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Emissivity measurements on reflective insulation materials

Emissionsgradmessungen an hochreflektierenden Isolationsmaterialien

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Abstract: The development and use of new thermal insulation products in many industrial sectors, ranging from building insulations to power generation or satellite applications, requires the accurate knowledge of the radiative properties of the investigated material, i. e. its emissivity. A major objective of the research project “Improvement of emissivity measurements on reflective insulation materials” within the framework of the European Metrology Programme for Innovation and Research was to improve and validate reference techniques for the measurement of the total hemispherical emissivity of low emissivity foils with an absolute measurement uncertainty below 0.03. The calibration and measurement procedures developed within this project shall lead to a significant benefit for industrial manufacturers of reflective foils as well as for the end-users of the industrial instruments used to characterize them.

Keywords: Emissivity, reflectivity, thermal insulation, reflective foil.

Zusammenfassung: Die Entwicklung und der Einsatz neuer Wärmedämmprodukte in vielen Anwendungen, von der Gebäudeisolierung bis zu Energieerzeugung oder Satellitenanwendungen, erfordert die genaue Kenntnis der Strahlungseigenschaften des verwendeten Materials, d. h. seines Emissionsgrades. Ein Hauptziel des Projekts “Improvement of emissivity measurements on reflective insulation materials” (EMIRIM) im Rahmen des Europäischen Metrologieprogramms für Innovation und Forschung (EMPIR) war die Verbesserung und Validierung von Referenztechniken für die Messung des hemisphärischen Gesamtemissionsgrades von Folien mit niedrigem

Emissionsgrad und die Reduzierung der Messunsicherheit auf unter 0,03. Die im Rahmen dieses Projekts entwickelten Kalibrier- und Messverfahren sollen sowohl bei industriellen Herstellern von Reflexionsfolien als auch bei Endnutzern von industriellen Messgeräten Anwendung finden.

Schlagwörter: Emissionsgrad, Reflexionsgrad, Wärmedämmung, hochreflektierende Folie.

1 Introduction

Reflective thermal insulation materials are expediently used in many industrial sectors ranging from renewable energy, building insulations, nuclear power generation to cryogenic and satellite applications. Their highly effective insulation properties are complimented by their light weight, small footprint and low particle contamination. Such products are generally made from aluminum foil applied often from both sides to different substrates and creating possibly multi-layer insulation constructions up to several dozen layers. The successful implementation and on-site quality control as well as the further development and improvement of the new insulation products are significantly based on the accurate knowledge of their radiative properties, i. e. its emissivity.

The commercially available portable instruments (emissometers or reflectometers) for on-site measurements as well as integrating spheres in combination with FTIR spectrometers are most often used for measuring the total hemispherical emissivities of the thermal insulation materials [1, 2, 3, 4]. However, the high uncertainties associated with different measurement techniques and experiment condition, which include not validated measurement and calibration procedures, inhibit technical progress for this field. The differences in results can be significant: a recent comparison of different measurement techniques made within the standardization CEN/TC 89/WG 12 showed discrepancies in the absolute emissivity from 0.02 to 0.08 on the same reflective foil [5]. Furthermore, the standard EN 16012 of the CEN/TC 89/WG 12 [5, 6]

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practically limits the possibility to measure low-emitting surfaces, recommending a rounding of measured emissivities lower than 0.05 to the value of 0.05. An improvement of the accuracy of emissivity measurements of reflective foils and a validation of the reference techniques are urgently needed in this field. Along with the direct improvement of the relevant sections of the standards EN 16012 and EN 15976, it is, therefore, a major objective of the project “Improvement of emissivity measurements on reflective insulation materials” (EMIRIM) within the framework of the European Metrology Programme for Innovation and Research (EMPIR) [7].

The Physikalisch-Technische Bundesanstalt (PTB), as a partner within the EMIRIM project, undertook to measure the emissivity of the highly reflecting insulation foils (emissivity below 0.1) with an absolute uncertainty of better than 0.03. The PTB routinely determines directional spectral and hemispherical total emissivities of different materials with low uncertainties in air and under vacuum conditions [8, 9]. However, the measurement of the crumpled foils is challenging because of the limited thermal contact of such samples with the heater. A poor thermal contact will result in an uncertain and non-uniform sample surface temperature and will dominate the overall measurement uncertainty. Therefore, a new sample holder based on the principle of vacuum mounting of the sample was designed within the EMIRIM project. In addition, the high reflective properties of the foils, especially at room temperature, result in very low detector signals, increasing the overall uncertainty budget as well. Thus, an accurate determination of the complete radiation budget is crucial in the case of the measurement of highly reflecting insulation materials.

2 Vacuum sample holder

The Emissivity Measurement in Air Facility (EMAF) has been operated at PTB for several years and has successfully been used in several research projects [10]. The EMAF was already used within the scope of the EMIRIM project to perform the high-accuracy emissivity measurements of the preliminary reference materials [1, 11]. Since the new vacuum sample holder for foils must be operated at the EMAF, its fitting in the existing sample holder was required. The latter is based on the two components [8]: a spherical enclosure made of copper and a sample heater inside this enclosure which provides a very good temperature uniformity of the sample. In combination with an accurate knowledge of the optical properties, thermal conductivities, geometry and temperatures of all materials, it

Figure 1: Photograph of the new vacuum sample holder for spectral emissivity measurement of the reflective thermal insulation foils. Front plate, pump connection, channels of the “fixation profile” and the holes for accommodation of the thermometers are labelled.

allows the precise calculation of the surface temperature of the investigated sample.

Usually, the sample is mounted on a vertical copper heating plate by three clamps. However, a similar mounting of the flexible, crumpled and thermally expanding foils is not realizable: it is obvious that three fixing points on the foil edges cannot provide a good thermal contact, especially over the entire sample area. Therefore, the new sample holder has been developed based on the principle of vacuum mounting of the thin foils. Its construction consists of several parts: a housing, a front plate, and a pump connection (Figure 1). The housing, made of aluminium, has a special internal channel structure that creates a vacuum inside of the sample holder by connection it to the pump. The front plate with a “fixation profile” for pumping air is mounted on the housing. A sample, which typically has a round shape with a diameter ranging from 50 mm to 90 mm, is placed on the “fixation profile” of the front plate. In addition, two holes for thermometers are provided in the housing for monitoring and regulating the sample temperature. The vacuum sample holder mounted at the EMAF can be seen in Figure 2. The hemispherical enclosure is presented here in an open position to illustrate the smooth surface of the foil, which indicates its good fixation on the vacuum sample holder.

The local temperature distribution of a sample mounted on the vacuum sample holder was investigated with a thermographic camera which was positioned in front of the sample holder using an additional positioning system. For these measurements, a light absorbent foil

Figure 2: Photograph of the setup for emissivity measurements in air (EMAF): two reference blackbodies, hemispherical enclosure in open position, translation stage and a reflective foil on the new vacuum sample holder.

“Metal Velvet” of company Acktar of 90 mm in diameter was selected. The high emissivity of this foil effectively minimizes the background reflections and consequently an undistorted temperature distribution on the optical surface could be measured. Additionally, the Data Reference Method (DRM) which is explained in detail in [12, 13] was applied to correct the nonuniformity of the imager system for a field of $90 \text{ mm} \times 90 \text{ mm}$.

The lateral distribution of the radiation temperature of the sample mounted on the vacuum holder operated at 40°C inside of a spherical enclosure is depicted in Figure 3. A slight increase of the radiation temperature can be seen towards the left side of the foil surface. Nevertheless, over the diameter of the field-of-view of the spectrometer the uniformity is better than 68 mK (standard deviation) for an angle of observation of 5° with respect to the surface normal (circle with 12 mm in diameter) and better than 121 mK (standard deviation) for an angle of observation of 70° with respect to the surface normal (ellipse with the major axis of 40 mm). This nonuniformity will not have a significant effect on the uncertainty budget and the achieved uniformity over the area observed area of the optical surface can be considered sufficient.

3 Reference thermal insulation foils

Within the EMIRIM project, the PTB and the Laboratoire national de métrologie et d'essais (LNE) have selected

Figure 3: Distribution of the radiation temperature image of vacuum sample holder operated at about 40°C with the mounted high-absorbent foil “Metal Velvet” recorded by a thermographic camera. The field-of-view of the spectrometer is shown for two angles of observation of 5° and 70° with respect to the surface normal. The observed temperature uniformity is 68 mK for the circular area with 12 mm diameter and 121 mK for the elliptical area with the major axis of 40 mm.

four foils for extensive investigation (Figure 4). Three foils are produced by the company ACTIS: polyethylene (PE) foil “colorless lacquered”, PE foil “copper lacquered” and mesh reinforced foil (aluminium color). In addition, the company Eurofoil has commercially provided the fourth sample: bare aluminium foil. The foils are shown in Figure 4 and their thicknesses and thermal conductivities are given in Table 1.

4 Emissivity of thermal insulation foils

The measurements of the directional spectral emissivity of four types of selected foils were performed at the EMAF at a foil temperature of 25°C , using the new vacuum sample holder described in Section 2. For the measurements, the spectrometer was equipped with a KBr broadband beam-splitter and a pyroelectric DLaTGS detector. According to the usual measurement procedure, the directional spectral emissivities were determined at different angles of observation ranging from 20° to 70° . Here, measurement at angles below 20° were omitted to avoid a self-reflection or self-observation of the detector in the spectrometer due to the high-reflective surfaces. The emissivity variation be-

Table 1: Thicknesses and thermal conductivities of the four selected foils. Values of the thermal conductivity specified for heat transport perpendicular to the surface of the foil.

Foils	Thickness (μm)	Thermal conductivity ($\text{W}/(\text{m}\cdot\text{K})$)
Foil 1: Bare aluminium foil	30	235 ¹
Foil 2: PE foil “copper lacquered”	80	0.103 ²
Foil 3: PE foil “colorless lacquered”	80	0.102 ²
Foil 4: Mesh reinforced foil	150	0.145 ³

¹Value from literature (European Aluminium Foil Association, 2019).²Value measured by PTB, applying a thermal constants analyzer (Hot Disk AB, 2019).³Value calculated based on information from the manufacturer (ACTIS Insulation Ltd, 2019). **Figure 4:** Photograph of the selected foils (from left to right): bare aluminium foil (Foil 1), PE foil “copper lacquered” (Foil 2), PE foil “colorless lacquered” (Foil 3) and mesh reinforced foil (Foil 4). **Figure 5:** Directional spectral emissivity of the PE foil “copper lacquered” measured at a temperature of 25 °C and under an angle of observation of 20° with respect to the surface normal. The spectral distribution of the standard uncertainty is shown in the lower part of the plot with the respective scale on the right-hand ordinate axis.

tween the surface normal and an angle of 20° is very small and can be neglected here.

Aluminium foil is a material with very high reflectivity and very low emissivity respectively and, therefore, shows very little self-radiation. To improve the signal-to-noise ratio, the recorded radiation was increased by the reflected radiation from the spherical enclosure. For this purpose, the spherical enclosure was operated at the highest possible temperature of 80 °C. Thus, the radiation exchange between the sample and the enclosure, as well as the influence of the latter on the final signal must be considered. This is done using the earlier developed multiple reflections method. This method considers all originating reflections inside the spherical enclosure to evaluate the final signal seen by the detector [14]. The multiple reflections between the sample and the sphere and the sphere with itself are mathematically considered as the sums of the individual radiation terms of all reflective orders using an in-

finite geometric series. As a result, the complete radiation budget can be accurately calculated and the uncertainty of the emissivity measurements of the low-emitting samples can be significantly improved.

The results of Foil 2 – PE foil “copper lacquered” – is presented in detail here. Figure 5 shows the directional spectral emissivity under an angle of observation of 20° (brown curve), whereas the associated standard uncertainty is depicted as blue curve and on the right-hand ordinate axis. The uncertainty of the directional spectral emissivity is calculated according to the GUM [6] for each specific condition and for each individual measurement, since many components depend on the measurement conditions. Furthermore, the uncertainties are spectrally dependent via Planck’s law and therefore vary strongly with wavelength. On the one hand side the standard uncertainty (Figure 5) increases slightly with the wavelengths. Furthermore, below 5.5 μm the measurement uncertainty

Figure 6: Directional spectral emissivity of PE foil “copper lacquered” under different angles of observation with respect to the surface normal.

is going up due to the low amount of emitted radiation at these low temperatures.

The results for observation angles of 20°, 30°, 50°, 60° and 70° with respect to the surface normal are depicted in Figure 6. An increase of the directional emissivities towards larger angles is clearly visible, which is a typical behavior for the low-emitting surfaces. From these results, the total directional emissivities are obtained by spectral integration. Furthermore, a theoretical model for the angular dependence of the directional total emissivity is used for the hemispherical emissivity. It is based on the sum of the Fresnel equations for two polarization directions as functions of the complex refractive index and an offset and is fitted to the experimental values. By angular integration of this model the hemispherical emissivity is obtained [8]. The uncertainty of the directional total emissivity $u(\varepsilon)$ is calculated – as a conservative estimate – as a difference $\varepsilon' - \varepsilon$ of the total directional emissivity ε' calculated as integral of the spectral directional emissivity with added uncertainty and of the total directional emissivity ε calculated as integral of the spectral directional emissivity without added uncertainty. In a very similar manner, the uncertainty of the total hemispherical emissivity is calculated. Again, as a conservative estimate as the difference of the two angular integrations of the two angular models fitted to the total emissivity with and without added uncertainties. Because both angular distributions converge towards 90° with low uncertainty the uncertainty of the hemispherical emissivity can be lower than of an individual total emissivity as it is here the case. The uncertainty of 0.022 for the hemispherical emissivity presented in Table 2 significantly improves the lowest achievable validated value

Figure 7: The directional spectral emissivity of the PE foil “copper lacquered” measured at the EMAF at a temperature of 25 °C and observed at an angle of 20°. This measurement is compared with the indirectly determined emissivity from a directional-hemispherical spectral reflectivity measurement (calculated as “1 – reflectivity”).

of 0.05 described in the standard EN 16012 of the CEN/TC 89/WG 12. All uncertainty contributions to the overall uncertainty budget of the directional spectral emissivity are presented in detail in [15].

To validate the emissivity results obtained by the direct EMAF measurements they were compared with reflectivity measurement using a different approach. The setup of PTB for the determination of optical properties, consisting of a gold-coated integrating sphere and a vacuum FTIR spectrometer [10], allows to obtain the directional-hemispherical spectral reflectivity in the NIR and MIR ranges. The consistency of the two independent measurements within their ranges of uncertainty is illustrated in Figure 7. For this comparison, the directional-hemispherical reflectivity measurements are presented here as “1 – reflectivity”. However, it must be stated that the directional-hemispherical reflectivity measurement exhibits the systematic sources of uncertainty which are aimed to overcome in the EMIRIM project, i. e., the systematic deviation due to the different angular distribution of the reflected light from the sample under test (foil) and the reference sample (gold mirror). This deviation is probably not fully corrected or considered in the uncertainty budget. So the directional-hemispherical reflectivity could probably have a systematic deviation.

Due their similar angular emissivity distribution and a comparable uncertainty in the measurement of their emissivity, the results on the other three selected foils are summarized in Figure 8 and in Table 2. The directional spectral emissivities of all four samples are depicted in Figure 8 for

Table 2: Directional total and hemispherical total emissivities of Bare aluminium (Foil 1), PE foil “copper lacquered” (Foil 2), PE foil “colorless lacquered” (Foil 3) and mesh reinforced foil (aluminium color) (Foil 4) in the wavelength range from 5 μm to 20 μm with their corresponding standard uncertainties $u(\epsilon)$.

Angle of observation	Bare aluminium ϵ (25 °C) Foil 1	$u(\epsilon)$	PE foil “copper lacquered” ϵ (25 °C) Foil 2	$u(\epsilon)$	PE Foil “colorless lacquered” ϵ (25 °C) Foil 3	$u(\epsilon)$	Mesh reinforced ϵ (25 °C) Foil 4	$u(\epsilon)$
20°	0.023	0.022	0.052	0.022	0.038	0.023	0.145	0.021
30°	0.017	0.022	0.051	0.023	0.040	0.023	0.145	0.021
40°	0.018	0.022	0.053	0.022	0.040	0.022	0.162	0.022
50°	0.043	0.022	0.088	0.022	0.071	0.022	0.167	0.021
60°	0.041	0.021	0.094	0.021	0.078	0.022	0.186	0.021
70°	0.053	0.022	0.126	0.021	0.108	0.022	0.213	0.020
ϵ hem	0.039	0.019	0.085	0.022	0.070	0.022	0.179	0.020

Figure 8: Directional spectral emissivity of four types of thermal isolation foils – Bare aluminium (Foil 1), PE foil “copper lacquered” (Foil 2), PE foil “colorless lacquered” (Foil 3), and mesh reinforced foil (aluminium color) (Foil 4) – measured at a temperature of 25 °C and observed under an angle of observation of 20° with respect to the surface normal.

one angle of observation of 20°. The bare foil (Foil 1) has the lowest directional spectral emissivity and a spectrally flat emissivity structure. The two polyethylene foils (Foil 2 and Foil 3) have a very similar spectral structure and approximately equal emissivity values. Finally, the mesh reinforced foil (Foil 4), as expected, has the highest emissivity, slightly increasing its effective emitting properties due to the ribbed surface (Figure 4).

The numerical values of the directional total and hemispherical total emissivities in the wavelength range from 5 μm to 20 μm and at a temperature of 25 °C of all four samples are provided in Table 2 together with their corresponding standard uncertainties $u(\epsilon)$. The low absolute measurement uncertainties, which vary in the range from 0.019 to 0.024, were achieved by reducing the uncer-

tainty of the sample surface temperature, which strongly depends on the thermal contact between the sample and the heater and is the dominant uncertainty contribution to the evaluation of the radiation budget. It has been significantly improved with the development of the new vacuum sample holder, which allows maintaining a good thermal contact even with thermally expanding and crumpled surfaces.

5 Uniformity of reference foils

Additionally, we investigated the uniformity of the directional spectral emissivity of each foil. These measurements were performed according to the usual measurement scheme, but for an angle of observation of 20° only. For these measurements, a smaller aperture size of the vacuum FTIR-spectrometer corresponding to the field-of-view of 8 mm was chosen. It was sequentially placed at the three positions (circles in the inset in Figures 9 and 10). This was accomplished by mechanically moving the sample on the vacuum sample holder. The center of each circular measurement area was placed at a distance of 20 mm from each other, so it was ensured that the three areas of measurement did not overlap.

The spectrally resolved emissivity is separately plotted for each of the three positions with the range of the standard uncertainty ($k = 1$) given as shaded areas. It should be noted that the purpose of this measurement was to check the uniformity of selected foils and not to determine their emissivity, which may slightly differ from the previous results (Table 2). The resulting curves exhibit no significant deviations in the wavelength range from 5 μm to 20 μm for all investigated foils. The obtained differences are not significant within the range of uncertainty, and the selected foils can be considered uniform.

Figure 9: Uniformity of the bare aluminium foil (Foil 1, left) and the PE foil “copper lacquered” (Foil 2, right) in terms of emissivity, measured at a temperature of 25 °C and at three positions on the sample surface (circles in the inset). The ranges of uncertainty are shown as shaded areas.

Figure 10: Uniformity of the PE foil “colorless lacquered” (Foil 3, left) and the mesh reinforced foil (Foil 4, left) in terms of emissivity measured at a temperature of 25 °C and at three positions on the sample surface (circles in the inset). The ranges of uncertainty are shown as shaded areas.

6 Conclusion

Within the EMIRIM project the PTB has determined the emissivity on highly reflective insulation foils with low uncertainties traceable to the International System of Units. The newly developed sample holder allows the vacuum mounting of the sample. It provides an excellent thermal contact of the investigated foils with the heater and, with it, a significant reduction of the main measurement uncertainty caused by the uncertainty of the sample surface temperature. Furthermore, the previously developed and discussed multiple reflection method perfectly contributed to the aim of this project, being an accurate evaluation procedure for the highly reflective materials lo-

cated inside of the temperature stabilized spherical enclosure.

The directional total emissivities of four different commercial thermal insulation foils were measured at a temperature of 25 °C: the bare aluminium foil (Foil 1) showed a hemispherical total emissivity of 0.039; the PE foil “copper lacquered” (Foil 2) and the PE foil “colorless lacquered” (Foil 3) revealed 0.085 and 0.070, respectively; finally, the mesh reinforced foil (Foil 4) has the highest hemispherical total emissivity of 0.179. The achieved absolute emissivity uncertainties of about 0.02 are a significant improvement in comparison to the requirements described in the relevant sections of EN 16012 and EN 15976. The good uniformity of thermal contact over the entire sample surface,

in combination with the accuracy of the measurements, made it possible to investigate the quality of the uniformity of the foils. Spectrally resolved emissivity measurements at three positions on the optical surface of each foil did not show any significant deviations within the range of the standard uncertainty ($k = 1$).

The progress made in the emissivity measurements of highly reflective insulation materials will lead to a benefit in many industrial sectors. It will support the control of the thermal performance of insulation products as well as the further development of new high-performance thermal insulation systems based on multi-low-emissivity-foils. On the other hand, the end-users from many industries will be able to perform reliable emissivity measurements with commercially available instruments at lower costs and with the validated uncertainties. New measurement procedures, including a new calibration procedure, which will be based on the results presented in this work, will allow measuring the hemispherical total emissivity of the reflective thermal insulation products with an uncertainty below 0.03.

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