EMIRIM

Improvement of emissivity measurements on reflective insulation materials

Publishable Summary for 16NRM06 EMIRIM
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Overview

Reflective foil insulations are used in the aerospace, transportation, and power generation industries where protection from thermal radiation is required and in thermal insulation systems for buildings where the EU conformance is required to Directive 2010/31/EU. The European standard EN 16012 describes methods for determining the thermal performance of reflective insulation products for buildings which manufacturers must declare. However, the standard has limited scope due to difficulties in obtaining reliable results for very low emissivity foils from different measurement techniques. Poor technique selection by insulation manufacturers can lead to inaccurate declarations of product properties. This project has developed SI traceable insulation foil emissivity measurements with 0.03 uncertainty which have great potential for inclusion in a revision of EN16012. This will help end users to select appropriate measurement methods more easily during development and certification of new and more efficient reflective insultation products.

Need

Reflective foils are used as heat protection screens and materials in many other applications apart from building insulation, e.g. in aircrafts, land vehicles, boats, space crafts, nuclear power generation, packaging, satellites and for protection of people exposed to intense thermal radiations. The principle of reflective insulation relies on the high sensitivity of the radiant heat power exchange between two surfaces not in contact to emissivity. The lower the emissivity of the surfaces, the lower the exchanged radiant power will be. In order to conform to Directive 2010/31/EU on the energy performance of buildings, producers of reflective insulation products must declare values of total hemispherical emissivity for the product's external surfaces. Declared emissivity values are used, in accordance with standards EN 16012 and ISO 6946, to calculate the thermal resistance of the insulation system under the condition of use. A comparison of measurement techniques organised by the standardisation group CEN/TC89/WG12, responsible for defining test methods and declaration rules for the thermal performance of reflective insulation products, showed high discrepancies with total hemispherical emissivity results ranging from 0.02 to 0.08 on the same reflective foil. These discrepancies can lead to the inadvertent under declaration of emissivity for reflective foils. The comparison included "integrating sphere" instruments and commercially available portable instruments (reflectometers). The sources of discrepancies were not explained, but likely sources were the geometrical, thermal and optical configurations of the measuring instruments and the type of reference sample used for calibration. Being unable to show that the measurement techniques evaluated were reliable when the emissivity is believed to be less than 0.05, CEN/TC89/WG12 set a limitation in EN 16012 that any 'measured' value of emissivity less than 0.05 has to be rounded up to 0.05. This limitation hampers new product development and market innovation since manufacturers cannot achieve the financial return from their investments in products with superior emissivity values below 0.05. To resolve this situation CEN/TC89/WG12 expressed a need for improvements to the accuracy of emissivity measurements of reflective foils used in insulation products. This project has addressed this by analysing carefully the sensitivities of the measurement techniques to the peculiarities of reflective foils, by defining the appropriate types of materials to be used for calibration, by improving reference techniques for calibration of the standards and by producing good practice guides for calibration of the end-users measuring instruments and for emissivity measurement on reflective foils. Recommendations were also given to CEN/TC89/WG12 for improvement of standard EN 16012.

Objectives

The overall aim of the project was to improve the measurement techniques recommended by the standards EN 16012 and EN 15976 and applied by end-users for the measurement of total hemispherical emissivity of reflective foils used in “reflective insulation” in order to obtain an uncertainty below 0.03 for emissivity below 0.1.

The specific objectives of the project were:

1. To analyse and test the different techniques and instruments used by end-users to characterise reflective insulation products. The sensitivity of these techniques in relation to the specificities of the reflective foils (specularity, angular diffusion, transparency, spectral properties, thermal inertia and non-flatness of surfaces) will be investigated to enable the definition of the most appropriate types of reference samples for ensuring traceability.
2. To improve and validate reference techniques based on different principles of measurement from at least two NMIs in Europe. The reference techniques will be able to measure total hemispherical emissivity below 0.1 with an uncertainty below 0.02. They will be applicable to materials with different ratios of specular reflectance/hemispherical reflectance.
3. To build new competencies in Metrology Institutes in order to produce appropriate calibrated reference samples for characterising end-users instruments and for ensuring traceability of measurements, via calibration and measurement procedures developed in the project. Calibrated reference samples will also be produced for partners involved in the measurement techniques characterisation.
4. To establish calibration and measurement procedures enabling the end-users to perform emissivity measurements on reflective foils with an uncertainty below 0.03 for emissivities below 0.1.
5. To participate to the revision of EN 16012 and EN 15976, via the provision to CEN/TC 89/WG12 and CEN/TC 254/WG14 of amendments based on the technical results of the project. To communicate technical reports and guidelines on the calibration and use of end-user techniques to CEN/TC 89/WG12 and CEN/TC 254/WG14. To disseminate the technical results of the project to the wider scientific and industrial community.

Progress beyond the state of the art

<u>Analysis and tests of the techniques used by end-users</u>

Prior to the project, the sensitivities to the peculiarities of reflective foils of the main end-user techniques for determining emissivity of reflective foils (integrating sphere systems, commercial emissometers) were not well known and a reliable assessment of uncertainties was necessary to reduce result variability. This project allowed the detailed characterisation of several industrially used instruments (integrating spheres and TIR100-2 emissometers) and produced reliable assessments of measurement uncertainties, coupled with a proposal for improved calibration and measurement procedures for low emissivity foils.

<u>Improvement and validation of reference techniques</u>

Prior to the project, no validated reference technique was available for the measurement of total hemispherical emissivity of low emissivity surfaces with an uncertainty below 0.02; the best-achieved uncertainties were around 0.03. This project’s adaptations, developments and improvements were made to existing reference techniques based on two different principles: calorimetry and measurement of total directional emissivity. These developments have allowed the measurement of total near-normal emissivity and total hemispherical emissivity with uncertainties of 0.02 on solid materials or foils with very low emissivities and various angular distributions of reflection.

<u>Building new competencies in Metrology Institutes for the production of appropriate calibrated reference samples</u>

In this project solid reference samples with low emissivities and various angular distributions of reflected radiation were produced using existing techniques (metal mirrors, very diffusing high reflective surfaces) or using a new technique based on precision machining of specifically structured surfaces on aluminium alloy.

<u>Establishment of calibration and measurement procedures for end-users</u>

The metrological characterisation of end-users measurement techniques have enabled us to establish calibration and measurement procedures for end-users and have been incorporated into guidelines for instrument calibrations and improved emissivity measurement of low emissivity foils with integrating spheres and TIR100-2 emissometers. The procedure for calculation of total hemispherical emissivity from the measured total near-normal emissivity, absent from standard EN 16012, has been included in this guideline.

Results

Analysis and tests of the techniques used by end-users

Measurements of the angular diffusion of the radiation reflected by reflective foils produced for the project were performed. The angular diffusion is highly correlated to the smoothness of the foil, from ± 2 to ± 3 degrees around specular direction for smooth foils to ± 30 degrees for a very structured foil (mesh for reinforcement). Those results show that the angular diffusions of foils are highly variable. Therefore, the emissivity measurement instruments should have a low sensitivity to the angular diffusion of the radiation reflected by the sample.

Emissivity measurement instruments mostly used by EU end-users are the TIR100-2 emissometer and integrating spheres associated to Fourier transform spectrometers; the two techniques are recommended in standard EN 16012. A detailed metrological characterisation of those techniques was performed. Detailed measurements of the angular variation of sensitivity of an integrating sphere were performed. The results obtained showed clearly that an integrating sphere can generate relative errors of a few percent on the reflectance measured when measuring the reflectance of a highly reflective sample. The error depends on the geometrical configuration of the incident beam, on the integrating sphere geometry configuration, on the surface tested and on the type of standard used for calibration. This finding highlighted the importance of the optical configuration when using integrating spheres.

The uncertainty budgets were established for measurements of total near-normal emissivity of reflective foils. For integrating spheres with appropriate optical configuration, the main source of uncertainty is the uncertainty on the total near-normal emissivity of the standard used for calibration. The limited sensitivity to the angular diffusion of the sample was demonstrated by comparing results obtained on a specular sample (Gold mirror) and on a diffusing sample (Infragold®), the two samples being calibrated. When the optical configuration is correct, an integrating sphere calibrated using a specular standard, can measure the total near-normal emissivity of a low emissivity polymer foil with an uncertainty around 0.025 (k=2). A TIR100-2 emissometer, calibrated in total near-normal emissivity using a metal mirror, can measure the emissivity of a low emissivity polymer foil with an uncertainty between 0.025 and 0.03 (k=2). The uncertainties were validated by a comparison on several low emissivity foils; the results from integrating spheres and TIR100-2 emissometers were compared to results from the reference spectroradiometric technique at PTB.

It was noticed that one of the integrating spheres characterised in the project had a poor optical configuration. The integrating sphere is integrated in an accessory commercialised to be fitted in a Fourier transform spectrometer and the optical configuration is not flexible. The defect was detected when measuring the emissivity of a low emissivity diffusing surface (Infragold® coating) by reference to a gold mirror, the emissivity result for the diffusing coating was lower by 0.022 than the reference value. This demonstrated that a way of detecting defects for an integrating sphere or other instrument is to have two low emissivity calibrated standards, one specular and one diffusing, and to perform emissivity measurement on one standard by reference to the other one.

This objective has been fully and successfully completed.

Improvement and validation of reference techniques

Three reference setups were adapted, improved or developed for measurement of total directional emissivity or total hemispherical emissivity with low uncertainty. PTB adapted a directional spectral emissivity measurement setup for the measurements on foils over the angular range 0° to 70°. A new sample holder based on the principle of vacuum mounting of the foils was built. It allows measurements of angular resolved emissivity on reflective foils with an uncertainty better than 0.02. The total hemispherical emissivity of foils is obtained by spectral integration with an uncertainty below 0.025. LNE built a new thermal guarded sample heating system for the reference calorimetric technique. The system is designed for heating two discs of the same material with a diameter of 100 mm. The calorimetric technique allows the direct measurement of total hemispherical emissivity with uncertainty below 0.02. One advantage of the calorimetric technique is that it measures directly the emissivity for all wavelengths and all directions. DTU developed a new setup based on the measurement of total directional emissivity. The directional radiance of the sample heated in an isothermal enclosure is measured with a wide band radiometer. The uncertainty is around 0.015 for smooth foils; the angular range is limited at 50° from normal to the sample surface.

The reference techniques were compared for total near-normal or total hemispherical emissivity measurements on low emissivity surfaces. The results were in good agreement with maximum differences below 0.015.

The spectroradiometric setup from PTB gives the angular distribution of total directional emissivity for angles from 10° to 70°, which allows the calculation of total hemispherical emissivity. The setup was used to quantify the uncertainty related to the model used to calculate the total hemispherical emissivity from the total near-normal emissivity. It was found that an uncertainty about 0.025 should be considered specifically for the calculation of total hemispherical emissivity using the model given in standard EN 12898. This uncertainty reflects the current level of knowledge and is based on few experimental results with quite large uncertainties. The spectroradiometric setup from PTB was also used for calibration of the standards used for the characterisations of the integrating spheres and TIR100-2 emissometers involved in the project.

This objective has been fully and successfully completed.

Building new competencies in Metrology Institutes for the production of appropriate calibrated reference samples

A tentative work at PTB to produce samples with "medium" emissivity was not successful. The surfaces were structured by laser ablation and gold coated giving emissivity about 0.2 but non-uniform. An important objective of the project was to produce solid reflective samples with angular diffusions at reflection similar to the ones of reflective foils. Three Metrology institutes (LNE, Aalto and PTB) and a technical research institute (FhG) collaborated to achieve this objective. The angular distribution of reflection of four reflective foils were measured by LNE using an existing goniometer and a new measurement procedure. For smooth reflective foils, the study done by Aalto to find by optical modelling the texture of a solid surface fitting the angular diffusions of smooth foils gave a structure at the dimensional level of roughness (Ra = 0.25 µm) not easily produced. For a very structured foil ("mesh reinforced foil"), Aalto was able, by adapting the optical modelling technique, to find a structure fitting the diffusion of the foil and to generate. a theoretical surface structure with bell-shaped hollows arranged in a hexagonal configuration. Two samples with corresponding coarse and fine structures were produced by precision milling followed by a light polishing and their total directional emissivity were measured around 0.05. The measured angular distributions of the directional spectral emissivities is similar to that of a reflective foil. The use of precision milling is innovative for producing surfaces with tailored radiative properties and is promising for producing solid surfaces with specific angular diffusions or with "medium" mean emissivities by machining tailored "cavities". Solid samples with tailored medium emissivities would be useful for calibrating and comparing emissivity measurement techniques. Additionally, nine sets of preliminary reference samples were produced, calibrated and distributed to partners performing emissivity measurements in the project.

This objective has been successfully completed.

Establishment of calibration and measurement procedures for end-users

The characterisations of integrating spheres and TIR100-2 emissometers showed that low emissivity metal mirrors are appropriate for calibration and that for integrating sphere particular attention should be paid for the optical configuration. For TIR100-2 instruments the calibration and measurement procedures are not changed, they were already calibrated at low emissivity with a metal mirror and the recommendations given by the manufacturer (INGLAS) are appropriate. The metal mirror and "black surface" used for calibration should be calibrated with traceability to SI. For infrared integrating spheres, a particular measurement procedure must be applied to correct for sensitivity variation related to the reflectance of the sample. Two good practice guides were written on project with recommendations for end-users with a particular focus on the measurement procedure and on the control of the optical configuration for integrating spheres. The application of recommended procedures allow the measurement of total near-normal emissivity of low emissivity foils with an uncertainty below or around 0.03 (k=2).

This objective has been fully and successfully completed.

Participation to the revision of EN 16012

The standard EN 16012 was analysed in detail at the start of this project. The main modifications proposed and comments are: integrating spheres should not be considered as reference techniques, it is not mentioned that the parameter measured with integrating spheres and TIR100-2 emissometers is the total near-normal emissivity and that the total hemispherical emissivity, the parameter to be declared for an insulation product, must be calculated, the model of calculation of total hemispherical emissivity defined in standard EN 12898 - March 2019 should be mentioned in EN 16012 so that all users use the same model, an uncertainty must be considered for the model for calculation of total hemispherical emissivity, the modification of the limit at 0.05

for the lowest emissivity value declarable for a thermal insulation product is not proposed by EMIRIM consortium because the overall uncertainty on the calculated total hemispherical emissivity is around 0.04 (k=2).

The recommendations were sent to the convenor of CEN/TC89/WG12 through draft improved versions of related sections of the standard and remarks for improvement of those sections. The recommendations for improvement of EN 16012 were also presented in detail and discussed during a workshop held in November 2020 and involving end-users from thermal insulation materials industry and some members of CEN/TC89/WG12.

Impact

Initial results from the project were presented at the 20th Symposium on Thermophysical Properties (United States, June 2018) via an oral presentation and intermediate results at the Tempmeko & TempBeijing 2019 Symposium (China, June 2019) as an oral presentation. At the 19th International Congress of Metrology (France, 2019) the project gave an oral presentation as well a poster presentation to promote project outcomes.

Two published open access papers present the results of a detailed characterisation of the TIR100-2 emissometer performed by PTB and the collaborative work performed to define the shape of the surface for production of solid samples with tailored angular diffusions of reflected radiation. Another paper on the comprehensive characterisation of the TIR100-2 emissometer giving a detailed uncertainty budget for emissivity measurements on reflective foils has been submitted for publication.

A project workshop was held in November 2018 to deliver training on '*Improvement of Emissivity Measurements on Reflective Insulation Materials'* to end-users from the thermal insulation industry, researchers and members of certification bodies as well as consortium members. The general content of the project and intermediate results on characterisations of end-user measurement techniques were presented and discussed. A second workshop in November 2020 for end-users from the thermal insulation industry, scientists and researchers presented project results, uncertainty budgets and recommended procedures for calibration and use of end-user emissivity measurement techniques. The proposals for improvement of standard EN 16012 were also presented in detail and discussed as well as the procedure for calculation of the total hemispherical emissivity from the measured total near-normal emissivity.

Impact on industrial and other user communities

The producers or distributors of thermal insulation materials for building wanting the "low emissivity effect" of their products to be considered by designers must declare a total hemispherical emissivity value to comply with EU Directive 2010/31/EU. Therefore, the project results benefit the producers of low emissivity insulation products, producers of flexible sheets for waterproofing buildings and organisations testing and certifying these products. End-user good-practice guides developed by the project are available from the project website.

The improvement of uncertainties for the measurement of low values of total hemispherical emissivity will facilitate the development of new high-performance thermal insulation systems like multi-layer-insulations (MLI) based on a stack of low emitting screens. These have various industrial and research applications including those where space is limited and weight penalties high such as satellites, space vehicles, aircrafts or applications where stacks of low emitting screens under vacuum can be used such as cryogenics and storage of liquefied gases. The improved knowledge of the performance and limitations of measurement techniques used industrially and the availability of appropriate calibrated samples will help end-users in the automotive, aeronautical and the space industries where reflective foil insulation is critical for the protection of people and infrastructures from intense thermal radiation. In these industries reliable emissivity measurements are often performed using commercially available instruments, and measurements with validated uncertainties will lead to improve demonstrations of products matching specifications. Therefore, many industrial sectors will benefit from the progress made in low emissivity measurements on reflective building insulation materials.

Impact on the metrology and scientific communities

In addition to presentations at scientific conferences and publications in scientific journals, this project and particularly the improvements developed at the reference level in National Metrology Institutes were presented to the Annual conference of the AK Thermophysik – GEFTA (Germany - 2018), EURAMET TC for Thermometry - Thermophysical Quantities of Materials WG and BIPM - CCT Task Group for Thermophysical Quantities (CCT-TG-ThQ).

This project has enabled the extension and improvement of three NMI measurement service capabilities for the calibration of emissivity standards with low uncertainties, that will facilitate the reliable validation and use of commercial emissivity measurement instruments in research and industry. The improved capabilities of PTB and DTU to measure, with low uncertainties spectral or total directional emissivities, will allow calibrations and measurements of emissivity at variable angles. The capabilities of most research laboratories or end-users are limited to measurement of spectral or total directional emissivity for a single direction close to normal. Therefore, PTB and DTU will be able to provide end-users with precise results at variable angles for applications in scientific research where angular emissivity values are required for precise heat balance calculations or applications of pyrometry or thermography for non-contact temperature measurements at non-normal directions. The calorimetric technique improved at LNE is rare even among research laboratories worldwide. It has the capability to directly measure the total hemispherical emissivity (all wavelengths, all directions) with a low uncertainty particularly for low emissivities. The improved calorimetric technique will be particularly useful for testing non-specular surfaces with very low total hemispherical emissivities. Potential applications include satellite measurement instruments that must be protected from parasitic thermal radiation.

Impact on relevant standards

The main objective of the project was to support CEN/TC 89/WG 12 to improve EN 16012 for the measurement of total hemispherical emissivity of low emissivity foils. Remarks and improved versions of the technical sections of the standard related to emissivity measurements were written and sent to convenor of CEN/TC 89/WG12 for potential incorporation into a revision of EN 16012 which will form a new EN standard for insulation products. The project addressed a lack of precision in the definition of the emissivity measured with the measurement techniques recommended in the standard which created the risk of manufacturers declaring undervalued emissivities for low emissivity insultation foils. It also addressed the absence that the total hemispherical emissivity is the parameter to be declared in specification for a thermal insulation product. The integrating sphere is mentioned in EN 16012 as a reference measurement technique, but its use with poor optical configurations may produce errors especially for reflective foils. This may inadvertently lead end-users to select a measurement technique that is quite difficult to use to obtain reliable results. Therefore, a recommendation for explaining the technical risks related to the integrating sphere technique was also provided.

In summary, the knowledge transferred from EMIRIM project to CEN/TC 89/WG12 should facilitate greater accuracy in emissivity measurement made by users applying standard EN 16012 and a reduction in the risk of measurement errors.

Good-practice guides for calibration and for emissivity measurement with integrating spheres and TIR100-2 emissometers were communicated to the convenor of CEN/TC 89/WG 12 and were presented and discussed during a workshop held in November 2020 involving end-users from the thermal insulation industry and members of the standards working group. These good-practice guides are available from the project website.

Longer-term economic, social and environmental impacts

The improvement of emissivity measurements on insulation products will help to increase the efficiency of thermal insulation systems for buildings, industries and home appliances. More reliable emissivity data will allow accurate calculations of heat transfer coefficients. In direct consequence, the results of the project will help in the reduction of energy consumption. The project will help to enhance consumer protection through the availability of insulation products with more accurate and validated declared performance values. More reliable and traceable emissivity measurements using commercially available instruments will help reduce the cost of product development and increase their performance. Reflective low emissivity surfaces are also used to protect people or structures from intense radiation sources such as a very hot surfaces; the reflective surface is then used in association with an insulation material or alone as a radiation screen. Typical applications are in the steel industry, glassmaking or firefighter protection. Radiation screens are also used widely to protect structures from thermal radiation emitted by hot elements. For example, from hot exhaust parts in vehicles, or the protection of critical elements or cables from fire. A particular area where a low emissivity is required is solar thermal collection, the lower the emissivity the higher the efficiency due to the reduction of thermal losses. For high tech solar absorbers low uncertainty on total hemispherical emissivity (around 0.02) is required for validating absorbers over long periods of use (ageing effect on emissivity) and for making investment decisions.

The improvements made and knowledge gained in this project for performing emissivity measurements will have positive applications in various areas particularly those related to energy saving and renewable solar powered energy production.

List of publications

Kononogova, Elena, Adibekyan, Albert, Monte, Christian and Hollandt, Jörg (PTB), Characterization, calibration and validation of an industrial emissometer, 2019, Journal of Sensors and Sensor Systems 8 (2019) , 233-242. https://www.j-sens-sens-syst.net/8/233/2019/

D. Lanevski, F. Manoocheri, A. Vaskuri, J. Hameury, R. Kersting, C. Monte, A. Adibekyan, E. Kononogova and E. Ikonen, "Determining the shape of reflectance reference samples for curved surface reflectors", Measurement Science and Technology, Volume 31, Number 5, 2020. doi.org/10.1088/1361-6501/ab68bf, https://iopscience.iop.org/article/10.1088/1361-6501/ab68bf

This list is also available here: https://www.euramet.org/repository/research-publications-repository-link/

<table>
<tr><td colspan="2">Project start date and duration:</td><td>01 June 2017, 42 months</td></tr>
<tr><td colspan="3">Coordinator: Jacques Hameury, LNE Tel: +33 1 30 69 12 18 E-mail: jacques.hameury@lne.fr
Project website address: http://projects.lne.eu/jrp-emirim/</td></tr>
<tr><td colspan="2">Chief Stakeholder Organisation:</td><td>CJHconsult Associates</td></tr>
<tr><td>Internal Funded Partners:
1. LNE, France
2. Aalto, Finland
3. DTU , Denmark
4. PTB, Germany</td><td>External Funded Partners:
5. FhG, Germany
6. FIW, Germany
7. IG, Italy
8. ZAE Bayern, Germany</td><td>Unfunded Partners:
9. ACTIS, France
10. INGLAS, Germany</td></tr>
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