EMPRESS: A European Project to Enhance Process Control Through Improved Temperature Measurement

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Abstract

A new European project called EMPRESS, funded by the EURAMET's European Metrology Programme for Innovation and Research (EMPIR), is described. The three-year project, which started in the summer of 2015, is intended to significantly enhance the efficiency of high value manufacturing processes by improving temperature measurement capability. The project consortium has 18 partners, from the metrology community, high value manufacturing industry, sensor manufacturing, and academia. Accurate control of temperature is key to ensuring process efficiency and product consistency, and is often not achieved to the level required for modern processes. Enhanced efficiency of processes may take several forms including: reduced product rejection/waste; improved energy efficiency; increased intervals between sensor checks/maintenance; and increased sensor reliability, i.e. reduced amount of operator intervention. Traceability of temperature measurements to the International Temperature Scale of 1990 (ITS-90) is a critical factor in establishing low measurement uncertainty and reproducible, consistent process control. Introducing such traceability in-situ (i.e. within the industrial process) is a theme running through this project.

Introduction

This article describes a project funded by the 2014 Industrial call of EURAMET's European Metrology Programme for Innovation and Research (EMPIR) entitled 14IND04 EMPRESS (enhanced process control through improved temperature measurement) [1]. This project both builds on the European Metrology Research Programme (EMRP) project HiTeMS [2] and introduces a number of completely new research approaches. The consortium comprises 18 partners from the metrology community (National Metrology Institutes (NMIs) and Designated Institutes (DIs)), high value manufacturing industry, and academia (Table 1). Through this project the uncertainty of temperature measurement in high value manufacturing processes are expected to be significantly reduced by developing new sensors which are less susceptible to calibration drift in-process; introducing traceability to the International Temperature Scale of 1990 (ITS-90) [3] directly into the process; and through developing novel calibration techniques.

Short Name	Organisation legal full name	Type of organisation	Country
NPL	National Physical Laboratory (Coordinator)	NMI	United Kingdom
BRML	Biroul Roman de Metrologie Legala	NMI	Romania
CEM	Centro Español de Metrología	NMI	Spain
CMI	Český Metrologický Institut Brno	NMI	Czech Republic
DTI	Teknologisk Institut	NMI	Denmark
DTU	Danmarks Tekniske Universitet	DI	Denmark
INRIM	Istituto Nazionale di Ricerca Metrologica	NMI	Italy
JV	Justervesenet	NMI	Norway
PTB	Physikalisch-Technische Bundesanstalt	NMI	Germany
Elkem	Elkem AS Technology	Company	Norway
Gamma	Gamma Forgiati SRL	Company	Italy
MUT	MUT Advanced Heating GmbH	Company	Germany
STRAT	University of Strathclyde – Advanced Forming Research Centre	University	United Kingdom
UC3M	Universidad Carlos III de Madrid	University	Spain
UCAM	University of Cambridge	University	United Kingdom
UOXF	University of Oxford	University	United Kingdom
BAE	BAE Systems	Company	United Kingdom
CCPI	CCPI Europe	Company	United Kingdom

Table 1. EMPRESS project partners.

The project has four scientific and technical objectives, one per workpackage (WP):

- WP1: To develop novel low drift temperature sensors for enhanced production and temperature control. The objective is to develop new sensors, suitable for high data capture rate, in the same format as current sensors. The target is to have in-process traceable uncertainty of better than 3 °C at temperatures around 1450 °C, and better than 5 °C at temperatures > 2000 °C over about 3 months continuous use.
- WP2: To develop non-drift contact sensors optimised for heat treatment applications to temperatures around 1350 °C. The sensors should be able to remain in service with a stability of better than 1 °C for at least 6 months. The sensors are expected to be trialled in-process in at least one industrial setting, e.g. heat treatment of gas turbine components.
- WP3: To develop traceable surface temperature measurement methods (contact methods) to enhance materials/chemical processing to around 500 °C. The methods should allow the calibration of surface temperature sensors, using at least one novel surface temperature approach and be used to demonstrate improved temperature measurement in at least two industrial settings, e.g. forming and forging.
- WP4: To develop an in-situ combustion standard of known temperature for the validation of flame temperatures. The combustion standard should have an uncertainty a factor of 10 lower than current methods and be tested in at least two industrial settings.

- WP5: To ensure that the outputs from the project are effectively disseminated to and exploited by the high value manufacturing sector (e.g. automotive, aerospace, casting, heat treatment, sensor manufacturers). To support the development of new, innovative products and services enhancing the competitiveness of EU industry.

In all the methods developed, the integrated involvement of national metrology institutes is intended to ensure traceability to the ITS-90.

The project

The European aerospace industry is a substantial entity, making a major contribution to the economy and supporting an extensive supply chain [4]. Key areas of research and development are: improving engine efficiency [5,6]; reducing airframe weight (via, in particular, very temperature sensitive forming processes and novel metal alloys with formidable forming challenges), and implementing more efficient production processes [6]. Nearly all of these require improved temperature measurement capabilities, either directly or indirectly [7]. These challenges are shared by many other high value manufacturing industries including manufacturing in automotive, marine manufacturing, and power generation [8]. The general scheme is summarised in Figure 1.

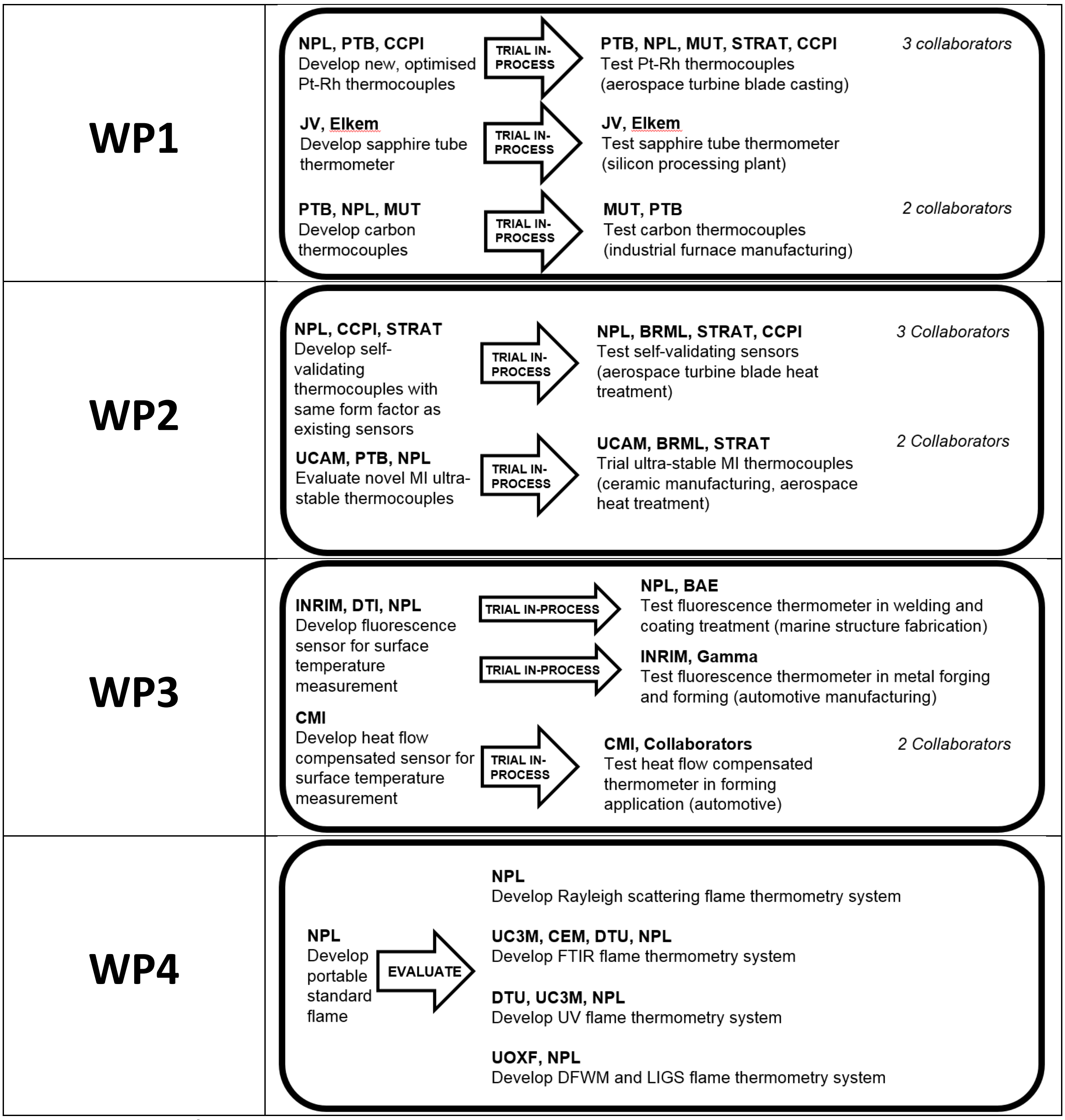


Figure 1. Schematic of the collaboration.

WP1: Low-drift contact temperature sensors

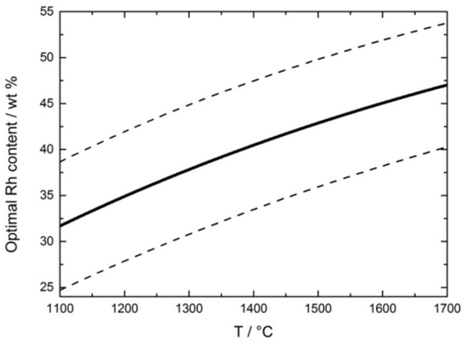
Thermocouple stability is a major issue [9] and is a major consideration in key standards e.g. AMS 2750E [10]. The aim of this WP is to identify new Pt-Rh thermocouples with superior thermoelectric stability to the standard Types R, S, and B [11] using high temperature fixed points (HTFPs) [13], and develop a reference function for the new optimal type to supplement the provisions of standards such as ASTM E1751 [12]. For environments where thermocouples are not suitable, optical-based sensors are under development using single-crystal sapphire light guides attached to a miniature blackbody.

A key development has been the establishment of two separate models of Pt-Rh thermocouple stability based on transport of Pt and Rh oxide vapour to guide the selection of Pt-Rh alloys in thermoelectric stability measurements [14,15]. Key predictions of the two models are shown in Figure 2. The left panel of Figure 2 shows the Rh composition (weight percent) as a function of temperature to ensure that, thanks to the nature of Pt and Rh oxide evaporation, there is no change to the wire composition, i.e. the wire remains thermoelectrically stable. The right panel of Figure 2 shows the composition that gives the optimum stability (the two axes show the two wire Rh compositions (weight percent)).

Stability measurements are being performed, using HTFPs, of 7-wire thermocouples, each wire having a different Pt-Rh alloy. At the time of writing the thermocouples are undergoing long-term exposure to selected high temperatures with periodic re-calibration to quantify the drift characteristics with unprecedented accuracy. The results to date of the long-term drift measurements at NPL (repeated in-situ calibrations with the Co-C HTFP, 1324 °C) and PTB (progressively increasing temperatures 1315 °C, 1350 °C, 1400 °C) are shown in Figure 3. The optimum thermocouple identified in this study is expected to be trialled in a suite of aerospace manufacturing applications including casting and heat treatment of gas turbine components.

At PTB a carbon thermocouple has been constructed. A preliminary reference function has been obtained up to about 400 °C (Figure 4). The device comprises of a closed-end carbon tube, within which sits a carbon rod which contacts the base of the tube, all encased in an inert gas atmosphere to prevent oxidation of the graphite. The temperature at the top is measured with a Pt-100 resistance thermometer to provide the reference junction temperature. Copper lead-out wires permit measurement of the voltage. Figure 4 also shows a low temperature trial reference function of the device. Development of the device is continuing in collaboration with industrial furnace manufacturer MUT.

At JV and Elkem a contact thermometer based on a blackbody and sapphire light guide has been constructed and is undergoing tests. The device and preliminary results are shown in Figure 5. Further developments are underway with the aim of implementing the device in-process at Elkem to address challenges in high volume silicon processing.



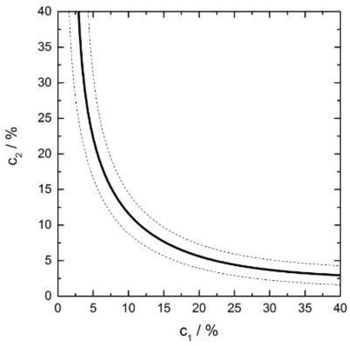


Figure 2. Predictions of the two different models. Left: optimal Rh content (i.e. composition) of a Pt-Rh wire as a function of temperature (vapour transport model). Right: prediction of the optimal composition of both wires at 1324 °C (empirical model). Dashed lines represent the uncertainty (coverage factor $k = 2$) of the solid line, i.e. 95 % of deviations are expected to lie within the two dashed lines.

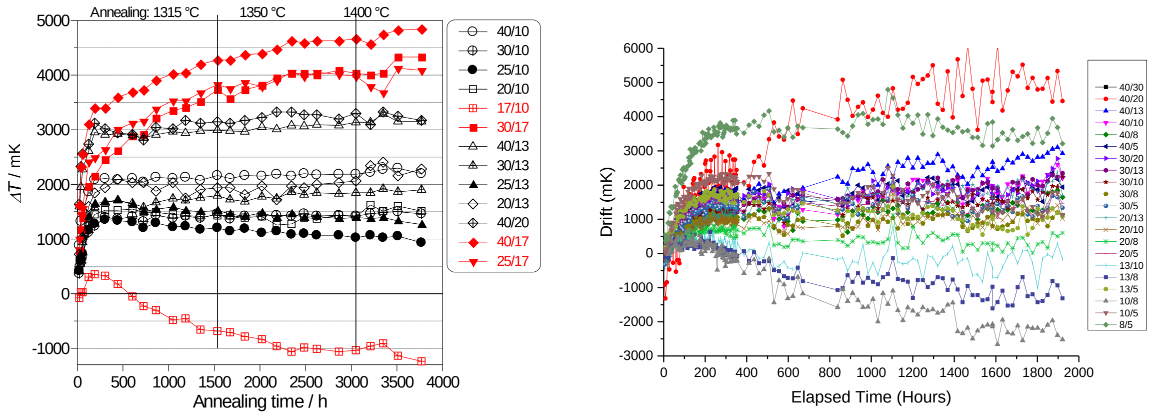


Figure 3. Pt-Rh thermocouple drift as a function of time, at PTB at the temperatures indicated across the top (left) and at NPL as measured in-situ with the Co-C HTFP (right).

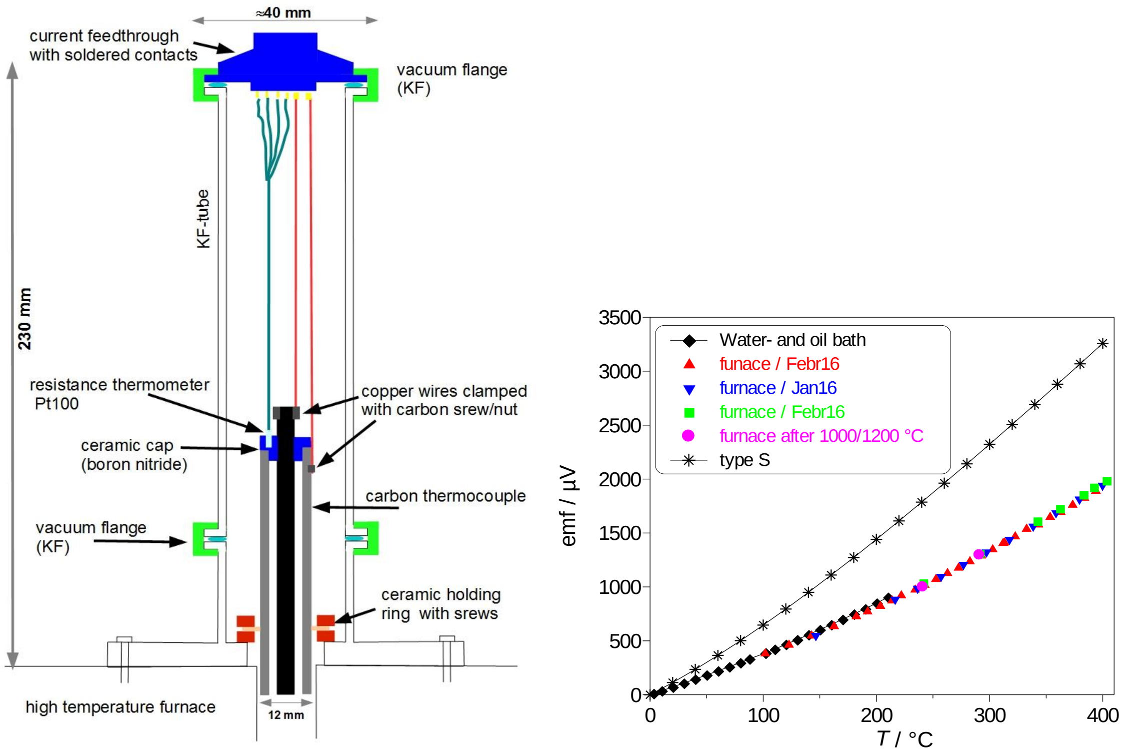


Figure 4. Graphite thermocouple (left) and low temperature reference function (right); the Type S thermocouple output is shown for comparison.

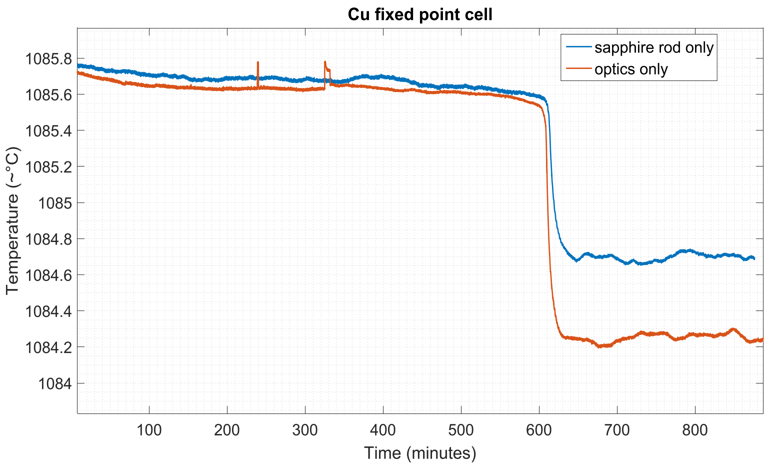


Figure 5. The JV-Elkem blackbody-based device. Left: indicated temperature of the sapphire rod blackbody thermometer during the copper freeze, showing data with and without optical-based corrections (the initial part of the freeze has been truncated). Right: Several instances of the device.

WP2: Zero-drift contact temperature sensors

For long-term processes, the unknown drift of the control or monitoring thermocouples can cause serious errors in the reading. Self-validating thermocouple prototypes, which make use of a HTFP reference mounted on the tip of a thermocouple to provide traceable *in-situ* calibration, have been successfully used with refractory thermocouples in a space application [16,17]. This project is expected to reduce the dimensions of the HTFP to a size commensurate with use in industrial environments.

An extremely small HTFP has been designed and constructed, being small enough to fit just below the twin-bore insulator of a noble metal thermocouple, inside an alumina sheath of inner diameter 5 mm, yet able to realise melting plateaus with a duration of minutes. Ingots of Cu and Co-C have been trialled. Figure 6 illustrates the integrated HTFP scheme. Figure 7 shows typical melting and freezing curves obtained with the new self-validating thermocouple, which are surprisingly well defined given the tiny size of the fixed point crucible. The next step is to demonstrate practical viability by proof-of-concept testing in a full-scale aerospace heat treatment facility at STRAT.

At UCAM, a novel ultra-stable mineral insulated, metal sheathed (MI) cable has been manufactured. The thermocouples have undergone rigorous assessment at PTB to characterise the effect of temperature cycling. Specifically this means increasing the temperature to 1350 °C by using heating rates of 5 K/min, keeping the temperature of 1350 °C stable over a period of 25 h and cooling down to 300 °C at a rate not exceeding cooling rates of -5 K/min. Altogether 90 cycles are intended. After 10, 20, 40, 60, and 90 cycles a stability test is performed at the melting point of Co-C HTFP. Industrial trials of the new thermocouples in an aerospace heat treatment facility are anticipated in early 2017.

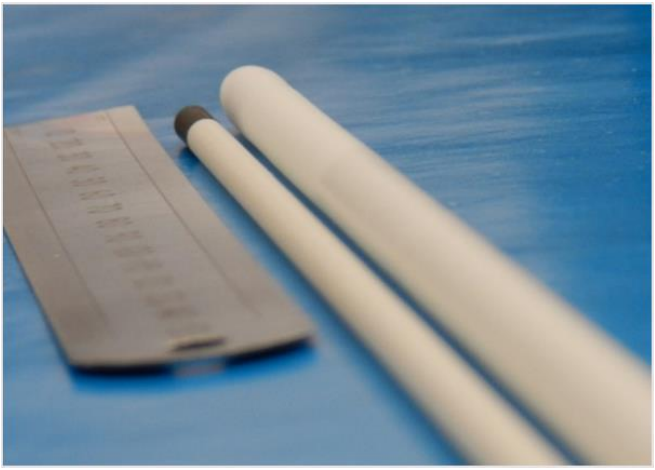


Figure 6. Integrated HTFP (to the left, seen at the end of the twin-bore ceramic tube) and thermocouple pictured alongside the ceramic protective sheath (to the right). The sheath has an outer diameter of 7 mm; the cell has an outer diameter of 4 mm and a length of 8 mm.

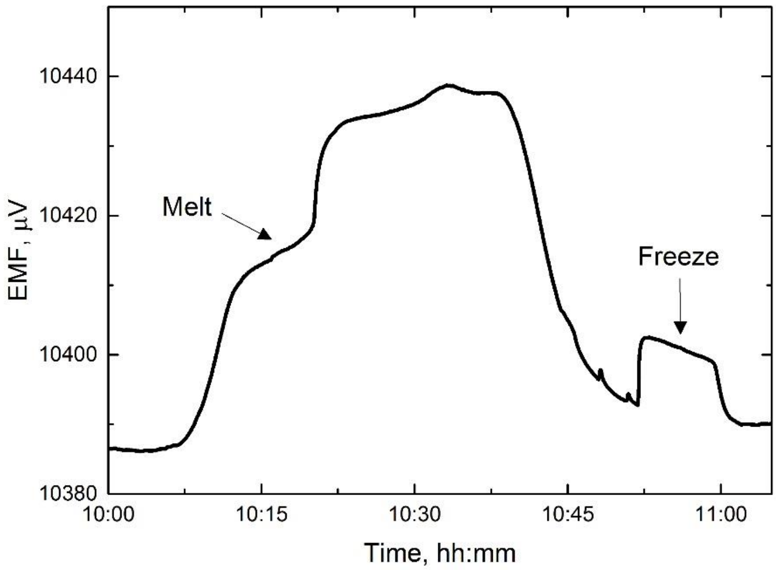


Figure 7. Typical melting and freezing curves obtained with the integrated miniature HTFP, showing Type S thermocouple emf as a function of time during melting and freezing.

WP3: Traceable surface temperature measurement with contact sensors

Despite contact surface probes having manufacturer stated accuracy of the order of 0.3 °C [18], in reality errors of tens of degrees may arise [19]. Standards governing coating of metals require surface thermometers accurate to ± 0.5 °C [20] which is currently unachievable.

An alternative is fluorescence thermometry. This involves irradiating a phosphor applied to the surface with light and observing the decay in the radiant intensity, which is a characteristic function of temperature. By painting a phosphor onto a surface, the surface temperature may be obtained without the heat flow problems associated with contact thermometers, and without the problem of unknown emissivity that besets non-contact thermometry. The goal of this WP is to develop two new techniques for traceably calibrating contact temperature probes based on fluorescence thermometry [21] (Figure 8), and a dynamically compensated heat flux probe.

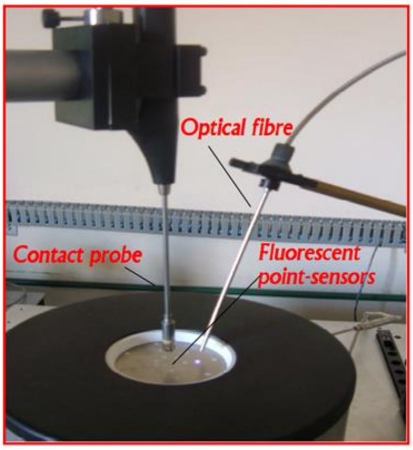


Figure 8. Prototype phosphor thermometer at INRIM.

The INRiM phosphor thermometer is shown in Figure 8. A thin layer of the temperature-sensitive phosphor is coated on the surface under test. The phosphor is irradiated using a laser diode. The optical signal is converted into an electrical signal to infer the fluorescence lifetime, τ [22,23] which is related to temperature through a pre-determined calibration. Mg_4FGeO_6:Mn has been selected [22,23] which can be used to more than 700 °C [24]. The temperature resolution of INRiM's technique is better than 0.05 °C up to 350 °C. The repeatability and uniformity both amount to 0.1 °C, and the achievable uncertainty is estimated to be within 1 °C, subject to further investigation [25].

NPL's device uses a bright LED (Figure 9). Figure 10 shows a measurement of τ for a sample at room temperature. Over the temperature range from 50 °C to 400 °C, the Type A uncertainty of 5 °C for measurement time of 1 s can be achieved, or 1 °C for measurement time of 20 s. The full uncertainties have not yet been determined and will include factors that depend on, for example, excitation strength, coating thickness, and bonding method. Improvements to the excitation scheme are anticipated which should reduce the measuring time required for a given uncertainty.

The NPL scheme is intended to be applied to welding challenges in marine manufacturing, while the INRiM device will be applied to hot-forging of aluminium alloy billets.

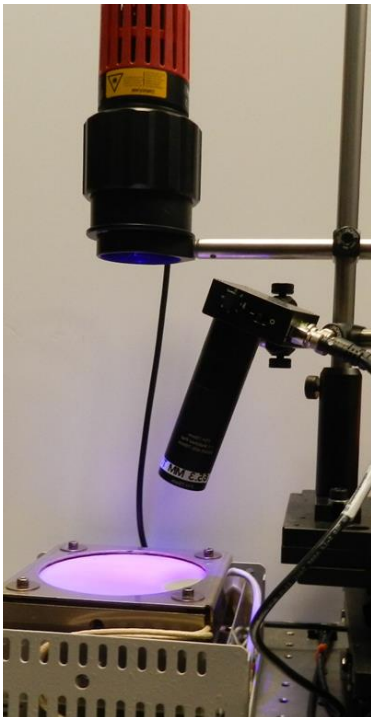


Figure 9. NPL phosphor thermometry system.

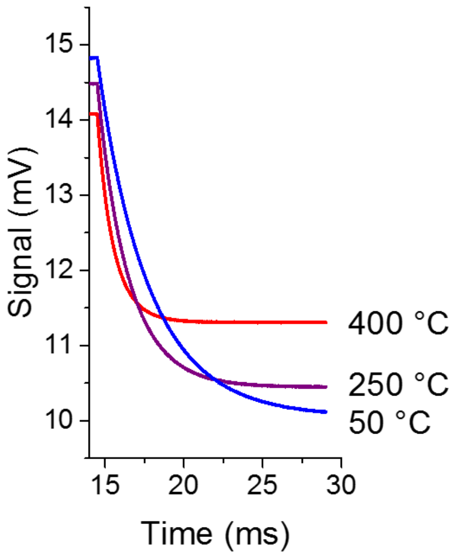


Figure 10. Phosphorescence decay curve for Mg_4FGeO_6:Mn.

CMI have developed a contact surface probe calibrator with interchangeable surface materials, in conjunction with a probe that eliminates heat flow errors by applying localised heating along the stem of the probe to cancel out heat flow away from the probe tip. The overall calibration uncertainty is currently under investigation.

WP4: Traceable combustion temperature measurement

The most accurate means of measuring temperatures of combustion processes are exotic optical methods, but uncertainties of up to 10% of temperature remain. This situation limits the efficiency of combustion processes [27] and hinders efforts to improve efficiency [26]. Thermocouple gas thermometry is subject to numerous errors [28]. To advance the state of the art, a standard flame is being commissioned, that has a known temperature traceable to ITS-90, and that can be transported to a user's site to validate their thermometry apparatus.

The NPL portable standard flame system consisting of a *Hencken* flat flame burner, low uncertainty mass flow controllers (for air and propane flow) and precision motorised XYZ translation stage has been commissioned. Additionally, NPL has commissioned a Rayleigh scattering thermometry system. Figure 11 shows the standard flame under interrogation. The NPL standard flame is expected to be measured using a number of optical techniques at several of the partner organisations.

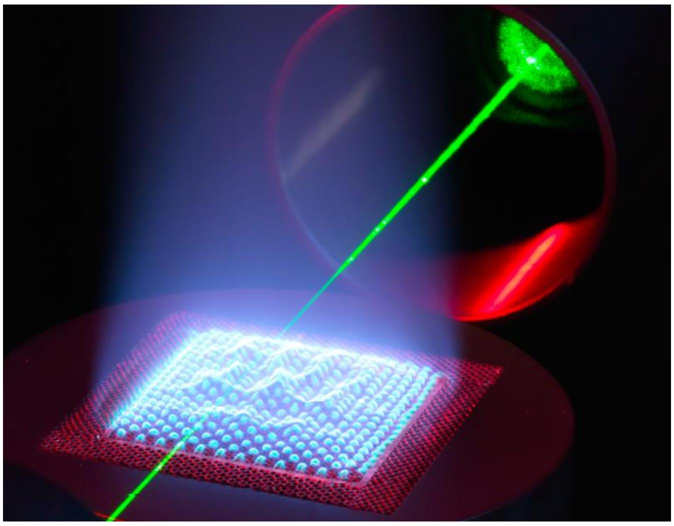


Figure 11. The NPL Standard flame. Photograph courtesy of NPL.

DTU are developing a UV spectrometer system to measure a) emission from the flame reaction zone (OH radical) and b) absorption in the post reaction zone (CO_2 and H_2O). At UC3M/CEM a hyperspectral imager is being used to measure flame spectra in the mid-infrared with the ability to acquire 2D images as opposed to a single point; temperature and combustion species can be retrieved at each pixel in the image. Figure 12 shows the emission spectra from a Bunsen burner flame measure at two points with the hyperspectral imager.

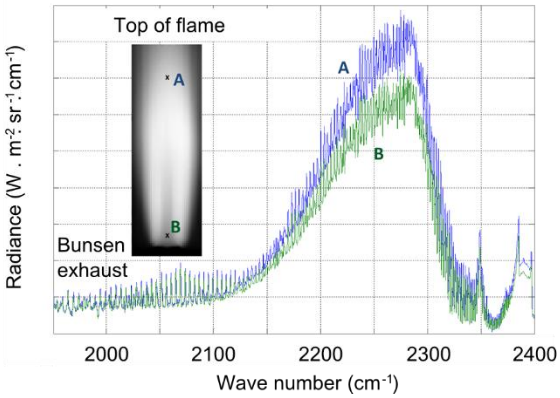


Figure 12. Emission spectra of a Bunsen burner flame at two points measured with the hyperspectral imager.

UOX are developing a number of laser based techniques to determine flame temperature. Preliminary studies have used resonant degenerate four-wave mixing (DFWM) generated in OH, in a methane air flame, established on a McKenna burner. Using a Nd:YAG laser pumped Optical Parametric Oscillator (OPO), spectra have been recorded in the region of 306 nm and the feasibility of recovering the flame temperature from relative line intensities in a Boltzmann plot, are being investigated. Laser induced thermal grating spectroscopy (LITGS) techniques are also being developed.

Summary

In summary, this three-year project, which has been running for about one year, is intended to develop a number of new temperature measurement techniques aimed at overcoming challenges in a suite of high value manufacturing applications and introducing traceability to ITS-90. The improvements are expected to be demonstrated in-process in high value manufacturing applications at users' sites.

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