Reference Correlations for the Thermal Conductivity of Solid BK7, PMMA, Pyrex 7740, Pyroceram 9606 and SS304

Konstantinos D. Antoniadis[1] · Alexandra Tyrou[1] · Marc J. Assael[1] · Xiaojing Li[2] · Jiangtao Wu[3] · Hans-Peter Ebert[4]

Abstract

Although in the case of fluids there are several reference correlations of low uncertainty for thermal conductivity, for solids there are very few and in a restricted temperature range. The available experimental data for the thermal conductivity of five widely used solids, BK7, PMMA, Pyrex 7740, Pyroceram 9606, and SS304, have been critically examined with the intention of establishing thermal conductivity reference correlations. All experimental data have been categorized into primary and secondary data according to the quality of measurement specified by a series of criteria. A new reference correlation is proposed for BK7, and improved ones for PMMA, Pyrex 7740, Pyroceram 9606 and SS304.

Keywords BK7 · PMMA · Pyrex 7740 · Pyroceram 9606 · Reference correlations · Solids · SS304 · Thermal conductivity

1 Introduction

In a recent paper [1], reference values and correlations for the thermal conductivity of fluids were reviewed. Internationally accepted "reference values" (known also as "standard reference values") serve two primary purposes. First, they can provide a

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✉ Hans-Peter Ebert
   Hans-Peter.Ebert@zae-bayern.de

1   Chemical Engineering Department, Aristotle University, 54636 Thessaloniki, Greece

2   School of Energy, Power and Mechanical Engineering, North China Electric Power University, Baoding 071003, Hebei, People's Republic of China

3   School of Energy and Power Engineering, Xi'an Jiaotong University, Xi'an 710049, Shaanxi, People's Republic of China

4   Bavarian Center for Applied Energy Research, ZAE Bayern, Magdalene-Schoch-Str. 3, 97074 Wuerzburg, Germany

means of confirming the operation and experimental uncertainty of any new absolute apparatus and the stability and reproducibility of existing absolute measurement equipment. Second, in the case of instruments operating in a relative way, they provide the basis to calibrate one or more unknown constants in the working equation. Reference values refer to the properties specified at a fixed-state condition (e.g., specific temperature and composition) or at a small number of such states. These values are often characterized by the lowest uncertainty possible at the time of their acceptance. "Reference correlations" often cover a wide range of conditions and are developed to achieve the lowest possible uncertainties (although perhaps higher than those of reference values).

The International Association for Transport Properties (IATP), formerly known as the Subcommittee on Transport Properties of the International Union of Pure and Applied Chemistry (IUPAC), has been proposing reference values and correlations for a wide selection of fluids, molten metals and salts [1]. In this work, we attempt to propose reference correlations for five solids, BK7, PMMA, Pyrex 7740, Pyroceram 9606 and SS304. All selected solids have in common that there are well defined, stable and available on the market for decades in high quality.

Finally, we note that the analysis that is described here is applied to the best available experimental data for the thermal conductivity of the five solids. Thus, a prerequisite to the analysis is a critical assessment of the experimental data. For this purpose, two categories of experimental data are defined: primary data, employed in the development of the correlation, and secondary data, used simply for comparison purposes. According to the recommendation adopted by IATP, the primary data are identified by a well-established set of criteria [1]. These criteria have been successfully employed to establish standard reference values for the viscosity and thermal conductivity of fluids over wide ranges of conditions, with uncertainties in the range of 1 % to 2 % [1]. However, in many cases, such a narrow definition unacceptably limits the thermodynamic states for which data can be represented. Consequently, within the primary data set, it is also necessary to include results that extend the correlations over a wide range of conditions, albeit with a poorer uncertainty, provided they are consistent with other more accurate data or with theory. In all cases, the uncertainty claimed for the final recommended data must reflect the estimated uncertainty in the primary information.

1.1 Data Analysis and Quality

For each solid, the selected primary data of the thermal conductivity, λ ($W \cdot m^{-1} \cdot K^{-1}$), are fitted as a function of the absolute temperature, T (K), to an equation of the form

$$\lambda = \sum_{i=0}^{4} c_i T_r^i \qquad (1)$$

where $T_r = (T/273.15)$, and the coefficients c_i ($W \cdot m^{-1} \cdot K^{-1}$) are determined. We note that measurements were weighted in the fitting process in inverse proportion to the square of their claimed uncertainty.

To determine the quality of the reference correlations, three more quantities are introduced. We have defined the percent deviation as $\text{PCTDEV} = 100\,(\lambda_{\text{exp}} - \lambda_{\text{fit}})/\lambda_{\text{fit}}$, where λ_{exp} is the experimental value of the thermal conductivity and λ_{fit} is the value calculated from the correlation. Thus, the average absolute percent deviation (AAD) is found with the expression $\text{AAD} = (\sum |\text{PCTDEV}|)/n$, where the summation is over all n measurements, and the bias percent is found with the expression $\text{BIAS} = (\sum \text{PCTDEV})/n$. Finally, the uncertainty at a 95 % confidence level (2σ) defined as $2\left(\frac{100}{\lambda_{\text{av}}}\right)\sqrt{\left[\sum_{n}(\lambda_{\text{exp}} - \lambda_{\text{fit}})^2\right]\Big/ n}$ is also shown. The variable λ_{av} represents the average thermal conductivity of each solid for the range examined.

All these quantities will be discussed for every solid in turn, and their values together with the proposed recommended values are given in Sect. 7.

1.2 Experimental Techniques

In this section, problems associated with the techniques employed for the primary thermal conductivity measurements of the specific solids considered are discussed.

a.　Guarded Hot-Plate method

In the case of the stationary Guarded Hot-Plate (GHP) method, the influence on measurement results, of thermal contact resistances and of radiative heat transfer, in general, need to be discussed.

The hard and stiff material induces thermal contact resistances at the interfaces between the glass specimen and the measurement device in the case of contact methods. Where for some dynamic methods, e.g., laser flash, this effect is not relevant, for some stationary methods, like the guarded hot-plate method, and it has to be taken into account. Uneven or non-parallel plates or specimen surfaces lead to small cavities at the interfaces. The temperature-dependent thermal and infrared-optical properties of the enclosed gas and of the surfaces result in a temperature-dependent thermal contact resistance, which decreases nonlinear with increasing temperature.

To minimize the contact resistance, thin flexible high thermally conductive sheets could be used as interlayers, e.g., Nickel gauze with wire diameters of 80 μm. A more precise correction of the effect is possible by thermal conductivity measurements for a given mean temperature and temperature difference between hot and cold plate under different gas atmospheres, preferably under high vacuum, or under nitrogen, or helium at atmospheric pressure. If there are cavities between plate and specimen surface, the corresponding thermal contact resistance varies with the use of atmospheres with different thermal conductivity.

$$\frac{1}{\Lambda_{\text{exp}}} = \frac{1}{\Lambda_{\text{s}}} + \frac{2}{\delta + \frac{\lambda_g}{d_{\text{int}}}} \qquad (2)$$

where Λ_{exp} is the measured heat transfer coefficient, Λ_{s} the heat transfer coefficient of the specimen, δ the radiative heat transfer coefficient in the gap, λ_{g} the thermal

conductivity of the gas, and d_{int} the effective, average thickness of the cavities at the interface between specimen and measuring plates. A nonlinear regression of Eq. 2 on the experimental data provides the constant thermal specimen resistance and thus the thermal conductivity of the specimen during the different measurements at constant mean temperatures (see Fig. 1).

A further complication for the determination of a true thermal conductivity values results in the semitransparency of BK7 at higher temperatures. In 1961, Gardon [4] gave an early overview of the influence of thermal radiation on the heat transfer in glasses. He stated that for stationary plate methods at higher temperatures apparent thermal conductivity values were determined which depend on the thickness of the investigated specimen. This fact, according to Gardon, has repeatedly led in the past to confusions regarding the wide spreading of measurement results by different investigators. In retrospect, this effect is also one reason of the unsatisfying result of the round robin test in 2002. In a good approximation, BK7 can be considered as a diathermic medium with a transparent band for wavelength below 2.8 μm while it is opaque above 5 μm [5, 6]. Between 2.8 μm and 5 μm, there is a semitransparent transition regime, depending on the thickness of the glass layer. The contribution of the radiative heat transfer to a determined thermal conductivity value can be estimated in the case of using a GHP setup. A one-dimensional configuration is assumed. The specimen is a slab enclosed by two parallel measuring plates of the GHP apparatus. The one-dimensional radiative heat flux density q_r is approximately determined by

$$q_r \approx 4 \, \sigma \, T_r^3 \, \Delta T \, F\left(\lambda_c, T\right) \quad = \lambda_{\text{app}} \, \Delta T \tag{3}$$

where σ is Stefan–Boltzmann constant, $T_r = (T_1^2 + T_2^2)(T_1 + T_2)/4$, is the radiative mean temperature, ΔT the temperature difference between the hot and cold plate of
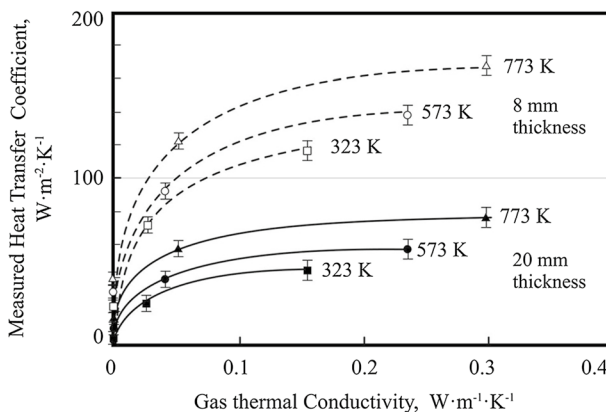

Fig. 1 GHP thermal conductivity measurement results, i.e., determined heat transfer coefficient (symbols), on BK7 at different mean temperatures as function of the thermal conductivity of the gas atmosphere (vacuum, nitrogen and helium) [2]. Two sets of specimens with thicknesses with 8 mm and 20 mm were investigated. Details of the GHP setup could be found in [3]. The lines indicate the theoretical description according to Eq. 2

the GHP, $F(\lambda_c,T)$ the black body fraction [7] with λ_c the cut-off wavelength (2.8 μm), and λ_{app} the apparent thermal conductivity.

Figure 2 shows the apparent radiative contribution to the true thermal conductivity due to the direct radiative heat exchange between the two measuring plates via the diathermic BK7 for two specimen with thicknesses of 8 mm and 20 mm. The black body fraction is also depicted. At 573 K only 2 % of the black body radiation is transmitted in the wavelength range below 2.8 μm. This fraction increases up to 10 % for a temperature of 773 K. With increasing thickness of the specimen the apparent radiative thermal conductivity contribution increases. The measured thermal conductivity values have to be reduced by this contribution, i.e., this corresponds to corrections of less than 1 % at 573 K and of 4 % at 773 K for the specimen with 8 mm thickness and 9 % for the 20-mm-thick specimen.

In the case of BK7 the applicability of the GHP method for the determination of the true thermal conductivity is limited to temperature below 573 K. Above 573 K, the measured values have to be corrected for the radiative contribution. The thicker the specimen, the higher the correction.

The GHP technique has successfully been employed by Ebert [2] for the measurement of the thermal conductivity of BK7, while Rudtsch and Hammerschmidt [8], and Boumaza and Redgrave [9] employed it for measurements in PMMA, Longo [10] for Pyrex 7740, and Filla et al. [11] for measurements on Pyroceram 9606.

The aforementioned discussion also holds for techniques that are based on the same principles such as the radial-heated cylinders, the axial heat conduction and the high-temperature longitudinal techniques, all employed for the measurement of the thermal conductivity of solids.

b.   Laser-flash method

The calculation of thermal conductivity values using values of density ρ, specific heat capacity c_p and thermal diffusivity a is carried out according to
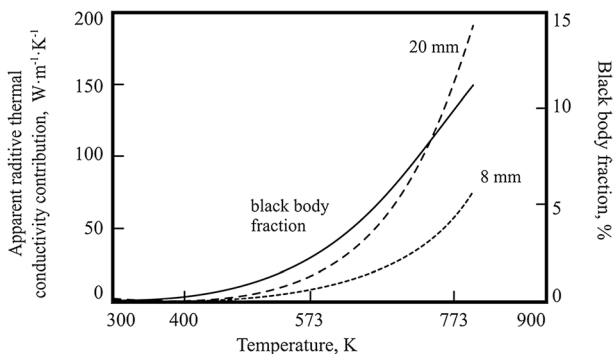

Fig. 2 Apparent radiative contribution to the true thermal conductivity due to the direct radiative heat exchange between the two measuring plates with emissity 0.8 via the diathermic BK7 (cut-off wavelength 2.8 μm) for two specimen with thicknesses of 8 mm and 20 mm as a function of temperature. The black body fraction as function of temperature is shown for a cut-off wavelength of 2.8 μm

$$\lambda(T) = a(T) \ \rho(T) \ c_p(T). \tag{4}$$

It is important that the thermal diffusivity value describes the true thermal diffusivity without any thermal radiation effect due to ballistic radiative heat transfer between the specimen front and rear side which could be critical for measurements on semitransparent media above room temperature. The influence of radiative heat transfer in the laser-flash experiment and correction methods was described in detail elsewhere [12–15].

In some cases, the ballistic radiative heat transfer is minimized by applying a low-emissivity coating on the specimen surfaces [2, 16, 17]. Therefore, for specimen used for measurements up to 873 K, the surfaces were first coated with a low-emissive gold layer and then with a graphite layer to optimize the absorption of the laser pulse energy at the front side and the contactless detection of thermal radiation at the rear side. The resulting measurement curves were evaluated according to the model of Carslaw and Jaeger [18].

Taking into consideration the aforementioned discussion, Göbel et al. [16], Hemberger et al. [17], and Ebert [2] have successfully employed the laser-flash technique to measure the thermal conductivity of BK7, and Gaal et al. [19] for measurements on Pyrex 7740 and Pyroceral 9606.

c.   Transient hot wire

In this technique, a current is applied to a fine wire (or strip) of known length which acts as both a heating element and a resistance thermometer. The wire is placed between two solid samples, a current is applied, and the temperature rise of the wire is measured as a function of time. The thermal conductivity is derived from the reciprocal of the slope of the linear portion of the plot of the temperature rise versus the logarithm of the time.

The main problem of this technique is the contact resistance between the heating wire (or strip) and the solid sample. A successful solution of this problem [20, 21] was to embed the wires in a thin soft silicon layer. The soft silicone layer eliminates the possibility of having an air layer between the wire and the solid, and thus it greatly enhances the contact between wire and silicone. In this arrangement, measurements of the temperature rise at very short times provide the properties of the silicone layer, while from measurements at longer times the properties of the solid are acquired. However, measurements are restricted to the highest temperature that the silicone remains soft (about 570 K). Assael et al. [20–25] employed this technique for measurements of the thermal conductivity of the five solids considered.

In other instruments with similar working principles, the heating element is placed in a thin polyimide layer, which in turn is placed between the solids, and the contact resistance is dealt through the evaluation of the raw data. Hammerschmidt and Meier [26] employed a transient hot bridge for measurements in BK7, Pyrex 7740, and PMMA, and Log, and Metallinou [27] and Gustafsson [28] employed the transient plane source to measure the thermal conductivity of

PMMA. Similar instruments were employed by Kubicar et al. [29, 30] for measurements on BK7 and PMMA, and Malinaric and Dieska [31] for measurements on PMMA.

d.   Other techniques

The principle of the 3ω method is the application of an alternating current of angular frequency ω through a thin metal heater line that has been directly deposited on an electrically insulated specimen. The method was extensively employed by Cahill and Pohl [32] for the measurement of thin films and solids. Nevertheless, the measurements of Cahill and Pohl [32] were not included in the primary datasets as (a) no uncertainty budget was given, and (b) measurements seemed to be higher than all others. In the case of Pyrex 7740, however, the measurements of Cahill [33] cover a very wide range and seem to agree well with the rest. Hence, regardless of the fact that no uncertainty is quoted, this set was included in the primary dataset, with a high estimated uncertainty.

Finally, we note that there are a few other heat transfer experiments employed for the measurement of the thermal conductivity of these solids. These are considered very carefully as usually no proper description, or uncertainty budget is included. Hence, the measurements on PMMA of Dawson et al. [34] (heat transfer experiment), on Pyrex 7740 by Matsumoto and Ono [35] (radiative heat exchanger technique), and the measurements on SS304 by Takahashi et al. [36] (steady-state direct electric heating), these were considered as primary data.

2 BK7

BK7 is a borosilicate glass produced by Schott AG, Germany, and is widely used for optical systems as it can be manufactured with outstanding homogeneity. In weight percent, it consists mainly of 70 % silica, 10 % boron oxide, 8.4 % sodium oxide and 8.4 % potassium oxide. Its density is 2504 kg·m^{-3} at 298 K [37], and it has isotropic thermophysical properties with excellent long-term stability as long as the material is not heated above its glass transition temperature of approximately 830 K [37].

In the case of BK7, an intercomparison among 11 European laboratories was organized by PTB [5], aiming to qualify it as a possible candidate reference material for thermal conductivity, produced in 2002 uncertainties up to 20 % at ambient temperature and 40 % at higher temperatures, which were far in excess of the laboratories' quoted uncertainties. Hence, this material is still under consideration.

2.1 Data Compilation

Table 1 shows, to the best of our knowledge, all the experimental datasets for the measurements of the thermal conductivity of solid BK7. In the table, the supplier of the sample, the technique employed, and the uncertainty quoted are also presented. Furthermore, the form in which the data are reported and the temperature range

Table 1 Datasets considered for the thermal conductivity of BK7 as a function of temperature

First author	Refs.	Publ. year	Supplied by	Technique employed	Uncertainty quoted (%)	No. of data	Temp. range (K)	Form of data
Primary data								
Cao	[38]	2016	na	3ω	6.0	1	298	T
Assael	[37]	2015	Schott AG	THW	2.0	1	298	T
Göbel	[16]	2013	Schott AG	LF (TD)	11.6	17	79–323	D
				LF (TD)	7.7	9	200–323	D
Hemberger	[17]	2010	Schott AG	LF (TD)	11.6<190 K 7.7>190 K	19	82–296	D
Assael	[25]	2008	Schott AG	THW	1.0	7	315–433	T
Hammerschmidt	[26]	2006	Schott AG	THB	2.0	1	293	T
Assael	[24]	2005	Schott AG	THW	1.5	9	295–498	T
Kubicar	[29]	2005	Schott AG	PTM (TD)	5.0	1	298	T
Ebert	[2]	2002	Schott AG	LF (TD)	7.7	11	296–773	D
				GHP	4.6–5.4	5	323–773	D
				THW	5.0	26	233–773	D
Secondary data								
Balachandar	[39]	2017	na	IVI (TD)	na	1	300	T

3ω 3ω technique, *GHP* Guarded Hot Plate, *IVI* Instantaneous Velocimetric Interferometer, *LF* laser flash, *PTM* pulse transient method, *TD* thermal diffusivity, *THB* transient hot bridge, *THW* transient hot wire, *D* diagram, *T* tabulated experimental data, *na* not available

covered is also noted. In the case of data in diagrams a free online standard digitizing software was employed to read the values (no influence on the quoted uncertainty was detected). The datasets have been classified into primary and secondary sets, as mentioned in Sect. 1. Following our aforementioned discussion, the guarded heat-flow measurements of Ebert [2], as well as the laser-flash measurements of Göbel et al. [16], Hemberger et al. [17], and Ebert [2] were very carefully performed and hence form part of the primary data. Primary data are also shown in Fig. 3.

In addition to these, Assael et al. [24, 25, 37] employed a transient hot-wire technique to measure the thermal conductivity of BK7 with uncertainty of 1.5 % and 1.0 %. To avoid contact resistances, the two 25-μm-diameter wires were embedded in a 1 mm flat silicone paste squeezed between two specimens of BK7. The method is absolute backed by a full theory. These measurements also form part of the primary dataset. A transient hot-wire was also employed by Ebert [2], with 5 % uncertainty, and these measurements also formed part of the primary dataset. Hammerschmidt and Meier [26] employed a very carefully designed, transient hot bridge with a 2 % uncertainty for the measurement of the thermal conductivity of BK7, while Kubicar et al. [29] employed a pulse-transient technique with a 5 % uncertainty. Both these sets also formed part of the primary dataset. Finally the more recent 3ω measurement of Cao et al. [38] was also included in the primary dataset.

2.2 Discussion

The primary data for the thermal conductivity, λ (W·m^{-1}·K^{-1}) shown in Table 1, were fitted as a function of the absolute temperature, T (K), to Eq. 1. Coefficients c_i

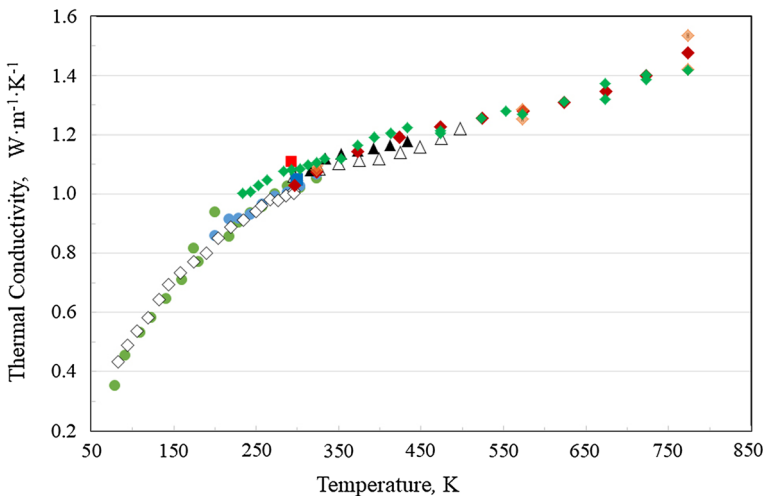**Fig. 3** Primary experimental thermal conductivity measurements for BK7, as a function of the temperature. (×) Cao et al. [38], (▲) Assael et al. [37], (●) Calorim., (●) MDSC Göbel et al. [16], (◇) Hemberger et al. [17], (▲) Assael et al. [25], (■) Hammerschmidt [26], (△) Assael et al. [24], (■) Kubicar et al. [29], (◆) LF, (◆) GHP, (◆) THW, Ebert [2]

are shown in Table 7 and recommended values obtained through Eq. 1 are shown in Table 8.

The uncertainty (at the 95 % confidence level) over the whole temperature range, 79–773 K, is 5.2 % (AAD is 2.3 % and BIAS is 0.0 %), dropping to 4.3 % over the restricted temperature range 273–773 K. Strictly speaking however, since the measurements in the range 79–233 K have an associated uncertainty of 11.6 %, this value should be employed for the uncertainty in this temperature range.

Figure 4 shows the percentage deviations of the primary thermal conductivity data of BK7 from those calculated by Eq. 1, as a function of the temperature. It can be seen that the proposed reference correlation for BK7 represents all primary data within the mutual uncertainties.

3 PMMA

Polymethyl methacrylate (PMMA) or Perspex® is an amorphous, colorless thermoplastic material of excellent optical transparency, and a luminous transmittance of about 92 %. It has good abrasion resistance and dimensional stability, but is brittle and notch sensitive. Its water absorptivity is very low when compared with other polymer materials, and its density is (1185 ± 5) kg·m^{-3} [37].

Because of its amorphous structure, PMMA was a preferred material in the 50 s and 60 s of the last century for the experimental verification of theoretical heat conduction models in amorphous polymers. Thus, measurements have been performed in temperatures as low as 1 K (e.g., Reese [40, 41], Berman [42]), however with
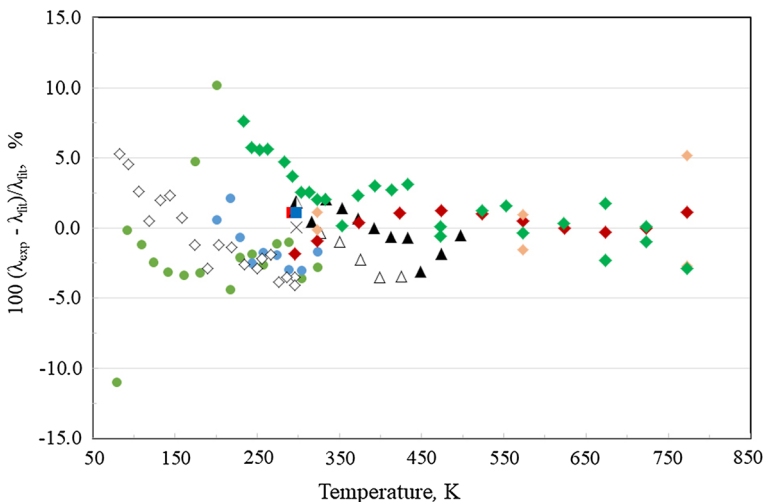

Fig. 4 Percentage deviations of the primary experimental thermal conductivity measurements for BK7, from those calculated by Eq. 1, as a function of the temperature. (×) Cao et al. [38], (⬬) Assael et al. [37], (●) Calorim., (●) MDSC Göbel et al. [16], (◇) Hemberger et al. [17], (▲) Assael et al. [25], (■) Hammerschmidt [26], (△) Assael et al. [24], (■) Kubicar et al. [29], (◆) LF, (◇) GHP, (◆) THW, Ebert [2]

higher uncertainties and large disagreements between each other. In this work, we concentrate in measurements over 90 K, as they are more reliable and can be considered as primary data for developing a reference correlation.

In 2001, Tye and Salmon at NPL (published in 2004 [43]), after a very careful investigation aiming to produce a candidate thermal conductivity reference polymer, proposed an equation for the thermal conductivity of PMMA, covering a temperature range 273–353 K, with an uncertainty of 1 % at the 95 % confidence level.

In 2004, Rudtsch and Hammerschmidt [8], for PTB, also aiming to propose a thermal conductivity reference material candidate, coordinated in PTB an intercomparison project for PMMA involving 17 European laboratories. Unfortunately, the thermal conductivity values produced even at 303 K, ranged from 0.16 $W·m^{-1}·K^{-1}$ to 0.21 $W·m^{-1}·K^{-1}$, while uncertainties ranged from 8 % to 13 % in the thermal conductivity values, which far exceeded the laboratories' quoted uncertainties. It was argued that the most probable reason for this discrepancy was the improperly treated effect of contact resistance (see also Sect. 2.1). To prove independently that the thermal contact resistance was adequately taken into account, in the same article, values obtained at PTB by two different methods (guarded hot plate and transient hot strip) were also reported.

In 2009, Rides et al. [44] for NPL also carried out an intercomparison project for PMMA, but no values were recommended. Finally, in 2014 Rohde et al. [45] published an inter laboratory (11 techniques: 9 laser-flash instruments, 1 hard disk, and 1 transient hot-wire instrument) comparison of thermal diffusivity for PMMA, with average values with an uncertainty of 4 % (at the 95 % confidence level), covering a temperature range 293–373 K.

Another point that ought to be discussed is the glass transition temperature of PMMA. Rides et al. in 2009 [44] stated that the glass transition range, measured by differential scanning calorimetry with heating at 10 $K·min^{-1}$, occurred between approximately 373 K and 403 K. Dixit et al. [46] in 2009 also employed a differential scanning calorimetry to investigate the glass transition temperature of PMMA, and a value of 356.95 K was quoted. Dos Santos [47] in 2013 measured a glass transition temperature of 386 K. Furthermore, they observed a drop in its thermal conductivity several degrees before the glass transition temperature value determined by DSC. This fact may be explained having in mind that the conventional glass transition is a kinetic (rate-dependent) manifestation of an underlying thermodynamic phenomenon, and corresponds to the temperature at which the forces that keep the connected chain segments of a solid polymer are overcome by the thermally induced movement within the experimental time scale. Hence, the value of such a property depends on the heating rate and the time that the material remains at a fixed constant temperature. For the thermal conductivity measurements of PMMA performed with the transient hot-wire technique, the actual glass transition temperature is attained around 360 K [46], and not at 385 K as determined by the classical differential scanning calorimetry.

Hence, following this discussion it seems best not to consider values of the thermal conductivity of solid PMMA over 360 K.

It should be also noted that stretched PMMA may have in extreme cases a 50 % increase in thermal conductivity in the direction of elongation [48].

3.1 Data Compilation

Table 2 shows, to the best of our knowledge, all the experimental datasets for the measurements of the thermal conductivity of solid PMMA. These are also shown in Fig. 5. As previously, in the table, the supplier of the sample, the technique employed, and the uncertainty quoted are also presented. Furthermore, the form in which the data are reported and the temperature range covered is also noted. The reference correlation of Tye and Salmon [43], as well as the average values proposed by Rohde et al. [45], are also shown on the top of the table. The datasets have been classified into primary and secondary sets.

The measurements of Assael et al. [24, 25, 37] were performed in a 2-Ta-wires transient hot-wire instrument. To avoid contact resistance the wires were embedded in a silicon layer, and squeezed between two samples of the solid. As the technique is absolute and its uncertainty is better than 2 %, these measurements were considered as primary data. The transient hot-bridge instrument developed by Hammerschmidt and Meier [26] operating with an uncertainty of 2 %, as well as the transient plane-source (hot disk) instrument of Rudtsch and Hammerschmidt [8], operating with an uncertainty of better than 2.2 %, were also part of the primary datasets. Similarly, of very low uncertainty, less than 2 % were the dynamic plane-source measurements of Malinaric and Dieska [31], the pulse transient method of Kubicar et al. [30], the transient heating technique of Lima e Silva et al. [49] and Jiang [50], and the radial heated cylinder measurements of Chen et al. [51]. All these measurements formed part of the primary dataset.

Following our previous discussion on guarded hot-plate instruments in Sect. 2.1, we also included in the primary datasets the guarded hot-plate measurements of Rudtsch and Hammerschmidt [8] and Shoulberg and Shetter [52], performed with an uncertainty of about 2 %, as well as the higher uncertainty measurements of Miller and Kortlar [53] and Boumaza and Redgrove [9].

Finally we did include in the primary dataset, the higher uncertainty measurements of Dawson et al. [34], Stefkova and Zmeskal [54], Log and Gustafsson [55], and Eiermann and Hellwege [48], as in their papers, a full description of the technique employed, the uncertainty of the instrument and the origin of the sample measured were given.

The 3ω measurements of Cahill and Pohl [32], and the guarded hot-plate measurements of Hattori [61], were not included in the primary dataset, as they did not quote the uncertainty of the measurements. Furthermore, the measurements of Cahill and Pohl [32] were consistently higher than the rest, while the measurements of Hattori were very much lower (see Fig. 5), which was also reported for other measurements that he also performed [62]. No uncertainty was also given for the single transient hot-wire measurement of Takeuchi and Suzuki [60]. Quite lower than anybody else were also the guarded hot-plate measurements of Zeng et al. [57] (see Fig. 5). Finally as secondary measurements, we considered the high-uncertainty measurements of Cao et al. [38], Qiu et al. [56], and Lin et al. [59], as well as the 2004 Malinaric [58] measurements as they were superseded by their 2009 measurements of lower uncertainty [31]. We note, that although we did include the single measurement of Cao et al. [38], in the primary dataset for BK7, in the case of

Table 2 Datasets considered for the thermal conductivity of PMMA as a function of temperature

First author	Refs.	Publ. year	Supplied by	Technique employed	Uncertainty quoted (%)	No. of data	Temp. range (K)	Form of data
Previous reference correlation/values								
Tye	[43]	2004	Reference correlation		1.0 (2σ)		273–353	E
Rohde	[45]	2014	Evonik Röhm GmbH (average values)	9 LF (TD) 1 HD 1 THW	4.0 (2σ)	9	293–373	D
Primary data								
Assael	[37]	2015	Degussa Röhm Plexiglas GmbH	THW	2.0	1	298	T
Malinaric	[31]	2009	na	DPS	1.5	1	296	T
Assael	[25]	2008	Degussa Röhm Plexiglas GmbH	THW	1.0	5	314–353	T
Dawson	[34]	2008	na	HTC	3.0	1	323	T
Hammerschmidt	[26]	2006	Degussa Röhm Plexiglas GmbH	THB	2.0	1	293	T
Stefkova	[54]	2006	Perspex	PTM	3.6	1	298	T
Assael	[24]	2005	Degussa Röhm Plexiglas GmbH	THW	1.5	6	308–353	T
Kubicar	[30]	2005	na	PTM	1.6	1	298	T
Rudtsch	[8]	2004	Degussa Röhm Plexiglas GmbH	TPS & GHP	2.2 < 243 K 1.5 > 243 K	16	203–353	T
Boumaza	[9]	2003	NPL	TPS	5.0	3	293–344	T
				GHP	4.0	3	293–344	T
Lima e Silva	[49]	1998	Perspex	TH	2.0	1	306	T
Chen	[51]	1995	na	RHC	1.2	1	292	T
Log	[55]	1995	Perspex, ICI, Inc	TPS	5.0	2	293	T
Miller	[53]	1993	na	GHP	5.0	1	296	T
Jiang	[50]	1991	na	TH	1.0	1	298	T

Table 2 (continued)

First author	Refs.	Publ. year	Supplied by	Technique employed	Uncertainty quoted (%)	No. of data	Temp. range (K)	Form of data
Eiermann	[48]	1962	Evonik Röhm GmbH	QSTP	4.0	91	95–363	D
Shoulberg	[52]	1962	Rohm-Haas Co.	GHP	2.0	16	303–363[a]	D
Secondary data								
Cao	[38]	2016	na	3ω	6.0	1	298	T
Qiu	[56]	2011	na	3ω	10.0	1	298	T
Zeng	[57]	2008	na	GHP	5.0	5	287–300	T
Malinaric	[58]	2004	na	DPS	3.6	1	293	T
Lin	[59]	1994	na	na	6.0	2	293, 303	T
Cahill	[32]	1987	Commercial grade	3ω	na	52	34–301	D
Takeuchi	[60]	1984	na	THW	na	1	291	T
Hattori	[61]	1962	na	GHP	na	5	301–363[a]	D

3ω 3ω technique, *DPS* Dynamic Plane Source, *GHP* Guarded Hot Plate, *HD* hot disc, *HTC* Heat Transfer Coefficient Method, *PTM* pulse transient method, *RHC* radial heated cylinder, *TH* transient heating, *THB* transient hot bridge, *THW* transient hot wire, *TD* thermal diffusivity, *TP* twin probe, *TPS* transient plane source, *QSTP* Quasi-Stationary Twin Plate Method, *D* diagram, *E* equation, *T* tabulated experimental data, *na* not available

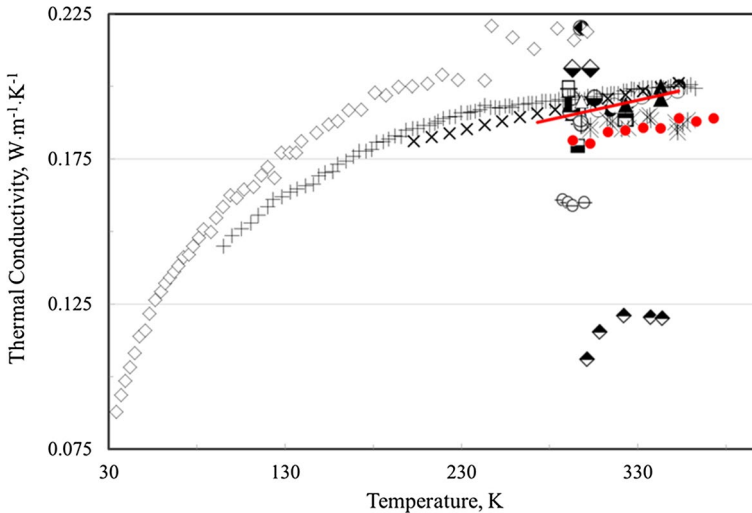[a]Restricted to a maximum temperature of 363.15 K

Fig. 5 Experimental thermal conductivity measurements for PMMA, as a function of the temperature. (◯) Assael et al. [37], (▮) Malinaric and Dienska [31], (●) Assael et al. [25], (�) Dawson et al. [34], (◆) Hammerschmidt and Meier [26], (△) Stefkova and Zmeskal [54], (○) Assael et al. [24], (⊕) Kubicar et al. [30], (x) Rudtsch and Hammerschmidt [8], (▲) Boumaza and Redgrove [9], (◖) Lima e Silva et al. [49], (◓) Chen et al. [51], (◗) Log and Gustafsson [55], (▭) Miller and Kotlar [53], (◐) Jiang et al. [50], (+) Eiermann and Hellwege [48], (✕) Shoulberg and Shetter [52], (◐) Cao et al. [38], (◆) Qiu et al. [56], (◒) Zeng et al. [57], (▮) Malinaric [58], (◆) Lin et al. [59], (◇) Cahill and Pohl [32], (▱) Takeuchi and Suzuki [60], (◆) Hattori [61], as well as the reference correlation of (━) Tye [43], and the proposed average values of (●) Rohde et al. [45]

PMMA the 6 % quoted uncertainty by Cao et al. [38] is large compared to the rest of the primary data used.

Figure 6 shows the primary datasets, shown also in Table 2, employed for the measurement of the thermal conductivity of PMMA.

3.2 Discussion

The primary data for the thermal conductivity, λ (W·m^{-1}·K^{-1}) shown in Table 2, were fitted as a function of the absolute temperature, T (K), to Eq. 1. The coefficients c_i (W·m^{-1}·K^{-1}) are shown in Table 7. In the same table the temperature range, T_{range} (K), of applicability of Eq. 1, derived from the corresponding temperatures in Table 2, is also presented.

As shown in Table 7, in the case of PMMA, the uncertainty (95 % confidence level) over the whole temperature range, 95–363 K, is 4.4 % (AAD is 1.8 % and BIAS is 0.3 %). Figure 7 shows the percentage deviations of the primary thermal conductivity data of PMMA from those calculated by Eq. 1, as a function of the temperature. It can be seen that the new reference correlation represents well within the mutual uncertainties, the previous reference correlation proposed by Tye and Salmon [43], the average values proposed by Rohde et al. [45], and all primary data.
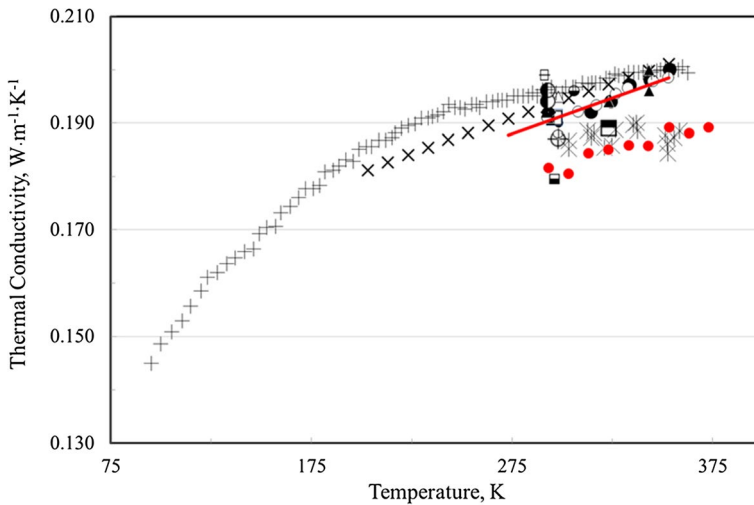
Fig. 6 Primary experimental thermal conductivity measurements for PMMA, as a function of the temperature. (○) Assael et al. [37], (▯) Malinaric and Dienska [31], (●) Assael et al. [25], (▮) Dawson et al. [34], (◆) Hammerschmidt and Meier [26], (△) Stefkova and Zmeskal [54], (○) Assael et al. [24], (⊕) Kubicar et al. [30], (x) Rudtsch and Hammerschmidt [8], (▲) Boumaza and Redgrove [9], (◒) Lima e Silva et al. [49], (◓) Chen et al. [51], (◑) Log and Gustafsson [55], (▭) Miller and Kotlar [53], (⏅) Jiang et al. [50], (+) Eiermann and Hellwege [48], (✕) Shoulberg and Shetter [52], (◐) Cao et al. [38], (◆) Qiu et al. [56], (⊖) Zeng et al. [57], (▯) Malinaric [58], (◆) Lin et al. [59], (◇) Cahill and Pohl [32], (⊟) Takeuchi and Suzuki [60], (◆) Hattori [61], as well as the reference correlation of (▬▬) Tye [43], and the proposed average values of (●) Rohde et al. [45]

Finally, recommended values obtained through Eq. 1, and the corresponding coefficients c_i from Table 7 are shown in Table 8.

4 Pyrex 7740

Pyrex 7740 is a well-known Type I, Class A borosilicate glass, which conforms to ASTM E438 [37]. Pyrex 7740 is an original product of Corning, USA, patented in 1915. It has a density of 2.23×10^3 kg·m^{-3} at 293 K [37] and consists approximately of 80.7 % silica, 13 % boron oxide, 4.0 % sodium oxide and 2.3 % aluminum oxide. It has a low coefficient of expansion, which allows to be manufactured in relative heavy walls giving it mechanical strength and thermal shock resistance, while retaining reasonable heat resistance. Moreover, it is highly resistant to chemical compounds such as strong acids, alkalis, etc., and can withstand temperatures up to 760 K [37]. Therefore, due to its excellent thermal and mechanical properties, Pyrex 7740 is used in many laboratory and industrial applications.

In 1966, Powel et al. [63] published reference values of the thermal conductivity with a 5 % maximum uncertainty covering a temperature range 100 K to 700 K. Three years earlier a National Bureau of Standards (NBS) complementary report by Flynn [64] stated probable thermal conductivity values for Pyrex 7740 from 273 K to 573 K which resulted from literature values, unpublished data and NBS measurements with
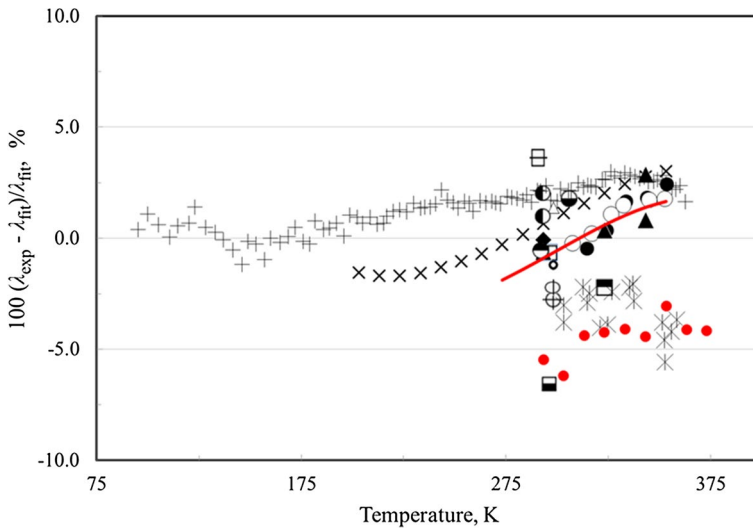
Fig. 7 Percentage deviations of the primary experimental thermal conductivity measurements for PMMA, from those calculated by Eq. 1, as a function of the temperature. (⬡) Assael et al. [37], (▮) Malinaric and Dienska [31], (●) Assael et al. [25], (▱) Dawson et al. [34], (◆) Hammerschmidt and Meier [26], (△) Stefkova and Zmeskal [54], (○) Assael et al. [24], (⊕) Kubicar et al. [30], (x) Rudtsch and Hammerschmidt [8], (▲) Boumaza and Redgrove [9], (◓) Lima e Silva et al. [49], (◓) Chen et al. [51], (◑) Log and Gustafsson [55], (▬) Miller and Kotlar [53], (⏀) Jiang et al. [50], (+) Eiermann and Hellwege [48], (✳) Shoulberg and Shetter [52], (◖) Cao et al. [38], (◆) Qiu et al. [56], (⊖) Zeng et al. [57], (▮) Malinaric [58], (◈) Lin et al. [59], (◇) Cahill and Pohl [32], (⊟) Takeuchi and Suzuki [60], (◆) Hattori [61], as well as the reference correlation of (▬) Tye [43], and the proposed average values of (●) Rohde et al. [45]

uncertainties of 5 % below 573 K and mentioned higher uncertainties above because of thermal radiation effects. Similar to BK7, Pyrex 7740 is transparent for thermal radiation at wavelengths below 2.7 μm and radiation effects are in the same order of magnitude occur, although they tend to be smaller than for BK7. In 1985, Hulstrom et al. [65] recommended values from round robin tests, characterized by a 10.3 % standard deviation (at the 95 % confidence level), covering a temperature range 323 K to 573 K.

In September 1990, the European Community Bureau of Reference (BCR) finally issued a certificate for Pyrex glass material [66]. This certified material is now available as CRM 039 from the European Union Institute of Reference Materials and Measurements (IRRM) in Geel, Belgium. However, it should be noted that this certificate refers only to a Pyrex glass and not specifically the 7740 grade. These certified values are characterized by a 1.7 % standard deviation at the 95 % confidence level.

4.1 Data Compilation

Table 3 shows, to the best of our knowledge, all the experimental datasets for the measurements of the thermal conductivity of solid Pyrex 7740. These are also shown in Fig. 8. As previously, in the table, the supplier of the sample, the technique

employed, and the uncertainty quoted are also presented. Furthermore, the form in which the data are reported and the temperature range covered is also noted. The reference correlation of Williams and Shawyer [66], as well as the reference values of Hulstrom et al. [65] and Powel et al. [63], are also shown on the top of Table 3, and in Fig. 8. The datasets have been classified into primary and secondary sets.

As discussed in the case of PMMA, the Pyrex 7740 measurements of Assael et al. [23, 25, 37] were performed in a 2-Ta-wires transient hot-wire instrument. To avoid contact resistance the wires were embedded in a silicon layer, and squeezed between two samples of the solid. As the technique is absolute and its uncertainty is better than 2.0 %, these measurements were considered as primary data. The transient hot-bridge measurements obtained by Hammerschmidt and Sabuga [67, 68] with an uncertainty of 2.5 %, as well as the laser-flash measurements of Gaal et al. [19], performed with a 2 % uncertainty, and the pulse-transient measurements of Milano et al. [69], performed with a 1.7 % uncertainty, were also part of the primary datasets, as full descriptions of the experimental setups were published (Fig. 9).

Following the discussion in Sect. 2.1, the guarded comparative-longitudinal-heat-flow measurements of Pillai and George [71], obtained with an uncertainty of 2 %, were also included in the primary dataset. Carefully designed guarded hot-plate instruments were also employed by Longo [10] operating at 3.8 % uncertainty, and by Ohmura [70], and Miller and Kotlar [53], with 5 % uncertainty. These were also part of the primary dataset. Two more sets were included in the primary dataset, as shown in Table 3; the short transient hot-strip measurements of Log and Metallinou [27] obtained with a 3 % uncertainty, and the radiative heat exchanger measurements of Matsumoto and Ono [35] with a 5 % uncertainty.

Finally, although the 3ω measurement of the thermal conductivity of PMMA performed by Cahill [33] was considerably higher than the rest, in the case of Pyrex 7740 they cover a very wide range and seem to agree well with the rest. Hence, regardless of the fact that no uncertainty is quoted, this set is the last one included in the primary dataset, with caution.

Examining the secondary data in Table 3, the guarded hot-plate measurements of Fujishiro et al. [77] were up to 40 % lower than the rest. The dynamic measurement access technique employed by Zawilski and Tritt [75] with no quoted uncertainty produced values which were up to 20 % higher than the rest. Similarly the calorimetric measurements of Yang et al. [79], with no quoted uncertainty, showed values that extended from -15 % to $+15$ %, from all other measurements. In the secondary dataset, the guarded hot-plate measurements of Himeno et al. [78] were also included, as the uncertainty quoted was 10 %. In addition, data from Flynn [64] were also assigned to the secondary data, as some of them were already taken into account for Powell's reference values. Campbell et al. [73] published in 2013 data between 93 K and 748 K derived by laser-flash measurements. The authors mentioned that due to the specific preparation, i.e., applying gold coatings at both sides of the specimen to avoid ballistic radiative heat transfer, at higher temperatures no radiation effects were observed. However, the data show at the lowest and highest temperatures deviations of -18 % and only in a medium temperature range, and their data were close to the proposed polynomial for the primary data. This and the fact that no clear statement of the uncertainties was given, led the decision to

Table 3 Datasets considered for the thermal conductivity of Pyrex 7740 as a function of temperature

First author	Refs.	Publ. year	Supplied by	Technique employed	Uncertainty quoted (%)	No. of data	Temp. range (K)	Form of data
Previous reference correlation/values								
Williams	[66]	1991	Reference correlation—CRM039		1.7 (2σ)	–	198–468	E
			Indicative values		na	–	148–198	E
Hulstrom	[65]	1985	Reference values		10.3 (2σ)	–	323–573	E
Powell	[63]	1966	Reference values		5.0	11	100–700	T
Primary data								
Assael	[37]	2015	Anter Corporation	THW	2.0	1	298	T
Ohmura	[70]	2011	na	GHP	5.0	4	282–333	D
Assael	[25]	2008	Anter Corporation	THW	1.0	7	314–432	T
Longo	[10]	2008	CRM 039	GHP	3.8 (2σ)	26	276–333	D
Assael	[23]	2004	Anter Corporation	THW	1.5	8	303–522	T
Gaal	[19]	2004	Anter Corporation	LF (TD)	2.0	1	293	T
Milano	[69]	2001	CRM 039	PTM	1.7	3	292–342	D
Hammerschmidt	[67]	2000	Corning	THB	2.5	36	194–573	D
Miller	[53]	1993	Corning	GHP	5.0	1	296	T
Log	[27]	1992	Corning	STHS	3.0	1	297	T
Matsumoto	[35]	1991	Corning	RHEM	5	21	303–353	D
Pillai	[71]	1991	Dynatech Corporation	GCL-HFM	2	21	327–742	D
Cahill	[33]	1990	na	3ω	na	50	33–501	D
Secondary data								
Cao	[38]	2016	na	3ω	6.0	1	298	T
Chen	[72]	2016	na	THW	na	1	300	T
Campbell	[73]	2013	Corning	LF	na	15	93–748	T
Tleoubaev	[74]	2005	na	GHP	na	2	298, 318	T

Table 3 (continued)

First author	Refs.	Publ. year	Supplied by	Technique employed	Uncertainty quoted (%)	No. of data	Temp. range (K)	Form of data
Zawilski	[75]	2001	na	DMA	na	89	46–299	D
Hisano	[76]	1998	na	TRAC	10–20	3	534–658	D
Fujishiro	[77]	1993	Corning	GHP (TD)	3.0	38	15–202	D
Himeno	[78]	1992	na	GHP	10	6	292–367	D
Yang	[79]	1992	Corning	ACCAL (TD)	na	45	20–310	D
Flynn	[64]	1963	na	–	5	7	273–573	T

3ω 3ω technique, *ACCAL* AC calorimetry, *DMA* Dynamic Measurement Access, *GHP* Guarded Hot Plate, *GCL-HFM* Guarded-Comparative-Longitudinal-Heat-Flow Method, *LF* laser flash, *PTM* pulse transient method, *RHEM* Radiative Heat Exchanger Method, *STHS* short transient hot strip, *TD* thermal diffusivity, *THB* transient hot bridge, *THF* transient heat flux, *THW* transient hot wire, *TRAC* thermal radiation calorimetry, *D* diagram, *E* equation, *T* tabulated experimental data, *na* not available
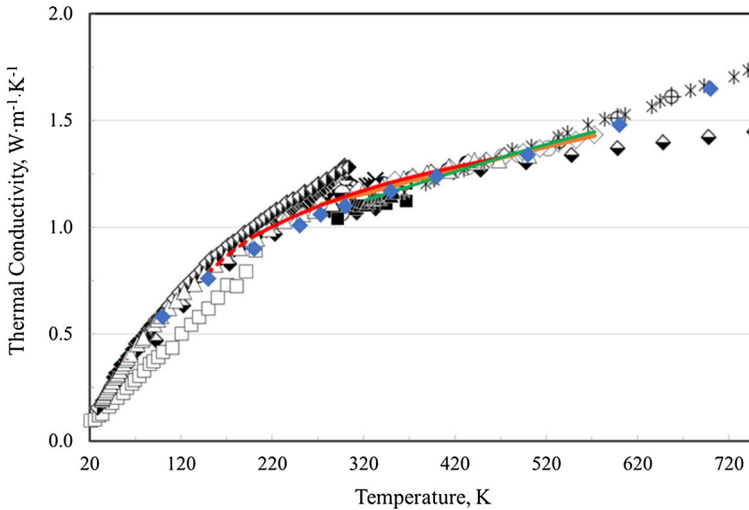
Fig. 8 Experimental thermal conductivity measurements for Pyrex 7740, as a function of the temperature. (⬡) Assael et al. [37], (◆) Ohmura [70], (●) Assael et al. [25], (x) Longo [10], (○) Assael et al. [23], (+) Gaal et al. [19], (◖) Milano et al. [69], (◇) Hammerschmidt and Sabuga [67], (⊕) Hisano and Placido [76], (◓) Miller and Kotlar [53], (◔) Log and Metallinou [27], (▲) Matsumoto and Ono [35], (✳) Pillai and George [71], (△) Cahill [33], (◖) Cao et al. [38], (◆) Chen et al. [72], (◆) Campbell et al.[73], (◑) Tleoubaev and Brezinski [74], (◀) Zawilski and Tritt [75], (□) Fujishiro et al. [77], (■) Himeno et al. [78], (◀) Yang et al. [79], (▬) Flynn [64], as well as the reference correlation (▬) and indicative values (▬ ▬) of Williams and Shawyer [66], the reference values of (▬) Hulstrom et al. [65], and the reference values of (◆) Powell et al. [63]

consider these data as secondary data. Finally, the single transient hot-wire measurement of Chen et al. [72], and the two modified guarded hot-plate measurements of Tleoubaev and Brezinski [74] were also included in the secondary data set, as they quoted no value for the uncertainty.

4.2 Discussion

The primary data for the thermal conductivity, λ (W·m^{-1}·K^{-1}) shown in Table 3, were fitted as a function of the absolute temperature, T (K), to Eq. 1. The coefficients c_i (W·m^{-1}·K^{-1}) are shown in Table 7, while recommended values are shown in Table 8.

As shown in Table 7, in the case of Pyrex 7740, the AAD is 1.4 % and the BIAS 0.0 %. The uncertainty (95 % confidence level) over the whole temperature range, 32 K to 742 K, is 3.7 %. As however, below 190 K, the correlation is only based on the values of Cahill [33], it would be wiser to assign an uncertainty of not less than 10 % in that temperature range. Even at temperatures above 573 K, the data should be considered with a higher uncertainty toward lower values, i.e., 10 %, since only few data are available and a detailed quantitative discussion of radiant heat transfer is still missing. Figure 10 shows the percentage deviations of the primary thermal conductivity data of Pyrex 7740 from those calculated by Eq. 1, as a function of the
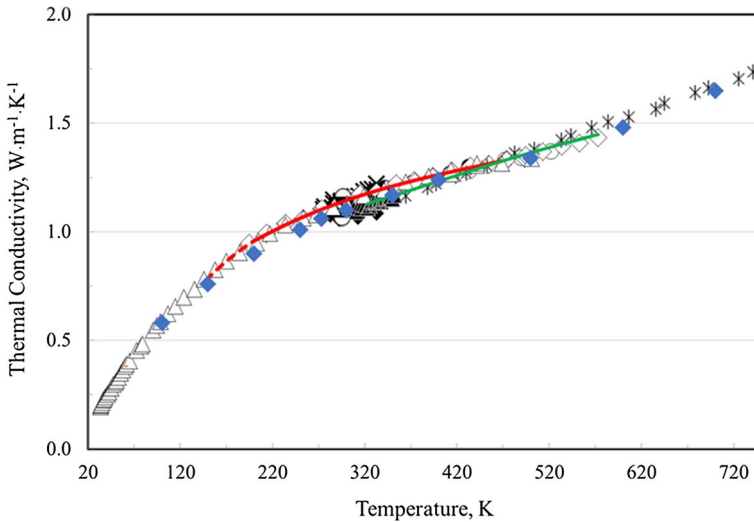
Fig. 9 Primary experimental thermal conductivity measurements for Pyrex 7740, as a function of the temperature. (○) Assael et al. [37], (◆) Ohmura [70], (●) Assael et al. [25], (x) Longo [10], (○) Assael et al. [23], (+) Gaal et al. [19], (◐) Milano et al. [69], (◇) Hammerschmidt and Sabuga [67], (◒) Miller and Kotlar [53], (◓) Log and Metallinou [27], (▲) Matsumoto and Ono [35], (✸) Pillai and George [71], (△) Cahill [33], as well as the reference correlation (▬) and indicative values (▬ ▬) of Williams and Shawyer [66], the reference values of (▬) Hulstrom et al. [65], and the reference values of (◆) Powell et al. [63]

temperature. It can be seen that the new reference correlation represents well within the mutual uncertainties, the previous reference correlation proposed by Williams and Shawyer [66] and Hulstrom et al. [65], the reference values proposed by Powell et al. [63], and all primary data.

5 Pyroceram 9606

Pyroceram 9606 is an opaque glassy ceramic with high strength and elastic modulus and an operating temperature covering the range 75 K to 1250 K. It is mechanically stable to 1450 K [80]. The polycrystalline ceramic consists primarily of silica (56 %), aluminum oxide (19.6 %), magnesium oxide (14.9 %), titanium oxide (8.6 %), and other (0.9 %) [81]. The density is 2.60×10^3 kg·m^{-3} at 293 K [63]. Originally, it was developed by Corning Glass Works (now Corning Inc.) in the 50s. It is particularly well defined and thermally stable, and it was proposed as a standard reference material for thermal conductivity by the National Institute of Standards and Technology (NIST), USA, in 1966 [63], and with lower uncertainty in a more restricted temperature range, in 1985 [65].

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 1025 K [80]. This certification was
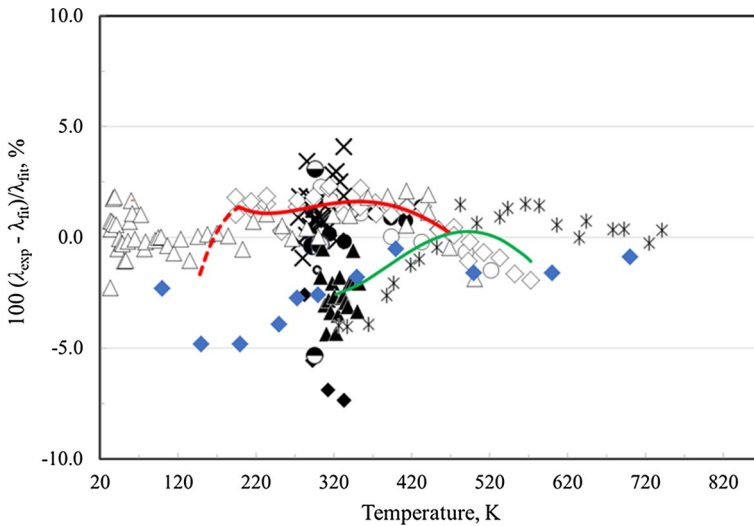
Fig. 10 Percentage deviations of the primary experimental thermal conductivity measurements for Pyrex 7740, from those calculated by Eq. 1, as a function of the temperature. (○) Assael et al. [37], (◆) Ohmura [70], (●) Assael et al. [25], (x) Longo [10], (○) Assael et al. [23], (+) Gaal et al. [19], (◐) Milano et al. [69], (◇) Hammerschmidt and Sabuga [67], (◑) Miller and Kotlar [53], (◗) Log and Metallinou [27], (▲) Matsumoto and Ono [35], (✳) Pillai and George [71], (△) Cahill [33], as well as the reference correlation (━━) and indicative values (■ ■) of Williams and Shawyer [66], the reference values of (━━) Hulstrom et al. [65], and the reference values of (◆) Powell et al. [63]

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). The uncertainty of the certified thermal conductivity value was ±6.5 %, while that of the thermal diffusivity was ±6.1 %.

5.1 Data Compilation

In Table 4, and Fig. 11, all published thermal conductivity measurements, to our knowledge, of Pyroceram 9606 are shown. In the same table and in the figure, the aforementioned 2007-reference correlation of Salmon et al. [80], the 1985-reference correlation of Hulstrom et al. [65], as well as the 1966-reference values proposed by Powell et al. [63], are also shown.

As previously we included in the primary dataset the measurements of Assael et al. [20, 21, 37] performed in a transient hot-wire instrument, with two Ta wires embedded in a silicone layer squeezed between two samples of the solid, to reduce contact effects. These measurements were obtained with an absolute uncertainty of better than 2 %. The transient plane source measurements of Feng and Li [82], obtained with a 1.8 % uncertainty, and the laser-flash diffusivity measurements of Gaal et al. [19], obtained with 3 % uncertainty, were also included in the primary dataset. Moreover, we included in the primary dataset, the guarded hot-plate

Table 4 Datasets considered for the thermal conductivity of Pyroceram 9606 as a function of temperature

First author	Refs.	Publ. year	Supplied by	Technique employed	Uncertainty quoted (%)	No. of data	Temp. range (K)	Form of data
Previous reference correlation/values								
Salmon	[80]	2007	Reference correlation—BCR 724		6.5 (2σ)		298–1025	E
Hulstrom	[65]	1985	Reference values		5.6 (2σ)	–	323–573	E
Powell	[63]	1966	Reference values		5–10	18	100–1400	T
Primary data								
Feng	[82]	2017	na	TPS	1.8	1	293	T
Assael	[37]	2015	Corning Inc.	THW	2.0	1	298	T
Salmon	[85]	2010	Interlaboratory AvVal		na	12	298–1273	T
Assael	[21]	2008	Corning Inc.	THW	1.0	6	314–414	T
Gaal	[19]	2004	Corning Inc.	LF (TD)	3.0 (2σ)	10	373–1274	T
Assael	[20]	2003	Corning Inc.	THW	1.5	15	298–570	T
Filla	[83]	1997	na	GHP	5.0	14	406–781	D
Matsumoto	[84]	1992	na	RHEM	3.5	–	293–393	E
Secondary data								
Cao	[38]	2016	na	3ω	6.0	1	298	T
Tleoubaev	[74]	2005	na	GHP	na	2	298, 318	T
Zawilski	[75]	2001	na	DMA	na	126	25–304	D
Filla	[11]	1999	na	GHP	5.0	15	403–1073	T
Hisano	[76]	1998	na	TRAC	10–20	50	699–863	D
Suliyanti	[86]	1995	na	LF (TD)	3.0	–	300–1200	E
Gustafsson	[28]	1991	Corning Inc.	TPS	na	4	295–473	T
Ohta	[87]	1991	Corning Inc.	LF (TD)	na	72	302–977	D
Cahill	[33]	1990	na	3ω	na	44	32–711	D

3ω 3ω technique, *DMA* Dynamic Measurement Access, *GHP* Guarded Hot Plate, *LF* laser flash, *RHEM* Radiative Heat Exchanger Method, *THW* transient hot wire, *TPS* transient plane source, *TRAC* thermal radiation calorimetry, *D* diagram, *E* equation, *T* tabulated experimental data, *na* not available
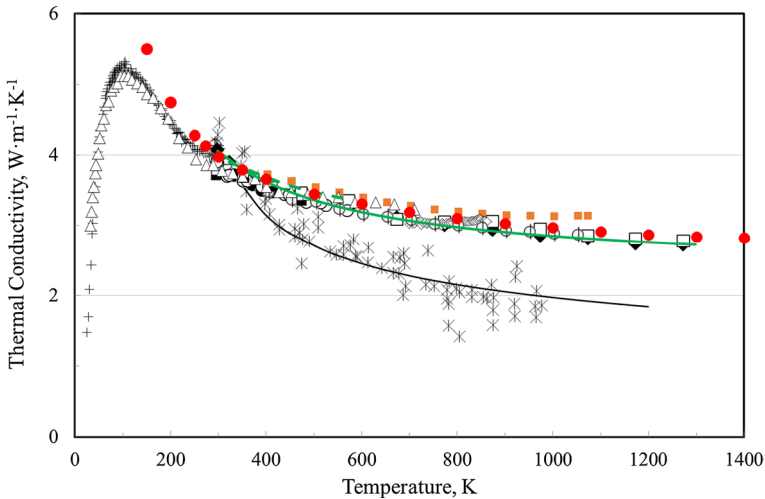
Fig. 11 Experimental thermal conductivity measurements for Pyroceram 9606 as a function of the temperature. ($\oplus$) Feng and Li [82], ($\bigcirc$) Assael et al. [37], ($\blacklozenge$) Salmon et al. [85], ($\bullet$) Assael et al. [21], ($\square$) Gaal et al. [19], ($\circ$) Assael et al. [20], ($\blacksquare$) Filla and Slifka [83], (- -) Matsumoto and Ono [35], ($\blacksquare$) Cao et al. [38], ($\ominus$) Tleoubaev and Brezinski [74], (+) Zawilski and Tritt [75], (Φ) Filla [11], ($\diamondsuit$) Hisano and Placido [76], (—) Suliyanti et al. [86], ($\blacksquare$) Gustafsson [28], ($\ast$) Ohta et al. [87], ($\triangle$) Cahill [33], (——) Salmon et al. [80], ($\blacksquare$ $\blacksquare$) Hulstrom et al. [65], ($\bullet$) Powell et al. [63]

measurements of Filla [83], and the radiative heat exchange measurements of Matsumoto and Ono [84], obtained with 5 and 3.5 % uncertainty. Finally, in the same table and figure, we showed the interlaboratory average values of the thermal conductivity of Pyroceram 9606, published by Salmon et al. [85].

In the secondary dataset, we allocated all measurements with uncertainty higher than 5 %, or no quoted uncertainty. We also included the laser-flash measurements of Suliyanti et al. [86] as they were distinctively lower than most other measurements. The data by Filla et al. [11] represent mean values derived by GHP measurements on different thick specimens. The derived values of thermal conductivity increase slightly with specimen thickness due to the influence of radiative heat transfer (cf. 2.1).

In Fig. 12, the percentage deviations of the primary thermal conductivity measurements from the reference correlation of 2007 proposed by Salmon et al. [80] as a function of temperature are shown. It is apparent that the associated uncertainty of 6.5 % at the 95 % confidence level can be improved.

5.2 Discussion

The primary data for the thermal conductivity, λ (W·m^{-1}·K^{-1}), shown in Table 4, were fitted as a function of the absolute temperature, T (K), to Eq. 1. The coefficients c_i (W·m^{-1}·K^{-1}) are shown in Table 7.
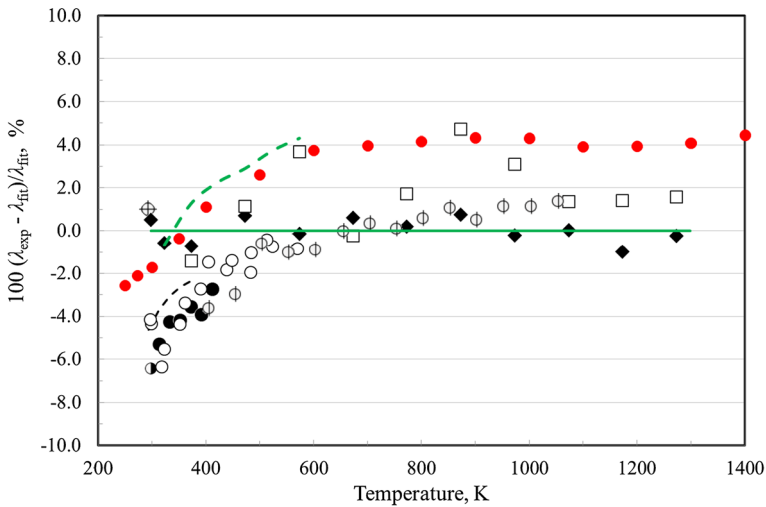
Fig. 12 Percentage deviations of the primary experimental thermal conductivity measurements for Pyroceram 9606, from those calculated by Salmon et al. 2007 [80] reference correlation, as a function of the temperature. (⊕) Feng and Li [82], (◑) Assael et al. [37], (◆) Salmon et al. [85], (●) Assael et al. [21], (□) Gaal et al. [19], (○) Assael et al. [20], (- -) Matsumoto and Ono [35], (φ) Filla [11], (▬) Salmon et al. [80], (▪ ▪) Hulstrom et al. [65], (●) Powell et al. [63]

As shown in Table 7, in the case of Pyroceram 9606, the AAD is 1.3 % and the BIAS − 0.4 %. The uncertainty (95 % confidence level) over the temperature range, 290 K to 1275 K, is 3.5 %. Figure 13 shows the percentage deviations of the primary thermal conductivity data of Pyroceram 9606 from those calculated by Eq. 1, as a function of the temperature. It can be seen that the new reference correlation represents well within the mutual uncertainties, the previous reference correlation proposed by Salmon et al. [80], the correlation of Hulstrom et al. [65], the reference values of Powell et al. [63], and all primary data. Furthermore it represents everything with the uncertainty of 3.5 % (at the 95 % confidence level) which is much better than the 6.5 % uncertainty of the previous reference correlation [80].

6 SS304

SS304 is a low-carbon member of the 18-8 type austenitic stainless steel [88], with a slightly higher chromium content for improved corrosion resistance. This steel is susceptible to intergranular corrosion in the temperature range 700 K to 1150 K, due to carbide precipitation. Its density is 8000 kg·m^{-3} at 293 K [88]. The 304L is a still lower carbon version of the 304 steel [88], with improved resistance to intergranular corrosion. The small difference of carbon content between SS304L and SS304 (see Table 5) is judged to be insignificant in altering the electrical and thermal properties [89].
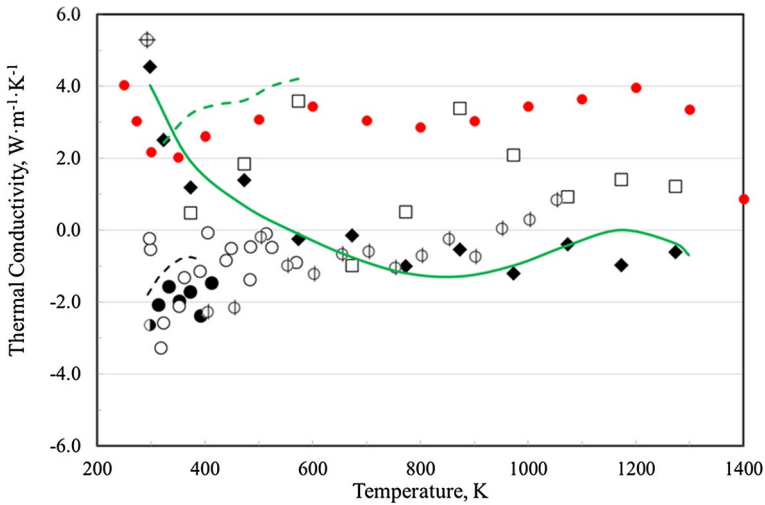
Fig. 13 Percentage deviations of the primary experimental thermal conductivity measurements for Pyroceram 9606, from those calculated by Eq. 1, as a function of the temperature. (⊕) Feng and Li [82], (◑) Assael et al. [37], (◆) Salmon et al. [85], (●) Assael et al. [21], (□) Gaal et al. [19], (○) Assael et al. [20], (- -) Matsumoto and Ono [35], (⬦) Filla [11], (━━) Salmon et al. [80], (▪ ▪) Hulstrom et al. [65], (●) Powell et al. [63]

6.1 Data Compilation

In 1977, Chu and Ho [89] examined 20 thermal conductivity sets of SS304 and seven sets for SS304L. These showed that the thermal conductivity of the two steels was virtually the same. The values they recommended covered a temperature range from 1 K to 1672 K (melting point) with an uncertainty of 5 %. Based mostly on the same datasets, in 1985 Bogaard [90] presented also a review paper on the thermal conductivity of SS304 between 100 K and 1707 K, with an uncertainty of 4 %. Since the values proposed by Bogaard cover an extensive temperature range with a good uncertainty, it

Table 5 Composition (mass%) of SS304 and SS304L [88]	AISI/ASTM 304 EN X2CrNi18010 EN 1.4301	AISI/ASTM 304L EN X2CrNi18010 EN 1.4301
C	≤ 0.08	≤ 0.03
Si	≤ 1.00	≤ 1.00
Mn	≤ 2.00	≤ 2.00
P	≤ 0.045	≤ 0.045
S	≤ 0.015	≤ 0.015
Ni	8.00–10.50	8.00–10.50
Cr	17.50–19.50	17.50–19.50
Fe	Makes up the rest	Makes up the rest

is interesting to investigate more recent measurements as to whether any improvement is possible.

In an attempt to investigate a slight anomalous infection of the thermal conductivity values reported by Bogaard [90] between 300 K and 500 K, Graves et al. [91], in 1991, measured the thermal conductivity of AISI 304L steel and found no such behavior. Graves employed two instruments: (a) a high-temperature longitudinal heat transfer instrument in Oak Ridge National Laboratory with a 1.5 % uncertainty (rising to 3 % over 700 K), and (b) a laser-flash apparatus of 1.5 % uncertainty. These measurements were part of the primary datasets. Assael et al. [22] employed an absolute 2-Ta wires transient hot wire in a thin silicon paste, and squeezed between two samples of the solid, to measure the thermal conductivity with an uncertainty of 1.5 %. These measurements were also included in the primary dataset.

As we are only interested in investigating the possibility of improving the uncertainty of the Bogaard et al. [90] reference correlation, we only considered in the primary dataset, measurements that are characterized by an uncertainty of less than 4 %. Only two other such sets are available to our knowledge. The measurement of Takahashi et al. performed in a steady-state direct electric heating instrument over the range 293 K to 449 K with an uncertainty of 3.6 % and the measurement of Blackwell obtained in an axial heat conduction instrument at 304 K with an uncertainty of 4 %. Hence, these two sets were also included in the primary dataset. The rest of the measurements of higher or no available uncertainty are included in the secondary dataset.

Table 6 shows all measurements of the thermal conductivity of SS304. These are also shown in Fig. 14.

6.2 Discussion

The primary data for the thermal conductivity, λ (W·m^{-1}·K^{-1}) shown in Table 6, were fitted as a function of the absolute temperature, T (K), to Eq. 1. Only the range 293 K to 546 K was employed as in this range there are at least two datasets. The coefficients c_i (W·m^{-1}·K^{-1}) are shown in Table 7.

As shown in Table 7, in the case of SS304, the AAD is 1.0 % and the BIAS -0.5 %. The uncertainty (95 % confidence level) over the restricted temperature range, 293 K to 546 K, is 2.7 %. Figure 15 shows the percentage deviations of the primary thermal conductivity data of SS304 from those calculated by Eq. 1, as a function of the temperature. It can be seen that in this restricted range, the new reference correlation represents well within the mutual uncertainties, the previous reference values proposed by Bogaard et al. [90], those of Chu and Ho [89], and all primary data. Furthermore, it represents everything with the uncertainty of 2.7 % (at the 95 % confidence level) which is much better than the 4 % uncertainty of the previous reference correlation [90].

Table 6 Datasets considered for the thermal conductivity of SS304 as a function of temperature, at 0.1 MPa

First author	Ref.	Publ. year	Supplied by	Technique employed	Uncertainty quoted (%)	No. of data	Temp. range (K)	Form of data
Previous reference correlation/values								
Bogaard	[90]	1985	Reference values		4.0	24	100–1707	T
Chu	[89]	1977	Reference values		5.0	33	1–1672	T
Primary data								
Assael	[22]	2003	Anter Corporation	THW	1.5	11	306–546	T
Blackwell	[92]	2000	na	AHC	4.0	1	304	T
Graves	[91]	1991	Jessop Steel Co.	HTL	1.5–3.0	13	333–976	T
			Jessop Steel Co.	LF (TD)	1.5	13	297–423	T
Takahashi	[36]	1983	na	SSDEH	3.6	24	293–449	D
Secondary data								
Dongmei	[93]	2017	na	LPM (TD)	9.0	22	30–282	T
Hadi	[94]	2017	na	GHF	10.0	1	323	T
Qiu	[56]	2011	na	3ω	6.8	1	298	T
Rempe	[95]	2008	na	LF (TD)	na	142	350–1426	D
Yao	[96]	2005	na	LRH	na	–	400–630	E
Sweet	[97]	1987	na	CFD (TD)	5.0	28	324–837	D

3ω 3ω technique, *AHC* axial heat conduction, *CFD* comparative and flash diffusivity, *GHF* guarded heat flow, *HTL* high-temperature longitudinal, *LF* laser flash, *LRH* laser rapid heating, *LPM* laser photothermal method, *SSDEH* steady-state direct electric heating, *TD* thermal diffusivity, *THW* transient hot wire, *D* diagram, *E* equation, *T* tabulated experimental data, *na* not available
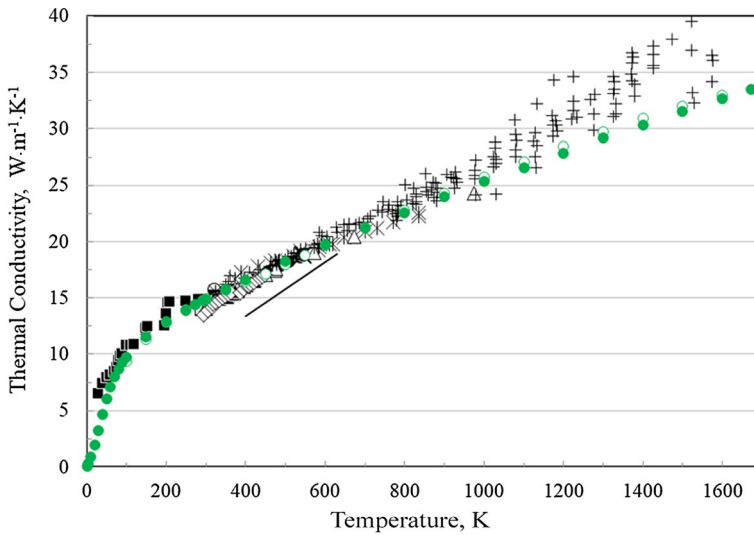
Fig. 14 Experimental thermal conductivity measurements for SS304, as a function of the temperature. (●) Assael and Gialou [22], (x) Blackwell et al. [92], (△) HTL, (▲) LF Graves et al. [91], (◇) Takahashi et al. [36], (■) Dongmei et al. [93], (+) Rempe and Knudson [95], (◓) Hadi et al. [94], (□) Qiu et al. [56], (—) Yao [96], (✱) Sweet et al. [97], as well as the reference values of (○) Bogaard [90], and the reference values of (●) Chu [89]

Table 7 Coefficients of recommended reference correlation for the thermal conductivity (Eq. 1), temperature range of applicability and associated uncertainties

	BK7	PMMA	Pyrex 7740	Pyroceram 9606	SS304
c_0 (W·m^{-1}·K^{-1})	−0.090 725 69	0.070 517 30	−0.063 949 789	6.021 828	3.232 264 61
c_1 (W·m^{-1}·K^{-1})	1.996 151	0.264 756 574	2.364 448 35	−3.034 446 57	23.693 573 7
c_2 (W·m^{-1}·K^{-1})	−1.185 182	−0.143 002 702	−1.779 834 23	1.247 200 06	−21.444 751
c_3 (W·m^{-1}·K^{-1})	0.319 537 9	−0.037 964 414	0.653 987 846	−0.245 199 53	10.258 952 4
c_4 (W·m^{-1}·K^{-1})	−0.028 831 42	0.037 034 680	−0.084 597 452	0.018 225 42	−1.752 379 7
T_{range} (K)	79–773	95–363	32–742	290–1275	293–546
AAD (%)	2.3	1.8	1.4	1.3	1.0
BIAS (%)	0.0	0.3	0.0	−0.4	−0.5
Uncertainty (95 % conf.)	5.2[a]	4.5	3.7[b]	3.5	2.7

[a] The value of 5.2 %, increases to 11.6 % over the temperature range 79–233 K, and drops to 4.3 % over the temperature range 273–773 K

[b] The value of 3.7 %, increases to 10 % at temperatures below 190 K, or higher than 573 K

7 Recommended Values

As already described, Table 7 shows for the five solids examined, the coefficients c_i of the recommended thermal conductivity reference correlations according to Eq. 1,
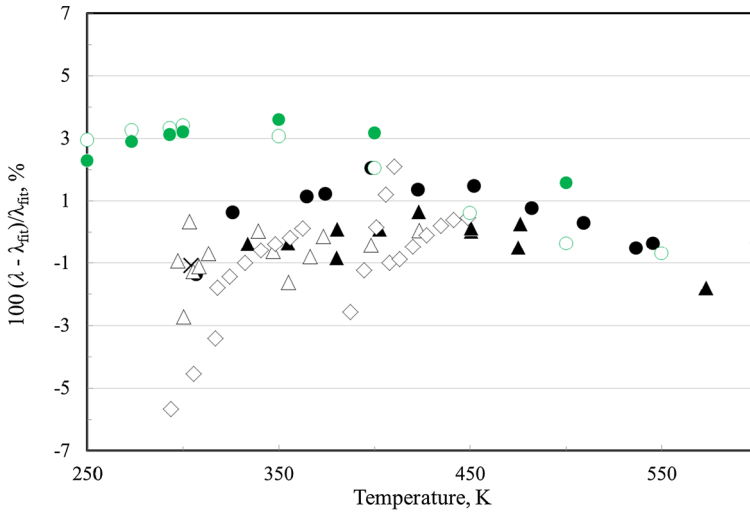
Fig. 15 Percentage deviations of the primary experimental thermal conductivity measurements for SS304, from those calculated by Eq. 1, as a function of the temperature. (●) Assael and Gialou [22], (**x**) Blackwell et al. [92], (△) HTL, (▲) LF Graves et al. [91], (▲) Takahashi et al. [36], (**+**), as well as the reference values of (◇) Bogaard [90], and the reference values of (●) Chu [89]

Table 8 Recommended values for BK7, PMMA, and Pyrex 7740

BK7		PMMA		Pyrex 7740	
T (K)	λ (W·m^{-1}·K^{-1})	T (K)	λ (W·m^{-1}·K^{-1})	T (K)	λ (W·m^{-1}·K^{-1})
79	0.395	95	0.144	32	0.190
100	0.496	100	0.147	50	0.313
150	0.698	150	0.170	100	0.594
200	0.853	200	0.183	150	0.798
250	0.968	250	0.190	200	0.946
293.15	1.043	293.15	0.192	250	1.051
300	1.053	300	0.192	293.15	1.120
350	1.116	350	0.195	300	1.129
400	1.162	363	0.196	350	1.191
450	1.198			400	1.246
500	1.228			450	1.302
550	1.258			500	1.362
600	1.291			550	1.429
650	1.329			600	1.504
700	1.376			650	1.584
750	1.431			700	1.665
773	1.459			742	1.728

its temperature range, T_{range}, of application, for each solid, as well as the AAD, the BIAS and the uncertainty at the 95 % confidence level of each correlation.

In Tables 8 and 9, recommended values of the thermal conductivity calculated from Eq. 1 and corresponding coefficients in Table 7, for each solid, are shown.

8 Conclusion

As described in this work, as a result of a careful examination of existing measurements, following conclusions were reached:

– In the case of BK7, a new reference correlation for its thermal conductivity is proposed. Its uncertainty (at the 95 % confidence level) over the whole temperature range, 79 K to 773 K, is 5.2 % (AAD is 2.3 % and BIAS is 0.0 %), dropping to 4.3 % over the restricted temperature range 273 K to 773 K. In the temperature range 79 K to 233 K, an uncertainty of 11.6 % should be employed because of the underlying uncertainties of the primary data in this range.

Table 9 Recommended values for Pyroceram 9606 and SS304

Pyroceram 9606		SS304	
T (K)	λ (W·m^{-1}·K^{-1})	T (K)	λ (W·m^{-1}·K^{-1})
290	3.94	293.15	14.32
293.15	3.92	300	14.43
300	3.90	350	15.24
350	3.71	400	16.10
400	3.57	450	17.03
450	3.45	500	18.00
500	3.35	546	18.87
550	3.27		
600	3.20		
650	3.14		
700	3.10		
750	3.05		
800	3.01		
850	2.98		
900	2.94		
950	2.91		
1000	2.87		
1050	2.84		
1100	2.81		
1150	2.78		
1200	2.76		
1275	2.75		

- An improved extended reference correlation is proposed for PMMA, with an uncertainty (at the 95 % confidence level) of 4.4 %, over the whole temperature range, 95 K to 363 K (AAD is 1.8 % and BIAS is 0.3 %).
- In the case of Pyrex 7740, a new reference correlation for its thermal conductivity is proposed with AAD 1.4 % and BIAS 0.0 %. Its uncertainty (at the 95 % confidence level) over the whole temperature range, 32 K to 742 K, is 3.7 %. Below 190 K, the correlation is only based on the values of Cahill [33]. Therefore, an uncertainty of not less than 10 % is assigned in that temperature range. At temperatures above 573 K, the data should be considered with a higher uncertainty toward lower values, i.e., 10 %, since only few data are available and a detailed quantitative discussion of radiant heat transfer is still missing.
- A lower uncertainty reference correlation is proposed for Pyroceram 9606 as actually stated in literature. Over the temperature range 290 K to 1275 K, the uncertainty (at the 95 % confidence level) is 3.5 % (AAD is 1.3 % and BIAS is −0.4 %).
- A lower uncertainty reference correlation but in a more restricted temperature range is proposed for the thermal conductivity of SS304. Over the temperature range 293 K to 546 K, the uncertainty at the 95 % confidence level is 2.7 % (AAD is 1.0 % and BIAS is −0.5 %).

This work provides reference correlations for thermal conductivity, which are helpful to improve the quality of thermal conductivity measurements on solids. It also shows in which areas there is still room for improvement, e.g., to increase measurements at low temperatures. Low-uncertainty measurements (<4%) are certainly required at temperatures lower than 233 K for BK7, lower than 200 K for PMMA, lower than 190 K for Pyrex 7740, and lower than 290 K for Pyroceram 9604 and SS304. Finally, the present work indicated in which cases derived thermal conductivity values need to be carefully analyzed (e.g., measurements on semitransparent glasses at higher temperatures).

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