

## Publishable Summary for 17IND04 EMPRESS 2 Enhancing process efficiency through improved temperature measurement 2

### Overview

The overall aim of the project was to enhance the efficiency of high value manufacturing processes by improving temperature measurement and control capabilities. Enhanced efficiency includes improved energy efficiency (and hence reduced emissions); improved product consistency (and hence reduced waste); increased sensor stability, reliability and longevity (and hence reduced operator intervention). All of the activities in the project were characterised by the implementation of traceability to the International Temperature Scale of 1990 (ITS-90) in-process. Such traceability is critical to establishing low measurement uncertainty, e.g.  $< 3$  °C, and reproducible process control. The main developments were the extension of traceable phosphor thermometry to 750 °C and 2D imaging, and combination with thermal IR imaging to overcome the problem of emissivity and background reflections; impartial determination of the stability of dual-walled mineral insulated, metal-sheathed thermocouples compared with conventional ones, and characterisation of a reference function for new Pt-40%Rh/Pt-6%Rh thermocouples; use of a portable standard flame to enable creation of inexpensive flame imaging apparatus, and in-process reduction of uncertainty in temperature control of waste incineration; development of several types of fibre-optic thermometer for various applications in harsh environments which are not amenable to other more conventional temperature sensors. These outputs have been extensively demonstrated in industrial processes throughout Europe.

### Need

High value manufacturing depends on accurate, traceable temperature measurement. This project sought to address four contemporary thermometry challenges in high value manufacturing (each of which relates to one of the project's technical objectives).

1. Below 750 °C many industrial processes require reliable surface thermometry e.g. welding, coating, forging and forming. Conventional non-contact surface thermometry techniques by thermal imaging/single spot radiometry were prone to large errors (generally tens of degrees) due to reflected thermal radiation and unknown emissivity. Surface contact thermometry approaches were also prone to equally large errors. New traceable approaches were needed.
2. Above 1200 °C sensor drift was a significant unaddressed issue for e.g. casting, forging and heat treatment, causing errors of tens and sometimes hundreds of degrees. There was therefore a need for more stable sensors and for the standardisation of at least one new thermocouple type to fill the gap between 1200 °C and 1800 °C.
3. The measurement of combustion temperature was extremely challenging for many reasons. For example, a contact probe (e.g. a thermocouple) will not reach thermal equilibrium with the flame because it radiates heat away faster than the flame can supply it, resulting in an under-reading of the temperature by hundreds of degrees. Non-contact thermometry was fraught with difficulties such as the three-dimensional nature of the flame and its strong spatial temperature variation. For this reason many users have resorted to complex and exotic laser-based systems to probe the combustion species directly with a wide range of different techniques. Traceability was almost non-existent; for example, thermocouple measurements of flame temperatures can be in error by hundreds of degrees. A verified 'standard flame' (such as that developed during EMPIR JRP 14IND04 EMPRESS, hereafter known as EMPRESS) that can be transported to users' sites was needed, as well as case studies to demonstrate its utility.
4. Many processes were not amenable to any conventional thermometry techniques due to e.g. inaccessibility, ionising radiation, electromagnetic interference, and contamination; here methods based on optical fibres were ideal, but there were no traceable calibration techniques, and in some cases (ionising radiation) the darkening of the fibre needs to be overcome by the development of practical 'hollow core' fibres.

By introducing traceability to ITS-90 directly into the process and through developing novel calibration techniques, as well as sensors with immunity to perturbing influences, the uncertainty of thermometry in various areas of industry has been substantially reduced from the current state of the art measurements, with a corresponding enhancement in the efficiency of the process. This project addressed the four contemporary thermometry challenges where the need for improvement was greatest: namely surface contact temperature probes, thermocouples, combustion thermometry, and optical fibres.

### Objectives

The overall goal of the project was to enhance the efficiency of high value manufacturing processes by improving temperature measurement and control capabilities.

The specific objectives of the project were to:

1. **Implement traceable surface temperature measurement in-process** – by developing accurate methods for phosphor thermometry for temperatures up to 750 °C. Such methods will also be combined with quantitative thermography in order to determine emissivity for temperature measurements over wide fields of view. The in-process target uncertainty for these techniques is less than 3 °C at 750 °C.
2. **Reduce uncertainty of temperature measurement in-process** – through developing and implementing improved and traceable low-drift temperature sensors for enhanced process efficiency and improved temperature control. This will address the traceability of optimised Pt-Rh thermocouples, and a reduction in the uncertainty of new in-process temperature sensors such as the double-walled mineral-insulated, metal sheathed (MI) thermocouples through mitigating insulation resistance breakdown and drift effects. The uncertainty will be less than 3 °C up to 1200 °C for the mineral-insulated, metal sheathed thermocouples. -
3. **Implement *in-situ* traceable combustion thermometry** – by validating an *in-situ* combustion reference standard (standard flame) of known temperature with an in-process target uncertainty of less than 0.5 %. In addition, to use the combustion reference standard to evaluate the linkage between portable standard reference flames, improved process temperature control, and an enhancement in process efficiency.
4. **Introduce traceable fibre-optic thermometry** – by developing reliable, accurate and validated methods for demonstrating the traceability of at least two different types of fibre-optic thermometry in hostile environments. In addition, to develop novel methods for fibre-optic thermometry, validated in at least one harsh environment with a target uncertainty of better than 5 °C up to 500 °C.
5. **To facilitate the take up of the technology and measurement infrastructure** developed in the project by the measurement supply chain, standard developing organisations, and end-users (e.g. petrochemical industry, nuclear power industry, cement manufacture, iron and steel manufacture, gas turbines and automotive industries, consumer electronics industry, metals industries, food and beverages industry, and healthcare).

### Progress beyond the state of the art

Good collaborative progress was made in EMPRESS, particularly in the development of working prototypes of several novel thermometers including the phosphor thermometer, several new types of ultra-stable thermocouples, a portable standard flame and a suite of flame and combustion temperature diagnostic techniques, and the demonstration thereof in real-world high value manufacturing settings.

In this project, the state of the art has been progressed further on several fronts: through further development of phosphor thermometry, of low-drift thermocouples, demonstration of a validated *in-situ* combustion reference standard for development of new flame imaging systems, and traceable fibre-optic thermometry.

On phosphor thermometry, the technique has now been fully extended to two dimensions to enable surface temperature mapping up to 750 °C, and a working prototype that does this has been produced. This has been combined with quantitative thermography to enable real-time determination of emissivity. Suitable phosphors to access this temperature range have been identified, traceably calibrated, and intercompared amongst participants. One of the partners won NPL's prestigious Rayleigh Award for excellence in measurement science, associated with a publication on the EMPRESS 2 phosphor thermometry outputs.

On the development of low-drift thermocouples, the project involved a large number of NMIs systematically determining the reference function of the Pt-40%Rh vs. Pt-6%Rh. A definitive reference function was determined and has been published in the peer-reviewed literature. It has also been submitted to the IEC committee responsible for standardising letter-designated thermocouples. The thermoelectric drift performance of the new dual-wall mineral insulated, metal sheathed (MI) thermocouple which featured in EMPRESS have been compared with regular single-walled MI thermocouples; such an independent impartial validation can only be achieved by a consortium of NMIs. It was demonstrated that Type N dual-walled thermocouples do indeed exhibit superior performance to that of single-walled thermocouples. Additionally a key performance limitation of the new MI thermocouples, namely insulation resistance breakdown, has been characterised, and a method for early identification of potential problems has been published.

On combustion thermometry, EMPRESS has enabled practical calibration of flame and combustion thermometry systems, at users' sites, fully traceable to ITS-90 by means of a fully characterised portable standard flame. This standard flame has been validated and is ready for use. This project saw the development of two practical devices for flame temperature measurement based on developments in EMPRESS, namely a practical low-cost Fourier Transform Infrared Hyperspectral Imaging System, and a practical sweeping IR flame thermometry system. Key experimental band spectra from the NPL standard flame were exploited to make this possible. In particular, the performance of the sweeping IR system was demonstrated at a waste incineration facility to show how the improved thermometry can be used to optimise the process and minimise harmful atmospheric emissions.

Finally, on traceable fibre-optic thermometry, the project developed a suite of new fibre-optic techniques with the aim of making traceable temperature measurements in harsh environments. This advanced the state of the art beyond current equipment, as most conventional fibre-optic thermometers were not suited to harsh environments such as high temperature (most do not work above 300 °C), or ionising radiation (gamma radiation causes excessive darkening of the fibre), and very few were supplied with traceable calibrations. A phosphor-tipped fibre-optic thermometer has already been developed and a new type of hollow-core fibre-optic has been tested for 'darkening' in an ionising radiation environment, showing a change in transmission in the key wavelength bands of less than 2 % following exposure to 4400 Gy. A sapphire Fibre-Bragg Grating thermometer was demonstrated in a very harsh silicon processing environment and proved to offer improved temperature stability in comparison with conventional techniques.

## Results

### *Implement traceable surface temperature measurement in-process*

STRATH developed a dual phosphor/pyrometer system to overcome the notorious problem of determining the emissivity associated with conventional pyrometer/thermal imaging. NPL also now has a reliable imaging (2D) phosphor thermometry system working, and traceably calibrated up to 750 °C, with uncertainty better than 3 °C over most of the temperature range. This has been trialled in several situations at NPL including photovoltaic panel development and tensile testing applications. INRIM has a fully developed phosphor thermometry system and focused on performing tests of the phosphor coating on real-world brake pads (at ITT) having different chemical compositions. Technical development proceeded in tandem with practical implementation, specifically in forging, brake disk manufacturing, and marine manufacturing (welding) applications. Discussions are currently ongoing with instrumentation manufacturers with a view to licensing the product. The objective has been successfully achieved.

### *Reduce uncertainty of temperature measurement in-process*

The Pt-40%Rh and Pt-6% Rh wire for the reference function determination was completed, drawing on the traceable measurements of ten NMI participants. The dual-walled MI cable was distributed amongst the partners in order to establish independent validation of the thermoelectric drift performance, and a common procedure was established. An extensive, impartial series of measurements was performed which demonstrated the superiority of Type N dual-walled thermocouples when compared with conventional single-walled thermocouples, and the results were submitted to the IEC committee concerned with standardisation of MI thermocouples. A method for early identification of potential insulation resistance breakdown of MI thermocouples has been published. The objective has been successfully achieved.

### *Implement in-situ traceable combustion thermometry*

The NPL portable standard flame was validated and the calibration re-checked. The low-cost multispectral imaging system under development at UC3M was tested with the portable standard flame, and better than 5 %

agreement was observed between the low-cost system and the 'reference' Fourier Transform Infrared system; this was traceably calibrated and compared against multispectral measurements to properly characterise the uncertainty contributions. This was an important milestone in the development towards an end-user device. DTU and B&W Vølund have performed very extensive measurements on-site at the Filborna waste-to-energy incineration plant in Helsingborg, Sweden, to demonstrate the utility of the Fourier Transform infrared thermometry in-process. The objective has been successfully achieved.

#### *Introduce traceable fibre-optic thermometry*

A working device to 650 °C has been developed with high temperature gold-coated fibres and achieved an uncertainty below the target of 5 °C. A hollow core fibre has been manufactured with geometry optimised for phosphor thermometry and completed testing in NPL's ionising radiation facility (gamma rays); the transmission losses amounted to less than 2 % for accumulated dose of 4400 Gy. A high temperature thermometer based on Fibre-Bragg Gratings (FBGs) was designed, the inscription of the gratings in the fibre completed, and the device constructed and exhibited an uncertainty of about 2 °C; the device was tested in an extremely harsh environment in a silicon processing environment where it exhibited very high stability. At CEM the fibre-optic DTS based on Brillouin Scattering was designed and constructed, and tests performed with aluminium and copper coatings performed, together with development of a traceable calibration technique based on immersion in a stirred liquid bath. The objective has been successfully achieved.

#### **Impact**

The project was industry-focused with the outputs being trialled in-process during the project's lifetime. Of particular note is the input to the discussions of the IEC committee responsible for thermocouple standard IEC 60751, input to the ASTM committee responsible for thermocouple standard E20, presentation to the NADCAP Heat Treating Task Group and SAE AMS AMEC aerospace committee meeting, input to three CCT guides, presentations at 25 international conferences on combustion thermometry, phosphor thermometry, spacecraft testing, thermodynamic modelling, thermal modelling, thermal imaging, nuclear decommissioning and measurement and control respectively. A Euramet summer school was held by partners, as well as a training course for university postgraduates; in total 16 training events were held. The EMPRESS 2 stakeholder community currently numbers 148, and a stakeholder community workshop was held by the consortium which attracted 121 delegates from across the globe. Two PhD students performed work as part of the project. 14 Open Access publications are now available and represent Technical reports, Articles in peer-reviewed journals and a Good Practice Guide. 50 other dissemination activities included trade journal articles, and workshops.

#### *Impact on industrial and other user communities*

The direct impact on partners will be very significant. The introduction of in-situ traceability to ITS-90 is embedded in all of the tasks of the project and this will ensure better consistency across the range of processes impacted. The longer term impact on industry will be wider. Improved temperature measurement will result in tighter process control leading to better, more consistent products which in turn enhances competitiveness. Examples of early impact include better characterisation of brake pad temperatures for automotive development applications, improved marine welding with reduced wastage and stronger end products, improved heat treatment of aerospace parts, lower uncertainty calibrations and thermocouples available from NMIs, new products for flame imaging instrumentation manufacturers, improved waste incineration facilities, new products for fibre optic thermometry manufacturers including traceability, improved characterisation of large scale industrial furnaces, more efficient large scale silicon processes, and more efficient large scale steel manufacturing processes. Early impact includes trials of **phosphor thermometry** in electro-thermal mechanical testing, photovoltaic device development, nuclear decommissioning and waste storage, marine manufacturing (welding pre- and post-heat treatment and annealing), temperature monitoring on forging tools, on-line and off-line temperature monitoring and control in forming, forging and heat treatment, and automotive braking system development. **Thermocouple thermometry** was demonstrated in quartz glass manufacturing, steel production for railways, float glass manufacturing, and industrial furnace and refractory insulation manufacturing. **Combustion thermometry** was demonstrated in the development of a low-cost thermal imaging system and in a commercial waste incineration process. **Fibre-optic thermometry** was demonstrated in the control of electron beams for dose planning in radiotherapy, high magnetic fields, and silicon processing for photovoltaic solar panel manufacture.

### *Impact on the metrology and scientific communities*

The consortium has representation on the Task Group on Guides on Thermometry of the BIPM Consultative Committee for Thermometry (CCT) which is responsible for promoting good thermometry practice and traceability to the SI by preparing and publishing guides on thermometry, with an emphasis on secondary thermometry, i.e. the subject of this project. The consortium will ensure that CCT members are made aware of the project's outputs. The consortium also has representation on the EURAMET Technical Committee on Thermometry (TC-T), including the Chair, and has been ensuring that the project's outputs are widely disseminated via EURAMET TC-T activities including the Euramet Summer School on Metrology, and presentations at the annual meeting and an associated workshop on the mise-en-pratique for the redefinition of the kelvin; a Euramet guide on thermocouple calibrations which span out of this work was also published. A member of the consortium chairs the Spanish National Accreditation Body, Entidad Nacional de Acreditación (ENAC) subcommittee for Temperature and Humidity (ENAC SCTC11). Standards bodies in Germany, Italy and Turkey are also linked to the project. In addition, a partner was president of the Institute of Measurement and Control (InstMC) during the project's lifetime, greatly facilitating the dissemination of the project's outputs.

There are a number of smaller NMIs in the consortium and their participation in this project contributed substantially to capacity building, particularly in the area of thermocouple calibration and the facilities and skills required for contributing to reference function determination.

### *Impact on relevant standards*

This project will have a significant impact by enabling industries to ensure a greater level of compliance with relevant standards. This was achieved through influencing committees including IEC TC 65/SC 65B/WG5 (thermocouple standards); ASTM (thermocouple standards); Nadcap (National Aerospace and Defence Contractors Accreditation Program) and SAE (specific industry-wide aerospace heat treatment standards – a presentation has been given at one of their meetings); Consultative Committee for Thermometry (wide ranging areas of interest – some partners are represented on the new working group associated with photonic thermometry, i.e. fibre-optic thermometry); EURAMET Technical Committee on Thermometry (TC-T, wide ranging areas of interest, outlined in the metrology section above, who have been given regular updates by the consortium); InstMC – Standards Policy Panel (promotion of excellence in instrumentation and control); IoP ISAT committee (promotion of new developments in sensing). Specific standards that were targeted are IEC 61515 (MI thermocouple cable manufacturing – some partners were represented at the last meetings of the IEC to discuss aspects of standardisation for the new double-walled thermocouples being studied in this project), ASTM E20 (MI thermocouple cable manufacturing; some partners were represented at a meeting to input into the E20 standard revision), IEC 62460 (Non letter designated thermocouple emf versus temperature relationship) and AMS 2750E (Aerospace heat treatment; some partners were represented at a meeting in 2018 to describe the project to aerospace heat treatment prime suppliers).

### *Longer-term economic, social and environmental impacts*

In regional terms, the EU is the world's largest exporter of manufactured goods. However, high energy prices have increased the economic importance of energy as an input in the production process. European firms have made great progress in improving efficiency, but this has not been enough to compensate for the increase in energy prices. Furthermore, there is growing evidence that the evolution of expertise in energy efficient processes, technologies and services means that this expertise itself can be exported, thus providing European business with a competitive edge.

The present project will assist in progressing towards these goals by demonstrating new and more accurate temperature measurement techniques to major manufacturers, such as Rolls-Royce, enabling them to better understand their processes and energy usage.

### **Publications**

1. The NPL portable standard flame: Operating instructions, NPL Report ENG 69, ISSN 1754-2987 <http://eprintspublications.npl.co.uk/8259/>
2. An investigation into a calibration scheme for a light pipe based temperature probe, Åge Andreas Falnes Olsen, Helge Mathisen, Sigurd Simonsen, 2018 Meas. Sci. Technol. 29 115004. Highlight: selected as one of the 2018 Highlights of Measurement Science and Technology <https://iopscience.iop.org/article/10.1088/1361-6501/aade6f>

3. Calibration of thermocouples: EURAMET Calibration Guide No. 8 Version 3.0, J.V. Pearce, N. Arifovic, J. Bojkovski, F. Edler, M. de Groot, C. Garcia Izquierdo, M. Kalemci, R. Strnad <https://www.euramet.org/publications-media-centre/calibration-guidelines/>
4. Validation of emission spectroscopy gas temperature measurements using a standard flame traceable to the International Temperature Scale of 1990 (ITS-90), G. Sutton, A. Fateev, Miguel A. Rodriguez-Conejo, Juan Melendez, Guillermo Guarzino, Int. J. Thermophys. 40 99 (2019) <https://doi.org/10.1007/s10765-019-2557-6>
5. A validated physical model of the thermoelectric drift of Pt-Rh thermocouples above 1200 °C, J.V. Pearce, Metrologia 57 025009 (2020) <https://doi.org/10.1088/1681-7575/ab71b3>
6. Design, construction and calibration of a novel phosphor-based fibre-optic thermometer from 0 °C to 650 °C, D. Lowe, G. Sutton, A. Sposito, G. Machin, J. Pearce, Measurement Science and Technology 32 094004 <https://doi.org/10.1088/1361-6501/abee53>
7. Mass Loss of Platinum-Rhodium Thermocouple Wires at 1324 °C, S. Uthayakumaar, S. Davidson, J. Pearce, Johnson Matthey Technology Review, Volume 65, Number 4, 1 October 2021, pp. 568-573(6) <https://doi.org/10.1595/205651321X16183288904988>
8. Some Predictions of a Validated Physical Model of Pt-Rh Thermocouple Drift Above 1200 °C, J.V. Pearce, Metrologia 58 035011 (2021) <https://doi.org/10.1088/1681-7575/abeb80>
9. Mapping of temperature and CO<sub>2</sub> column density in a standard flame by multispectral imaging, J. Meléndez, J. Talavante, G. Guarzino, F. López, Proceedings Volume 11743, Thermosense: Thermal Infrared Applications XLIII; 117430V (2021) <https://doi.org/10.1117/12.2585805>
10. Pt-40%Rh versus Pt-6%Rh thermocouples: an emf-temperature reference function for the temperature range 0 °C to 1769 °C, F. Edler, J. Bojkovski, C.G. Izquierdo, M.J. Martin, D. Tucker, N. Arifovic, S.L. Andersen, L. Sindelarova, V. Zuzek, International Journal of Thermophysics Volume 42, Article number 150 (2021) <https://doi.org/10.1007/s10765-021-02895-w>
11. Miniature iron-carbon eutectic point crucible for the calibration of thermometers, V. Žužek and J. Bojkovski, Measurement Volume 181, August 2021, 109619 <https://doi.org/10.1016/j.measurement.2021.109619>
12. Thermoelektrische Eigenschaften von Pt-40%Rh/Pt-6%Rh Thermoelementen, Frank Edler and Petra Ederer, tm - Technisches Messen, (2021) <https://doi.org/10.1515/teme-2021-0042>
13. Correlation between insulation resistance and temperature measurement error in Type K and Type N mineral insulated, metal sheathed thermocouples, Pearce, J., Tucker, D., Izquierdo, C.G. et al., Int J Thermophys, (2022) <https://doi.org/10.1007/s10765-021-02967-x>
14. Multispectral Mid-Infrared Camera System for Accurate Stand-Off Temperature and Column Density Measurements on Flames, Meléndez, J.; Guarzino, G., Sensors, (2021), <https://doi.org/10.3390/s21248395>

This list is also available here: <https://www.euramet.org/repository/research-publications-repository-link/>

|   |                           |                         |
|---|---------------------------|-------------------------|
| Project start date and duration:  |                           | 01 May 2018, 42 months  |
| Coordinator: Dr Jonathan Pearce, NPL    Tel: +44 (0)20 8943 6886    E-mail: <a href="mailto:jonathan.pearce@npl.co.uk">jonathan.pearce@npl.co.uk</a>  |                           |                         |
| Project website address:<br><a href="https://www.strath.ac.uk/research/advancedformingresearchcentre/whatwedo/collaborativeprojects/empress2project/">https://www.strath.ac.uk/research/advancedformingresearchcentre/whatwedo/collaborativeprojects/empress2project/</a> |                           |                         |
| Internal Funded Partners:   | External Funded Partners: | Unfunded Partners:      |
| 1 NPL, United Kingdom   | 11 CNR, Italy             | 20 ACERINOX, Spain      |
| 2 CEM, Spain  | 12 Elkem, Norway          | 21 B&W Volund, Denmark  |
| 3 CMI, Czech Republic   | 13 IPHT, Germany          | 22 BAE, United Kingdom  |
| 4 DTI, Denmark  | 14 ITT, Italy             | 23 CCPI, United Kingdom |
| 5 DTU, Denmark  | 15 MUT, Germany           | 24 JM, United Kingdom   |
| 6 INRIM, Italy  | 16 STRATH, United Kingdom | 25 Sensia, Spain        |
| 7 JV, Norway  | 17 UC3M, Spain            |                         |
| 8 PTB, Germany  | 19 UCAM, United Kingdom   |                         |
| 9 TUBITAK, Turkey   | 19 UoS, United Kingdom    |                         |
| 10 UL, Slovenia   |                           |                         |
| RMG: -  |                           |                         |