

# Slip Casting of Cordierite and Cordierite–Mullite Materials

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## Abstract

*The obtention of cordierite and cordierite–mullite ceramics by slip casting is studied. Aqueous slips with solids contents up to 65 wt% were used. The effect of the deflocculant content and the mixing time on the rheological behavior was studied, showing always a shear thickening behavior, which can be deleterious for demolding and handling. The rheological behavior became Newtonian by reducing the solid content up to 60 wt% and adding a plasticizer. This led to higher green densities (near 63% th). Different firing cycles were tested to improve the sintered density, reaching values between 96.5 and 97.5% th. In all cases, both the green and the sintered densities were significantly higher than those obtained by axial pressing. © 1998 Elsevier Science Limited. All rights reserved*

## 1 Introduction

Cordierite ( $2\text{MgO}\cdot 2\text{Al}_2\text{O}_3\cdot 5\text{SiO}_2$ ) and cordierite-based glass ceramics are promising materials for electronic packaging because of their low dielectric constant and thermal expansion. Moreover, cordierite is employed in fast-heating furnaces due to its excellent thermal shock resistance. However, because of its poor mechanical properties a second phase is often added to improve them. According to this, and having in mind the high mechanical performance of mullite, this material can be added in order to reinforce the cordierite matrix.<sup>1,2</sup>

On the other hand, the cordierite and cordierite–mullite materials are difficult to sinter without sintering aids. Unfortunately, the thermal expansion and electrical properties of cordierite are degraded if such additives are employed.<sup>3,4</sup> For this reason

there is an actual necessity to improve the steps of powder processing before sintering to reach higher green densities. Colloidal processing of ceramics is being increasingly considered for the production of more homogeneous, denser materials, with the additional advantage that allows the obtention of complex shaped parts.<sup>5,6</sup> Between the colloidal forming processes, slip casting is the most widely used for many kinds of ceramic powders<sup>7,8</sup> because of the high reliability of the process and its simplicity.

The aim of this work is to develop cordierite and cordierite–mullite materials by slip casting in order to reach higher densities than those obtained by pressing, normally used for the obtention of these materials.

## 2 Experimental

Commercially available powders of cordierite (CORCR Baikowsky, France) and mullite (MULCR Baikowsky, France) were used. The characteristics of these powders are summarized in Table 1. The contents of the elements were determined by wet chemical analysis. Particle sizes and specific surface areas were measured using a Coulter LS 130 analyser and a Monosorb Quantachrome equipment, respectively. The real densities were determined by He picnometry (Multipicnometry, Quantachrome, USA).

Scanning electron microscopy (SEM) observations reveal that the cordierite powder [Fig. 1.(a)] has an heterogeneous morphology composed by submicronic particles and flat and/or rod-like bigger particles.

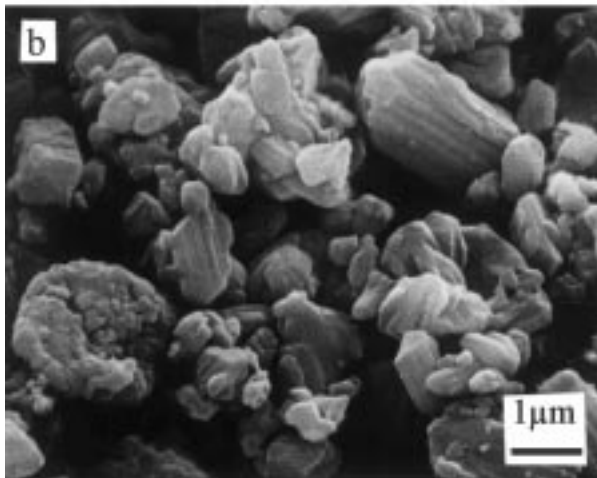
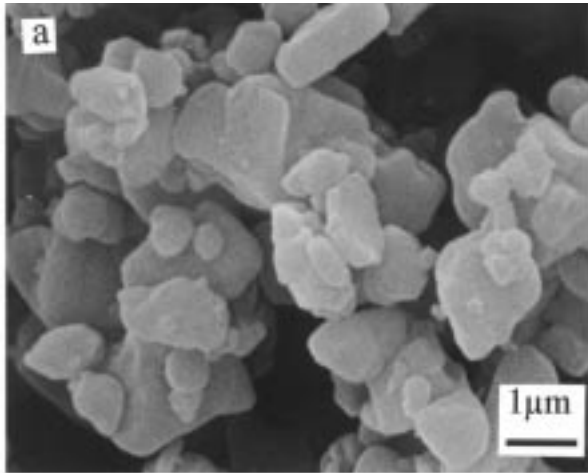
The relatively coarse starting mullite [Fig. 1.(b)] was subjected to milling in order to break down the agglomerates and to reduce the shape factor to allow a better particle packing during colloidal

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**Table 1.** Characterization of cordierite and mullite starting powders

<i>Powder</i>	<i>Cordierite</i>	<i>Mullite</i>
Chemical analysis (wt%)		
W.L.	0.46	0.13
SiO <sub>2</sub>	51.00	27.20
Fe <sub>2</sub> O <sub>3</sub>	0.03	0.02
TiO <sub>2</sub>	0.02	0.01
Al <sub>2</sub> O <sub>3</sub>	35.60	72.70
CaO	0.11	0.07
MgO	12.80	0.008
Na <sub>2</sub> O	0.11	0.06
K <sub>2</sub> O	0.002	0.003
Specific surface area (m <sup>2</sup> g <sup>-1</sup> )	2.5	2.3
Mean particle size (μm)	2.1	2.7
Pyconometric density (g cm <sup>-3</sup> )	2.6	3.05

forming.<sup>9</sup> Furthermore, the milling assures a low particle size of the mullite to allow a more homogeneous distribution of this phase in the cordierite matrix. Milling was performed using a nylon-coated attritor mill with alumina balls for 7 h. The milled powder had a mean particle size of 1.2 μm and specific surface area of 6.4 m<sup>2</sup> g<sup>-1</sup>.

**Fig. 1.** Scanning electron microscopy (SEM) micrographs of the starting (a) cordierite and (b) mullite powders.

The cordierite–mullite mixtures were prepared with the as-received cordierite and the 7 h milled mullite powders. Aqueous slips of cordierite and cordierite–mullite were prepared at solids loadings up to 65 wt%, using a ball mill with alumina jar and balls.

The rheological behaviour of the slips was studied considering the pH, the concentration of deflocculant, the solid content, the milling time and the addition of plasticizers in order to modify the rheology of the slips. A carbonic acid based polyelectrolyte (Dolapix CE64, Zschimmer–Schwarz, Germany) was selected among a variety of commercial deflocculants. Additions of a high molecular weight (i.e. 20 000) plasticizer (polyethylene glycol, PEG, Compound 20M, Carbowax, USA) were also studied. The viscosity measurements were performed using a concentric cylinders viscometer (Haake, Rotovisco RV20, Germany) at a constant temperature of 25°C. The measurements were made increasing the shear rate from 0 to 2700 s<sup>-1</sup> in 2 min, with a plateau of 1 min, and decrease to 0 s<sup>-1</sup> in 2 min. The equipment software allows the simultaneous analysis of the rheological data, considering a number of mathematical and rheological regression models. All the measured flow curves were analysed in the whole shear rate range to select the best fitting model.

The slips showing the desired rheological behavior were cast onto plaster of Paris molds to obtain green samples in the form of solid disks (2 cm diameter, 1 cm height). After drying in air for 48 h the green density was measured by the Hg immersion method.

The casts were sintered in an electrical furnace with MoSi<sub>2</sub> heating elements at 1450°C/2 h, using different firing cycles (Table 2). The microstructures of the sintered materials were observed by scanning electron microscopy (SEM) on polished and thermally etched surfaces. The thermal etching was made at 1400°C/30 min, with a heating rate of 20°C min<sup>-1</sup>. The apparent densities (δ<sub>s</sub>) of the sintered samples

**Table 2.** Sintering cycles

<i>Sintering cycle</i>	<i>Heating rate and maximum temperature</i>		<i>Cooling rate</i>
I	25°C min <sup>-1</sup> 800°C	5°C min <sup>-1</sup> 1450°C/2 h	10°C min <sup>-1</sup>
II		5°C min <sup>-1</sup> 1450°C/2 h	5°C min <sup>-1</sup>
III		1°C min <sup>-1</sup> 1450°C/2 h	1°C min <sup>-1</sup>
IV	1°C min <sup>-1</sup> 500°C/2 h	1°C min <sup>-1</sup> 1450°C/2 h	1°C min <sup>-1</sup>

were measured by the Archimedes method in water. The relative density percentages ( $\% \delta_s / \delta_{th}$ ) were calculated from the real densities ( $\delta_{th}$ ) of the powders heated at 1450°C.

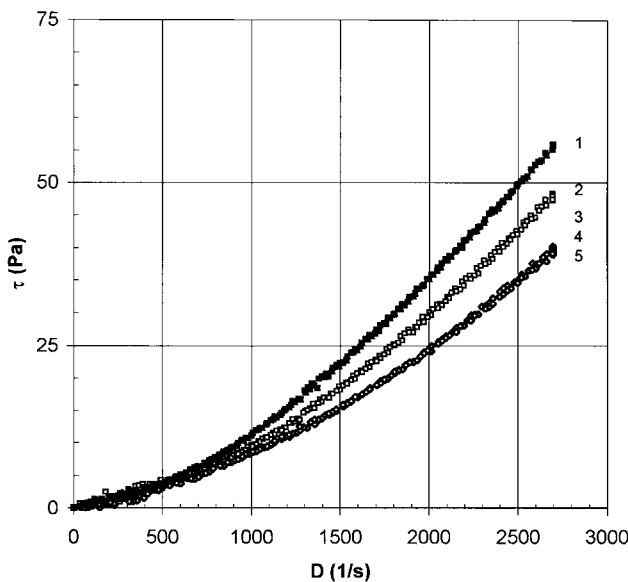
### 3 Results and discussion

#### 3.1 Slip properties

Cordierite slips with a solid loading of 65 wt% have been prepared with different concentrations of deflocculant in order to search the best dispersing conditions. Figure 2 shows the corresponding flow curves obtained after 4 h treatment in ball mill. It can be seen that all the slips have a shear-thickening behavior without any tixotropic cycle. Up to a concentration of 0.5 wt% deflocculant, the behavior can be adjusted by the Casson model [eqn (1)]

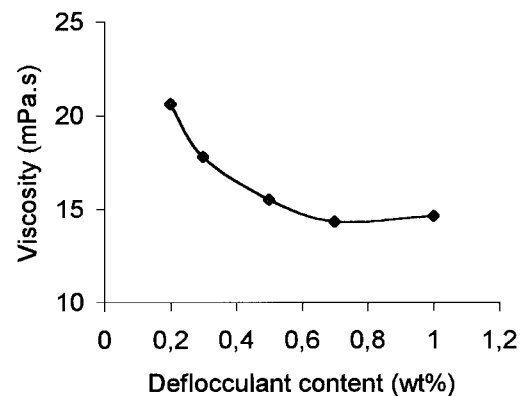
$$\tau^{1/2} = \tau_0^{1/2} + (\eta \cdot \gamma)^{1/2} \quad (1)$$

where  $\tau$  is the shear stress,  $\tau_0$  is the yield point,  $\eta$  is the viscosity and  $\gamma$  is the shear rate. For concentrations of deflocculant higher than 0.5 wt%, the best fitting corresponds to a Newtonian regression, but some thickening is still appreciated. On the other hand, the viscosity values (Fig. 3) decrease when the deflocculant content increases until 0.5 wt% (referred to solids) but further additions do not produce significant changes in the viscosity. Nevertheless, the pH of the slips decreases for increasing deflocculant concentrations (from 9.3 for 0.2 wt% to 8.9 for 1.0 wt%).



**Fig. 2.** Flow curves of 65 wt% cordierite slips with additions of deflocculant of (1) 0.2, (2) 0.3, (3) 0.5, (4) 0.7 and (5) 1.0 wt% referred to solids.

Although the resulting viscosity is very low, the dilatant behavior can difficult the casting process, specially demoulding and further manipulation of the green cast. One problem associated to dilatancy is related to the fact that mixing is difficulted, which in practice generates problems for the preparation of a stable slip, because the more energetic is the mixing process, the higher is the viscosity. On the other hand, a basic requirement of the slip is that particle-particle repulsive forces are necessary to assure the slip stability, whereas during casting the basic requirement in the cake is to promote particle-particle attraction. The first can be easily controlled by means of the addition of deflocculant. In our case, the deflocculant is a polyelectrolyte in which stabilization is achieved by two simultaneous mechanisms: (1) the presence of charges in the deflocculant molecules, promoting electrostatic repulsion; and (2) the deflocculant chains adsorb onto the particle surfaces, providing also a steric hinderance. However, the required repulsion in the cast cannot occur until the residual moisture has been reduced to a level sufficient for the repulsion effects of the adsorbed deflocculant to be exceeded by the Van der Waals forces. This depends on the slip rheology and strongly influences the handleability of the cast. Ceramic slips should be preferably pseudoplastic, because the viscosity is lower at higher shear rates, and thus mixing and pouring into the mold are easier. At lower rates the viscosity is higher, retarding sedimentation and facilitating wall thickness formation. Dilatant slips behave in the opposite sense. At higher shear rates the problems mentioned above occur, but there are also important limitations at low or zero shear rates, where the viscosity is minimum, then promoting possible sedimentation effects and, more important, greatly prolonging casting and demolding times. When no external forces are operating the piece can spontaneously deform.



**Fig. 3.** Viscosity of 65 wt% cordierite slips as a function of deflocculant content measured at the maximum shear rate (2700 s<sup>-1</sup>).

In the present study, the dilatancy is associated to the size and the morphology of the starting powders [Fig. 1(a)]. Flat and elongated particles can easily form open structures when they are subjected to high shear forces, or referring to the rheological measurement, when shear rate increases.<sup>9</sup>

Another factor to be considered in order to improve the homogeneity and reduce the dilatancy is the homogenization time, which must be also optimized. This has been performed after the most effective concentration of deflocculant has been established (0.5 wt%) for a fixed mixing time. Figure 4 shows the flow curves of slips with 65 wt% solids and 0.5 wt% deflocculant ball milled during different times from 1 to 24 h. The minimum viscosity was obtained after 4-6 h milling. For shorter or longer times some tixotropy is observed in the flow curve. In addition to the viscosity measurements, the particle size distribution of fresh slips milled at different times has been also determined. The registered mean values are summarized in Table 3, as well as the corresponding pH values. It can be observed that both the pH and the mean particle size are maintained constant up to 4 h milling time, whereas longer times give place to an increased pH and some reduction in the particle size. This suggests a soft milling which should be accompanied by an increased surface area of the particles, thus originating new surfaces uncovered by the deflocculant. This is confirmed by the pH variation, since an increased concentration of deflocculant should shift down the pH towards the isoelectric point contrarily to the observed in this case.

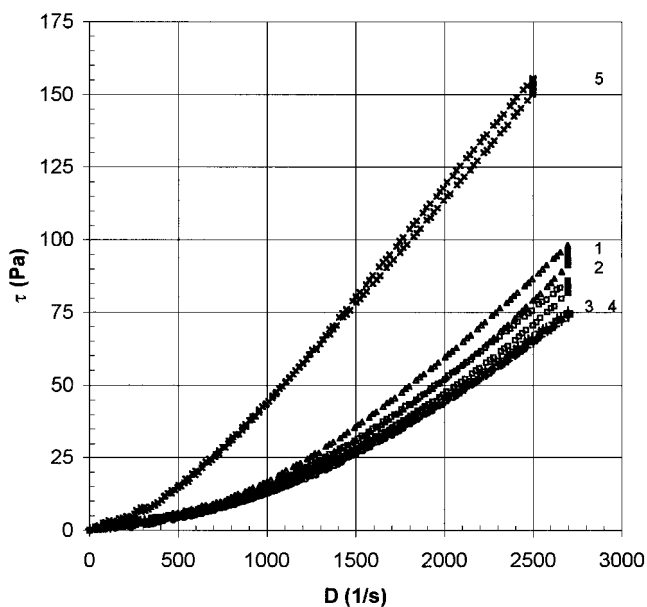
In order to modify the shear thickening behavior, the addition of a plasticizer has been con-

**Table 3.** Evolution of pH and mean particle size with milling time

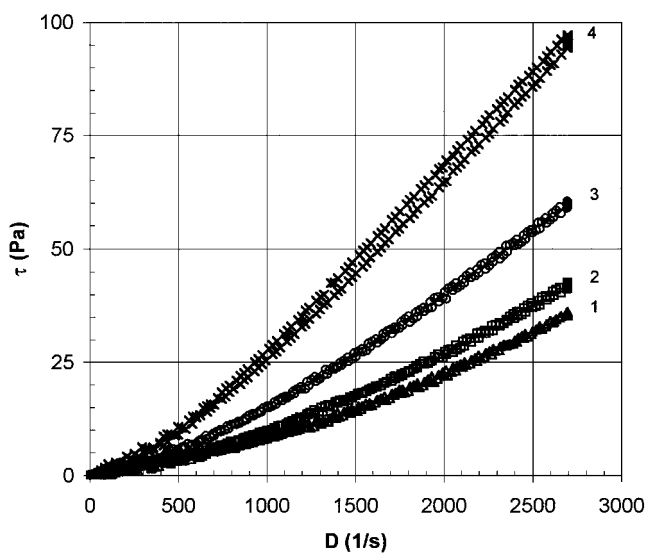
Milling time (h)	pH of the slip	Mean particle size ( $\mu\text{m}$ )
0	—	2.6
2	9.14	1.98
4	9.18	1.98
6	9.20	1.89
24	9.63	1.83

sidered. Plasticizers of low or very low molecular weight are commonly employed in the ceramic industry as dispersing aids, because the small chains allow a high degree of motion. On the other hand, high molecular weight plasticizers are more rigid and, if the concentration is high enough, can produce some binding effect. This produces an increase in the viscosity. A polyethylene glycol (PEG) with a molecular weight of 20 000 has been used at different concentrations over slips prepared with 65 wt% solids and 0.5 wt% deflocculant. The addition of increasing concentrations of plasticizer produces a progressive increase in the viscosity. For additions of 2 wt% PEG, this increase is significantly high (36 mPas) and, the most important, the shear thickening behavior is reduced. The flow curves of these slips containing plasticizer are plotted in Fig. 5. For small PEG additions, the best fit is again obtained for Casson model, whereas for 1 wt% PEG or more, the best fitting is reached for the Newtonian model, although some curvature in the flow curves is still evident. The pH of the slip (9.3) practically does not change when PEG is added (9.2 for additions of 2 wt% PEG).

According to the above analysis, it would be possible to change the rheology by the simultaneous



**Fig. 4.** Flow curves of cordierite slips after mixing in a ball mill for (1) 1, (2) 2, (3) 4, (4) 6 and (5) 24 h.



**Fig. 5.** Effect of plasticizer on the flow curve of cordierite slips with (1) no addition of plasticizer and with additions of (2) 0.5, (3) 1.0 and (4) 2.0 wt% plasticizer.

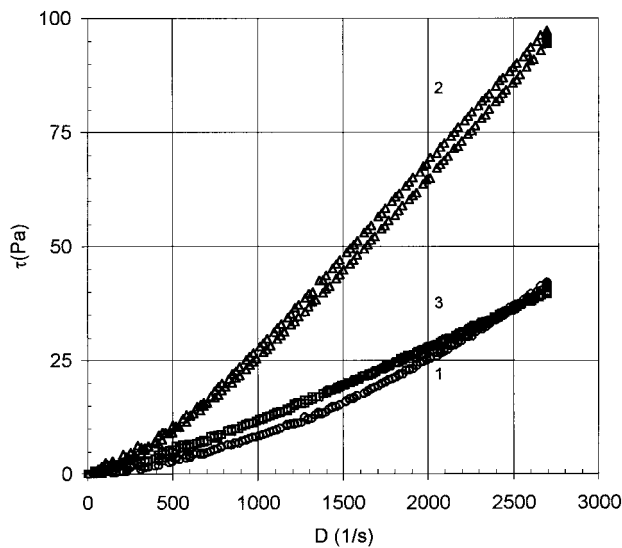
variation of both the solid content and the processing additives. The effect of solid loading is obvious. Low concentrated slips are Newtonian and increasing concentrations in solids produce deviations from this Newtonian behavior. In our case, and considering the very low viscosities obtained, a small reduction in solid loading will give Newtonian slips. Figure 6 plots the flow curves of the slips with different solid contents showing the effect of the addition of plasticizer. The starting well-dispersed slip (65 wt% solids, 0.5 wt% deflocculant) presents shear thickening behavior and low viscosity, as mentioned before. When 2 wt% PEG is added, the shear thickening practically disappears, although viscosity significantly increases. Once the shear thickening is reduced, the viscosity can be also reduced by decreasing the solid content of the slip from 65 to 60 wt%. It can be seen that the final viscosity is very similar to that of the starting slip but the dilatancy is strongly reduced, thus simplifying handling and enhancing the casting behaviour. The slips with 65 wt% solid loading fit to Casson flow, as described by eqns (2) and (3) for slips without PEG and with PEG, respectively.

$$\tau^{1/2} = 1.53 + 0.22\gamma^{1/2}(\text{mPa}) \quad (2)$$

$$\tau^{1/2} = 0.412 + 0.150\gamma^{1/2}(\text{mPa}) \quad (3)$$

The slip with PEG and 60 wt% solid loading follows the Newtonian law given below

$$\tau = 0.138\gamma(\text{mPa}) \quad (4)$$

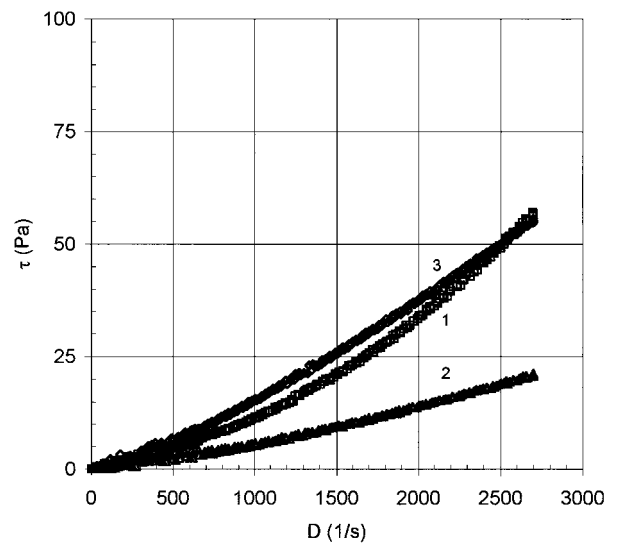


**Fig. 6.** Modification of the rheological behavior of (1) 65wt% cordierite slips by (2) adding 2.0wt% plasticizer and (3) further decrease in solid loading up to 60 wt% solids.

Once the processing conditions for cordierite slips have been established, the effect of the addition of 10 wt% mullite on the cordierite matrix has been also studied. Aqueous mullite slips at 65 wt% solid loading were prepared using different concentrations of the same deflocculant employed for the cordierite. Mixing time was fixed at 4 h in all cases. Table 4 shows the viscosity and the pH of the mullite slips as a function of deflocculant concentration. These data show that the viscosity is very low for 0.5 wt% deflocculant, as in the case of cordierite.

Taking into account the above results, the cordierite–mullite slips have been prepared with the two same solid contents used for only cordierite (60 and 65 wt%), 0.5 wt% deflocculant, with 2 wt% PEG and without PEG, and 4 h ball milling. In Fig. 7, the flow curves of cordierite–mullite slips with 60 and 65 wt% solids without plasticizer and the corresponding to 60 wt% solids with 2 wt% PEG are plotted. The combined effect of lower solid content and addition of a plasticizer enables to change the rheological behavior reducing the shear thickening and maintaining the same order of viscosity value. The reduction of the solid content to 60 wt% produces also a low viscosity and slip Newtonian behavior. By adding the plasticizer to this slip the viscosity becomes similar to that obtained for 65 wt% slips, but with a nearly Newtonian behavior. The reduction in solids promotes the change of rheological behavior to a Newtonian model.

It must be noted that the viscosities of cordierite–mullite slips (20.5 mPas at 2700 s<sup>-1</sup>) are higher than those obtained for both the cordierite slips and the mullite ones (14.6 and 12.4 mPas, respectively, at 2700 s<sup>-1</sup>).



**Fig. 7.** Modification of the rheological behavior of (1) 65 wt% cordierite–mullite slips by (2) decreasing solid content to 60 wt% and (3) further addition of 2.0 wt% plasticizer.

**Table 4.** Variation of pH and viscosity (at 2700 s<sup>-1</sup>) of 65 wt% mullite slips using different concentrations of deflocculant

Deflocculant wt%	0.1	0.2	0.3	0.5	1.0
pH	8.0	8.3	8.5	8.7	8.7
Viscosity (mPas)	17.7	13.5	12.8	12.4	11.8

According to these results, slip casting was performed for either the cordierite or the cordierite-mullite materials at two different conditions: (1) slips with 65 wt% solids, and (2) slips with 60 wt% solids and 2 wt% PEG, respectively. In all cases the deflocculant content was fixed at 0.5 wt% and the mixing time was 4 h. Under these conditions the shear thickening behavior can be minimized.

### 3.2 Properties of cast and sintered materials

The modification of the rheological properties of the slips influences the casting behavior and, consequently, the relative green density. In general, higher solid content slips give denser green casts.

Cordierite obtained from 60 wt% slips with 2 wt% PEG was labelled as C1, and that obtained from 65 wt% slips without PEG, as C2. Similarly, CM1 corresponds to cordierite-mullite prepared from 60 wt% slips and 2 wt% PEG, and CM2 to 65 wt% slips without PEG. In all the slips, 0.5 wt% deflocculant was added.

The green density of C2 casts was 1.60 g cm<sup>-3</sup> (61.5%th). Despite of the reduction of the solids content (60 wt%), when PEG was added the green density slightly increased to 1.63 g cm<sup>-3</sup> (62.7%th). The relative green densities of cordierite-mullite casts also increased from 62.1%th to 63.2%th when PEG was added. These values are considerably higher than those obtained by axial pressing, thus confirming a better rearrangement of particles during casting.

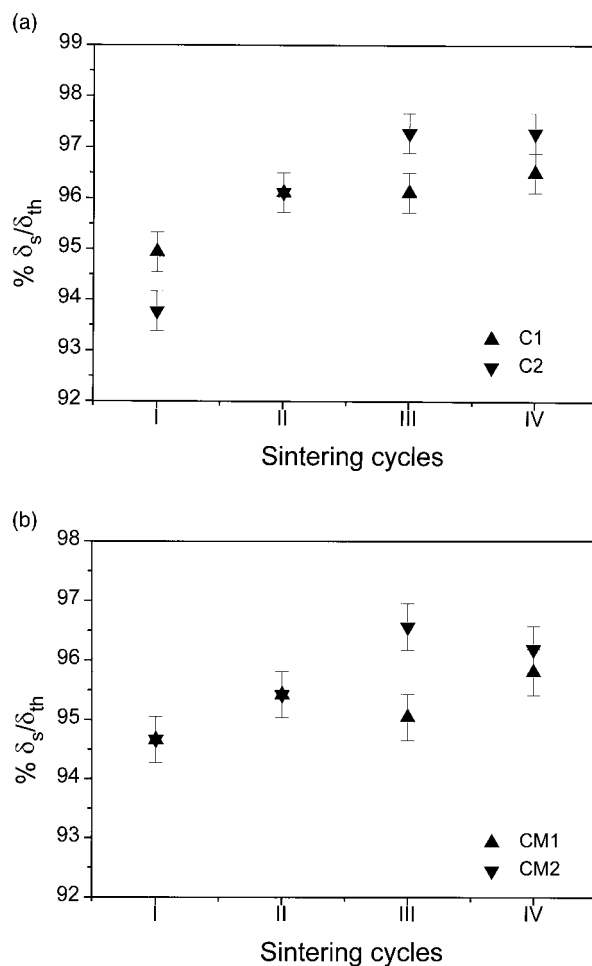
The relative density of the sintered materials was calculated relating the measured density of the samples by water immersion with the theoretical densities of cordierite (2.57 g cm<sup>-3</sup>) and cordierite-10 wt% mullite (2.62 g cm<sup>-3</sup>) as determined by He picnometry after treating the dry powders at 1450°C/2 h.

Figure 8 shows the relative densities of cordierite [Fig. 8(a)] and cordierite-mullite [Fig. 8(b)] samples sintered according to the heating cycles given in Table 2. The relative densities of cordierite samples C1 and C2 sintered using the faster cycle (I) are 94.9 and 93.8 %th, respectively. These values are lower than those attained using the other firing schedules (heating rates of 5 and 1°C min<sup>-1</sup>). Samples show isolated cracks and other defects, while

no defects were observed in samples sintered following the slow cycles. Obviously, this heating rate is too fast to allow burning out of the organics without damage. It is concluded that a fast heating cycle, usual for uniaxial pressed samples, is not useful for slip cast samples.

For slow heating rates, a difference between sintered samples C1 (with PEG) and C2 (without PEG) was additionally observed. In sample C1 no changes were measured in the obtained densities using both II and III sintering cycles, however, a slight increment of the density was measured when it was included a dwell at 500°C (cycle IV). On the other hand, in sample C2 the densification was higher for decreasing heating rates, but a step at 500°C (cycle IV) did not improve the degree of densification. This difference must be related to the fact that a low temperature dwell helps to remove the organic additive in C1 sample.

After sintering, the relative densities of cordierite-mullite materials showed a similar trend to that observed in sintered cordierite samples in relation to the firing cycle used and the presence of the plasticizer [Fig. 8(b)]. The density values increased



**Fig. 8.** Relative density of (a) cordierite and (b) cordierite-mullite cast specimens heated according to cycles shown in Table 2.

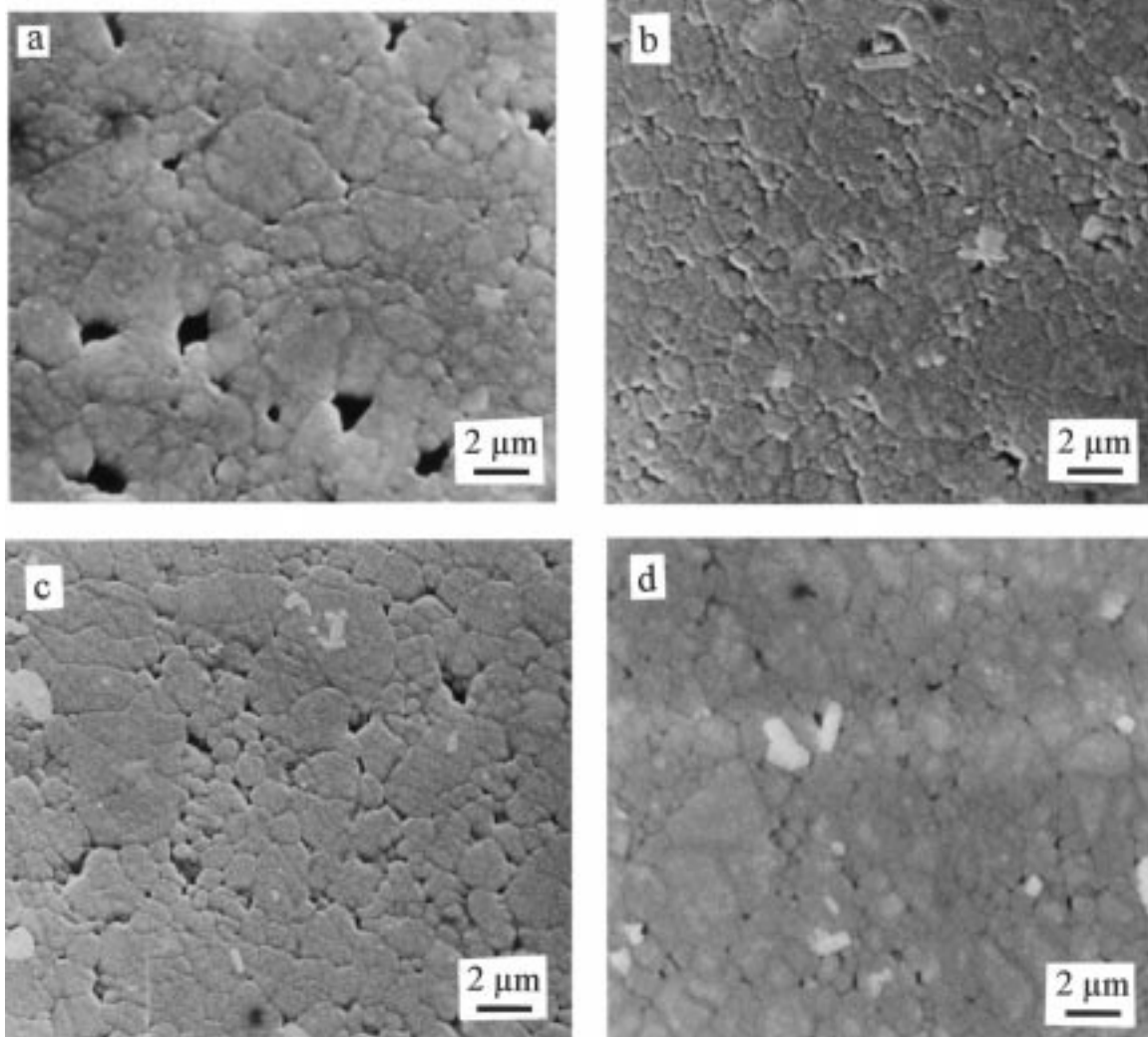
as the heating rate diminished. Additionally, the sample CM1 with 2wt% PEG, reached a greater density when cycle IV was employed, while in CM2 no changes were registered.

Comparing the cordierite and cordierite-mullite relative densities it can be noted that the values corresponding to composite materials are slightly lower although the cast densities are higher. This could be attributed to the presence of mullite grains in the cordierite matrix<sup>1,3,10,11</sup>

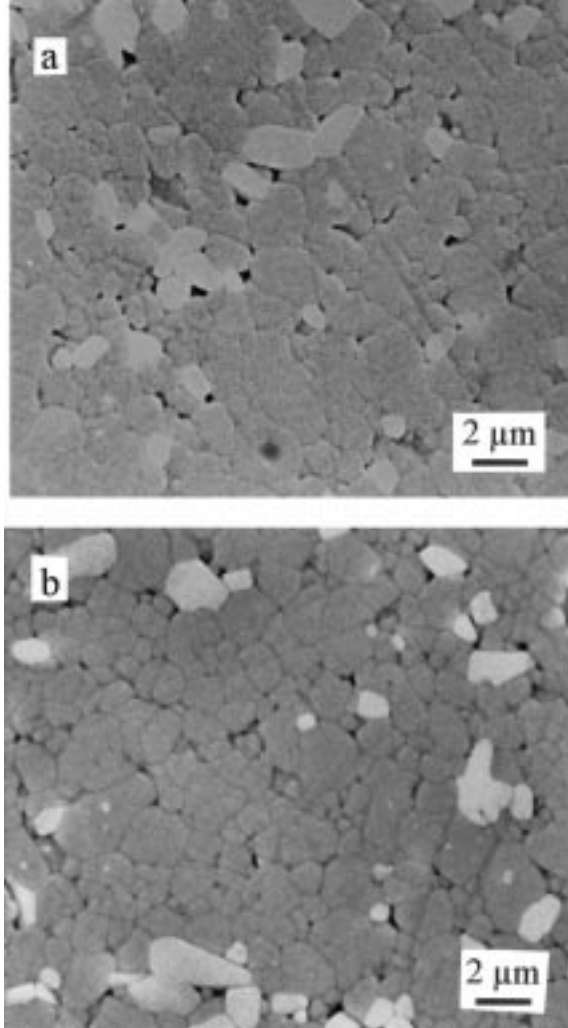
The green and sintered relative densities of cordierite and cordierite-mullite materials prepared by slip casting were significantly higher than those attained from uniaxial pressed green compacts (55.4 and 94%, respectively, for cordierite and 56.1 and 94.3% th for cordierite/mullite). This demonstrates the improved consolidation provided by colloidal processing techniques. Other aspect to be mentioned is that in both series of samples, the materials with 2wt% PEG (C1 and CM1) gave slightly lower densities than the samples without PEG (C2 and CM2). The most important is that both alternative routes can be useful for different

applications. For drain casting of open shaped pieces (manufacturing of crucibles, containers, etc.) or solid pieces of small thickness are desired, there is no a real necessity to add plasticizer. However, the production of bigger or thicker pieces is limited by the initial dilatancy of the slip, because the casting time increases very much and handleability of the cast is poor. For such situations, the addition of a plasticizer is useful in order to reduce the dilatancy, facilitating the whole processing cycle.

SEM microphotographs of the microstructures of thermally etched C1 samples sintered following cycles I, III and IV are given in Fig. 9(a), (b) and (c), respectively. Equiaxial cordierite grains, 2–3  $\mu\text{m}$  in size, are present. A decrease in porosity occurs in parallel with the densification and no coarsening of the grains are detected as the heating rate is lower (cycles III and IV) with respect to the faster heating treatment (cycle I). No significant differences can be observed between the microstructures of samples C1 [Fig. 9(c)] and C2 [Fig. 9(d)] sintered following the firing cycle IV, although in C1 sample, 2wt% PEG was added.



**Fig. 9.** SEM micrographs of C1 samples sintered according to cycles (a) I, (b) III and (c) IV, and (d) of sample C2 following cycle IV.



**Fig. 10.** SEM micrograph of cordierite–mullite samples heated according to cycle IV obtained (a) from 65 wt% slip without plasticizer and (b) from 60 wt% slip containing 2 wt% plasticizer.

The surface of the thermally etched cordierite–mullite sample [Fig. 10(a) and (b)] showed equiaxial cordierite grains and also elongated mullite grains, which are difficulting the densification process. This can be observed in the microstructure, where a higher porosity is detected around the elongated mullite. This is confirmed by the measured densities reported before.

#### 4 Conclusions

It is possible to change the rheology of either cordierite or cordierite–mullite slips decreasing the solid content (from 65 to 60 wt%) and adding a plasticizer (2 wt% PEG) as a rheology modifier. In addition, the viscosity of the obtained nearly Newtonian slip is increased, enhancing the casting and draining behavior, and consequently, demolding and handling. Furthermore, the relative green densities increase when 2 wt% PEG is added. The values of cordierite–mullite casts are higher than

those obtained for cordierite casts thus confirming their better casting behavior.

The final relative densities of cordierite samples sintered using the fast heating cycle, are notably lower than those attained using the firing schedules with slow heating rates. For the slow heating rates, in sample C1 no changes are measured in the obtained densities using both slow sintering cycles, however, a slight increment of the density is measured when a dwell at 500°C is included, while in C2 this step does not improve the degree of densification. The relative densities of the sintered cordierite–mullite materials show a similar trend, although the values are lower even though the cast densities are higher. In all the cases, both the green and the sintered densities obtained by slip casting are significantly higher than those attained from uniaxial pressed green compacts.

A decrease in porosity occurs in parallel with the densification and no coarsening of the grains is detected as the heating rate decreases with respect to the fast heat treatment. The microstructures of the cordierite samples show equiaxial grains and the cordierite–mullite samples, equiaxial mullite and cordierite grains and also elongated mullite grains.

In spite of the effect of the addition of PEG is very important in the slip behavior, the sintered densities are slightly lower with respect to the samples without PEG.

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