WHITE PAPER

NETZSCH

Qualifying the New NETZSCH EPS Standard Material for HFM Instruments: A Question of Reproducibility

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Introduction

When discussing the reproducibility of measurement results in general, what is known as the "reproducibility conditions" must always be taken into account. Measuring exactly the same specimen several times with exactly the same measuring device is usually not considered to be a reproducibility test, but a repeatability test. In order to draw conclusions about the reproducibility, one requires – at a minimum – measurements on multiple specimens of the same type and additionally, if possible, measurements using different devices. Such a reproducibility study is described in the following:

NETZSCH-Gerätebau GmbH has recently qualified its own factory standard for the calibration and validation of heat flow meter (HFM) instruments in the temperature range between 8°C and 40°C: NETZSCH EPS, which is made of expanded polystyrene (see figure 1). Numerous NETZSCH EPS specimens with the dimensions 600x600x25 mm, 300x300x25 mm and 200x200x25 mm were investigated using five different NETZSCH Heat Flow Meter devices of the HFM 446 type (one HFM 446 *Large*, two HFM 446 *Medium* and two HFM 446 *Small* instruments). The NETZSCH EPS specimens had mean densities in the range from 24.5 to 28 kg/m^3. The HFM measurements were carried out at mean temperatures of 8°C, 16°C, 24°C, 32°C and 40°C using a temperature gradient of 20 K and a load of 2 kPa. In order to calibrate the HFM devices, three different certified reference samples (one specimen 600x600x25 mm, one specimen 300x300x25 mm and one specimen 200x200x25 mm) of the NIST SRM 1450d type were used, which are fiber boards.

1 Photo of a 300 mm x 300 mm x 25 mm NETZSCH EPS specimen

Experimental Results

Figure 2 shows the thermal conductivity values of 30 different NETZSCH EPS specimens:

- 10 specimens of the size 600x600x25 mm were measured with an HFM 446 *Large*
- 10 specimens of the size 300x300x25 mm were measured with an HFM 446 *Medium*
- 10 specimens of the size 200x200x25 mm were measured with an HFM 446 *Small*

These measurements resulted in three main observations: Firstly, the thermal conductivity values are higher for higher mean sample temperatures; this finding was, of course, expected. Secondly, the thermal conductivity values do not depend – within about ±0.5% standard deviation – on the specimen size or measuring device, which demonstrates a reproducibility of the results of about ±0.5%. Thirdly, the relatively low scatter of the data even allows for the identification and quantification of a slight dependence of the thermal conductivity on the density of the specimens. The lower thermal conductivity at higher density is a known property of such EPS materials; it can be explained by the fact that less radiation is contributed to the thermal conductivity [1].

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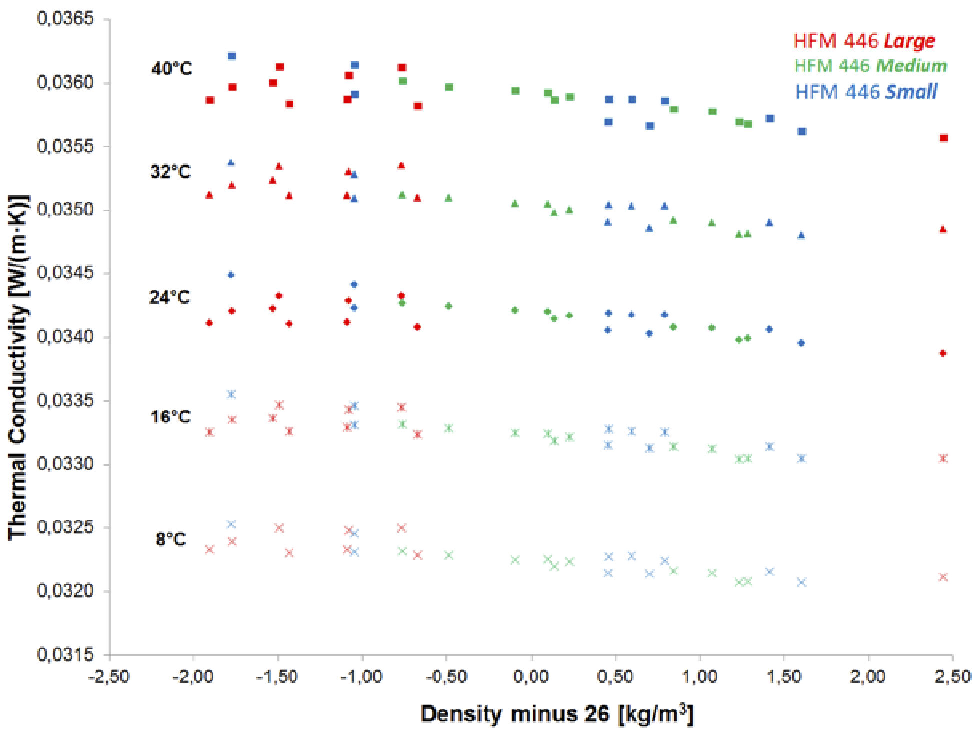
Based on the data such as shown in figure 2, the mean thermal conductivity of NETZSCH EPS can be described as a function of two parameters, the mean sample temperature and density. This allows for calculating the "nominal" thermal conductivity for each individual specimen in future measurements. Figure 3 displays the temperature-dependent part, which exhibits the typical linear behavior.

Another reproducibility test is depicted in figure 4. Over a period of about 2 to 3 weeks, 100 different NETZSCH EPS specimens of the size 300x300x25 mm were measured with the same HFM 446 *Medium* device at a mean sample temperature of 40°C. The calibration of the HFM was validated from time to time using the same specimen of NIST SRM 1450d with which the device had originally been calibrated. The thermal conductivity values obtained are compared with the nominal values, which refer to the certified reference value at 40°C in the case of NIST SRM 1450d, and the expected density-dependent value at 40°C (see above) in the case of NETZSCH EPS. The thermal conductivity values for NETZSCH EPS shown in figure 4 are in agreement, within ±0.5%, with the nominal values – in particular, if the individual sample density is taken into consideration, because this clearly reduces the standard deviation. These results again demonstrate the excellent reproducibility of measurements carried out with the NETZSCH HFM 446 in general – and furthermore confirm a correct and representative description of the thermal conductivity values of NETZSCH EPS.

Regarding the qualification of NETZSCH EPS, it should be noted that the thermal conductivity values determined via HFM measurements were reproduced for an exemplary pair of 300x300x25 mm samples using two different Guarded Hot Plates devices – one of the NETZSCH GHP 456 type and another of the NETZSCH GHP 456 HT type – with an absolute accuracy of about ±1.5%. Furthermore, two exemplary 500x500x25 mm EPS samples were investigated at the FIW Munich institute, D-82166 Gräfelfing, Germany, also using HFM and GHP instruments; the results were in agreement with the thermal conductivity values obtained by NETZSCH within about ±1.0% (GHP) / ±0.5% (HFM). In addition, an investigation was conducted regarding the long-term stability of the NETZSCH EPS thermal conductivity values; over the course of several dozens of HFM and GHP measurements at 40°C, no significant change in thermal conductivity was observed.



2 Temperature- and density-dependent thermal conductivity of 30 different NETZSCH EPS specimens

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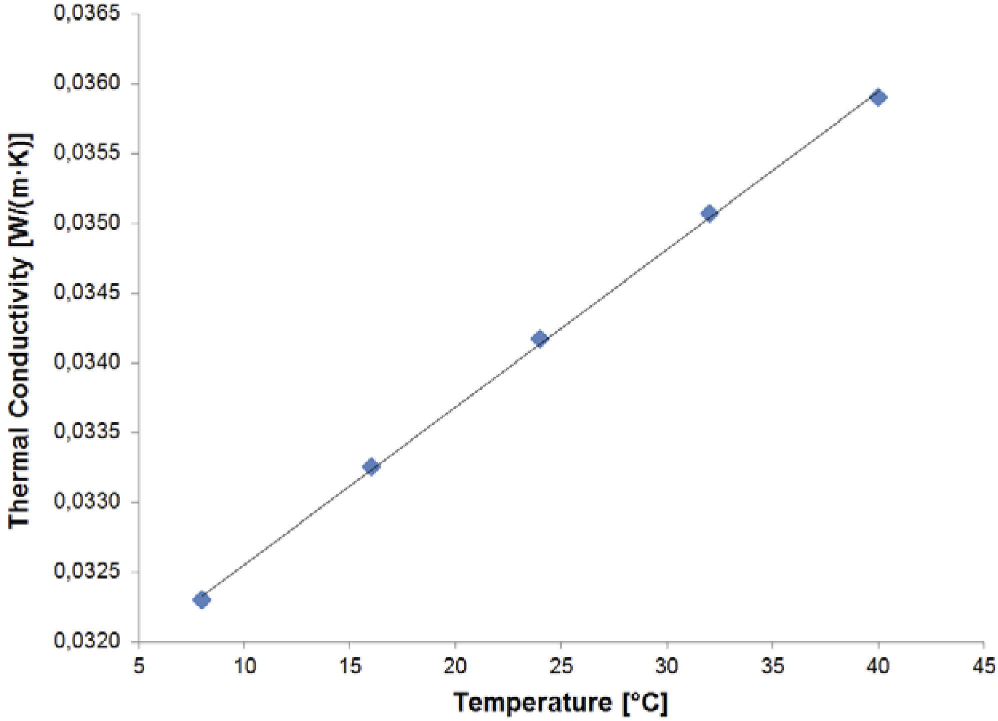
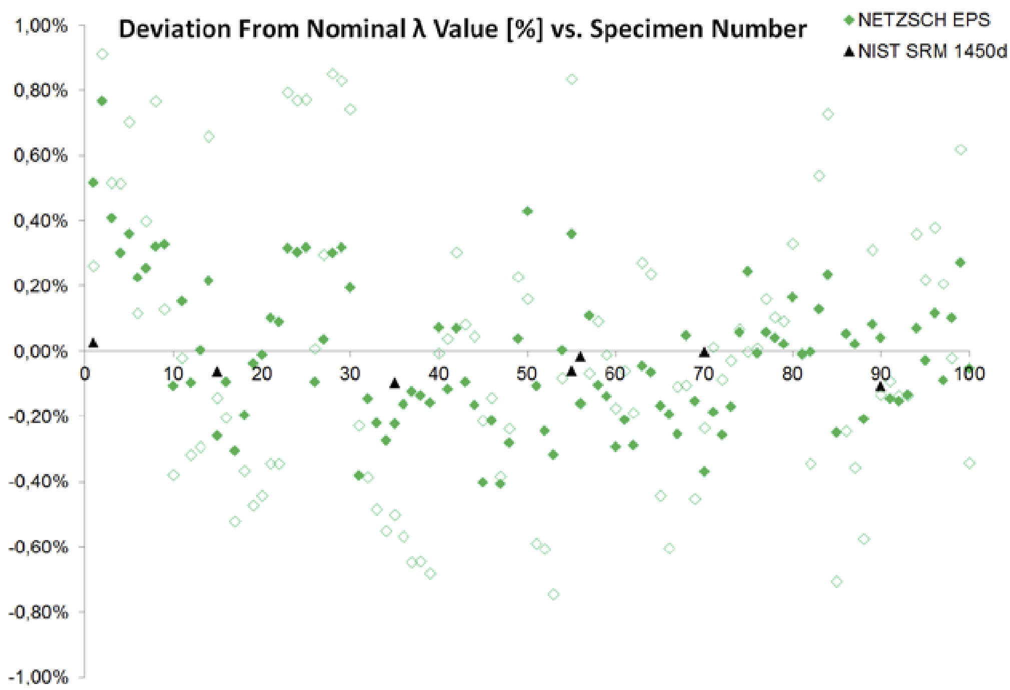


3 Mean thermal conductivity of NETZSCH EPS for a density of 26 kg/m³ as a function of temperature



4 Relative deviation of the thermal conductivity λ from nominal values of 100 different NETZSCH EPS specimens of the size 300x300x25 mm measured with the same HFM 446 *Medium* device at 40°C mean sample temperature. In the case of the solid green full symbols, the sample density for each individual NETZSCH EPS sample was considered for calculating its nominal λ value, whereas a constant density of 26 kg/m³ was assumed for all samples in the case of the green outline symbols. The black triangles reflect calibration checks done with an NIST SRM 1450d specimen.

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Summary

A total of more than 300 different NETZSCH EPS specimens of the sizes 600x600x25 mm, 300x300x25 mm and 200x200x25 mm were measured with five different NETZSCH heat flow meter devices of the types HFM 446 *Large*, HFM 446 *Medium* and HFM 446 446 *Small*. These HFM devices were calibrated using three different certified reference samples of the type NIST SRM 1450d. Measurements were carried out in the temperature range between 8°C and 40°C. Reproducibility of the thermal conductivity values was observed at about ±0.5%, thus fulfilling the requirements set by HFM standards [2, 3].

These results allowed for the qualification of NETZSCH EPS as a new standard material for the calibration and validation of HFM instruments.

References

[1] J. Schellenberg and M. Wallis, Dependence of Thermal Properties of Expandable Polystyrene Particle Foam on Cell Size and Density, Journal of Cellular Plastics, Volume 46, 2010, p.209-222

[2] ASTM C518: Standard Test Method for Steady-State Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus.

[3] ISO 8301: Thermal insulation – Determination of steady-state thermal resistance and related properties – Heat flow meter apparatus.

The Authors

Dr. Alexander Schindler has worked in the fields of experimental physics, thermal analysis and thermophysical properties for over 20 years. At NETZSCH, he has been employed in the Applications Laboratory as well as in the Hardware and Software Development. He is a known expert in thermal characterization methods and their applications.

Michael Düngfelder studied mechanical engineering (M. Eng.) and is a specialist for production techniques and numerical simulations like FEM or CFD. Since April 2016, he has been employed at NETZSCH as one of the main development and construction engineers for the HFM *Lambda* Series which can be applied for the investigation of insulating materials.

Dr. Jan Hanss studied chemistry at the University of Hamburg and received is Ph.D. in the field of inorganic chemistry. He has then held a permanent position at the University of Augsburg (solid state chair) for 13 years. Since 2012, he has been Head of the NETZSCH Applications Laboratory.

Dr. Martin Brunner studied physics and received his Ph.D.in the field of materials science in 2011. Since then, he has worked at NETZSCH and has been appointed Director of Research & Development in 2018.

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