

THE EFFECT OF TEMPERATURE ON RHEOLOGICAL PROPERTIES OF CERAMIC PASTE

JANA TRÁVNÍČKOVÁ, JIŘÍ HAVRDA, FRANTIŠEK OUJÍŘÍ

*Department of Silicate Technology, Institute of Chemical Technology,
Suchbátarova 5, 166 28 Prague 6*

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The effect of temperature on the value of apparent viscosity was studied on a ceramic paste based on α -Al₂O₃—ZrO₂. This relationship was evaluated by the capillary viscometer method. It was found that over the temperature range involved, the paste was described by the constitutive equation of a general viscoplastic liquid with temperature-dependent parameters. The power law index and the yield stress are linear functions of temperature and the consistency parameter depends exponentially on temperature.

INTRODUCTION

Knowledge of the constitutive equation and the temperature dependence of apparent viscosity are prerequisites for a quantitative description of the non-isothermal flow of ceramic paste, or of isothermal flow at various paste temperatures. A change in paste temperature results from energy dissipation during the flow. The change in temperature is also associated with a change in properties, particularly that of apparent viscosity. Under isothermal conditions, the apparent viscosity or its dependence on the shear rate can be determined by the capillary viscometer method [1]. Measurements of the rheological behaviour of ceramic pastes indicate that the pastes exhibit a yield stress. Their behaviour is best described by the constitutive equation in the following form:

$$\tau = \tau_0(T) + \eta(S, T) \gamma, \quad (1)$$

where τ is stress, τ_0 is the yield stress, γ is the shear rate; for apparent viscosity it holds that

$$\eta(S, T) = K(T) S^{(n(T)-1)/2}, \quad (2)$$

where K is the consistency parameter, n is the power law index, and for parameter S it holds that

$$S = \gamma^2. \quad (3)$$

The present study had the purpose to determine the effect of temperature on the parameters of equation (2) and on the value of yield stress τ_0 .

TEMPERATURE DEPENDENCE OF THE PARAMETERS OF THE CONSTITUTIVE EQUATION

The available findings of the effect of temperature on the parameters of equation (2) indicate that the assumption of their independence of temperature is met only with some liquids and within a narrow temperature interval (2, 3, 4). With respect to expressing the temperature dependence of apparent viscosity it was found [5]

that a satisfactory fit is obtained with the relationship analogous to the exponential function of a purely viscous liquid in the form

$$\eta = A_1 \exp(B_1/(T - T_0)), \quad (4)$$

where A_1 , B_1 and T_0 are constants. Study [6] established a significant temperature dependence of the consistency parameter K . Compared to this, the temperature dependence of yield stress, τ_0 , and that of the power law index, are negligible. The explanation is based on the idea that the yield stress is brought about by temperature-independent mechanical solidification of the liquid. Generalization of this idea was denied by the discovery of liquids showing a temperature dependence of τ_0 [7, 8, 9, 10, 11, 12], expressed by the equation

$$\tau_0(T) = \tau_{0i} T_i / T, \quad (5)$$

or

$$\tau_0(T) = \tau_{0i} \exp[-Z(T - T_i)], \quad (6)$$

where τ_{0i} is the yield stress value at temperature T_i , and Z is a constant.

The temperature dependence of the consistency parameter K is given in the form

$$K(T) = A \exp(B/T), \quad (7)$$

or

$$K(T) = K_i / [1 + Z(T - T_i)^n], \quad (8)$$

where K_i is the value of the consistency parameter at temperature T_i , and n is the power law index.

EXPERIMENTAL

The temperature dependence of the parameters of equation (2) and that of $\tau_0 = f(T)$ was carried out by the capillary viscometer method [1]. Capillaries having the diameter $D = 4.2 \times 10^{-3}$ m and lengths $L_1 = 5 \times 10^{-2}$ m, $L_2 = 4 \times 10^{-2}$ m, $L_3 = 8 \times 10^{-2}$ m and $L_4 = 10 \times 10^{-2}$ m were used in the measurements. The mean flow velocities of the paste through the capillary varied over the interval of $\bar{v}_x \in \langle 9.92 \times 10^{-4} \text{ m s}^{-1}; 3.92 \times 10^{-2} \text{ m s}^{-1} \rangle$. The mixture of $\alpha\text{-Al}_2\text{O}_3$ (80 wt. %) and ZrO_2 (20 wt. %), cf. Table I, was prepared by homogenizing

Table I
Characteristics of the oxides employed

Designation	Particle size μm	Content %	Specific surface area g m^{-2}
$\alpha\text{-Al}_2\text{O}_3$	0.1—0.4	83	10.8
ZrO_2	0.1—0.6	68	15.0

in ethyl alcohol for 48 hours. After drying, the surface of the particles was activated by an addition of organic substances. The mixture was pasted by an addition of 10 wt. % plasticizer. The tank of the capillary viscometer, filled with the evacuated paste, was tempered in a thermostat at working temperature T , which was varied over the interval of $\langle 19^\circ\text{C}; 29^\circ\text{C} \rangle$. In the course of the experiment, maintenance of isothermal flow was checked by measuring temperature at the capillary inlet

and outlet. The data obtained by the measurement were then processed by the procedure described in [1]. The resulting relationship

$$p = f(4\bar{v}_x/R), \tag{9}$$

where p is pressure, R is the capillary radius, was corrected for terminal effects ($\Delta p = p_{L_i} - p_{L_{i+1}}$; $\Delta L = L_i - L_{i+1}$). On introducing

$$\tau_s = R \Delta p / 2L, \tag{10}$$

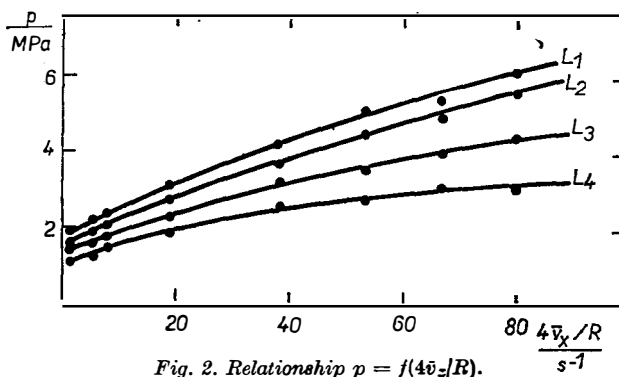
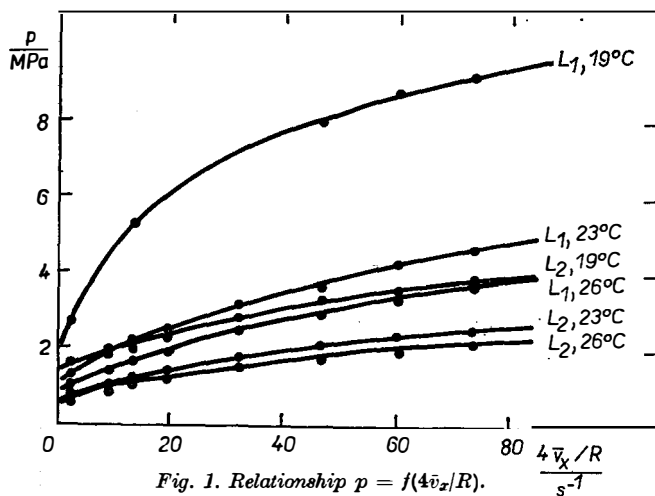
the following relationship was evaluated:

$$\tau_s = f(4\bar{v}_x/R), \tag{11}$$

where τ_s is shear stress at the capillary wall. Conversion of the quantity $(4\bar{v}_x/R)$ to the shear rate at the wall $(dv_x/dr)_s$ yielded the diagram of the constitutive equation, i.e. the function

$$\tau_s = f(dv_x/dr)_s. \tag{12}$$

The experimental values (9) and the evaluated ones (11), (12) are plotted in Figs. 1, 2, 3 and 4 for the individual temperatures.



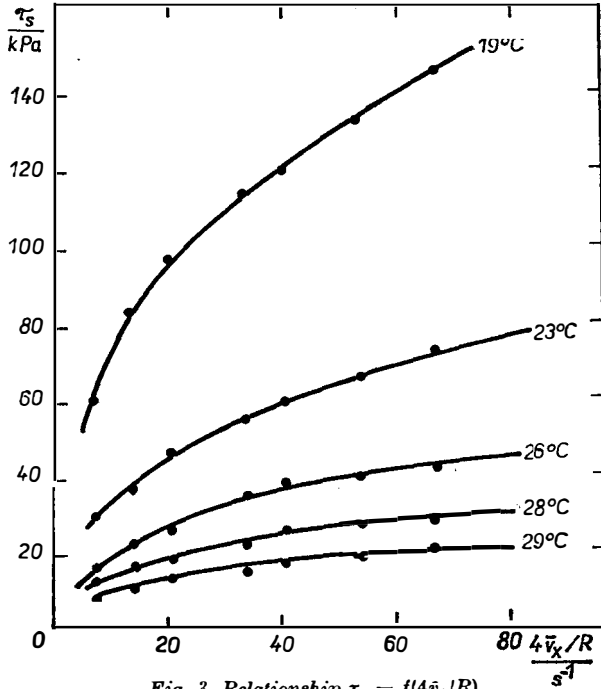


Fig. 3. Relationship $\tau_s = f(4\bar{v}_x/R)$.

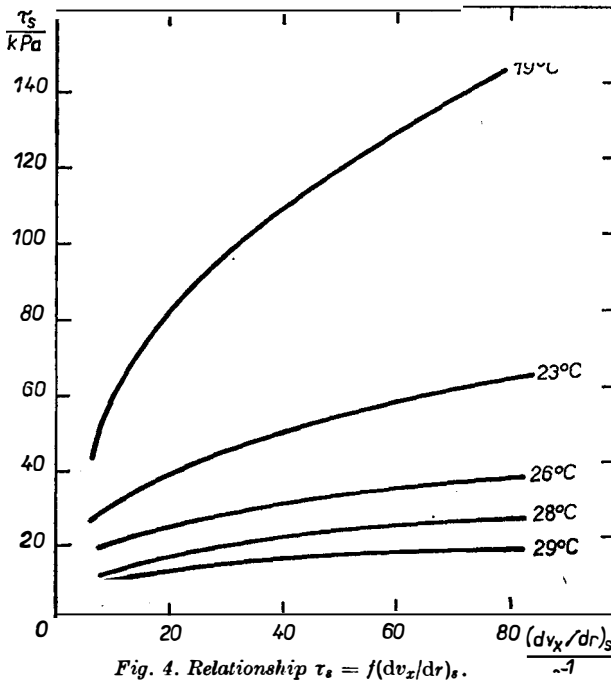


Fig. 4. Relationship $\tau_s = f((dv_x/dr)_s)$.

CONCLUSION

The graphic plot of the constitutive equations in Fig. 4 was approximated by equations (1) and (2) for all the temperatures using the values of constants listed in Table II. The temperature dependences of the constants listed in Table II were approximated in the following forms:

$$K = 0.845 \exp(56/T) \text{ kPa s}^n, \quad (13)$$

$$n = 0.98 - 2.55 \times 10^{-2}T, \quad (14)$$

$$\tau_0 = (26.86 - 0.84T) \text{ kPa}. \quad (15)$$

Table II

Values of the constants of the constitutive equation for $\alpha\text{-Al}_2\text{O}_3\text{-ZrO}_2$ paste at various temperatures

T °C	K kPa s ⁿ	n	τ_0 kPa
19	16.32	0.487	10.30
23	9.06	0.410	8.20
26	8.00	0.305	5.80
28	6.00	0.297	2.90
29	5.80	0.215	2.10

It can be concluded that a change in temperature over the interval $T \in \langle 19^\circ\text{C}; 29^\circ\text{C} \rangle$ does not bring about any change in the material model in the case of the $\alpha\text{-Al}_2\text{O}_3\text{-ZrO}_2$ paste. The temperature dependence of the constants of equations (1) and (2) are described by equations (13), (14) and (15), and their courses are in agreement with the characteristics of these relationships published so far.

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VLIV TEPLOTY NA REOLOGICKÉ VLASTNOSTI KERAMICKÉ PASTY

Jana Trávníčková, Jiří Havrda, František Oujíří

Katedra technologie silikátů, Vysoká škola chemicko-technologická, 166 28 Praha 6

V práci je sledován vliv teploty na reologické chování keramické pasty na bázi $\alpha\text{-Al}_2\text{O}_3\text{—ZrO}_2$. Metodou kapilárního viskozimetru byla pro různé teploty pasty určena závislost viskozity na gradientu rychlosti deformace a vyhodnocena konstitutivní rovnice. Bylo zjištěno, že v proměřeném rozsahu teplot $T \in < 19^\circ\text{C}; 29^\circ\text{C} >$ je chování pasty popsáno konstitutivní rovnicí obecné viskoplastické kapaliny s teplotně proměnnými parametry. Idnex toku n a mez toku τ_0 jsou lineární funkcí teploty, koeficient konzistence K závisí na teplotě exponenciálně. Průběhy teplotních závislostí reologických parametrů jsou v souladu s publikovaným charakterem těchto závislostí.

Obr. 1. Závislost $p = f(4\bar{v}_x/R)$.

Obr. 2. Závislost $p = f(4\bar{v}_x/R)$.

Obr. 3. Závislost $\tau_s = f(4\bar{v}_x/R)$.

Obr. 4. Závislost $\tau_s = f(d\bar{v}_x/dr)_s$.

ВЛИЯНИЕ ТЕМПЕРАТУРЫ НА РЕОЛОГИЧЕСКИЕ СВОЙСТВА КЕРАМИЧЕСКОЙ ПASTЫ

Яна Травничкова, Иржи Гаврда, Франтишек Оуири

Кафедра технологии силикатов Химико-технологического института 166 28 Прага 6

В работе исследуется влияние температуры на реологическое поведение керамической пасты на базе $\alpha\text{-Al}_2\text{O}_3\text{—ZrO}_2$. Посредством капиллярного вискозиметра установили для разных температур пасты зависимость вязкости от градиента скорости деформации и предложили конstitutivное уравнение. Было установлено, что в измеряемом диапазоне температур $T \in < 19^\circ\text{C}; 29^\circ\text{C} >$ поведение пасты описывается конstitutивным уравнением общей вискозопластической жидкости с температурно изменяемыми параметрами. Показатель потока и предел потока τ_0 являются линейными функциями температуры, коэффициент консистенции K зависит от температуры экспоненциально. Ходы температурных зависимостей реологических параметров находятся в согласии с опубликованным характером данных зависимостей.

Рис. 1. Зависимость $p = f(4\bar{v}_x/R)$.

Рис. 2. Зависимость $p = f(4\bar{v}_x/R)$.

Рис. 3. Зависимость $\tau_s = f(4\bar{v}_x/R)$.

Рис. 4. Зависимость $\tau_s = f(d\bar{v}_x/dr)_s$.

METÓDA DYNAMICKÉHO VÁŽENIA využívajúca zmeny rezonančnej frekvencie kremenného výbrusu získaného v rovine AT patrí medzi bežne používané metódy merania hrúbky a rýchlosti nanášania tenkých vrstiev. Tento spôsob sledovania hrúbky naparovaných tenkých vrstiev využíva i zariadenie MSV 1843/M vyrábané Ústavom pre prístrojovú techniku — MIKI v Budapešti. Prístroj automaticky riadi proces naparovania tenkých vrstiev pre aplikácie v elektronike a optike. Spracovanie výsledkov merania v mikroprocesorovej jednotke umožňuje určiť rýchlosť naparovania s rozlíšením $0,01 \text{ nm s}^{-1}$. Naparovacia automatika umožňuje naprogramovanie až 15 parametrov pre každú vrstvu a uchovanie týchto parametrov až pre 10 technologických postupov nanášania tenkých vrstiev. Hrúbku vrstiev je možné merať v intervale $0\text{—}99,99 \mu\text{m}$, rýchlosť naparovania je nastaviteľná od $0,01$ do $99,99 \text{ nm s}^{-1}$. (Sďelovací technika 3/88)

M. Uhrík