Test Method

Intercomparison of thermal conductivity and thermal diffusivity methods for plastics

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ABSTRACT

An intercomparison of measurements of the thermal conductivity and thermal diffusivity of two poly(methyl methacrylates) is reported. A wide variety of methods were used: temperature wave analysis, laser flash, transient plane source (Hot Disk®), transient line-source probe, and heat flux meter methods. Very good agreement of thermal conductivity results and, separately, of thermal diffusivity results was obtained. Similarly, good agreement between thermal conductivity and thermal diffusivity results, when converted using specific heat capacity and density values, was also obtained. Typically, the values were within a range of approximately ±10%. Considering the significant differences between the methods and the requirements on specimen dimensions, the level of agreement between results was considered to be good.

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1. Introduction

Reliable thermal properties of polymers are essential for accurate design, whether for process simulation or product performance. The well-established guarded hot plate and heat flux meter techniques for thermal conductivity [1–3] are suited to large, thick specimens. For these methods, testing times can be long due to the need for thermal equilibrium, which can be of the order of 24 h, and temperature gradients across the specimen can be large. Furthermore, these steady state methods are not suited to testing molten polymers that, in addition to other practical factors related to the molten state, are likely to degrade over the course of the extended test duration that is required to achieve thermal equilibrium. In comparison, transient or non-steady state techniques for thermal conductivity and thermal diffusivity such as the transient plane source [4,5], temperature wave analysis [6–9], laser flash [10,11] and line-source probe [12–14] methods[1] are appealing in that the test time is comparatively short, small specimens can be measured, and formed products, e.g. films, sheets and mouldings, can be tested. Indeed, the choice of technique will be influenced by the available specimen geometry. Furthermore, transient techniques can provide other benefits, e.g. the transient plane source is reported to be able to determine the thermal conductivity, thermal diffusivity and the anisotropy of these properties [5].

Several transient techniques are currently being drafted as ISO standards [5,9,11]. However, the comparability of

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[1] For convenience the term transient is used herein to denote techniques that are non-steady state.

data from these methods for plastics is largely unknown. Nunes dos Santos [15] compared results obtained using laser flash and hot wire techniques for a range of polymers. For PMMA there was up to a 20% difference in thermal diffusivity values and up to a 10% difference in thermal conductivity values between the two methods. However, significant differences of up to a factor of two in thermal conductivity were also obtained. The laser flash yielded lower values than the hot wire for LDPE and higher values for HIPS, illustrating the difficulties in thermal properties measurement for polymers. Sombatsompop and Wood [16] concluded that the thermal conductivity of polymers was influenced by the density, molecular structure, level of crystallinity and thermal history of the polymers, thus complicating the comparison of methods where, for example, differences in specimen preparation may influence thermal properties. Furthermore, the repeatability of measurements was found to be material and test condition specific. Dawson et al. [13] and Urquhart et al. [17] reported that the repeatability of line-source probe measurements of HDPE over the melt and solid phases was up to ±16%, but for a liquid polymer (PDMS) it was significantly lower at ±1.4% (95% confidence levels) – a significant difference for the same technique. For the transient techniques used in this intercomparison, estimated uncertainties up to 10% and repeatabilities up to 6% were reported (Table 1).

To address the issue of comparability of results, an intercomparison of the measurement of thermal conductivity and thermal diffusivity of polymers was initiated to obtain precision data over a range of temperatures. Furthermore, to facilitate the comparison of the various techniques the specific heat capacity and density values of the materials were measured to enable calculation of thermal conductivity values from thermal diffusivity values, and vice-versa.

The objectives of this intercomparison were to assess the repeatability and reproducibility of the various transient thermal conductivity and thermal diffusivity techniques, some of which are covered by ISO 22007 Parts 1–4 [3,5,9,11] and of other techniques that may also be incorporated into this series of standards in the future. It is envisaged that the findings of this intercomparison will be incorporated into ISO 22007 as information on precision or as part of the precision statements, and will contribute to the development of all parts of this standard.

2. Materials

The intercomparison was based on the testing of two poly(methyl methacrylate) (PMMA) materials, one in the form of a cast sheet and the other in the form of an extruded sheet for which samples in pellet form were also available. These are referred to herein as "cast" and "extruded" respectively.

The cast PMMA sheet was of Sumipex 000, Lot. 6621114, 2 mm in thickness and supplied by Sumitomo Chemical. The glass transition range, measured by differential scanning calorimetry (DSC) with heating at 10 °C/min, occurred between approximately 100 °C and 130 °C. The polymer degraded above 220 °C. As the cast sheet was directly polymerized in-situ no equivalent pellet grade having the same molecular weight distribution was available.

The extruded PMMA was an extrusion grade with melt mass flow rate of 1.6 g/10 min (230 °C/3.8 kg). The glass transition range, measured by DSC in heating at 10 °C/min, occurred between approximately 90 °C and 130 °C. For this material, typical die process temperatures are in the range 220 °C to 240 °C, with decomposition occurring above 280 °C, but recommendations to participants were to keep the material to below 240 °C.

For the intercomparison a testing procedure was provided to participants and, where relevant, testing was to be carried out following the draft ISO 22007 series of standards [3,5,9,11].

3. Experimental methods

3.1. General

The intercomparison involved the following methods for thermal conductivity and thermal diffusivity:

Thermal conductivity and thermal diffusivity
  Transient plane-source method – Hot Disk® – ISO/DIS 22007-2
Thermal diffusivity
  Temperature wave analysis – ISO/DIS 22007-3
  Laser flash – ISO/DIS 22007-4
Thermal conductivity
  Transient line-source method – ASTM D5930
  Heat flow meter – ASTM E1530

Table 1
Details of the test methods and specimen conditions applied to this study.

Lab. ref.	Method	Thermal conductivity/ diffusivity	Nominal specimen thickness (mm)	Specimen size (mm; φ, diameter)	Pre-treatment	Uncertainty estimate (95% confidence level)
L1	Hot Disk®	λ, α, (Cp)	2, 3	φ 5; φ 10		λ, 2–5%; α, 5–10% (repeatability 1–2%) [5]
L2	Temperature wave analysis	α	0.01	3×5		5%[a] (repeatability < 1%) [9]
L3	Laser flash (cast PMMA only)	α	2	Disk	Silver paint (30 μm)	3–5% (repeatability < 1%) [11]
L4	Laser flash	α	1.14, cast; 1.49, extruded	Disk	Sputtered graphite	3–5% (repeatability < 1%) [11]
L4	Transient line-source probe	λ	Moulded in-situ	Formed in barrel	Moulded in-situ	(Repeatability 3–6%) [14]
L4	Heat flow meter	λ	2, 3			5% [18]
L5	Heat flow meter	λ	2, 3	φ 80		(Repeatability 3%) [19]

[a] Values given for sputtered sensors. For non-sputtered sensors uncertainties were estimated to be in the range 5–10%.

For the temperature wave analysis, laser flash and Hot Disk measurements, the draft standards for thermal conductivity and thermal diffusivity, ISO/DIS 22007 parts 2–4 were used.

For the specific heat capacity (Cp) data, differential scanning calorimetry measurements were performed. Heat capacity values were also determined using the Hot Disk method that separately determines both thermal conductivity and thermal diffusivity, from which the volume heat capacity and, given the density, the specific heat capacity were calculated. For density values, both the Archimedes principle and mass and length dimensional measurements were performed. For the temperature dependence of density, linear thermal expansion measurements were performed, the results of which were used to calculate the bulk temperature dependence.

The thermal conductivity and thermal diffusivity methods used in this intercomparison are described below.

3.2. Transient plane-source – Hot Disk®

The transient plane-source or Hot Disk method [4,5] uses a thin resistive element, in the form of a bifilar spiral, which acts as both a heat source and a temperature sensor. The element is sandwiched between two planar specimens of the same material. A known amount of electrical power is supplied to the element and its change in resistance as a function of time, due to its increase in temperature, is recorded. From this, both the thermal conductivity and thermal diffusivity can be determined, providing criteria concerning the penetration depth of the temperature transient into the specimen are met. The method can be used to determine the anisotropy of thermal conductivity and thermal diffusivity. Further details of the test method and data analyses can be found in ISO/DIS 22007-2 [5].

3.3. Temperature wave analysis

The temperature wave method [6–9] is used for measuring the thermal diffusivity in the through-thickness direction of films or thin sections of specimens. It can be used to measure the thermal properties of polymers in either their solid and molten states. The specimens are typically 1 μm to 1 mm in thickness and 10 mm by 10 mm in area. The principle of the technique is to measure the phase shift of an oscillating temperature wave travelling through the specimen by using a two-phase lock-in amplifier. The temperature wave is generated and sensed by electrical resistors, typically sputter coated onto opposing sides of the specimen, with one resistance element acting as the heater and the opposing element as the sensor. The thermal diffusivity is calculated from the temperature wave's phase shift across the specimen as a function of the angular frequency of the temperature wave and the specimen thickness.

A specific issue related to this intercomparison is that the specimens had to be reduced in thickness to enable the temperature wave analysis method to be used. This obviously has implications for any potential effect of microstructure dependence of thermal diffusivity, if there is a through-thickness variation in microstructure, as only a small part of the thickness of the original specimens was tested. Further details of the test method and data analysis can be found in ISO/DIS 22007-3 [9].

3.4. Laser flash

The principle of the laser flash method is to irradiate the front side of a specimen using a short energy pulse, typically provided by a laser, and record the subsequent temperature rise on the rear side of the specimen using an infra-red detector [10,11]. From the shape of the temperature–time curve of the rear side and the specimen thickness, the thermal diffusivity of the specimen can be determined. The specimens are typically 10 mm to 20 mm in diameter and 1 mm to 3 mm thick. Due to the transparent nature of the PMMA specimens, they had to be coated prior to testing to ensure absorption and emission at the front and rear faces respectively: silver paint and graphite were used by the participants in this intercomparison. Further details of the test method and data analysis can be found in ISO/DIS 22007-4 [11].

3.5. Transient line-source

The transient line-source probe technique [12–14], also referred to as the needle probe method, is a development of the hot wire method but is suited for testing plastics in both their molten and solid states. A linear heating element and thermocouple are sheathed in a casing in the form of a needle, typically 50 mm to 100 mm in length and 1.5 mm to 2 mm in diameter. The heater element lies along the length of the needle with a thermocouple at the mid-length position. The probe is immersed in the specimen and held until temperature equilibrium is achieved at the required temperature. Then, a known amount of energy is dissipated by the line-source heater, achieved by applying a voltage across the resistance heater for a period of time, resulting in a heat wave propagating radially into and through the specimen. The thermal conductivity is determined from the temperature change of the probe with time: a test specimen of higher conductivity will result in a more rapid transfer of heat from the probe, and thus a lower rate of temperature rise of the probe with time, compared with a lower conductivity specimen. The finite probe dimensions, end effects and other non-linearities require the probe to be calibrated against a reference material of known thermal conductivity. For the results presented here, PDMS was used as the calibrant [3]. A specific issue related to this intercomparison was that the test specimens had to be formed in the cylindrical heated barrel of the instrument and thus any previous microstructure associated with the plate specimen would have been lost. Details of the test method and data analysis can be found in ASTM D5930 [14].

3.6. Heat flow meter

The principle of the heat flow meter method, a variant of the guarded hot plate technique, is that a heat flux transducer is used to measure the heat flow through the specimen mounted between two plates, a 'hot' and a 'cold'
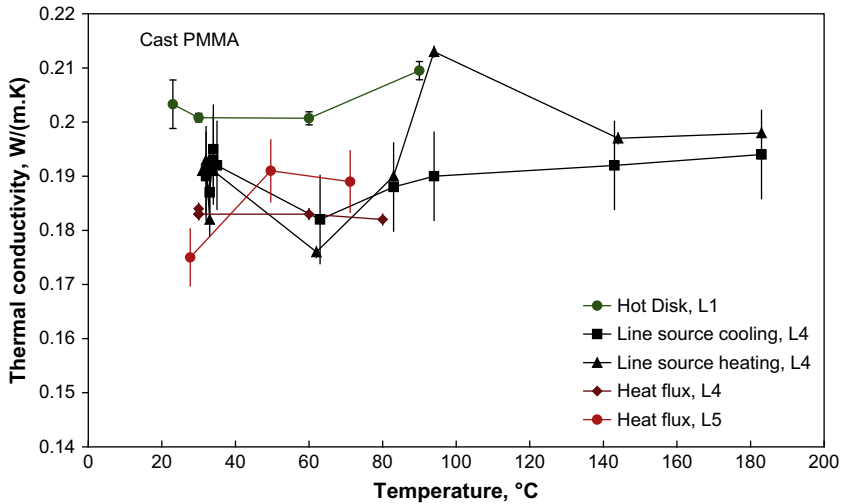
Fig. 1. Thermal conductivity of the cast PMMA measured by transient line-source probe, Hot Disk and heat flux meter (by two laboratories) methods.

plate. The method is a quasi-steady state method and thus the instrument and specimen must be allowed to reach thermal equilibrium before measurements are made. From measurements of the heat flux, temperature difference across the specimen and specimen thickness the thermal conductivity can be determined. The method is a comparative method and thus the instrument must be calibrated using a specimen of known thermal conductivity. Principles of the test method and analysis can be found in ASTM E1530 [2].

4. Results

The directly measured properties of thermal conductivity and thermal diffusivity are presented in Figs. 1 and 2 for the cast PMMA, and in Figs. 3 and 4 for the extruded PMMA. Scatter bars are presented indicating the level of repeatability of results, at the 95% confidence level, the values being obtained from repeat tests. For the cast material, the variation in thermal conductivity results were, for any given temperature, within a range of approximately $\pm 7\%$, with the maximum variation occurring at $\approx 30\,^{\circ}C$ (Fig. 1). Only the transient line-source probe data were obtained above the glass transition temperature (Tg) and showed a relatively constant value with increasing temperature. Below Tg the data obtained by the Hot Disk method were slightly higher, by approximately 6%, than data obtained by the transient line-source probe and both heat flow meter methods – see later discussion on the effect of anisotropy. The effect of temperature on thermal conductivity was small, on the basis of these results, with little clear variation over the test temperature range: any systematic variation being masked by the repeatability and reproducibility of the methods. For thermal diffusivity (Fig. 2), the temperature wave analysis, laser flash and Hot Disk methods yielded results that were, for any given
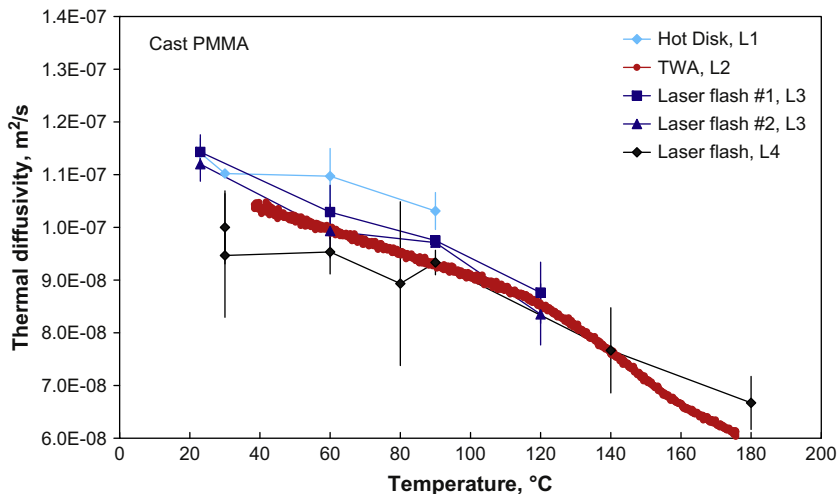

Fig. 2. Thermal diffusivity of the cast PMMA measured by Hot Disk, laser flash (by two laboratories), and temperature wave analysis methods.
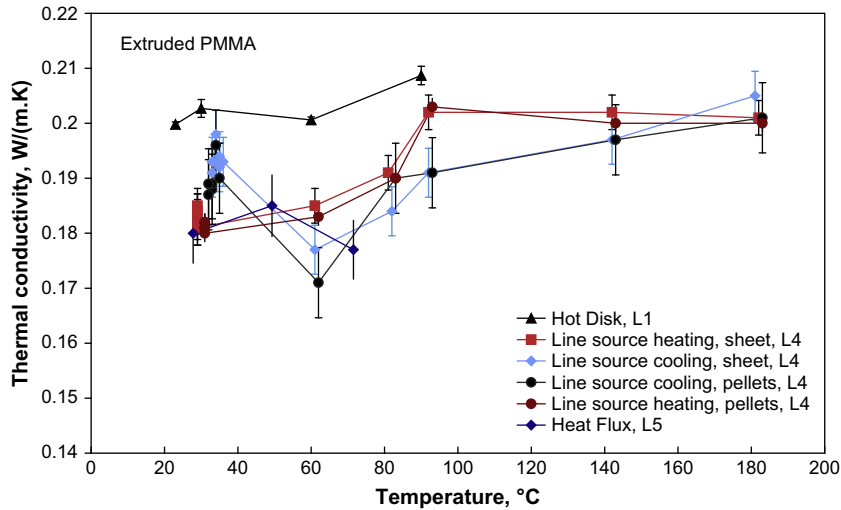
Fig. 3. Thermal conductivity of extruded PMMA measured by transient line-source probe, Hot Disk and heat flux meter methods.

temperature, within a range of approximately ±9%, with the maximum variation occurring at ≈30 °C. All thermal diffusivity data directly measured by the transient methods exhibited a clear decrease in value with increasing temperature.

Results for the extruded PMMA material exhibited similar magnitudes and trends to the results for the cast PMMA (Figs. 3 and 4). Thermal conductivity results varied by up to ±7%, and thermal diffusivity results by up to ±4%. In most cases, the differences observed in the measured thermal conductivity or thermal diffusivity values were comparable to the 95% confidence limits for the repeatability of the methods (Table 1).

Thermal conductivity λ can be calculated from thermal diffusivity α, and vice-versa, given the specific heat capacity C_p and density ρ values of the specimen at the same temperature using Eq. (1).

$$\lambda = \rho C_p \alpha \qquad (1)$$

In order to make this conversion, and thus comparison of directly measured thermal conductivity results with thermal diffusivity results, measurements were also made of specific heat capacity and density as part of the inter-comparison. Differential scanning calorimetry measurements of specific heat capacity are presented for the cast and extruded PMMA materials in Figs. 5 and 7 respectively. Mean values with scatter bars representing the average two standard deviation ranges in values (i.e. ±7% for the cast PMMA and ±6% for the extruded PMMA) are also presented at select temperatures. Both materials exhibited similar behaviour, with increasing C_p values with increasing temperature, except that the cast material exhibited a peak on passing through the T_g (at approximately 120 °C) for the first test run on a specimen (L5 results). This peak was
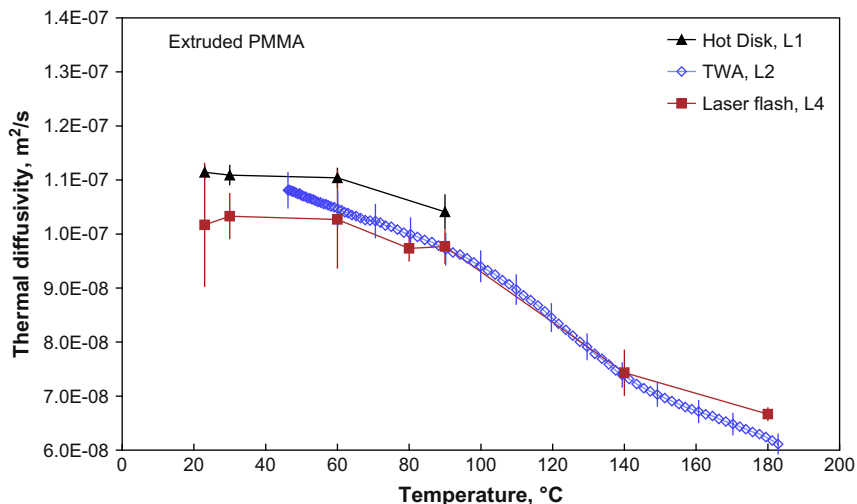

Fig. 4. Thermal diffusivity of extruded PMMA measured by Hot Disk, laser flash and temperature wave analysis methods.
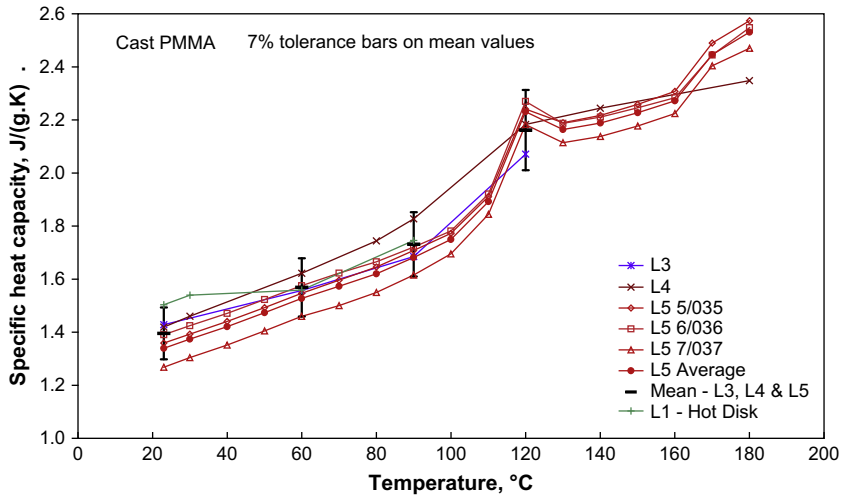
Fig. 5. Specific heat capacity, Cp, of the cast PMMA measured by DSC.

minimized or absent from repeat tests on the same specimen. This phenomenon, considered to be caused by residual stresses, potentially complicates the comparison of DSC results where different procedures were used for testing. Also, it obviously complicates the conversion from thermal conductivity to thermal diffusivity, or vice-versa, where again the thermal histories of the specimens are likely to be different for the different thermal properties test methods which differ quite markedly.

The specific heat capacity values determined at 23 °C, 60 °C, 90 °C and 120 °C for both the cast and extruded PMMA materials had 95% confidence levels (two standard deviations) of up to approximately ±10%, but more usually of the order of ±7%. The uncertainty in specific heat capacity determination was estimated by one laboratory to be of the order of ±4%. However, specimen preparation and possibly moisture content were both considered to have affected the results for the cast PMMA, but not for the extruded PMMA.

The densities of the materials were measured by three laboratories at or near 23 °C and determined at elevated temperatures by one laboratory using linear thermal expansion data. The results at 23 °C gave a repeatability of ±0.3% and a reproducibility of ±1.0% (95% confidence level) (Fig. 6). Values for the densities of the cast and extruded PMMA materials at 23 °C differed by less than 0.1%.

These specific heat capacity and density data were used to calculate thermal conductivity values from thermal diffusivity values, and vice-versa, using temperature dependant values throughout. The temperature dependence of density for the extruded PMMA was assumed to be the same as the temperature dependence of density of the extruded PMMA. Thus directly measured thermal conductivity results and calculated thermal conductivity values, based on directly measured thermal diffusivity, density and specific heat capacity values, are presented in Fig. 8 for the cast PMMA and Fig. 10 for the extruded PMMA. Similar plots of measured and calculated thermal diffusivity results
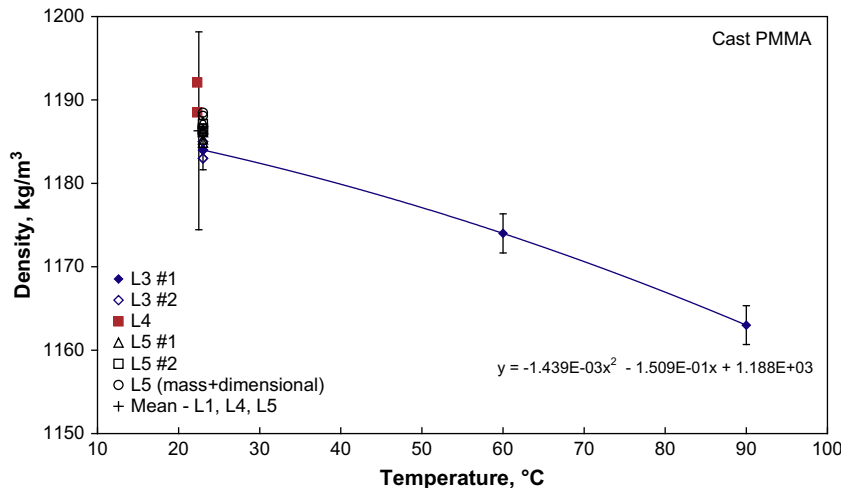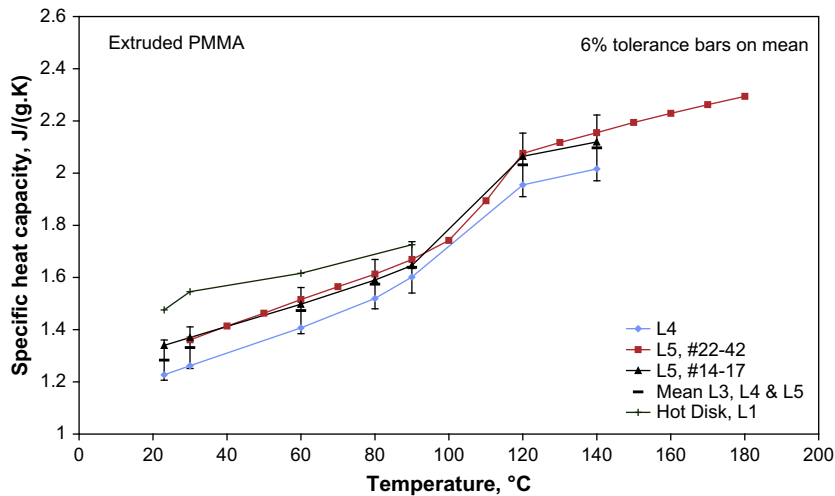

Fig. 6. Density of the cast PMMA.

Fig. 7. Specific heat capacity, Cp, of extruded PMMA measured by DSC.

are presented in Figs. 9 and 11 for the cast and extruded PMMAs respectively. The calculation of thermal conductivity from thermal diffusivity, and vice-versa, was only performed for temperatures up to 90 °C as this was the upper limit for which density values were determined. Extrapolation of data into and beyond the glass transition region was considered too unreliable.

For the cast PMMA material, there was generally very good agreement of the directly measured and calculated results, both for thermal conductivity and thermal diffusivity (Figs. 8 and 9 respectively), with scatter not being significantly greater than that exhibited for directly measured values only. This was also the case for the extruded PMMA results, Figs. 10 and 11.

The uncertainties in C_p and density, estimated at approximately $\pm10\%$ and $\pm1\%$ respectively (95% confidence levels), can be combined to give an overall uncertainty in

the calculation of $\pm10\%$: the contribution due to the uncertainty in the density is negligible. These uncertainties will obviously contribute to the uncertainties in the calculated values, although there does not appear to be a systematic and consistent shift in calculated data from directly measured values, suggesting that the conversion factors involving density and specific heat were accurate.

5. Discussion and conclusions

The results of this intercomparison indicate a good level of agreement considering the significant differences in the various methods. Some of the key differences are summarised in Table 1. Although all participants were supplied with nominally identical sheet samples, the actual thickness of the specimens used in testing had to be tuned to the test method to enable measurements to be made or to
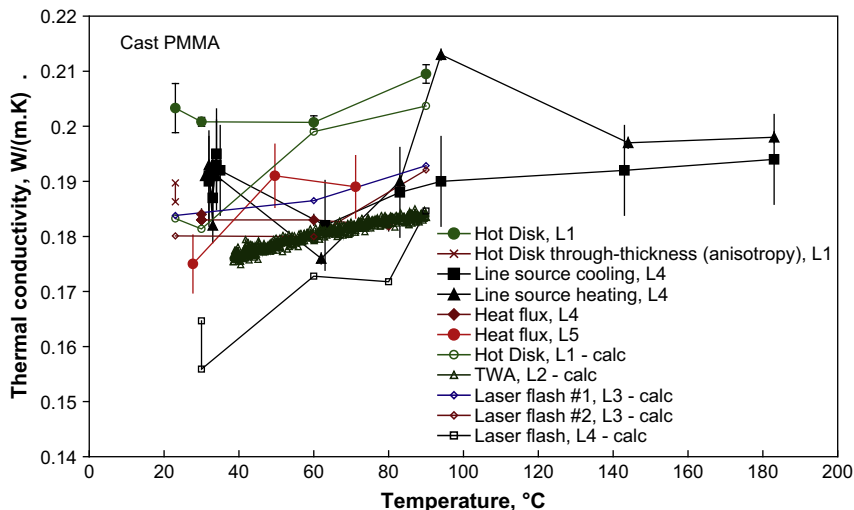

Fig. 8. Thermal conductivity of the cast PMMA measured directly by heat flux meter (by two laboratories), transient line-source probe and Hot Disk methods, and calculated from thermal diffusivity measured by laser flash (by two laboratories), temperature wave analysis and Hot Disk methods.
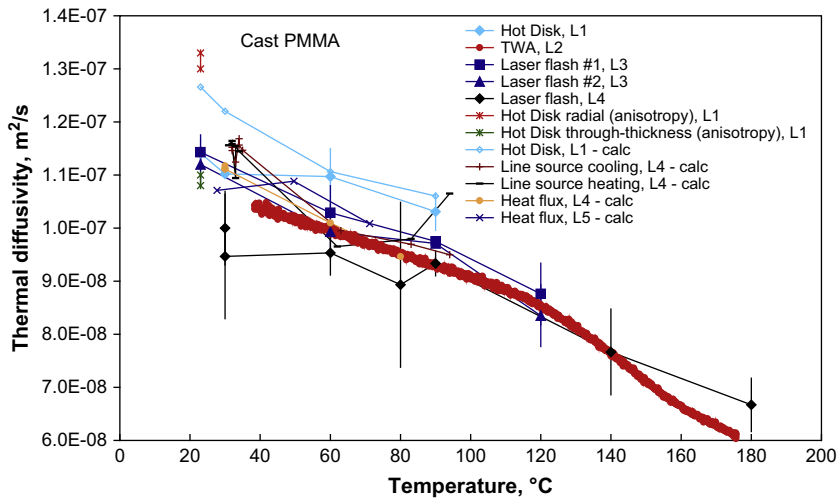
Fig. 9. Thermal diffusivity of the cast PMMA measured directly by Hot Disk, laser flash (by two laboratories) and temperature wave analysis methods, and calculated from thermal conductivity measured by Hot Disk, heat flux meter (by two laboratories) and transient line-source probe methods.

ensure that assumptions made in the analyses were valid. For example, the temperature wave analysis method is suited to thin films or sheets of thickness of approximately 100 μm and thus the specimens had to be cut to this thickness prior to testing. For the laser flash method, the 3 mm thickness of the extruded PMMA sheets was found to be too large for reliable measurements and so these specimens were cut thinner. Similarly, for the Hot Disk method it is necessary to comply with the thickness requirements for testing, related to the depth of penetration of the thermal transient into the specimen. For the thinner 2 mm specimens this was achieved by stacking two specimens together to achieve a sufficient thickness for reliable measurement.

In addition to specimen thickness issues, further differences in the methods exist. For laser flash of PMMA, as the specimen is transparent its surfaces had to be treated

with an opaque material prior to testing, otherwise measurements could not be made. The effect of contact resistances in contact methods (transient line-source probe, heat flow meter, temperature wave analysis, Hot Disk) potentially differed: in some methods the contact resistance can be corrected for. Furthermore, for the heat flow meter method an average test temperature has to be assigned to the test due the large temperature difference across the specimen, and thus an average thermal conductivity value is determined and assigned to that average temperature. In transient methods the temperature transient is typically less than 10 °C and errors due to non-linear dependence of thermal properties on temperature are potentially reduced.

There is significant evidence that the microstructure of polymers can affect their thermal properties [15,16,20], in particular for semi-crystalline materials. For the transient
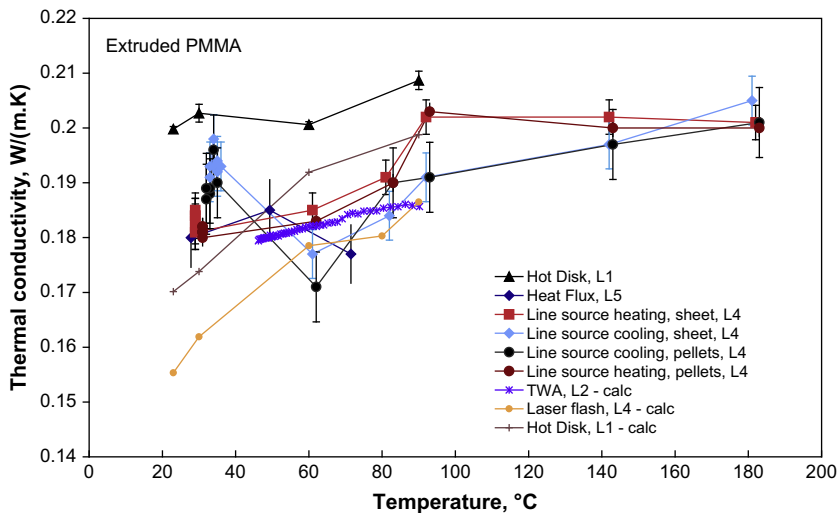

Fig. 10. Thermal conductivity of the extruded PMMA measured directly by transient line-source probe, Hot Disk and heat flux meter methods, and calculated from thermal diffusivity by laser flash, temperature wave analysis and Hot Disk methods.
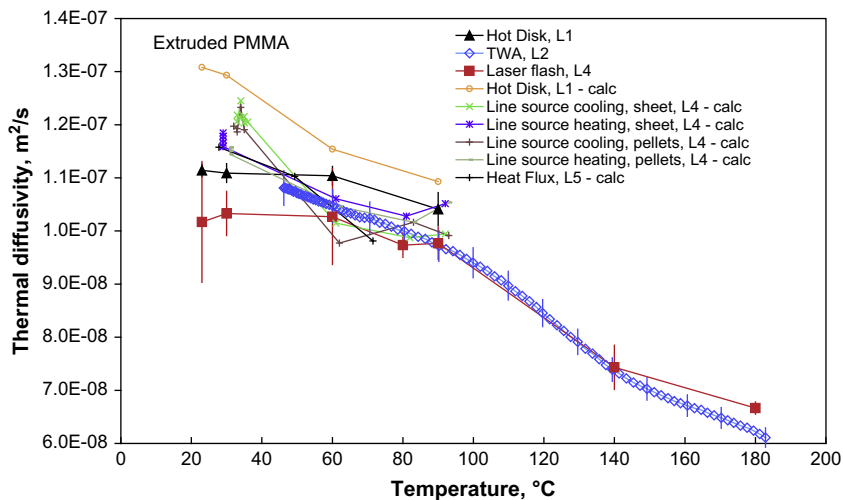
Fig. 11. Thermal diffusivity of the extruded PMMA measured directly by Hot Disk, laser flash and temperature wave analysis methods, and calculated from thermal conductivity measured by Hot Disk, heat flow meter and transient line-source probe methods.

line-source probe method, the specimen had to be formed in-situ by melting in a heated barrel prior to testing in order to achieve good thermal contact between the specimen and the probe sensor. Thus any effect that differences in microstructure may have on the thermal properties would potentially result in differences between the transient line-source probe data and those generated by other methods that used the as-provided sheet material. Line-source measurements on the cast PMMA were made on cut-up sheet samples, whereas the tests on the extruded PMMA were made on the original pellets as used to extrusion form the sheets. Thus, there were differences in the thermal history of the specimens that may also have affected the results.

It is clear that there are many differences between the methods and also the specimens tested, each of which will potentially have an effect on the measured thermal conductivity or thermal diffusivity values. However, in comparing the results between methods, generally the results are in very good agreement with differences being of the same order as the estimated uncertainties for the methods (Table 1). The thermal conductivity or thermal diffusivity values calculated using Eq. (1) were also in good agreement with directly measured values, indicating the accuracy of the specific heat capacity and density data.

The thermal conductivity and thermal diffusivity values measured using the Hot Disk method were generally slightly higher than the values obtained by other methods, although this difference was within the scatter obtained by some of the methods. The Hot Disk results presented are for bulk thermal conductivities where the bulk thermal conductivity is given by Eq. (2):

$$\lambda_{bulk} = \left(\lambda_{in-plane} \cdot \lambda_{through-thickness}\right)^{1/2} \qquad (2)$$

Thus, the measured 'bulk' thermal conductivity is a function of the through-thickness and in-plane thermal conductivities. If the in-plane value was higher than the

through-thickness value then by using this bulk approach the thermal conductivity measured using the Hot Disk method would be higher than the true through-thickness value as measured by the other techniques (except line-source probe). Furthermore, where this anisotropy effect is observed in measurements it will be carried through when calculating thermal conductivity from thermal diffusivity values, and vice versa. Preliminary results using the Hot Disk method indicated that the cast PMMA had a greater degree of anisotropy of thermal properties than the extruded PMMA, with thermal conductivities in the through-thickness direction being some 20% lower than in the in-plane direction for the cast PMMA, whereas the difference was of the order of 2% for the extruded PMMA. However, it was expected that the extruded PMMA sheet would have greater anisotropy with molecular alignment in the extrusion direction, and that the cast PMMA would have relatively little orientation. This was indeed demonstrated by annealing experiments which showed that the extruded PMMA relaxed more significantly, evidenced by bowing of the annealed sheet samples, than the cast PMMA. The reasons for this discrepancy in apparent anisotropy are not clear and require further study.

As the Hot Disk method can measure both the thermal conductivity and thermal diffusivity of the same specimen provided the material is isotropic, the volume heat capacity can be determined from the ratio of these values Eq. (1). However, for specimens with any anisotropy the heat capacity values calculated this way would be less accurate compared with values obtained for isotropic specimens, and strictly speaking should not be used. However, for the purposes of this intercomparison, volume heat capacity values were determined and converted to specific heat capacity values, using density, and plotted along with specific heat capacity values measured by DSC (Figs. 5 and 7) for the cast and extruded PMMAs respectively. The level of agreement is considered good. The Hot Disk values were approximately 5% to 15% higher for the extruded PMMA and up to 7% higher for the cast PMMA. Considering the

estimated uncertainty of 10% for DSC measurement these values compare reasonably well.

In summary there was very good agreement of thermal conductivity results with values being within a range of approximately ±7%. Also, very good agreement of thermal diffusivity results were obtained with values being within a range of approximately ±9%. Furthermore, there was also good agreement between thermal conductivity and thermal diffusivity values, when thermal conductivity values were converted to thermal diffusivity values (and vice versa) using specific heat capacity and density, even though the uncertainties in specific heat capacity values were of the order of ±10%. The level of agreement achieved demonstrates the validity of these various methods for thermal conductivity and thermal diffusivity measurement, notwithstanding the significant differences between the techniques and specimen preparation.

Acknowledgements

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