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Thermally Enhanced Slip Casting of Alumina Ceramics

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Abstract: Slip casting is a process which is very suitable for the production of large components and thin-walled bodies of complex shape but which is also time and energy intensive. One method of increasing the casting rate is by heating the slip. This has been examined via the use of both microwave and convectional heat energy for 72 wt% nitric acid dispersed aqueous alumina slips. It has been found to provide an increase in the casting rate over ambient temperature processing, with a gain of 55% when using microwave energy and 20% using a convection oven at 45°C. The enhanced rate is apparently achieved via two mechanisms. At temperatures less than approximately 40°C it is probably due to a reduction in the slip viscosity enabling easier water movement through the cast layer and mould. At higher temperatures there is a change in the dispersion state of the slip. It appears that as the slip temperature increases above ambient the slip becomes increasingly flocculated and that above about 45°C this influences the casting rate. By 65°C the slip is fully flocculated. A further enhancement in the casting rate is achieved with microwave energy over convectional heat energy. This is attributed to the rate of movement of water *through* the mould being increased more than the rate of water uptake *by* the mould. This in turn is believed to be due to the plaster of paris being essentially transparent to microwave radiation so that the only significant heating that occurs in the mould is due to the presence of the water. Since the water in the mould cannot move around as freely as it can in the slip, local temperature gradients can rapidly develop. This causes the water front within the mould to move faster than with convectional heating. No evidence has been found that this accelerated rate has any detrimental effect on the cast body produced. © 1998 Elsevier Science Limited and Techna S.r.l.

1 INTRODUCTION

Slip casting is a process used to consolidate ceramic powders into sophisticated shapes with high green densities. Currently it is used mainly for the production of large components or for those having thin walls or a shell of complicated contours where the costs involved preclude the use of other techniques such as die pressing or isostatic pressing.[1] It is also used for short production run items or development trials due to the relative inexpense of the mould material.[2] The main disadvantage of the process is that it is time intensive with the speed of casting ranging from hours to days for large objects. Hence technologies are required which can increase the casting rate without introducing heterogenities into the component produced.

Methods of accelerating the colloidal filtration process have been sought by a number of researchers, many of them with a significant degree of success.[3–10] The basis behind most of the methods is manipulation of the parameters which control casting; these can be expressed generically by the equation:

$$L^2 \propto \frac{P.t}{\eta}$$

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where L is the thickness of the body, P the differential pressure across the system, t the casting time and η the slip viscosity. That is, since the rate of formation of the cast body is proportional to the differential pressure and inversely proportional to the viscosity of the slip a change in either of these parameters will affect casting rate. Research by a number of authors[4,7,11] has therefore shown that the major factors that affect the casting rate are the application of a pressure to the slip (or vacuum to the mould); the solid content of the slip; the resistance to fluid flow of both cast and mould (these are primarily controlled by the porosity of the cast layer and mould); and the viscosity of the slip.

Traditionally the solid content of the slip is kept as high as is practically possible whilst ensuring that the viscosity is suitable for casting to occur satisfactorily. This maximises the casting rate by minimising the amount of water to be absorbed by the mould and reduces shrinkage of the body on drying. The permeability of the cast can be controlled by the state of dispersion of the suspension and there have been studies on casting using all types of slips. Tsao and Haber[12] studied dispersed and partially flocculated suspensions of alumina whilst Chang *et al.*[13] looked at filter casting of dispersed, coagulated and fully flocculated alumina suspensions. The studies found that the relative densities of the dispersed, partially flocculated and coagulated slips were similar whilst the flocculated slip resulted in a much lower green density, but a much faster casting rate. These packing results were also confirmed by Velamakanni and Lange[14] with alumina. However they also revealed that the permeability of the casts prepared from coagulated slips (achieved via salt additions) were greater than those cast from dispersed slips. Although the permeability increased with increasing salt additions, they never reached the level of porosity achieved with flocculated slips. Although the use of partially flocculated or coagulated slips decreases the casting time by increasing the permeability of the body to fluid flow, it has been shown that fully deflocculated slips can produce bodies with superior green microstructures.[10] This is of importance if high quality and reliable ceramic components are required.

The state of dispersion of the slip and its viscosity can also be affected by the application of heat to the slip. Herrman and Cutler[15] investigated the slip casting of alumina using a convection oven and determined that although there was a casting rate increase as expected, the rate achieved was out of proportion with purely viscosity effects. They attributed the extra increase to a change in the dispersion state of the slip. Further investigations by Gastic and Kostic[16] on silica slips indicated that while elevated temperatures produced an increase in casting rate it resulted in a decrease in the cast density, a result confirmed by Binner[17] for alumina slips. However, recent research[18] contradicts these results and indicates that under the right conditions elevated temperature casting can produce a body with a similar green density to that produced at ambient temperature but at a faster casting rate.

This paper discusses the effect that elevated temperature has on the casting rate and the cast body produced when slip casting is carried out using electrostatically dispersed alumina slips using both a microwave and convection oven.

2 EXPERIMENTAL

2.1 Slip preparation and characterisation

Aqueous Alcoa A16-SG alumina slips containing 72 wt% (40 vol%) solids were dispersed electrostatically using nitric acid at a pH of 4. The slips were deagglomerated using ultrasonic energy and subsequently deaired prior to the pH, rheology and solid content being measured. The rheological characteristics of the slips were determined using a Bohlin 88 BV concentric cylinder viscometer with the shear stress–shear rate curves obtained over the shear rate range of $18.5–1122\,s^{-1}$ for both up and down shear sweeps. The equilibrium viscosity of the slips was determined at a steady shear rate of $18.5\,s^{-1}$ at temperatures ranging from 20–65°C. The viscosity of the slips used were in the range 120–140 MPa s when tested at ambient temperature (20°C).

The packing density, which can yield useful information on a slips' state of dispersion, was measured by sedimenting the slips in sealed, graduated perspex tubes at temperatures ranging from 7–65°C. The height of the gradually settling sediment was measured with time until a constant reading was noted over 48 h. Unfortunately, due to the length of the trials it was not possible to analyse the effect that microwave heating had on the slips' sedimentation behaviour.

2.2 Casting

Cup shaped moulds, see Fig. 1, were produced by mixing plaster of paris with deionised water in the ratio 3:2. A reservoir was placed on top of the mould in the form of a perspex graduated tube. This allowed the temperature of the slip to be monitored and the casting kinetics, from the

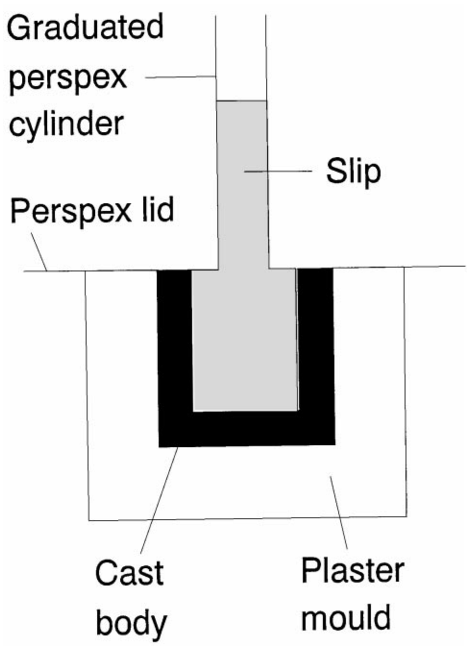

Fig. 1. Schematic of the slip casting apparatus.

amount of slip entering into the mould cavity, to be determined.

Slip casting was undertaken at 7, 20, 45 and 65°C using a fridge, a convection oven and a domestic 650 W microwave oven with the average thickness of the cast being used to determine the casting rate achieved. For elevated temperature casting the slip and mould were heated separately to the required temperature prior to casting. The solid content and the rheological behaviour of the slips were checked once casting was complete to determine if they had changed as a result of the casting process.

The densities of the bodies produced at each casting temperature were measured using the Archimedes principle on green casts, on casts fired at 1100°C for 1 h and after sintering for 2 h at 1540°C. Diametral strength tests[19] were performed on green bodies and those 'biscuit-fired' at 1100°C, and c-ring tests[20] were performed on sintered bodies. The microstructures at the mould–cast interface were examined using a scanning electron microscope on bodies heat treated for 1 h at 1100°C and also sintered for 2 h at 1540°C.

2.3 Water flow through the moulds

The uptake of water by the mould and progression of water through the mould were monitored in a number of experiments, in some cases by using dyes added to the alumina slip. The experiments were performed at 20°C, 45°C and 65°C with the elevated temperatures being achieved via both a convection and microwave oven. The temperatures of the wet and dry regions of the mould were also monitored. In addition, in some experiments the outer surfaces of the mould were covered with a tight fitting polymer film.

A separate series of experiments were also performed in which the amount of water contained in the capillary pores of a mould was determined. A cylinder of plaster of paris 22.25 mm in diameter and 175 mm long with the bottom 15 mm sealed using tape was suspended in a beaker of water containing dye such that only the bottom 10 mm were below the level of the water. This ensured that water could only enter the plaster via the bottom surface. The distance travelled up the cylinder by the water and the mass of water in the cylinder were recorded after 20 min. The experiment was performed with both mould and water at 20°C, 45°C and 65°C, the latter using both the microwave and convection ovens. From the results the fraction of the wet part of the mould occupied by water was calculated.

3 RESULTS AND DISCUSSION

3.1 Casting rates

The relationship of (cast thickness)^2 against time is seen in Fig. 2 with the casting rates (gradients) presented in Table 1. It is observed that elevated temperature casting yielded an increase in the casting rate. This could be due to two reasons; a change in dispersion state of the slip and/or a change in the effective permeability of the cast or mould.

3.2 Rheological behaviour of the slips

A decrease in the shear stress and corresponding reduction in the dilatancy onset point was noted on heating the slips with a minimum being observed at

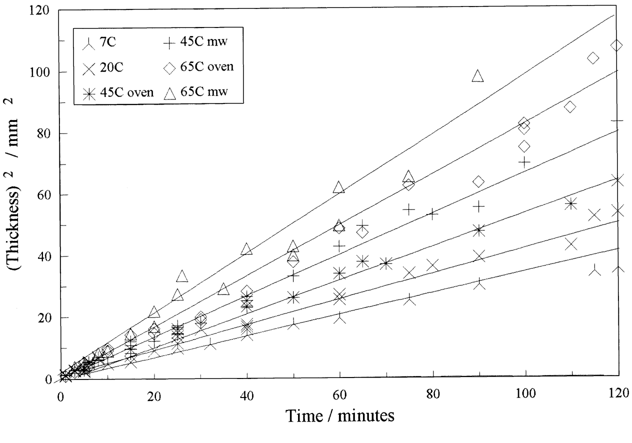

Fig. 2. L2 vs time for slip casting at different temperatures.

Table 1. Casting rates (gradient of thickness^2 against time) for bodies cast with nitric acid dispersed 72 wt% alumina slips as a function of casting temperature

Casting temperature (°C)	Casting rate		Casting rate at temperature ÷20°C
	Gradient	R^2	
20	0.45	0.98	1.00
45 mw	0.70	0.99	1.54
Oven	0.54	0.99	1.19
65 mw	0.96	0.95	2.12
Oven	0.81	0.98	1.79

45°C. The results were similar regardless of the heating method used, see Figs 3 and 4. These rheology results were confirmed by the viscosity measurements, Fig. 5, which also indicated a change in slope occurs at 40–45°C. This is not believed to be due to water evaporation as the solid content of the slips was remeasured after the tests and was found to have remained constant. Thus the change is believed to indicate a change in the state of dispersion of the suspension towards partial flocculation or coagulation. The results of the sedimentation tests, Fig. 6, suggest that the lower the temperature the slower the degree of settling, ie the greater the degree of dispersion. This implies that a degree of partial flocculation or coagulation occurs with increasing temperature although the lack of an increase in viscosity below about 40°C (Fig. 4) would suggest that this move towards partial flocculation is not significant below this temperature. At 65°C the slip had a large sediment height and virtually clear supernatant (the supernatant remained cloudy for the tests at 7–45°C) which was taken to indicate that the slip had become fully flocculated by 65°C.

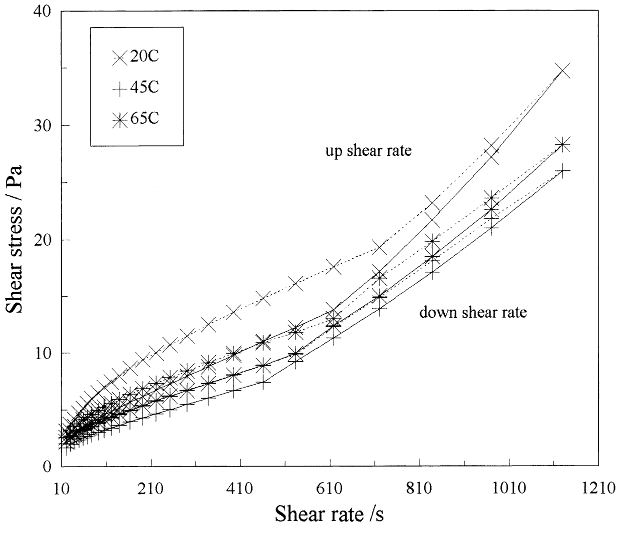

Fig. 3. Shear stress/shear rate curves for acid dispersed slips heated via microwave energy.

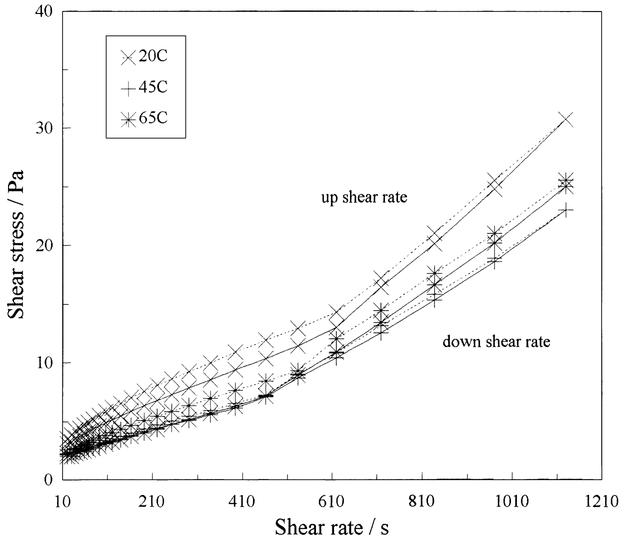

Fig. 4. Shear stress/shear rate curves for acid dispersed slips heated via convention oven.

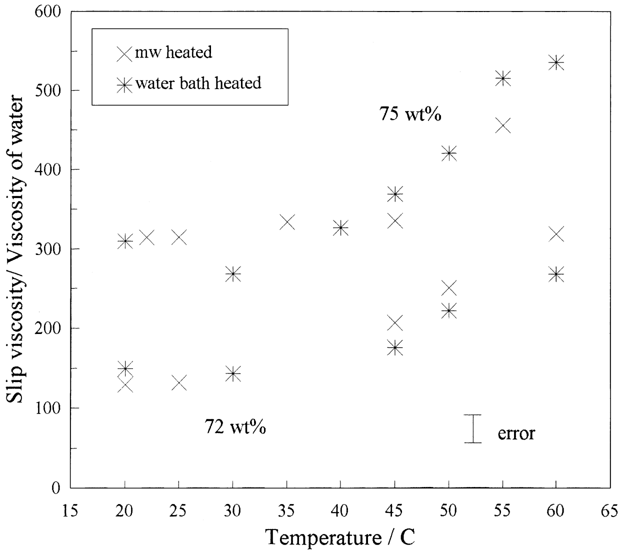

Fig. 5. Relative viscosities of acid dispersed slips as a function of temperature.

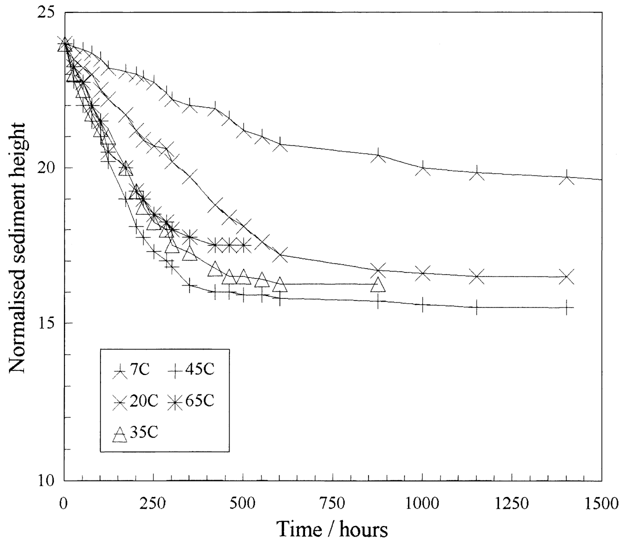

Fig. 6. Sediment heights for acid dispersed slips as a function of time at various temperatures.

Hence between 20°C and about 40°C a significant factor behind the accelerated casting rate is probably the reduction in viscosity of the slip as casting temperature is increased. However between 45°C and 65°C the faster casting rate is believed to be more likely to be associated with the change in the state of dispersion of the slip. However neither of these explanations can account for the differences between the microwave-assisted castings and those performed using convectional heating since no significant variation in slip viscosity with heating mechanism was observed.

3.3 Water flow through the moulds

During the casting process water is removed from the slip by the mould leaving behind a particulate cast at the mould–slip interface. The variables that control the rate of water extraction are: the volume fraction of voids in the mould; the permeability of the mould and the suction pressure arising from the mould, i.e. the capillary suction pressure.

The porosity of the mould is an inherent property based on the plaster: water ratio used and the mixing conditions. It will not alter significantly with temperature during casting. Similarly the permeability of the mould is a function of the pore structure and therefore is also assumed to remain constant. Thus the only factor that could alter at elevated temperatures is the suction pressure exerted by the mould.

The water flow experiments revealed that as the slip casting temperature increases both the rate of water absorption by the mould and the rate of water movement through the mould is faster, in agreement with the casting results described above. The use of microwave energy also resulted in faster absorption/movement than convectional heating for the moulds open to the atmosphere suggesting that the suction pressure arising from the moulds is greater (since it is known that the form of energy used to heat the slip does not affect the viscosity). However, if the moulds were covered then the faster rates achieved at elevated temperature became identical for the two types of heating method used, that is, the additional effect caused by the use of microwaves was lost.

The measurements of water uptake obtained using the suspended plaster of paris cylinders yielded similar results. The distance that the water travelled up the plaster followed the same trend as the castings; elevated temperature was greater than 20°C and the water in the plaster heated with microwave energy travelled further compared to that in the convection oven. The quantitative measurements of water uptake shown in Table 2 indicate that at higher temperatures the extra mass of water absorbed occupies a proportionately larger volume, ie the fraction of the wet mould occupied by water decreases. This means that the rate of movement of water *through* the mould is increased more than the rate of water uptake *by* the mould, particularly when using microwave energy to accelerate the casting process.

For castings performed in the convection oven there was no significant difference in the temperature of the mould in the dry or wet sections. However, temperature measurements made in the microwave oven showed that the dry section of the mould rarely exceeded 35°C regardless of the temperature of the slip, whilst the temperature of the water at the water front could be significantly higher. This is attributed to the plaster of paris being essentially transparent to microwave radiation so that the only significant heating that occurs in the mould is due to the presence of the water. In addition, since the water in the mould cannot move around as freely as it can in the slip, local temperature gradients can rapidly develop. This causes the water front within the mould to move faster than with convectional heating and may also result in an increase in vapour pressure. This mechanism is consistent with the result noted above, that when the moulds were covered the benefit from microwave heating was lost. When water enters into the pores of the plaster air must be displaced. When the mould was covered then an equilibrium pressure would build up which would eventually reduce the casting rate. With microwave heating, the more rapid movement of the water front and increase in vapour pressure would cause the effect to occur much faster, eliminating any advantage from using microwaves.

3.4 Characterisation of the casts produced

The green densities of the bodies cast from the slips were all within the range 2.38–2.47 g cm^{-3} (i.e. 60.6–62.8% of theoretical) with no discernable trend present with either an increase of casting time

Table 2. Water movement within mould as a function of temperature after 20 min

Casting temperature (°C)	Height of water movement up mould (mm)	Mass of water in mould (g)	Fraction of mould occupied by water
20	42.2	5.79	0.353
45 mw	56.8	7.29	0.330
Oven	57.0	7.33	0.331
65 mw	64.4	7.68	0.307
Oven	60.8	7.71	0.326

Table 3. Strength results for slip cast bodies

Casting temperature (°C)	Green bodies	Partially sintered bodies		Fully sintered bodies	
	Tensile strength (MPa)	Tensile strength (MPa)	Weibull modulus	Tensile strength (MPa)	Weibull modulus
20	0.198 SD=0.027	21.06 SD=4.02	4	203 SD=19.07	6
45 mw	0.185 SD=0.010	20.78 SD=2.63	6	191 SD=8.01	4
Oven	0.207 SD=0.026	22.76 SD=3.78	4	187 SD=21.62	4
65 mw	—	18.97 SD=2.91	4	201 SD=9.89	5
Oven	—	20.11 SD=1.75	6	193 SD=7.15	5

or temperature. Each body reached ~98% of its theoretical density on sintering. Scanning electron microscopy also revealed no apparent difference in the microstructures of the partially or fully sintered casts as a function of casting conditions, however it is possible that the process of drying and sintering eliminated the distinguishing features. Further work using an environmental scanning electron microscope (ESEM) on the wet green body would be required to determine whether this is the case.

The mechanical strength measurements are seen in Table 3. The standard deviation and Weibull modulus values obtained indicate that there is a relatively large degree of scatter in the data as expected for such measurements made on non-optimised systems. However, no trend is observable either in the level of reliability or in the strength values themselves as a function of casting conditions. Thus despite the differences observed in the states of dispersion of the casting slips and casting rates, there appears to be no discernable differences in the final microstructures or properties of the bodies themselves.

4 CONCLUSIONS

Elevated temperature slip casting of 72 wt% acid dispersed alumina slips results in an increase in the casting rate with no evidence that this accelerated rate has any detrimental effect on the cast body produced. The enhanced rate is apparently achieved via two mechanisms. At temperatures less than approximately 40°C it is probably due to a reduction in the slip viscosity enabling easier movement through the cast layer and mould. At higher temperatures there is a change in the dispersion state of the slip. It appears that as the slip temperature increases above ambient the slip becomes increasingly flocculated and that above about 45°C this influences the casting rate. By 65°C the slip is fully flocculated.

A further enhancement in the casting rate is achieved with microwave energy over convectional heat energy. This is attributed to the rate of movement of water *through* the mould being increased more than the rate of water uptake *by* the mould. This in turn is believed to be due to the plaster of paris being essentially transparent to microwave radiation so that the only significant heating that occurs in the mould is due to the presence of the water. Since the water in the mould cannot move around as freely as it can in the slip, local temperature gradients can rapidly develop. This causes the water front within the mould to move faster than with convectional heating.

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