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

Background

Recent developments in polymer, aerogel and fibrous based composite systems used for thermal protection have shown the potential to provide thermal performance that is several-fold better than conventional insulation materials. However, there is currently no reliable metrology framework with which to evaluate their performance, including the validation of their use in safety critical engineering. Measurements using existing industrial techniques have shown a significant level of scatter, sometimes over 100 % for these new types of thermal protection. The lack of traceability in thermal performance metrology results in the manufacturers of these advanced materials not having the confidence to invest in these new materials and/or not being able to demonstrate their performance to engineering certification authorities.

Traceable qualification of improvements in high-performance thermal protection systems will have significant and far reaching impact across most of Europe’s Key Enabling Technologies and industries. Requirements for validated and traceable measurements are especially critical in advanced manufacturing industries such as aerospace and in safety systems for industrial facilities and transportation, where a lack of reliable thermal property data leads to either extra cost from over engineering or to a higher risk of industrial disasters.

This Joint Research Project will establish a thermal conductivity measurement infrastructure that addresses the whole traceability chain. It aims to achieve three times better agreement between reference laboratories and to evaluate the viability of improving transient and other industrial measurement techniques up to 800 °C.

Need for the project

Despite European government having spent the last fifteen years putting into place new regulations (EU NO. 305/2011) and mandatory standards (including EN 14303 to EN 14309) with the aim of making reliable thermal performance data available to industrial users, the current level of agreement between reference laboratories is still three times worse than the maximum 5 % allowed in these regulations. Implementation of these regulations urgently requires the science underpinning thermal conductivity measurements to undergo a step-change improvement.

Measurement of thermal conductivity for the purpose of research and development can often produce scatter of over 100 %, which means advanced manufacturing industries cannot reliably select or develop materials for their engineering applications. They are therefore obliged to make much more use of costly full-scale testing during the development of technologies such as aerospace components, structural fire safety systems and process plants. 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 is the main limiting factor on the predictive power of thermal modelling.

Scientific and technical objectives

The existing metrological techniques for temperatures up to 800 °C are sound and provide a good foundation for development, but there are many aspects that are still not sufficiently advanced. The final measured thermal conductivity value is very sensitive to the effects of these remaining metrology issues. Until these remaining metrology 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 Joint Research Project focuses on the traceable measurement and characterisation of modern advanced thermal protection materials. The top-level scientific objectives are:

EMRP
European Metrology Research Programme
Programme of EURAMET

The EMRP is jointly funded by the EMRP participating countries within EURAMET and the European Union

- Develop new techniques for the next generation of national standard instruments.
- Identify and develop the first-of-their-kind reference materials with a thermal conductivity in the range 0.02 $W{\cdot}m^{-1}{\cdot}K^{-1}$ to 1 $W{\cdot}m^{-1}{\cdot}K^{-1}$ and aiming at temperatures up to 800 °C.
- Systematically investigate the limitations of the transient and other industrial techniques.
- For the first time theoretically and numerically determine the effect of radiant heat transfer on thermal resistance/conductivity measurements at high temperatures.
- Ensure that the knowledge generated within the project will be disseminated to other reference laboratories, industrial users, standards organisations and engineering designers.

Expected results and potential impact

The new measurement infrastructure established in this Joint Research Project will immediately provide the metrology that is urgently needed for the implementation of new European regulations (No. 305/2011) for insulation products used in industrial installations. New European products standards for industrial insulation (EN 14303 to EN14309, EN 14313 and EN 14314) require manufacturers of insulation products marketed in Europe to CE mark their product and declare the thermal conductivity/resistance values to a level that is three times tighter than current level of agreement between reference laboratories. A dramatic improvement in the current level of agreement, from 15 % to 5 %, will allow European manufacturers to meet these mandatory CE marking requirements and to market their high-temperature insulation products to European industry.

New composite thermal protection materials promise to be thinner, lighter, stronger and thermally more efficient than conventional materials. This offers enormous scope for improvements in performance, efficiency and in the competitiveness of European advanced manufacturing, including the aerospace and automotive 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 new infrastructure will provide reliable thermal conductivity measurement data and will enable the designers of fire engineering (Structural Eurocodes EN 1990 to EN 1999) and transportation safety systems to select the best performance thermal protection materials. This will help to reduce the rate of industrial disasters/incidents and save lives. The accurate thermal conductivity measurements that will become possible through this project will also enable the uptake of innovation and it will help many Key Enabling Technologies to progress through Technology Readiness Levels four and five.

- Develop new techniques for the next generation of national standard instruments.

The project is progressing towards achieving the new national standard level metrological instrumentation for thermal conductivity measurements that go beyond the state of the art. LNE has designed the new hot plate and the new cold plates for updating its homemade HTGHP. LNE has also performed for the first time the in-situ calibration of a set of 30 N type thermocouples that will be used to calibrate the temperature sensors of a commercial guarded hot plate and to measure temperature uniformities of hot and cold plates. MKEH has developed a theoretical heat transfer model for its new technique for measuring high temperature thermal conductivity. The heat transfer model allows the calculation of axial and radial heat flows at the boundaries of the metering section using temperature distributions measured along two radial grooves machined in each face of the sample. CMI and LNE have measured the emissivity of 2 high temperature paints with total hemispherical emissivity above 0.8, which are suitable for increasing the emissivity of the heater plates up to 800 °C. The 2 paints have the commercial references: AREMCO HiE Coat 840-M and Pyromark 2500 Flat Black. NPL and LNE have measured the thermophysical properties of the three new heater plate materials that were recommended by PTB. At temperatures up to 400 °C, both the silicon infiltrated silicon carbide (SiSiC CS11T from Ceramdis) and Machineable Aluminium Nitride (SHAPAL™ Hi M-soft) are opaque and have higher thermal conductivity than the nickel 201 that is currently used. CMI has presented the knowledge generated in EMRP HiTeMS (IND01) to the Thermo project, with regard to the drift and stability issues of base metal thermocouples. All partners participated in workshops on the uncertainty budget of the high-temperature guarded hot plate. PTB has developed an analytical mathematical model for the calculation of the standard-uncertainty of the thermal conductivity measured using a GHP.

NPL has improved and tested a new small guarded hot plate dedicated to measuring the thermal conductivity of thin polymer composites. This small guarded hot plate has a novel new type of heater plate, which includes features designed to reduce uncertainties when measuring thin specimens. The initial validation measurements following these improvements have given measured values within 2 % of the certified reference value for a polymethylmethacrylate, which is well within the combined uncertainty for the certified values and the target uncertainty for the new facility. This demonstrates that thermal conductivity of thin polymer composites or other thin materials with medium thermal conductivity can be measured accurately in steady-state conditions. The new small guarded hot plate is being developed as a national standard facility to provide traceable thermal conductivity measurements of thin materials (down to 2 mm thick) with medium thermal conductivity, and it will be used to provide a reference for other techniques, such as those based on transient methods.

NPL has developed a technique for measuring specimen surface temperature using thin butt-welded thermocouples positioned in a 0.1 mm diameter groove machined across the faces of specimens. The technique could be very interesting for many other applications where the temperature sensors must be as unintrusive as possible. NPL has also demonstrated experimentally that a malleable indium sheet with a particular textured surface (a commercial product) is more efficient than silicone paste or copper filled grease in reducing thermal contact resistance. This indium sheet is much easier to use than pastes and grease and can solve many problems where thermal contact resistance must be reduced or controlled.

The step-change improvements that will be made to reference instruments will enable European National Measurement Institutes to offer industry access to more comprehensive high quality facilities. They 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.

- Identify and develop reference materials with a thermal conductivity in the range 0.02 $W{\cdot}m^{-1}{\cdot}K^{-1}$ to 1 $W{\cdot}m^{-1}{\cdot}K^{-1}$ and aiming at temperatures up to 800 °C.

To resolve discrepancies and to enable agreement between reference laboratories, NPL is leading the development of high temperature thermal conductivity reference materials. So far, the initial tests of a short list of candidate high-temperature thermal conductivity reference materials have been completed. In total, NPL, MKEH, CMI, LNE and PTB carried out 15 test tasks agreed in the test matrix at the beginning of the project. A new approach has been developed in the project, which 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. In April 2014, NPL presented a paper titled “Provisional assessment of candidate high temperature thermal conductivity reference materials in the EMRP ‘Thermo’ project” at the 32nd International Thermal Conductivity Conference and the 20th International Thermal Expansion Symposium that was held at Purdue University, Indiana, USA. It has been peer-reviewed and is now being published by Purdue University. One of the reviewers of the paper commented “This paper describes a comprehensive analysis of thermo-mechanical properties in high temperature proof materials of particular interest. … This report of the systematic material characterizations is crucial to publish.” NPL’s presentation was then disseminated to a regulatory scheme for European commercial laboratories, the CEN Key-marking scheme via the chairman of the scheme. Based on initial tests on material composition and microstructure changes, dimensional stability, mechanical stability, chemical stability and uniformity, the two most promising candidate reference materials, a high density calcium silicate (HDCaSi-N) and a low density calcium silicate (LDCaSi) were selected for next stage full characterisation. These two materials are dimensionally, mechanically and chemically stable, more robust and easier to handle than others. However, the specimens need to be selected to meet the requirement for material uniformity in terms of density, i.e. density variation within 2 %. Stocks of the two selected candidate reference materials were purchased by NPL and CMI. The cutting plans and technical specifications for machining and heat treatment were agreed between all partners and subcontractors in Hungary. To speed up the preparation of the specimens, MKEH is coordinating the machining, heat treatment and dimensional and mass measurements of specimens.

First-of-their-kind reference materials will be developed with thermal conductivity in the range 0.02 $W{\cdot}m^{-1}$ K^{-1} to 1 $W{\cdot}m^{-1}{\cdot}K^{-1}$ and with a target maximum temperature of 800 °C. These reference materials will be made available to European industries and throughout the world to provide traceability to national standards of thermal conductivity. They will be used by reference laboratories to enable them to resolve discrepancies and to enable agreement between reference laboratories which use Guarded Hot-Plates. It is expected that the agreement will be improved by a factor of three from 15 % to 5 %, thus enabling European industry to meet the mandatory European regulations (No.305/2011) and to market their products.

- Systematically investigate the limitations of the transient and other industrial techniques.

Limitations and uncertainties in industrial measurement techniques will be investigated for their use in measuring the thermal conductivity of advanced thermal protection materials. PTB prepared a critical review of industrial techniques for thermal conductivity measurements of thermal protection materials. The manuscript was submitted to a peer-reviewed journal for publication. LNE wrote a review report on the application of the laser flash technique to anisotropic materials. The knowledge gained will be disseminated through consultancy to help industrial and academic users to select appropriate thermal conductivity measurement tools for research and design purposes.

- Theoretically and numerically determine the effect of radiant heat transfer on thermal resistance/conductivity measurements at high temperatures.

PTB is leading 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. LNE has performed normal hemispherical spectral transmittance and quasi-normal hemispherical reflectance measurements on five high temperature insulation materials at room temperature. The study has shown that it is possible to obtain experimentally radiative properties data on high temperature insulation materials. LNE compiled a list of suitable thermal protection materials to be characterised for their radiative properties. Based on experimental results, MKEH 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. CMI developed a numerical model to investigate the appropriate tolerance of the surface flatness of heater plates.

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.

- Ensure that the knowledge generated within the project will be disseminated to other reference laboratories, industrial users, standards organisations and engineering designers.

The knowledge gained in this project will be disseminated to standard committees, e.g. BIPM CCT WG9, EURAMET TC-T and CEN/TC 89 through presentations at international conferences and at committee meetings, and through recommendations to standards. In response to the CEN/TC 89 N 1526, the EMRP SIB52 Thermo project submitted comments to CEN/TC89 on CEN/TS 15548-1 (a technical specification for thermal conductivity measurement at elevated temperatures). A new heater plate material identified in the EMRP SIB52 Thermo project was recommended to the Technical Committee. Comments also made on the revision needed for specimen thickness measurements and for the temperature difference across specimen(s). Dissemination of the knowledge gained in the project will enable CEN/TC 89 to complete the new European measurement standards for thermal resistance/conductivity measurements of insulation at high temperatures. The European Standard Technical Committee, CEN/TC89 is in the process of establishing a new Working Group (WG14) to follow the work in the project. Two training videos have been made available to the public on the project website. Four workshops and three technical assessment visits were held to disseminate the knowledge among the consortium. One external workshop was held in the 32nd International Thermal Conductivity Conference. NPL’s presentation at the conference has been disseminated to a regulatory scheme for European commercial laboratories, the CEN Key-marking scheme.

JRP start date and duration:	01 June 2013 – 36 months
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