

An assessment of a new type of capacitance dilatometer for measurement of the thermal expansion of solids between 273 and 620 K

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Abstract. The construction of an apparatus for measuring the thermal expansion of solids, based on the three-terminal capacitor method, in the temperature range between 273 and 620 K is described. The device enables measurements to be made on samples regardless of their shape, size, conductivity or dielectric constant. The operating range can easily be extended down to 77 K requiring no changes in essential parts. Replacement of the sample is easy and fast with no need to dismantle the capacitor. The minimal change in length of the sample that can be resolved is 0.3 nm, which yields the sensitivity of 2×10^{-6} for a 100 μm long sample. The dilatometer has been calibrated using silicon, palladium and copper as the reference materials. The thermal expansion coefficient can be determined with an accuracy of $\pm 1.5\%$.

1. Introduction

Quite a number of various techniques have been developed for the precise measurement of the variation of the thermal expansion coefficient with temperature. The capacitor method is the most sensitive method available ($\Delta l/l_0 \approx 10^{-10}$) (for the optical method $\Delta l/l_0 \approx 10^{-7}$ – 10^{-8} , for the x-ray method $\Delta a/a_0 \approx 10^{-6}$ and for the inductive method $\Delta l/l_0 \approx 10^{-6}$).

Various types of capacitance dilatometer have been reported in the literature (Miller *et al* 1971, Kroeger and Swenson 1977, Willemsen *et al* 1977, Villar *et al* 1980), but they are rarely intended for work at low as well as at high temperatures (Pott and Schefzyk 1983, Johansen *et al* 1986). Relatively large specimens are sometimes required for greater accuracy (Kroeger and Swenson 1977, Villar *et al* 1980); some dilatometers (Villar *et al* 1980, Pott and Schefzyk 1983) involve springs, screws, supports or glues in their construction which may cause irreproducibilities owing to plastic deformation when heated. To ensure mechanical stability of the specimen and electrode assembly relatively large forces are sometimes applied to the sample (Johansen *et al* 1986) which may cause spurious results near the phase transition (Willemsen *et al* 1977). The majority of published papers treat the contribution of the cell (background signal) to the thermal expansion by measuring the expansion of a standard sample and ascribing the difference between measured and literature values to the cell. This procedure

might be inconvenient in cases where the thermal expansion of the cell depends on the length of the sample (Pott and Schefzyk 1983).

We intended to build a capacitance dilatometer in such a way as to reduce most of the above-mentioned imperfections. Our dilatometer enables measurements to be made well above room temperature (600–700 K) as well as down to 77 K. The dilatometer can work with samples of almost arbitrary shape, because we do not use the sample as one of the electrodes. A sensitivity of 10^{-6} can be achieved even with samples only 100 μm long. We have ensured that only a small force acts on the sample by choosing light materials for device construction. The total loading on the sample amounts to 0.04 N and is constant by construction. The background signal is measured directly in the course of measurement on investigated samples. The replacement of samples is easy and fast, and there is no need to remove the capacitor when mounting the sample. The capacitor is separated from the heater and held at a constant temperature. This is an advantage regarding the thermal noise, which can diminish the advantage of the high sensitivity of this method, especially at temperatures well above room temperature.

2. Construction of the dilatometer

The construction of the dilatometer is shown in figure 1(a). The main parts are an outer quartz tube

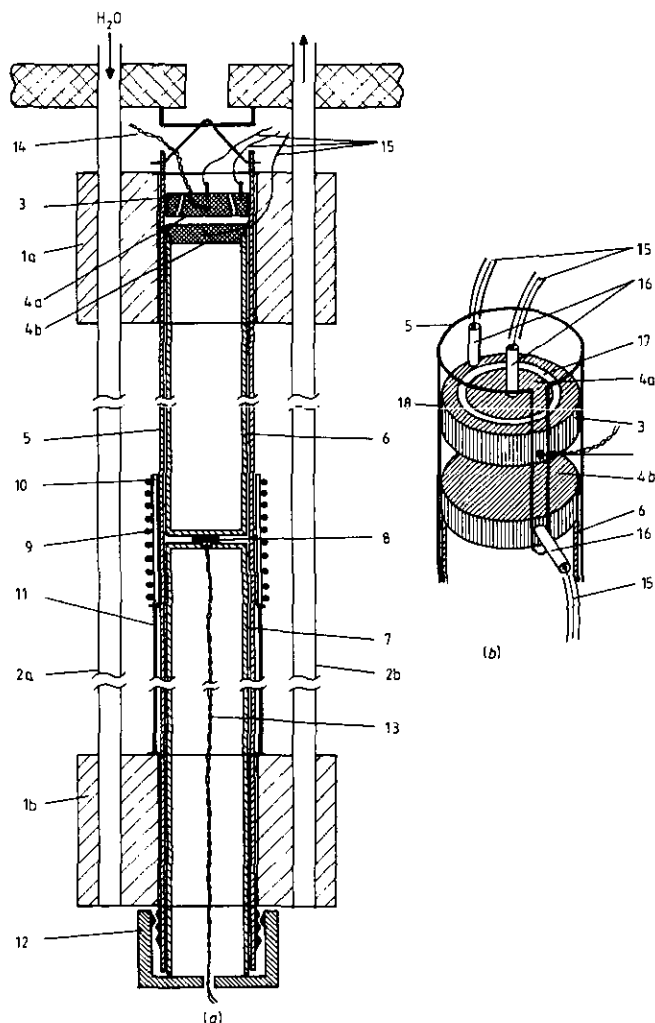


Figure 1. (a) Scheme of the dilatometer: 1a and 1b, copper blocks; 2a and 2b, copper tubes for conveyance of water; 3, guard ring; 4a and 4b, capacitor plates; 5, outer quartz tube; 6, inner quartz tube (electrode holder); 7, inner quartz tube (sample support); 8, sample; 9, heater; 10, quartz heater holder; 11, quartz support; 12, quartz screw cap; 13, sample thermocouple; 14, electrode thermocouple; 15, miniature coaxial cables. (b) A detail of the construction related to the electrodes: 16, miniature Cu bars; 17, Stycast 2850 GT; 18, Araldite. The remaining numbers as in (a).

(250 mm long, inner and outer diameters being 9 and 11 mm respectively), and two inner quartz tubes 9 mm in diameter (one serving as an electrode holder, the other being the sample support). To ensure tight sliding and to prevent tilting of the tubes the outer surface of the inner tubes was rubbed against the inner surface of the outer tube with an abrasive paste (0.25 μm diamond paste) in between. Quartz discs were fused at one end of each inner tube and polished to a mirror finish perpendicularly to the tube axes. A low-potential electrode (with a guard ring) is fixed to the outer quartz tube, while the high-potential, displacive one is attached to the upper inner tube with Araldite epoxy resin. Figure 1(b) shows a detail of the electrode construction. Electrical contact to the high-potential electrode prevents its angular movement. We have chosen aluminium

for the electrode material, which in addition to the thin walls of its quartz holder (0.3 mm) produces a load to the sample of only 4 g. The outer tube is closed at the bottom with a quartz screw cap (figure 1(a)) which allows adjustment of the initial electrode separation.

The whole quartz part of the device is inserted through massive copper blocks (40 mm in diameter) as shown in figure 1(a). Copper blocks are water cooled and are used for keeping the electrodes at a constant temperature, as well as as an electrical shield.

The heater is wound bifilarly around the cylindrical quartz support and is sited in the middle between the copper blocks. The whole dilatometer assembly is sealed in a quartz tube closed at one end, to ensure good thermal insulation from the surroundings.

The high-potential, movable electrode is an aluminium disc, 9 mm in diameter, 2 mm thick and polished to a mirror finish. The fixed, low-potential, guard-ring electrode (figures 1(a) and (b)) is made of two pieces glued with Stycast 2850 GT with catalyst 11 (Emerson & Cuming). The central electrode shaped like a truncated cone is positioned centrally in the matching guard ring. With the epoxy resin hardened, the electrode is rubbed until no electrical contact can be detected between central and the guard electrode. Finally the electrode is highly polished. The diameter of the central electrode is 7 mm and the gap width 0.03 mm. Electrical connections to the electrodes inside the dilatometer are made through miniature coaxial cables.

Following the work done by Heerens (1986), related to the linearity of capacitance transducers and conditions therefore imposed upon the gap width, thickness of the electrodes and typical plate separations, we have chosen a thick geometry for our electrodes. Considering the gap width of our capacitor, the typical plate separation which ensures the linearity if the electrodes were thin, should have been at least 150 μm . Designing the electrodes in a thick geometry allows the typical separation to be about 50 μm thus giving an increase in $\Delta C/\Delta d$ of almost one order of magnitude.

The temperature of the specimen is measured by a copper-constantan thermocouple placed in the centre of the quartz disc supporting the sample. The thermocouple is glued with a high-temperature cement (Astrocera, $T_{\text{max}} = 1500 \text{ K}$) of good thermal conductivity. The thermocouple is inserted slightly downwards (a few micrometres) with respect to the quartz disc surface to ensure that neither the thermocouple nor the cement contributes to the measured thermal expansion. Another thermocouple (copper-constantan) is attached to the low-potential electrode with varnish (General Electric). Both thermocouples were calibrated against a platinum resistance thermometer (100 Ω at 273 K, Degussa) and a Pt-Pt Rh 13% thermocouple (Degussa) in the temperature range of interest. The best polynomial fit (maximum error $\Delta T = 0.02 \text{ K}$) through calibrated points was used for determination of temperature in the measurements.

The heating is provided by a programmable Kepco power supply. The temperature gradient along the furnace was measured and the region of constant tempera-

ture, at least 5 mm wide, was found in the middle of the furnace. A test was made with the device not insulated from the surroundings under conditions of maximal heat dissipation, i.e. at the maximum temperature achieved later in the measurements. The temperature stability at the sample position under measurement conditions was found to be ± 10 mK. Cooling of the electrodes is provided by water flow through the copper blocks. We used ultra-cryostat which gives steady water flow of 2 l min^{-1} and an ultimate temperature stability of circulating water of ± 10 mK. The water pump is a combined suction and pressure one. This ensures (with connection tubes of the same length for conveyance of liquid) zero net pressure on the circulating liquid, thus eliminating undesirable vibrations which might otherwise arise.

In order to extend the operational range of the dilatometer to low temperatures it is sufficient to use a closed circulating cycle of liquid nitrogen or trap-cooled He gas instead of water. Because all the materials used are also suitable at low temperatures this rearrangement is an outward one.

The capacitance was measured by the standard arrangement for three-terminal capacitors. The capacitor was connected in one arm of a Wayne-Kerr four-decade capacitance bridge. A Stanford Research lock-in amplifier was used as a null detector for the bridge. The reference signal (1 V, 1 kHz) for the bridge was provided by a lock-in amplifier. The output of the lock-in amplifier was fed to a Keithley digital voltmeter via an RS-232 interface. The temperatures of the sample and the electrodes were measured by the same voltmeter. Data acquisition was performed by the system developed and built by Aviani *et al* (1987).

Using a 1 V signal at 1 kHz the sensitivity of the method was determined to be 5×10^{-5} pF, which in terms of dilatation corresponds to a resolution of 0.3 nm at $C = 7$ pF, i.e. 2×10^{-6} in relative length change for a 100 μm long sample. The temperatures were measured with a resolution of ± 10 mK, which is limited by the temperature stability of our system.

Electrical connections between the top cover of the dilatometer and the bridge were made by double shielded BNC cables. Adequate care was also taken in proper grounding and shielding in the rest of the measuring assembly (Morrison 1967).

3. Measuring procedure

The sample was placed (with no use of glues or grease) on the flat surface of the sample holder and then carefully inserted in the dilatometer body. Initial separation of the electrodes was adjusted by the quartz screw cap. Heating was provided by continuous programmed current increase through the non-inductively wound heater. When the sample was mounted the dilatometer was sealed in the quartz can and evacuated to a pressure of 10^{-3} Pa and subsequently filled with argon.

The heating rate was varied from 0.4 K min^{-1} to 3 K min^{-1} and irreproducibilities arising from non-

stationary heat transport through the system were observed when the heating rate exceeded 1.5 K min^{-1} . The presented results were collected by computer, taking one reading per minute, with the temperature increased continuously at a rate of 1.5 K min^{-1} .

4. Calculation of results

The capacitance of an ideal parallel-plate capacitor with circular plates, assuming no distortion of the field at the edges, is given by

$$C = \epsilon_0 \epsilon_r \frac{\pi r^2}{d} \quad (1)$$

where ϵ_0 and ϵ_r are dielectric constants of vacuum and the medium respectively, r is the radius of the plates and d is the plate separation. In our experiments we used argon gas as the medium, and since its dielectric constant differs slightly from unity even at atmospheric pressure we used $\epsilon_r = 1$ in calculation of the results.

4.1. Distortion of the electric field at the edges of the plates

For a real capacitor the electric field is never uniform near the edges of the plates. In practice this can be eliminated to a great extent by enclosing the capacitor plates in grounded guard rings. The corresponding expression for the capacitance of such a capacitor which also takes into account the correction due to the dielectric filling the gap (Brown and Bulleid 1978) is

$$C = \epsilon_0 \frac{\pi r^2}{d} \left[1 + \frac{\epsilon_s d}{\epsilon_s d + 0.22w} \frac{w}{r} \left(1 + \frac{w}{2r} \right) \right] \quad (2)$$

where d is the plate separation, w is the gap between the central and the guard electrode and ϵ_s is the dielectric constant of Stycast. Retaining only terms linear in w/r , expression (2) reduces to

$$C = \epsilon_0 \frac{\pi r^2}{d} (1 + \delta) \quad (3)$$

$$\delta = \epsilon_s \frac{w}{r} \frac{d}{\epsilon_s d + 0.22w} \quad (4)$$

For our dilatometer $r = 3.5$ mm, $w = 0.03$ mm, $\epsilon_s = 6.3$ (value at 1 kHz quoted by manufacturers) and a typical value for d is 0.05 mm. The calculated value for δ is therefore 0.8% which we have taken as a constant correction due to the weak dependence of δ on d .

4.2. Thermal expansion of the capacitor plates

The change of the radii of the capacitor plates with temperature affects the capacitance and consequently the calculated plate separation. Taking this into account the change in plate separation is

$$\Delta d = \epsilon_0 \pi \left(\frac{r_2^2}{C_2} - \frac{r_1^2}{C_1} \right) \quad (5)$$

where r_i ($i = 1, 2$) are the radii of the plate at temperatures T_i , and C_i the corresponding capacitances. Substituting $r_2 = r_1 + \Delta r$ and retaining only terms linear in $\Delta r/r_1$ (Pott and Schefzyk 1983), one obtains

$$\Delta d = -\varepsilon_0 \frac{\pi r_1^2}{C_1} \left(\frac{C_2 - C_1}{C_2} - 2 \frac{\Delta r}{r_1} \frac{C_1}{C_2} \right). \quad (6)$$

Since we have designed the dilatometer with the aim that the capacitor plates be kept at constant temperature, in an ideal case the second term in the equation (6) should vanish due to $\Delta r = 0$. In reality the optimum we could reach was that for a temperature change of 400 K at the sample position we obtained a total temperature change of the capacitor plates of 1.5 K. With typical values for $C_1 = 7$ pF, $C_2 = 9$ pF and $\Delta r/r_1$ being the expansion of aluminium over a temperature interval of 1.5 K, one finds the contribution of the second term in relation (6) to be at most 0.1%. Compared with the first term, which varies from 5% to 20% (depending on the expansion of investigated material), it may be neglected.

4.3. Non-parallelism of the capacitor plates

The correction of the capacitance due to the non-parallelism of the plates can be estimated from the expression involving explicit dependence on the amount of tilt (Pott and Schefzyk 1983) which gives

$$C = \varepsilon_0 \frac{\pi r^2}{d} \frac{2d^2}{a^2} \left\{ 1 - \left[1 - \left(\frac{a}{d} \right)^2 \right]^{1/2} \right\} \quad (7)$$

where a is the amount of tilt, defined so that the plate separation varies from $d - a$ to $d + a$. Expression (7) yields a maximal capacitance for $d = a$, given by

$$C_{\max} = 2\varepsilon_0 \frac{\pi r^2}{a} \quad (8)$$

while d , expressed in terms of C_{\max} , becomes

$$d = \varepsilon_0 \frac{\pi r^2}{C} \left[1 + \left(\frac{C}{C_{\max}} \right)^2 \right]. \quad (9)$$

According to relation (8) the amount of tilt can be determined by measuring C_{\max} . For our capacitor the maximal measurable capacitance before the capacitor plates were brought into electrical contact was 105 pF. Relation (8) therefore determines a to be 7 μm . With a typical capacitance $C = 7$ pF the error in determining the plate separation if we do not include correction for tilting is about 0.4% (relation (9)).

4.4. Calculation of Δl and β

The thermal expansion of the sample, Δl , is related to the change of the plate separation Δd by

$$\Delta l_{\text{sample}}(T) = -\Delta d_{\text{meas}}(T) + \Delta d_{\text{BS}}(T) + \Delta l_{\text{quartz}}(T). \quad (10)$$

The second term in relation (10) represents the contribution of the background signal, which will be discussed in section 4.5.

The thermal expansion of the sample is the relative

expansion with respect to the wall of the outer quartz tube. The third term in relation (10) represents the thermal expansion of quartz of the same length as the sample and is given by

$$\Delta l_{\text{quartz}} = l_0 \beta_q \Delta T \quad (11)$$

where l_0 is the length of the sample, ΔT is the change in temperature of the sample and β_q is the expansion coefficient of fused quartz.

The thermal expansion coefficient β has been determined by taking the derivative of measured $\Delta l/l_0$ versus T curves. The method used for calculation of the derivative is based on least-squares fitting of the parabola through a set of n points ($n > 3$), and taking the analytical derivative (slope of the tangent to the parabola) in the middle of the interval. The derivative in each measured point is obtained by moving the set of n points along the whole experimental curve.

4.5. Signal of the dilatometer with no sample

Besides the sources of possible errors in determining plate separation discussed so far, there is a problem of determining the contribution of the device itself to the change in plate separation.

Unlike previously published treatments of the background signal (Johansen *et al* 1986, Pott and Schefzyk 1983, Villar *et al* 1980) we have chosen to measure it straightforwardly in the course of measurement on investigated samples. To achieve that it was necessary to determine reliable parameters in terms of which the background signal can be expressed. We have performed measurements in a dynamic vacuum as well as in an inert gas atmosphere. Temperature at the sample position (T_s), temperature of the electrodes (T_{el}) and the plate separation d were measured simultaneously.

Results obtained in dynamic vacuum, in both the heating and cooling cycles, are shown in figure 2. The plate separation as a function of T_s and T_{el} is shown in figures 2(a) and (b) respectively. The d versus T_{el} curve exhibits well defined linear dependence, contrary to the d versus T_s curve. The change of the electrode temperature is ≈ 15 K for the corresponding change of T_s of 350 K. Figure 2(d) displays a delay in change of T_{el} with respect to T_s . Temperature at the sample position increases when heating is initiated while weak change in d or T_{el} is measured. This suggests that temperature T_s is not a correct parameter for the plate separation to be correlated with. In addition, the thermal expansion coefficient determined from the d versus T_{el} curve, $\Delta d/l_0 \Delta T$, where l_0 is the length of the aluminium electrodes, is found to be 22×10^{-6} which is in agreement with the literature value for the thermal expansion coefficient of aluminium (Kirby *et al* 1972). Therefore we may conclude that the whole background signal can be ascribed to the length change of the electrodes. The correctness of this conclusion is illustrated in figure 2(c) where the difference between the measured d versus T_s curve (background signal) and the one calculated from the relation

$$d_{\text{BS}}(T_s) = d_0 + k_v(T_{el} - T_{0el}) \quad (12)$$

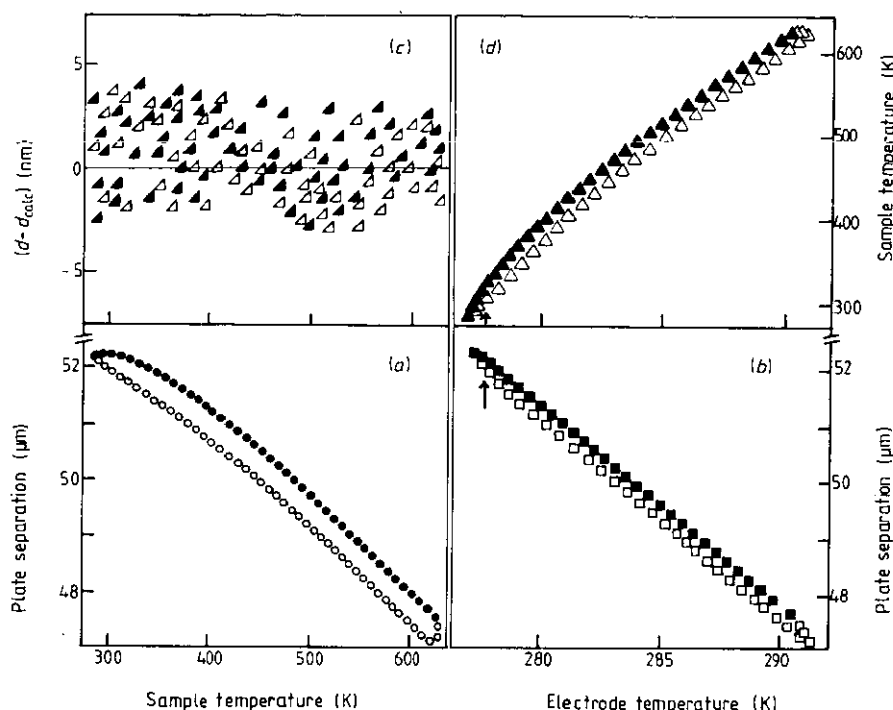


Figure 2. Background signal of the dilatometer in vacuum; heating, full symbols; cooling, open symbols. (a) Plate separation as a function of sample temperature. (b) Plate separation as a function of electrode temperature. (c) The difference between measured and calculated plate separation, as explained in the text, as a function of sample temperature. (d) Sample temperature versus electrode temperature dependence.

is shown. T_{0e1} and d_0 are the initial values of electrode temperature and plate separation respectively, and k_v is the slope determined from the d versus T_{e1} curve obtained in vacuum. The scattering of the data is within the range which can be expected regarding the temperature stability of the aluminium electrodes (± 10 mK). Reproducibility of the results can be expressed through variation of the slope k_v , determined from independent measurements under nominally the same conditions, which is found to be less than 0.1%.

The results obtained on cooling display hysteresis. Upon reversing heating into cooling the temperature T_s begins to decrease. Meanwhile the temperature of the electrodes increases by 1 K before decreasing too. This delay in response to thermal cycling is due to the low thermal conductivity of the quartz separating the electrodes from the heater. In that narrow temperature interval T_s decreases while T_{e1} increases, which results in an additional decrease in plate separation (contrary to the rest of the heating-cooling curve where the expansion of the outer quartz tube and the electrodes itself gave the contribution to the plate separation change in the same direction). The d versus T_{e1} curve during cooling is parallel to the heating one except in the narrow temperature interval at the end of the cycle, which coincides with the beginning (indicated by arrows in figures 2(b) and (d)) of the narrowing of the hysteresis displayed in the T_s versus T_{e1} curve. At the end of the heating-cooling cycle the system reaches the initial

conditions regarding T_s , T_{e1} and d , which confirms the reproducibility and the mechanical stability of the system.

Overall changes in d as well as in T_{e1} are relatively large in vacuum, compared with typical total measured length changes (see section 5). According to the results obtained in vacuum the background signal can be decreased by reducing the overall temperature change of the electrodes. This can be achieved in the inert gas atmosphere thus introducing the heat transport by convection.

Typical d versus T_{e1} and d versus T_s signals obtained in an inert gas atmosphere are shown in figures 3(a) and (b) respectively. As one can see, heating of the electrodes is reduced by one order of magnitude ($\Delta T_{e1} \approx 1.5$ K), while change of the plate separation itself is decreased even more ($\Delta d \approx 0.2$ μm compared with 4 μm in vacuum). Qualitatively this can be explained bearing in mind that convection permits better heating of the outer quartz tube with respect to the inner one in the region outside the heater. As a consequence the net relative expansion of the outer tube causes the upper electrodes to move apart. An average difference in temperature of 1 K along the tubes outside the heater can account for the observed effect. Because of the poor thermal conductivity of quartz such a difference can be expected.

To confirm the above explanation we have carried out the measurements of the background signal for various pressures of the inert gas (60, 133, 1333, 2700 Pa).

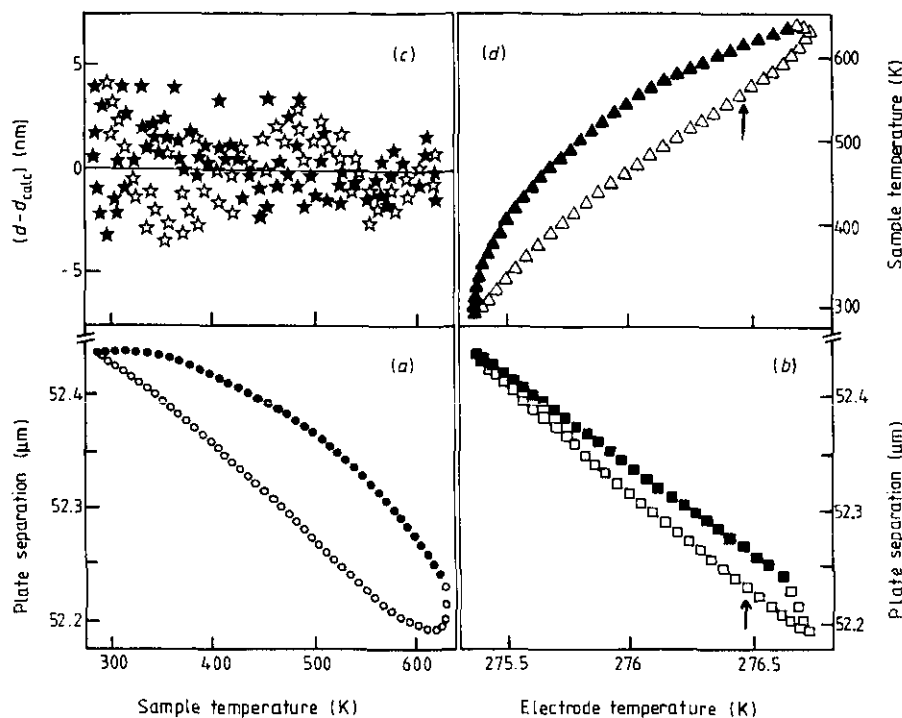


Figure 3. As figure 2 but for the dilatometer in argon atmosphere.

The overall change of T_{e1} as well as of the slope k_{ig} of the d versus T_{e1} curve decreases with increasing pressure up to 700 Pa. For pressures larger than 1333 Pa neither the overall change of T_{e1} nor the slope k_{ig} vary significantly, which is in accordance with the properties of dilute gases; namely, the thermal conductivity of a dilute gas rises with increasing pressure up to ≈ 133 Pa, after which it is no longer pressure dependent.

Figure 3(d) is analogous to figure 2(d) where T_s versus T_{e1} dependence is shown. In an inert gas atmosphere the delay in the change of T_{e1} with respect to T_s is even more pronounced than in a vacuum.

The results obtained on cooling in an inert gas atmosphere exhibit hysteresis too. The 'overheating' of the electrodes at the end of the heating cycle is reduced to 0.3 K compared with that observed in vacuum. The hysteresis displayed in the T_s versus T_{e1} curve begins to narrow earlier (indicated by the arrow in figure 3(d)) than in vacuum conditions due to the presence of the inert gas. This coincides with the deviation from linear dependence in the d versus T_{e1} curve, as has already been observed for the cooling curve in a vacuum. The discussion for the remaining features is analogous to that made for the corresponding curve in a vacuum.

Figure 3(c) shows the difference between measured d and d_{BS} (relation (12)). As for heating, d_{BS} was obtained using slope k_{ig} determined from the heating curve in an inert gas atmosphere. The cooling d versus T_{e1} curve has been fitted to a parabola and its coefficients $k_{ig}^{(1)}$ and $k_{ig}^{(2)}$ were used for obtaining d_{BS} (expression analogous to (12)).

The presented measurement of the background signal was carried out in an argon atmosphere under a pressure

of 2700 Pa. The slope of the d versus T_{e1} curve depends only on the pressure of inert gas and is reproducible under conditions of constant pressure for heating rates not greater than 1.5 K min^{-1} .

5. Experimental results and discussion

Measurements of thermal expansion were performed on copper (polycrystalline, Metal Crystals & Oxides Ltd, 5N purity), and palladium (polycrystalline, Metal Crystals & Oxides Ltd, 5N purity) samples in an inert gas atmosphere ($p = 2700 \text{ Pa}$).

Since the expansion coefficient of fused quartz is a function of temperature and annealing treatment we have used silicon (single-crystal, Wacker, $\rho_{Si} = 100 \text{ } \Omega \text{ cm}$) to calibrate β_q of quartz (following equations (10) and (11)).

The silicon sample was in the form of a 1 mm thick slab, $2 \times 3 \text{ mm}^2$ in cross section. The copper and palladium samples were 1 mm and 1.5 mm thick discs, 6 mm and 3 mm in diameter respectively.

The results of measurements of the thermal expansion for palladium and copper are shown in figures 4(a) and 5(a). Relative deviations ($\pm 0.5\%$) of experimental data for thermal expansion from tabulated values (Kirby *et al* 1972) are shown in insets 4(b) and 5(b).

The thermal expansion coefficients obtained for palladium and copper are shown in insets 4(c) and 5(c), together with the polynomial fits to the literature values of β (Kirby *et al* 1972). The absolute deviation between the presented results for β and the literature ones is at most $2.5 \times 10^{-7} \text{ K}^{-1}$. Thermal expansion

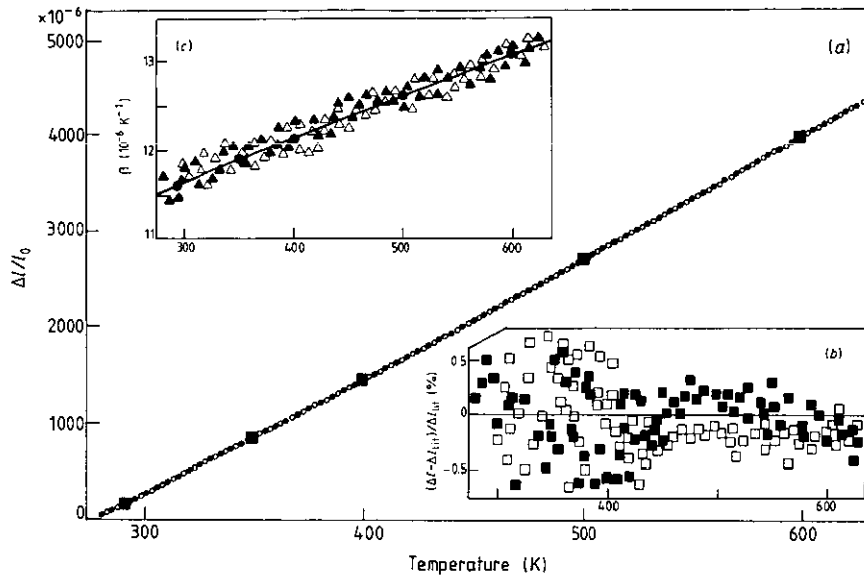


Figure 4. (a) Thermal expansion $\Delta l/l_0$ of palladium as a function of temperature: ●, heating; ○, cooling (data obtained in this work); ■, literature values (Kirby *et al* 1972). (b) Relative deviation between present data and polynomial fit through the literature data (Kirby *et al* 1972) of the thermal expansion of palladium as a function of temperature; ■, heating; □, cooling. (c) Thermal expansion coefficient of palladium as a function of temperature; ▲, heating; △, cooling (values obtained in this work); ●, literature values (Kirby *et al* 1972). The line shows the polynomial fit through the literature values (Kirby *et al* 1972).

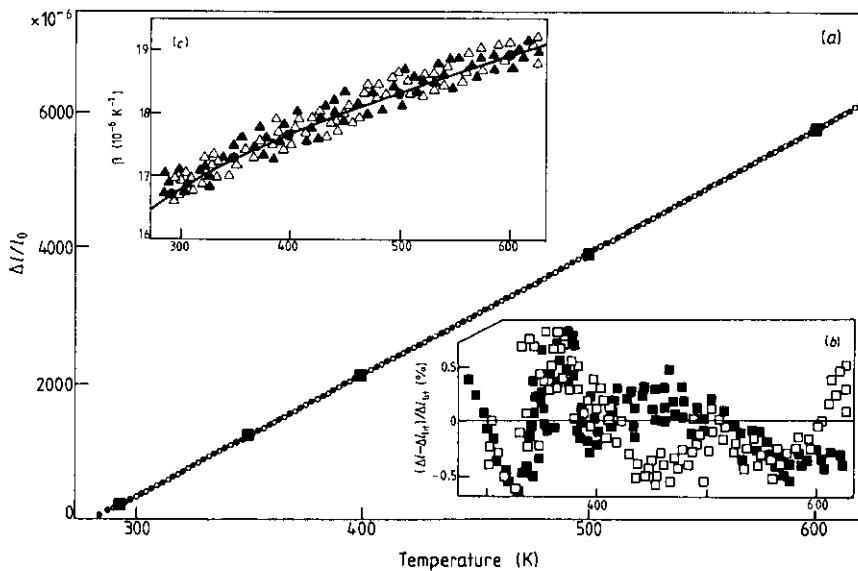


Figure 5. As figure 4 but for copper instead of palladium.

coefficients were determined with a relative accuracy of $\pm 1.5\%$.

The presented results were obtained under the most unfavourable conditions regarding the heating rate and the size of the samples in order to test the limitations of the device. Besides the presented ones we have performed measurements with smaller samples and slower heating rates to examine whether the relatively short

isothermal zone of the heater or the heating rate would influence the results. We have measured thermal expansion of silicon and copper samples, $1 \times 1 \text{ mm}^2$ in cross section and of $300 \text{ }\mu\text{m}$ thickness, with a heating rate of 0.5 K min^{-1} . The observed differences are within the range of discrepancies already declared in the text for the presented results and cannot be definitely ascribed to an improved uniformity of specimen heating.

The results obtained for the background signal point to the direction in which some further improvements of the device could be made. The thickness of the electrodes could be decreased, which would reduce the magnitude of the background signal. This could be done to the extent that they could still be considered thick. Simple thin aluminium coating on the quartz disc, apparently the most efficient solution, might not be the most convenient—as discussed in section 2, a thin electrode geometry would require further reduction of the gap width (it is already smaller than usually reported in quoted literature) in order to retain the same sensitivity and linearity of the device. In the present arrangement no attempt is made to restrict convection upward since its effect tends to reduce the magnitude of the background signal. On the basis of present knowledge it would be possible to optimize the electrode thickness and the convection (with a ceramic fibre plug) which might result in an almost complete annulment of contributions to the background signal.

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