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## 1 Executive Summary

### Introduction

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.

### The Problem

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. Implementation of these regulations urgently requires the science underpinning thermal conductivity measurements to undergo a step-change improvement.

Before the start of the project, there were no thermal conductivity reference materials available for investigating the causes of the disagreement between the reference laboratories. The current standard documents do not address the challenges of thermal conductivity measurements at elevated temperatures. The CEN Technical Committee 89 set up Working Group 14 to prepare proposals for resolving accuracy and reproducibility problems with measurements on high temperature insulation, and to develop CEN/TS 15548-1 into a new standard specifically to address thermal conductivity measurements at temperatures up to 850 °C. However, the lack of knowledge about measurements at high temperatures prevented CEN/TC89/WG14 from completing the development of the new EN standard.

Today's thermal modelling capabilities provide an opportunity to design industrial systems far more efficiently, but a lack of accurate thermal properties data for materials was the main factor limiting the predictive power of thermal modelling. High quality data must be made available to allow designers to precisely engineer systems, rather than just adopting a costly over-engineering approach. The existing metrological techniques for temperatures up to 850 °C are sound and provide a good foundation for development, but there were many aspects that were still not sufficiently advanced. The final measured thermal conductivity value is very sensitive to the effects of these remaining metrology issues. Until these issues are resolved, thermal conductivity metrology within Europe is still a long way from meeting the requirements of industry and regulation. It is essential that these issues are resolved through improved measurement technology.

### The Solution

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.

### 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 in the region.

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.

## 2 Project context, rationale and objectives

### 2.1 Project context and rationale

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 structural 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 led 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.

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.

### 2.2 Objectives

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

- To determine the effect of radiant heat transfer on thermal conductivity measurements at high temperatures. Heat transfer within many insulation products includes a combination of all three modes (conduction, convection, radiation), but there is currently insufficient knowledge of the

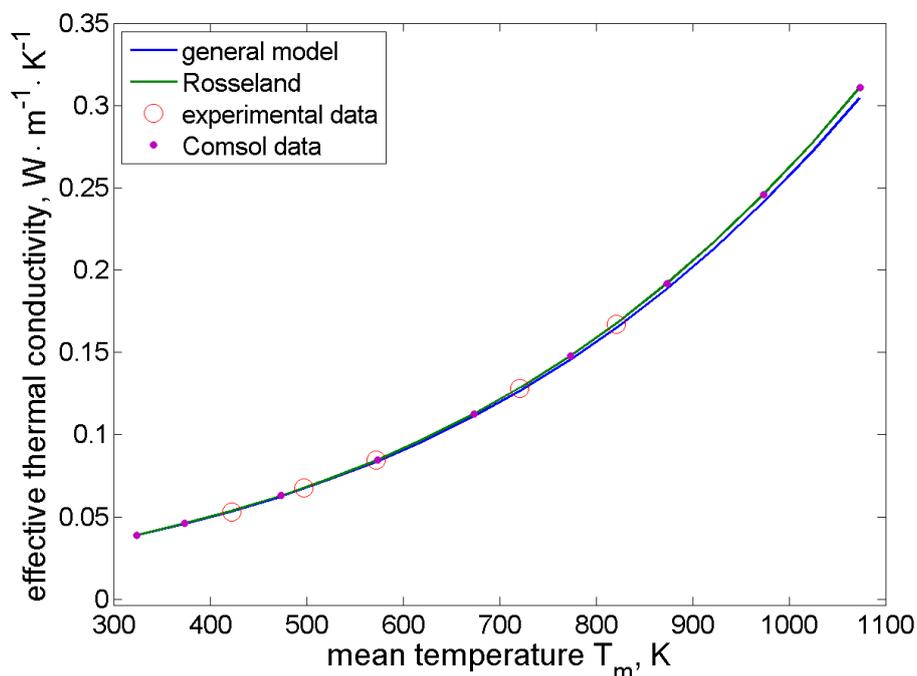
magnitude of radiant heat transfer. This knowledge is required to be able to specify the tolerance in surface emissivity of measurement apparatus components, to improve measurement uncertainties, to investigate the anomalies between reference laboratories, and to finalise the new European measurement standard for thermal conductivity measurements of insulation at high temperatures.

- To improve thermal guarding systems and temperature sensor arrangements to obtain temperature measurements that are more representative of the specimen boundary conditions within regions of heat flux. Temperature sensors used in these applications need to be as thin as possible to minimise distortion of the temperature field, but they also need to have both a high level of batch agreement and long-term calibration stability. Newly developed thermocouples need to be tested for these specific measurement applications.
- To identify and characterise coatings that can withstand abrasion from contact with the specimen; withstand thermal cycling up to 850 °C and remain securely bonded to plate materials; and provide a consistent high emissivity (0.8 or higher is required) with a specified value/tolerance. We will also identify new heater plate materials that will provide improved dimensional stability despite repeated temperature cycling, while still providing the required temperature uniformity across critical components.
- To overcome thermal contact resistance issues in thin sections of composite materials. Existing steady-state techniques cannot provide reliable thermal measurements due to distorted heat flux. This is caused by differential expansion and temperature sensors that do not represent the specimen surfaces without affecting their temperature fields.
- To identify and characterise reference materials with an appropriate level of thermal conductivity ( $0.02 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$  to  $1 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ ), aiming at temperatures up to 800 °C, and are suitable for the investigation of anomalies between laboratories. Reference materials must meet ISO Guide 34 guidelines for long-term property stability/uniformity/reproducibility and should be robust and allow the comparison of apparatus of different geometries.
- To assess the viability of using transient techniques in thermal conductivity measurements of advanced composites and insulation materials. Composite thermal protection materials, such as carbon fibre reinforced types, often have significant differences between thermal conductivity in the through-thickness direction and that in the in-plane direction. This JRP will provide uncertainty evaluation and limitations on the use of the transient and other industrial techniques for thermal protection materials. This will enable industry to select appropriate measurement techniques for characterising thermal protection materials and to estimate measurement uncertainties.

### 3 Research results

**Objective 1: To determine the effect of radiant heat transfer on thermal conductivity measurements at high temperatures. Heat transfer within many insulation products includes a combination of all three modes (conduction, convection, radiation), but there is currently insufficient knowledge of the magnitude of radiant heat transfer. This knowledge is required to be able to specify the tolerance in surface emissivity of measurement apparatus components, to improve measurement uncertainties, to investigate the anomalies between reference laboratories, and to finalise the new European measurement standard for thermal conductivity measurements of insulation at high temperatures.**

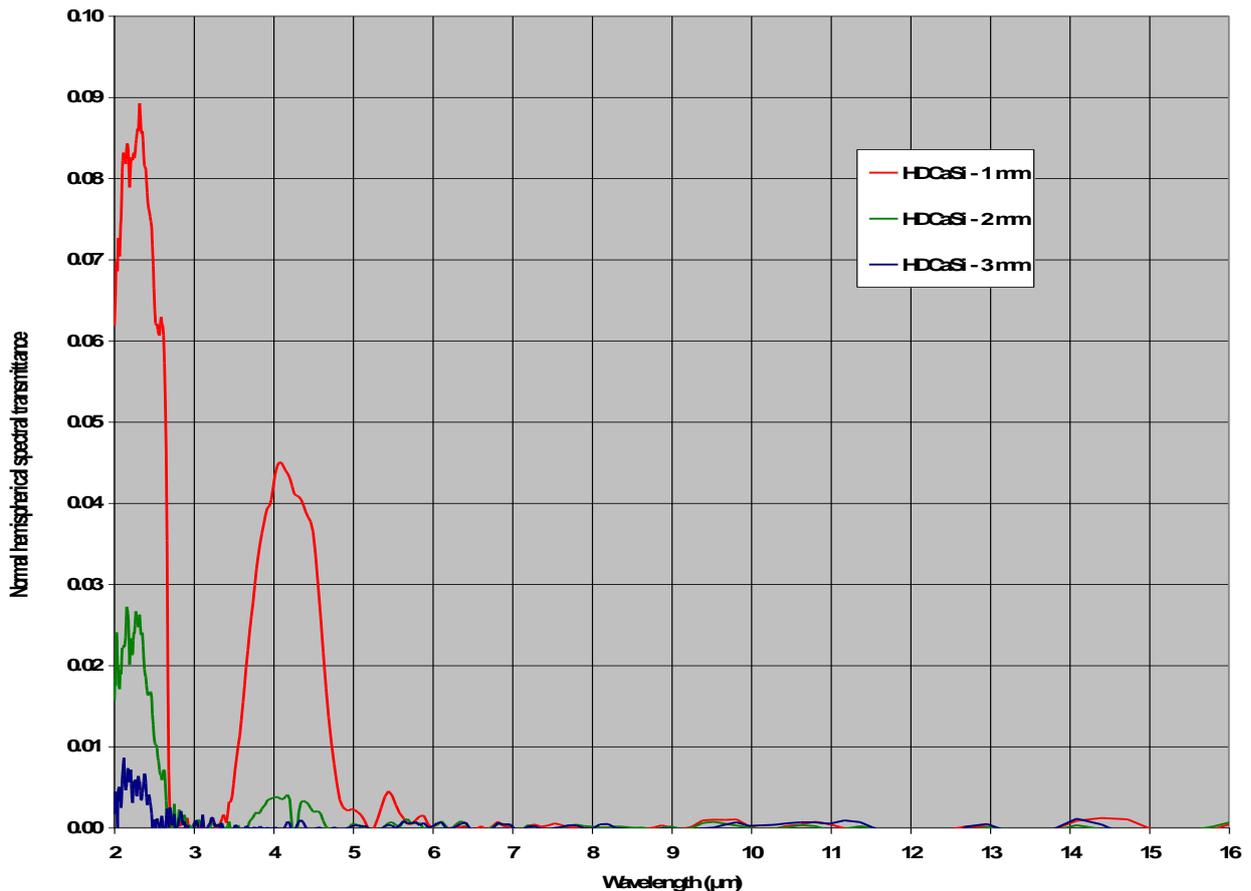
PTB led the investigation of the effect of radiant heat transfer on thermal resistance/conductivity measurements, aiming at temperatures up to 800 °C. As a result of a literature survey, PTB identified a theoretical model describing relatively simple analytical expressions of the complex behaviour of the combined conductive and radiative heat transfer through a highly porous semi-transparent material. A FEM model developed by PTB allows the estimation of the radiative contribution to the overall heat transfer through porous semi-transparent samples. The results of this numerical simulation agree well with the previously identified analytical prediction models, see Fig. 1. Two further numerical models, a very simple one and a FEM model, were developed by CMI to investigate the possible tolerances of the heater plate surface flatness and the heater plate surface emissivity with regard to their effect on the results of thermal conductivity measurements. NPL provided measurement data for validating the models developed at PTB and CMI. BFKH performed an experimental study that shows the effect of applying lateral radiative screens to decrease the radial heat-flow along the gap between the metering zone heater and the guard zone heater. Based on experimental results, BFKH has developed a theoretical model to estimate the radiative heat transfer between the metering zone heater and the guard zone heater of its in-house built HTTCMA (High-Temperature Thermal Conductivity Measurement Apparatus).



**Figure 1 Good agreement of the models of the complex behaviour of the combined conductive and radiative heat transfer through a highly porous semi-transparent material with experimental data**

LNE has performed normal hemispherical spectral transmittance and quasi-normal hemispherical reflectance measurements on five high temperature insulation materials at room temperature. The five high-temperature insulation materials are: low-density calcium silicate, amorphous silica, high-density calcium silicate, exfoliated vermiculite and needle mats. The first four materials were porous and the last one was fibrous. Transmittance measurements were performed on thin samples to get significant transmittance in the spectral bands of transparency. Measurements were performed using an integrating sphere in order to collect diffuse transmitted or reflected radiation. The normal hemispherical spectral transmittance of high-density calcium silicate material (HDCaSi-N) is shown in Fig. 2. The study has shown that it is possible to obtain

experimentally radiative properties data on high temperature insulation materials. Radiative properties can be measured accurately only on materials at room temperature, but radiative properties don't vary much with temperature as long as the material structure doesn't change. Thus, radiative properties obtained at room temperature allow the prediction of difficult situations for the measurement of thermal conductivity at high temperature with a guarded hot-plate. The experimental data of radiative properties obtained on those samples are usable for modelling radiant heat transfers in insulation materials.



**Figure 2 Normal hemispherical spectral transmittance results on high density calcium silicate (HDCaSi-N)**

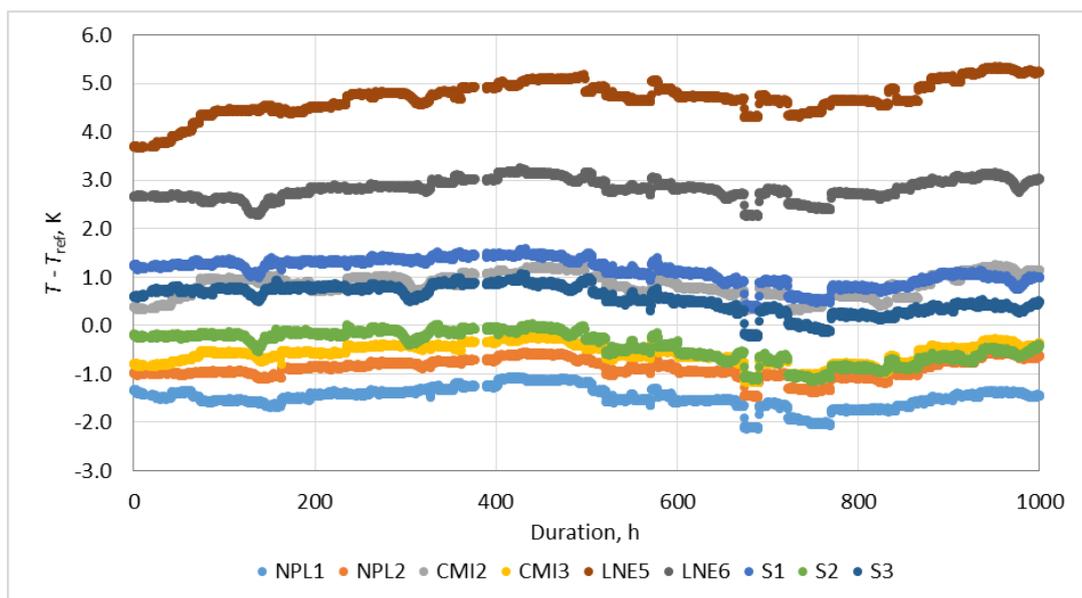
This objective has been achieved, as CMI, LNE and PTB delivered four co-authored presentations and posters at the Nineteenth Symposium of Thermophysical Properties in Boulder CO, USA in June 2015, based on the research in the Thermo project. The measurement of radiative properties and modelling will provide industrial and academic modellers with reliable reference data to allow them to validate their heat transfer models of thermal protection materials. It will also provide the knowledge needed by the reference laboratories that use High-Temperature Guarded Hot-Plates to improve the quality of their thermal conductivity measurements.

**Objective 2: To improve thermal guarding systems and temperature sensor arrangements to obtain temperature measurements that are more representative of the specimen boundary conditions within regions of heat flux. Temperature sensors used in these applications need to be as thin as possible to minimise distortion of the temperature field, but they also need to have both a high level of batch agreement and long-term calibration stability. Newly developed thermocouples need to be tested for these specific measurement applications.**

An adequate thermal guarding system is essential for minimising the heat loss/gain at the edge(s) of the specimen(s). BFKH has designed and implemented in their High-Temperature Thermal Conductivity

Measurement Apparatus (HTTCMA) a new passive thermal guarding system made of reflective layers. The new passive thermal edge guarding system has helped to improve the quality of the thermal conductivity measurements in the HTTCMA at BFKH.

Temperature sensors used in HTGHP need to be as thin as possible to minimise the distortion of the temperature field to be measured. At the same time the sensors need to be stable and accurate to provide reliable measurements of temperature difference across the thickness of the specimen(s) and to ensure the establishment of a uniform 1-D heat flow through the specimen(s). CMI investigated the long term stability of selected type S and type N mineral insulated metal sheathed (MIMS) thermocouples usable from 70 °C to 850 °C in HTGHPs. In one of the two batches of thermocouples tested for stability, three type S MIMS thermocouples, S1 to S3 with an outer diameter of 1.5 mm and six type N MIMS thermocouples with an outer diameter of 1 mm, were placed in an air furnace together with a reference type S thermocouple at a nominal temperature of 825 °C. The reference thermocouple was placed in the furnace for the whole duration of the measurements. Before and after the measurements in the air furnace, the reference thermocouple was calibrated at fixed points (Zn, Al, Ag) to verify its stability. To eliminate the effect of the furnace drift, all MIMS thermocouples were compared to the reference thermocouple in terms of the temperature calculated according to EN 60584-1 from the respective output voltages. The temperature deviations from the reference thermocouple are presented in Fig.3. The conclusion of the study is that the stability of all investigated thermocouples during the exposure to temperature above 800 °C for 250 or 1000 hours (depending on the measurement batch) was within  $\pm 1$  °C.



**Figure 3 Temperature difference from the temperature measured by a reference thermocouple at a nominal temperature of 825 °C**

The inhomogeneity of the MIMS thermocouples was studied by CMI in the project. Prior to and after the measurements at high temperature, the homogeneity of the thermocouples was measured in an oil bath at the temperature 200 °C. The variations in response due to inhomogeneity (over 30 cm) are within  $\pm 1$  °C. No significant differences in stability between MIMS type S and type N thermocouples were observed. The higher the temperature, the higher the drift of the thermocouples, so the results obtained by heating the thermocouples at 850 °C initially and testing stability at 825 °C give stability results applicable to the thermocouples used from 70 °C to 850 °C. Type N MIMS thermocouples can be recommended for use in HTGHP, as they have a considerably lower price and a higher sensitivity compared to type S. Additional investigations of factors influencing the uncertainty of the temperature measurement related to the installation of thermocouples in heater plates (e.g. immersion depth) were carried out at CMI. To achieve a high level of batch agreement, LNE have shown that the measurement of the electrical resistance of the loop of thermocouples is a way to select a batch of thermocouples with close responses. LNE has also tested a new method to check at high temperatures the offset of the thermopile that is used to control the centre-gap

temperature imbalance. The method uses two metal specimens with high thermal conductivity in order to have a very isothermal hot plate and this method is quite easy to apply.

A Good Practice Guide was developed based on the experimental results obtained on thin MIMS thermocouples that were supplied by LNE, NPL and CMI. It provides recommendations for the selection, preparation and calibration of temperature sensors to be used in high temperature thermal conductivity measurement devices. This objective was met, as the [Good Practice Guide](#) has been made reader-friendly for the public and is available on the Thermo website. All NMIs developing HTGHPs and the wider scientific community that use fine gauge thermocouples for temperature measurements at elevated temperatures will benefit from the knowledge disseminated in the Good Practice Guide.

**Objective 3: To identify and characterise coatings that can withstand abrasion from contact with the specimen; withstand thermal cycling up to 850 °C and remain securely bonded to plate materials; and provide a consistent high emissivity (0.8 or higher is required) with a specified value/tolerance. We will also identify new heater plate materials that will provide improved dimensional stability despite repeated temperature cycling, while still providing the required temperature uniformity across critical components.**

A European framework for characterising the performance of thermal protection materials has been developed and validated in the project, including many technical advances in measurement technology and capability. The High-Temperature Guarded Hot-Plate (HTGHP) technique has been improved or introduced at participating NMIs. Before the start of the project, NPL was the only European NMI that had a validated HTGHP with the expanded relative uncertainty  $\pm 5\%$  ( $k=2$ ) at 800°C. The project has achieved the new national standard level metrological instrumentation for thermal conductivity measurements.

LNE now have a validated National Standard level HTGHP. LNE firstly upgraded their in-house-constructed HTGHP for the measurement of thermal conductivity in the temperature range 70 °C to 800 °C. LNE then carried out systematic performance checks of their two high-temperature guarded hot-plates – their in-house-constructed LNE HTGHP and a commercial HTGHP. Further developments were made on the in-house constructed LNE HTGHP after the first comparison (the Round-Robin) in the project and this led to an improved quality of measurements, as shown in the Star-shape comparison later in the project. The assessments of uncertainties of the HTGHP measurements have given expanded relative uncertainties ( $k=2$ ) of 4 % to 5 % from 150 °C to 650 °C for their in-house-constructed guarded hot-plate, 7 % to 10 % from 150 °C to 500 °C for the commercial high-temperature guarded hot-plate. The LNE in-house-constructed HTGHP, see Fig. 4 has become the National Standard HTGHP in France.



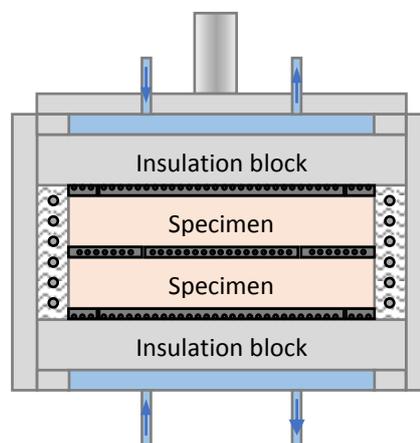
**Figure 4 The LNE in-house-constructed High-Temperature Guarded Hot-Plate (HTGHP) has become the National Standard in France**

BFKH now have a National Standard level High-Temperature Thermal Conductivity Measurement Apparatus (HTTCMA), see Fig. 5. BFKH have been developing a new technique for high temperature thermal conductivity measurements up to 850 °C. BFKH firstly modified their in-house designed HTTCMA and then carried out systematic performance checks of the apparatus. The assessments of measurement uncertainties have given expanded relative uncertainties ( $k=2$ ) of 5 % to 6 % from 150 °C to 650 °C for the HTTCMA at BFKH.



**Figure 5 BFKH’s first in-house designed High-Temperature Thermal Conductivity Measurement Apparatus (HTTCMA)**

During the 3-year project, CMI completed the design and construction of their first HTGHP, see Fig. 6 functioning up to 800 °C. CMI performed first inter-comparison measurements of thermal conductivity up to 650 °C using the high-temperature guarded hot-plate built during the project, and the measured values agreed with NPL within  $\pm 5$  % up to 650 °C. The knowledge transfer among partners significantly reduced the amount of time for CMI to develop their new HTGHP.



**Figure 6 CMI's first in-house-designed High-Temperature Guarded Hot-Plate (HTGHP)**

NPL improved and carried out an initial validation of their new National Standard Small Guarded Hot-Plate (SGHP), see Fig. 7 for testing thin specimens of polymers and composites.



**Figure 7 The new NPL Small Guarded Hot-Plate (SGHP) for thin polymer composites**

One of the difficulties in HTGHP measurements arises particularly from the limitations of the materials which can be used at high temperatures to make the hot and the cold plates of the instrument. In HTGHPs, the hot and the cold plates are all used at high temperatures and are subjected to heat cycles up to high temperatures. The material constituting them must be mechanically and chemically stable to keep a high level of flatness of the plates and it must have a high thermal conductivity to get uniform temperatures over the plates. In order, firstly to reduce the thermal contact resistance between the specimens and the hot and cold plates, and secondly to have all the HTGHPs working with similar experimental conditions, it is highly recommended that the surfaces of the hot and cold plates have a total hemispherical emissivity above 0.8. During the last decades, new advanced ceramic materials with high thermal conductivity have been developed and are now available commercially as well as high temperature paints with high emissivity.

Studies have been carried out in the Thermo project with the aim of finding and testing new advanced materials and high emissivity coatings for the hot and cold plates of HTGHPs working up to 800 °C. Three ceramic materials, a silicon infiltrate silicon carbide SiSiC CS11T produced by Ceramdis GmbH (Switzerland), a machinable aluminium nitride SHAPAL® Hi M-soft produced by Tokuyama Corp. (Japan), and a sintered aluminium nitride AlN 180 produced by FINAL Advanced Materials (France) with high thermal conductivity at ambient temperature were suggested by PTB for further tests in terms of thermal conductivity at higher temperatures and opacity to thermal radiation. The parameters measured by LNE and NPL on the candidate materials are the density at room temperature, the thermal expansion coefficient up to 800 °C and the thermal conductivity up to 800 °C. The transparency of the three thin ceramic material specimens was also measured by LNE in the infrared spectral range from 1 µm to 17 µm. Fig. 8 presents the thermal conductivity curves of Nickel 201 alloy (the plate material currently used by NPL, NIST and LNE in their HTGHPs) and of the three selected ceramic materials. Above 600 °C, Nickel 201 alloy has a higher thermal conductivity than the three ceramics. Below 600 °C, the SiSiC CS11T and the sintered AlN 180 have a thermal conductivity significantly higher than Nickel 201 but the sintered AlN 180 shows a wide transparency spectral band at short wavelengths (below 6.5 µm).

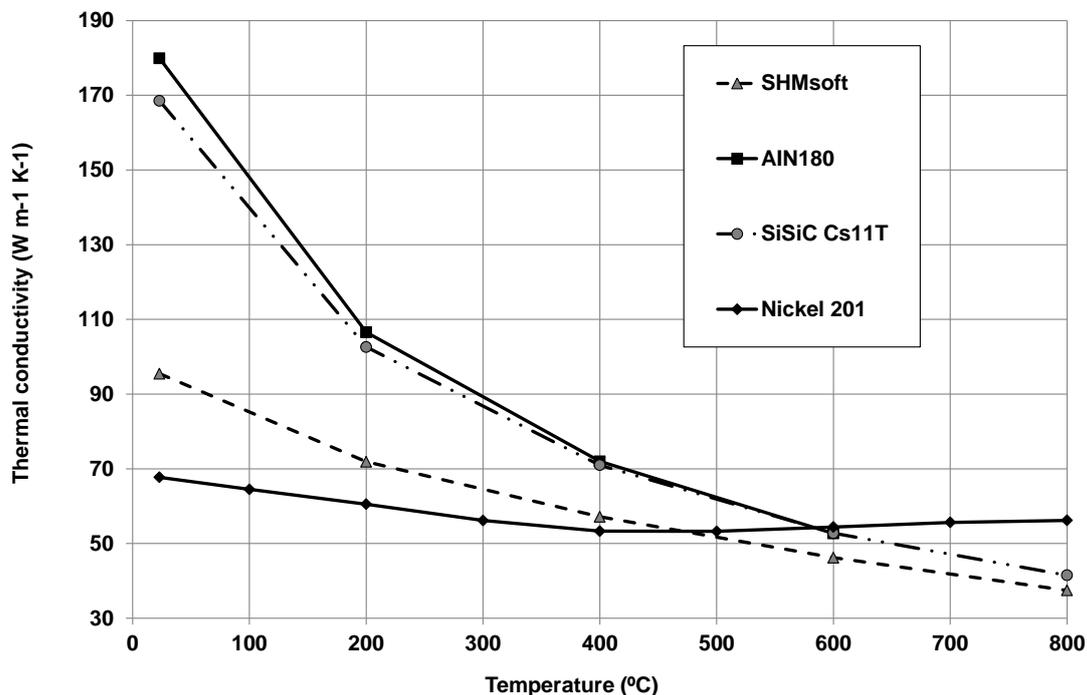
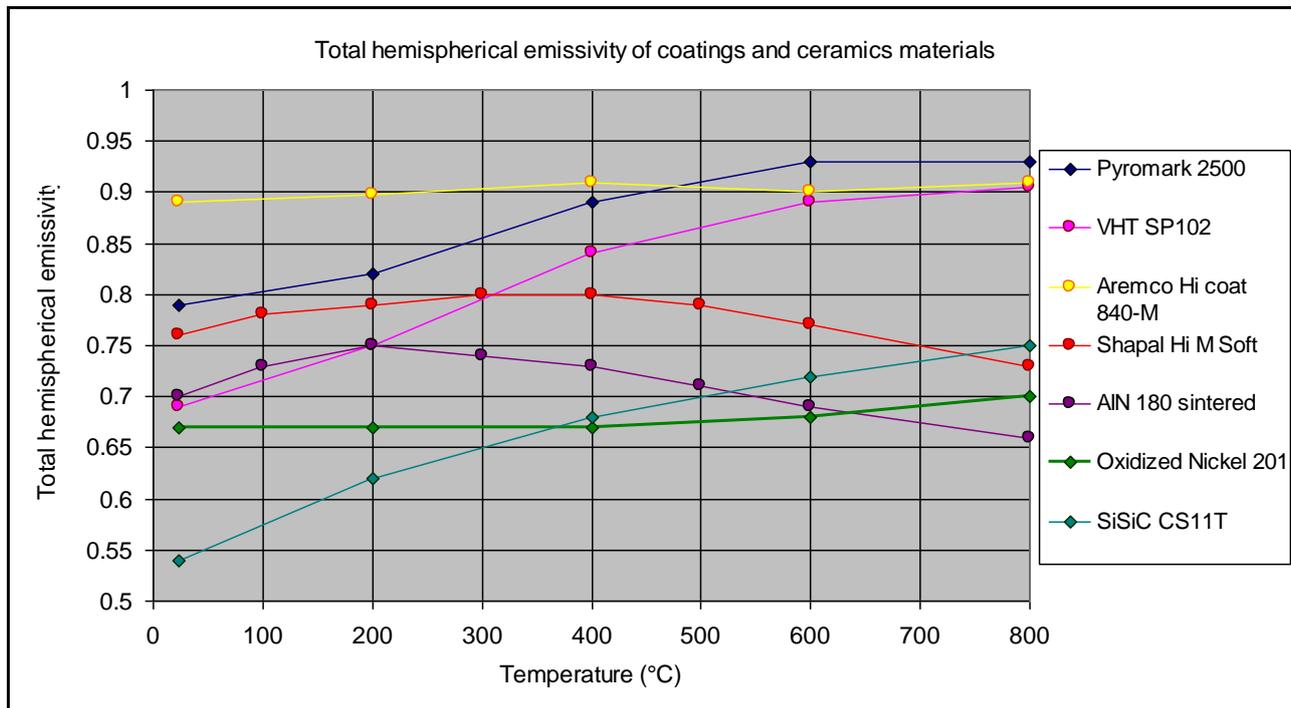


Figure 8 Thermal conductivity of Nickel 201 and of the three selected ceramic materials

Three commercially available high temperature black paints were selected for the testing of the total hemispherical emissivity and durability at high temperatures at LNE: the Pyromark® High Temperature Paint 2500 Flat Black produced by LA-CO Industries INC (USA), the VHT Flame Proof SP102-Flat Black paint produced by VHT Products Company (USA) and the AREMCO HiE Coat 840-M produced by Aremco Products Inc. (USA). The Pyromark 2500 Flat Black paint sample was prepared by NPL and was heated up to 850 °C before sending to LNE for tests. Another way to get a high emissivity of the plates is to make the plates with a material having a high emissivity and keep the material uncoated or for plates made of metal to produce a stable oxide layer at the surface by heating the metal plates at high temperatures in air. The emissivity of oxidised Nickel 201 alloy and of the two ceramics SiSiC Cs11T and SHAPAL Hi M-Soft were measured at LNE. The total hemispherical emissivity curves of the tested coatings, oxidised Nickel 201 and ceramic materials are shown in Fig. 9. The stability of the total hemispherical emissivities of the paint were tested after the paints were maintained at 830 °C and successively in contact with each of the four types of insulation materials (low density calcium silicate, amorphous silica, high density calcium silicate, and a needle mat of silica fibres) for 24 hours. The total hemispherical emissivities of the paints were stable within 0.035, less than the measurement uncertainty of the total hemispherical emissivity results.

LNE coated the plates of a HTGHP with the Aremco Hi coat 840-M and performed thermal conductivity measurements on a pair of very flat specimens of a high density calcium silicate insulation material up to 650 °C. After the test, it was noticed that the colour of the surfaces of the specimens had changed from white before the test to light yellow after the test. An energy-dispersive X-ray spectroscopy (EDX) test performed by NPL showed that chromium and copper were present in trace amounts at the surface of the yellowed specimens. The Aremco Hi coat 840-M paint contains copper chromite which has probably been converted into chromate (yellow) during the heating up to 650 °C. The influence of the reaction between the Aremco paint and the insulation material on the thermal conductivity result was not quantified. Pyromark® 2500 paint has been used in the current NPL HTGHP and has not shown any reaction with the insulation materials tested in the apparatus. The VHT Flame Proof SP102-Flat Black paint has currently been used in the LNE HTGHP and CMI HTGHP; it has shown good compatibility with calcium silicates insulation materials and good durability.



**Figure 9 The total hemispherical emissivity curves of the tested coatings, oxidised Nickel 201 and ceramic materials**

For the high-temperature high-emissivity coatings, two, among the three tested paints, can be suggested for coating the hot and cold plates of HTGHPs, the two paints are the Pyromark 2500 Flat Black and the VHT Flame Proof SP102-Flat Black. They have a total hemispherical emissivity above 0.8 respectively for temperatures above 100 °C and above 300 °C, and with good durability in use. The tests performed do not give a service life for the paints; the service life depends highly on the heat cycles undergone by the coatings. Both paints still have to be used for a longer time and tested in contact with a large variety of insulation materials to be fully validated for HTGHPs up to 850 °C. The user of a HTGHP must check periodically the state of the coatings of the plates and renovate them when degraded.

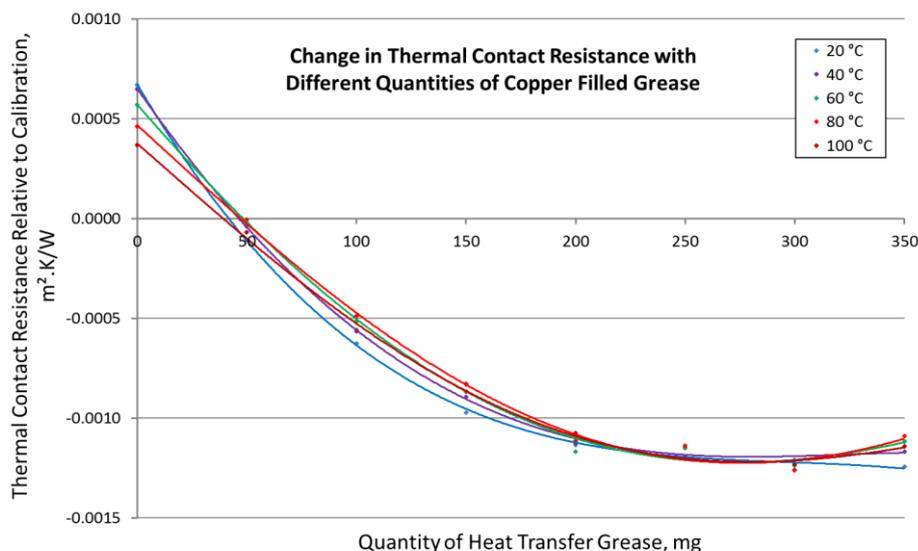
This objective was achieved. A presentation on this study was delivered by LNE at the 20th European Conference on Thermophysical Properties (ECTP 2014). LNE developed a guideline for validating the in-situ thickness measurements in the HTGHP, and a good practice guide on the performance check and validation of the HTGHP. NPL and other partners have co-authored in a guideline recommendations for the design of a new generation of high temperature guarded hot-plate based on the main technical results of the project. At the end of the project, two manuscripts were submitted to the International Journal of Thermophysics for publication as peer-reviewed papers. One paper titled: Identification and Characterization of New Materials for Construction of Heating Plates for High-Temperature Guarded Hot-Plates was submitted by LNE, with other partners as co-authors. Another paper titled “Design Guideline for New Generation of High-Temperature Guarded Hot-Plate” was submitted by NPL, again with other partners as co-authors. The knowledge disseminated in the papers will enable NMIs, other industrial or academic laboratories, and instrument manufacturers to select the appropriate heater plate materials and coatings when designing/upgrading their HTGHPs. In fact, NPL, LNE and CMI have already used the outputs of the study in the development/upgrading of their HTGHPs. The step-change improvements that have been made to reference instruments enable European National Measurement Institutes to offer more comprehensive high quality measurement facilities for European Industry. The National Standard level HTGHPs will also be used to provide measurement services to European industry to enable them to meet the mandatory regulations and may also contribute to the European Space Agency program and FP7 Clean-Sky Call projects for the design and testing of thermal protection components and systems.

**Objective 4: To overcome thermal contact resistance issues in thin sections of composite materials. Existing steady-state techniques cannot provide reliable thermal measurements due to distorted heat flux. This is caused by differential expansion and temperature sensors that do not represent the specimen surfaces without affecting their temperature fields.**

The issue of thermal contact resistance is especially significant for measuring thermal conductivities of polymers and polymer composites, as they have a reasonably high coefficient of thermal expansion. When these materials are subject to a large temperature gradient through their thickness during a thermal conductivity measurement, the thermal expansion may cause the hot surface of the specimen to be larger than the cold surface. This differential expansion can cause the specimen to bow and create air gaps between the specimen surfaces and the plates of a measurement apparatus. To obtain reliable thermal conductivity measurements of thin polymer composites using the Guarded Hot-Plate method, the thermal contact resistance between the surfaces of the specimens and the heater plates need to be minimised.

NPL has developed a new variation on the established guarded hot-plate technique for steady-state measurements of thermal conductivity. This new guarded hot-plate has been specifically designed for making measurements on specimens with a thickness that is practical for advanced industrial composite materials and applications. During the development of this new guarded hot-plate, NPL carried out an experimental investigation into methods for minimising the thermal contact resistance between the test specimen and the plates of the apparatus. This experimental investigation included tests on different thermal interface materials for use in another NPL facility based on a commercial guarded heat flow meter apparatus (Holometrix and with model number TCA-200LT-A) conforming to standard ASTM E1530-11. The investigation started by establishing a baseline calibration for the guarded heat flow meter using 50 mg of zinc oxide filled silicone grease applied to both surfaces of eight reference specimens with known values of thermal conductivity.

Studies were carried out on the effect of applying different quantities of the type of heat transfer compound suggested in ASTM E1530-11 (clause 10.7.3), and also the effect on thermal resistance of alternative types of thermal interface products. The optimum quantities of two silicone greases were determined and a silicone grease filled with copper was found to offer the best combination of repeatability, a small hysteresis effect and a low thermal contact resistance. Fig. 10 shows the change of thermal contact resistance (relative to the calibration) with different quantities of copper filled grease. NPL recommended that the interface products from this study that gives the best combination of repeatability, small hysteresis effect and low thermal contact resistance would be the silicone grease filler with copper with 350 mg applied to each specimen surface. However, the application of the grease is time consuming, messy and unsuitable for specimen materials that would absorb the silicone.



**Figure 10 The change of thermal contact resistance (relative to the calibration) with different quantities of copper filled grease**

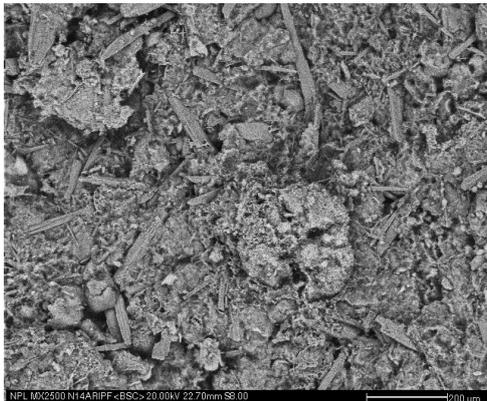
In addition, two products based on a textured indium foil and pyrolytic graphite sheet were found to offer similar or better reductions in thermal contact resistance, but with quicker, easier application and the advantages of protecting the apparatus plates from damage and being useable with specimen materials that would otherwise absorb silicone grease. The best alternative to silicone greases for temperatures up to about 100 °C, offering advantages in terms of quick application and protecting the apparatus plates from damage, would be the textured indium foil. A potential alternative for thermal conductivity measurements at temperatures up to 250 °C is the pyrolytic graphite sheet, which offers significant reductions in thermal contact resistance over the temperature range 20 °C to 250 °C and is nearly as easy to handle as the textured foil products. However, these advantages also come at the expense of slightly poorer repeatability.

The objective was met, as NPL has published a paper titled “Techniques for Reducing Thermal Contact Resistance in Steady-State Thermal Conductivity Measurements on Polymer Composites” in the International Journal of Thermophysics, 37(11), 1-13, DOI 10.1007/s10765-016-2119-0, 2016. The paper is co-authored with an international manufacturer of thermal interface materials, the Indium Corporation. The knowledge disseminated in this peer-reviewed paper will help test laboratories using the GHP method to choose the appropriate thermal interface materials for minimising thermal contact resistance. It will also help wider industrial applications, e.g. power electronics where thermal contact resistance is a dominant factor, to choose the thermal interface materials.

**Objective 5: To identify and characterise reference materials with an appropriate level of thermal conductivity ( $0.02 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$  to  $1 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ ), aiming at temperatures up to 800 °C, and are suitable for the investigation of anomalies between laboratories. Reference materials must meet ISO Guide 34 guidelines for long-term property stability/uniformity/reproducibility and should be robust and allow the comparison of apparatus of different geometries.**

Despite the European Union having put into place new regulations (EU NO. 305/2011) and mandatory standards with the aim of making reliable thermal performance data available to industrial users, the level of agreement between reference laboratories at the outset of the project was 3x worse than the maximum 5 % allowed in these regulations. Implementation of these regulations urgently require the science underpinning thermal conductivity measurements to undergo a step-change improvement. Before the start of the project, there was no thermal conductivity reference material available to aid the investigation of the causes of the disagreement between the reference laboratories.

One ‘first-of-its-kind’ insulation/refractory type European high-temperature thermal conductivity reference material was developed during the project, in order to resolve discrepancies and to enable agreement between reference laboratories. NPL has led the development of high temperature thermal conductivity reference materials, with contributions from all partners and collaborators. The research started with a survey of potential candidate materials, which led to a short list of four candidates: low density calcium silicate (LDCaSi), Amorphous Silica (AmSi), high density calcium silicate (HDCaSi-N), and exfoliated vermiculite (EV). The initial tests (including 15 test tasks) on material composition and microstructure changes, dimensional stability, mechanical stability, chemical stability and uniformity were completed on the short list of four candidates, during which a new approach was developed in the project. The new approach combines both thermal metrology and material science in the assessment of candidate high temperature thermal conductivity reference materials. It enables science to go beyond what could be achieved separately. The X-Ray Fluorescence Spectrometry (XRF), and X-ray Diffraction (XRD) analysis were applied to reveal the composition of each material. The microstructure of each material, before and after heat treatment, is illustrated in the micrographs obtained using a Scanning Electron Microscope (SEM). Fig. 11 is the backscattered electron images of fractured surfaces of HDCaSi-N. The images labelled as (a) and (b) show the as received in-plane and the through-thickness fracture surfaces, respectively. The images labelled as (c) and (d) show the samples after the first heat treatment as in-plane and through-thickness fracture surfaces, respectively. The images labelled as (e) and (f) show the samples after three runs in the Thermogravimetric Analysis apparatus (TGA) as in-plane and through-thickness fracture surfaces, respectively. The Fig.11 (c) to (f) show some circular black features which have been identified as the holes formed by the melting of the glass fibres seen in Fig. 11 (b). Fig. 11 shows that the microstructure of HDCaSi-N remains stable after three runs in the TGA.



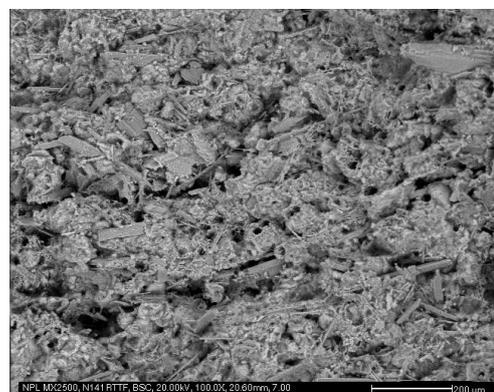
(a)



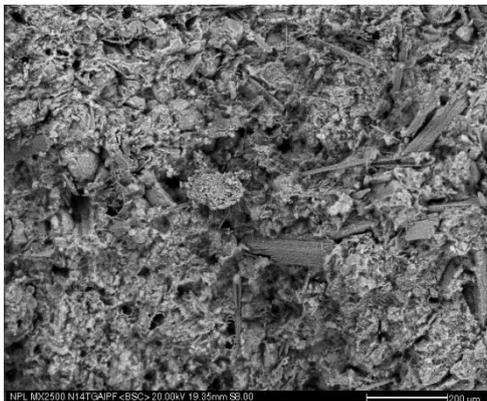
(b)



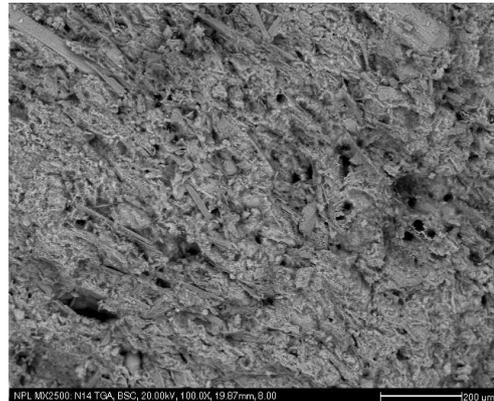
(c)



(d)



(e)



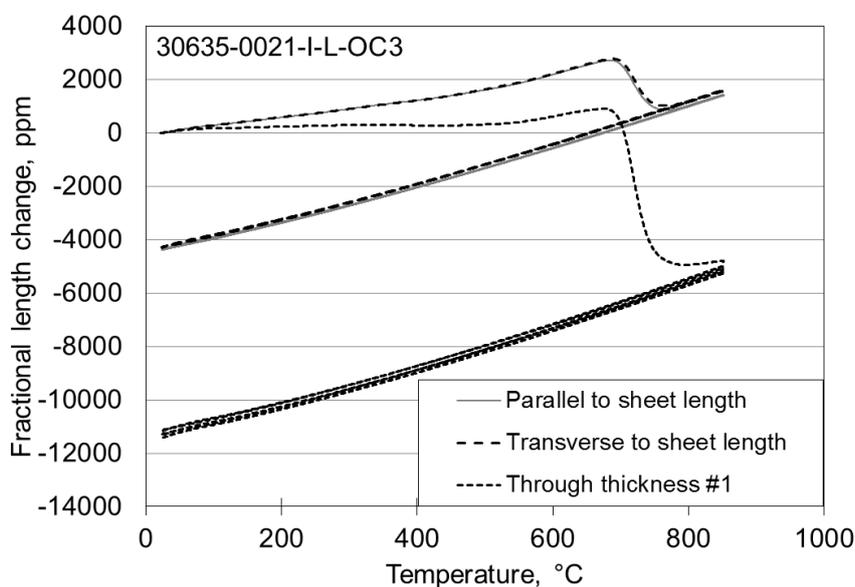
(f)

**Figure 11 Backscattered electron images of fractured surfaces of HDCaSi-N: (a) as received, in-plane, (b) as received, through-thickness; (c) after 1st heat treatment at 850 °C for 24 hrs, in-plane; (d) after 1st heat treatment at 850 °C for 24 hrs, through-thick; (e) post TGA, in-plane; and (f) post TGA, through-thickness.**

Based on the initial test results, stocks of the two selected candidate reference materials, HDCaSi-N and LDCaSi were purchased by NPL and CMI. The cutting plans and technical specifications for machining and heat treatment were agreed by all partners. BFKH coordinated with two subcontractors in Hungary and completed the machining, heat treatment up to 850 °C, and dimensional and mass measurements of 76-off HDCaSi-N specimens and 50-off LDCaSi specimens.

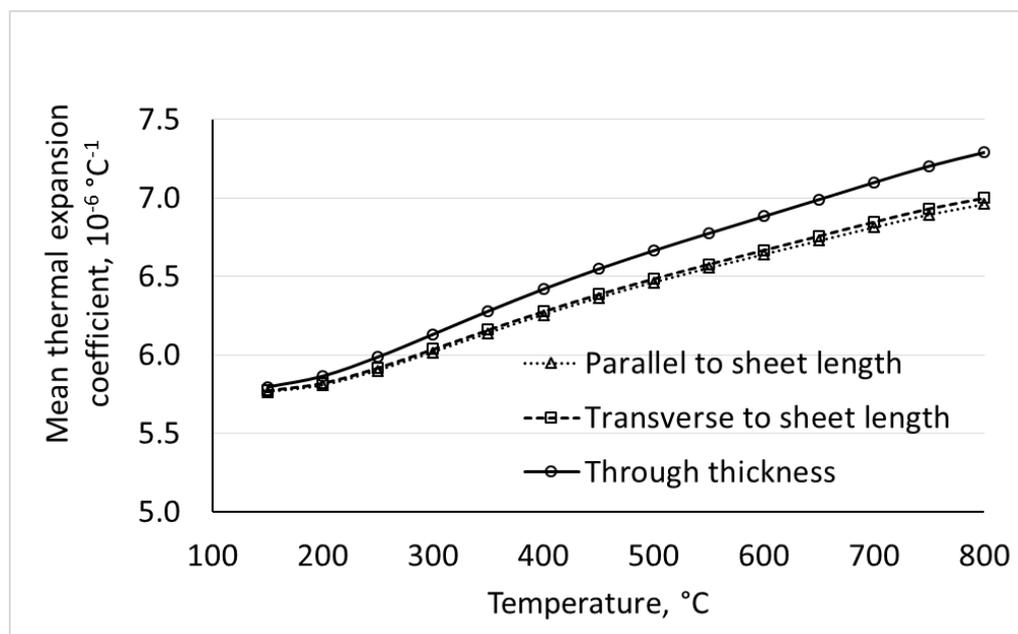
To check the mechanical stability under thermal cycling and uniformity of the newly purchased batch of candidate reference material, HDCaSi-N, thermal expansion tests were performed on eighteen specimens

prepared from board Nos. 0013, 0016 and 0021. From each board six specimens were prepared from two lateral locations: three of them from the left hand side and another three of them from the right hand side of the board. Within each set of three specimens prepared from the same location, one specimen was prepared for thermal expansion testing in the through-thickness direction, and two specimens were prepared with axis in plane, one of them is parallel to the sheet/board length and the other transverse to the sheet/board length. Thermal expansion measurements were made by NPL using a Linseis twin push-rod alumina dilatometer calibrated using platinum and alumina reference materials. The typical thermal expansion curves in three orthogonal directions for the candidate reference material HDCaSi-N (batch No. 30635) are shown in Fig. 12. The fractional length change over the 1st thermal cycle shows that the material underwent significant shrinkages both in-plane (parallel and transverse to sheet length) and through thickness in the first thermal cycle, the latter direction showing greater changes. The sharp change in fractional length at around 700 °C is due to the dehydroxylation of the material. The XRD measurements had been performed on HDCaSi-N samples in the provisional assessments revealed that one of the major phases, hydrated calcium silicate ( $\text{Ca}_2\text{SiO}_4 \cdot \text{H}_2\text{O}$ ) in fresh samples became calcium silicate ( $\text{CaSiO}_3$ ) after the specimen had been heat treated at 850 °C. The fractional length changes recorded for the 2nd and 3rd thermal cycles show that the HDCaSi-N material is mechanically stable.



**Figure 12 Typical thermal expansion curves for the HDCaSi-N (batch No. 30635) during the three thermal cycles up to 850 °C**

The mean thermal expansion coefficients averaged over the six specimens measured from 150 °C to 850 °C for each direction is shown in Fig. 13. As the thermal expansion coefficients in the two in-plane directions, parallel and transverse to the sheet length are the same (well within the uncertainty of 2 %), a single in-plane value for thermal expansion can be used. The values of mean thermal expansion coefficients have been used by NPL to estimate the amount of free bowing of the HDCaSi-N specimens due to the effect of differential thermal expansion caused by the temperature difference across the specimen. It is recommended to use smaller lateral dimension specimens, with a smaller temperature drop through the thickness of the specimen to minimise the amount of specimen bowing.



**Figure 13 Mean thermal expansion coefficient of HDCaSi-N (batch No. 30635)**

NPL completed a series of assessments on the HDCaSi-N and LDCaSi specimens during the full characterisation stage. However, around a quarter of the LDCaSi specimens were damaged during a transportation from BFKH to NPL – indicating the LDCaSi is less robust than the HDCaSi-N. All partners agreed to proceed with developing HDCaSi-N as an insulation/refractory type European high-temperature thermal conductivity reference material, because it is robust, mechanically and chemically stable and uniform.

NPL developed a new non-destructive acoustic based test method for mapping the density distribution within a specimen and have used it in the selection of candidate reference specimens. It is the first time that the density mapping technique has been applied in the field to improve the quality of the reference materials and inter-comparisons. After a systematic selection process at NPL, in total 15-off HDCaSi-N specimens have been selected for further detailed characterisation and the inter-comparisons.

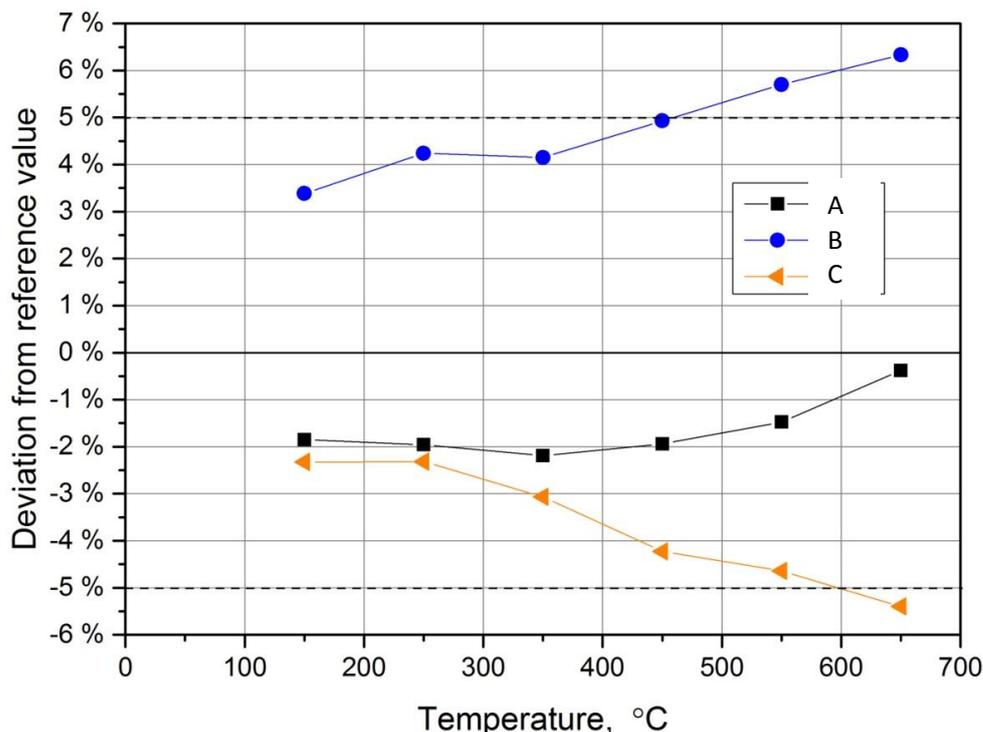
The results of thermal conductivity tests at NPL on 5 out of the 15 selected HDCaSi-N specimens showed that their thermal conductivity values differed by less than 2 % from the average values measured from 150 °C to 650 °C. The 2 % difference is less than half of the measurement uncertainty of the NPL HTGHP. Therefore, these specimens are considered to be identical or uniform in terms of thermal conductivity by the NPL HTGHP. Although the bulk density of the specimens varied from 800 kg·m<sup>-3</sup> to 813 kg·m<sup>-3</sup>, no thermal conductivity dependence on density was detected using the NPL HTGHP. The results indicate that the criteria used in the selection process were sufficient to ensure uniformity across the candidate reference specimens.

A Round-Robin inter-comparison was first undertaken among the National Standard Instruments at NPL, LNE, CMI and BFKH and was started in Oct. 2015; and in Feb. 2016 a Star-shape comparison was then undertaken among the European reference laboratories NPL, LNE, CMI, BFKH and FIW. All the 15-off HDCaSi-N reference specimens were used in the inter-comparisons. Both comparisons were finished and the provisional results were reported and analysed at the same time at the end of the project. PTB, as an independent party (that did not participate in the thermal conductivity measurements in the comparisons), carried out the data analysis of the comparisons.

In the Round-Robin comparison, all participating laboratories gained experience and this led to improved results in the Star-shape comparison that was started a few months later.

The provisional results of the thermal conductivity measurements at NPL, LNE and FIW in the Star-shape comparison, see Fig. 14, showed that their 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. Please note that the HTTCMA at BFKH is based on a new technique using a 2-D axisymmetric heat transfer model, which is different from the 1-D heat transfer model used in the HTGHPs at LNE, NPL, CMI and the Collaborator FIW. 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.



**Figure 14 Provisional results from FIW, LNE and NPL in the Star-Shape comparison**

The objective was achieved in that the newly validated HDCaSi-N reference material specimens fulfilled the criteria in ISO Guide 34 and met the revised maximum temperature 650 °C. The heat treatment and thermal expansion characterisation of the specimens was carried out at temperatures up to 850 °C. The uniformity of the specimens in terms of thermal conductivity was assessed up to 650 °C and it was concluded that the selected 15 specimens were considered to be identical or uniform by the NPL HTGHP in terms of thermal conductivity. The provisional results of NPL, LNE and the collaborator FIW in the Star-shape comparison have shown that 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.

NPL and other partners of the project have published two peer-reviewed papers. One paper titled “Provisional Assessment of Candidate High Temperature Thermal Conductivity Reference Materials in the EMRP ‘Thermo’ Project” was published in the Proceedings of the 32nd International Thermal Conductivity Conference and of the 20th international Thermal Expansion Symposium. Another peer-reviewed paper titled “Characterisation of a High-Temperature Thermal Conductivity Reference Material” was published in the International Journal of Thermophysics. NPL has led the knowledge transfer from the Thermo project to the CEN/TC89 WG14 and all partners contributed. The technical inputs from the Thermo project have been captured by CEN/TC89 WG14 and have been included in the working draft of the new EN measurement standard prEN 15548-1.

**Objective 6: To assess the viability of using transient techniques in thermal conductivity measurements of advanced composites and insulation materials. Composite thermal protection materials, such as carbon fibre reinforced types, often have significant differences between thermal conductivity in the through-thickness direction and that in the in-plane direction. This JRP will provide uncertainty evaluation and limitations on the use of the transient and other industrial techniques for thermal protection materials. This will enable industry to select appropriate measurement techniques for characterising thermal protection materials and to estimate measurement uncertainties.**

In the project, limitations and uncertainties in industrial measurement techniques have been investigated for their use in measuring the thermal conductivity of advanced thermal protection materials.

LNE wrote a recommendation report on the use of the laser flash technique for anisotropic materials. The laser flash technique can be used for the measurement of thermal conductivity (indirect technique) of anisotropic materials with 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.

PTB developed an analytical mathematical model for the calculation of the standard-uncertainty of the thermal conductivity measured using a GHP. PTB also developed a numerical finite-element model for modelling heat transfers in a double-specimens circular GHP. This model allowed the effect of each parameter to be studied, and it helped to quantify the uncertainties that were not easily calculable. PTB led the work to put together a comprehensive list of factors that contribute to the uncertainties in the HTGHP measurement, thus enabling the closer comparison of measurement capabilities among different laboratories.

PTB, LNE and FIW reported the uncertainty analysis of the two industrial HTGHPs: the "GHP Titan" manufactured by Netzsch Gerätebau GmbH, Selb (Germany) and operated by LNE; and "GHP-750-250", an in-house build instrument by Forschungsinstitut für Wärmeschutz e.V. (FIW), Munich (Germany). The second apparatus is operated by FIW themselves in their role as a collaborator of EMRP Thermo.

The objective was achieved, as a good practice guide on selecting and using industrial techniques for measurements of the thermophysical properties of protection materials was written jointly by LNE and PTB. The main recommendations are given below. Industrial techniques for the measurement of the thermal resistance or thermal conductivity of thermal protection materials can be classified in two categories: the steady state techniques (guarded hot-plates, heat-flow meters) and the transient techniques. The steady state techniques allow the measurement of the thermal resistance of any thermal protection material provided the thermal resistance of the specimens is high enough compared to the thermal contact resistance with the plates. Practically, guarded hot-plates and heat-flow meters allow the measurements of thermal conductivities below 1 or 2 W m<sup>-1</sup> K<sup>-1</sup>. The apparent thermal conductivity can be calculated from the measured thermal resistance assuming that the material is homogeneous. Steady state industrial techniques are usually limited to 800 °C. Industrial transient techniques are usually based on heat transfer models assuming a homogeneous and isotropic material (transient hot wire, transient plane source) or at least a material homogeneous in the direction of heat transfer (laser/xenon flash). So those transient techniques are limited to homogeneous materials or materials with little size singularities uniformly distributed. The maximum size of singularities must be defined regarding mean heat penetration depth in the material and dimensions of the sensors. Transient techniques can be used under harsh conditions (temperatures up to 3000 °C, in vacuum, high pressure,). Industrial transient techniques are usually less expensive than steady state techniques. All those techniques must be used by staff with high competencies in heat transfer.

In addition, PTB and all other partners co-authored a peer-reviewed paper "Critical Review of Industrial Techniques for Thermal-Conductivity Measurements of Thermal Insulation Materials" that was published in Int. J. Thermophys in Apr. 2015. The knowledge disseminated to the wider scientific community will help industrial and academic users to select appropriate thermal conductivity measurement tools for research and design purposes. The authors also summarised a collection of relevant international standards for measurement techniques/instruments, Guarded Hot-Plate (GHP), Heat Flow Meter (HFM), Transient Hot Wire (THW), Transient Plane Heat Source (TPS), and Laser Flash/Xenon Flash (LFA/XFA), for thermal conductivity and thermal diffusivity, see Table 1. It states that unfortunately, not all of the related standards from different organisations are compatible to each other. Currently there is no adequate measurement

standard for the thermal conductivity of insulation materials at high temperatures. So far, the vast majority of adequate instruments are self-made. In order to critically assess the abilities and disabilities of existing instruments, international round robins and inter-comparisons as well as good practice guides and related standards are urgently needed.

**Table 1 Relevant international standards for measurement techniques/instruments for thermal conductivity and thermal diffusivity**

GHP	HFM	THW	TPS	LFA/XFA
ISO 8302	ISO 8301	ISO 8894-1 ISO 8894-2	ISO 22007-2	
EN 1946-2 EN 12664 EN 12667 EN 12939	EN 1946-3 EN 12667 EN 12 EN 12939	EN 993-15		EN 821-2
ASTM C177 JIS A 1412-1	ASTM C518 JIS A 1412-2	ASTM C1113		ASTM E1461

EN: European Standard (CEN: European Committee for Standardization)  
 ISO: International Organization for Standardization  
 ASTM: American Society for Testing and Materials  
 JIS: Japan Industrial Standard (JSA: Japanese Standards Association)

**Summary:**

- There is a good agreement of the models that were used by PTB for the complex behaviour of the combined conductive and radiative heat transfer through a highly porous semi-transparent material with experimental data. It is possible to obtain experimentally radiative properties data on high temperature insulation materials. The experimental data of radiative properties obtained on those samples are usable for modelling radiant heat transfers in insulation materials.
- Type N MIMS thermocouples can be recommended for use in HTGHP, as they have a considerably lower price and higher sensitivity compared to type S. The experiments of CMI show that there is no gain from using S type metal-sheathed thermocouples regarding performance in stability. To achieve a high level of batch agreement, LNE have shown that measuring the electrical resistance of the loop of thermocouples is a way to select a batch of thermocouples with close responses.
- The metrological developments from this project have led to new National Standard level HTGHPs that are able to provide high quality thermal conductivity measurements for European Industry. The LNE in-house-constructed HTGHP has become the National Standard HTGHP in France. BFKH now have a National Standard level High-Temperature Thermal Conductivity Measurement Apparatus (HTTCMA). During the 3-year project, CMI has completed the design and construction of their first HTGHP functioning up to 800 °C; and has performed their first inter-comparison measurements. The knowledge transfer among partners significantly reduced the amount of time for CMI to develop their new HTGHP. NPL improved and carried out an initial validation of their new National Standard Small Guarded Hot-Plate (SGHP) for testing thin specimens of polymers and composites.
- The studies of heater plate materials for high temperature use show that above 600 °C, Nickel 201 alloy has a higher thermal conductivity than the three ceramics, a silicon infiltrate silicon carbide SiSiC CS11T produced by Ceramdis GmbH (Switzerland), a machinable aluminium nitride SHAPAL® Hi M-soft produced by Tokuyama Corp. (Japan), and a sintered aluminium nitride AlN 180 produced by FINAL Advanced Materials (France). Below 600 °C, the SiSiC CS11T and the sintered AlN 180 have a thermal conductivity significantly higher than Nickel 201 but the sintered AlN 180 shows a wide transparency spectral band at short wavelengths (below 6.5 µm).
- For the high-temperature high-emissivity coatings, two, among the three tested paints, can be suggested for coating the hot and cold plates of HTGHPs, the two paints are the Pyromark 2500 Flat Black and the VHT Flame Proof SP102-Flat Black. They have a total hemispherical emissivity above

0.8 respectively for temperatures above 100 °C and that above 300 °C, and with good durability in use. The tests performed do not give a service life for the paints; the service life depends highly on the heat cycles undergone by the coatings. Both paints still have to be used for longer time and tested in contact with a large variety of insulation materials to be fully validated for HTGHPs up to 850 °C.

- The interface products from this study that gives the best combination of repeatability, small hysteresis effect and low thermal contact resistance would be the silicone grease filler with copper with 350 mg applied to each specimen surface. However, the application of the grease is time consuming, messy and unsuitable for specimen materials that would absorb the silicone. The best alternative to silicone greases for temperatures up to about 100 °C, offering advantages in terms of quick application and protecting apparatus plates from damage, would be the textured indium foil. A potential alternative for thermal conductivity measurements at temperatures up to 250 °C is the pyrolytic graphite sheet, which offers significant reductions in thermal contact resistance over the temperature range 20 °C to 250 °C and is nearly as easy to handle as the textured foil products. However, these advantages also come at the expense of slightly poorer repeatability.
- The first-of-its-kind thermal conductivity reference materials – 15-off HDCaSi-N reference specimens have been developed and validated in the project and have been used by the European reference laboratories to improve their HTGHPs. Technical know-how has been gained on the development and validation of insulation/refractory type high temperature thermal conductivity reference materials. The new approach that combines both thermal metrology and material science in the assessment of candidate high temperature thermal conductivity reference materials, has enabled science to go beyond what could be achieved separately. A new non-destructive acoustic based test method for mapping the density distribution within a specimen was used in the selection of candidate reference specimens. It is the first time that the density mapping technique has been applied in the field to improve the quality of the reference material specimens and inter-comparisons. The values of mean thermal expansion coefficients have been used by NPL in thermal stress analysis to estimate the amount of free bowing of the HDCaSi-N specimens due to differential thermal expansion. It is the first time that a thermal stress analysis method has been applied in the field to quantify the bowing of rigid specimens in measurements using HTGHPs. It is recommended to use smaller lateral dimension specimens, with a smaller temperature drops through the thickness of the specimen to minimise the amount of specimen bowing.
- The provisional results of NPL, LNE and the collaborator FIW in the Star-shape comparison of thermal conductivity measurements of selected HDCaSi-N reference specimens have shown that 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. 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.
- A critical review paper of industrial techniques for thermal-conductivity measurements of thermal insulation materials was published in the International Journal of Thermophysics. A good practice guide on selecting and using industrial techniques for measurements of the thermophysical properties of protection materials was developed in the project.
- The laser flash technique can be used for the measurement of thermal conductivity (as an indirect technique) of anisotropic materials with 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.
- PTB developed an analytical mathematical model for the calculation of the standard-uncertainty of the thermal conductivity measured using a GHP. PTB also developed a numerical finite-element model for modelling heat transfers in a double-specimens circular GHP. The model allows the study of the effect of each parameter and it helps to quantify the uncertainties that are not easily calculable analytically. A comprehensive list of factors contributing to uncertainties in the HTGHP measurement is now available and this enables the closer comparison of measurement capabilities among different laboratories.

#### 4 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 the 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 of materials (available on the project website), 4 technical assessment visits and consultancies. 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 32<sup>nd</sup> 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 designs 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  $\pm 5\%$  ( $k=2$ ) 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
- BFKH now have a National Standard level HTTCMA up to 850 °C
- CMI completed the design and construction of their first HTGHP functioning up to 800 °C

The new high density calcium silicate (HDCaSi-N) thermal conductivity reference materials developed and validated in the project have been made available to the European reference laboratories and an industrial laboratory, FIW for comparisons of measurement techniques and for improvement of European equivalence. The industrial laboratory, FIW is a collaborator of the project, that provides testing services for insulating materials in the construction industry. 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 at NPL, LNE and FIW 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 and (ii) the use of transient and other industrial methods 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 in the region.

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.

## **5 Website address and contact details**

A public website is available at: <http://projects.npl.co.uk/thermo/>

The contact person for general questions about the project is Dr Jiyu Wu, ([jiyu.wu@npl.co.uk](mailto:jiyu.wu@npl.co.uk)).

## **6 List of publications**

- U. Hammerschmidt, J. Hameury, R. Strnad, E. Turzó-Andras, J. 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)
- J. Wu, R. Morrell, T. Fry, S. Gnaniyah, D. Gohil, A. Dawson, J. Hameury, K. Alain, U. Hammerschmidt, E. Turzó-András, R. Strnad, A. 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.

- C. Stacey, M. J. Parfitt, A. J. Simpkin, J. Wu; Design of a Guarded Hot-Plate for Measuring Thin Specimens of Polymer and Composite Materials, in Proceedings at the 32nd International Thermal Conductivity Conference and 20th international Thermal Expansion Symposium (on 27 April to 1 May 2014, IN, USA), pp129-135, Purdue University Press, Oct. 2015.
- 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, Published on-line: Oct. 2016.
- J. Wu, R. Morrell, C. Allen, P. Mildeova, E. Turzó-András, U. Hammerschmidt, E. Rafeld, A. Blahut, J. Hameury; Characterisation of high-temperature thermal conductivity reference materials, Int J Thermophys 38:66 DOI 10.1007/s10765-017-2200-3. (2017).