DETERMINATION BY A DIRECT MEASUREMENT MODEL OF UNCERTAINTY IN DILATOMETER MEASUREMENTS

J.Mª. Martín Algarra (*), A. Belda Peña (**), G. Mallol Gasch(**)

(*) Metrology and Calibration Laboratory, Cegelec Comsip España S.A., Castellón, Spain

(**) Instituto de Technología Cerámica, Asociación de Investigación de las Industrias Cerámicas, Universidad Jaume I, Castellón, Spain

SUMMARY

Documented measuring instrument calibration is a standard quality requirement (see UNE EN ISO 9001). Calibration should provide measurement data as well as the uncertainty that is associated with them.

Dilatometers are widely used instruments for measuring linear thermal expansion in the ceramic branch. They measure the coefficients of expansion of test specimens based on temperature and length measurements. DIN 51 045, in its description of the test for the determination of thermal expansion of solids, provides an example of the determination of uncertainties in measurement, involving temperature and length measurements, and the respective uncertainties of their measured magnitudes.

At Ceramic Testing Laboratories, the calibration of dilatometers and generation of correction tables for these instruments is done by using standard materials, whose coefficient of expansion has a known value and uncertainty, the example for the calculation of measurement uncertainty as set out in DIN 51 045 not being applicable. The present study shows a practical example of the application of a method for the calculation of measurement uncertainty, to a dilatometer calibrated using standard materials, based on our own experiments, applying the criteria set out in ISO TAG4/WG3(June 1992).

The determination of the coefficient of expansion of a specimen, whether a body or a glaze, is performed in a dilatometer. Such an instrument consists of a tubular kiln that is controlled by an electronic regulator which measures and controls the temperature in the working area. The length measurement is typically obtained by a system of sensing rods that bring the test specimen (in the kiln) into contact with the length measuring system. The

main drawback, from a measurement point of view, as far as this instrument is concerned, is that the sensing rods of the length measuring system need to cross the area between the test specimen that is subjected to the testing temperature, and the measuring system, which is at room temperature. This means that the real expansion of the sensing rod during the test is not known. Prior to use, the instrument must run a test on a sample whose coefficient of expansion is known, which enables a correction to be made for the sensing rod length. However, besides the determined expansion of the rods, and the application of these data as a correction to subsequent tests performed with the instrument involved, the results also depend upon further factors such as:

- Initial length of the test specimen and the standard material used for the readout correction.
- Expansion of the base on which the test is run.
- Heating rate of the test specimens.
- Heating capacity of the test specimen and the standard material used for the readout correction.
- Connection between the sensing rod and the test specimen or the standard material used for the readout correction.
- Closeness of the test specimen and the standard material used for the readout correction.
- Pressure of the sensing rod on the test specimen.

Optimum operating conditions are achieved when the physical properties of the test specimen and those of the standard material used for the correction are alike.

The materials used as reference materials are samples whose physico-chemical characteristics have been specially engineered. In this particular case, organisations like the NIST have developed and marketed standard materials that are sufficiently well characterized to allow using them as reference materials in such applications for determining the coefficients of expansion. Moreover, DIN 51 045 provides lists of expansion values for various types of commonly used standard materials in dilatometric testing, as well as the uncertainty involved in the determination of these expansions. However, the uncertainty calculation set out in DIN 51 045 as an example, is really rather impracticable in industrial dilatometric testing because the length measurement in the example is an absolute measurement, which is usually not readily accessible to the industrial operator, as this requires highly sophisticated measuring techniques. In order to avoid this drawback, a simpler measuring model has been derived $x_i=f(\lambda i)$, where x_i is the coefficient of expansion to be determined, li is a function parameter of the standard material used for correcting the measuring instrument, and f is the measurement function. With this measurement model, the determination of the uncertainty measurement of the proposed model is performed as set out in ISO TAG4/WG3, yielding the final equation:

$$U(x_i)_{k=2} = 2\sqrt{u^2(\lambda) + \frac{s^2(x_i)}{N} + \frac{\varepsilon^2}{3}}$$

where N is the number of performed measurements, $u(\lambda)$ the uncertainty of the value λ, $u(x_i)$ the uncertainty in the measurement of x_i and $s^2(x_i)$ the variance in the measurement of x_i.

In using the dilatometer, readouts are given by the instrument, which automatically performs the corrections based on the standard material used, using these values without any subsequent correction. In the experimental stage, it was found that there were differences between the dilatometer readouts and the theoretical values of the coefficients of expansion found in the literature. This error was included in the uncertainty assessment as a Type B contribution, reducing it to a variance by assuming that it contributes as an equiprobable distribution (see ISO TAG4/WG3). By calling the error e, the above equation is obtained for calculating the expanded uncertainty.

Summing up, it may be stated that the experimental determination of uncertainties in dilatometer measurements is perfectly feasible from the practical point of view of a testing laboratory, by applying the criteria set out in ISO TAG4/WG3, thus meeting the UNE EN ISO 9001 requirement of knowing and assessing test measurement uncertainty. The determination of the measurement uncertainty requires an initial experimental effort, which, when suitably documented yields Type B data according to the terminology of INC-1 of the BIPM, appropriate for use in uncertainty calculations. Wise use of these data allows saving time in experimentation. The advantages of this experimental uncertainty determination lie in its rapidity, simplicity and ease of performance in ceramic laboratories, making it possible to thus have the necessary reference materials available with each instrument. The uncertainty determination of an instrument cannot be easily performed in a practical way by applying the DIN criteria, as the necessary reference conditions are usually unavailable (dimensions, temperature). Systematic calibration of measuring instruments allows detecting whether any variations have arisen (in the case of dilatometers, contamination of the base, aging of thermocouples, variation of the measuring position, etc.), which might bias the outcomes on determining the coefficient of thermal expansion of materials.