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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 program ‘European Metrology Program for Innovation and Research,’ is described. The 3 year project, which started in the summer of 2015, is intended to substantially augment the efficiency of high-value manufacturing processes by improving temperature measurement techniques at the point of use. The project consortium has 18 partners and 5 external collaborators, from the metrology sector, high-value manufacturing, 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 recalibration/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.

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1 Introduction

This article describes a project funded by EURAMET's European Metrology Program for Innovation and Research (EMPIR) called EMPRESS (enhanced process control through improved temperature measurement) [1]. By building on the European Metrology Research Program (EMRP) project HiTeMS [2] and introducing a number of completely new research approaches, it aims to confer substantial improvements to process control across a wide range of high-value manufacturing applications. There are 18 partners in the consortium, as well as 5 external collaborators, from a range of sectors including the metrology community, high-value manufacturing industries, and universities (Table 1). The goal is to reduce the uncertainty of temperature measurement employed for process control in high-value manufacturing, by developing new sensors which are more robust against in-process calibration drift, and new calibration methods to overcome existing shortcomings. A key feature of the activities is the

Table 1 EMPRESS project partners

Short name	Organization legal full name	Type of organization	Country
NPL	National Physical Laboratory (Coordinator)	NMI	UK
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	UK
UC3 M	Universidad Carlos III de Madrid	University	Spain
UCAM	University of Cambridge	University	UK
UOXF	University of Oxford	University	UK
BAE	BAE Systems	Company	UK
CCPI	CCPI Europe	Company	UK

introduction of traceability to the International Temperature Scale of 1990 (ITS-90) [3] directly into the process.

The project has four work packages (WP), each representing a key objective:

- WP1: To develop new temperature control sensors with high data acquisition rate, with high stability to enhance production. A key criterion is to retain the format of existing sensors to simplify their introduction in-process. Traceable in-process uncertainty is of $<3^{\circ}\text{C}$ at temperatures below 1450°C , and $<5^{\circ}\text{C}$ at temperatures above 2000°C . These figures pertain to durations of about 3 months.
- WP2: To develop ultra-stable contact thermometers that are suitable for long-term, slowly varying heat treatment applications. These will be applicable to temperatures up to about 1350°C . Target drift rates are $<1^{\circ}\text{C}$ for a minimum of 6 months. In-process tests are envisaged in several real-world processes, such as heat treatment of turbine blades for gas turbine applications.
- WP3: To develop surface temperature measurement techniques (contact methods) that are properly traceable. This is to enhance processes such as welding, coating, and forming up to about 500°C . A new type of apparatus is envisaged to enable superior calibration of contact probes employed for surface temperature measurement, and trials are envisaged in several high-value manufacturing applications.
- WP4: To develop and characterize a 'portable standard flame,' i.e., a combustion standard of known temperature that can be used to validate the often complex flame and combustion thermometry apparatus found in industry.

All activities will be characterized by the introduction of traceability to the ITS-90, through the close involvement of national metrology institutes.

2 The Project

The European aerospace industry is a substantial entity, making a major contribution to the economy and supporting an extensive supply chain [4]. Priority research areas include improving the efficiency of gas turbine engines [5,6]; reducing airframe weight (via, in particular, very temperature-sensitive forming processes and novel metal alloys with formidable forming challenges), and improving the efficiency of manufacturing processes [6]. Nearly all of these require, in some way, better capability for improving temperature measurement and control, 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 summarized in Fig. 1.

2.1 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]. In this WP, at least one new Pt–Rh thermocouple will be identified which can offer better thermoelectric stability to the standard types (i.e., R, S, and B) [11] using high-temperature fixed points (HTFPs) [13] and develop a draft reference function for the optimized thermocouple type. Where developments

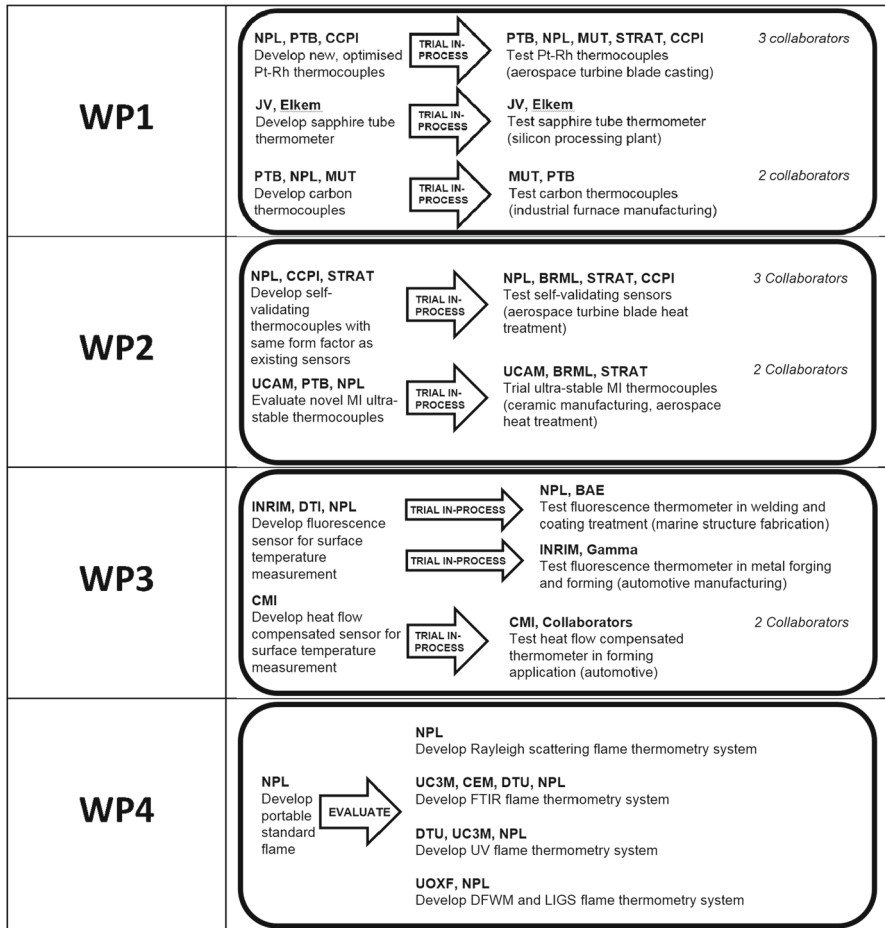


Fig. 1 Schematic of the collaboration

confer a significant benefit, they may be used to augment standards such as ASTM E1751 [12]. For environments where thermocouples are not suitable, optical-based sensors are under development which make use of pyrometric measurement methods of a miniature blackbody coupled with single-crystal sapphire light guides/sheathing.

A key development has been the establishment of two separate models of Pt–Rh thermocouple stability based on transport of Pt and Rh oxide vapor to guide the selection of Pt–Rh alloys in thermoelectric stability measurements [14, 15]. Key predictions of the two models are shown in Fig. 2. The left panel of Fig. 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 in the wire composition, i.e., the wire remains thermoelectrically stable. The right panel of Fig. 2 shows the composition that gives the optimum stability [the two axes show the two wire Rh compositions (weight percent)].

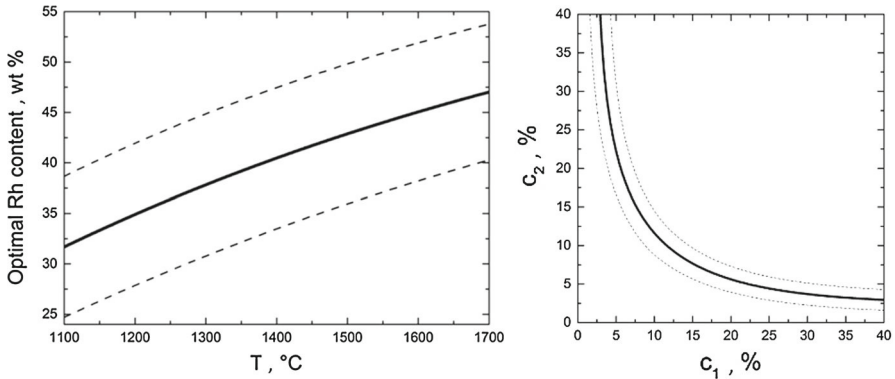


Fig. 2 Predictions of the two different models. *Left* optimal Rh content (i.e., composition) of a Pt–Rh wire as a function of temperature (vapor transport model). *Right* prediction of the optimal composition of both wires at 1324 °C (empirical model). *Dashed lines* represent the uncertainty (coverage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

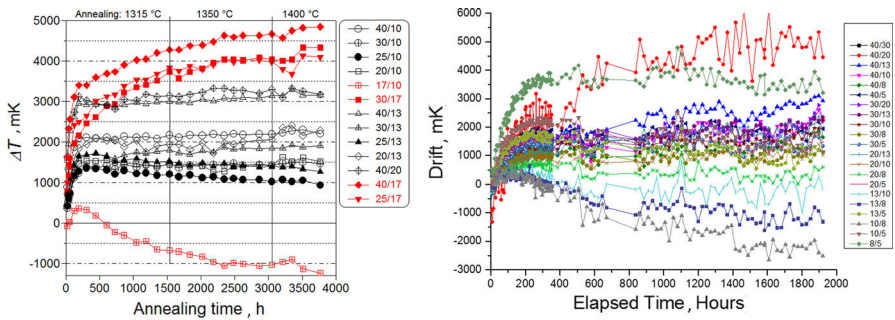


Fig. 3 Pt–Rh thermocouple drift as a function of time, at PTB at the temperatures indicated across the *top* (*left*) and at NPL as measured *in situ* with the Co–C HTFP (*right*)

Stability measurements are being performed, using HTFPs, of 7-wire thermocouples, each wire comprising a unique 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 Fig. 3. The optimum thermocouple identified in this study is expected to be trialed 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 tentative reference function has been determined up to about 400 °C (Fig. 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 inhibit graphite oxidation. 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. Develop-

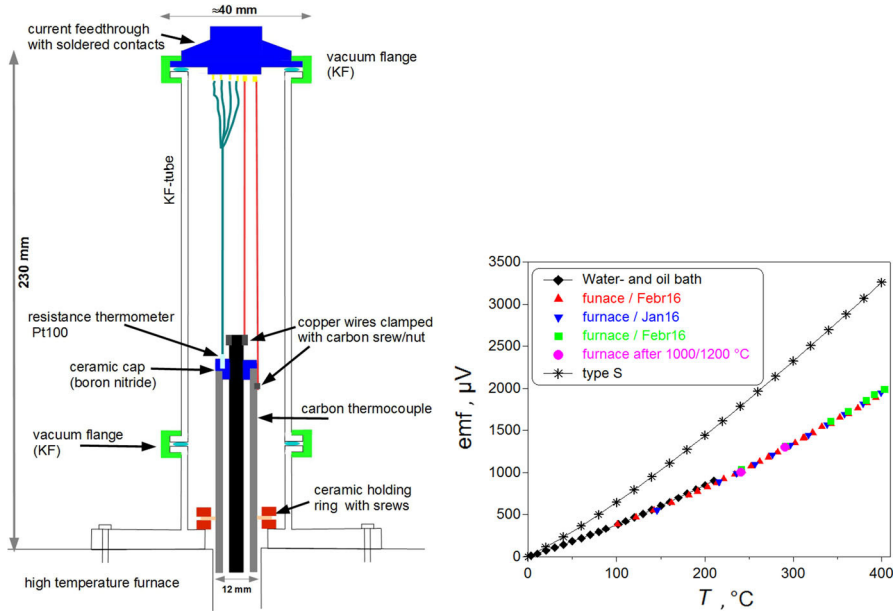


Fig. 4 Graphite thermocouple (left) and low-temperature reference function (right); the Type S thermocouple output is shown for comparison

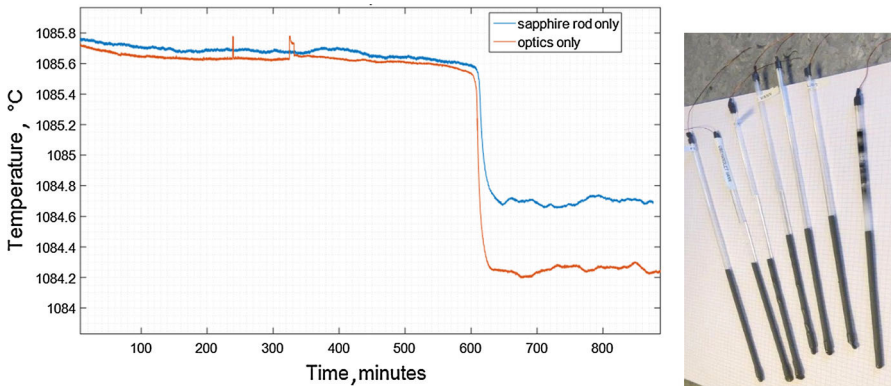


Fig. 5 The JV-Elkem blackbody-based device. Left Indicated temperature of the sapphire rod blackbody thermometer during the copper freeze, showing data with and without optical-based corrections (the initial part of the freeze has been truncated). Right Several instances of the device

ment 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 Fig. 5. Further developments are underway with the aim of implementing the device in-process at Elkem to address challenges in high-volume silicon processing.

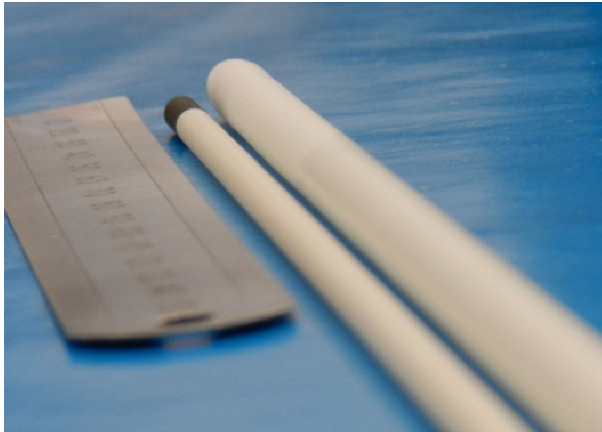


Fig. 6 Integrated HTFP (to the *left*, seen at the end of the twin-bore ceramic tube) and thermocouple pictured alongside the ceramic protective sheath (to the *right*). The sheath has an outer diameter of 7 mm; the cell has an outer diameter of 4 mm and a length of 8 mm

2.2 WP2: Zero-Drift Contact Temperature Sensors

For longer-duration processes such as heat treatment, errors in the reading caused by control or monitoring thermocouple drift can cause serious problems. By mounting an HTFP over the measurement junction of a thermocouple, an *in situ* calibration can be performed. Early prototypes of these self-validating thermocouples saw great utility in the space industry [16, 17]. In this project, the dimensions of the fixed-point cell will be substantially reduced to enable it to be placed in the relatively small thermocouple sheaths used in industry.

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 realize melting plateaus with a duration of minutes. Ingots of Cu and Co–C have been trialed. Figure 6 illustrates the integrated HTFP scheme. Figure 7 shows typical phase transition plateaus obtained using the new self-validating thermocouple, which are surprisingly well defined given the minuscule dimensions 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 characterize the effect of temperature cycling. Specifically, this means increasing the temperature to 1350 °C by using heating rates of 5 K·min⁻¹, 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⁻¹. Altogether 90 cycles are intended. After 10, 20, 40, 60, and 90 cycles, a stability test is performed at the melting point of the Co–C HTFP. Industrial trials of the new thermocouples in an aerospace heat treatment facility are anticipated in early 2017.

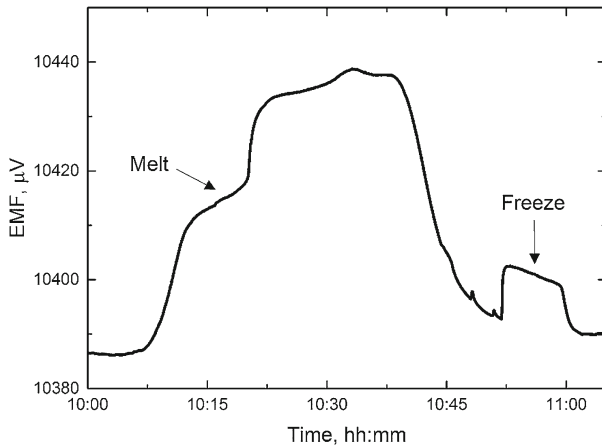


Fig. 7 Typical melting and freezing curves obtained with the integrated miniature HTFP, showing Type S thermocouple emf as a function of time during melting and freezing

2.3 WP3: Traceable Surface Temperature Measurement with Contact Sensors

Despite contact surface probes often being supplied with quoted accuracies of $<1^\circ\text{C}$ [18], real-world use can easily give rise to errors of $>10^\circ\text{C}$ [19]. Metal coating standards impose requirements of $\pm 0.5^\circ\text{C}$ [20] which is currently unachievable.

A relatively recent alternative surface temperature measurement technique is based on the fluorescence of a coating on the surface. A phosphor coating can be illuminated, and the way the radiant intensity subsequently decays provides a measure of the temperature through relatively well-understood physics. This overcomes the heat transfer difficulties that beset contact probes and the emissivity difficulties of non-contact thermometry. Two new methods will be developed for the traceable calibration of contact surface probes [21] (Fig. 8), and separately, a practical dynamically compensated heat flux probe will be developed.

The INRiM phosphor thermometer is shown in Fig. 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 predetermined calibration. $\text{Mg}_4\text{FGeO}_6 : \text{Mn}$ has been selected [22,23] which can be used for 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 (Fig. 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

Fig. 8 Prototype phosphor thermometer at INRIM

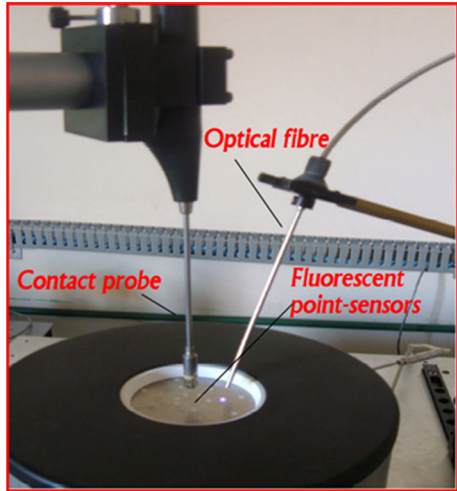
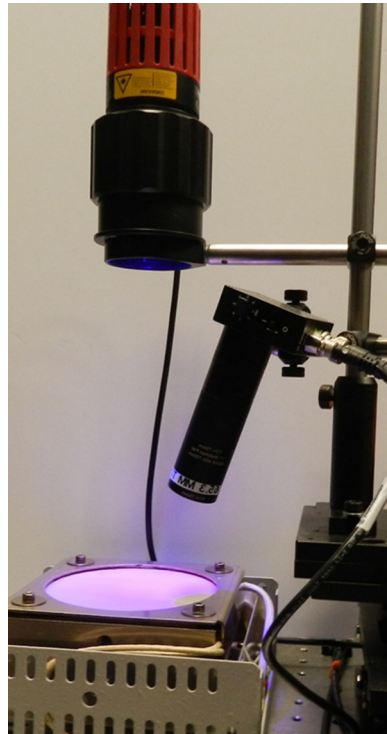


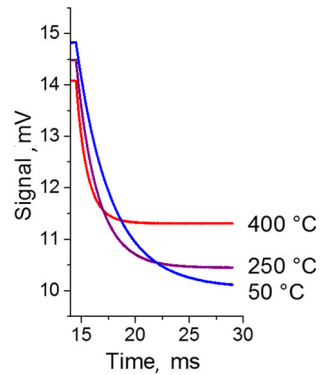
Fig. 9 NPL phosphor thermometry system



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 aluminum alloy billets.

Fig. 10 Phosphorescence decay curve for $\text{Mg}_4\text{FGeO}_6 : \text{Mn}$



CMI has developed a contact surface probe calibrator with interchangeable surface materials, in conjunction with a probe that eliminates heat flow errors by applying localized 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.

2.4 WP4: Traceable Combustion Temperature Measurement

Often in industry, combustion processes are measured using exotic laser-based methods, but uncertainties of the order of 10% of the temperature remain. The efficiency of combustion processes is therefore restricted [26,27]. Thermocouple gas thermometry is subject to numerous errors [28]. To advance the state of the art, a standard flame is being put into service; by extensive characterization at NMIs across Europe, it will be assigned a temperature that is traceable to ITS-90. The portable standard flame can then be taken to a user's site and used to validate the user's 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-motorized 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 organizations.

DTU is 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 UC3 M/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.

UOX is 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

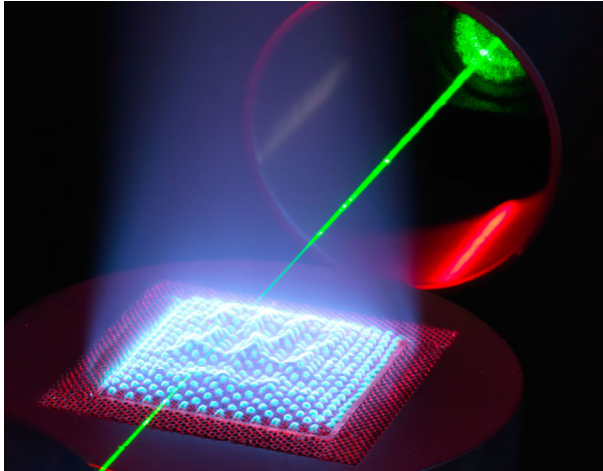


Fig. 11 The NPL standard flame. Photograph courtesy of NPL

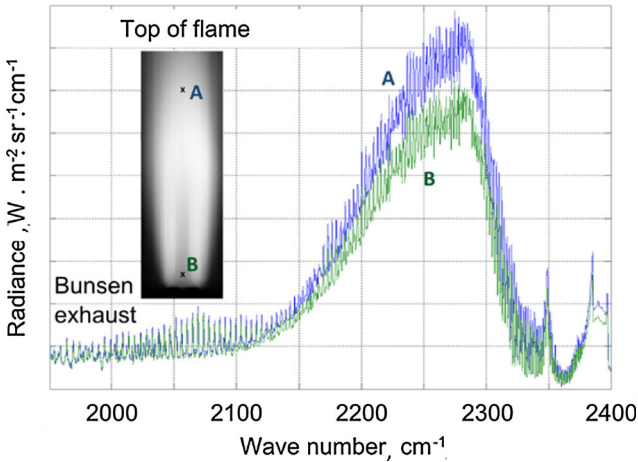


Fig. 12 Emission spectra of a Bunsen burner flame at two points measured with the hyperspectral imager

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 is being investigated. Laser-induced thermal grating spectroscopy (LITGS) techniques are also being developed.

3 Summary

In summary, this 3 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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