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UNCERTAINTY OF THE THERMAL CONDUCTIVITY MEASUREMENT USING THE TRANSIENT HOT WIRE METHOD

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Abstract

This work deals with uncertainty analysis of the thermal conductivity measurement using the transient hot wire method. The characterization is made from a sample of low-density, polyethylene BRALEN SA 200-22. The utilized experimental data are obtained from the test measurements performed on the air at room temperature. The sources of measurement errors are analyzed and the uncertainty of the measured value of the thermal conductivity is evaluated. The analysis shows that in the present case the uncertainty of the thermal conductivity measurement is about ±3.3% for 68% confidence level.

Keywords: measurement uncertainty, thermal conductivity, transient hot wire method

Introduction

The reliability of every measurement confirms a quantitative statement of its uncertainty that accompanies it. General rules for evaluating and expressing uncertainty in measurement, which can be followed at various levels of accuracy, have been established as the GUM method (Guide to the Expression of Uncertainty in Measurement) [1, 2]. The method has been adopted by various regional metrology and related organizations worldwide.

The GUM approach has been followed in expressing the uncertainty of an estimation of several thermophysical properties including thermal conductivity using the transient hot strip technique [3] or the guarded hot plate technique [4] as well as thermal diffusivity using the laser flash method [5].

Here we present the uncertainty analysis of the thermal conductivity measurement using the transient hot wire method [6], which is a standard test method for measuring thermal conductivity. The hot wire method has been successfully applied to

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the measurement of liquids and gases [7–9] and also to solids [10] with considerable variability in accuracy of result.

General classification of uncertainty components

Every measurement is affected by measurement errors that cause the difference between the measured value of the estimated property (in our case the thermal conductivity) and its true value. The true value associated with the measured property is an idealized notion, which cannot be determined. It is only an approximation or an estimate of the value subjected to the measurement. The value assumed to be a true value usually comes from various independent measurements.

The uncertainty of the result of a measurement generally consists of several components, which may be grouped into two categories according to the method used to estimate their numerical values:

Type A standard uncertainties are evaluated by the statistical analysis of a series of observations. An evaluation may be based on any valid statistical method for treating data, i.e. calculating the standard deviation of the mean of a series of independent observations; using the method of least squares to fit a curve to data in order to estimate the parameters of the curve and their standard deviations; and then carrying out an analysis of variance in order to identify and quantify random effects in certain kinds of measurements.

A Type B evaluation of standard uncertainty is usually based on scientific judgment using all the available relevant information, which may include previous measurement data; experience with, or general knowledge of; the behavior and property of relevant materials and instruments; manufacturer's specifications; data provided in calibration and other reports; and uncertainties assigned to reference data taken from handbooks.

All the individual uncertainties influence the uncertainty of the result measurement and they should therefore be combined. The combined standard uncertainty $u_c(y)$ of a measurement result y represents the estimated standard deviation of the result. It is obtained by combining the individual standard uncertainties u_i arising from a Type A or a Type B evaluation, using the usual method for combining standard deviations based on the law of propagation of uncertainty [1, 2].

If the probability distribution characterized by the measurement result is approximately normal (Gaussian), then it is believed with an approximate level of confidence of 68 % that the measurement result (measurand Y) is greater than or equal to $y-u_c(y)$, and is less than or equal to $y+u_c(y)$, written as $Y=y\pm u_c(y)$.

Hot wire method

The simple measurement consists of measuring the temperature rise *vs.* time evaluation of an electrically heated wire embedded in a tested material. The thermal conductivity is derived from the resulting change in the temperature over a known time interval.

The ideal analytical model assumes an ideal – infinite thin and infinite long line heat source (hot wire), operating in an infinite, homogeneous and isotropic material with uniform initial temperature T_0. If the hot wire is heated for the time t=0 with constant heat flux q per unit wire length, the radial heat flow around the wire will occur. The temperature rise $\Delta T(r,t)$ in any distance r from the wire as a function of time describes the simplified equation [11]

$$\Delta T(r,t)=\frac{q}{4\pi k}\ln\frac{4at}{r^2C} \tag{1}$$

where k is the thermal conductivity, a thermal diffusivity and C=exp(γ), with γ the Euler's constant. The thermal conductivity is calculated from the slope S of the temperature rise $\Delta T(r,t)$ *vs.* the natural logarithm of the time ln$t$ evolution using the formula

$$k=\frac{q}{4\pi S} \tag{2}$$

Several corrections have been introduced to account for the heat capacity of the wire, the thermal contact resistance between the wire and the test material, the finite dimension of the sample and the finite dimension of the wire embedded in the sample [12, 13].

The hot wire method is in accordance with the way of measurement of the temperature increase and the place of the temperature sensor utilized in three main variations, known as the resistance technique [14], the standard (cross) technique [15] and the parallel wires technique [16].

Experimental apparatus

The utilized computer-controlled experimental apparatus, that allows the determination of the thermal conductivity of solid, powders and granular materials is described in detail elsewhere [17]. It allows the utilization of one of three measurement techniques: the standard cross wire technique, the resistance potential lead method and the probe modification of hot wire method.

In the present study the results of measurement obtained using the cross technique are analyzed. A wire cross is embedded in ground grooves between two equally sized samples. The cross consists of a linear heat source – the kanthal wire 0.4 mm in diameter (Bulten Kanthal AB) and of a spot welded thermocouple, K type, made from Ni–NiCr wires (Heraeus) 0.1 mm in diameter which acts as the temperature sensor. The hot spot of the thermocouple is in direct contact with the heating wire and it is placed in the center of the sample. The cold junction is put on the reference place in the Dewar cup at 0°C (Fig.1).

The current flowing through the heating wire is produced by the stabilized regulated direct current supply Z-YE-2T-X (Mesit) operated by a PC via the remote control unit JDR-1 (Mesit). The setting of the optimal current mainly depends on the sample thermal properties and dimensions and is chosen to have a hot wire temperature rise between 5–10°C. A high resolution data acquisition board PCL-818HG

(Advantech) with a lock in pre-amplifier Z-35 (Metra) is used for serial measurements of the transient *emf* of the thermocouple, and the transient voltage corresponding to the temperature rise. A proportional feedback temperature controller regulates the temperature of the electro-resistive furnace. The apparatus allows measurement in air or in a controlled environment, under atmospheric pressure, in the temperature range from room temperature up to 1200°C.

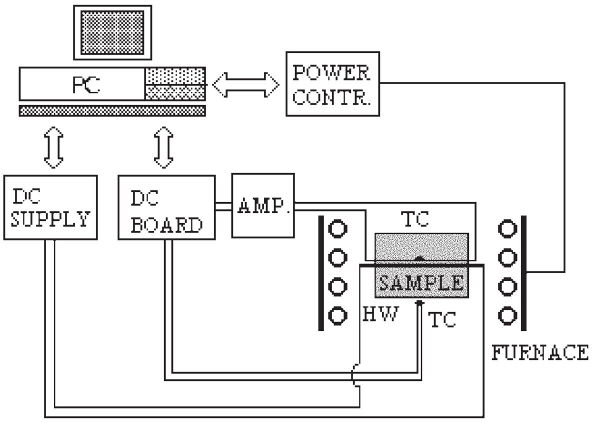

Fig. 1 Block diagram of the apparatus

The data reduction considers the calculation of the slope of the temperature rise *vs.* time evolution in logarithmic scale. This is performed using the least-squares-fitting of the data. Because of the simple model it is necessary to find a linear portion of the temperature rise curve. This is done easily by calculating the slope at various time intervals that the software easily allows to be set. The correct time interval is indicated by the independence of the slope value at the time interval used.

Samples

The measurements have been carried out on plastic samples made of low-density polyethylene (BRALEN SA 200-22). The Research Institute for the Processing and the Application of the Plastic Materials (VUSAPL Nitra) has prepared the samples from granulates produced by Slovnaft by compressed moulding. The 'wire cross' was embedded between two sample blocks of 50×100×100 mm. The thermal contact was improved using the silicon sink compound paste (Dow Corning 340). Independent measurements and comparison of experimental and analytical temperature rise *vs.* time data show that the samples' dimensions are satisfactory for reliable thermal conductivity measurements in the experimental arrangement. We have carried out measurements utilizing two different currents – 0.6 and 0.7 A, with the temperature rise kept at the level of 5°C. The measurements were performed at room temperature in air under atmospheric pressure.

To compare the results achieved, the thermal conductivity of the test material (from the same sample) has been measured on the guarded heat flow meter TCHM LT (Holometrix) installed in the Austrian Research Centers in Seibersdorf. This value has been taken as the reference value of the thermal conductivity k_{ref} of the sample material. The accuracy of the reference thermal conductivity value k_{ref} is about 3%.

Experimental results

Figure 2 presents the typical temperature rise *vs.* time evolution. We can see that the linear portion of the curve starts above 50 s and it can be confirmed that it does not finish below 300 s.

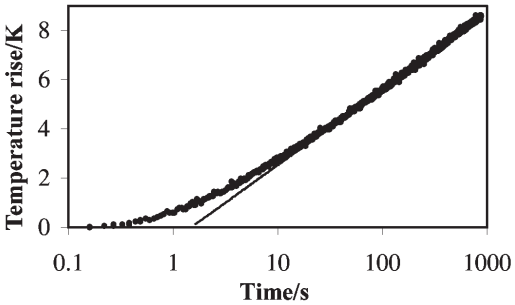

Fig. 2 Typical experimental temperature rise *vs.* time data and its least-squares-fit

Table 1 Results of the thermal conductivity measurement

No.	k_1/W m^{-1} K^{-1}	k_2/W m^{-1} K^{-1}
1	0.321	0.313
2	0.299	0.319
3	0.295	0.309
4	0.329	0.312
5	0.326	0.309
6	0.330	0.313
7	0.312	0.312
8	0.322	0.311
9	0.323	0.307
10	0.322	0.313
mean	0.3180	0.3119

Table 1 summarizes the results of the thermal conductivity for two sets of measurements performed by independent measurements on the same test sample. The thermal conductivity values presented here are values calculated for each measurement as the average of five values, obtained from a least-squares-fit of the linear part of the recorded temperature rise *vs*. time data. The fitting was performed at the time intervals 50–200, 80–200, 50–300, 80–300 and 100–300 s in order to check the measurement independency of the chosen time interval.

Table 2 The measured thermal conductivity values and their comparison

No.	k/W m^{-1} K^{-1}	U/W m^{-1} K^{-1}	u/%	k_{ref}/W m^{-1} K^{-1}
1	0.318	0.0121	3.08	0.319
2	0.312	0.0034	1.07	0.314
mean	0.315			0.317

The thermal conductivity results achieved as well as the calculated standard deviations are summarized in Table 2. We see a very good agreement with the reference values k_{ref}.

Uncertainty analyses

Calculation of the thermal conductivity is in the transient hot wire method performed estimating the slope of the measured temperature rise *vs.* time evolution in logarithmical scale over a defined time interval. The main sources of the thermal conductivity measurement uncertainty are connected with the measurement of the temperature, the stability of the time axis, the stability of the power supply and the satisfaction of the experimental conditions as they are supposed in the analytical model. The main sources of the non-measurement errors cause differences between the real conditions and the assumptions of the analytical model i.e., that the heating wire has finite non-zero diameter and the real heat capacity, that there is a thermal barrier between the wire and the sample, and between the temperature sensor and the wire, that the sample and the wire have finite dimensions and that the heat exchange between the sample surface may occur there. The random component of the uncertainty is evaluated statistically by analyzing the repeated measurement.

Type A uncertainty

The relative standard deviation values in Table 2 represent Type A uncertainties. It can be conducted that value 3.1% represents the Type A uncertainty component.

Type B uncertainty

Because of several sources of this type of uncertainties, all the components will be discussed individually.

Temperature measurement

The temperature is measured using the K type thermocouple made from spot-welded Ni–NiCr wires. The manufacturer specifies that its typical accuracy is better than

0.4% of the measured value. If we take account of the uncertainties in the thermocouple *emf* measurement that is in accordance with the PCL-818HG data acquisition board manufacturer of order 0.08%, we may estimate the uncertainty of the temperature measurement at value 0.5%. It is supposed that the effect causes mainly systematic error in the temperature measurement. If we calculate the thermal conductivity using the slope of the temperature rise *vs.* logarithm of the time evolution, the influence of the uncertainty of the thermal conductivity estimation on the uncertainty in the temperature measurement is negligible. We can guess that the uncertainty of the thermal conductivity is better than 0.1%.

Time base stability

The time scale is based on the PCL-818HG data acquisition board time system. The manufacturer specifies the stability and the uncertainty as better than 0.01%. The error is so small that we do not have to consider it as a source of the thermal conductivity uncertainty.

Power supply

In the measurement we let the stabilized direct current flow through the heating wire. The current is produced by the stabilized power source Z-YE-2T-X working in the stabilized current supply mode. The manufacturer specifies the current stability at level of 0.05%. The influence on the thermal conductivity uncertainty is less than 0.1%.

Non-measurement errors

The ideal analytical model considers the ideal line heat source – the infinitely long one with no heat capacity embedded in an infinite medium. The ideal thermal contact between the wire, the sample and the temperature sensor (zero thermal contact resistance) is considered here. Deviations of real experimental conditions from those considered in the theory cause a deformation of the temperature rise curve.

Non-linearity of the beginning of the graph of $\Delta T(r,t)$ *vs.* $\ln t$

Non-linearities of the initial part of the data are caused by the finite radius and non-zero heat capacity of the wire. The thermal contact resistance between the hot wire and the sample and the thermal contact resistance between the hot wire and the temperature sensor have a similar influence. To overcome this effect we need to find the certain minimum time $t_{\min}$ which corresponds to the beginning of the linear part of the curve. Time $t_{\min}$ can be determined either analytically – calculated with respect to the complex theory [17]. In our approach we utilize the interactive calculating of time $t_{\min}$ searching the linear part of the graph of the temperature rise *vs.* the natural logarithm of the time calculating the slope as a function of time. We cannot directly evaluate the uncertainty of the thermal conductivity estimation caused by these effects. We try to eliminate these effects experimentally as much as possible (by using thin wires, and by improving the thermal contact using a silicon paste) and because of the method of data reduction, based on the checking of the least-squares fit of the data to the analytical model, we suppose that uncertainty is included in the random uncertainty (here presented as Type A).

Deformation of the end of the graph of $\Delta T(r,t)$ *vs.* ln*t*

Deformation of later parts of the experimental curve mainly results in the influence of finite dimensions of the sample and finite length of the hot wire. Heat exchange at the sample surface can only be eliminated when the thermal conductivity value is calculated from that part of the temperature rise curve that is not influenced by outer boundary conditions. Practically this means performing the least squares fitting on the linear part of the curve $\Delta T(r,t)$ *vs.* ln*t*. Similarly to time t_{min}, the maximal time t_{max} can be calculated analytically (based on the thermal diffusivity of the shape and the material) or could be found by interactive searching [17]. In our experiment we use the second approach. To eliminate the other boundary effects experimentally we use relatively large samples. We eliminate the influence of the finite length of the hot wire by the measurement of the temperature evaluation in the center of the sample. We consider that the influence of the accuracy of the measurement on these effects is in our case negligible.

All the A and B Type components of the uncertainty are considered to be independent. Using the law of uncertainty propagation we can ensure that the combined standard uncertainty of the thermal conductivity is better than 3.3%.

Conclusions

This study presents the uncertainty analysis of the thermal conductivity measurement using the transient hot wire method. The series of test measurements performed on the plastic sample BRALEN SA 200-22 in air at room temperature show that the combined standard uncertainty of the thermal conductivity measurement is better than 3.3% within a 68% confidence level. The thermal conductivity values achieved are in accordance with the reference value that confirms the reliability of the measurement using the apparatus described.

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