EMPRESS: A pan-European project to enhance process efficiency through improved temperature measurement

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Abstract

The ground-breaking results of a European project called EMPRESS, funded by the EURAMET's European Metrology Programme for Innovation and Research (EMPIR) are described. This three-year project, led by the UK's National Physical Laboratory, started in May 2015, and is intended to significantly enhance the efficiency of high value manufacturing processes by improving temperature measurement capability and implementing traceability to the SI unit of temperature, the kelvin, in-process. Key developments, demonstrated in process, include new ultra-stable thermocouples, surface temperature measurement based on phosphor thermometry, and a portable 'standard flame' for traceable combustion thermometry.

1 Introduction

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 measurements to the International Temperature Scale of 1990 (ITS-90) [1] is a critical factor in establishing low measurement uncertainty and reproducible, consistent process control [2]. Introducing such traceability *in-situ* (i.e. within the process) is a theme running through the EMPRESS project.

The project has four key scientific and technical objectives. Each objective targets a specific problem concerning process temperature measurement. Through fulfilling all four objectives, process efficiency across many industries will experience a step change improvement; each of them have applicability to gas turbine instrumentation. These objectives are:

- To develop novel low drift temperature sensors for enhanced production and temperature control. The objective is to develop novel sensors, in the same format as current sensors, up to 2000 °C. Key outputs include the identification of a Pt-Rh thermocouple composition with optimum stability, thermocouples with graphite thermoelements trialled in industrial furnace manufacturing, and sapphire tube blackbody-based thermometers trialled in silicon processing.
- To develop non-drift contact sensors optimised for heat treatment applications to temperatures around 1350 °C with a stability of better than 1 °C for at least 6 months. Key outputs include self-validating Pt-Rh thermocouples, and ultra-stable double-walled mineral insulated, metal sheathed thermocouples. Both sensors have been trialled in gas turbine heat treatment applications, ceramic manufacturing, and/or metal forging/forming processes.
- To develop traceable surface temperature measurement methods to enhance materials/chemical processing to around 500 °C. A key output is the development of a reliable non-contact emissivity free thermometer that has been trialled in marine and automotive manufacturing environments.
- To develop a portable combustion standard of known temperature for the validation of flame temperature measurement techniques. A key output is the existence of a portable standard flame for traceable calibration of users' flame and combustion diagnostic thermometry systems.

This project is nearing completion: an update on the progress of the project, and its outputs, is presented here, with an emphasis on the developments and in-process trials applicable to gas turbine instrumentation. Firstly the partners and the organisation of the project is presented. The four technical work packages are then presented, with a summary of the outputs so far. The paper concludes with a summary and some information for those interested in finding out more.

2 The project

The consortium comprises 18 partners across Europe from the metrology community, high-value manufacturing industry and academia, as shown in Table 1. In addition there are five collaborators who are involved with the project on a specific activity; these include Bodycote Heat Treatments (UK and Romania), Imperial College London, ICPE-CA Romania, and the Korea Research Institute of Standards and Science (KRISS, South Korea). A schematic of the activities and collaboration scheme is shown in Table 2.

Short name	*Full name*	*Type*	*Country*
NPL	National Physical Laboratory	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 – AFRC	University	UK
UC3M	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

Table 1: Participants. NMI: National Metrology Institute, typically an organisation responsible for maintaining the national measurement infrastructure such as standards, traceability and dissemination thereof; DI: Designated Institute, an organisation which assumes some (often specialised) capabilities of an NMI.

There are four technical work packages (WP) which reflect the four objectives given in the introduction, namely (1) low-drift contact temperature sensors, (2) zero-drift contact temperature sensors, (3) traceable surface temperature measurement with contact sensors, and (4) traceable combustion temperature measurement.

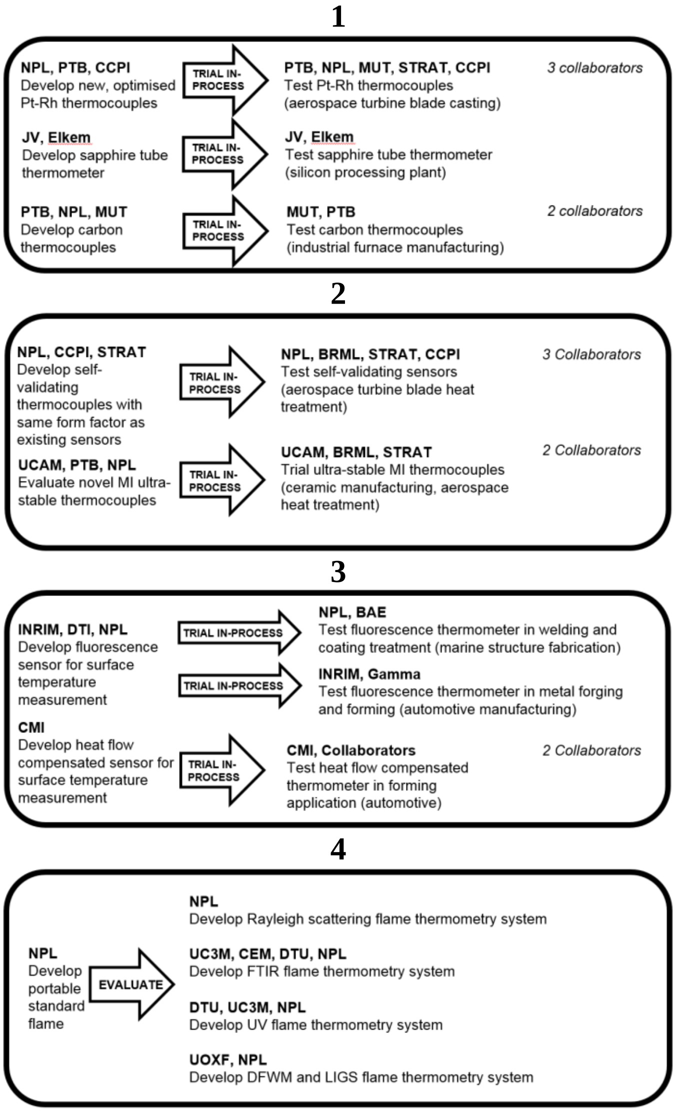

Table 2: Schematic of the activities, by work package.

2.1 WP1: Low-drift contact temperature sensors

Thermocouples are of major importance for monitoring and control of processes. Their stability is an important consideration, particularly at high temperatures >900 °C, and is a key factor in compliance with standards e.g. AMS 2750E [3]. There are two main types of thermocouple used in high value manufacturing, namely the base metal (Type K and N) mineral insulated, metal sheathed thermocouple which is a hermetically sealed, flexible cable for use to about 1200 °C; and the noble metal (Type R, S and B) thermocouple based on Pt-Rh alloys.

The wire composition of the commonly used Pt-Rh thermocouples Type R, S and B is, in all cases, the subject of historical accidents [4]. None of the thermocouples, to the authors' knowledge, have been the subject of objective optimisation. In this WP, high temperature fixed points

(HTFPs[1]) [5,6], for accurate calibration of thermocouples to 1500 °C, were employed as *in-situ* references to enable accurate monitoring of calibration drift. Seven-wire thermocouples using wires having a composition of Pt-5%Rh, Pt-8%Rh, Pt-10%Rh, Pt-13%Rh, Pt-20%Rh, Pt-30%Rh, Pt-40%Rh (at NPL) and Pt-10%Rh, Pt-13%Rh, Pt-17%Rh, Pt-20%Rh, Pt-25%Rh, Pt-30%Rh and Pt-40%Rh (at PTB) were placed in a local heat treatment furnace, while immersed in a high temperature fixed point for *in-situ* calibration (NPL only; PTB used an external fixed point cell to check the stability periodically). All possible pairs of wires (i.e. thermocouples) were measured using a switched nanovoltmeter. The thermocouple was then subjected to long-term heat treatment at about 1315 °C or 1482 °C, with periodic calibrations at 1324 °C (the Co-C eutectic point) and 1492 °C (the Pd-C eutectic point). PTB performed extra measurements of the stability at steadily increasing temperatures from about 1315 °C up to about 1450 °C. An example of the drift measurements performed in this way is shown in Figure 1.

In combination with new models of drift mechanisms [7,8], it was shown that the most stable thermocouple for use between about 1300 °C and 1500 °C is the Pt-40%Rh versus Pt-6%Rh thermocouple [7]. As this is a non-standard thermocouple, three partners (NPL, PTB, CEM) and one collaborator (KRISS) are now performing preliminary measurements of the emf-temperature relationship to enable it to be used practically.

For applications related to silicon processing, which is performed at temperatures often exceeding 1500 °C, contact thermometers based on a blackbody and sapphire light guide has been constructed by JV and Elkem, and are undergoing tests in a silicon processing application at Elkem. The devices are shown in Figure 2.

At PTB, a graphite (carbon) thermocouple has been constructed in collaboration with MUT, and a tentative reference function has been determined, at least up to about 1500 °C. This consists of a closed-end carbon tube. A carbon rod is inserted into the tube so that the only point of contact is at the bottom, with otherwise clear space all around. The device is encased in argon gas to inhibit graphite oxidation. The reference junction temperature is obtained by measuring the temperature at the top with a Pt-100 resistance thermometer. Copper lead-out wires then permit measurement of the thermocouple voltage.

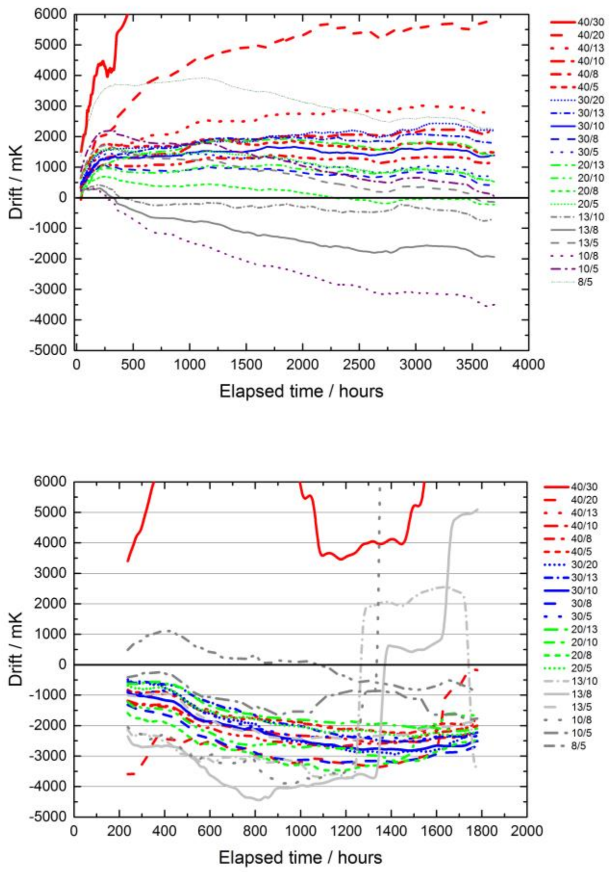

Figure 1: Stability of each Pt-Rh thermocouple combination as a function of elapsed time at ~1315 °C (top) and ~1482 °C (bottom).

Figure 2: The JV-Elkem blackbody-based thermometers. Photograph courtesy of JV.

2.2 WP2: Zero-drift contact temperature sensors

For longer-duration processes such as heat treatment, the stability of the thermocouple is at a premium. One approach to eliminating thermocouple drift is to perform in-situ calibrations. Practically, this can be achieved using a miniature high temperature fixed point and mounting it in such a way that it surrounds the measurement junction of the

[1] An HTFP consists of a graphite crucible containing a metal-carbon alloy, having a phase diagram characterised by a eutectic point, which yields an invariant melting temperature. Periodically recalibrating the thermocouple while immersed in the HTFP hence yields a measure of the calibration drift.

thermocouple. Early prototypes saw great utility in the space industry [9]. These were further developed in the European Metrology Research Programme "HiTeMS" [10]. In this project an extremely small HTFP has been designed and constructed by NPL and CCPI, being small enough to fit within a recrystallized alumina sheath of inner diameter 5 mm (this represents a de-facto standard size in a wide range of heat treatment applications involving Pt-Rh thermocouples) [11]. Despite the tiny size of the fixed-point cell, melting and freezing plateaus having durations of several minutes are observed; this can be seen in Figure 3. Several of these 'self-validating' thermocouples are now undergoing real-world trials in several high value manufacturing environments in aerospace and ceramic manufacturing plants facilitated by STRAT and BRML.

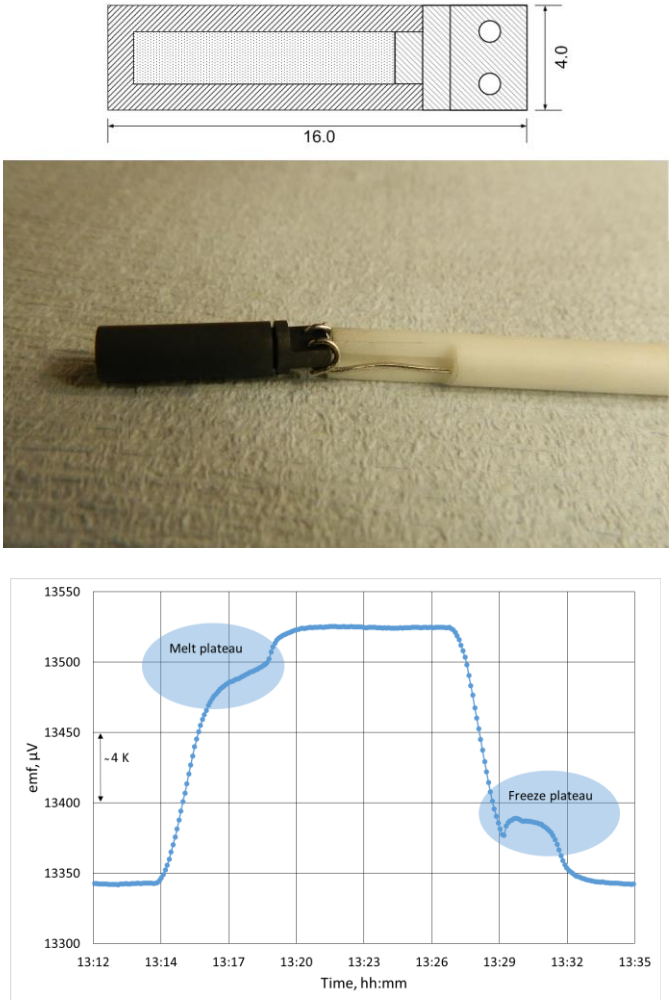

Figure 3: Self-validating thermocouple featuring a miniature fixed-point cell (top two figures; dimensions in mm). Melting/freezing curve as measured with a miniature Co-C ingot (bottom). Photographs courtesy of NPL.

At UCAM, a new ultra-stable mineral insulated, metal sheathed (MI) thermocouple cable has been developed. Although specifically designed for use in gas turbines, the applications are considerably more wide-ranging. The innovation employs the use of a double-walled sheath; by lining the outer steel sheath with an inner sheath comprising a material that mitigates contamination of the thermoelements by the sheath material, the calibration drift is dramatically reduced [12]. Rigorous assessment at UCAM – with supporting validation at PTB – to characterise the performance under temperature cycling was completed. Following this, thermal cycling and high pressure argon gas quenching at STRAT in an industrial vacuum heat treatment furnace, and industrial trials, are now underway in representative aerospace heat treatment furnaces.

2.3 WP3: Traceable surface temperature measurement with contact sensors

The options for measuring reliable surface temperatures are currently limited. Non-contact radiance based methods are free from spurious heat flow and other perturbing influences, but suffer from the problem of the unknown emissivity of the surface and contamination from background thermal radiation, which can lead to very large uncertainties (easily in excess of 10 °C and even 100 °C, at relatively modest temperatures). Contact probes, on the other hand, influence the surface with which they are in contact, and suffer from a range of errors associated with heat transfer within the probe and between probe and surface [13]. Real-world use can give rise to errors >10 °C. Given the possible magnitude of these errors, metal coating and heat treatment standards (e.g. pre- and post-welding) have requirements which are extremely difficult, if not impossible, to meet with current temperature sensing capabilities.

A non-contact non-radiance based surface temperature measurement technique, known as phosphor thermometry, is a promising solution which is coming of age. While the method has been known for a relatively long time [14], it is only now, as part of this project, where rigorous investigation of the uncertainty sources (and so work to make the method traceable to national standards) is being undertaken, is the technique becoming trustworthy and reliable. The technique relies on measuring the fluorescent emission of a coating of a phosphor applied to the surface. By briefly exciting a thin layer of phosphor on the surface using, for example, a UV/violet LED or laser diode, then measuring the fluorescence lifetime by observing the subsequent decay in emitted light, the temperature can be determined[2].

In this project NPL and INRIM have developed practical phosphor thermometry apparatus using the phosphor Mg_4FGeO_6 [15-17] along two lines: firstly, by interaction with a thin layer of phosphor coated on the surface to be measured, and secondly by adaptation to a calibrator plate which can be used to calibrate contact surface temperature probes e.g. thermocouples with a well characterised calibrator plate surface temperature. It is anticipated that the former will provide a real world demonstration of reliable phosphor thermometry in industry whilst the latter will form part of a

[2] There are other approaches to using phosphors for thermometry as well, but the one described here is the most common approach.

calibration service available to end-users to enhance the accuracy and traceability of conventional contact thermometer calibrations. The repeatability and temperature uniformity of both approaches amount to around 1 °C. A typical setup is shown in Figure 4.

The NPL scheme is currently being applied to welding challenges in marine manufacturing, including development of techniques for applying the phosphor coating to a range of different metal surfaces supplied by BAE. The INRIM device is being applied to hot forging to aluminium alloy billets for automotive applications in collaboration with Gamma.

In addition to this, CMI have performed an extensive characterisation of existing surface temperature probe calibrators, as well as the subjective aspects of conventional probes' use e.g. how hard the sensor is pressed to the surface and the angle of contact. CMI have also further developed a heat-flux compensating probe which is undergoing characterisation.

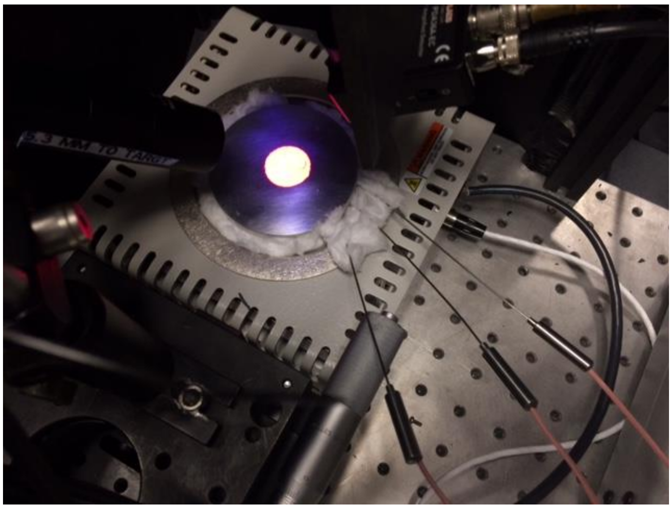

Figure 4: Phosphor coating (bright circular spot) undergoing excitation by a laser. Photograph courtesy of NPL.

2.4 WP4: Traceable combustion temperature measurement

Combustion processes are not generally amenable to conventional thermometry techniques such as thermocouples or radiation thermometers [18]. They normally require more sophisticated apparatus based on laser-based methods, but these are often subject to uncertainties of 10% or more. This restricts optimisation of combustion processes, since temperature is probably the most important parameter governing their efficiency, e.g. in internal combustion engines and gas turbines. NPL has developed a 'standard flame' which comprises a burner with very well characterised gas composition and a high degree of spatial uniformity. Being portable, the flame can be taken to a user's site and used to validate thermometry apparatus. The aim of this WP is to use the standard flame as a transfer standard to validate a wide range of different thermometry systems.

The NPL portable standard flame system [19] comprises a Hencken flat-flame burner, high accuracy mass flow controllers for the flow of air and propane, and a motorized XYZ translation stage. NPL has also commissioned a Laser Rayleigh Scattering thermometry system to characterise it. Figure 5 shows the burner flame.

DTU has developed a UV spectrometer to measure both the emission from the flame reaction zone (OH radical) and absorption in the post-reaction zone (CO_2 and H_2O). At UC3M and CEM, a hyperspectral imager has been developed to measure 2D images in the mid-infrared; each pixel in the image contains information on the temperature and combustion species. UOX has developed an IR Laser Induced Grating Scattering (LIGS) thermometer interrogating H_2O transitions at 3 μm in a methane air flame established on a McKenna burner to measure the temperature. Measurements of the portable standard flame are underway at the time of writing.

So far the cross-comparisons and characterisation of the portable standard flame have yielded excellent agreement across institutions (agreement within the combined standard uncertainties, ~ 1% of temperature). It is envisaged that at the end of the project the device will become available for deployment to end-users' sites to provide traceable calibrations on a routine basis.

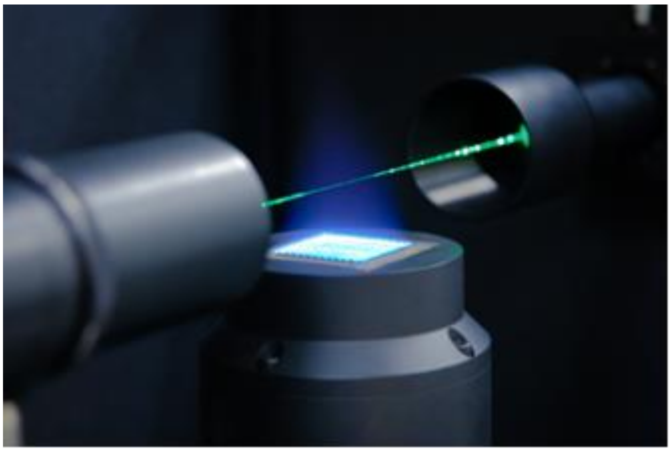

Figure 5: The NPL standard flame in the process of being interrogated using Laser Rayleigh Scattering. Photograph courtesy of NPL.

3 Summary

This three-year project is intended to develop a number of new temperature measurement techniques aimed at solving process control and monitoring challenges across a range of high-value manufacturing applications. Each activity is characterised by the introduction of traceability to ITS-90 in-process. To demonstrate the improvements in real-world environments, devices are being trialled at end-users sites.

For those interested in finding out more about the project, there is a website (www.strath.ac.uk/afrc/projects) and an introductory paper [20]. There is also a Stakeholder Community associated with the project, comprising a continuously growing group (currently 114) of interested parties across government, academia and industry. Membership is free of obligations and will entail regular updates on progress in the project, and an invitation to two workshops where project outputs will be presented and discussed, as well as plentiful networking opportunities. Interested parties are encouraged to join the stakeholder community which can simply be done by supplying their email address to the author.

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References

[1] H. Preston-Thomas, "The International Temperature Scale of 1990 (ITS-90)", Metrologia **27**, 3-10 (1990); erratum 107

[2] M. Sene, I. Gilmore, J.-T. Janssen, "Metrology is key to reproducing results", Nature **547**, 397, 27 July 2017

[3] Aerospace Material Specification (AMS) 2750 REV. E—Pyrometry

[4] Thermocouple Temperature Measurement, P.A. Kinzie, John Wiley & Sons, 1973. ISBN 0-471-48080-0

[5] G. Machin, "Twelve years of high temperature fixed point research: A review", 9th International Temperature Symposium, AIP Conf. Proc. **1552**, 305 (2013)

[6] J.V. Pearce, "Extra points for thermometry", Nature Physics **13**, 104, January 2017

[7] J.V. Pearce, A. Smith, C.J. Elliott, A. Greenen, Relating composition and thermoelectric stability of Pt-Rh alloy thermocouples, Int. J. Thermophys. **38**, 26 (2017)

[8] J.V. Pearce, "Optimising Pt-Rh thermocouple wire composition to minimise composition change due to evaporation of oxides", Johnson Matthey Technology Review **60**(4) 238-242 (2016)

[9] J.V. Pearce, C.J. Elliott, G. Machin, O. Ongrai, "Self-validating Type C thermocouples using high temperature fixed points", 9th International Temperature Symposium, AIP Conf. Proc. **1552**, 595 (2013)

[10] G. Machin, K. Anhalt, M. Battuello, F. Bourson, P. Dekker, A. Diril, F. Edler, C. Elliott, F. Girard, A. Greenen, L. Kňazovická, D. Lowe, P. Pavlásek, J. Pearce, M. Sadli, R. Strnad, M. Seifert, E.M. Vuelban, "The research outcomes of the European Metrology Research Programme Project: HiTeMS – High Temperature Measurement Solutions for Industry", Measurement **78** 168-179 (2016)

[11] C.J. Elliott, A.D. Greenen, D. Tucker, T. Ford, J.V. Pearce, "A Slimline Integrated Self-Validating Thermocouple: Initial Results", Int. J. Thermophys. **38**, 141 (2017)

[12] M. Scervini and C. Rae, "An Improved Nickel Based MIMS Thermocouple for High Temperature Gas Turbine Applications", Journal of Engineering for Gas Turbines and Power, September 2013, Vol. 135, p. 091601.

[13] R. Morice, E. András, E. Devin, T. Kovacs, "Contribution for the calibration and the use of surface temperature sensors", in Proceedings of TEMPMEKO 2001, 8th International Symposium on Temperature and Thermal Measurements in Industry and Science, ed. By B. Fellmuth, J. Seidel, G. Scholz (VDE Verlag, Berlin, 2002), pp. 1111-1116

[14] S.W. Allison and G.T. Gillies, "Remote thermometry with thermographic phosphors: Instrumentation and applications, Rev. Sci. Instrum. **68**(7), 2615-2650 (1997)

[15] L. Rosso, V.C. Fernicola, A. Tiziani, "A Calibration System for Fluorescence Thermometry" in Temperature: Its Measurement and Control in Science and Industry, Vol. **7**, Part 2, pp. 1045-1050, ed. by G. Strouse, W. Tew (American Institute of Physics, New York, 2003)

[16] L. Rosso, V. Fernicola, "Time and Frequency Domain Analyses of Fluorescence Lifetime for Temperature Sensing, Rev. Sci. Instrum. **77**, 034901 (2006)

[17] A.H. Khalid, K. Kontis, "Thermographic Phosphors for High Temperature Measurements: Principles, Current State of the Art and Recent Applications", Sensors (Basel) **8**, 5673-5744 (2008)

[18] J.C. Jones, in Thermal Measurements: The Foundation of Fire Standards, L.A. Gritzo and N. Alvares, ASTM STP1427 (2003)

[19] G. Sutton, A. Levick, G. Edwards, D. Greenhalgh, "A combustion temperature and species standard for the calibration of laser diagnostic techniques", Combustion and Flame **147**, 39-48 (2006)

[20] J.V. Pearce, "EMPRESS: A pan-European project to enhance manufacturing process efficiency through improved temperature control", Measurement and Control, Vol. **49** No. 8 pp. 252-255, October 2016