Testing the Accuracy of the GHP 456 and HFM 446 with the New ERM-FC440 Reference Material

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Introduction

The new reference material for thermal conductivity, ERM-FC440, is the successor to the well-known IRMM-440 reference material, which is no longer available. ERM-FC440 was certified by the Institute of European Reference Materials, ERM®, located in Belgium [1]. The material is intended for quality control and assessment of the method performance of guarded hot plate (GHP) measurements as well as for calibration of heat flow meter (HFM) instruments [2].

Properties of ERM-FC440

ERM-FC440 resin-bonded glass fiber boards are available in three different sizes:

- 30 cm x 30 cm (ERM-FC440a)
- 50 cm x 50 cm (ERM-FC440b)
- 60 cm x 60 cm (ERM-FC440c)

The mean thickness of ERM-FC440 is (28.65 ± 0.15) mm under a load of 0.25 kPa and (28.27 ± 0.19) mm under a load of 1.5 kPa. The densities of all ERM-FC440 samples are in the range of 130 and 148 kg/m^3 [2]. The thickness and density of each individual sample plate at 0.25 kPa are stated on each reference material certificate.

ERM-FC440 is certified for its thermal conductivity in the temperature range from -10°C to 70°C [2]. In addition, indicative thermal conductivity values are given in the range from -150°C to -10°C. The temperature-dependent thermal conductivity λ of ERM-FC440, as stated in the certificate, is expressed as

$$\lambda\,[\mathrm{W/(m{\cdot}K)}] = 0.03104 + 1.1 \cdot 10^{-4} \cdot T\,[°\mathrm{C}] \quad (1)$$

for the entire temperature range from -150°C to 70°C.

1 ERM-FC440 material in original packaging (left) and with NETZSCH laser marking (right)

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The expanded uncertainty is 1.1 % in the range from -10°C to 70°C and 1.9% to 1.1% in the range from -150°C to -10°C. Figure 2 displays the nominal thermal conductivity λ of ERM-FC440 according to equation 1 as well as the uncertainty budget.

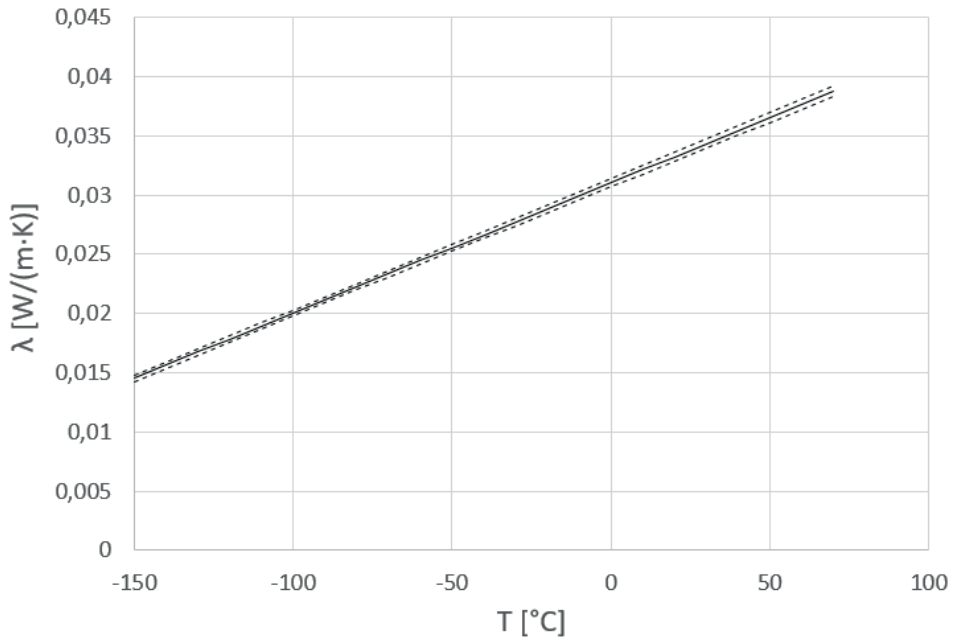
Thermal Conductivity Results

1. Results Obtained with the GHP 456

The guarded hot plate (GHP) technique is an absolute method without any calibration of the thermal conductivity. In the two-plate mode, the thermal conductivity λ is calculated from the power Q flowing into the hot plate with metering area A, the temperature gradient across the two samples ΔT as well as the mean sample thickness d as follows:

$$\lambda = \frac{Q}{2A} \cdot \frac{d}{\Delta T} \qquad (2)$$

GHP measurements on ERM-FC440 samples were carried out using a NETZSCH GHP 456 *HT Titan*® equipped with liquid nitrogen cooling. ERM-FC440a specimens with serial numbers 001, 002, 003, and 005 were used for the GHP tests. The pair of specimens 001+002 and the pair 003+005 were each measured simultaneously in the two-plate mode. The temperature gradient across the samples was 30 K at temperatures below -10°C, and 20 K at 10°C and higher temperatures. The specimens had rigid spacers in their corners with a length identical to the nominal sample thickness in order to ensure a defined sample thickness.



2 Temperature-dependent nominal thermal conductivity of ERM-FC440 (solid line) including expanded uncertainty (dashed lines).

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Figure 3 depicts the GHP measurement results: In the temperature range between -150°C and 70°C, the relative deviation from the nominal thermal conductivity values calculated from equation 1 is less than ± 1.3% for all measurement data except for one point at -150°C which deviates by -2.2%. These results are in accordance with the expected accuracy of the GHP 456.

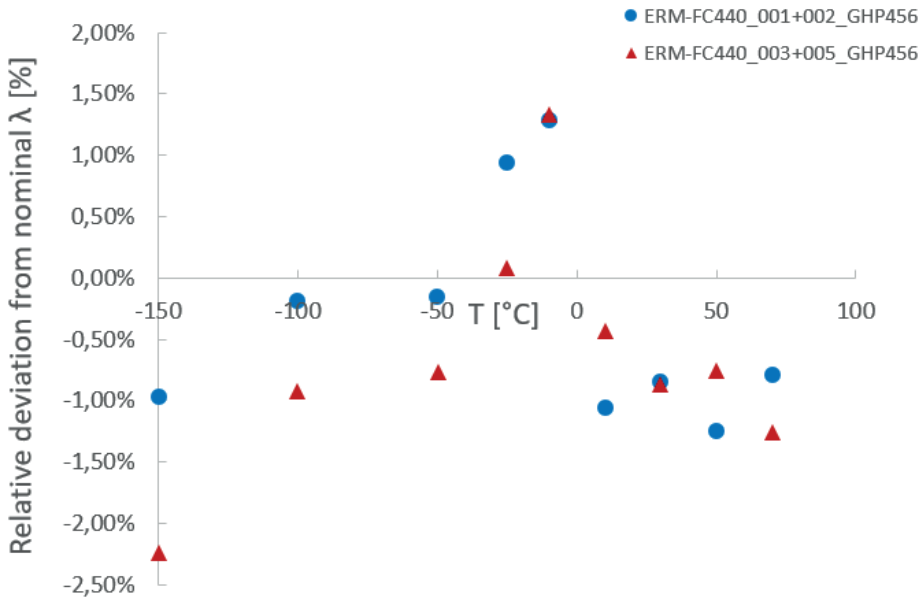
2. Results Obtained with the HFM 446

The heat flow meter (HFM) technique is a relative method based on calibration of the heat-flux sensors using a reference material with known thermal conductivity. The unknown thermal conductivity λ of a sample is calculated from the heat flux per area Q/A and the temperature gradient ΔT across the sample with mean thickness d according to the Fourier's equation for one-dimensional heat flow as follows:

$$\lambda = \frac{Q}{A} \cdot \frac{d}{\Delta T} \qquad (3)$$

The HFM measurements on the ERM-FC440 samples were carried out using the NETZSCH HFM 446 *Lambda* Eco-Line *Small*, *Medium* and *Large* instruments.

Two HFM 446 *Large* devices with serial numbers 0009 and 0010 at different locations were used to test the ERM-FC440c specimens with serial numbers 004 and 005. Two HFM 446 *Medium* devices with serial number 0007 and 0009 were used to test the ERM-FC440a specimens with serial numbers 001, 002, 003 and 005. Three HFM 446 *Small* devices with serial numbers 0086, 0087 and SOA-002 were used to test ERM-FC440 specimens with the size 20 cm x 20 cm cut out of the ERM-FC440c board with serial number 005 after the measurements in the HFM 446 *Large* were completed. For documentation purposes, each 20-cm board was assigned an identification number with five digits, which was lasered on the front face (see also figure 1 on the right). All devices were calibrated with either NIST SRM 1450d or IRMM440. The measurements were performed at mean temperatures between -10°C and 70°C with a temperature gradient across the samples of 20 K and a contact pressure of maximum 2 kPa.



3 Relative deviation of the temperature-dependent thermal conductivity from the nominal values of two pairs of ERM-FC440a specimens (serial numbers 001+ 02 and 003+005), measured with a GHP 456 *HT Titan*® in the two-plate mode.

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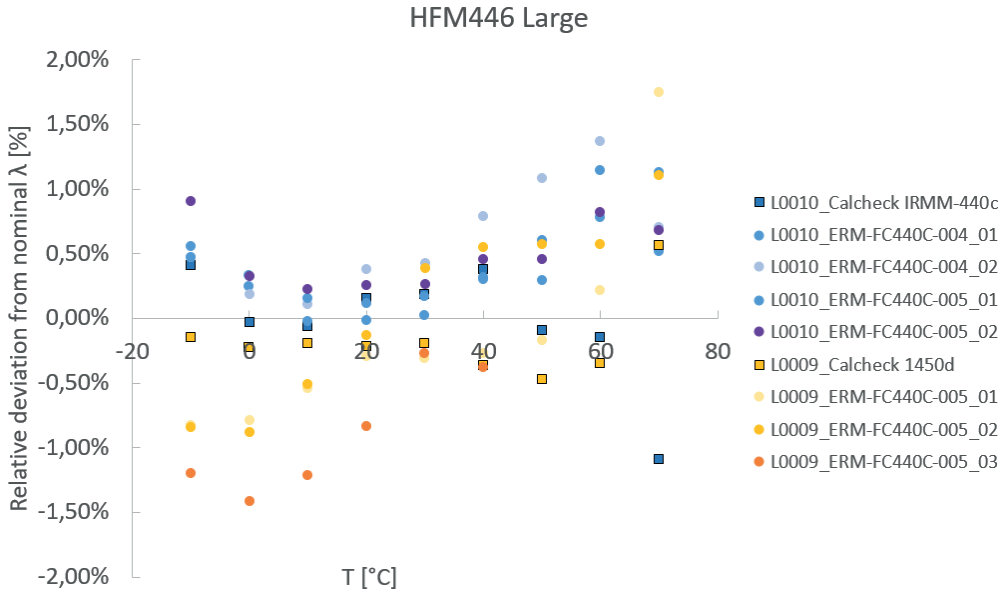
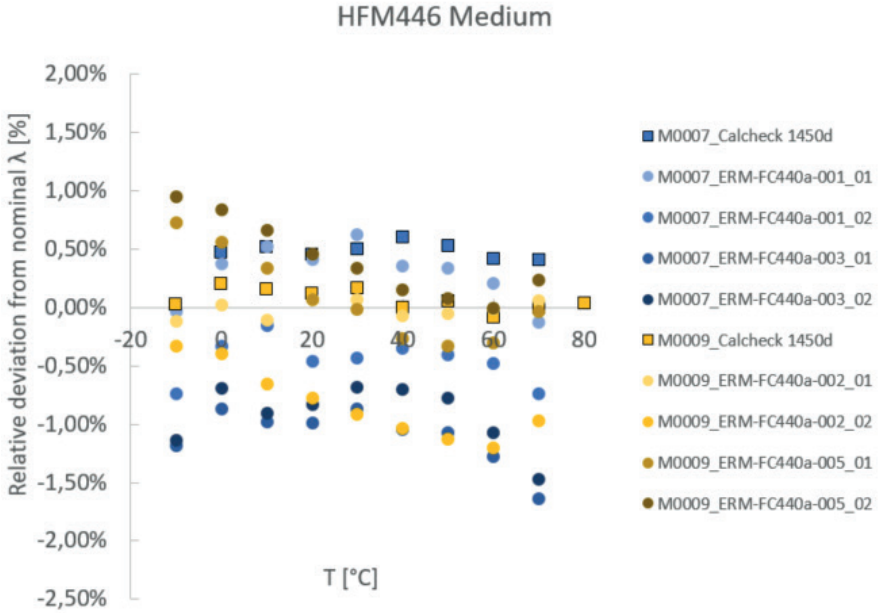
Figures 4 to 6 depict the measurement results from all HFM instruments. Across the entire temperature range, the relative deviation from the nominal thermal conductivity values calculated using equation 1 is within ± 1.5% for most measurement results, except for some measurement points at the highest temperature of 70°C. All results are in accordance with the expected accuracy of ± 2% for the HFM 446 heat flow meters.



4 Relative deviation of the temperature-dependent thermal conductivity from the nominal values of two ERM-FC440c specimens (serial numbers 004 and 005) measured with two HFM 446 *Large* devices.



5 Relative deviation of the temperature-dependent thermal conductivity from the nominal values of four ERM-FC440a specimens (serial numbers 001, 002, 003 and 005) measured with two HFM 446 *Medium* devices.

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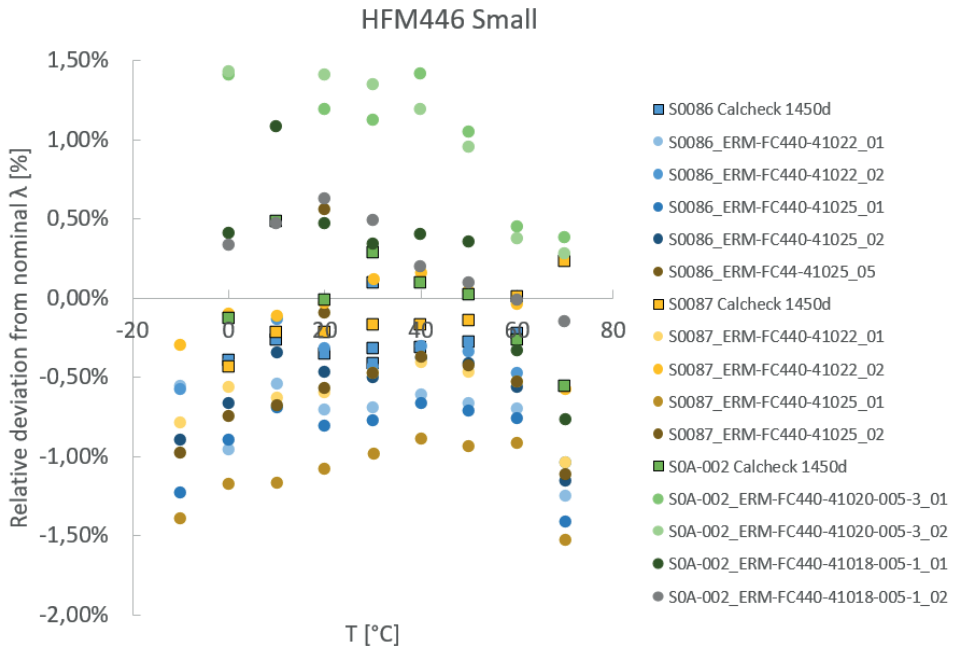

6 Relative deviation of the temperature-dependent thermal conductivity from the nominal values of the four 20 cm x 20 cm large ERM-FC440 specimens (cut out of board serial number 005) measured with three devices of type HFM 446 *Small*.

Summary

The thermal conductivity of the new reference material for thermal conductivity, ERM-FC440, was investigated in the temperature range between -150°C and 70°C applying one GHP 456 and several HFM 446 devices. Almost all results were in agreement within ± 1.5% with the nominal values, reflecting the accuracy of GHP 456 *Titan*® and HFM 446 *Lambda* instruments by NETZSCH.

References

[1] European Reference Materials (ERM®), European Commission, Joint Research Centre, Directorate F – Health, Consumers and Reference Materials, Retieseweg 111, 2440 Geel, Belgium.
[2] T. Linsinger, The certification of the thermal conductivity of a resin bonded glass fiber board: ERM®-FC440a, ERM®-FC440b and ERM®-FC440c, EUR 30859 EN, Publications Office of the European Union, Luxembourg, 2021, ISBN 978-92-76-42259-4, doi:10.2760/759309, JRC126677.

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