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An Improved Application of the Transient Hot-Wire Technique for the Absolute Accurate Measurement of the Thermal Conductivity of Pyroceram 9606 up to 420 K

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Abstract This article describes the final refinements of a novel application of the transient hot-wire technique developed for the absolute, accurate measurements of the thermal conductivity of solids. Although the technique was originally developed five years ago, these new refinements allow a full understanding of the method and hence the performance of measurements with an absolute uncertainty of less than 1 %. New measurements of Pyroceram 9606 up to 420 K are reported. The maximum deviation of the present measurements is 0.54 %, while their standard deviation at the 95 % confidence level is 0.25 %. Since May 2007, Pyroceram 9606 is a European Commission certified thermal-conductivity reference material, designated as BCR-724, with an uncertainty of ±6.5 % at the 95 % confidence level.

Keywords BCR-724 · Ceramic · Pyroceram 9606 · Solid · Thermal conductivity · Transient hot wire

1 Introduction

Over the last five years in a series of papers [1–4], a novel application of the transient hot-wire technique for the measurement of the thermal conductivity of solids was described. The advantages of the proposed application are:

(a) Contact resistance between wire sensor and solid was minimized, as the wire sensor was placed in a soft silicone paste between two blocks of the solid. At very short times, the generated heat wave is confined to the paste; hence, the properties

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of the paste can be obtained. Knowing those, at larger times the properties of the solid can be obtained.

(b) The method is based on a full theoretical model with equations describing the heat generated in the wire, and the heat transferred in the intermediate layer and the solid. The equations are solved by a finite-element method applied to the exact geometry, over the entire temporal rise of the temperature.

(c) The technique can be validated by employing the sensor for measurements of the thermal conductivity of reference liquids.

(d) Very small temperature rises are needed, typically 3 K–4 K over a period of 10 s.

The measurements were shown to be absolute and characterized by a low uncertainty. The technique was employed for the measurement of the thermal conductivity of various solids such as Pyroceram 9606 [2,3], Pyrex 7740 [3], and Stainless Steel AISI 304L [4].

In this article the following refinements are presented:

- The use of COMSOL FEM software to accurately describe the geometry of the sensor (for measurements from 20 μs to 10 s).
- The use of stainless-steel spacers to define exactly the distance between the two solid samples.
- The use of a new silicone paste of higher thermal conductivity to allow the calculation of the thermal conductivity of the solid with lower uncertainty.

Finally, the technique is applied to the measurement of the thermal conductivity of Pyroceram 9606 up to 420 K. Pyroceram 9606 is a glassy ceramic, originally developed by NASA, and since it is particularly well defined and thermally stable, it was proposed as a standard reference material for thermal conductivity by the National Institute of Standards and Technology (NIST), USA [5]. Moreover, since May 2007, Pyroceram 9606 is supplied by the European Commission Institute for Reference Materials and Measurements (http://www.irmm.jrc.be/) as a certified thermal-conductivity and thermal-diffusivity reference material (designated as glass ceramic BCR-724) up to 1,025 K [6]. This certification was the outcome of a research project, funded by the European Union under the 'Competitive and Sustainable Growth' program ('HTCRM—High Temperature Certified Reference Materials,' Contract SMT4-CT98-2211/2003 [7]). The uncertainty of the certified thermal conductivity value was ±6.5 %, while that of the thermal diffusivity was ±6.1 %. Therefore, measurements of very low uncertainty are still in need today.

2 Experimental

In the transient hot-wire technique, the temporal rise of a thin wire placed in the test material, initially at equilibrium, is observed following the application of a step voltage across the wire. The wire acts both as a heat source producing a time-dependent field within the material, and as a thermometer, registering the temporal resistance change and thus its temperature rise. To eliminate end effects, two wires differing only in length are employed. In the specific case of measurements of the thermal conductivity of solids [1–3], in order to minimize contact resistance between the wire sensor and

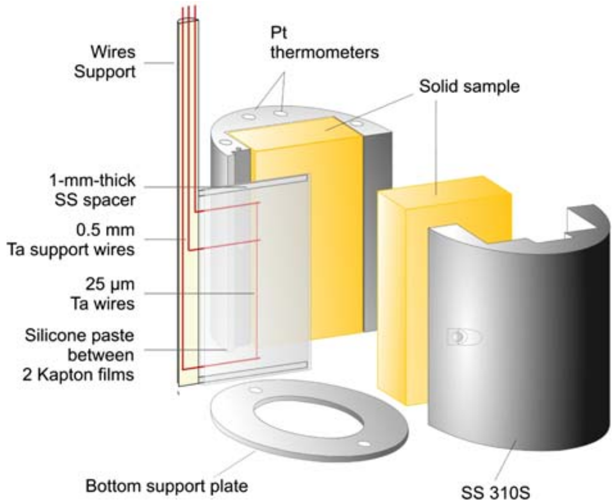

Fig. 1 The new sensor

the solid, the wire sensor is placed inside a flat layer of soft silicone paste which in turn is squeezed between two blocks of the solid. Heat is transferred from the wire via the silicone paste to the solid.

In Fig. 1, the new sensor, composed of two 25-μm-diameter Ta wires, is shown. The two wires, of 2 cm and 5 cm lengths, are spot-welded on 1 mm Ta support wires which are flattened to 0.5 mm at their ends. Two 1 mm-thick-SS strips on the top and bottom of the sensor ensure that the solid samples are kept exactly 1 mm apart, and hence the paste thickness is also equal to 1 mm. In a specially made base, two 25-μm-thick polyimide films (Kapton, DuPont) (10×5) cm are held while silicon paste (AS1803, ACC Silicones) is laid over them. The sensor with the wires is placed on top of one of them, while the other is placed over it. Then the specially made base is squeezed tight to 1 mm. After about 1 week, the silicone paste becomes harder but still retains some elastic properties. This produces an elastic layer of exactly 1 mm thickness, with Kapton on the outside, and the silicone paste with the two wires on the inside. Furthermore, this assembly ensures that the Ta wires are in the middle of the silicone layer. To measure the thermal conductivity, the sensor is squeezed between the two pieces of the solid sample.

The whole arrangement is placed in the center of an accurate, vertical three-zone tubular furnace (Carbolite, Model TVS 12). The temperature is determined using two platinum-resistance thermometers embedded on the top and bottom of the half cylinder. The thermometers were calibrated versus a Class I certified, Tinsley platinum resistance thermometer, to a maximum uncertainty of less than ±80 mK (all temperatures refer to ITS-90). Preliminary measurements with platinum resistance thermometers placed in the position of the wires, showed a 0.4 K maximum difference between the top and the bottom of the sensor cylinder and a 0.05 K maximum difference radially. These differences have no significant effect in the final quoted uncertainty of the thermal-conductivity measurement.

During a single run, 1,000 temperature versus time points are registered usually from 20 μs up to 10 s, with a very accurate bridge described elsewhere [1]. In this technique in general, there are five unknown parameters:

- the thickness of the paste (which, in the new sensor, is exactly equal to 1 mm)
- the paste's thermal conductivity and (density × specific heat capacity) product, and
- the solid's thermal conductivity and (density × specific heat capacity) product.

The parameters concerning the paste properties are obtained from measurements at short times (20 μs–0.3 s). Having acquired those, the parameters related to the solid can be obtained from measurements at longer times (0.3 s–10 s).

In this work, the COMSOL Multiphysics V.3.2b finite-element package is employed to fully describe the complete geometry of the sensor. The COMSOL package has the advantage that its mesh can consist either triangular or quadrilateral elements. This allows precise modeling of the sensor, i.e., the wire, the silicone paste, the Kapton film (negligible effect), and the solid, resulting in perfect agreement between the experimental points and the calculated temperature versus time curve from 20 μs to the last time measurement. Hence, full advantage of the complete temperature versus time curve is obtained, as will be seen in the figures in this article.

3 Measurements

3.1 Validation of the Technique

An advantage of the proposed configuration is that it can also be employed to measure the thermal conductivity of fluids. Liquid toluene has been proposed by the Subcommittee on Transport Properties of the International Union of Pure and Applied Chemistry as a standard thermal-conductivity liquid with an uncertainty of ±0.6 % [8].

In this case, the wires in their support were placed in toluene at 302 K and 1,000 temperature versus time points were obtained. To calculate the thermal conductivity of toluene, the COMSOL package was employed, but instead of the properties of the silicone layer, the Kapton film, and the solid, the properties of liquid toluene were used. In Fig. 2, the temperature rise versus the logarithm of the time is shown. The thermal conductivity value obtained from the superimposition of the COMSOL calculated values and the experimental ones, deviates by 0.4 % from the aforementioned recommended value, whereas the product (density × specific heat capacity) was found to deviate by ±0.3 % from literature values [9,10].

As already mentioned, the technique employed is an absolute technique. The final uncertainty is associated with the uncertainties in the following variables:

(a) Uncertainty in the voltage supplied to the wires. A digital voltmeter (HP 34401A) is employed with an uncertainty of ±1 μV. This results in an uncertainty in the final thermal conductivity value of the order of 10^{-6} %.
(b) Uncertainty in the temperature coefficient of resistance of the tantalum wires. This was obtained by measuring accurately the resistance of the wires at known

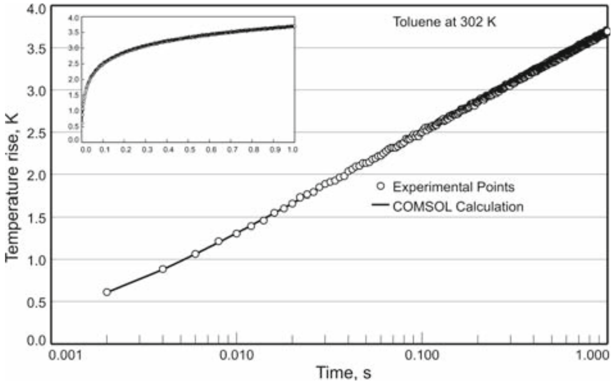

Fig. 2 Temperature rise as a function of time for toluene at 302 K

temperatures. The resistance of the wires was measured with a ±0.01 % uncertainty, while the temperature was recorded by a Class I (±1 mK) certified Tinsley platinum resistance thermometer. Hence, the temperature rise is obtained with an uncertainty of better than ±20 mK, which results to an uncertainty of the final value of the thermal conductivity of less than ±0.05 %.

(c) Uncertainty in the real time. Time is recorded in a 16-bit mode, i.e., with an uncertainty of ±1 μs. However, in the thermal conductivity calculation, the logarithm of the real time is involved. This results in an uncertainty in the final thermal conductivity value of ±0.001 %.

Taking into account the aforementioned discussion as well as the uncertainty in the temperature of the furnace discussed previously, the estimated absolute uncertainty of the technique is better than ±1 %. This estimate concurs very well with the measured value for the thermal conductivity of toluene.

According to the aforementioned discussion, the uncertainty in the measurement of the product (density × specific heat capacity) is about ±5 %.

3.2 Pyroceram 9606

3.2.1 Results and Uncertainty

Following the toluene measurements, the wires were placed in the silicone paste, and the sensor, prepared as described in the previous section, was placed between two blocks of Pyroceram 9606 as shown in Fig. 1. The two blocks of Pyroceram 9606 were made by Corning Inc., New York and purchased from Anter Corporation, Pittsburgh, USA.

The results for the thermal conductivity and the product (density × specific heat capacity) are shown in Table 1. Figures 3 and 4 show a typical run at 353.46 K. Figure 3 shows 1,000 measurements between 0.001 s and 1 s. The first part of the curve is related to the properties of the tantalum wire, while the second part refers to the silicone paste. Similarly in Fig. 4, 1,000 measurements between 0.01 s and 10 s are shown.

Table 1 Measured properties of Pyroceram 9606 as a function of temperature

T(K)	$\lambda(\mathrm{W \cdot m^{-1} \cdot K^{-1}})$	$\Delta\lambda^{a}$ (%)	$\rho C_p(\mathrm{kJ \cdot m^{-3} \cdot K^{-1}})$	$\Delta\rho C_p^{b}$ (%)
314.18	3.76	−0.20	1,986	0.03
333.74	3.71	0.35	2,103	−0.30
353.46	3.63	−0.05	2,233	0.61
373.05	3.58	0.20	2,310	−0.17
392.90	3.50	−0.54	2,388	−0.27
413.21	3.48	0.29	2,466	0.19

[a] $\Delta\lambda = 100 \times [(\lambda - \lambda_{\mathrm{fit}})/\lambda_{\mathrm{fit}}]$, with λ_{fit} from Eq. 1

[b] $\Delta\rho C_p = 100 \times [(\rho C_p - \rho C_{p,\mathrm{fit}})/\rho C_{p,\mathrm{fit}}]$, with $\rho C_{p,\mathrm{fit}}$ from Eq. 2

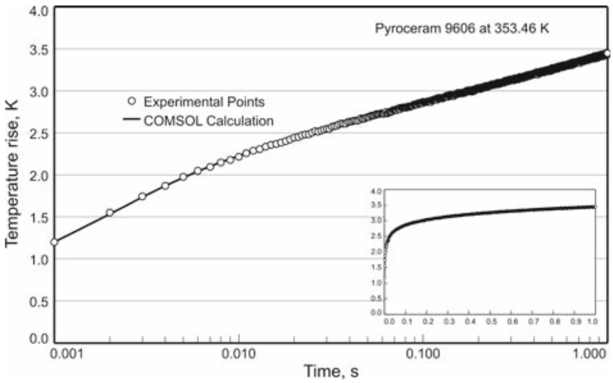

Fig. 3 Temperature rise as a function of time for Pyroceram 9606 at 353.46 K up to 1 s

In this case, the first part of the curve refers to silicone paste while the second part to the properties of Pyroceram 9606.

To analyze the sensitivity of the technique to the thermal conductivity of Pyroceram 9606, simulations were carried out varying the value of its thermal conductivity by ±1 % around the value which provides the best representation of data. Figure 5 shows the results obtained, where it can be seen that the differences in the fit arising from this small change in the thermal conductivity of the solid affects only the very long times of the temperature rise of the wire and has no effect upon the heat transfer process inside the sensor. After $t > 1$ s, the fit to the experimental data achieved by the perturbed values of the thermal conductivity of Pyroceram 9606 is discernibly worse than for the optimal value ($\lambda = 3.63\,\mathrm{W \cdot m^{-1} \cdot K^{-1}}$).

The preceding discussion has provided strong evidence that the heat transfer model developed for the description of the hot-wire sensor is entirely consistent with the practical operation of the sensor over a time range of five orders of magnitude as the heat pulse transmits through four different materials. Following the uncertainty analysis performed in the case of toluene, it is possible to assert that the uncertainty in the

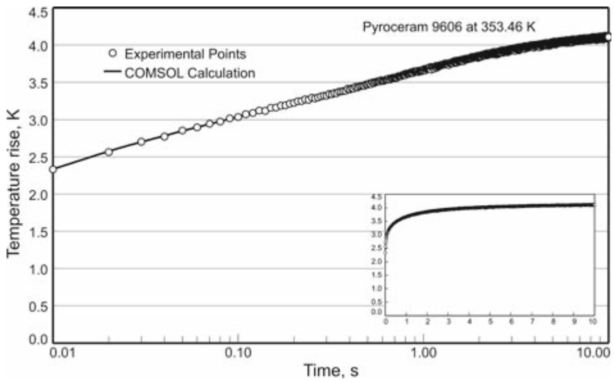

Fig. 4 Temperature rise as a function of time for Pyroceram 9606 at 353.46 K up to 10 s

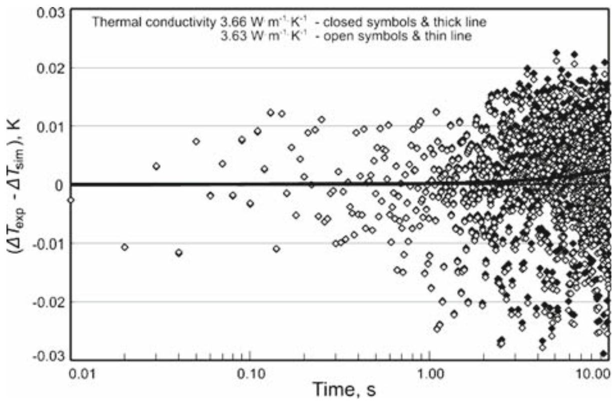

Fig. 5 Differences between experimental and simulated temperature rise for 1% change in the optimum thermal conductivity value for Pyroceram 9606

thermal conductivity of the solid determined by this technique is better than ±1 %. For the same reasons, the uncertainty in the measurement of the product (density × specific heat capacity) is determined to be ±5 %.

One last point that should be mentioned is the choice of a silicone paste. The ideal silicone paste should be of high thermal conductivity and still retain its elastic properties. A silicone with low thermal conductivity prevents the heat transfer from the wire to the solid and therefore the solid's thermal conductivity barely affects the evolution of the temperature of the wire. This effect is more intense and affects the uncertainty of the technique, when the solid sample is a relatively good thermal conductor. Furthermore, the elasticity of the paste is important as it ensures good contact between the sensor and the solid. The use of pastes with low elasticity resulted in the appearance of contact resistance between the solid and the sensor.

Table 2 Coefficients of Eqs. 1 and 2

Eq. 1		Eq. 2	
$\lambda(298.15\,\text{K})\ (\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1})$	3.83	$\rho C_p(298.15\ \text{K})\ (\text{kJ}\cdot\text{m}^{-3}\cdot\text{K}^{-1})$	1,873
a_0 (−)	1.485	b_0 (−)	−1.078
a_1 (−)	−0.6596	b_1 (−)	2.990
a_2 (−)	0.1745	b_2 (−)	−0.912

3.2.2 Analysis of Results

The thermal conductivity, λ $(\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1})$, values shown in Table 1, were fitted as a function of the absolute temperature $T(\text{K})$ to an equation,

$$\lambda = \lambda(298.15\,\text{K}) \sum_{i=0}^{2} a_i \left(\frac{T}{298.15}\right)^i, \tag{1}$$

where the thermal conductivity, $\lambda(298.15\ \text{K})$ $(\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1})$, and the coefficients a_i (−) are given in Table 2. The maximum deviation of the experimental points presented in Table 1 from the above equation is 0.54 %, and the standard deviation at the 95 % confidence level is 0.25 %.

The values for the product (density × specific heat capacity), $\rho C_\text{p}(\text{kJ}\cdot\text{m}^{-3}\cdot\text{K}^{-1})$, shown in Table 1, were also fitted as a function of the absolute temperature $T(\text{K})$ to an equation,

$$\rho C_p = \rho C_p(298.15\,\text{K}) \sum_{i=0}^{2} b_i \left(\frac{T}{298.15}\right)^i, \tag{2}$$

where the product (density × specific heat capacity), $\rho C_\text{p}(298.15\,\text{K})$ $(\text{kJ}\cdot\text{m}^{-3}\cdot\text{K}^{-1})$ and the coefficients b_i (−), are given in Table 2. The maximum deviation of the experimental points presented in Table 1 from the above equation is 0.61 %, and the standard deviation at the 95 % confidence level is 0.26 %.

4 Discussion

In discussing the present thermal-conductivity measurements that extend to 420 K, it should be remembered that the technique is absolute and is based on a full theory with an uncertainty of better than 1 %. Moreover the maximum deviation of the experimental points presented in Table 1 from Eq. 1 is 0.54 %, and the standard deviation at the 95 % confidence level is 0.25 %. In Fig. 6, the deviations of the present measurements for the values calculated by Eq. 1 are shown. In the same figure, our previous values [2,3] (two sets with different silicone pastes) with an uncertainty of better than ±2 % are included. The agreement is excellent.

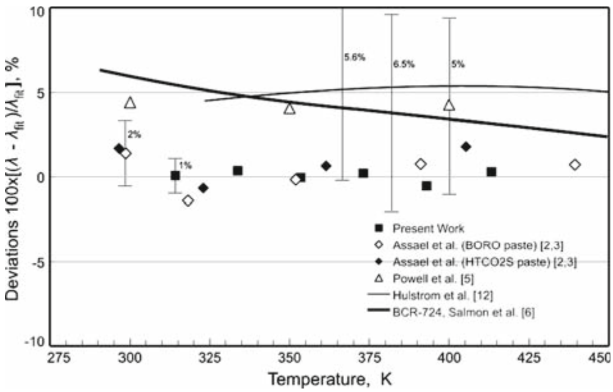

Fig. 6 Percentage deviations of the thermal conductivity values from those calculated by Eq. 1

The history of establishing Pyroceram 9606 as a certified thermal-conductivity standard of low uncertainty is quite long, as it is a very stable well-defined solid. Three major projects will be discussed here. Although in all cases the temperature reached was over 1,000 K, the discussion will be restricted to 450 K which is the highest temperature of the present measurements.

In 1966, Powell et al. [5] proposed Pyroceram 9606 as a thermal conductivity reference solid with an uncertainty of ±5 % over the temperature range from 200 K to 1,000 K (no confidence level was specified). In this work, six laboratories took part with only one set from direct thermal-conductivity measurements [11], whereas all other measurements were of thermal diffusivity performed by the laser-flash or radial heat-flow techniques. The deviations of the recommended values of Powell et al. [5] from the values calculated by Eq. 1, are shown in Fig. 6. The maximum deviation from the present set is 4.3 %, which is well within the mutual uncertainties. This comparison is quite interesting as in 1966, techniques were not as advanced as today.

In 1988, Hulstrom et al. [12], following an interlaboratory measurements program reported new recommended thermal conductivity values of Pyroceram 9606 up to 570 K, with ±5.6 % uncertainty at the 95 % confidence level. In referring to the previous values by Powell et al. [5], Hulstrom quotes ‘It would appear that the overall state of the art in thermal-conductivity measurements for materials in this range has changed little over the last 18 years.’ In the round-robin project there were seven participants:

(a) South Dakota School of Mines, USA
(b) Dynatech R/D Company, Cambridge, Massachusetts, USA
(c) Properties Research Laboratory, USA
(d) Sandia National Laboratories, USA
(e) Austrian Research Centre at Seibersdorf, Austria
(f) General Electric Company, USA
(g) University of Manchester, UK

The techniques employed by these participants were:

– Guarded hot-plate technique
– Flash thermal-diffusivity technique
– Comparative technique

The guarded hot-plate technique was operated as an absolute technique for the direct measurement of the thermal conductivity of Pyroceram 9606 by two laboratories with an uncertainty of better than 15 %. Measurements between the two laboratories differed by 14 %. Flash thermal-diffusivity measurements on the other hand, obtained in an absolute way, were found to differ in excess of 20 %. Finally, measurements performed by the comparative technique, were found less susceptible to errors and a spread of the values of about ±5 % was observed. To obtain the recommended values, an iterative algorithm was adopted according to which all data that deviated by more than two standard deviations from the master fit were excluded. The deviations of the recommended values by Hulstrom et al. [12] from the values calculated by Eq. 1 are shown in Fig. 6. The maximum deviation from the present set is 5.3 %, which is still within the mutual uncertainty of the values.

As already discussed, since May 2007, Pyroceram 9606 is supplied by the European Commission Institute for Reference Materials and Measurements (http://www.irmm.jrc.be/) as a certified thermal-conductivity and thermal-diffusivity reference material (designated as glass ceramic BCR-724) up to 1,025 K [6]. This certification was the outcome of a research project, funded by the European Union under the 'Competitive and Sustainable Growth' program ('HTCRM—High Temperature Certified Reference Materials,' Contract SMT4-CT98–2211/2003 [7]). The uncertainty of the certified thermal conductivity value was ±6.5 %, while that of the thermal diffusivity was ±6.1 % at the 95 % confidence level.

In this project, the following laboratories participated:

(A) Austrian Research Centre GmbH, Materials Technology, Seidersdorf, Austria
(B) CeramRes, Queen's Road, Stoke-on-Trent, UK
(C) Corus R&D, Ceramics Research Centre, Ijmuiden, the Netherlands
(D) Constantine the Philosopher University, Physics Department, Slovakia
(E) Forschungsinstitut fur Wärmeschutz, Gräfelfing, Germany
(F) INSA, Centre de Thermique de Lyon, Vilieurbanne cedex, France
(G) Forschungsinstitut fur Kerntechnik und Energiewandlung, Stuttgart, Germany
(H) Institute for Reference Materials and Measurements, EC, Geel, Belgium
(I) Laboratoire National d' Essais, Trappes Cedex, France
(J) Netzsch Gerätebau GmbH, Selb/Bayern, Germany
(K) National Institute of Science and Technology, Building and Fire Research, Gaithersburg, USA
(L) National Physical Laboratory, CBTM, Teddington, UK
(M) Physikalisch-Technische Bundesanstalt, Dept. 3.102, Braunschweig, Germany
(N) Sociétè Francaise de Céramique, Ceramique Industrielles, Paris, France

The thermal conductivity measurements included in the project were obtained by the following techniques:

- Three sets by the guarded hot-plate technique (G, L, M),
- Four sets by the parallel transient hot-wire technique (B, C, L, N),
- One set by the resistive transient hot-wire technique (L), and
- One set by the transient hot strip (M).

Five more sets of measurements (A, G, I, J, L) were included for the thermal-diffusivity certification.

In this round-robin project, for the first time, the transient techniques for the measurement of the thermal conductivity dominate the picture. Unfortunately the situation has not improved. The uncertainty has increased to ±6.5 % at the 95 % confidence level. In Fig. 6, the deviations of the certified values by IRMM from the values of Eq. 1 are shown. Although the deviations are still within the mutual uncertainties, some points must be made in relation to these certified values.

Around 300 K, the deviations of seven sets of measurements from the proposed correlation are between −2 % and −4 %, whereas the other two sets (laboratories L and N) are between +5 % and +11 %. Although the guarded hot-plate instruments seem to be properly designed, it is the author's belief that the transient hot-wire instruments should not be operated in the parallel mode if low uncertainty is required.

In the parallel mode of the transient hot-wire instrument instead of the wire acting both as sensor and a thermometer, a thermocouple is placed usually 2 cm from the heating wire to register the temperature. The heating wire is usually 0.35 mm–0.50 mm in diameter made of platinum with a length of about 20 cm, resulting in a resistance of about 0.1 Ω–0.2 Ω, which in turn requires high operating power (in the present work, two 25 μm tantalum wires of about 5 cm and 2 cm lengths are employed, resulting in a total resistance of 20 Ω and less than $3\,W \cdot m^{-1}$ power). The large power and the low resistance can easily produce larger uncertainties. Moreover, the use of a single wire instead of two enhances the errors associated with the end effects of the wire.

Another point that should be added is that, in this form of the parallel mode to obtain the thermal conductivity, only a part of the temperature versus time curve is used instead of the whole curve. Hence, the choice of this 'part' affects the uncertainty of the results. Furthermore, the uncertainty of the method is also affected by the appearance of contact resistance when the wire and the thermocouple are embedded in the solid. The contact resistance still exists even if powders are employed.

Following the aforementioned discussion, it is the authors' belief that techniques employed in proposals for reference thermal-conductivity materials should be further developed so as to minimize errors.

5 Conclusions

In this article the instrumentation for the measurement of the thermal conductivity of solids has been described. Evidence has been adduced to support the consistency between the theoretical model and the practical operation of the sensor. Following an uncertainty analysis, it is possible to assert that the uncertainty in the thermal conductivity of the solid determined by this technique is better than ±1 %, while the uncertainty in the measurement of the product (density × specific heat capacity) is ±5 %.

Measurements of the thermal conductivity of Pyroceram 9606 up to 420 K are presented and discussed in relation to recently proposed certified thermal-conductivity values for this material.

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