Calibration of the Horizontal Pushrod Dilatometer

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Abstract

In this paper we present calibration of a horizontal pushrod alumina dilatometer and explain the principle of the dilatometer. We performed calibration of the linear expansions sensor using etalon materials platinum, steel and sapphire and estimated the correction function for alumina and zero. We also performed calibration of the temperature-measuring sensor and estimated the corresponding correction function for temperature. Thermal field homogeneity was measured using a thermocouple. The measurement uncertainty was estimated for the case of inhomogeneous heating of the sample. We conducted the example measurement using the correction functions mentioned above.

Key words: linear thermal expansion coefficient, pushrod dilatometer, calibration

1 Introduction

The thermal expansion coefficient is a very important material property. Tracking of the materials expansion in the dependence of temperature provides us with important information for praxis and research. (for example [1, 2]). An expansion of a material is measured by an apparatus called a dilatometer. The principle of differential dilatometers lies in a parallel measurement of the expansion of an unknown sample and of a known sample for which we have known values of the thermal expansion coefficient. By comparing the expansion of these two samples we are able to estimate the thermal expansion coefficient of an unknown sample. The most commonly used comparison material is the alumina Al_2O_3 (up to 1500 °C) [3]. Dilatometers differ in their geometry of the imposition of the sample (horizontal or vertical) and in the manner of measuring the geometrical changes of the sample and its recording. In this paper we present the calibration of the horizontal pushrod dilatometer.

2 Principle of the apparatus

The thermal expansivity of materials depends on their chemical composition, structure of the material and temperature. Thermal expansion of materials is characterized by the linear thermal expansion coefficient (LTEC) α, which is defined as [3, 4, 5]

$$\alpha = \frac{1}{l_0}\frac{dl}{dt},\tag{1}$$

where l_0 is the length of the sample at the beginning of measurement, dl is the change of the samples length resulting from the temperature change dt. In the praxis, the

temperature change dt is realized by a sufficiently small temperature change $\Delta t = t_2 - t_1$. We show the principle of our dilatometer on the figure 1.
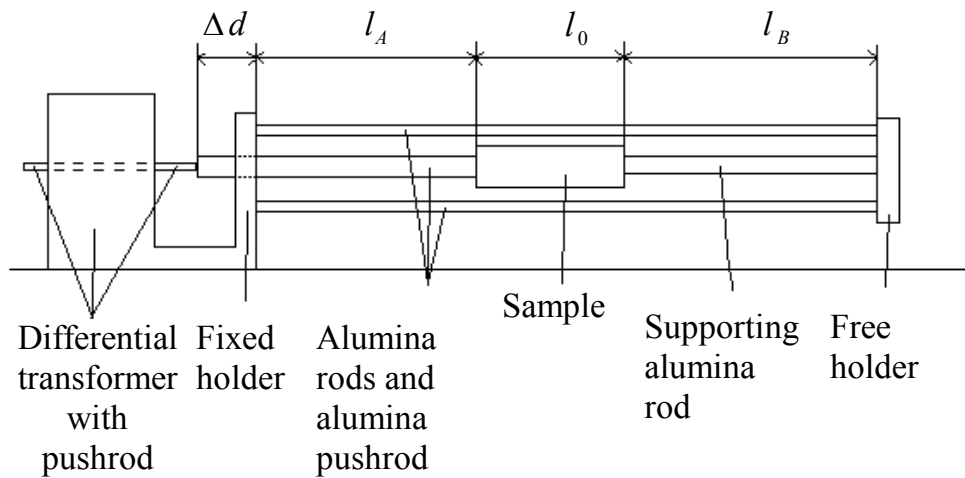

Fig.1 - Scheme of the dilatometer

The dilatometric system consists of alumina rods (Degussa, Germany) that are fastened to the fixed holder (aluminium). A free holder (aluminium) is at the opposite end of these rods. A supporting alumina rod is fastened into the free holder with a zeroing screw. In the central part of the dilatometric system, the sample is being held between the supporting alumina rod and the other alumina rod that acts as a piston pushing to the pushrod of a differential transformer (INPOS, ZPA Jinonice – Nová Paka, Bohemia [6]). The differential transformer measures the deflection Δd of the central rods and the sample. The middle part is held in the furnace. The silicium carbide rods are used as the heating element, and the working temperature range is from 20 °C up to 1200 °C. Temperature is measured by a thermocouple that touches the sample. All data is stored by a PC that also operates the power of furnace by a regulator.

Using the differential measurement method we measure the expansion of a known material (alumina) and an unknown material (sample). In the parts l_A and l_B all alumina rods expand to the right direction (because the left holder is fixed), and only the central part l_0 is important. The length of the sample l_0 is equally long as the alumina length l_c at the beginning of a measurement. At higher temperatures the alumina expands to the right and also the unknown sample, but the sample has a different LTEC, so it expands (or contracts) also to the left. This movement is measured by the differential transformer as deflection Δd. Then the deflection $\Delta d = \Delta l_0 - \Delta l_c$, where Δl_0 means the expansion of the sample and Δl_c the expansion of alumina, can be easily calculated.

The expansion of the alumina is well known for the whole temperature range of our furnace, so we can estimate the expansion of the sample from the known Δl_c and the measured Δd. Finally, for the relative expansion $\varepsilon = \dfrac{\Delta l_0}{l_0}$ we obtain the equation:

$$\frac{\Delta l_0}{l_0} = \frac{\Delta d}{l_0} + \frac{\Delta l_c}{l_0}.$$

(2)

3 Calibration of the apparatus

We have calibrated the induction detector using three reference materials. Homogeneity of the thermal field in the furnace was measured, and considerations about the impact of the inhomogeneity of the thermal field were calculated. Calibration of the thermocouple was also performed.

3.1 Calibration of the induction detector

Calibration was performed using the reference materials steel X10NiCrMoTiB 1515, sapphire 59° Sehnit SA25 and the pure platinum. We measured these materials and obtained their deflections Δd. From the known values of the relative expansion of these materials we were able to get the relative expansion of the reference alumina. However, these values included not only the correction for alumina but also hidden systematic errors of our apparatus. So, using the polynomial fitting with the help of a program Mathcad 2001 we obtained the corrections as a function of the temperature T for these materials and then calculated the values for the mean correction function for our apparatus.

The final equation for the correction function is:

$$\left(\frac{\Delta l_c}{l_0}\right)(T)_{corr.} = \left(\frac{\Delta l_c}{l_0}\right)(T)_{etalon} - \left(\frac{\Delta d}{l_0}\right)(T)_{measured}.$$

(3)

Such correction functions were made four times for the steel, two times for the platinum and two times for the sapphire. The example for the steel in Figure 2 shows the difference between the etalon data and the measured data:
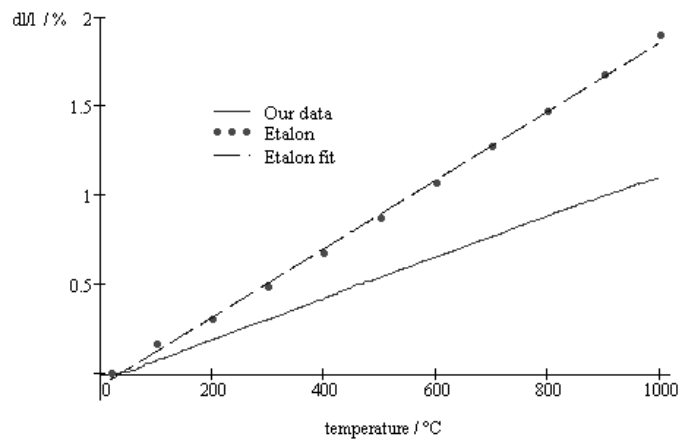

Fig. 2 Calibration for the steel X10NiCrMoTiB 1515

The correction is the difference between the solid and the dashed curve. All calibration functions and the mean calibration function are drawn in Figure 3. We used the third

order polynomial fitting for obtaining these calibration functions. The final mean calibration function has the following form:

$$\left(\frac{\Delta l_c}{l_0}\right)(T)_{\text{corr.}} = -4,162.10^{-11}.T^3 + 1,553.10^{-7}.T^2 + 6,478.10^{-4}.T - 1,400.10^{-2}. \qquad (4)$$

Equation (2) changes finally into:

$$\left(\frac{\Delta l_0}{l_0}\right)(T)_{\text{sample}} = \left(\frac{\Delta d}{l_0}\right)(T)_{\text{measured}} - \left(\frac{\Delta l_c}{l_0}\right)(T)_{\text{corr.}} \qquad (5)$$
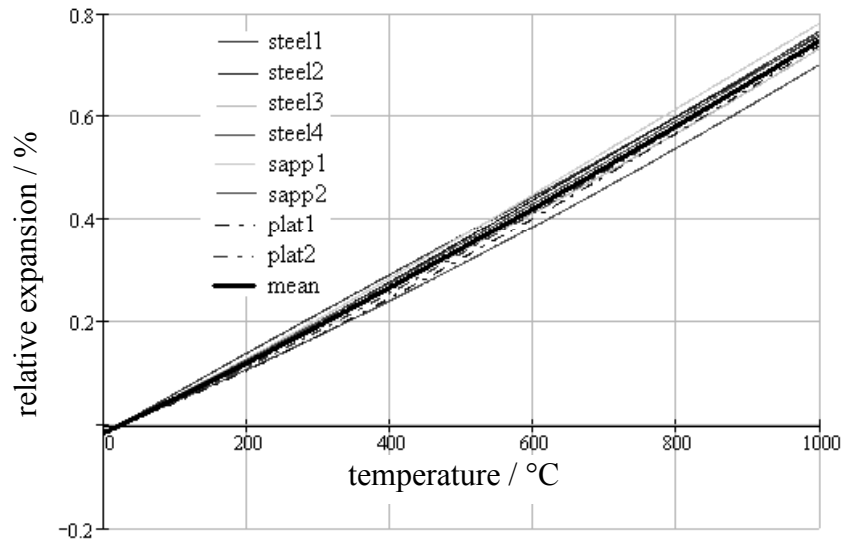

Fig 3 Correction functions and the mean correction function

3.2 Homogeneity of the thermal field

In accordance with the norms [7, 8] the temperature deviations in the furnace near the sample have to be less then 5 °C. Therefore the measurement of the temperature along the dilatometric system was performed. We used a thermocouple to measure it along the places where the sample usually lies. We performed the measurements at constant temperatures 300, 600 and 900 °C. At these constant temperatures, the thermocouple was moved through the whole part of the dilatometric system that is placed in the furnace.

At the edges of the furnace the deviations were greater than 5 °C, but in the central part, where the sample is usually placed, the temperature deviations were in the permitted range. With the higher temperatures the temperature deviations were higher too.

Nevertheless, we performed an additional calculation of the systematic error of the measurement resulting from the temperature field inhomogeneity. Let the temperature in the middle of the sample be T_m. Suppose that temperature T sinks towards the edges of the sample in accordance with the function

$$T(x) = T_m - \eta x^2, \qquad (6)$$

where x is a position along the samples length and η ($[\eta] = \mathrm{K.m}^{-2}$) is the coefficient of inhomogeneity of the thermal field. Let the initial temperature be T_0 (for example 20 °C). Then the temperature difference $\Delta T = T - T_0$ in the place x is

$$\Delta T = (T - T_0) = (T_m - \eta\, x^2) - T_0 .$$

We suppose that LTEC α is in the segment dx constant. Segment dx will expand by $\alpha.dx.\Delta T = \alpha.\left[(T_m - T_0) - \eta\, x^2\right] dx$. Summation of such expansions will be the total expansion of the sample. We obtain it by integrating the last formula in the boundaries from 0 to l, where l is the length of a one half of the sample

$$\Delta l = 2\alpha (T_m - T_0) l - 2\alpha \eta \frac{l^3}{3} , \tag{7}$$

where multiplication by two takes note of the two sample halves, but Δl is already the expansion of the whole sample. When the thermal field is homogeneous, $\eta = 0$, and we obtain the well-known expression $\Delta l = 2\alpha l (T_m - T_0)$. Then the calculation of the relative systematic error of the expansion of the sample is

$$\gamma_{\Delta l} = \frac{(\Delta l)_{\text{homog}} - (\Delta l)_{\text{nehomog}}}{(\Delta l)_{\text{homog}}} = \frac{2\alpha l \Delta T - (2\alpha l \Delta T - \dfrac{2\alpha \eta l^3}{3})}{2\alpha l \Delta T} = \frac{\eta l^2}{3\Delta T} . \tag{8}$$

Let us estimate the systematic error induced by inhomogeneity of the thermal field. If $\Delta T = 200\,°\mathrm{C}$ (sample temperature $\sim 220\,°\mathrm{C}$), the length of one half of the sample is $l = 50\,\mathrm{mm}$ and the temperature of the edges of the sample differs by $5\,°\mathrm{C}$ from the temperature of the middle of the sample, then we can obtain the coefficient $\eta = 2000\,\mathrm{K.m}^{-2}$ from the equation (6) and the relative systematic error from the equation (8), which then is $\gamma_{\Delta l} = 0{,}83\,\%$. According to the equation (8) this error reduces with higher temperatures, at the samples temperature of 1000 °C ($\Delta T \cong 980\,°\mathrm{C}$) it is $\gamma_{\Delta l} = 0{,}17\,\%$.

In the real experimental conditions the temperature field inhomogeneity has an increasing tendency, which means that the coefficient η also increases. These two influences partially compensate each other. The true value of $\gamma_{\Delta l}$ is a function of the temperature and should be calculated after the experimental estimation of the coefficient η using the equation (6).

3.3 Calibration of the thermocouple

We use the thermocouple NiCr/Ni for the temperature measurement. It is placed closely to the sample. It measures the temperature of the furnace, not the temperature in the sample. According to [9], the difference between the temperature measured in the furnace and the real temperature in the kern of the sample can be up to hundreds of degrees of Celsius for metals, and at most 10 °C for other materials. The reason is the radiation from the sample. Hence it is material dependent. According to [4, p.171] we should take the furnace temperature as the sample temperature because the properties of most materials are not so much temperature dependent that this could be a problem.

But it is possible to partially improve the temperature measurement. We measured the temperature of the melting point of the aluminium. According to the well-known value 660,32 °C we were able to calculate a primitive linear correction function for the temperature from two points. One was the temperature of 20 °C, which is the starting temperature of our measurements when the furnace temperature is equal to the sample temperature. The second point is the temperature of melting of the aluminium measured by the thermocouple. Thermocouple showed us approximately 669,3 °C, when the aluminum was melting in the furnace. From this, we obtained this temperature correction:

$$t_{corrected} = 1,014 . t_{measured} - 0,281 . \qquad (9)$$

This correction should be refined in the future by measurements of other well-known temperature dependent effects.

4 Conclusions

The results of calibration of the dilatometer are formulae (4), (5) for calculating the relative expansion from measured linear displacement and formula (9) for calculating the temperature from thermo-emf measured by thermocouple.

The corrections we presented in this paper enable us to precisely measure the LTEC of various materials with measurement uncertainty less than 3 % (calculated in accordance to norm [7]).

Acknowledgement

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