Applicability of the Transient Plane Source Method To Measure the Thermal Conductivity of Low-Density Polyethylene Foams

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ABSTRACT: The thermal conductivity at constant pressure of a collection of crosslinked, closed-cell polyethylene foams were measured at room temperature with the transient plane source (TPS) method. The experimental results were compared with those determined by a standard steady-state technique. The results showed that the values measured by the TPS method follow the same trends as those measured by a heat-flow meter. Therefore, with the TPS technique it is possible to observe the influence of structural characteristics such as cell size, black carbon content in foams, density, and so forth on thermal conductivity. However, the values obtained by the transient method were approximately 20% higher than those given by the standard method. Possible reasons for these variations are discussed. © 2004 Wiley Periodicals, Inc. J Polym Sci Part B: Polym Phys 42: 1226–1234, 2004

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INTRODUCTION

Thermal conductivity is an important property in the application and use of cellular plastics. This property changes extensively depending on the density, cellular structure, and morphology of the polymeric matrix and so forth.[1–4] Moreover, thermal conductivity also exhibits a strong dependence on temperature and pressure.

To analyze the thermal properties of cellular polymers, it is necessary to evaluate the three mechanisms whereby, under the influence of a temperature gradient, energy can be transported from one region of space to another. These mechanisms are radiation, conduction through the solid and gaseous phases, and convection. These processes of heat transfer are often very important in a wide variety of scientific and industrial applications. Because of this reason, a number of different experimental techniques have been developed[5,6] to measure the thermal conductivity for different experimental conditions and for different materials. The fact that in most practical situations all three heat-transfer mechanisms are present greatly complicates the process of measurement of this property.

The overall range of thermal conductivity for cellular plastics is one order of magnitude above approximately 0.015 W/m K [the lowest value for a low-density (<50 kg/m^3) phenolic urethane or polyisocyuretane closed-cell insulation product
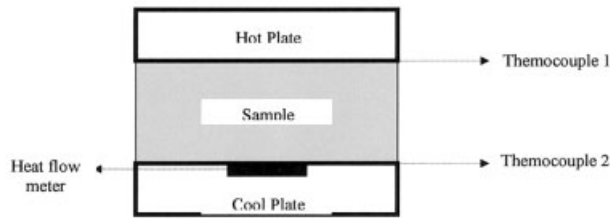
Figure 1. Diagram of a heat-flow meter.

containing a low thermal conductivity blowing agent (CFC, HCFC, etc.) in the cells]. Practically, there is no ideal method of measurement for this range of thermal conductivity, λ. It is necessary to choose a procedure depending on factors such as the expected conductivity, the shape of the material, its density, and its availability in a suitable size to be considered representative of the bulk or for the application under analysis.

To measure thermal conductivity or a related property by a steady-state or a transient method, the experimental arrangement must simulate a solution to the basic heat-conduction equation for a homogeneous isotropic solid:

$$\frac{1}{k}\frac{\partial T}{\partial t} = \nabla^2 T + \frac{A(x,y,z,t)}{\lambda} \qquad (1)$$

where T is the temperature, t is the time, k is the thermal diffusivity ($k = \lambda/\rho c_P$), c_P is the specific heat capacity at constant pressure, ρ is the density of the sample, and $A(x,y,x,t)$ is the heat generation per unit volume per unit time in the medium.

For longitudinal unidirectional heat flow, no radial losses, and without power supplied into the solid, analysis of the steady-state term leads to a lineal dependence between temperature and dimension. Then the heat per unit time and unit area through a sample can be determined by Fourier's law:

$$Q_a = G\lambda\frac{\Delta T}{d} \qquad (2)$$

where Q_a is the heat flow generated by the application of a temperature difference (ΔT) between the two sides of the sample material, separated over a distance d, and G is a constant, evaluated by calibration, for a given apparatus (see Experimental). Figure 1 shows a diagram of a typical heat-flow meter.

The steady-state techniques have found wide applications. In fact, there are a number of standard methods based on this procedure (ASTM C177 or ISO DIS 8302, ASTM C518 or ISO 8301, and ASTM F433).

Whether the sample is a solid, the heat generated in the upper plate is not all conducted to the lower plate. Thus, it is necessary in all cases to account for heat losses. Moreover, it is not always true that the heat flow is normal to the heat surfaces and there is a small gap between the two heater surfaces and those of the sample. This gap contributes to the reduction of the heat transferred (interfacial heat-transfer resistance).

There are a wide number of commercial equipments designed to work under steady-state conditions, for instance, the guarded hot plate, the unguarded hot plate, different arrangements for determining linear heat flow, or radial heat flow.[5] Although the heat-flow meters are relatively fast in operation, there is still a need to reduce such times, particularly for quality-control applications.

Transitory methods are based on the analysis of the transient term solution of eq 1, which relates change in temperature with time. Hot wire, transient hot strip, and transient plane source are techniques based on measuring the sample behavior in the transient regime of heat flow.

These methods have several advantages; for example, it is possible to obtain values of thermal conductivity, thermal diffusivity, and specific heat simultaneously. The range of measuring of these properties is wide (ca. 0.02–150 W/m K). Other benefits are that these methods can be used to measure properties of inhomogeneous and/or anisotropic materials, can offer the ability to measure in small samples, and measurements are in general fast.

The transient methods are not yet standard. However, several efforts are being developed to standardize these transient methods.[7] In the last few years, different kinds of materials (butadiene rubber compounds, pineapple leaf fiber-reinforced composites, and highly porous building materials) have been characterized by the transient plane source (TPS) technique.[4,8–12]

In this work, an experimental study on the thermal conductivity of a collection of low-density polyethylene (LDPE) foams, produced from a high-pressure nitrogen gas solution process, is presented. Measurements were performed both with a steady-state technique and with TPS. The main goal of this investigation was to check and
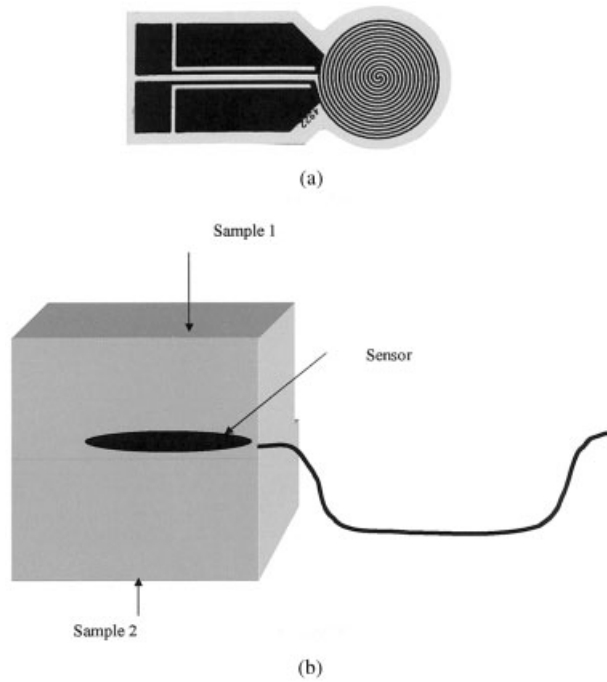
Figure 2. (a) Diagram of a TPS sensor and (b) experimental arrangement of the sensor and samples.

analyze the ability of the TPS technique as a scientific instrument for the analysis of the thermal properties of low-density foams, while trying to point out the advantages and disadvantages of this technique as compared with a standard method (heat-flow meter) widely known and used for both scientific and industrial investigations and for quality assurance.

THEORY OF TPS

Measurements of both thermal conductivity and thermal diffusivity are possible by means of the hot-disk method.[8] In this method, the TPS element behaves both as a temperature sensor and as a heat source. The TPS element consists of an electrical conducting pattern of thin nickel foil (10 μm thick) in the form of a double spiral, which resembles a hot disk, embedded in an insulating layer made of Kapton (70 μm thick) [Fig. 2(a)]. Two samples of the same material are located in contact with the two faces of the sensor [Fig. 2(b)].

A constant electric power is supplied to the hot-disk sensor, and the increase in temperature $\overline{\Delta T(t)}$ is directly related to the variation in the sensor resistance [$\Delta R(t)$]. The time-dependent resistance [$R(t)$] is given by[8]

$$R(t) = R_0 \lfloor 1 + \alpha \overline{\Delta T \tau)} \rfloor \qquad (3)$$

where R_0 is the hot-disk resistance in the beginning of the recording (initial resistance), α is the temperature coefficient of resistance of the nickel foil, and $\overline{\Delta T(\tau)}$ is the temperature increase of the sensor expressed in terms of an only variable τ, defined as

$$\tau = (t/\theta)^{1/2}$$

$$\theta = a^2/k \qquad (4)$$

where $t(s)$ is the measurement time from the start of the transient heating; θ is the characteristic time, which depends both on parameters of the sensor and the sample; a (millimeters) is the hot-disk radius; and k (millimeter squared per second) is the thermal diffusivity of the sample.

Assuming an infinite sample and the conductive pattern to be in the Y–Z plane of a coordinate system, the temperature rise at a point (y,z) at time t due to an output of power per unit area Q is given by an expression obtained from the heat-conduction equation solution[8,13]

$$\Delta T(y,z,t) = (8\pi^{3/2}\rho c_P)^{-1} \int_0^t dt[k(t - t')]^{-3/2}$$

$$\times \int_S dy'dz' Q(y',z',t')\exp\{-[(y - y')^2 + (z - z')^2]$$

$$\times [4k(t - t')]^{-1}\} \quad (5)$$

where S is the total area of the conducting pattern that is exposed to a certain temperature increase.

Previous expression can be simplified by taking $k(t - t') = \sigma^2 a^2$:

$$\Delta T(y,z,t) = (4\pi^{3/2}a\lambda)^{-1} \int_0^\tau \frac{d\sigma}{\sigma^2} \int_A dy'dz' Q(y',z',t')$$

$$\times \exp\left\{-\frac{[(y - y')'^2 + (z - z')^2]}{4\sigma^2 a^2}\right\} \quad (6)$$

In the case of disk geometry, consisting of n concentric ring sources, ΔT can be related to $\overline{\Delta T(\tau)}$ through the equation

$$\overline{\Delta T(\tau)} = P_0(\pi^{3/2}a\lambda)^{-1}D(\tau) \qquad (7)$$

where P_0 is the total output power and $D(\tau)$ is a geometric function obtained from eq 5 given by the next equation

$$D(\tau) = [n(n+1)]^{-2} \int_0^\tau \frac{d\sigma}{\sigma^2}$$

$$\times \left(\sum_{l=l}^{n} l \left\{ \sum_{k=1}^{n} k \cdot \exp\left[\frac{(-l^2+k^2)}{2\sigma^2 n^2} \right] L_0\left(\frac{lk}{2\sigma^2 n^2} \right) \right\} \right) \quad (8)$$

in which L_0 is the modified Bessel function.[8,9,13]

Thermal conductivity can be obtained by fitting the experimental data to the straight line given by eq 7, and thermal diffusivity is calculated from eq 4 considering the τ value determined in the previous fit. Finally, the heat capacity was derived from previous values with the relation $k = \lambda/\rho c_P$, where ρ is the sample density. In this article the diffusivity and c_P values are not discussed.

EXPERIMENTAL

A rapid K heat-flow meter from Holometrix was used for the thermal-conductivity measurements under steady heat-flow conditions. The experiments were performed in accordance with the ASTM C518 and ISODIS 8301 methods. The measurements were performed in square samples of 30 cm side and 11 mm thick. A dispersion less than 1% in two consecutive readings was taken as the criterion to ensure that the measurements were made under steady-state conditions. The time lapse between readings was 20 min. Measurements were performed at 24 °C with a temperature difference between plates of 30 K. The time for conducting an experiment was approximately 7 h. Therefore, if three data are needed to characterize one material, the time per material is approximately 21 h.

The performance of any heat-flow-meter apparatus is unique. It depends on factors such as the type and form of heat-flow transducer, its thermal resistance in relation to the specimen resistance, possible contact resistances in the system, heat losses, position of temperature sensors, and the overall calibration factor. To minimize this effect,

a calibration is needed (determination of the constant G). A fibrous board insulation material (NIST SRM 1450c) was used as the standard material. The calibration constant is an apparatus constant. The major advantage of the *in situ* calibration is that the specimen may be considered somewhat self-guarding, and the heat losses can be considered to be eliminated for each particular condition at which calibration is undertaken.

A hot-disk TPS, thermal-constant analyzer, was used for the measurement of thermal conductivity of LDPE foams. Experiments were carried out at room temperature (24 ± 2 °C). The measurements were performed in two square samples of 36 mm side and 11 mm thick. The samples were much smaller than those used for the heat-flow meter. The thickness of the samples should preferably not be less than the diameter of the hot-disk sensor, and this must always be much larger than the porosity or the cellular structure of the sample if the material is not dense or homogeneous (ca. an order of magnitude higher than the cell size). A disk-shaped TPS sensor with a diameter of 12.806 mm provided with 16 rings was used in all measurements. The TPS element was made of 10-μm-thick nickel foil with electric insulation on each side of 30-μm Kapton.

The calculations of the thermal properties were performed according to the equations outlined in theory of TPS. The first 50 points of each recording (total number of points in each measurement was 200) were not used in each calculation. This procedure reduced the effect of the contact resistance between the sensor and sample.

MATERIALS

The product code, base polymer, measured density (ρ_f), mean cell diameter (ϕ), thickness L, and apparent color of the industrial materials under study are summarized in Table 1.

Foams produced from 100% LDPE and blends of LDPE and high-density polyethylene (HDPE) were examined. The density ranged between 15 and 83 kg/m³ and the cell diameter was between 313 and 1000 μm. The thickness of the foams was approximately 11 mm. Moreover, foams of different colors were characterized; in particular, black foams with different black carbon contents were used in this analysis. The solid sheets that were used to manufacture some of the foams were also considered in this investigation.

Table 1. Main Characteristics of the Foams under Study

Foams	Chemical Composition	Apparent color	ρ_f (kg/m^3)	ϕ (μm)	L (cm)	θ_{carbon} (%)
LD15W	100% LDPE	White	16.7	313	1.12	0
LD24W		White	24.6	312	1.02	
LD29W		White	30.7	528	1.11	
LD33 (1)W		White	32.0	424	1.10	
LD50CNB		Black	52.3	910	1.04	12
LD60 G		Green	58.5	773	1.02	0
LD70B		Black	69.5	528	1.10	2
LD24(FC)		Black	23.6	315	1.02	
LD24(LC)		Black	23.7	956	1.02	2
LD33 (2)W		White	31.4	377	1.10	0
LD33B		Black	32.5	337	1.10	2
LD sheet W		White	910	—	0.93	0
LD sheet B		Black	910	—	1.03	2
HL79(1)W	50% LDPE	White	81.0	1006	0.99	0
HL79(2)W	+ 50% HDPE	White	83.0	1076	1.70	
HL sheet W		White	926	—	1.31	

ρ_f is the foam density, ϕ is the cell size, L(cm) is the thickness, and θ_{carbon} is the additional black carbon content in the polymeric matrix.

These foamed samples were crosslinked, closed-cell polyolefin foams manufactured by a high-pressure nitrogen gas-solution process and were provided by Z-Foams Plc. (Croydon, United Kingdom). In this process, a polyolefin is compounded with a peroxide curing agent and extruded as a thick sheet, which is passed through a hot oven to effect crosslinking (gel content was ca. 40%). Slabs are cut from the extruded sheet and placed in an autoclave where they are subjected to high pressure (several hundred bars) nitrogen gas at temperatures above the polymer softening point. Under these conditions, the nitrogen dissolves in the polymer. At the end of the solution stage and after cooling, the pressure is reduced to zero gauge. The slabs, now containing nitrogen gas for expansion, are then placed under low pressure in a second autoclave and again heated above the polymer melting point. Release of the pressure then results in full expansion. By altering the saturation gas pressure, the amount of gas dissolved in the polymer and thus, the final foam density, is varied. The cell size can also be controlled by changing some industrial process parameters.

RESULTS

The thermal conductivity for all the foams under study as a function of the foam density, obtained by both methods, is shown in Table 2 and Figure 3.

Table 2 also includes the experimental power, measuring times, and time between experiments used for each material. The whole time for an experiment in which five recordings were measured was approximately 2.5 h.

The first important result is that the trends observed by both methods as a function of foam density are very similar (Fig. 3). Therefore, if the interest is focused on the analysis of the relationships between structure and thermal properties, it seems clear that very similar conclusions would result from the data obtained by both techniques. The qualitative influence of structural parameters, such as cell diameter, density, black carbon content, kind of base polymer, and so forth, that it is possible to deduce from the data collected by the TPS method were similar to those we analyzed in previous investigations[1–3] in which the measurements were performed under steady-state conditions.

In that research, the effect of several structural characteristics that influences the thermal conductivity was studied. It was established that cell size and black carbon content were the two most important factors that affect the thermal conductivity through their effect on the radiative heat-flow contribution. In particular, it was proven that increasing the cell size results in higher conductivities and adding a low proportion of black carbon content reduces the conductivity.

Table 2.

Foams	λ_{RK} (W/m K) Rapid K	λ_{HD} (W/m K) Hot Disk	$\Delta\lambda$ (%)	t_m (s)	σ
LD15W	0.0374	0.0439	14.8	30	0.0002
LD24W	0.0372	0.0468	20.5	30	0.0002
LD29W	0.0441	0.0516	14.6	30	0.0003
LD33 (1)W	0.0407	0.0503	19.1	40	0.0001
LD50CNB	0.0413	0.0530	22.0	40	0.0005
LD60G	0.0475	0.0599	20.7	40	0.0002
LD70B	0.0456	0.0581	21.5	50	0.0004
LD24(FC)	0.0364	0.0460	20.8	30	0.0003
LD24(LC)	0.0413	0.0497	16.9	30	0.0004
LD33 (2)W	0.0401	0.0519	22.6	40	0.0003
LD33B	0.0364	0.0484	24.7	40	0.0002
LD Sheet W	0.2140	0.3729	42.6	110	0.0015
LD Sheet B	0.2140	0.3773	43.3	110	0.0033
HL79(1)W	0.0565	0.0705	19.9	40	0.0005
HL79(2)W	0.0592	0.0767	22.8	40	0.0003
HL Sheet W	0.2246	0.4727	52.5	90	0.0048

λ is the thermal conductivity for all the foams under study. $\Delta\lambda$ (%) is the difference between the values of λ obtained with hot disk and rapid K equipments, $\Delta\lambda = 100 \cdot (1 - \lambda_{RK}/\lambda_{HD})$. t_m (s) is the measuring time for each sample. Five measurements were obtained, and the time between experiments was 30 min. The deviations of these five measurements are also given in the table (σ). The applied power was 0.02 W.

Similar trends can be inferred from the experimental data measured by the TPS method. The thermal conductivity of the low-density polyethylene foams (LD) measured by the TPS method as a function of the foam density is displayed in Figure 4. The black foams have a slightly lower conductivity than the white foams of similar density. Moreover, for this kind of material, reducing the density involves an improvement of the thermal-insulation capability.
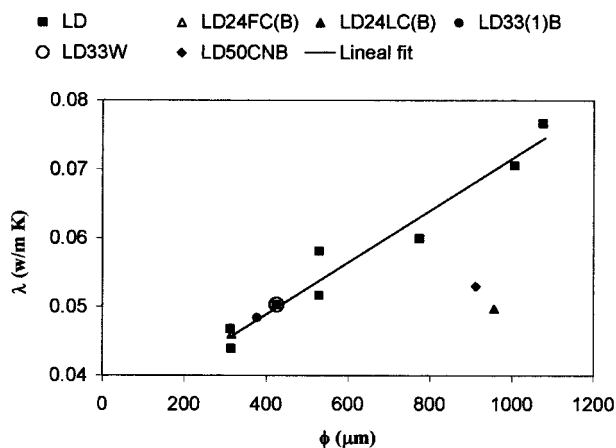
Figure 5 depicts the thermal conductivity of the foams measured by the TPS method as a function of the cell diameter. A linear increase of this thermal property was observed for the LD white foams. It was also possible to observe that black foams present lower values of the conductivity. As explained previously, these trends were due to the thermal-radiation heat flow. The out-
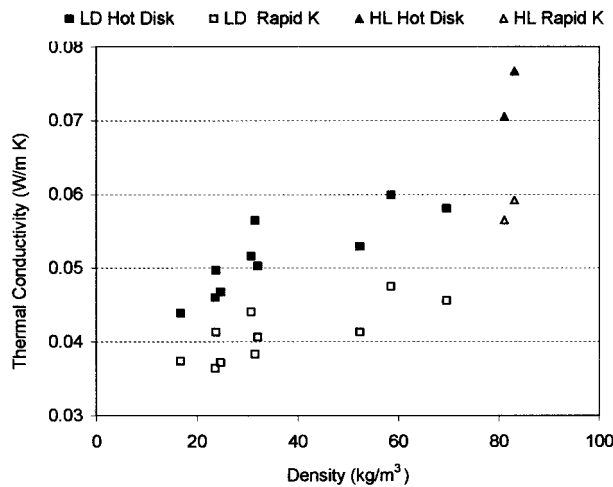
Figure 3. Experimental thermal conductivity, obtained by both the hot-disk and rapid K methods, as a function of the density for all foams under study.
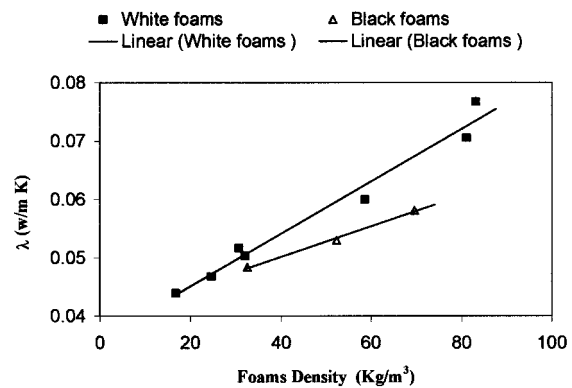
Figure 4. Thermal conductivity of the white and black LD foams measured by the TPS method as a function of foam density; linear fits for the white and black foams are included.

Figure 5. Thermal conductivity measured by the TPS method as a function of the cell diameter; the linear fit corresponds to the LD white foams.

come of reducing the cell size is to increase the number of cell walls the radiation has to pass across. As the heat flow by radiation is absorbed or scattered in the cell walls, reducing the cell diameter results in a lower conductivity. Moreover, adding black carbon to the cell walls increases dramatically the extinction coefficient of the polymer; therefore, the radiation is more absorbed in black foams decreasing the heat flow.

For instance, the value of thermal conductivity obtained for the LD24(LC) foam (see Table 2), the material with a large cell size (956 μm), was clearly higher than the value of thermal conductivity for the LD24(FC) sample. This last material had cells of smaller diameter (315 μm). A similar result was found when the influence of the black carbon content was analyzed. In Table 2 the values of thermal conductivity obtained in a white LD33 foam and in a black LD33 foam are collected. This last sample presented a low content (2 wt %) of black carbon in their composition and had a thermal conductivity 6.7% smaller than the LD33 white foam.

HL foams have higher thermal conductivities than LD ones. The main reason for this difference is the chemical composition of the polymer matrix of HL materials. These foams are produced from a blend of LDPE and HDPE. The HDPE phase has a higher thermal conductivity than the LDPE phase, which increases the thermal conductivity of the blend.

As previously mentioned, the preceding trends can be deduced from both the data measured by TPS and the standard technique. From a scientific point of view, the effect of cell size and black carbon content on the conductivity are due to the contribution of the radiation term on the whole conductivity. For these materials, it can be stated that TPS technique detects the radiation heat flow.

However, the thermal-conductivity values obtained with the TPS method are always higher than the values obtained with the rapid K equipment. The differences were for almost all the foams under study approximately 20% (Table 2). Also, these differences were clearly higher (ca. 40–50%) for the solid sheets.

To analyze the previous results, it is convenient to introduce several notions on the precision of these two techniques.

Several interlaboratory studies were carried out in an attempt to determine the precision of the steady-state technique. Various types and sizes of heat-flow meter apparatus were involved in the American Society for Testing and Materials/National Mineral Wool Association round robin on fibrous blankets.[14] The results indicated that the overall precision was +2.5%, a similar order of the guarded hot plate. This was confirmed by a study[15] on random specimens of expanded polystyrene materials. Although differences of 1–2% were found in the absolute values for five different specimens, all laboratories ranked the specimens in the same order illustrating the value of the method for comparative studies.

As far as we know, there are no detailed studies on the precision of equipments based on the TPS method. However, the precision on the line-source method, a procedure not equal but based on similar principles, was evaluated at 300 K on several materials.[16] These included cellular materials including a well-aged polystyrene board. The results of the investigations were not encouraging. They indicated standard deviations of some 20% for the different foams, and for insulations the differences in results exceeded 10% from the accepted values obtained with steady-state methods.

In our opinion, several reasons could explain the differences between the λ values measured by both techniques.

A first contribution has its origin on the temperature gap between the heat-flow meter surfaces and those of the sample. Figure 6 illustrates this concept. The temperature difference contributed to the reduction of the heat transferred by conduction and radiation.
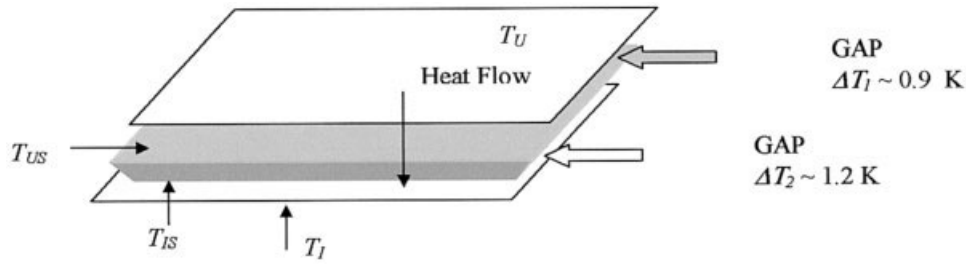
Figure 6. Diagram showing the temperature difference between the plates of the rapid K equipment and the temperature of the sample surface (contact resistance).

It is possible to estimate a corrected thermal conductivity from the gap temperature and the measured conductivity. With the Fourier law, the following relationships can be deduced:

$$\lambda_{\text{corrected}} = \frac{\lambda_{\text{measured}}(T_{\text{U}} - T_{\text{I}})}{(T_{\text{U}} - T_{\text{I}}) - (\Delta T)} \tag{9}$$

$$\Delta T = \Delta T_1 + \Delta T_2; \quad \Delta T_1 = T_{\text{U}} - T_{\text{US}};$$

$$\text{and } \Delta T_2 = T_{\text{IS}} - T_{\text{I}}$$

where $\lambda_{\text{measured}}$ is the thermal conductivity determined by applying the procedure described in the Experimental section; $\lambda_{\text{corrected}}$ is the value of the thermal conductivity corrected by the influence of the temperature difference previously mentioned; ΔT_1 is the temperature difference between sample and heater surface in the hot plate; ΔT_2 is the temperature difference between the sample and heater surface in the cold plate; T_{U} and T_{I} are the temperatures of the hot and cold plates, respectively; and T_{US} and T_{IS} are the temperatures of the sample surface in the hot and cold plates, respectively.

The temperature differences ΔT_1 and ΔT_2 were measured for the sample LD33(1). The results for these measurements are collected in Table 3. The temperature gap increased approximately 7% the value of the thermal conductivity.

The temperature gap was much smaller in the hot-disk equipment. As mentioned in the Experimental, in this method this gap was reduced by cutting the first points in each transient recording.

The temperature gap could also be one of the reasons for the large differences between the conductivity of the solid sheets measured by both methods. The solid sheets as compared with the foams were very stiff. As a consequence, a small deviation from a completely flat surface resulted in a considerable increase of the temperature gap existing in the rapid K measurements.

A second possible cause of difference between the values obtained for the thermal conductivity with the heat-flow meter and the TPS method is the thermal state of the sample in which the equipment measured the values. Rapid K measures a stationary heat flow, whereas TPS measures the transient regimen. This dissimilarity could result in a systematic difference between the values of the thermal conductivity measured by both methods.

Other possible sources of discrepancy are the slight differences in the average temperature of the samples; the size of the samples is much smaller in the transient method, which in case of inhomogeneous samples could also influence the difference between the methods and so forth. A deep analysis of these differences is outside the

Table 3.

Foams	L (cm)	T_{U} (°C)	T_{US} (°C)	Gap between T_{U} and T_{US} (K)	T_{I} (°C)	T_{IS} (°C)	Gap between T_{I} and T_{IS} (K)	$100*$ $(1 - \lambda_{\text{corrected}}/\lambda_{\text{measured}})$
LD33W	1.10	39	38.1	0.9	9	10.2	1.2	7

L(cm) is the sample thickness, T_{U} is the temperature of the hot plate, T_{I} is the temperature of the cool plate, T_{US} is the temperature of the face in contact with the hot plate, and T_{IS} is the temperature of the face in contact with the cool plate.

scope of this work. However, these differences should be analyzed systematically in the future.

CONCLUSIONS

The thermal conductivity of a collection of closed-cell polyethylene foams has been measured by both a steady-state method and a TPS technique. Both methods gave similar trends for this property, an important result, which validated the TPS method as a tool for analyzing the structure–property relationships for these two phase materials. However, it has been also shown that there is a systematic difference between the numerical values given by the two methods. Several possible sources for these differences have been proposed.

The TPS technique seems to be a powerful technique for comparative studies of the thermal properties of insulating materials because of several factors such as fast measurements, possibility of using small samples, potential analysis for heterogeneity and anisotropy, and so forth. However, considerable thought and effort must be expended to analyze the absolute values given by the method.

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