SIMPLE PUSH-ROD DILATOMETER FOR DILATOMETRY OF CERAMICS

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Abstract
The push-rod dilatometer with inductive transducer and alumina rod arrangement is appropriate instrument for evaluating the dilatometric behavior of fired and green ceramics. The calibration was carried out by the help of the reference steel NiCrMoTiB sample. Also, the influences of the thermal field inhomogeneity, zero drift, temperature shift, mechanical load on the sample, position of the sample, as well as accuracy of the measurement of the sample length and temperature were analyzed and evaluated. The relative uncerntainty of the measurement was calculated, its value is 0.58 %.

Key words: dilatometer, calibration, uncerntainty

1 Introduction

An accurate understanding of behavior of ceramics can provide insights on firing processes, the influence of additives and raw materials, densification and sintering properties, reaction kinetics, phase transitions, glaze development, and thermal shock. Thermodilatometry is very suitable method for investigation of such processes in ceramics, e.g. sintering [1]. Sintering is accompanied with vanishing of the porosity, which is connected with shrinkage of the sample measurable by dilatometer. Because dilatometric patterns of most raw ceramic constituents are known, dilatometric curve can be useful for estimation of the composition of the green sample [2, 3].

A survey of dilatometric results obtained for fired ceramics show that their relative thermal expansion is $\Delta l/l_0 < 1$ % in the temperature region 20 – 1300 °C. If sintering of the green sample takes place, one can expect contraction $\Delta l/l_0 > 12$ % in this temperature region. Also some phase changes are accompanied with large shrinkage, e.g. intensive rebuilding of the crystal lattice at dehydroxylation and creation of the new phase at 950 °C in clay ceramics. As it follows from that, a large scale of the measurement, rather than extreme sensibility, is necessary for thermodilatometry of the green ceramics. A resolving power of 1×10^{-6} m is fully sufficient for most investigations. A push-rod dilatometer with inductive transducer meets these requirements very well.

In this paper we describe a low-cost dilatometer, its calibration, evaluation of the systematic errors and calculation of the uncertainty of the measurement.

2 Construction of dilatometer

Different known techniques of measurement of thermal expansion of solid materials are shortly described in [4, 5] but no design of the dilatometric cell is similar to that used in this work. A dilatometric cell consists of four carrier alumina rods (with diameter 3.0 mm) that are fastened to two holders made of duralumin and the push-rod and supporting rod. Inductive differential transducer (INPOS, ZPA Jinonice, Czech republic) is fixed to the one holder (see Fig. 1). The construction is light and no one holder is firmly connected with the frame of the apparatus. The dilatometric cell freely passes through windows of the furnace with the ceramic fiber insulation. The furnace is made from porous alumina bricks and silicon carbide rods are used as heating elements. Central part of the dilatometric cell with the sample is located in the furnace. The sample is placed between two rods: one of them is push-rod and the other is supporting rod, which is fastened to the second holder. The supporting rod can be shifted together with

the sample and push-rod with a zeroing screw. The inductive differential transducer measures the changes of the pushrod position Δd, i.e. the changes of the length of the sample.

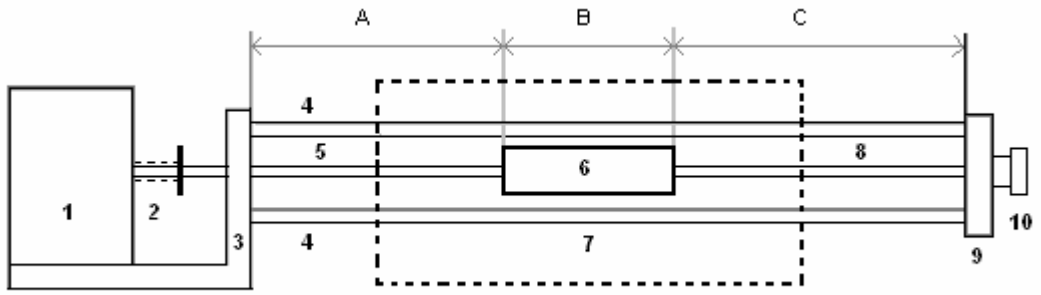

Fig. 1 – Schematic diagram of the dilatometer. 1 – inductive transducer, 2 – spring, 3 – holder, 4 – carrier alumina rods, 5 – push-rod, 6 – sample, 7 – furnace, 8 – supporting rod, 9 – holder, 10 – micrometric zeroing screw

The working temperature range of the dilatometer is 20 – 1200 °C, ambient atmosphere is the air. Temperature is measured by thermocouple Pt-PtRh10%, which is placed beside the sample. Output signal from the difference transformer and temperature are stored by PC, which also operates the power of furnace [6].

The dilatometer exploits a measuring the difference between the expansion of alumina carrier rods and the sample made from material, which dilatometric behavior is unknown. The deflection is proportional to the change of the electrical output signal of the transducer ΔU and we can write

$$\frac{\Delta l_s}{l_0} = \frac{k\Delta U}{l_0} + \left(\frac{\Delta l}{l_0}\right)_{cor} \qquad (1)$$

where l_0 is the initial length of the sample, Δl_s is the change of the length of the sample and Δl_{cor} is the sum of the known change of the length of the carrier alumina rods and systematic errors (e.g. zero drift).

3 Calibration of the dilatometric cell

Because the used method is not absolute, the calibration of the instrument is necessary [7-10]. The calibration was performed using the reference steel X10NiCrMoTiB 1515 delivered by Physikalisch-Technische Bundesanstalt, Berlin. This material has been intensively studied in the recent past. Results of round-robin tests we used as the reference dilatometric curve [11], which can be expressed by the polynomial function

$$\left(\frac{\Delta l}{l_0}\right)_{ref} = 2.001\times10^{-9}t^2 + 1.8\times10^{-5}t - 4.5\times10^{-4} \quad , \qquad (2)$$

where t is a temperature in °C. Correlation coefficient of the fitting is $R = 0.9997$.

The steel etalon with dimensions ∅5 × 30.00 mm and heating rate 5 °C/min were used for the calibration, which was repeated 5 times. The results are shown in Fig. 2.

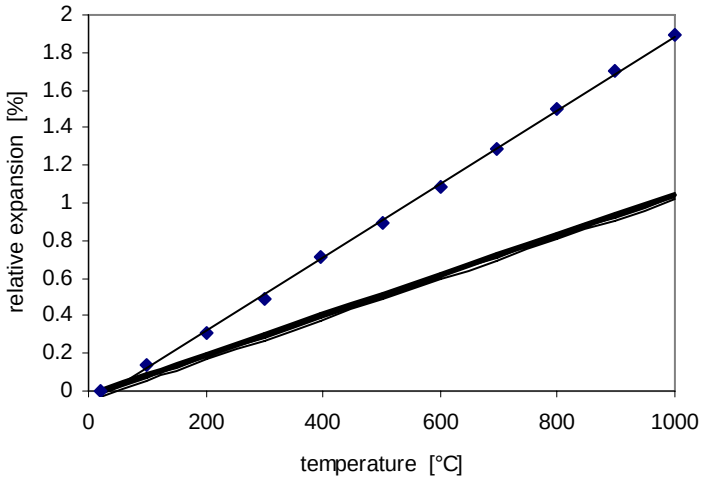

Fig. 2 – Dilatometric curve of the steel reference sample plotted from reference values (♦) [11], measured curves (5 thin lines), and mean curve (thick line)

We used the polynomial fitting to obtain the analytical expression of the correlation function. The function has the form

$$\left(\frac{\Delta l}{l_0}\right)_{cor} = 2.00\times10^{-9}t^2 + 7.00\times10^{-6}t - 1.57\times10^{-4} \ . \qquad (3)$$

Correlation coefficient of the fitting is $R = 0.9999$.

From Eqs. (1), (2) and (3) and from measured voltages ΔU at actual temperatures we calculated the coefficient $k = 2.46$ mm/V.

4 Sources of systematic errors

a) Inhomogeneity of thermal field. The thermal field along the sample was measured at constant temperatures 300, 600, 900 and 1150 °C. It was found that the temperature deviations between the center of the sample and its ends were less than 3 °C, which is in the permitted interval according to the standard [12]. For $\Delta t = 3$ °C and the length of the sample $l_0 = 50$ mm and $\alpha \approx 5\times10^{-6}$ 1/K we obtain the systematic error 1.33×10^{-3} %.

b) Mechanical pressure on the sample. Reliable mechanical contact between the push-rod and the sample is sustained by the spring. A consequence of that is compressive force acting on the sample as well as on the push-rod and supporting rod which leads to their contraction. The carrying rods are expanded under this load. Taking into account the spring compressive force $F = 0.2$ N and dimensions of the rods and ceramic sample (which does not contain glassy phase) as well as temperature dependences of their Young's moduli, the sum of these deformations gives the systematic error $\sim1\times10^{-4}$ % at temperatures below 1000 °C.

5 Sources of random errors

Every dilatometric measurement assumes the measuring of the length of the sample l_0 at the room temperature t_0. According to [12], the length l_0 should be measured with relative error $\Delta l / l_0 < 0.2$ %. Using a micrometer with resolution 0.01 mm we can measure the length of the sample used in the apparatus (50 mm) with relative error $\Delta l / l_0 < 0.02$ %.

We also found, that measured values $(k\Delta U / l_0)$ are not ideally reproducible as it can be seen in Fig. 2. The obtained dilatometric curves are located in a belt, the width of which responds to relative error ~0.06 %. This error is caused by instability of ΔU.

The accuracy of the temperature measurement is ~0.3 °C. The relative error of the temperature measurement is not constant and becomes less as the temperature increases. For the temperatures higher than 500 °C is $\Delta t / t \approx 0.06$ %

The sample position in the dilatometric cell is strongly determined by its construction, therefore it does not influence the value of $\Delta l_s / l_0$.

A total systematic error is ~0.145×10^{-2} % and a total random error is ~0.14 %. The systematic error is 100 times less so the random error can not be take into account.

6 Uncerntainty of the measurement

General rules for evaluating uncerntainty in measurement have been established as the GUM method [13]. The uncerntainty are grouped into categories A and B. Uncerntainties A are calculated by statistical methods and uncerntainties B are estimated by scientific judgement based on experience, manufacturer′s specifications, knowledge of behavior and properties of apparatus and materials, and other relevant information.

If the value Y is determined through directly measured values X_1, X_2, … X_p and the relationship $Y = F(X_1, X_2, ...X_p)$ exists, then the uncerntainty of the type A or B can be calculated. For a dilatometric measurement described above, equations given in [GUM] can be simplified and gain forms

$$u_{Ay} = \sqrt{\sum_{i=1}^{p} A_{x_i}^2 u_{Ax_i}^2} \quad , \quad u_{By} = \sqrt{\sum_{i=1}^{p} A_{x_i}^2 u_{Bx_i}^2} \quad , \quad \text{where} \quad A_{x_i} = \frac{\partial F(X_1, X_2, ...)}{\partial X_i} \quad . \qquad (4)$$

The values u_{Ax_i} and u_{Bx_i} are standard deviations of the measurement of the values X_i belonging to the group A or B. The values A_{x_i} are sensitive coefficients. Then the combined (total) uncerntainty is

$$u_{Cy} = \sqrt{u_{Ay}^2 + u_{By}^2} \quad . \qquad (5)$$

To determine the uncerntainty, we exploited a methodology after [14-16]. From Eq. (1), Eq. (3) and $k = 2.46$ mm/V we have a formula for calculating the relative thermal expansion of the sample

$$\frac{\Delta l_s}{l_0} = \frac{2.46\,\Delta U}{l_0} + (2\times10^{-9}t^2 + 7\times10^{-6}t - 1.57\times10^{-4}) . \qquad (6)$$

The Eq. (6) serves for determination of the sensitivity coefficients of the directly measured values ΔU, l_0 and t. The calculations were made for t = 500 °C. The inductive transducer voltage output corresponding to the actual Δl_s was ΔU = 63.5 mV and was measured with the accuracy of 0.5 mV. The voltage ΔU = 63.5 mV belongs to Δl = 156 μm. The results of the determinations of the sensitivities coefficients and uncerntainties $u(\Delta l_s)$ and relative uncerntainties are given in Tab. 1.

Tab. 1 – Uncerntainties of the dilatometric measurement

Source of uncerntainty	Type	Standard deviation $u(x_i)$	Sensitivity coefficient A_i	$u(\Delta l_s)$ µm	Relative uncerntainty %
Measurement l_0	B	5 µm	0.009	0.045	0.11
Measurement ΔU	B	0.5 mV	2.46 µm/mV	1.23	0.45
Measurement t	B	0.3 °C	1.0 µm/°C	0.30	0.02
Total uncerntainty				1.575	0.58

The relative total uncerntainty was calculated as a ratio of total uncernainty to Δl = 271 µm which is the expansion of the steel etalon at 500 °C.

7 Summary

The push-rod dilatometer with inductive transducer and new alumina rod arrangement described in this paper presented itself as an appropriate instrument for evaluating the dilatometric behavior of fired and green ceramics (e.g. [17]). The calibration was carried out by the steel X10NiCrMoTiB 1515 reference sample. Also, the sources of errors were evaluated and the relative error of the dilatometric analysis was estimated as 0.14 %. The relative uncerntainty of the measurement was also calculated. Its value is 0.58 %.

Acknowledgement

This work was supported by the grants VEGA 1/3179/06 and VEGA 1/3117/05.

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