Improved metrology of thermophysical properties at very high temperatures

1st Newsletter

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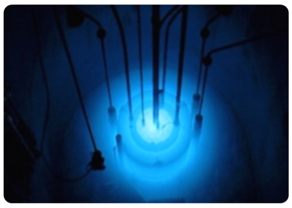
Introduction



This is the first newsletter of the EMPIR project "Improved metrology of thermophysical properties at very high temperatures" (Hi-TRACE). This three-year project was launched in July 2018 with the ambition to establish a metrological infrastructure composed of reference facilities providing industries with thermophysical properties data traceable to the SI for any solid material up to 3000 °C.

The objective of this project is to address the needs of space, glass, nuclear and aeronautics industries which develop new refractory materials and new processes that should securely and reliably work at very high temperatures. Although some apparatus for measuring thermophysical properties at such temperatures already exist in the concerned industries, it is often impossible to ensure the reliability and traceability of the measured data due to a lack of appropriate standard reference materials and reference facilities available to validate these measurement techniques. In order to fill this gap, the EMPIR project Hi-TRACE aims at developing reference facilities and new methodologies for measuring the thermal diffusivity, specific heat, normal emissivity and temperature of fusion of solid materials up to 3000 °C and the thermal contact resistance above 1000 °C.

Project description

The Hi-TRACE project is composed of four work packages, one for each thermophysical property: thermal diffusivity (WP1), specific heat (WP2), emissivity & temperature of fusion (WP3), thermal contact resistance (WP4). The structure of all these technical work-packages is similar:

Step 1 Development of reference facilities by the involved National Metrology Institutes (NMIs).

Step 2 Metrological validation of these reference facilities through inter-laboratory comparisons using pure solid materials, and validation of the existing setups and measurement techniques used by the industrial and academic partners against the newly developed facilities.

Step 3 Characterisation of refractory materials of technological significance at very high temperatures.

Hi-TRACE brings together eleven partners from five European countries: four NMIs, three industrial partners, two universities, and two research centres. It is coordinated by the French National Metrology Institute (LNE). With an overall budget of about 1.6 million Euros, it is receiving funding through EMPIR, the European Metrology Programme for Innovation and Research, which is co-financed by the Participating States and the European Union's Horizon 2020 research and innovation programme.

The Hi-TRACE consortium

Scientific and industrial impacts

An advisory board has been set up to regularly review project progress and to provide guidance in terms of relevance for the end users. Particular importance is placed upon ensuring the widest possible dissemination of the knowledge generated within the project, including to CEN and ISO standards bodies, as well as collecting feedback from end users, such as instrumentation manufacturers, actors in aerospace, nuclear energy, additive and conventional manufacturing involving very high temperatures.

Based on the project's results, a good practice guide for measuring the thermal diffusivity up to 3000 °C using the laser flash method will be issued. To further spread the results to the community a workshop for end-users will be organised and e-learning modules will be prepared to allow for an online tutorial. Finally, the datasets generated in the inter-laboratory comparisons will be made available through open repositories, as long as they are not covered by confidentiality agreements with the industrial partners.

A first dissemination action of the Hi-TRACE consortium was a specific training session related to thermophysical properties measurements provided to young researchers coming from European NMIs (from Turkey, Slovakia, Greece, Bosnia, Serbia, Italy) during the Thermal Metrology Summer School organized by Euramet TC-T in Thessaloniki on 17-21 September 2018.

Conferences and congress

Since its beginning, the project and the first achievements have been presented through oral presentations and posters at the following international events.

Tempmeko 2019 (10-14 June 2019)

TEMPMEKO CHENGDU CHINA 2019

http://www.tempmeko2019.cn

CIM 2019 (24-26 September 2019)

https://www.cim2019.com

34th ITCC (17-20 June 2019)

https://thermalconductivity.org/

Selected research highlights

Reference facility for thermal diffusivity measurements up to 3000 °C

The laser flash device of LNE has been modified to operate at very high temperatures by improving the inductive furnace used and by implementing new bi-chromatic radiation thermometers that have been calibrated up to 3000 °C. A specific filter has also been integrated in the HF generator in order to reduce high-frequency electromagnetic interferences (> 250 kHz) and to obtain exploitable curves at high temperature.

A protocol has been proposed to monitor in-situ the long term stability of the radiation thermometers using high temperature fixed-point cells positioned at the location of the specimens. This protocol has been tested up to now with three miniaturized metal-carbon eutectic fixed point cells (Pd-C at 1492 °C, Pt-C at 1738 °C and Ir-C at 2290 °C) designed at LNE. Preliminary measurements of thermal diffusivity have been performed on a graphite specimen from 2000 °C to 2995 °C.

LNE has also started the adaptation of this laser flash apparatus to specific heat measurements by drop calorimetry where the tested specimen is heated in the inductive furnace and dropped in a heat-flux calorimeter. The energy calibration of the calorimeter is achieved by electrical substitution using a new home-made *in-situ* electrical calibration system implemented in the heat-flux calorimeter (Calvet thermopiles).

High-temperature diffusivimeter of LNE

Pulse heating facility for specific heat measurements above 2300 °C

High temp. pulse heating facility of VINS

The pulse heating technique employs the temperature measurement either by radiation or contact thermometry. The facility developed at VINS uses both ways of temperature measurement which provide the data of the specimen spectral normal emissivity up to the temperature limit of the contact thermometry.

Above that limit, the obtained data are extrapolated, letting the recorded radiance specimen temperature to be converted to the absolute one. The latter values are then used to obtain the desired specific heat and other properties at very high temperatures, i.e. above 2300 °C.

The proposed principle of the simultaneous use of contact and radiation thermometry has been tested on a specimen of pure polycrystalline tungsten. Preliminary results show good agreement with literature data and suggest that radiance to absolute temperature conversion needs to be considered carefully due to its potentially significant influence on the final total measurement uncertainty of the measured properties.

Experimental Platform for dynamic material research ExdyMa

Based on a conventional laser flash set-up, FHWS plans to implement additional features in its ExdyMa facility to assess other thermophysical properties and to allow for advanced evaluation of layered systems.

In a first step an IR radiation thermometer for additional optical front side temperature measurement has been implemented allowing the simultaneous measurement of the front and back side temperature measurement after heating the sample with an instant laser pulse. It is intended to use the measurement of the front side temperature measurement for additional information to allow for a more detailed investigation of layered materials and, in particular, of the thermal resistance between the layers.

In further steps additional 2D measurement systems will be included in order to enable a localization of bad adherence between the layers and to quantify lateral heat flows.

Front side temperature measurement in ExdyMa

Drop calorimetry for specific heat measurements up to 3000 °C

The drop calorimeter at PTB uses a solid copper block as a receiver. The increase in temperature of the copper block caused by the rapidly dropped sample of a higher temperature is measured. Knowing the initial temperatures of the sample and of the copper block allows the calculation of the sample's specific heat.

At the envisaged high temperatures, it is mandatory to perform all experiments in vacuum or under a protective atmosphere. To avoid opening the instrument to remove the sample after it has been dropped into the calorimeter, a lifting mechanism was constructed enabling consecutive experiments. A finite-element calculations support the corrections necessary for the determination of the sample temperature which decreases due to radiation during its fall into the calorimeter. Similarly, the finite-element calculation serves to optimise the calorimeter design with the aim to model the temperature fields and thus the heat losses during the experiment and the electrical calibration.

Inverse heat transfer models for quantifying thermal contact resistance in layered materials at high temperatures

Delamination in the interlayers of multi-layer systems can cause a degradation in functionality, stability and life span of that system. This is even more pertinent for systems at high temperatures. Laser flash analysis (LFA) has long been used for measuring thermal diffusivity of layered materials at high temperatures. One of the aims of the project is to develop multi-layer reference artefacts with and without debonded regions for validating the thermal characterization of such systems using LFA.

As a first step, NPL further developed a control volume based forward numerical heat transfer model for multi-layer material systems in LFA measurements. It has been validated against FEA simulation using the COMSOL MultiPhysics package and literature. The NPL model considers the thermal interface conductance between partially or fully debonded layers, without needing the details of surface topography and other interface material properties. The model has been used for sensitivity study to aid in the design of multi-layered reference artifacts. The simulation study has found that the area and thermal conductance of the defect had the greatest effect on the heat transfer through the multi-layered material systems, with shape and radial location having a significant but lesser effect in comparison. The results of the study were presented at the 34th ITCC in June 2019 in USA.

In the second step, an inverse heat transfer model is being developed to calculate the thermal interface conductance between partially or fully debonded layers from the thermogram obtained in LFA experiments. The inverse heat transfer model is being validated using two multi-layered material systems, a BCR724 (Pyroceram 9606)-Al tape- BCR724 (Pyroceram 9606), and a pure Cu-tape-pure Cu system at room temperature. At the same time, the high-temperature multi-layered material systems are being developed and the selected systems will be fully characterized next.

Upcoming events

Workshop for stakeholders

A workshop will be organized on 8th September 2020 at ZAE Bayern in Würzburg (Germany). The goal of this workshop will be to present the main results of the project to stakeholders and end-users and to get feedback from them.

More information about the workshop will be available on the project's website (https://hi-trace.eu) as soon as the program is finalized.

Members of the Hi-TRACE consortium will be present at the following conferences:

- **European Conference on Thermophysical Properties - ECTP 2020**
  14-17 September 2020, Venice, Italy

- **International Confederation for Thermal Analysis and Calorimetry - ICTAC 2020**
  30 August - 04 September 2020, Krakow, Poland

Contact details

For enquiries about the project, its partners, results and stakeholder relations, please contact Bruno Hay (project coordinator) at LNE (bruno.hay@lne.fr). This newsletter together with all information about the project is also available online on the project's website at https://hi-trace.eu.

Advisory board

Are you interested in the results of the Hi-TRACE project and do you wish to be informed regularly? Join us as a member of the advisory board of the project and benefit from synergies within this project.

To register as an advisory board member, please contact Bruno Hay (bruno.hay@lne.fr).

The consortium members at the second progress meeting in Belgrade (Serbia) in April 2019

EMPIR EURAMET
The EMPIR initiative is co-funded by the European Union's Horizon 2020 research and innovation programme and the EMPIR Participating States