Final Publishable JRP Summary for SIB52 Thermo
Metrology for thermal protection materials

Overview

Recent advances in thermal protection used in the aerospace, oil and gas industries include the development of polymers, aerogels and fibrous based composite systems. These new materials are thinner, lighter and stronger, and provide a thermal performance several times better than conventional insulation materials.

As part of EU No. 305/2011 (regulation for construction products), the EU has introduced material performance regulations to ensure that materials can be robustly tested against well-defined criteria and to ensure that performance matches specifications, but current testing capabilities are unable to meet these requirements. There is a need for better measurement techniques for thermal protection materials that conform to the EU standards, so ensuring safety and quality across manufacturers, thus saving lives, saving energy and reducing costs.

This project established a framework for thermal conductivity measurements of thermal protection materials up to 650 °C, to support the implementation of the EU regulations for construction products. The improved measurement technique enables designers of fire engineering for buildings (Structural Eurocodes EN 1990 to EN 1999) and transportation safety systems, such as engine housing of aircrafts, to select the best performing thermal protection materials. The application of the best multifunctional thermal protection materials is able to both reduce energy consumption during normal use, and to protect structural integrity during a fire situation. Therefore, it will ensure that the load-bearing capacity of structures will be maintained for a long enough time to allow the evacuation of people.

Need for the project

Reliable thermal conductivity measurement data is important for fire safety engineering and transportation safety systems to help reduce the severity of accidents and so save lives. Underperforming thermal protection products could lead to structures being exposed to over excess heat and causing failures of structual integrity and resulting in loss of life. The use of new polymer, aerogel and fibrous based composite materials with better heat resistant properties than conventional materials, such as fire bricks or boards routinely used in process plants, needs to be accurately tested and characterised at the temperature used in service, up to 650 °C.

Measurements of thermal conductivity have produced scatter of over 100 % for the new types of thermal protection material. This has lead to a lack of confidence and investment, an inability to demonstrate performance for certification, and costly full-scale testing during the development of aerospace components and fire safety systems for buildings and industrial processes.

The European Union has put into place new regulations in the Construction Products Regulation EU No. 305/2011 and mandatory product standards with the aim of making reliable thermal performance data available to industrial users. However, the level of agreement between reference laboratories at the outset of the project was outside the requirements of the new EU regulations. Implementation of these regulations urgently required the science underpinning thermal conductivity measurements to undergo a step-change improvement.

At the start of the project, many aspects of the Guarded Hot Plate technique were still not sufficiently advanced for high temperature measurements. These included the stability of temperature sensors and plate materials, and lack of reference materials. The final measured thermal conductivity value is very sensitive to the effects of the remaining measurement issues. Until they are resolved, thermal conductivity measurements within Europe are still a long way from meeting the requirements of industry and regulation.

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Thus there was a need for a thermal conductivity measurement infrastructure that addresses the whole traceability chain, to obtain better agreement between reference laboratories by establishing traceability and to improve industrial measurement techniques up to a target temperature of 650 °C.

Scientific and technical objectives

To address these requirements, the project focused on the traceable measurement and characterisation of modern advanced thermal protection materials. The project's top-level scientific objectives were:

1. To gain a better understanding of the limitations and uncertainties in industrial measurement techniques when used for measuring the thermal conductivity of advanced thermal protection materials.
2. The identification and development of appropriate techniques for the measurement of the thermal conductivity of advanced insulation and composites to provide traceability for industrial measurements.
3. The resolution of discrepancies in measuring advanced insulation and composites, aiming at agreement between the reference laboratories, which use Guarded Hot-Plates (GHPs), to be improved by a factor of three from 15 % to 5 %, thus enabling industry to meet the mandatory requirements of new EU regulations.

Results

High-Temperature Guarded Hot-Plate (HTGHP) technique is a steady-state absolute method that measures material thermal conductivity based on Fourier's Law. The determination of thermal conductivity by this method involves the measurement of the mean temperature difference between the opposite sides of parallel faced specimens at steady state condition when a constant, unidirectional heat flux density of known magnitude is passed normally through the specimen. Many of the measurement challenges involved with making thermal conductivity measurements arise from the complexity of heat transfer through porous insulation at high temperatures, and the requirement to confine small amounts of heat flux to pass through a defined 'metering' area within the specimen. Critical apparatus components require materials that have specific combinations of thermal properties and can remain dimensionally stable while being cycled to high temperatures. Temperature sensors need to pass through significant temperature gradients and therefore must have a minimum cross sectional area, which is in direct conflict with the normal approach to achieve a stable calibration.

1: To gain a better understanding of the limitations and uncertainties in industrial measurement techniques when used for measuring the thermal conductivity of advanced thermal protection materials.

Limitations and uncertainties in industrial measurement techniques such as laser flash technique, heat flow meter and HTGHP were investigated for their use in measuring the thermal conductivity of advanced thermal protection materials.

Investigations into the laser flash technique identified that it could be used for the measurement of thermal conductivity as an indirect technique for anisotropic materials (materials with a physical property which has a different value when measured in different directions) with the following severe limitations. For the laser flash technique the material must be able to be homogenised in the direction of heat transport. For an anisotropic material, the measurement must be performed along a principal axis of the material. For composite materials the density of fibres or other "singularities" must be uniform at the scale of the thickness of the specimen.

An analytical mathematical model was developed for the calculation of the standard-uncertainty of the thermal conductivity measured using a Guarded Hot-Plate (GHP). A finite-element model for heat transfers in a double-specimen circular GHP was also developed. This model allowed the effect of each parameter to be studied, and it helped to quantify the uncertainties that were not easily calculable.

An uncertainty analysis of the two selected industrial HTGHPs: the "GHP Titan" manufactured by the Netzsch Gerätebau GmbH operated by LNE, and "GHP-750-250", an in-house build instrument by Forschungsinstitut für Wärmeschutz e.V. was reported. For example, for thermal conductivity measurements of high density calcium silicate (HDCaSi-N) specimens at 500 °C the expanded relative measurement uncertainties ($k=2$) for the "GHP Titan" is 10 %.

The project achieved its objective to gain a better understanding of the limitations and uncertainties in industrial measurement techniques used for measuring the thermal conductivity of advanced thermal protection materials, and the outcomes will help industrial and academic users to select appropriate thermal conductivity measurement techniques for research and design of products.

2: The identification and development of appropriate techniques for the measurement of the thermal conductivity of advanced insulation and composites to provide traceability for industrial measurements.

The HTGHP technique was improved at NMI partners. An in-house-made HTGHP at LNE was upgraded for the measurement of thermal conductivity in the temperature range 70 °C to 800 °C. Systematic performance checks of the in-house made and the commercial 'GHP Titan' as per objective 1 were carried out. The assessments of uncertainties of the HTGHP measurements gave expanded relative uncertainties of 4 to 5 % from 150 °C to 650 °C for the in-house HTGHP, and 7 % to 10 % from 150 °C to 500 °C for the commercial HTGHP. The in-house-made HTGHP has become the National Standard apparatus in France.

A new technique for high-temperature thermal conductivity measurements was developed at BFKH. The assessments of measurement uncertainties gave expanded relative uncertainties of 5 % to 6 % from 150 °C to 650 °C for the High-Temperature Thermal Conductivity Measurement Apparatus (HTTCMA).

CMI completed the design and construction of their first HTGHP functioning up to 800°C, and also performed their first inter-comparison measurements of thermal conductivity of HDCaSi-N specimens up to 650 °C. The measured data from CMI and NPL deviate within 5% from the reference value up to 650°C.

NPL improved and carried out an initial validation of the new National Standard Small Guarded Hot Plate (SGHP) for testing thin specimens of polymers and composites. Experimental investigations of thermal interface materials for reducing thermal contact resistance demonstrated that the best alternative to silicone greases would be a textured indium foil product. It offered advantages in terms of quick application, protecting the apparatus plates from damage and was effective up to 100 °C.

The project collated a comprehensive list of factors contributing to uncertainties in HTGHP measurements enabling the closer comparison of measurement capabilities among different laboratories.

The project achieved the objective of developing thermal conductivity measurement techniques for advanced insulation and composites to provide traceability. New and improved national standard level metrological instrumentations, such as HTGHPs, SGHP and HTTCMA for thermal conductivity measurements are now established at participating NMIs. They are now able to provide traceable thermal conductivity measurements of thermal protection materials for industry.

3: The resolution of discrepancies in measuring advanced insulation and composites, aiming at agreement between the reference laboratories, which use GHPs, to be improved by a factor of three from 15 % to 5 %, thus enabling industry to meet the mandatory requirements of new EU regulations.

Before the start of the project, there was no thermal conductivity reference material available to investigate the causes of the discrepancies between reference laboratories using GHPs.

The project developed a 'first-of-its-kind' insulation/refractory high-temperature thermal conductivity reference material. This began with a survey of potential candidate materials, which led to a short list of four candidates. Initial tests on the four candidate materials were completed, during which a new approach was developed to combine both thermal metrology and material science in the assessment of materials. A high density calcium silicate (HDCaSi- N) was validated for use as a high-temperature reference material with low thermal conductivity. The specimens were robust, mechanically and chemically stable and uniform and heat treated up to 850°C prior to detailed characterisations.

The results of thermal conductivity tests on 5 out of the 15 selected HDCaSi-N specimens showed that their thermal conductivity values differed by less than 2 % from the average values measured in the temperature range 150 °C to 650 °C. This is less than half of the measurement uncertainty of the HTGHP.

Inter-laboratory comparisons on HTGHPs were undertaken using HDCaSi-N reference specimens. The inter-laboratory comparisons (between NPL, LNE and collaborator FIW) showed that the measurement data deviated from the reference value within 5 % up to 450 °C, and within 6.5 % up to 650 °C. Unfortunately, the heater plate of the HTGHP at CMI was found to be significantly warped and could not be included. Some consistent discrepancy was seen in the HTTCMA, and the cause for this needs further investigation. However, thanks to the new HDCaSi-N reference specimens, it could be ruled out that the difference was caused by a difference in the test specimens.

The objective of 5 % agreement between reference laboratories was achieved up to 450 °C. From 450 °C to the revised maximum temperature 650 °C the agreement was 6.5 %, slightly above the 5 % target.

Actual and potential impact

The outputs from this project will benefit insulation manufacturers, standards organisations (such as CEN/TC89/WG14) and engineering designers working on aircraft and buildings.

Dissemination of results

5 peer-reviewed papers were published in journals such as the International Journal of Thermophysics (IJOT) and the Proceedings of the International Thermal Conductivity Conference (ITCC). Two additional papers were submitted to the IJOT for publication as peer-reviewed papers.

The project partners delivered 18 presentations at conferences including TEMPMEKO 2013, 32nd ITCC conference, the 20th European Conference on Thermophysical Properties (ECTP), the 19th Symposium of Thermophysical properties, IMEKO 2015 and TEMPMEKO 2016 Conference.

The project's results were also disseminated amongst reference laboratories and stakeholders through 7 training workshops on HTGHP uncertainty budgets and high temperature thermal conductivity measurement, 2 training videos on thickness and flatness measurement of materials (available on the project website) and 4 technical assessment visits. This enabled the reference laboratories, e.g. CMI, BFKH, FIW, NPL and LNE to improve their measurement facilities and capabilities, and to provide more comprehensive measurement services to help European manufacturers to meet mandatory CE marking requirements in the new Construction Product Regulations (EU No. 305/2011).

Knowledge of technical improvements of HTGHP technique was also disseminated to the wider scientific community via an external Workshop at the 32nd ITCC conference and scientific publications.

Contribution to standards

A European Standard Committee CEN/TC 89 established a new Working Group (WG14) to follow the work in the project so that the knowledge generated in the project could be quickly transferred in the development of the new measurement standard, EN 15548-1. CEN/TC89 is responsible for the standardisation in the field of thermal performance of buildings and building components and the development of standards for the European Construction Products Regulations. All partners contributed to the first and second CEN/TC 89 WG14 meeting in reviewing the Annex-A (Limits for equipment performance and test conditions) of CEN/TS 15548-1(Thermal insulation products for building equipment and industrial installations — Determination of thermal resistance by means of the guarded hot plate method — Part 1: Measurements at elevated temperatures from 100 °C to 850 °C). The outputs of this project have been incorporated by CEN/TC 89 WG14 into the working draft standard document prEN 15548- 1. Once it becomes an EN standard, it will be part of the EU Construction Product Regulations. This standard will help test laboratories to improve the quality of their high-temperature thermal conductivity measurements using GHPs. It will also help instrument manufacturers to improve the design of their HTGHPs. Ultimately, it will help the implementation of the European Construction Product Regulations.

Early impact

Before the start of the project, NPL was the only European NMI that had a validated National Standard level HTGHP with ±5 % ($k=2$) expanded relative uncertainty at 800 °C. The measurement infrastructure developed in this project has resulted in increased numbers of European NMIs being able to perform thermal conductivity measurements using HTGHP at higher temperatures. The project went beyond the state of the art by achieving new national standard metrological instrumentation for thermal conductivity measurements:

- LNE now have a validated National Standard level HTGHP (objective 2)

- BFKH now have a National Standard level HTTCMA up to 850 °C (objective 2)
- CMI completed the design and construction of their first HTGHP functioning up to 800 °C (objective 2)

The new high density calcium silicate (HDCaSi-N) thermal conductivity reference materials developed in objective 3 have been made available to European reference laboratories and an industrial laboratory, the collaborator FIW, who provide testing services for insulating materials in the construction industry, for comparisons of measurement techniques and for improvement of European equivalence. The provisional results of the inter-comparisons on measurements of the new reference material HDCaSi-N specimens have shown that the measured thermal conductivity data deviated from the reference values within 5 % up to 450 °C and within 6.5 % up to 650°C. The good agreement in measurements will enable laboratories to confidently provide more comprehensive high quality thermal conductivity tests for industry than were previously possible.

Good practice guides covering (i) the design and validation of the HTGHP (objective 2) and (ii) the use of transient and other industrial methods (objective 1) have been made available to NMIs who maintain or develop National Standard instruments, as well as to instrument manufacturers and end users from industry and academia. They are available via the project web site.

Potential future impact

New National Standard level HTGHPs developed in the project now provide more comprehensive high quality thermal conductivity measurements, enabling European industry to meet the mandatory European Construction Product Regulations (EU No.305/2011). Measurement demand is increasing, particularly from industries in Eastern Europe, as more and more international insulation manufacturers set up plants in the region. The new National Standard measurement facilities, e.g. the HTGHP developed at CMI will enable them to meet the measurement demands from industry.

The new infrastructure developed in this project will provide reliable thermal conductivity measurement data and this will enable the designers of fire engineering for buildings (Structural Eurocodes EN 1990 to EN 1999) and transportation safety systems (e.g. aircraft engine housing) to select the best performance thermal protection materials. Using the best multifunctional thermal protection materials not only reduces energy consumption during normal use, but also protects structural integrity during a fire situation. Therefore, it will ensure that the load-bearing capacity of structures will be maintained for a long enough time to allow the evacuation of people so will save lives.

Requirements for validated and traceable measurements for insulation materials are especially critical in advanced manufacturing industries. Traceable thermal conductivity data will enable engineering companies to rigorously demonstrate the performance of their new technologies to potential customers, which is now an essential part of the procurement supply chain within the energy, aerospace and defence sectors. The current lack of reliable thermal property data leads to either extra costs from over engineering when too much material is used or higher risk when too little material is used.

The new measurement infrastructure established in this project will provide a framework to enable the manufacturers of advanced thermal protection materials to have the confidence to invest in the development of new, innovative products and to be able to demonstrate their performance to engineering certification authorities.

List of publications

[1]. Ulf Hammerschmidt, Jacques Hameury, Radek Strnad, Emese Turzó-Andras, Jiyu Wu; Critical Review of Industrial Techniques for Thermal Conductivity Measurements of Thermal Protection Materials, International Journal of Thermophysics: Volume: 36, Issue: 7, Pages: 1530-1544, Published: JUL 2015. DOI 10.1007/s10765-015-1863-x (2015)

[2]. Jiyu Wu, Roger Morrell, Tony Fry, Sam Gnaniah, Dipak Gohil, Angela Dawson, Jacques Hameury, Koenen Alain, Ulf Hammerschmidt, Emese Turzó-András, Radek Strnad, Aleš Blahut; Provisional Assessment of Candidate High Temperature Thermal Conductivity Reference Materials in the EMRP 'Thermo' project, in Proceedings at the 32nd International Thermal Conductivity Conference and 20th

international Thermal Expansion Symposium (on 27 April to 1 May 2014, IN, USA), pp142-153, Purdue University Press, Oct. 2015.

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[4]. C. Stacey, A. J. Simpkin, R. N. Jarrett; Techniques for Reducing Thermal Contact Resistance in Steady-State Thermal Conductivity Measurements on Polymer Composites, International Journal of Thermophysics: Volume: 37, Issue: 11. DOI 10.1007/s10765-016-2119-0, Oct. 2016.

[5]. Jiyu Wu, Roger Morrell, Crispin Allen , Petra. Mildeova, Emese Turzó-András, Ulf Hammerschmidt, Eva Rafeld, Aleš Blahut, Jacques Hameury, Characterisation of high-temperature thermal conductivity reference materials, Int J Thermophys (2017) 38:66 DOI 10.1007/s10765-017-2200-3. (2017).

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