

Rheological characterization of water-based slurries for the tape casting process

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Abstract

Rheological properties of aqueous tape casting slurries have been investigated as they strongly affect the tape casting process and the quality of the final product. The aqueous slurries consist of yttria stabilized zirconia and a polymeric latex emulsion as the binder. The viscosity, its time dependent behavior and the strength of the internal structure of the slurry were characterized by static and dynamic measurements using a cone-plate viscosimeter. The results were compared with the properties of the pure latex emulsion binder. The ceramic slurries exhibit the desired strong pseudoplastic, but non-thixotropic behavior. Due to the high solids content the slurries show distinctive elastic properties below a critical shear stress. Above the critical shear stress the internal structure is broken up and the viscous properties become predominant. The paper demonstrates that the measurement of the viscosity, its time dependent behavior and the strength of the internal structure of the slurry are excellent tools to characterize the suitability of a ceramic slurry for the tape casting process. © 2002 Elsevier Science Ltd and Techna S.r.l. All rights reserved.

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1. Introduction

The tape casting process is an economical method to produce thin, flat ceramic components such as substrates, capacitors, piezoactuators, sensors, etc. [1,2]. For each of these products different tape casting slurries are used. Their rheological behavior depends on the type and concentration of powder, binder, solvent and other organic additives such as dispersants or wetting agents. The rheology strongly controls the quality of the final product. In comparison with organic solvents water-based systems have the advantage of low toxicity and environmental safety. But the replacement of organic solvents by water has a strong influence on the rheological behavior of the slurry. Water exhibits a higher surface tension than organic solvents and the

solubility of binders in water is also limited. Therefore polymeric emulsion binders are often used, which have the disadvantage to exhibit a higher viscosity [3]. These emulsion binders, which are colloidal dispersions of a polymer in water, had been used to fabricate slurry formulations with high solids contents [4].

In comparison with organic solvent based tape casting slurries, aqueous tape casting systems have a smaller tolerance to minor changes in processing parameters such as casting composition, drying conditions, or film thickness [5]. Thus, it is of utmost importance to understand and control the rheology of water-based tape casting slurries.

The contents of water and organic additives in tape casting slurries should be as low as possible. Thus, the amount of water which has to be evaporated during drying and of organic additives, which have to be burnt out, is reduced. This minimizes the risk of defect formations. In addition, a low content of water results in high particle loaded slurries with high viscosities. On the other hand, the viscosity of the slurries must be low enough in order to ensure a homogeneous mass flow under the blade during the casting process. In addition the slurry should exhibit pseudoplastic behavior: during

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passing the blade, the viscosity is decreased due to shear forces, and immediately after the blade the viscosity increases rapidly to suppress uncontrolled flow and to prevent sedimentation of the ceramic particles. Thixotropy and any other time dependent behavior are undesired, because in this case the rheological behavior would be dependent on the pre-treatment of the slurry.

On an atomistic scale the viscosity of a slurry is determined by small forces between the suspended particles. In an undisturbed slurry these forces build up an internal structure. With increasing shear rate the viscosity is reduced, until a constant level is reached. Dynamic measurements of the viscosity give information about the internal structure and its changes during shearing. For viscoelastic substances the shear module consists of two parts. The storage module G' describes the elastic properties of the substance. The loss module G'' gives the fraction of the applied stress which produces viscous deformation.

In this work aqueous tape casting slurries for the manufacturing of thin electrolytes for the Solid Oxide Fuel Cell (SOFC) [6], were examined by rheological measurements. Viscosity, its time dependent behavior and the strength of the internal structure of the slurries were characterized and discussed with regard to their usability for the tape casting process. Because binders strongly affect the rheological properties of slurries [7–9], the rheological behavior of the slurries was compared with those of the pure binder emulsion.

2. Experimental procedure

2.1. Starting materials and slurry formulation

Tape casting slurries were prepared from 8 mol% yttria stabilized zirconia (TZ8Y, Tosoh-Zirconia Corp., Japan), distilled water and a water-based polymeric emulsion (Mowilith DM765 S, Hoechst Perstorp, Sweden). The average particle size of the ceramic particles was $0.3\ \mu\text{m}$.² The binder emulsion contained 50 mass% polymer,² the binder tangles in the emulsion have a diameter of $0.05\text{--}0.15\ \mu\text{m}$.² The powder was dispersed by the addition of 2 wt.% of a polyelectrolyte (Dispex, Allied Colloids Ltd., UK) and ball milling for 66 h. Sedimentation tests and measurements of the zeta-potential showed that an excellent dispersion of the powder was achieved. After addition of the binder further mixing for 24 h was performed. Slurries with different contents of solid and binder were investigated for their rheological behavior (Table 1). The slurries only differ slightly in the composition but they show great differences in their rheological properties.

² Data from suppliers.

2.2. Rheological measurements

The experiments were carried out using a stress-controlled rheometer (UDS 200, Paar Physica, Austria) with a cone-plate system (angle 2.5°) at $22\ ^\circ\text{C}$. The viscosity, thixotropy and internal structure of the slurries were characterized.

After pre-shearing of the slurry at a shear rate of $5\ \text{s}^{-1}$ for 60 s, viscosity curves were measured by increasing the shear rate continuously in 120 s from 5 to $300\ \text{s}^{-1}$.

The thixotropy was characterized by the recovery time of the viscosity after a steep decrease from a high to a low shear rate: After pre-shearing at $300\ \text{s}^{-1}$ for 60 s the shear rate was immediately reduced to $1\ \text{s}^{-1}$. The dependence of the viscosity η on time can be described by an exponential function:

$$\eta(t) = A - B \exp\left(-\frac{t}{\text{THIX}}\right) \quad (1)$$

in which the parameters A and B are given by the viscosities before ($\eta(0) = A - B$) and after ($\eta(\infty) = A$) the steep decrease of the shear rate which occurs at the time $t = 0$. The variable THIX (dimension: seconds) characterizes the thixotropic behavior: with increasing value of THIX the thixotropic behavior of the slurry increases.

Dynamic measurements were carried out to characterize the changes of the internal structure of the slurry with time and to determine the strength of this internal network, which is given by a critical shear stress [10]. The experimental steps were as follows: pre-shearing at $100\ \text{s}^{-1}$ for 60 s, oscillation at a frequency of 1 rad/s ($\approx 0.17\ \text{Hz}$) with a low amplitude of 0.1 Pa to avoid destruction of the internal structure. During 25 min the value of the storage module was recorded. Directly after this the stress amplitude was increased logarithmically up to 500 Pa, at a constant frequency of 1 rad/s.

2.3. Tape casting, green sheets and sintered substrates

After degassing of the slurry tape casting was carried out on a tape casting machine with two stationary blades and with a moving polypropylene carrier film. The blade gap was adjusted to $150\ \mu\text{m}$. After drying at

Table 1
Composition of examined tape casting slurries

	Water (total) (vol.%)	Powder (vol.%)	Binder ^a + additives (vol.%)
Emulsion binder	50	—	50
Slurry A	53.4	18.0	28.6
Slurry B	51.3	18.5	30.2
Slurry C	52.7	18.8	28.5

^a Without water.

room temperature the geometric green density of the tape was measured. The thickness was determined with a microscope after breaking samples which were frozen in liquid nitrogen.

The binder burnout of the green sheets took place with a heating rate of 25 K/h up to 650 °C, followed by pre-sintering at 1200 °C for 3 h (heating rate 60 K/h). The final sintering was carried out in another furnace at 1400 °C for 3 h with a heating rate of 300 K/h.

The fracture surfaces of the sintered sheets were examined by scanning electron microscopy. Measuring the density after the Archimedes' principle gave great errors due to the very thin and light samples. A better wetting behavior of the liquid on the ceramic by using terpineol (Merck, Germany) improved the results only slightly. Thus, the sintered density could only be assessed by the porosity of the fracture surfaces.

3. Results

3.1. Rheological characterization

3.1.1. Viscosity curves

Fig. 1 shows the viscosity curves of the pure emulsion binder and the aqueous zirconia slurries. The emulsion binder itself already shows the desired strong pseudo-plasticity. The addition of ceramic particles increases the viscosity of the slurries, but their general rheological behavior is still like the one of the pure binder emulsion. The slurries only differ slightly in their compositions

(Table 1) but their viscosities and yield stresses are quite different.

The curves in Fig. 1 include information about a suitable casting speed. During tape casting the slurry exhibits a certain shear stress or shear rate, respectively, depending mostly on the casting velocity and gap height. One can estimate the shear rate during the tape casting process by the ratio of the tape velocity and the height of the blades. The casting speed should not be too small, because at these shear rates the gradient of the viscosity versus shear rate curve (Fig. 1) is very steep. This means that small changes of the shear rate result in great changes of the viscosity, and therefore in changes of the fluid mechanic conditions in the casting head. For this reason the shear rate must be high enough to be in the less steep range of the curve. For example, for slurry A this is approximately the range above 35 s⁻¹. Fig. 2 informs about the desired casting velocity to obtain such shear rates in dependence of the gap height. In the typical processing window for tape casting the viscosity is still on a relatively high level. At these shear rates the slurry can be cast, but the high viscosity hinders sedimentation.

The examined slurries have a yield stress like a Bingham body but they behave non-newtonian. There exist various models to describe this behavior. The Casson model [11] combines an empirical power-law function to describe the non-linear behavior ($\tau \propto \dot{\gamma}^n$) of a slurry with a yield stress τ_0 :

$$\sqrt{\tau} = \sqrt{\tau_0} + a\sqrt{\dot{\gamma}}. \quad (2)$$

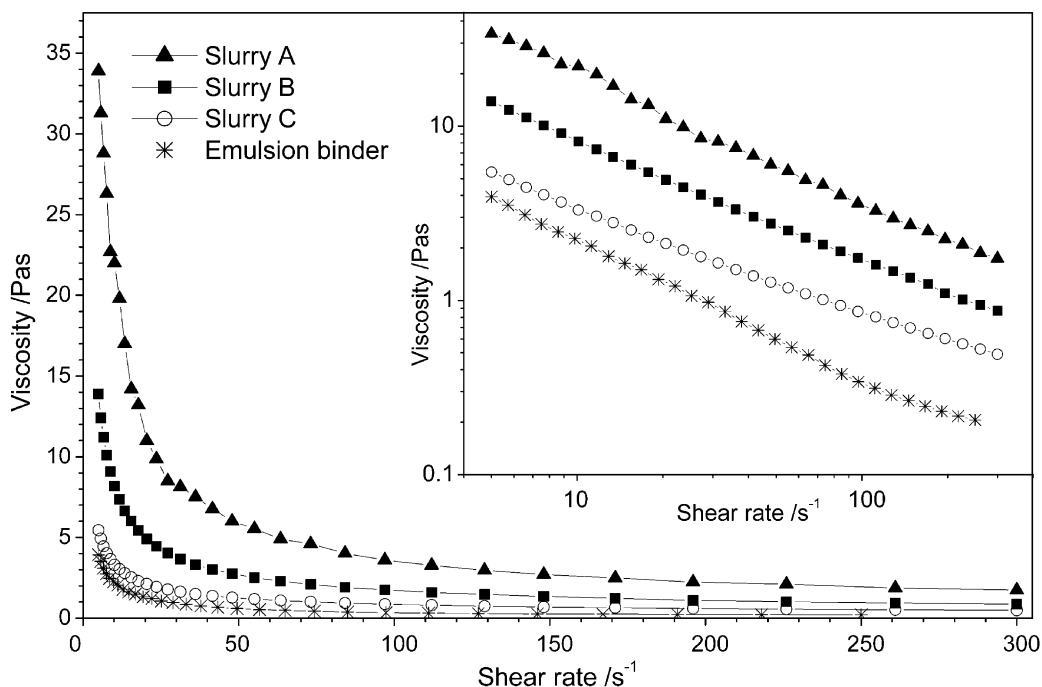


Fig. 1. Viscosity curves of the slurries after pre-shearing at 5 s⁻¹ for 60 s.

The parameter a is a constant. The slurry will not flow if the applied shear stress is lower than the yield stress τ_0 . Eq. (2) describes satisfactorily the properties of many filled systems over a rather wide range of shear rates [12]. Fitting the experimental data of Fig. 1 by Eq. (2) correlation coefficients of at least 0.99 are obtained indicating that the viscosity behavior of the examined slurries can be described very well by the Casson model (Table 2). The yield stresses τ_0 for the slurries given by Eq. (2) are relatively high. Nagata et al. [13] described that a high shear-thinning behavior and a high yield stress indicate a poor dispersion of the suspension. These characteristics are also valid for slurries A and B. This means that in spite of the good electrosteric dispersion of the powder binder-bridging flocculation occurs [13].

3.1.2. Time dependent behavior

The slurries exhibit scarcely no time dependent behavior, because the viscosity increases almost immediately after the steep decrease of the shear rate (Fig. 3). The experimental data were fitted using Eq. (1). As a result

Table 2

Yield stress and regression coefficient obtained by approximation of the measured viscosity curves after the Casson-model and parameter THIX obtained by thixotropy measurements

	τ_0 /Pa	Corr.Koeff.	THIX/s
Emulsion binder	17	0.988	0.12
Slurry A	140	0.990	0.16
Slurry B	56	0.992	0.16
Slurry C	19	0.995	0.06

the THIX values are obtained which represent the degree of thixotropy. These values are low (Table 2) which means, that the structure is able to rebuild itself in a very short time (< 1 s). These results were varified by additional experiments in which viscosity curves were measured at increasing and decreasing shear rates. No hysteresis could be detected which confirmed that the slurries exhibit the desired non-thixotropic behavior.

During a long time oscillation of 25 min almost no changes in the internal structure of the slurries occurred with time (Fig. 4). The storage module remained nearly constant during this period. This means that these slurries build up their internal structure very fast and do not change it afterwards. This time independent behavior is preferred in tape casting technology, because these slurries will not change their properties during processing. Slurry B exhibits a higher elastic behavior than slurry C or the pure binder emulsion. The high values of G' of slurry A could not be measured because the values were out of the instruments measuring range for these experimental conditions.

3.1.3. Internal structure

Fig. 5 shows the modules G' and G'' in dependency on the shear stress amplitude. The storage module G' of the slurries is much higher than that of the pure emulsion binder and reaches a maximum value of 4000 Pa. This is caused by the high solid content which increases the elastic properties of the slurry. At low shear stresses the modules remain nearly constant and G' is larger than G'' , which means that the viscoelastic slurries behave more elastic than viscous. The storage module G' exhibit a sudden decrease at a critical shear stress τ_C . The

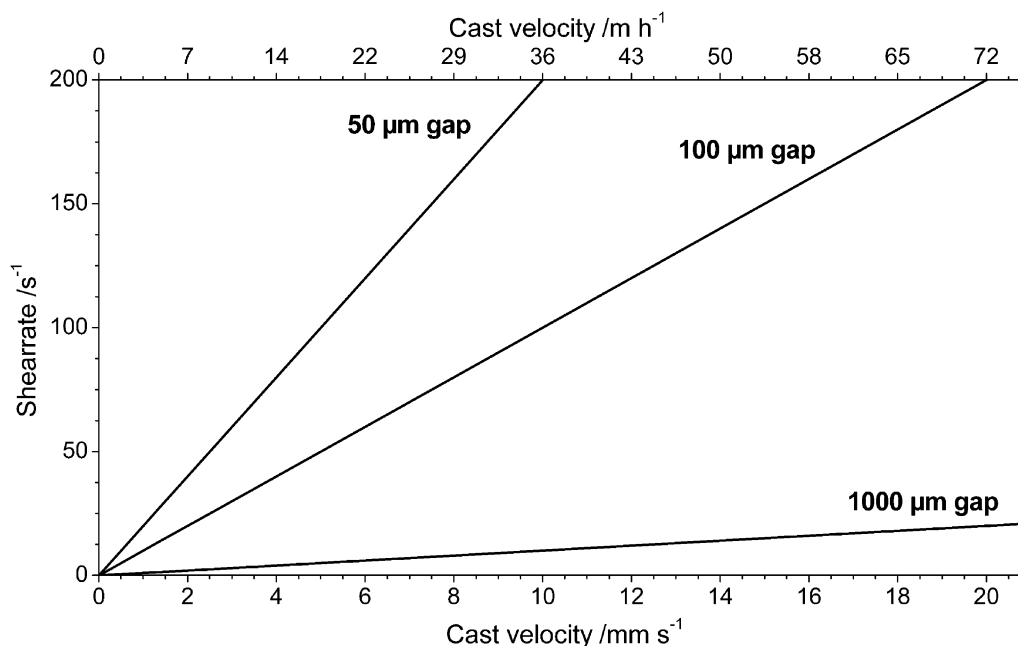


Fig. 2. Dependency of cast velocity, gap height and shear rate.

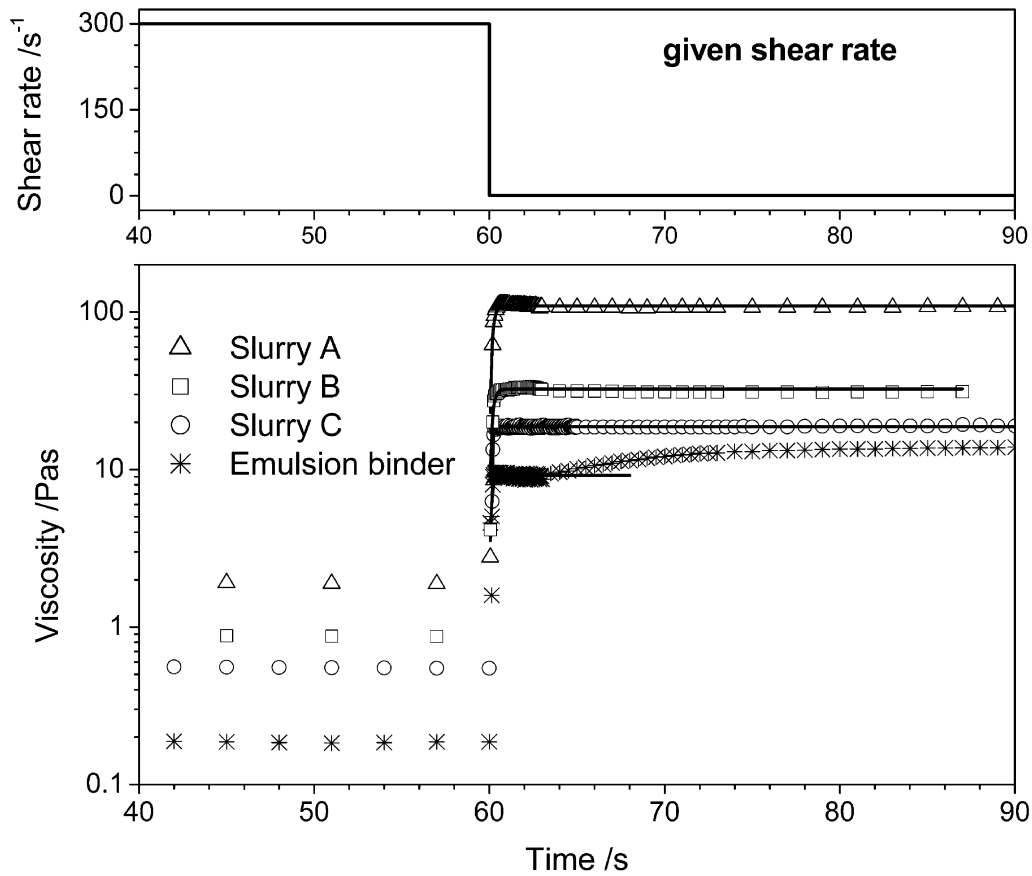


Fig. 3. Top: given shear rate: steep decrease from 300 to 1 s^{-1} at $t = 60$ s; bottom: viscosity increase of the slurries as a response to the change of the shear rate, (—: fitted functions).

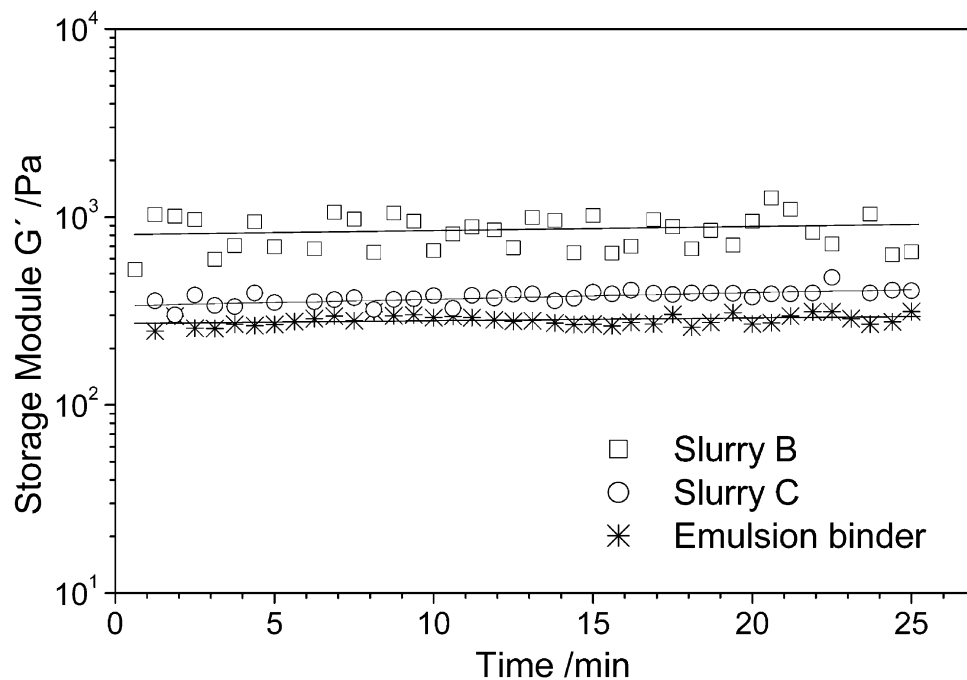


Fig. 4. Storage module G' as a function of time in long time oscillation experiments at a frequency of 1 rad/s with an amplitude of 0.1 Pa.

occurrence of this critical stress has its origin in the internal structure of the slurry [14]. Above τ_C the weak attractive forces between the powder and/or binder particles are broken up by the external shear stress, which destroys the internal network. The elastic properties of the slurry become very low and the storage module decreases more than three orders of magnitude.

The loss module G'' reaches a maximum at this point. This is also due to the breaking up of the weak interconnections between the particles in the slurry at these shear rates: the energy is used to break these networks. Energy which is now transferred into the system, cannot be stored as elastic deformation like before. The tendency for viscous deformation increases and both moduli decrease. The same behavior of the moduli was found in similar oscillation experiments with alumina slurries with the same binder [15,16].

For tape casting the slurry must spread the carrier to carry out the process. This means a shear stress greater than τ_C must be applied to the slurry. Table 3 lists the critical shear stresses of the examined substances taken

at the maximum of the loss module curves (Fig. 5). The values are comparable with the calculated yield stresses obtained from the viscosity curves (Table 2). The deviations are due to different pre-treatments and measuring methods [17], but the results show the same tendency: slurry A with the highest yield stress has the highest critical stress, slurry C has the lowest critical stress. For tape casting the critical shear stress τ_C should be high enough, thus the gravitational forces on the powder are too weak to cause viscous deformation in the slurry, which could result in sedimentation of the particles. On the other hand, τ_C must not be too high because the slurry must flow under the shear conditions which occur during passing the doctor blades. Slurry A has already a high value for τ_C . Higher values would lead to slurries which will not flow out of its reservoir under gravitational forces.

From oscillation experiments a complex viscosity was calculated [18]. And by dividing stress and absolute value of the complex viscosity $|\eta^*|$ a shear rate γ_C can be obtained (Table 3). This approximated value of the

Table 3
Critical shear stress, calculated complex viscosity and shear rate from the oscillation experiments

	τ_C /Pa	$ \eta^* /10^3$ Pa s	γ_C /s ⁻¹
Emulsion binder	5	0.2	0.02
Slurry A	165	0.8	0.07
Slurry B	65	0.7	0.03
Slurry C	15	0.3	0.03

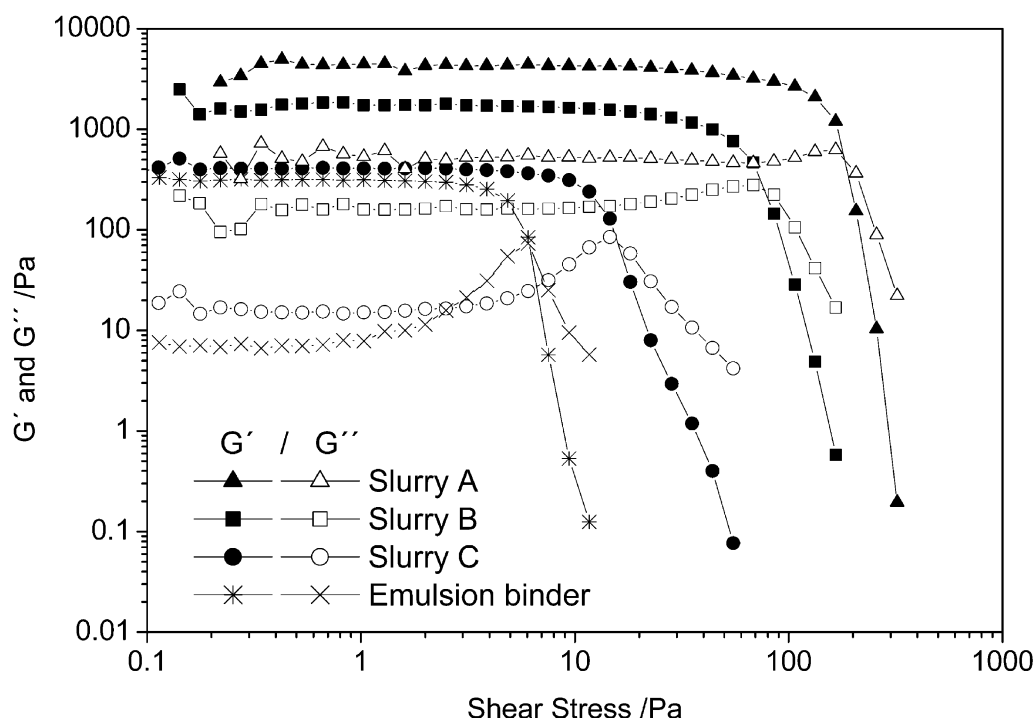


Fig. 5. Storage and loss module as a function of shear stress during oscillation at 1 rad/s with stepwise increasing stress amplitude.

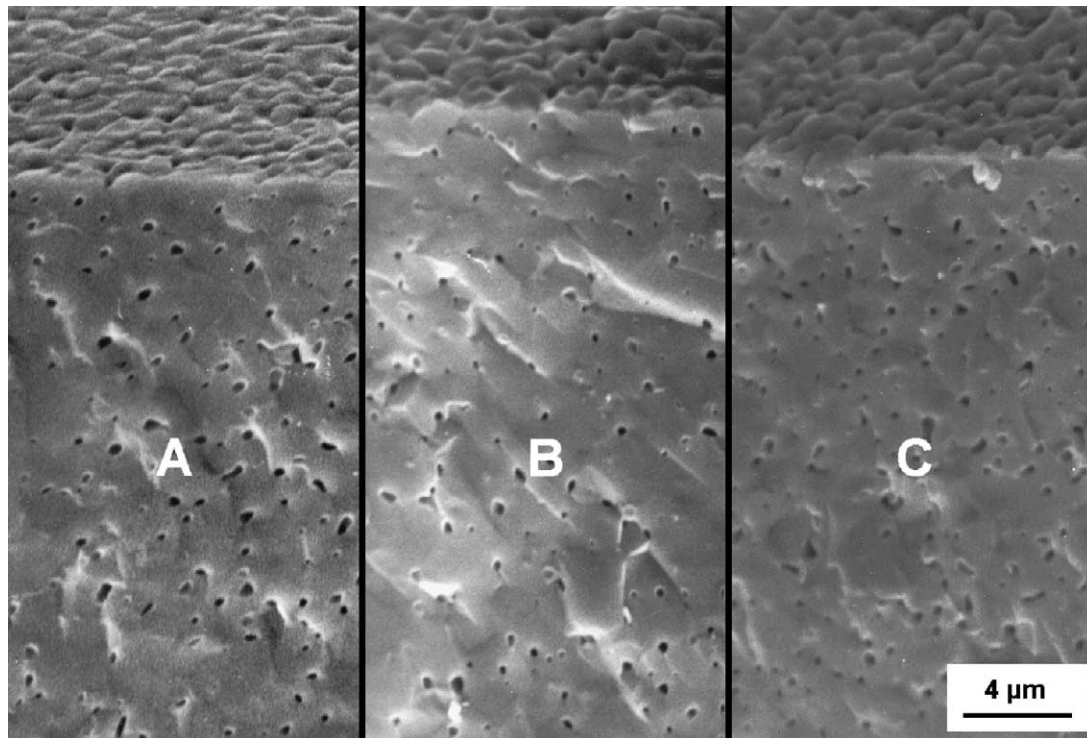


Fig. 6. Scanning electron micrographs of fracture surfaces of sintered sheets made of slurries A, B and C.

Table 4

Binder content, thickness, green density and linear shrinkage during sintering of green sheets of slurries A–C

	$V_{\text{powder}}/(V_{\text{binder}} + V_{\text{powder}})/\%$	Thickness/ μm	Green density/ g cm^{-3}	Sintering shrinkage ^a / $\%$
A	38.6	48	2.76 ± 0.08	26.9 ± 0.3
B	38.0	51	2.65 ± 0.09	31.3 ± 0.5
C	39.8	46	2.79 ± 0.06	25.3 ± 0.5

^a Linear shrinkage in horizontal direction.

shear rate can be used to determine the necessary cast velocity at a given gap height to overcome the critical stress. Very low shear rates are sufficient to break up the internal structure of the slurries. But independent of this, the viscosities of the slurries are still very high. Thus, greater shear rates are required to decrease the viscosity to get a castable slurry (s. Fig. 1).

3.2. Sintered substrates

After sintering a dense microstructure was obtained. Only a small amount of closed porosity remained (Fig. 6). The density was at least 97% of theoretical density. The substrates made of the different slurries hardly show any difference although the green sheets had different green densities (Table 4).

The different green densities of the green sheets were compensated by a different sintering shrinkage. The lower the green density the higher was the shrinkage during sintering (Table 4). The green density or the shrinkage seems only to be dependent on the powder to

binder ratio. Slurry C has the highest powder content and developed the highest green density and lowest sintering shrinkage. This is consistent with the thickness of the green sheets, which were all cast using the same height of the blades. With slurry B the lowest green density was obtained, which means that the thickness and shrinkage of the tape must be the highest.

4. Discussion

4.1. Rheological characterization

The presented results demonstrate that rheological measurements are an appropriate and important tool to decide if the rheological properties of a slurry match the demands of the tape casting process. These kind of measurements are of course applicable to other processes like, for example, slip casting or low pressure casting. To get the desired rheological information of a tape casting slurry it is sufficient to measure the viscosity

curves, to investigate the thixotropic behavior by time dependent experiments and the internal structure of the slurry by dynamic measurements.

The viscosity curves of the slurries showed the desired pseudoplastic behavior which is strongly determined by the pseudoplastic behavior of the pure emulsion binder with its very small polymer particles. This behavior is enhanced by the high content of fine grained ceramic powder.

The experimental condition to apply a steep decrease in shear rate to the slurry is a simple and fast test to examine the grade of thixotropy in slurry development. Furthermore the results of this test are relative independent of the type of measurement equipment used to characterize the rheological behavior of ceramic slurries. The slurries used for this study exhibit almost no thixotropic or any other time dependent behavior as it was also shown for a comparable powder by Fang et al. [19]. Such properties are desired for the tape casting process.

The internal structure of the slurries, examined by dynamical measurements without destroying the structure, showed almost no changes with time which indicates that the properties of the slurries will be constant during processing

But the slurries exhibit distinctive elastic properties below a critical shear stress τ_C indicated by a high storage module. Above τ_C the internal structure is broken up. This is necessary to increase the viscous behavior of the slurries to carry out the tape casting process. τ_C should be high enough to prevent sedimentation of the ceramic particles after casting the slurry. On the other side the viscosity must be low enough, thus the slurry can still be cast. It was observed that the binder content in the slurries tends to increase the internal structure much more than changes in the powder content. Thus, the viscosity of a slurry with a small water fraction, but a low binder to powder ratio, can be less than that of a slurry with a higher water fraction, but higher binder to powder ratio.

The investigated slurries show great differences between their rheological properties although their composition is quite similar. Nevertheless, all slurries match the demands for tape casting: pseudoplasticity, no thixotropy, time stability and a critical shear stress in ranges practicable for tape casting. All experiments clearly show that slurry A has the highest internal structure indicated by the highest viscosities, storage module, critical shear stress and grade of thixotropy, whereas slurry C has in all cases the lowest values even if slurry C exhibits the highest ratio of powder to organic additives. The low viscosity of slurry C has its origin in the fact, that it has the lowest amount of polymeric emulsion. It was shown, that the binder emulsion itself behaves strongly pseudoplastic, even at the high water amount of 50 vol.%, due to a relatively weak dispersion of the binder tangles. Thus, the higher the binder content in the slurry, the higher the viscosity.

The described characterization of the slurries demonstrates that slurry C is the most suitable slurry for tape casting. The data also showed that it is not sufficient to look at the composition of the slurry to choose the most suitable slurry for tape casting. Due to the different effects of water, binder and powder content and their interactions no simple correlation exists between composition and tape casting behavior.

4.2. Properties of tape cast sheets

All slurries were cast with the same parameters but the green densities of the green sheets are different due to the different composition and rheological behavior of the slurries. The sheets with the highest green density and therefore lowest sintering shrinkage were obtained from slurry C which had the lowest binder to powder ratio of the different slurry compositions. Because of the low viscosity slurry C gave the thinnest green sheet, too.

Slurry B has the highest binder to powder ratio. Therefore its green density was the lowest and the thickness of the green sheets was greater than that of the sheets from the other slurries. In contrast to this its exhibit only intermediate values. This is due to the small amount of water in this slurry (s. Table 1). During sintering a lower green density was compensated by a higher sintering shrinkage. Because of the highly sinter-active zirconia powder, the same dense microstructure was obtained for all compositions.

5. Conclusion

The paper demonstrates that the measurement of the viscosity, its time dependent behavior and the strength of the internal structure of the slurry are excellent tools to characterise a ceramic slurry with regard to its usability for the tape casting process. All investigated slurries match the demands for tape casting: pseudoplasticity, no thixotropy, time stability and a critical shear stress in a usable range.

The viscosity measurements of all investigated slurries showed the desired pseudoplasticity. This rheological behavior is caused by the highly pseudoplastic emulsion binder and the high content of submicron powder particles.

To check if the slurries exhibit any thixotropy effects or time dependent behavior, a steep decrease in shear rate is applied to the slurries. The time for the viscosity of the slurry to reach again a constant value quantifies the ability to rearrange their internal structure. The viscosity of the examined slurries followed almost immediately the change in shear rate indicating a negligible grade of thixotropy.

By observing the rheological properties without destroying the structure of the slurry during a long time oscillation measurement, it was confirmed that the

properties of the slurries will remain constant during the tape casting process.

Dynamical measurements with increasing shear amplitudes lead to a break-up of the internal structure at a certain shear stress, which quantifies the strength of the internal structure. On the one hand this critical shear stress must be low enough thus the slurry can be cast. On the other hand, a sufficient high critical shear stress shall prevent sedimentation of the powder particles due to gravitational forces. The investigated slurries fulfilled these conditions. In addition it was found that the strength of the internal structure of the investigated slurries was more dominated by the binder content than by the amount of water or powder.

Green density and thickness of the tape cast sheets was influenced by the rheological properties of the slurries. A high green density reduces the sintering shrinkage. The maximum green density was obtained from the slurry with the highest powder to binder ratio (slurry C). This slurry also showed the lowest viscosity and weakest internal structure.

The data showed that it is not sufficient to look at the composition of the slurry to choose the most suitable slurry for tape casting. Due to the different effects of water, binder and powder content and their complex interactions no simple correlation can be made between composition and tape casting behavior.

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