Final Publishable JRP Summary for IND01: HiTeMs
High temperature metrology for industrial applications (>1000 ºC)

Overview

This project developed techniques for industrial users to more accurately measure temperatures above 1000 ºC. This research advanced the state of the art in temperature measurement by developing methods to compensate for sources of uncertainty in radiation thermometry, to re-calibrate contact thermometers *in situ*, and to develop and characterise the performance of non-standard contact thermometers. These new measurement techniques will be used across a range of industries to better control high-temperature processes, improving energy efficiency, ensuring uniform product quality, and minimising emissions.

Need for the project

The accurate measurement of high-temperatures is vital to a wide range of EU industries. Using the right temperature ensures that (i) processes are energy-efficient, minimising production costs and greenhouse gas emissions; (ii) that components produced at high-temperatures consistently meet quality standards (e.g. are not damaged through overheating), and (iii) that process temperatures do not exceed safety standards (for instance in the nuclear industry). Ultimately, the more accurate measurement of temperatures will support improved EU industrial competitiveness whilst minimising its environmental impact.

Although high-temperature processes are used routinely in industries such as aerospace/space (~1300-3000 ºC); nuclear (~1800->2500 ºC); refractory metals production (>2500 ºC); silicon carbide, carbon and carbon composites production (>2800 ºC); iron, steel, glass and ceramics production (2000 ºC); measuring temperatures above 1000 ºC is difficult. Improved measurement techniques are required that accurately and consistently measure temperatures above 1000 ºC, and do so *in-situ* (during the process), to ensure that high-temperature processes can be controlled effectively.

Industry use two broad approaches to measure high-temperatures: **contact thermometry** that uses temperature sensors, such as thermocouples and **non-contact thermometry** that measures temperature at a distance by analysing the thermal radiation an object emits at different temperatures. Both approaches present challenges at high temperatures. The performance of thermocouples changes considerably over time (mainly through undetected but significant drifts) and the hostile environments of high-temperature processes restricts access for servicing and re-calibration. Novel thermocouples based on non-standard materials could be better suited to high temperature applications, but their use is limited as their performance is not fully understood. Non-contact-thermometry offers many advantages over contact thermometry in high temperature environments and while it is increasingly used in a range of industries, it suffers from a number of sources of measurement uncertainty such as unknown emissivity (different materials emit light differently) and high levels of background thermal radiation.

Scientific and technical objectives

The project aimed to improve the reliability of temperature measurement at high temperatures and to address specific problems associated with the hostile environment for both contact and non-contact thermometry. Its objectives were to develop:

Contact thermometry:

1. <u>Rigorous, traceable techniques to enable lifetime testing and stability evaluation of contact temperature sensors above 1000 ºC</u>. There was no agreed method of drift and lifetime testing of thermocouples. The project will develop a lifetime and drift testing capability, including the development of an authoritative EURAMET guide for performing such tests.

2. <u>Self-validating contact temperature sensors for use from 1000 ºC to above 2000 ºC demonstrated.</u> Contact thermometry sensors drift, sometimes very rapidly, when exposed to high temperatures. The

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project will improve in-process temperature measurement by an uncertainty factor of 10 or more through developing novel self-validating thermocouples.

3. A methodology for the rigorous determination of 'reference functions' for non-standard thermocouple types. Thermocouples suitable for high-temperature processes and harsh environments are currently not used as their reference functions have not been defined. The project will develop tools in the EU national measurement institute (NMI) community to rapidly define the reference functions of non-standard thermocouples (and potentially other temperature sensors).

Non-contact thermometry:

4. Traceable and accurate radiation measurement techniques for *in-situ* surface high temperature measurements above 1000 ºC. Temperature measurement by radiation thermometry is a rapidly growing measurement method in a broad spectrum of industries. The project will develop solutions to the significant accuracy limiting factors; unknown emissivity, background radiation and size of source effect.
5. *In-situ* validation methods for non-contact thermometry to 2500 °C, including demonstrating novel drift correcting techniques. At high-temperatures, radiation thermometry accuracy is limited by window contamination (which also requires scheduled stoppage for cleaning or replacement), and shifts in calibration. The project will demonstrate *in-situ*, quasi-real time correction of these two intractable problems by incorporating known, stable high-temperature references within the industrial process.
6. A demonstration of *in-situ* traceable temperature measurements for exotic thermal processing methods such as laser hardening. The project will demonstrate traceable and accurate non-contact thermometry in a challenging industrial setting, where it has not been achieved before. Laser hardening has been selected as it is an industrial technique used widely throughout the EU.

Results

This pan-European project developed a range of techniques and capabilities to more effectively measure high temperatures in hostile environments. Significant developments were made in the following areas:

1. Rigorous, traceable techniques to enable lifetime testing and stability evaluation of contact temperature sensors above 1000 ºC

Methods in use before this project were *ad-hoc*, often differing between sensor manufacturers and users, leading to misunderstanding of results. The project developed a standard experimental process and NMI facilities traceable to the International Temperature Scale of 1990 (ITS-90) to test the lifetime and drift of base metal thermocouples and the drift of noble metal thermocouples. This work specifically enabled drift to be accurately measured for base metal thermocouples up to 1300 °C (short-term) and 1100 °C (long-term) and for noble metal thermocouples up to 1820 °C (short-term) and 1720 °C (long-term). Lifetime and drift results were obtained for a range of available base thermocouples, these results have been published, or are in press, for use by industrial users.

A EURAMET guide was developed to explain how to perform such tests. This is available for free download from the EURAMET website, allowing both NMIs and manufacturers to adopt the technique.

2. Self-validating contact temperature sensors for use from 1000 ºC to above 2000 ºC demonstrated

At high temperatures the accuracy of contact thermometers can drift rapidly and dramatically. The objective was to develop thermocouples that can self-calibrate *in situ* removing the need to remove them for calibration. This was achieved by developing and validating two novel *in-situ* calibration methods:

In the first approach, fixed points with known melting points were used as references, incorporated within the measurement junction of the thermocouple. These could be then incorporated within the measurement process to detect thermocouple drift *in-situ*. The project identified suitable materials to be used as miniature high-temperature fixed points (HTFPs) at temperatures above 2000 °C in non-oxidising environments, and up to 1800 °C in oxidising environments. The former were demonstrated in combination with mineral insulated metal sheathed (MIMS) W/Re thermocouples. The detection and correction of measurement drift was successfully demonstrated with *in-situ* traceability with uncertainties as low as 2-3 °C possible.

The second approach combined an electrical noise thermometer and a thermocouple into one device, to provide a second measurement of temperature alongside the thermocouple. Although electrical noise thermometers are accurate, they make temperature measurements too slowly for most industrial processes. The project demonstrated the concept of checking and re-calibrating the thermocouple periodically (every 20 minutes) against an electrical noise thermometer. The detection and correction of measurement drift as low as around 2 °C was successfully demonstrated.

Both approaches demonstrated the proof-of-concept for *in-situ* thermocouple calibration. This work is being carried forwards by EMPIR project EMPRESS to refine these techniques into commercial thermocouples appropriate for use in industrial settings.

3. A methodology for the rigorous determination of 'reference functions' for non-standard thermocouple types.

High temperature processing is inevitably accompanied by harsh environments and operating conditions that make reliable temperature measurement challenging. Some environments are not suitable for the thermocouple types that are currently available (such as nuclear reactors) and the use of non-standard thermocouple types could be beneficial. However the lack of reliable accurate reference functions for such sensors has limited their uptake.

The project developed a European distributed capability for determining low uncertainty reference functions for thermocouples with demonstrated traceability to the ITS-90. The facility for determining the reference function was based on a number of high temperature fixed points and radiation thermometer comparators. These were distributed in four NMIs throughout Europe (CEM, LNE-CNAM, NPL and SMU). The facility's effectiveness was demonstrated through determining the reference function of the non-standard Pt-40%Rh / Pt-20%Rh thermocouple (which is useful for continuous use up to 1800 °C and occasional use to 1850 °C).

4. Traceable and accurate radiation measurement techniques for *in situ* surface high temperature measurements above 1000 ºC.

The accuracy of non-contact radiation thermometers is limited by unknown emissivity of the surface being measured, background radiation, and the size of source effect. Although these are challenging technical problems, some progress was made towards their mitigation.

To deal with the problem of unknown emissivity, multi-wavelength thermometry at short wavelengths and an active two colour thermometer was developed, and demonstrated in the laboratory.

Gold-cup radiation thermometers (1a) are used extensively in industry, and can, in principle, be used to simultaneously mitigate emissivity and reflected (background) radiation. However, the results provided by this type of radiation thermometer is significantly influenced by the surface geometry, with tubular and sloping surfaces commonly encountered in process industries. VSL built a facility (1b-d) where gold-cup radiation thermometers could be characterized properly with different surface geometries (1c-d). Characterisation results have shown that the uncertainty associated with the measurement on a curved surface at a temperature of 700 °C could be as high as 3.5 °C (k = 2), which accounts for more than half of the total uncertainty (6.1 °C) of the measurement. For sloping surfaces, an uncertainty contribution of the surface geometry amounts to 0.4 °C (at a maximum angle of 20°), with the overall uncertainty of 5.4 °C. This facility is available for use by industry to help characterise the uncertainty in the use of gold cup pyrometers in particular industrial settings.

In addition a new approach to characterising size-of-source effect (SSE), one of the significant contributors to the measurement uncertainty in radiation thermometers, was developed, as well as new approaches to determining surface emissivity.

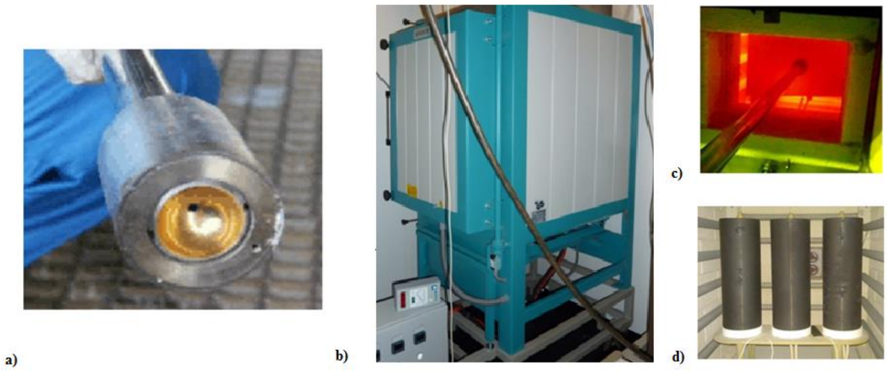

Figure 1 Facility for industrial size-of-source characterization developed at VSL.

<u>5*. In situ* validation methods for non-contact thermometry to 2500 °C, including demonstrating novel drift correcting techniques.</u>

One of the difficulties at the highest temperatures is to monitor and control temperature in harsh environmental conditions. For radiation thermometers the most likely cause of measurement drift over time are intrinsic stability of the thermometer itself or progressive contamination of the viewing window.

The project developed robust high-temperature fixed point blackbodies, which produce known temperatures between 1000 ºC and 2500 ºC. These were successfully tested in a hostile environment at the French Alternative Energies and Atomic Energy Commission (CEA) to determine the effect of progressive window contamination (though they could equally be used for determining the stability of the radiation thermometer).

The cell design is shown in Figure 2.

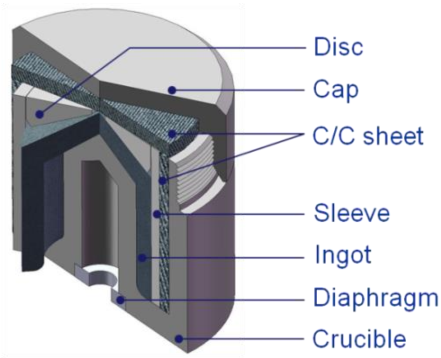

Figure 2 Design of the rugged HiTeMS HTFP cell for use for *in-situ* corrections for window transmission and radiation thermometer drift.

High temperature fixed point blackbodies were constructed from Co-C (~1324 °C), Ru-C (~1953 °C) and Re-C (~2474 °C). Their robustness was demonstrated through severe thermal cycling in the laboratory – no breakage was observed – even when very rapid and very wide temperature excursions were experienced. A number of these HTFPs were then transported to CEA and successfully used *in-situ* to demonstrate the possibility of window transmission measurement in real time. Understanding the change in window transmission is very important at the CEA facilities as they are used for essential nuclear power safety testing.

<u>6. A demonstration of *in-situ* traceable temperature measurements for exotic thermal processing methods such as laser hardening.</u>

Laser hardening is an industrial mass production technique in which components are heat treated with a high-powered laser that results in a thin surface layer which is much harder than the untreated material. A precise temperature must be used, typically within a range of 20 °C at 1200 °C, to ensure the right hardening is achieved. Although accurate temperature measurement and control is vital, no standardised temperature measurement technique is available. The objective was to develop a means to calibrate *in-situ* the radiation thermometers that are used to control laser heat treatment because the temperature measurement devices are mechanically and electrically integrated into complex machine systems and so cannot be easily removed for recalibration. Such calibration is important because pyrometer output changes due to window and fibre optic bundle transmission changes over time – affecting the quality of the process control.

The project developed and validated a mobile induction-heated fixed point device for calibration at 1000–1500 °C *in-situ.* It was used to evaluate the absolute accuracy of the temperature measurement systems at two industrial sites using laser hardening - Fraunhofer IWS and Alotec Dresden GmbH, Germany. Large deviations of up to several 10 ºC were found, due to large uncertainties introduced from the continuous contamination of the optical components from process fumes and mechanical damage to the optical fibres.

Finally to improve measurement accuracy, and to ensure robust traceability to ITS-90, the emission spectra of a number of materials commonly subjected to laser hardening were determined in order to establish a database of emissivity values.

Actual and potential impact

The HiTeMS project achieved significant impact during the lifetime of the project, and we anticipate our results will have a substantial ongoing impact, as temperature measurement is pervasive in a broad range of industries.

Dissemination of results

To ensure the uptake of the techniques developed, the consortium engaged in a variety of activities to ensure the user community were well informed of the project's developments. 27 papers were published in science and trade journals in the Czech Republic, Germany, The Netherlands, Spain and the UK (listed in the next section). And results were presented at international conferences and workshops; including; two industry workshops held during the project, attended by NMI researchers, industrialists (potential users) and sensor manufacturers; and a number of presentations were given at trade fairs, including the 2012 Hannover trade fair.

Early impact on industry

The EURAMET guide for lifetime drift testing of thermocouples is currently being used by a number of contact thermometer manufacturers to test their products. The performance details they produce will help industrial users to select the most appropriate thermocouples for their processes.

During the production of the guide, a number of manufacturers provided sample thermocouples for testing. Feedback on their performance has enabled the manufacturers to better understand the limitations of their sensors, allowing them to improve their performance.

Potential future impact on industry

The approaches we demonstrated for re-calibrating thermocouples *in situ* will be further developed in EMPIR project EMPRESS. Self-calibrating thermocouples suitable for use in industrial settings will be constructed and trailed by 2018.

The facility for determining reference functions for novel thermocouples has allowed us to investigate new thermocouple types, and will be used by the EMPIR EMPRESS project to develop a high-temperature, low/no drift thermocouples. These thermocouples will be trialled in industry before the end of the project in 2018. An industrial partner is keen to exploit this development.

The facility for investigating gold cup pyrometer performance is now available for industrial users to estimate and address the sources of temperature uncertainty in industrial processes.

The use of high-temperature fixed point blackbodies to determine window transmission has the potential to significantly improve temperature measurement for essential nuclear safety testing, leading to the safer use of nuclear reactors for power generation.

The demonstration of accurate *in-situ* temperature measurement during laser hardening has the potential to dramatically improve process control in this growing industry.

Over the longer-term we expect the approaches developed here will be adopted by industry, or further refined in current and future EURAMET projects, to more accurately measure temperature in high-temperature processes. These techniques will improve process control and safety, and product quality; ultimately resulting in gains in industrial competitiveness, and reductions in environmental impact, for EU industry.

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