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WIDE RANGE DETERMINATION OF THERMAL CONDUCTIVITY BY DIFFERENTIAL SCANNING CALORIMETRY

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ABSTRACT

A differential scanning calorimeter was used for the determination of the thermal conductivities of various solid materials having a wide range of values. The tested materials were teflon, duraluminium and electrolytic copper. This method provides a reliable way to measure λ in the range 0.200–400 W m^{-1} K^{-1} for temperatures between 40 and 100 °C.

INTRODUCTION

Differential scanning calorimetry (DSC) has already been applied for the determination of thermal conductivity [1–3]. Polymeric materials and glasses have been studied most extensively. Our laboratory has frequently been requested to perform rapid determinations of the thermal conductivity of various materials. Therefore we decided to explore the applicability of the DSC technique over a wide range of conductivity values. Teflon, duraluminium and copper, which have low, intermediate and high values of the thermal conductivity respectively, were tested in the range 40–100 °C.

EXPERIMENTAL

Teflon, duraluminium and electrolytic copper were commercially available from Mexican suppliers. Samples were machined in cylindrical shapes (diameter, 6.25 mm; height, 24.36 mm). The diameter of the samples was the same as that of the DSC cell. Smaller diameters which produce heat leakages that are difficult to evaluate were not considered.

The calorimeter was a Perkin–Elmer 1B with the cover of the sample holder assembly replaced with a modified aluminium cover similar to that

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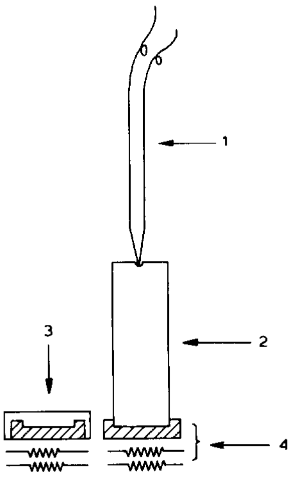

Fig. 1. Schematic diagram representing measurement using the "direct" method: 1, thermocouple; 2, sample cylinder; 3, teflon cover; 4, power compensation device of the calorimeter.

described by Chiu and Fair [1]. High purity indium and tin were used for temperature and power calibration which have been described elsewhere [4].

The temperatures at the top of the samples were measured using chromel–alumel thermocouples and they were calibrated using a platinum resistance thermometer at the Metrology Laboratory in this Research Centre. In all cases silicone grease was used to provide good thermal contact at the cell–cylinder–thermocouple junctions.

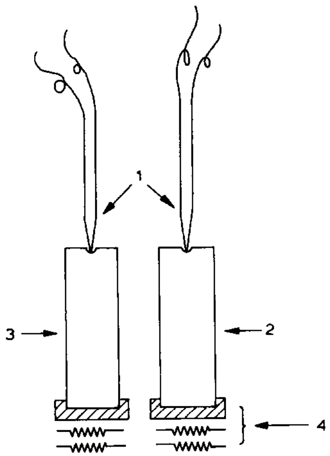

Fig. 2. Schematic diagram representing measurement using the "differential" method: 1, thermocouples; 2, sample cylinder; 3, reference cylinder; 4, power compensation device of the calorimeter.

Austenitic stainless steel (SRM 1461), provided by the National Bureau of Standards, was used as reference material with certificate values of thermal conductivity.

"Direct" and "differential" methods were tested and they are schematically represented in Figs. 1 and 2, respectively. In the "direct" method the cylinder of the sample to be tested was placed in the sample cell and one piece of teflon was placed in the reference cell to minimize heat leakage. The heat flux was determined directly from the steady state signal of the calorimeter, taking into account leakage through the teflon from a blank run. In the "differential" method, a cylinder of the reference material with the same geometric dimensions as the sample was used. The difference in the heat flux through the cylinders (with their bases maintained at the same temperature) was taken from the calorimetric signal in relation to the zero line, both at steady state and at the temperature of measurement.

RESULTS AND DISCUSSION

In the "direct" method the thermal conductivity was calculated using the Fourier equation

$$Q = \lambda \frac{A}{l} \Delta T$$

where Q is the heat flux, λ is the thermal conductivity, A is the cross-sectional area, l is the height of the cylinder and ΔT is the temperature difference between the bottom and the top of the cylinder.

In the case of the differential method, thermal conductivity was calculated using the equation

$$\lambda_x = \frac{\lambda_r \Delta T_r - (l/A)(Q_r - Q_x)}{\Delta T_x}$$

which results from the application of the Fourier equation to the sample (x) and reference (r) cylinders. The quantity $Q_r - Q_x$ is directly provided by the calorimeter signal. $\Delta T_r = T - T_r$ and $\Delta T_x = T - T_x$, where T is the temperature at the bottom of the cylinders (taken from the calorimeter) and T_r and T_x are the temperatures at the top of the reference and sample cylinders (both measured with the thermocouples).

The thermal conductivity of the reference material at the experimental temperature was calculated from the equation

$$\ln \lambda(\text{in W m}^{-1}\text{K}^{-1}) = A + B(T/\text{K}) + C(T/K)^2$$

with $A = 2.035$, $B = 2.700 \times 10^{-3}$ and $C = 0.206 \times 10^{-5}$; this equation was obtained by fitting the certificate values of λ in the range 200–400 K.

TABLE 1

Experimental results of thermal conductivity using the "direct" method

T (°C)	Experiment number 1	2	3	4	5	λ ($W\ m^{-1}\ K^{-1}$)
Teflon						
40	0.285	0.287	0.283	0.280	0.285	0.284 ± 0.002 [a]
60	0.281	0.286	0.279	0.276	0.283	0.281 ± 0.004
80	0.283	0.284	0.275	0.279	0.284	0.281 ± 0.004
100	0.275	0.281	0.282	0.277	0.280	0.279 ± 0.002
Duraluminium						
40	168.635	169.527	168.123	168.033	168.582	168.58 ± 0.60
60	174.540	174.366	173.551	173.787	174.576	174.16 ± 0.47
80	178.417	178.078	178.994	179.222	178.054	178.55 ± 0.54
100	181.243	181.305	180.548	182.003	181.286	181.28 ± 0.51
Electrolytic copper						
40	395.576	396.736	395.728	395.464	395.866	395.87 ± 0.51
60	394.555	395.101	394.863	395.239	394.442	394.84 ± 0.33
80	393.702	394.368	393.552	393.801	393.107	393.71 ± 0.45
100	392.336	392.078	392.888	392.595	391.848	392.35 ± 0.40

[a] Uncertainties represent one standard deviation of the mean.

TABLE 2

Experimental results of thermal conductivity using the "differential" method

T (°C)	Experiment number 1	2	3	4	5	λ ($W\ m^{-1}\ K^{-1}$)
Teflon						
40	0.274	0.280	0.274	0.277	0.275	0.276 ± 0.002 [a]
60	0.274	0.271	0.276	0.277	0.274	0.274 ± 0.002
80	0.273	0.276	0.272	0.276	0.272	0.274 ± 0.002
100	0.274	0.272	0.277	0.273	0.276	0.274 ± 0.002
Duraluminium						
40	167.360	166.857	167.808	167.153	167.227	167.28 ± 0.36
60	173.011	173.572	172.683	173.289	173.130	173.14 ± 0.33
80	178.003	177.885	177.770	178.325	178.467	178.09 ± 0.29
100	181.200	181.559	180.672	181.001	181.208	181.13 ± 0.31
Electrolytic copper						
40	395.217	394.977	395.068	395.567	394.716	395.11 ± 0.31
60	394.170	393.863	394.358	394.525	393.604	394.10 ± 0.38
80	393.041	393.403	392.910	393.316	392.695	393.07 ± 0.29
100	391.235	391.786	391.411	391.525	390.603	391.31 ± 0.45

[a] Uncertainties represent one standard deviation of the mean.

TABLE 3

Thermal conductivity values of duraluminium in W m^{-1} K^{-1}

T (°C)	"Direct" (this work)	"Differential" (this work)	Ref. 11	Ref. 12	λ_{int} [a]
0			159	159.331	159.28
20			165	165.146	164.97
40	168.58(0.27) [b]	167.35(0.28)			169.70
60	174.16(0.21)	173.13(0.22)			174.27
80	178.55(0.13)	178.49(0.21)			178.40
100	181.28(0.23)	181.04(0.25)	181	181.428	181.68
200			194	194.221	194.20

[a] Interpolated values from ref. 12. [b] Numbers in parentheses are the standard deviations.

Tables 1 and 2 show the thermal conductivities determined by the "direct" and "differential" methods. For each experiment, the cylinders and thermocouples were replaced so that the results are totally independent of each other.

Tables 3 and 4 show a comparison of our results with those obtained from the literature. We also interpolated values for temperatures where no data were available from the compiled values of the National Bureau of Standards. It can be seen from these tables that the "differential" method produces lower values for λ and smaller standard deviations than the "direct" method. We expected the differential method to be more reproducible; however, the λ values obtained using both methods are the same within the standard deviation.

For teflon, the literature values of the thermal conductivity [6–10] range between 0.165 and 0.418 W m^{-1} K^{-1}; our values are in good agreement with those obtained by Hsu et al. [5] (0.272 W m^{-1} K^{-1}) for their as-received sample. We also observed the well-known temperature-independent behaviour of λ for this material.

TABLE 4

Thermal conductivity values of electrolytic copper in W m^{-1} K^{-1}

T (°C)	"Direct" (this work)	"Differential" (this work)	Ref. 13	Ref. 12	Ref. 14	λ_{int} [a]
0			401.00	395.420	393	401.20
20			398.29			398.20
40	395.87(0.23) [b]	395.24(0.25)				397.36
60	393.84(0.15)	394.06(0.19)				396.42
80	393.71(0.20)	393.02(0.22)	395.46			395.46
100	392.35(0.18)	395.57(0.21)	394.49	391.931	385	394.50
200			389.42			389.43

[a] Interpolated values from ref. 13. [b] Numbers in parentheses are the standard deviations.

In the case of duraluminium, our results are in excellent agreement with values in the literature [11,12] and with values interpolated from ref. 12. This agreement is probably due to the fact that λ is not very sensitive to the chemical composition of the alloy. In contrast, for electrolytic copper (a pure metal) our values are lower than those reported in refs. 12–14. The difference cannot be explained by experimental error. We believe that it is due to variations in the purity of the materials studied by different researchers; nevertheless, the decrease in λ with temperature is well detected by our experiments.

From this study we conclude that it is possible to obtain reliable results of thermal conductivity for solid materials in the range 0.200–400 W m^{-1} K^{-1} for temperatures of at least 100 °C using differential scanning calorimetry. This is the maximum temperature tested in this study and at higher temperatures heat loss by radiation may become more important.

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