RHEOMETRY OF CONCENTRATED CERAMIC SUSPENSIONS - STEPS FROM MEASURED TO RELEVANT DATA PART 2. ROTATIONAL VISCOMETER WITH COAXIAL CYLINDERS - BINGHAM MODEL

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The knowledge of flow behavior of ceramic suspensions is an important condition for control of ceramic wet processing. The flow behavior can be stated by the rheological measurements. The aim of the measurements was to obtain the flow curve and to propose suitable and simple rheological model for the flow behavior description. The Bingham model is the simplest model used for description of rheological behavior of plastic ceramic materials. Using this model, the relation of shear stress τ *and shear rate* $\dot{\gamma}$ *can be expressed by the following relation:* $\tau = \mu_p\dot{\gamma} + \tau_0$ *for* $|\tau| \geq \tau_0$ *where* τ_0 *is the yield stress and* μ_p *stands for plastic viscosity. The several arrangements of rotational viscometer method with coaxial cylinders were used in rheological measurements. The procedure of experimental data evaluation from rotational viscometer with coaxial cylinders to obtain parameters* τ_0 *and* μ_p *is presented.*

INTRODUCTION

Dry powder processing is currently the main technique employed in the ceramics industry for the manufacture of ceramic materials. There is a trend to move towards wet colloidal processing in order to overcome problems of microstructural heterogeneity or flaws associated with dry powder processing [1]. Wet colloidal processing uses surface chemistry controls to manipulate interparticle forces in order to achieve the desired rheological properties and solid concentration. Excellent control of the interparticle forces is essential for good processing [2]. In the manufacture it is desirable to have the ability to tailor the rheological properties of the suspensions to suit the particular process. Knowledge of rheological behaviour is essential in designing or selecting equipment for storing, pumping, transporting, milling, mixing, atomizing, and forming a ceramic system. Rheological measurement are a integral part of the research and development of slurry systems, and rheological tests are used in programs for monitoring and controlling the consistency and behaviour of slurries for casting, spray-drying, or glazing and pastes for surface printing and decorating.

Ceramic slurries are commonly multicomponent systems that are relatively complex in structure and often poorly characterized, Particles may range from granular sizes to colloids. Added electrolytes and polymers may significantly change interparticle forces and state of dispersion. The interparticle spacing depends directly on the concentration of particles (solid loading), the state of dispersion, and the particle packing. Particle coagulation and polymer flocculation may produce a structural linkage, a gel structure that varies with the mixing procedure and the shear history prior to measuring the rheological properties. Flow produced during a rheological measurement may alter the microstructure system [3].

In this work we will discuss fundamentals and techniques used to determine the relevant rheological properties of these important systems. The first part of our series dealing with the rheometry of concentrated ceramic suspensions was presented in [4].

THEORETICAL

The rotational viscometer method with coaxial cylinders is mostly used in rheological measurements. The aim of the measurement is to obtain the flow curve and to propose suitable and simple rheological model for the flow behavior description.

The Bingham model is the simplest model used for description of rheological behavior of plastic ceramic materials. Using this model, the relation of shear stress τ and shear rate $\dot{\gamma}$ can be expressed by the following relation:

$$\tau = \mu_p \dot{\gamma} + \tau_0 \quad for \quad |\tau| \geq \tau_0 \tag{1}$$

where τ_0 is the yield stress and μ_p stands for plastic viscosity. The common configuration of rotational viscometer with coaxial cylinders consists of an inner rotating cylinder with radius R_1 and the outer stationary cylinder with the radius R_2 - see Figure 1. The values of shear stress τ_1 and Newtonian shear rate $\dot{\gamma}_{1N}$ on inner cylinder are usually obtained from rheological measurements, where the following relation was reported in [5]

$$\dot{\gamma}_{1N} = \frac{2\omega}{\left(1-\kappa^2\right)} \tag{2}$$

where ω is angular velocity of inner cylinder and κ is inner to outer radius ratio.

As it was shown in [5] when $\dot{\gamma}_{1N}$ exceeds its critical value

$$\dot{\gamma}_{1Nk} = \left(\frac{1}{\kappa^2} - \frac{2}{1-\kappa^2}\ln\frac{1}{\kappa}\right)\frac{\tau_0}{\mu_p} \tag{3}$$

the measured material flows in the whole gap between cylinders. The comparison of Newtonian and Bingham velocity profiles calculated by equations presented in [5] for case that $\dot{\gamma}_{1N} \geq \dot{\gamma}_{1Nk}$ and $\kappa = 0.75$ is shown in Figure 2. In this figure $u^* = u/u_1$ (u is tangential velocity and u_1 tangential velocity of inner cylinder) and the dimensionless coordinate y^* is defined by relation $y^* = y/(R_2 - R_1)$ (y is radial distance from rotating cylinder). From this figure it can be seen that Bingham's velocity profile is more curved than Newtonian one and from it follows that also real shear rate on inner cylinder surface $\dot{\gamma}_1$ is greater than Newtonian $\dot{\gamma}_{1N}$.

It was also shown in [5] that in case that $\dot{\gamma}_{1N} \geq \dot{\gamma}_{1Nk}$, the dependence of τ_1 on $\dot{\gamma}_{1N}$ is linear and can be described by equation

$$\tau_1 = \tau_{01} + \mu_p \dot{\gamma}_{1N} \tag{4}$$

where

$$\tau_{01} = 2\frac{\ln(1/\kappa)}{1-\kappa^2}\tau_0 \tag{5}$$

From Equation (4) it can be seen that dependence of τ_1 on $\dot{\gamma}_{1N}$ is a straight line with the slope μ_p and intercept τ_{01} from which value τ_0 can be calculated on the basis of Equation (5).

The dependence of τ_{01}/τ_0 ratio on κ is shown in Figure 3. From this figure it follows that the value of τ_{01}/τ_0 ratio increases with decreasing κ. From Figure 3

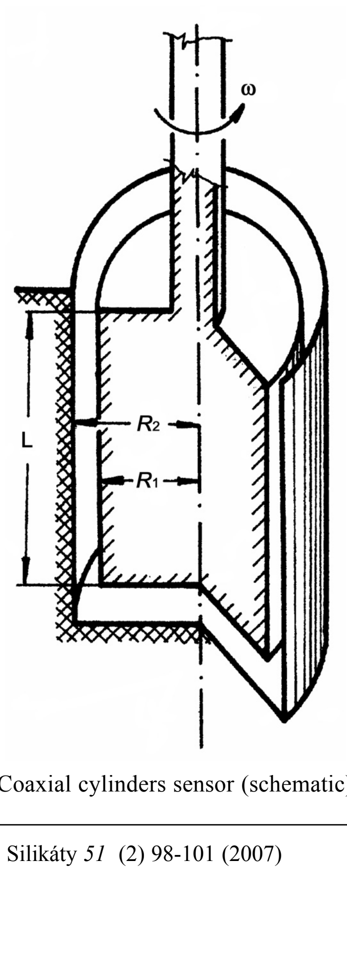

Figure 1. Coaxial cylinders sensor (schematic).

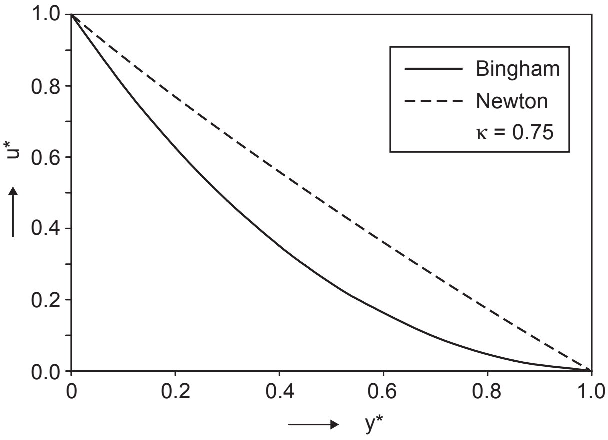

Figure 2. Comparison of Newtonian and Bingham velocity profiles.

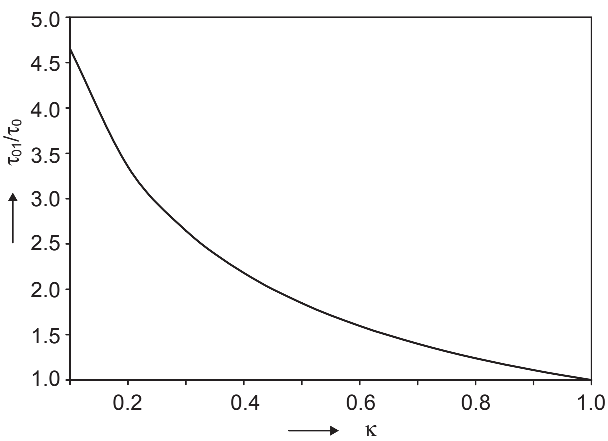

Figure 3. Dependence of τ_{01}/τ_0 ratio on κ value.

and Equation (5) it follows that for systems with $\kappa < 0.97$ the ratio τ_{01}/τ_0 is greater than 1.03. It means that measurements for such systems should be corrected if error less than 3% is required.

Combining Equations (1) (4) and (5) the following relation between values of shear rates holds

$$\dot{\gamma}_1 = \dot{\gamma}_{1N} + \frac{1}{\mu_p}(\tau_{01} - \tau_0) = \dot{\gamma}_{1N} + \frac{\tau_0}{\mu_P}\left(\frac{2\ln(1/\kappa)}{1-\kappa^2} - 1\right) \quad (6)$$

EXPERIMENTAL

Rheological behavior of 2 types of clay and water suspension was determined in experiments. A result of crystalline phase identification by X-ray diffraction of both clays is shown in Table 1. The particle size distribution of clay 1 is shown in Figure 4. This distribution was obtained from measurement in particle size analyzer A22 Compact (Fritsch). The mean volumetric particle diameter d_p = 3.2 µm was also obtained from this analysis. The particle size distribution of clay 2 is shown in Figure 5. The mean volumetric particle diameter d_p = = 6.5 µm was received for clay 2.

The particles in suspension didn't settle thanks to the size and concentration and therefore rotary rheometer with coaxial cylinders could be used for measurements. The measured suspension samples were prepared by gradual adding of clay into the batch mixed by high shear stress impeller. After adding all necessary solid phase, the batch has been dispersed for several minutes in addition. This procedure was done to prepare homogenous suspension without particle conglomerates, which appear in such concentrated fine particle suspensions and which would influence the rheological measurements.

Table 1. Phase composition of studied clay types.

Phase	Clay 1	Clay 2
Clay minerals	59 wt. %	78 wt. %
Quartz	41 wt. %	22 wt. %

The suspension of clay 1 with mean volumetric concentration 40 % vol. of solid phase was used in the rheological experiments. The experiments were carried out using rheometer RV1 (Haake) and by usage two configurations of cylinder system with different diameters ratio. The diameter ratio was κ = 0.72 at configuration Z31 and κ = 0.95 at configuration Z41. The suspension of clay 2 with mean volumetric concentration 30 % vol. was used in the rheological measurements. The experiments were carried out using two different rheometers: RC20 (RheoTec) and Rheotest 2 (VEB MLW). One configuration CC48 of coaxial cylinders with diameters ratio κ = 0.98 was used with rheometer RC20. Two configuration of cylinder system with different diameters ratio were used in measurements with Rheotest 2. At configuration S1 the diameter ratio was κ = 0.98 and at configuration S2 the ratio was κ = 0.94. Rheometers RC20 and RV1 were controlled by PC and the measurement were done automatically according to a predefined measure program of shear rate course. Measurements with rheometer Rheotest 2 were done manually.

RESULTS

The experimental dependences of τ_1 on $\dot{\gamma}_{1N}$ obtained by measurements with suspension of clay 1 in configrations Z31 and Z41 are depicted in Figure 6. The values obtained at $\dot{\gamma}_{1N} \geq \dot{\gamma}_{1Nk}$ were evaluated by Equation (4) and corresponding equations obtained by linear regression are presented in this figure.

The real values of $\dot{\gamma}_1$ were calculated from equation (6). The dependence τ_1 on $\dot{\gamma}_1$ with Equation (1) characterizing rheological behavior of measured suspension is shown in Figure 7.

The similar dependences obtained by measurements with suspension of clay 2 are presented in Figures 8 and 9.

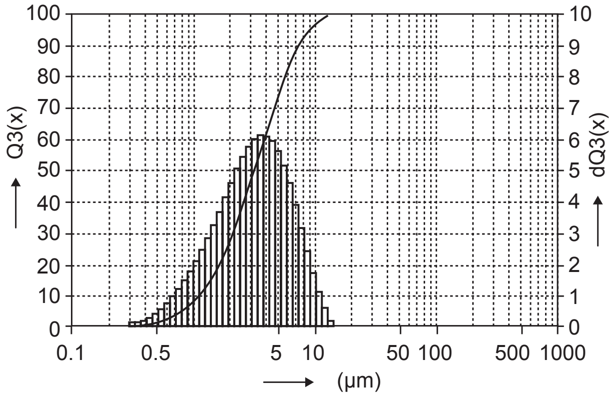

Figure 4. Particle size distribution of the clay 1.

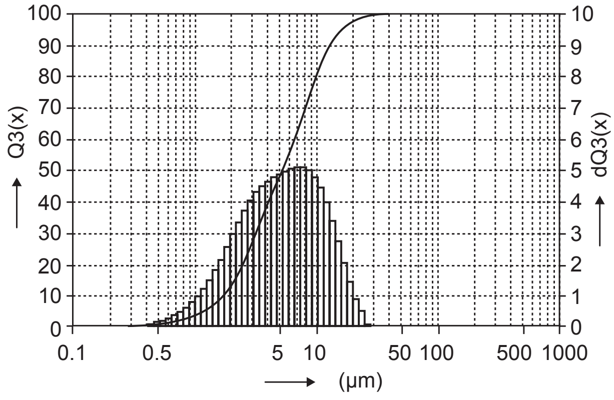

Figure 5. Particle size distribution of the clay 2.

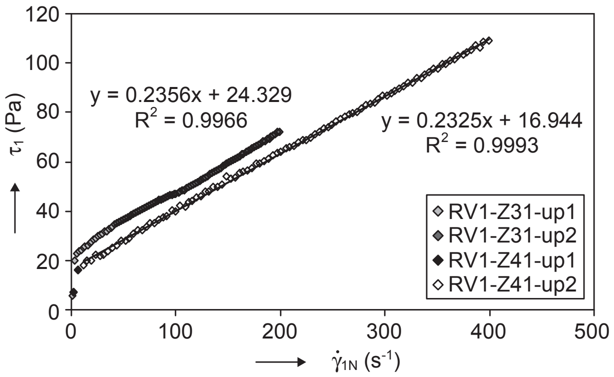

Figure 6. Experimental dependences of τ_1 on $\dot{\gamma}_{1N}$ obtained by measurements with suspension of clay 1 in configurations Z31 and Z41.

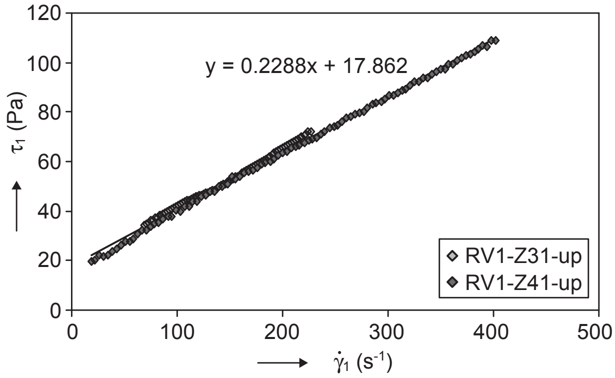

Figure 7. Real flow curve for suspension of clay 1 (interlayed by Bingham model).

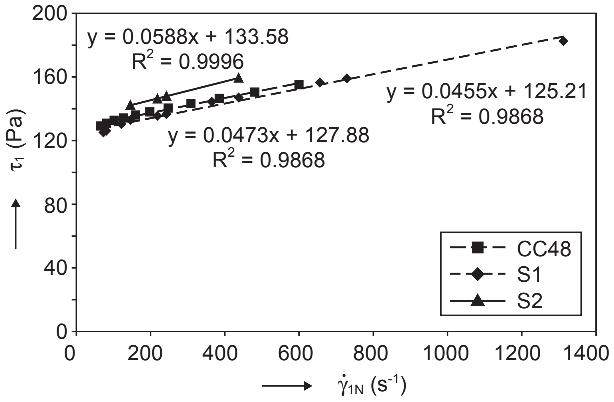

Figure 8. Experimental dependences of τ_1 on $\dot{\gamma}_{1N}$ obtained by measurements with suspension of clay 2.

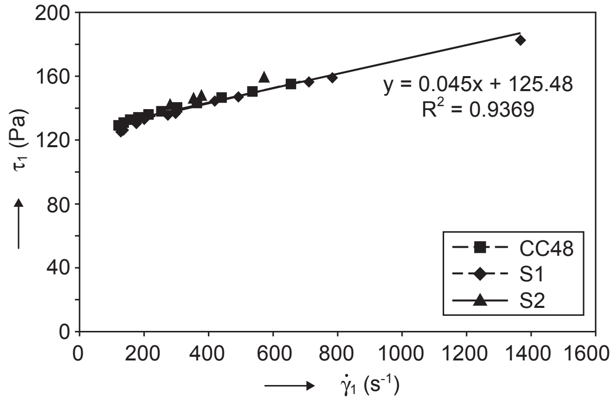

Figure 9. Dependence τ_1 on $\dot{\gamma}_1$ for suspension of clay 2.

CONCLUSIONS

The following procedure can be recommended for measurements evaluation. The values μ_p and τ_{01} can be obtained by linear regression of measured data τ_1 and $\dot{\gamma}_{1N}$ (see equation (4)). The yield stress τ_0 can be calculated from τ_{01} using equation (5).

When measurements with more coaxial cylinders configurations are done, the values of real shear rates on inner cylinder $\dot{\gamma}_1$ should be calculated from equation (6) and all couples τ_1 and $\dot{\gamma}_1$ should be depicted in common diagram, from which resulting values μ_p and τ_0 can be obtained by linear regression of Equation (1) related to radius R_1.

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References

1. Lange, F.F.: J.Am.Ceram.Soc. *72*, 3 (1989).
2. Leong, Y.K.: Materials & Design *15*, 141 (1994).
3. Reed J. S., Introduction to the Principles of Ceramic Processing, p.227, J. Wiley & Sons, Inc., New York 1988.
4. Andertová J., Rieger F.: Ceramics-Silikáty *50*, 163 (2006).
5. Rieger, F., Mašín, L.: Acta Technica ČSAV *35*, 129(1990).

REOMETRIE KONCENTROVANÝCH KERAMICKÝCH SUSPENZÍ - OD MĚŘENÝCH K RELEVANTNÍM HODNOTÁM. ČÁST 2 - ROTAČNÍ VISKOZIMETR SE SOUOSÝMI VÁLCI - BINGHAMSKÝ MODEL

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Znalost tokového chování keramických suspenzí je důležitým předpokladem řízení keramických technologií zabývajících se zpracováním keramických prášků v kapalné fázi. Tokové chování systémů se stanoví reologickými měřeními. Práce se zabývá měřením reologického chování koncentrovaných keramických suspenzí. Je uveden postup vyhodnocení naměřených dat na rotačním viskozimetru se dvěma souosými válci pro suspenze, jejichž tokové chování (závislost smykového napětí τ na smykové rychlosti $\dot{\gamma}$) je popsáno binghamským modelem ve tvaru $\tau = \mu_p\dot{\gamma} + \tau_0$ pro $\tau \geq \tau_0$, kde τ_0 je počáteční napětí a μ_p plastická viskozita. Měření byla provedena na několika uspořádáních rotačního viskozimetru se souosými válci. Je navržena korekce dat získaných přímým měřením pro nenewtonské látky s cílem získat relevantní tokovou křivku a určit parametry binghamského modelu.