ORIGINAL PAPER

Verification of thermal conductivity measurements using guarded hot plate and heat flow meter methods

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Abstract

The guarded hot plate (GHP) and the heat flow meter (HFM) methods are two widely used techniques to measure the thermal conductivity (λ) of insulation specimens. In this study, the λ measurement of the two methods was validated using the certified reference material (CRM) IRMM-440 and an expanded polystyrene (EPS) board at temperatures from − 10 to 50 °C. The GHP apparatus was a commercially available apparatus for 300 mm × 300 mm specimens, and the HFM apparatus for 900 mm × 900 mm specimens was built in-house. The λ of the CRM measured using the GHP was within 2% of the certified λ value. Subsequently, the λ values of specimens of nominally identical EPS with 20 mm thicknesses were measured using both the GHP and the HFM methods. A comparison of the λ measurements showed that the two methods were consistent to within 3%. Finally, the λ values of EPS specimens of various thicknesses from 50 to 200 mm were measured using the HFM method. Although the specimens were nominally identical, values of λ for the specimens with thicknesses of 20−150 mm were increased by 0.09% mm^{-1} at − 10 °C and by 0.07% mm^{-1} at 10 °C.

Keywords Thermal conductivity · Guarded hot plate · Heat flow meter · Certified reference material · Expanded polystyrene

1 Introduction

In an effort to save energy and improve the thermal efficiency of buildings, the use of an insulator with good thermal performance and optimal thickness is important when constructing industrial, commercial, and residential buildings. Many related studies on the design of insulation materials and the analysis of thermal performance have been reported [1–5]. Thus, valid tests of the thermal properties of insulators are important to properly obtain various thermal properties of building materials [6, 7]. The most common methods for measuring the thermal conductivity and the thermal resistance for insulators are using a guarded hot plate (GHP) or a heat flow meter (HFM). International standards, such as ISO 8302 and ASTM C177 for GHP and ISO 8301 and ASTM C518 for HFM, contain details of the test methods and procedures for thermal conductivity measurements that have been used over the past 20 years.

Figure 1 shows schematic diagrams of the principles of the GHP and the HFM methods. The two methods represented by the apparatus are widely used to measure the thermal conductivity of insulation materials such as bricks and glass with thermal conductivities below 1.5 W m^{-1} K^{-1}. Among many different realizations of GHP and HFM, Fig. 1a and b shows the double-specimen type for the GHP and the single-specimen type for the HFM in this study.

In the GHP method, the amount of heat P in the metering section is measured, where P is the required heat to maintain the temperature difference ΔT between the hot and the cold plates. The thermal conductivity (λ) is obtained as follows [8, 9]:

$$\lambda = \frac{Pd}{2A\Delta T}, \tag{1}$$

where A is the area of the metering section, d is the average specimen thickness, and the factor 2 in the denominator is due to the double-specimen structure of the apparatus.

With the HFM method, the amount of heat flux across a specimen is measured when the two sides of the specimen

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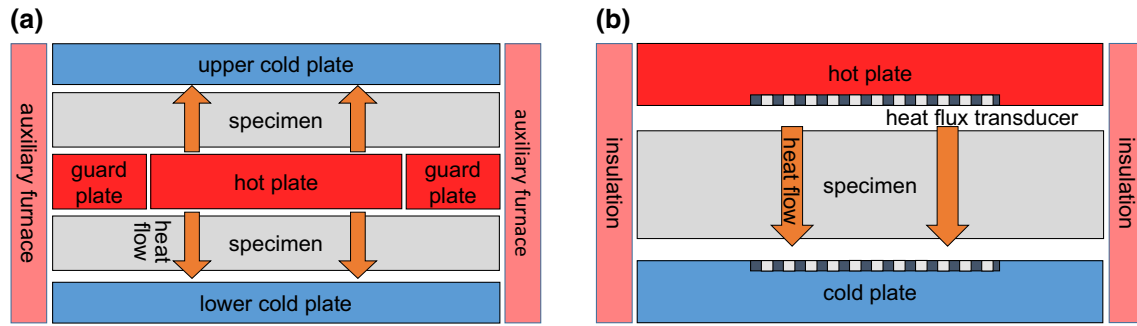   Research Institute of Standards and Science, Daejeon 34113,
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Fig. 1 Schematic diagrams of the principles of the **a** GHP and the **b** HFM methods

(of thickness d) are maintained at a temperature difference ΔT. The heat flux is measured by using calibrated heat flux transducers for which the voltage output V is proportional to the heat flux across the transducer. The thermal conductivity can be obtained from the HFM measurement as follows [10, 11]:

$$\lambda = \frac{Vd}{S\Delta T} \tag{2}$$

where S (unit: V m^2 W^{-1}) is the sensitivity of the transducer determined via a calibration procedure using a certified reference material (CRM) with a known thermal conductivity value.

Because the GHP method is a primary measurement in the context that it does not require a CRM with a known thermal conductivity, its traceability is from the SI units of electrical power (watt), length (meter), and temperature (kelvin). The commercially built GHP apparatuses that many test laboratories use comprise a user-friendly integration of hardware and software. Therefore, users have difficulty controlling or accessing each component of the parameters in the measurement, which makes evaluating the uncertainty of the λ measurements difficult for the users, most of whom accept the accuracy of the apparatus from the specifications (normally a 1–2% accuracy in measurements under optimal measurement conditions). Meanwhile, the HFM is a secondary measurement method for the thermal conductivity, where the measurement traceability comes from the calibrated sensitivity of the heat flux transducers. Therefore, it requires an insulation material with a known thermal conductivity, such as a CRM.

In this study, we linked the measurements from the GHP and the HFM apparatuses to verify the λ measurements. The GHP measurement was first verified within its relative uncertainty of 2% using a CRM with a known λ. Then, an insulation material commonly used in buildings was measured using both methods to compare the results

of the thermal conductivity measurements. The maximum difference between the results of the two methods for the specimens of identical thicknesses was less than 3%. The HFM measurements on insulation specimens with various thicknesses showed temperature- and thickness-dependent results that were consistent with results previously published by others. This verification process can give confidence in the measurements of λ and the assigned uncertainties for both GHP and HFM measurements by checking the internal consistency of the measurements from the two apparatuses and the agreement with values in the existing literature.

2 Experimental

2.1 GHP and HFM apparatus

In this study, we used one GHP and one HFM apparatus. Figure 2 shows photographs of the two apparatuses, and Table 1 summarizes their physical characteristics. The GHP apparatus in this work was the commercially available GHP 456 Titan (Netzsch, Germany). The apparatus can operate with a mean specimen temperature range of − 160 to 250 °C and can accommodate a pair of specimens with dimensions of 300 mm × 300 mm. The expanded uncertainty (with expansion factor $k = 2$) of the measured λ from the specification of the apparatus is 2%. (The uncertainty throughout this paper is the expanded uncertainty when $k = 2$.)

Figure 3 shows an example of data obtained from the measurement of λ using the GHP apparatus. The operation starts with the stabilization of the temperature by circulating liquid nitrogen and powering up the heaters in the heater plates. Under most measurement conditions, the temperature stabilizes to the target value in the order of the guard plate, lower cold plate, and upper cold plate, at which point the hot plate temperature is precisely controlled. The temperature was obtained using 29 platinum resistance thermometers (PRTs) at various positions on the plates (including 10 PRTs
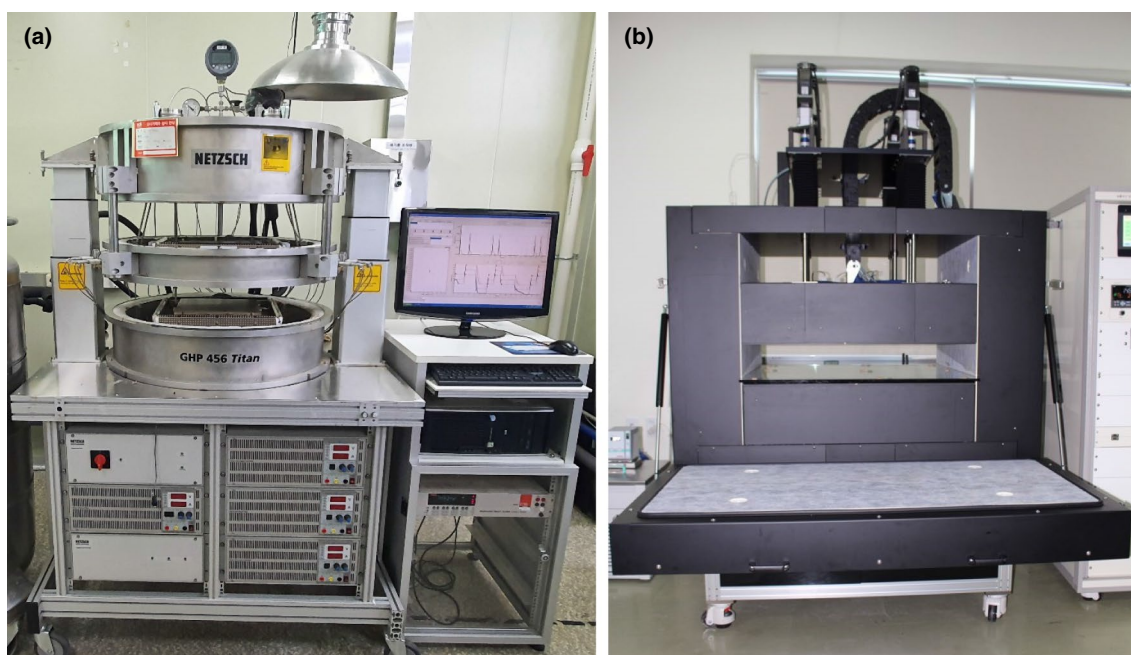
Fig. 2 Photographs of the **a** guarded hot plate and **b** the heat flow meter apparatus in this work

Table 1 Physical characteristics of the guarded hot plate (GHP) and the heat flow meter (HFM) apparatus in this work

Attribute	GHP	HFM
Temperature range (°C)	− 160 to 250 °C	− 10 to 70 °C
Measurement type	Double specimen	Single specimen
Specimen dimensions	300 mm × 300 mm	900 mm × 900 mm
Metering section	150 mm × 150 mm	300 mm × 600 mm × 2 sides
Heater plate	Aluminum	Aluminum
Temperature sensor	29 PRTs[a]	10 PRTs[a]
Cooling method	Liquid nitrogen	Circulated bath
Secondary guard	Auxiliary furnace	Insulation

[a]Platinum resistance thermometers

Fig. 3 Temperatures at different parts of the GHP during measurements at 20, 30, and 40 °C with $\Delta T = 20$ K. Inset: stabilized thermal conductivity acquired from the apparatus at 20 °C (HP: hot plate; GP: guard plate; UCP: upper cold plate; LCP: lower cold plate; AF: auxiliary furnace)

on the hot plate), where readings were acquired every minute. The measurement of λ is completed when the standard deviations of the mean hot plate temperature, heater power at the hot plate, and λ are less than 0.01 K, 0.005 W, and 0.001 W m^{-1} K^{-1}, respectively, for the data during the last 1 h. The measurement at one mean specimen temperature takes approximately 3 h for the first set and 2 h for subsequent ones.

The in-house-built HFM apparatus in this study can accommodate a large-area specimen of 900 mm × 900 mm to measure λ of thick insulation materials [12]. The details of the apparatus and the uncertainty assessment are shown in our previous publication [12]. The uncertainty in the
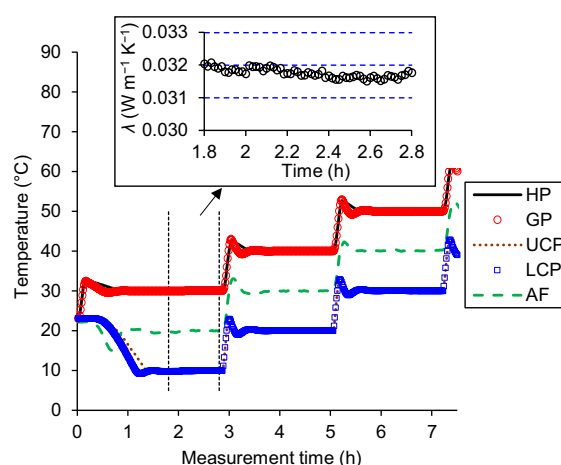
thermal conductivity measurements evaluated using CRM IRMM-440 is 1.4% in the temperature range from − 10 to 50 °C. The measurement procedure for the HFM apparatus is similar to that for the GHP apparatus. The temperatures of the various plates are controlled by circulating a liquid (a mixture of water and ethylene glycol) via a thermostat, which is temperature-controlled from − 20 to 80 °C. The temperatures of the hot and the cold plates (i.e., the two faces of the specimen) are measured using 5 PRTs on each

plate. The temperature and heat flux are measured every 5 s, and the mean values of the measurements over 5 min are recorded. The measurement of λ is complete when the relative standard deviation of the measured λ values for at least 1 h is less than 0.5%. The measurement of one mean specimen temperature takes approximately 10 h (including the time for the initial temperature stabilization), and 7 h are required for each subsequent measurement temperature. The time for temperature stabilization is longer than that for the GHP because of the large dimensions of both the HFM apparatus and the specimen. For precise measurements, the λ value for only one or two mean specimen temperatures was measured each day.

Figure 4 summarizes the traceability chain of the two apparatuses and the verification scheme in this work. The traceability aspects of the two apparatuses are independent of each other (the traceability of GHP from other physical quantities and HFM from a CRM), as depicted by the two rectangles with dashed boundaries in Fig. 4. As reported in Sects. 3.1 and 3.2, two specimens were used to link and verify the measurement results of the two methods.

2.2 The IRMM-440 CRM and EPS board

Two specimens were used to measure the λ in this work. The first is IRMM-440, which is a thermal conductivity CRM made of a resin-bonded glass fiber board [13]. In our previous work, this specimen was cut to dimensions of 900 mm × 900 mm and used to calibrate the heat flux transducers of the HFM to provide traceability for the measurement [12]. In this work, two nominally identical specimens of IRMM-440 were cut to 300 mm × 300 mm for the λ measurement via the GHP. The thickness of the specimen was 35 mm, and the densities of the two specimens were 69 and 77 kg m^{-3}. The certified value of λ for the CRM is a quadratic function of temperature, and the value at 20 °C is 0.031 597 W m^{-1} K^{-1}.

The reported relative uncertainty of λ of the IRMM-440 CRM is 1.0% at − 10 ℃ and 0.8% at 50 ℃. The specimens were dried at 60 °C for 24 h in an oven and subsequently weighed. Then, the specimens were soaked in air at room temperature for 24 h and weighed again to check whether the change in mass was less than 0.5%. The thickness of the specimens was measured via a caliper before the specimens were loaded into the GHP apparatus. The results of the λ measurements at temperatures from − 10 to 50 °C and a comparison with the certified value of the CRM are presented in Sect. 3.1.

Another specimen in this work was an expanded polystyrene (EPS) board from the same batch, whose thermal conductivity was approximately 13% higher than that of the IRMM-440 CRM. The EPS specimens were cut to dimensions of 300 mm × 300 mm for the GHP and 900 mm × 900 mm for the HFM methods, and λ was measured using both apparatuses. The density of the specimen was 21 kg m^{-3}, and the thickness was 20 mm for both the GHP and the HFM measurements (see Sect. 3.2 for the results). However, additional EPS specimens with various thicknesses (50, 100, 150, and 200 mm) were also used for the HFM measurements (see Sect. 3.3 for the results).
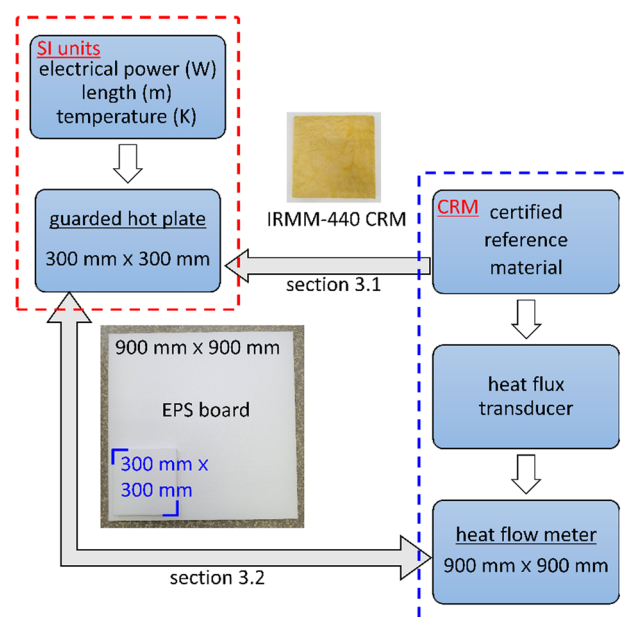
3 Results and discussion

3.1 Verification of the thermal conductivity of IRMM-440 using the GHP methods

The thermal conductivity of IRMM-440 was measured using the GHP apparatus for mean specimen temperatures from − 10 to 50 °C with an identical pair of specimens for 7 measurement sets conducted over a 9-month period. The relative change in the density of the specimen between the beginning and the end of the entire measurement procedure was less than 0.11%. Forty-seven measurements in 7 sets with 3–17 measurements in a set were made. The temperature difference between the hot and the cold plates was 20 K.

Figure 5a shows the results of the λ measurements (λ_{meas}) of the CRM using GHP and the certified value of λ_{CRM}. Figure 5b exhibits plots of the same data expressed as the relative deviation of the measured λ_{meas} with respect to the certified λ_{CRM}. The results in Fig. 5, especially Fig. 5b, clearly show that all GHP measurements were consistent with the certified value of the CRM to within 2%. The average value
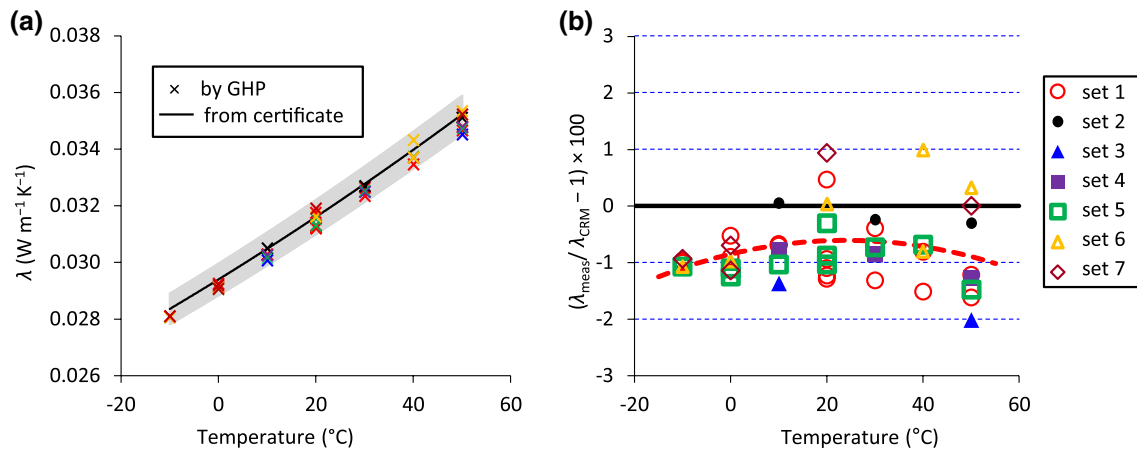

Fig. 4 Traceability chains for the GHP and the HFM methods and the specimens to cross-check the measurement results

Fig. 5 **a** λ for the IRMM-440 CRM, as measured using GHP, compared to the certified value. The shaded area represents an error of $\pm 2\% \times \lambda$. **b** The same data expressed as the relative deviation of the measured λ, λ_{meas}, with respect to the certified λ_{CRM}. The dashed line represents a quadratic fit to the relative deviation

of the relative deviation was -0.77%, and the root-mean-squared deviation from the $\lambda_{\mathrm{meas}} = \lambda_{\mathrm{CRM}}$ line was 0.98%. The quadratic fit to the deviation in Fig. 5(b) shows that the deviation was -0.6% at 20 °C, which increased (in magnitude) to approximately -1% near the lower and the upper ends of the measured temperature range.

The reproducibility of the measurements, taken from the pooled standard deviation calculated from multiple measurements at identical nominal temperature, was 0.63%. This result is consistent with the technical specifications provided by the manufacturer of the GHP, which states that the reproducibility of the measurement is better than 1%.

3.2 Measurement of λ of the EPS board using the GHP and the HFM methods

The thermal conductivity of the EPS board with a thickness of 20 mm was measured using the GHP and the HFM methods in the temperature range from -10 to 50 °C. Figure 6a shows 39 measurement points for the GHP method (the filled circles that overlap). At 20 °C, λ was measured 18 times with 11 different ΔT values of $15-30$ K. No observable systematic dependence of the measured λ value on the choice of ΔT was found. At other temperatures, ΔT was fixed to 20 K.

The same EPS board with identical nominal thickness and dimensions of 900 mm $\times$ 900 mm was measured via the HFM method. The measurements were conducted in two sets
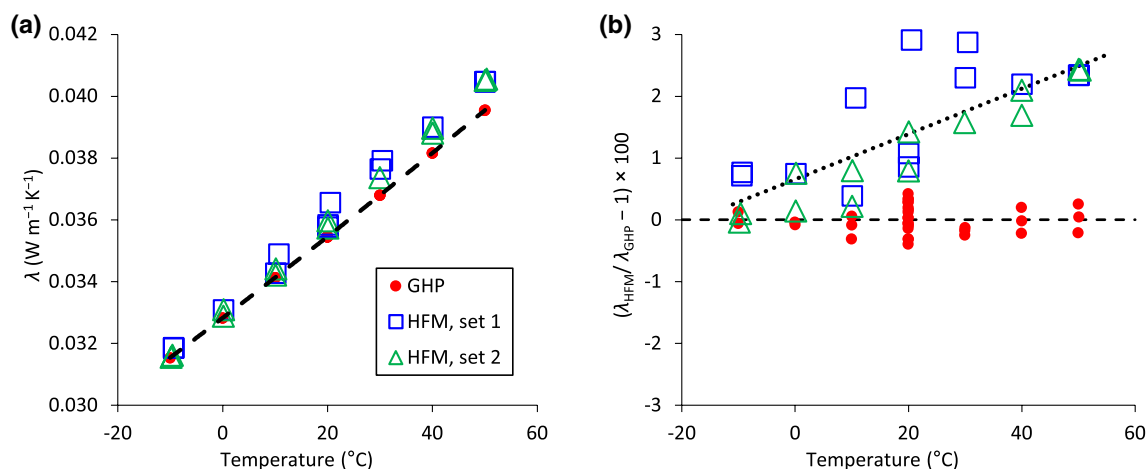

Fig. 6 **a** Measurement of λ of the EPS with a thickness of 20 mm using the GHP and the HFM methods. The dashed line represents a second-order polynomial in T that fits the GHP measurement data. **b** The same data expressed as the relative deviation of λ, as measured using the GHP and the HFM methods, from the fitted line obtained using the GHP method. The dotted line represents the linear fit of the relative deviation of two sets of HFM measurements

separated by 50 days, for a total of 26 measurement points at temperatures from -10 to 50 °C. The results of the measurements are shown in Fig. 6a as open symbols. Figure 6b depicts the same data expressed as the relative deviation of the GHP and the HFM measurements from the fitted average of the GHP measurements. The dotted line in Fig. 6b represents a linear fit to the deviation data of the HFM method and shows a weak temperature dependence from 0.2% at -10 °C to 2.4% at 50 °C. The relative deviation was less than 3% for all measurements. The pooled standard deviation taken from the measurements at identical nominal temperatures was 0.59%.

From Fig. 6a, the relative increase in the measured λ_{HFM} with increasing temperature was approximately 0.43% K^{-1} in the measured temperature range. This value is close to the previously observed increase of 0.41% K^{-1}, where the increase in λ of the EPS board was 6.1% when the temperature increased from 10 to 25 °C [14].

International standard ISO 10456 states that the measurement at one temperature can be converted to estimates at other temperatures using conversion coefficients given for various thicknesses and conductivities of the EPS specimen at temperatures from 0–30 °C [15]. From Table A.2 of ISO 10456, the conversion coefficient f_T is $(0.0034–0.0036)$ K^{-1}, where a factor $\exp[f_T(T_2 - T_1)]$ can be multiplied to convert from λ at T_1 to λ at T_2, which translates to the temperature dependence of λ that is equivalent to $(0.40–0.43)$ % K^{-1}. This temperature dependence is also consistent with our EPS measurements.

The HFM apparatus was calibrated using the same CRM based on the value from the certificate. As shown in Fig. 5b, the certified value of the CRM is higher than the value measured using our GHP by $(0.5–1.0)$%. Therefore, some of the differences between the GHP and the HFM measurements in Fig. 6b (where $\lambda_{HFM} > \lambda_{GHP}$) can be attributed to this deviation. If the HFM is calibrated based on the measured value of the CRM via the GHP method instead of the certified value, then the differences in Fig. 6b will be reduced.

3.3 Measurement of λ of EPS with various thicknesses using HFM

In addition to the EPS board with a 20 mm thickness, specimens of identical size and material with various thicknesses of 50–200 mm were measured using the HFM apparatus. Figure 7a shows the results of the thermal conductivity measurements for specimens, while Fig. 7b presents the same data expressed as the relative deviation of the HFM measurements with various thicknesses with respect to the GHP measurement of the 20 mm EPS board.

From the HFM measurement, the λ of a specimen with a 50 mm thickness at 30 °C is 0.0394 W m^{-1} K^{-1}. According to the previous HFM measurement by Lakatos [16], EPS specimens with similar densities (EPS 150 in Ref. [16]) and identical thickness have a λ of 0.0390 W m^{-1} K^{-1} at 30 °C. The difference between the two measurement results is only 1%, which is well below the measurement uncertainty of the HFM.

Additionally, Fig. 7a shows a slight decrease in the temperature dependence (slope) of λ when the thickness is increased from 20 to 150 mm. This result is also consistent with the stated conversion coefficient f_T according to ISO 10456, where the suggested f_T is 0.0041 K^{-1} for thicknesses < 20 mm and gradually decreases to 0.0034 K^{-1} for thicknesses > 100 mm [15].
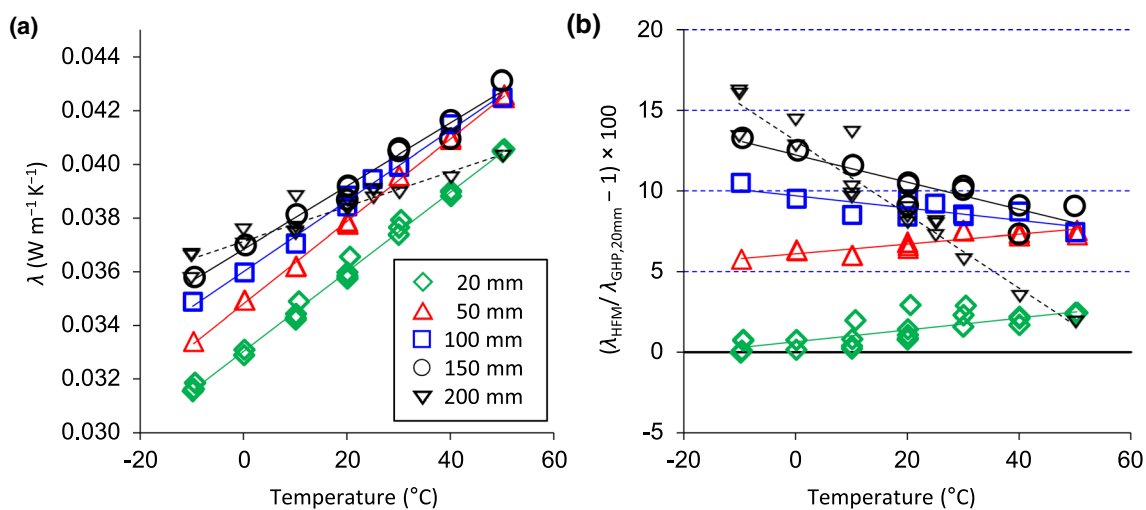

Fig. 7 a Thermal conductivity of EPS boards with various thicknesses is measured via the HFM method. **b** The same data expressed as the relative deviation of the HFM measurements with respect to the GHP measurements of the EPS specimen with a 20 mm thickness. The lines represent linear fits to the data for each thickness

For 20–150 mm thicknesses at a fixed temperature, the measured λ_{HFM} as a function of the specimen thickness monotonically increased by the relative amounts of 0.09% mm^{-1} at -10 °C and of 0.07% mm^{-1} at 10 °C. λ_{HFM} deviated from this trend for a 200 mm-thick specimen, especially when the temperature was higher than 10 °C.

A similar increase in the value of λ for a thicker specimen was observed in the HFM measurements of Gnip et al. [14], where a 3% increase in the measured λ was observed at 10 °C when the thickness of the EPS specimen was increased from 50 to 100 mm (i.e., 0.06% mm^{-1}). Lakatos et al. [17] also reported that an 80 mm-thick specimen had a measured λ larger than a 50 mm-thick specimen by (3.5–7.1)% at 17 °C when the HFM method was used (i.e., 0.12–0.24% mm^{-1}). However, they found that λ decreased when the specimen's thickness was further increased from 80 to 100 mm.

In Fig. 7a, for the specimen with a 200 mm thickness, λ increased with increasing temperature as it did the other specimens, but the slope was significantly lower than those for thinner specimens. The uncertainty in the measurements made using the HFM method increases when the thickness of the specimen is increased. International standard ISO 8301 specifies that the thickness of a specimen must be less than one-eighth of the plate dimension (clause 2.3.2 in Ref. [18]). For the 900 mm×900 mm plate in this work, this value corresponds to a specimen thickness of 113 mm. Thus, the measurements for the 150 and 200 mm specimens do not comply with ISO 8301, and a larger HFM apparatus is required for these thick specimens. Therefore, the change in the measured λ can be attributed to either a genuine dependence on the specimen's thickness or an artificial effect of the thickness on the measurement method.

4 Conclusions

In the verification process of the GHP measurements, the measured value of λ for CRM IRMM-440 at temperatures from -10 to 50 °C showed an average difference of 0.77% from the certified value and a maximum difference of 2% in this temperature range. Measurements of the EPS specimen using both the GHP and the HFM methods showed an average difference of 1.4% and a maximum difference of below 3% in the same temperature range. The expanded uncertainty in the assigned thermal conductivity value of the CRM was 0.8–1.0%, while those in the GHP and the HFM measurements were 2% and 1.4%, respectively. When these uncertainties are considered, the verifications of the thermal conductivity measurements reported herein are consistent with one another and with the certified value for the CRM.

Thick EPS specimens (up to 150 mm) at temperatures from -10 to 50 °C showed a monotonic increase in λ with increasing thickness, and the dependence of λ on the thickness was similar to that in previous reports. Furthermore, our results show that the dependence is stronger at low temperatures and weaker at high temperatures. For a 200 mm-thick specimen, the measurement results indicate that the specimen was too thick for the 900 mm×900 mm HFM apparatus used in this research.

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