Thermochimica Acta, 12 (1975) 102–104

Note

Calibration, precision and accuracy of a Dupont thermomechanical analyzer

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(Received 16 December 1974)

Thermomechanical analysis (TMA) is rapidly attaining the status of a standard method for measuring linear expansion as a function of temperature in diverse fields[1–4]. The transducer in both the DuPont and Perkin-Elmer TMA analyzers is a linear variable differential transformer (LVDT). This is not an absolute transducer and, although linear, must be calibrated. In addition, system variables such as the expansion of the quartz measuring probe and quartz sample stage introduce unique individual instrument reponse factors into the calibration curve.

The simplest and most direct method for calibration of TMA expansion (y-axis) is by following the expansion of some isotropic elemental sample as a function of temperature. Two standards are required for statistical analysis. Aluminum (pure) and conductivity grade copper bus are satisfactory after relieving internal sample stresses induced by the rolling and drawing operations of manufacture. The linear coefficients of expansion are known[5,6]. As a result of recent studies conducted at this laboratory a body of calibration data on these two metals has been obtained which permits some statistical manipulation.

EXPERIMENTAL

The aluminum sample was employed as received with the instrument from the manufacturer. The copper standard was a cylinder turned from OFHC copper. Stresses were relieved by heating the cylinder to 1000°C in powdered alumina with free access to air. The sample was quenched in room temperature tap water. The ends were faced off and lightly filed to remove tool marks. The length of the aluminum standard, 0.7550 ± 0.0005 cm, was measured with a Zeus micrometer. The length of the copper standard, 0.6165 ± 0.0005 cm was measured with a Starrett vernier micrometer.

The 941 TMA module was connected to a DuPont 900 thermoanalyzer module and run in the expansion mode. The y-axis or expansion scale was set at sensitivity

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0.008 for all runs. The system thermocouples used an ice reference. A heating rate of 5°C min^{-1} was employed for all runs with the samples allowed to cool to room temperature at the normal power off furnace cooling rate. The aluminum data were acquired over a three-month period at random intervals. The copper sample was run over a three-day period. Both samples will express the total system error, i.e., probe error and repositioning errors. The scale factor was calculated for the 0.04 scale in mixed notation, i.e., cm sample expansion per inch chart. At first blush this may appear a bad case of mixed units. However, the manufacturer furnishes charts graduated only in inches. To ignore these marks would be inefficient. We have found the mixed unit system most convenient since the initial answers appears in terms of cm of sample length.

RESULTS

The calibration factors as calculated from ten runs on aluminum and twelve runs on copper are shown in Table 1. The average value from the aluminum measurements was found to be 7.943×10^{-3} cm sample expansion per inch of chart at

TABLE 1

SUMMARY OF TMA CALIBRATION FACTORS ON ALUMINUM AND COPPER

Conversion factor for 0.04 scale cm $in.^{-1} \times 10^{-3}$

Aluminum[a]	*Copper*[b]
7.666, 7.817	7.310, 8.122
7.837, 7.892	7.872, 7.995
7.581, 8.008	7.637, 7.107
8.283, 7.780	7.525, 7.753
8.292, 8.271	7.753, 7.310
Average = 7.943×10^{-3} cm sample $in.^{-1}$ chart	7.753, 7.753
$\sigma = 0.261 \times 10^{-3}$	Average = 7.658×10^{-3} cm sample $in.^{-1}$ chart
	$\sigma = 0.298 \times 10^{-3}$

[a] $\beta = 24.1 \times 10^{-6}$ $°C^{-1}$ at 77°C. [b] $\beta = 16.6 \times 10^{-6}$ $°C^{-1}$ at 25°C.

sensitivity 0.04 with a sigma of 0.261×10^{-3} cm $in.^{-1}$. For copper the average was found to be 7.658×10^{-3} cm sample expansion per inch of chart with a sigma of 0.298×10^{-3} under the same conditions. These were calculated from the slope of the TMA plot in the temperature range 30 to 80°C over which the TMA records were relatively linear.

A Student's t test for the significance of this difference gave a value of $t = 2.392$ and $\phi = 20$. $P < 0.05$ which shows that the values differ significantly. Thus, the two values cannot express the same number if the variance is given correct weight. Such a condition is perplexing. In seeking to find the source of inter-sample error (assuming the samples were aluminum and copper) the authors explored the accuracy of the

linear expansion data on the two metals used in the computation. A value of 24.1×10^{-6} per °C (at 25°C) had been used for copper[5]. The plots had appeared sensibly linear over that range in all cases. A value of 23.2×10^{-6} per °C at 27°C was found in the *AIP Handbook* for aluminum[6]. Using this value the average calibration constant obtained from the aluminum measurements becomes 7.646×10^{-3} cm in.$^{-1}$ which is in excellent agreement with the value of 7.658×10^{-3} cm in.$^{-1}$ obtained from copper.

CONCLUSIONS

The calibration constant determination for the DuPont TMA can be determined to four significant figures with a sigma of about 0.28. Two separate calibrations with copper and aluminum agree to within 0.156% on a total of twenty-two measurements. Particular effort must be extended towards obtaining correct values for the expansion coefficients of the standards. These replicates probably represent average operation of an unmodified instrument.

ACKNOWLEDGMENT

This work was supported in part by G. E. Purchase Orders 169-03941, -05456 and -02139.

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