements were performed for slips with a range of properties. Fig. 8 shows plots of cake thickness versus time for 36 vol % alumina slips at several viscosities. It can be seen that as the viscosity of the slip increases, so does the cake thickness. This is as expected, since an increase in viscosity is indicative of decreasing interparticle repulsion and increased floc formation. Slips with small interparticle repulsion will cast into cakes having a low packing density [10]. Cakes with a lower packing density, or conversely a higher void volume, allow the water to filter through the cake more rapidly, thereby increasing the casting rate [4, 11, 12].

Alumina slips of different specific gravities also were studied. Fig. 9 shows the casting rate of four A-16 slips at different specific gravities with the same viscosity. It can be seen from Fig. 9 that when the viscosity is held constant, slips with the lowest specific gravity cast the fastest. This is explained by the fact that the particles in the lower-specific-gravity slips must be more flocculated for their viscosities to be equal to the viscosities of higher-specific-gravity slips. As indicated

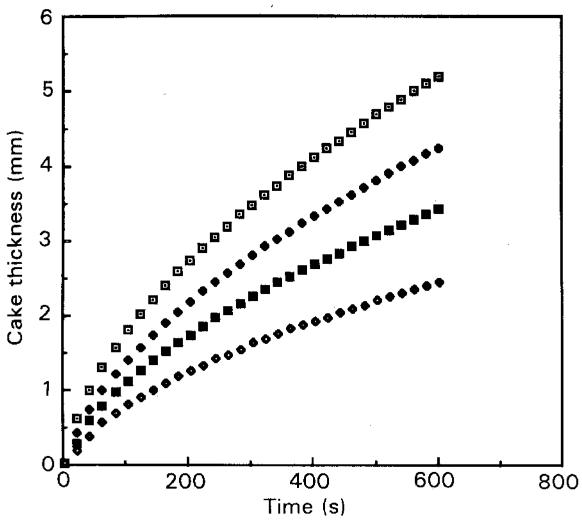

Figure 9 Casting rate of A-16SG alumina as a function of solid loading: (◻) 33 v/o, (◆) 36 v/o, (■) 39 v/o, and (◆) 43 v/o. All slips were deflocculated to 170 mPa s.

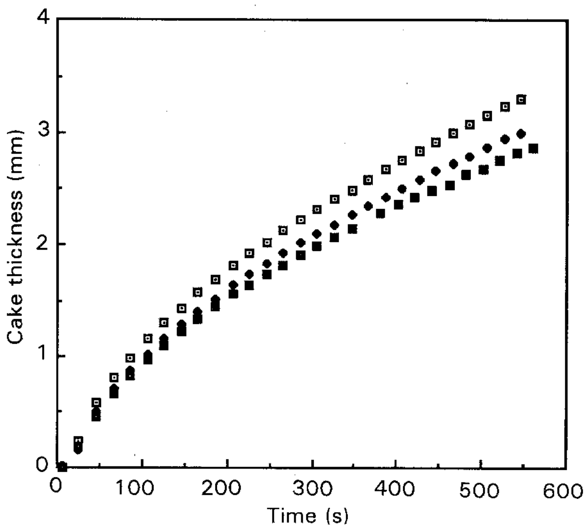

Figure 10 Effect of mould moisture on cake thickness: (◻) dry, (◆) 9.6% H_2O, (■) 19.5% H_2O.

earlier, a more flocculated slip is expected to cast into a cake which has a higher void volume, thereby allowing a higher filtration rate.

The casting rate as a function of mould moisture content also was examined; the results are shown in Fig. 10. It can be seen that the casting rate decreased as a function of mould moisture content. These results may be explained by considering the presence of water in the plaster capillaries. As the water content in the capillaries increases, the absorption rate of the mould decreases [12]. The casting rate therefore decreases according to the amount of free water present.

3. Conclusion

An ultrasonic technique for measuring the casting kinetics of A-16SG alumina slips was described. It was found that the variation of ultrasonic velocity as a function of solid loading and viscosity was not significant. A transducer frequency of 1 MHz was found to be the optimum for monitoring the casting kinetics of A-16SG alumina slips. The technique was used to monitor the casting rate as a function of slip solid loading and viscosity as well as mould moisture content. It was shown that by maximizing the ultrasonic signal with respect to resolution and intensity, an accurate and precise measurement of the casting rate can be obtained. The practical consequences of this are faster determination of casting rates for experimental purposes and improved quality control. Also, transducers can be used during drain casting to determine when drainage should take place, thereby maintaining close tolerances in the finished piece.

Acknowledgements

The authors would like to acknowledge funding by the Ceramic Casting Technology Program in the Center for Ceramic Research at Rutgers, The State University of New Jersey, which is partially funded by the New Jersey Commission on Science and Technology. The technical assistance of Arun Mahlev (Ultran, Incorporated), Dale Niesz, and Joseph Keller are also acknowledged.

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Received 28 January
and accepted 20 November 1992