Calibration of a Push Rod Dilatometer

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The linear thermal expansion coefficient (TEC) is a key property of condensed matter. In particular, it is of crucial importance for the thermo-mechanical behaviour of solid components and respective numerical simulation. TEC is widely measured with push rod dilatometers which are also in use for detecting glass transition, phase transition and sintering temperatures. As they are affected by numerous complex error sources, push rod dilatometer measurements are mainly applied as a comparative method based on calibration with reference materials [1]. Accuracy values of TEC ≈ ±2 % are reported generally for measurements using a heating rate of 3K/min and secondary reference materials for calibration [1] and ±2% as maximum error for a silicon single crystal calibrated with fused silica (5K/min) [2]. About ±4% accuracy is given in ref. [1] for sample holder correction based on tabular TEC values of the sample holder material.

The presented work was aimed to reveal the maximum error of a Netzsch 402C push rod dilatometer used in our lab when each sample measurement is corrected by a reference sample previously measured analogously in terms of sample dimension and heating regime.

For that sake, four different reference materials have been measured repeatedly up to 30 times each and calibrated against each other. Measurements comprised single measurements and series of repeated runs and were spread over a period of several months providing different climatic conditions. Experimental variations were combined randomly in order to cover all potential error effects as e.g. initial furnace temperature distribution, moisture, the sample-reference difference in thermo physical properties and their effect on measured TEC. In order to estimate the TEC maximum error, all different reference samples were measured and calibrated against all reference samples regardless of their TEC value and the time of their measurement. Alternatively, the maximum error is obtained for calibration with reference samples under more restricted conditions.

The long-term repeatability (sample holder correction) was ≈ $\pm 0.09 \cdot 10^{-6} K^{-1}$, $\pm 0.11 \cdot 10^{-6} K^{-1}$, $\pm 0.27 \cdot 10^{-6} K^{-1}$, $\pm 0.14 \cdot 10^{-6} K^{-1}$ for fused silica, corundum, sapphire, and platin, respectively. The maximum error obtained for fused silica, corundum, sapphire and platinum randomly calibrated against each other was as high as $\pm$ 0.35 10^{-6} K^{-1}. Better TEC accuracy thus requires the use of reference materials almost identical to the sample material in terms of heat capacity, thermal radiation coefficient, heat conduction, volume, size and weight.

[1] Valentich, *J. Therm. Anal.* 11 (1977) 387-403.
[2] Yamada *et al.*, *Measurement Sci. Technol.* 12 (2001) 2121-9.