Publishable Summary for 14IND04 EMPRESS
Enhancing process efficiency through improved temperature measurement

Overview

Most industrial processes need to be maintained at a specific temperature to maximise efficiency; and accurate control of temperature ensures process efficiency. By improving temperature measurement techniques for selected applications, this project aimed to enhance the efficiency of high value manufacturing processes in terms of reduced wastage, improved yield, more consistent processing, increased intervals between sensor checks and maintenance, increased reliability, improved energy efficiency and reduced greenhouse gas emissions. The developments were trialled in-process at end users' facilities to solve documented manufacturing problems in high value manufacturing environments and introduce *in-situ* traceability to the International Temperature Scale of 1990 (ITS-90). A commercial partnership between a commercial and academic project partner has been formed to develop a new double wall MI thermocouple, several companies are looking to license the know-how associated with the use of phosphor thermometry, and the portable standard flame is now an artefact available to end-users.

Need

Manufacturing contributes over €8 trillion of value added to the global economy, of which 22 % is due to the European Union (EU). Modern high value manufacturing is a vital part of the economy of numerous EU countries; e.g. aerospace, where key areas of research and development are improving engine efficiency by maximising the temperature rise through the combustor and reducing airframe weight by using new materials and implementing more efficient production processes, which require improved temperature measurement.

The EC Energy Efficiency Plan [COM(2011) 109] outlines the importance of enhancing efficiency to increase the competitiveness of European manufacturing industry, which accounts for about 25 % of the EU's energy consumption. The need for better process control has been brought to the fore by the EU's introduction of a legislative framework to enforce emissions levels of new vehicles [COM(2007) 19] by imposing fuel efficiency targets [COM(2010) 656] in recognition of this sector's vast contribution (20 %) to overall EU emissions.

Important gaps exist in temperature measurement capability in the aerospace and energy sectors, particularly in heat treatment and forging of components, casting of exotic single-crystal alloys and combustion temperature measurement. Challenges remain for manufacturers in mitigating drift of thermocouples, which are used almost exclusively for process control. Processing of refractory materials, such as ceramics and nuclear fuel, is still inefficient above 1800 °C due to temperature control. Semiconductor processing is not always amenable to the use of thermocouples for process control, so to address this gap, an optical solution is needed. Accurate temperature measurement is also required in combustion and flame processes, which is important for manufacturing (e.g. steel and cement) and for heat engines (e.g. internal combustion engines and gas turbines) used in transport. Currently, traceability of combustion thermometry to ITS-90 does not exist.

An area of growing importance in advanced manufacturing is surface temperature measurement where uncertainties are unacceptably large. Growing use of light composite materials and aluminium alloys introduces new challenges associated with welding, forging and forming; placing high demands on surface temperature measurement techniques to ensure adequate pre- and post-welding/forming heat treatment. Improved forming techniques are identified as a priority in the EU publication 'Factories of the future'. The demand for ever stronger steel structures in marine and submarine applications, including offshore structures for e.g. renewable energy and oil/gas platforms, is also growing, which places high demand on welding quality and strength.

The EMPIR initiative is co-funded by the European Union's Horizon 2020 research and innovation programme and the EMPIR Participating States

Objectives

The overall aim of this project was to improve process efficiency by improving the accuracy of temperature measurements in industrial manufacturing environments.

The specific objectives were to:

1. Reduce the uncertainty of temperature measurement in-process through developing more stable (lower drift) temperature sensors over several applicable temperature ranges (optical-based sensors to 1800 °C, better noble metal thermocouples to 1800 °C and carbon thermocouples to over 2000 °C). Target uncertainties are of better than 3 °C at temperatures around 1450 °C and better than 5 °C at temperatures > 2000 °C.
2. Implement *in-situ* traceability of temperature measurement in-process through developing ultra-low drift sensors, including a novel mineral insulated, metal-sheathed thermocouple type and *in-situ* self-validating thermocouples at around 1350 °C, which will remain in service, with a stability of better than 1 °C, for at least 6 months.
3. Implement traceable surface temperature measurement in-process, to around 500 °C with a target uncertainty of better than 5 °C, with contact sensors by developing novel surface temperature measurement techniques based on phosphor thermometry; use this to demonstrate improved temperature measurement in at least two industrial settings.
4. Introduce traceability to combustion temperature measurement by development, characterisation and traceably calibrating a portable standard flame, to have an uncertainty of less than 1 %, a factor of 10 lower than current methods, which will then be used to calibrate industrial users' combustion thermometry equipment.
5. Contribute to impact via trials of new developments in-process, contributions to international standards and collaboration with, and dissemination of research outputs to, end-users in the high value manufacturing sector.

Progress beyond the state of the art

Thermocouples are the most widely used temperature sensor in high value manufacturing applications, but are known to exhibit unpredictable calibration drift, so their performance limits the efficiency of many advanced manufacturing processes, objectives 1 and 2 focused on improving this aspect. They are typically made of two different metal alloy wires.

<u>Objective 1 Novel, low drift temperature sensors</u>

Pt-Rh thermocouples have the lowest uncertainties for contact temperature measurement up to 1800 °C but have never been subject to optimisation and developing new, more stable alloys of Pt-Rh for thermocouples will substantially improve drift performance.

Above approximately 1600 °C, only the W-Re thermocouple contact sensor is routinely available and this is subject to substantial drift of tens of degrees. The state of the art at temperatures up to 2000 °C is being progressed through the development of carbon thermocouples in this project.

For many processing environments where thermocouples are not suitable, the only current solution is radiation thermometry and this project has advanced the state of the art by developing optical-based sensors using single-crystal sapphire light guides. Metrological characterisation of the sapphire tube thermometer was performed.

<u>Objective 2 Ultra-low drift sensors</u>

Below approximately 1300 °C, the 'work horse' of industrial temperature measurement in industry is the Type N MI cable thermocouple, but this is plagued by stability issues above approximately 900 °C; a new mineral-insulated metal-sheathed (MI) cable as a more stable substitute for Type N thermocouples has been implemented. The improved Pt-Rh thermocouples described above for objective 1 are also a good candidate for this.

<u>Objective 3 Traceable surface temperature measurement methods</u>

Surface temperature measurement with contact thermometry is currently problematic and the current use of temperature sensitive crayons to determine metal surface temperature prior to welding is highly subjective and non-traceable; surface temperature probes are subjective and poorly characterised. Infra-red thermometry is beset by emissivity problems and not often practical. In this project, two new techniques, were developed, and trialled in-process, for traceably calibrating contact surface temperature probes. Phosphor thermometry was selected for robust metrological assessment and a industrially relevant method was developed.

Objective 4 In-situ combustion standard

Currently, the most accurate means of measuring the temperature of combustion processes is achieved with laser diagnostic methods. These have uncertainties of the order of 10 % and are not traceable to the ITS-90. The use of thermocouples is prone to numerous severe errors. To advance the state of the art, a standard flame was commissioned and characterised. A number of optical systems based on IR emission hyperspectral imaging, UV absorption spectroscopy and non-linear optical techniques for measuring the standard flame were also commissioned. The portable standard flame was circulated amongst the partners to enable assessment of its temperature.

Results

The results of this project provide new sensing techniques for better control of high value manufacturing and other processes.

Objective 1 Novel, low drift temperature sensors

Good progress was made towards this objective, although the performance of sensors at the upper end of the thermocouple range >2000ºC was not reached because the graphite thermocouples could not be stabilised above about 1600 °C.

Seven different Pt-Rh alloy two-wire thermocouples were constructed and measured to find the most stable combination, and the Pt-6%Rh vs. Pt-40%Rh thermocouple was identified as the optimum combination. Four of these thermocouples were constructed and stabilised then measurements to establish a draft reference function of the Pt-40%Rh vs. Pt-6%Rh thermocouples have been performed at NPL, PTB, CEM and KRISS, the Korean NMI, a collaborator on the project. A preliminary reference function has been submitted for publication.

A novel sapphire tube thermometer with better mechanical stability than current technology permits together with a new optical readout system was tested at 900 °C, 1000 °C and 1100 °C in two different furnaces at JV. The optical signal was compared with the temperature recorded by a calibrated thermocouple, but the results still reveal a dependence on immersion. Initial work on modelling of the light intensity has been carried out, but so far it has not been possible to conclude on the most appropriate compensation strategy. Initial results suggest the device has promise for silicon processing, but further improvements are needed before it can be a practical option for end users.

Two additional thermocouples made of glassy carbon in combination with conventional graphite materials were constructed and tested for its thermoelectric stability from 1300 °C up to 1800 °C at PTB. The results exhibit a better thermoelectric stability than the previously investigated combinations of the materials. Further measurements to determine the long-term stability at 1500 °C under industrial conditions and by using the ruggedised industrial design of MUT have been performed, which suggest that the device is most suitable for use up to 1500 °C.

Objective 2 Ultra-low drift sensors

Good progress was made towards this objective, although the double-walled mineral insulated, metal sheathed thermocouples did not demonstrate the expected stability (within 1 °C) – although they came close (within between 2 °C and 3 °C) which is already a remarkable achievement.

The main exploitable results for this objective are: (a) the realisation of the self-validating principle within a thermocouple suitable to be used in industry; and (b) redesign of the Co-C fixed point cell. The latter has been proven to operate correctly and efforts are underway to enhance its robustness. The self-validating thermocouples have been developed and constructed with a major thermocouple manufacturer (one of the project partners), who have assisted with construction of robust thermocouple assemblies. Industrial trials have shown promising results, with a clearly observable melting/freezing plateau providing a self-validation point measured *in-situ*, and stability/robustness has proven to be excellent. A licensing opportunity has been realised.

A new mineral-insulated metal-sheathed (MI) cable as a more stable substitute for Type N thermocouples has been developed and prototype devices are currently being trialled in industrial aerospace heat treatment furnaces. Staff have been trained in their use at an aerospace heat treatment facility, and some promising results have been obtained concerning long-term stability.

Objective 3 Traceable surface temperature measurement methods

Two surface temperature measurement methods were developed and one of these was in two industrial settings. The target uncertainty of better than 5 ºC at 500 ºC was achieved.

The phosphor thermometry developments have progressed rapidly; dedicated electro-optical systems have been developed for measuring the decay constant of the phosphorescent signals and thus infer temperature for surface temperature sensor calibration. Samples of steel have been provided for trials of the new phosphor thermometer on the actual material used in the construction of large-scale marine craft, demonstrating early engagement of end users; and in-situ measurements have been performed which show the practical utility of phosphor thermometry. In addition, a calibrator for conventional contact surface probes, which uses phosphor thermometry to determine the surface temperature directly, has been constructed and demonstrated.

Additionally a dynamically compensated surface probe using a reliable phosphor thermometer has been constructed and its performance has been demonstrated to be fit for purpose.

Objective 4 In-situ combustion standard

The portable standard flame has been characterised; and optical thermometry systems based on IR emission hyperspectral imaging, UV absorption spectroscopy and non-linear optical techniques have been commissioned. An uncertainty of 0.5% was achieved, surpassing the objective of a 1% uncertainty and meeting the objective.

Measurements have been made on the portable standard flame using the hyperspectral imager system of UC3M and the UV high-resolution absorption spectrometer at DTU. A comparison between the flame temperatures measured by UC3M and NPL indicate excellent agreement (typically better than 1 %) between the two independent methods. This is the first time that such measurements have been made and these will be disseminated to the spectroscopy community via peer-review publication. Additionally, a UV flame thermometry system has been developed at DTU; this instrument can provide a novel method of flame thermometry and species determination.

The portable standard flame has been calibrated and its medium term (6 months) stability assessed. A re-calibration following trials at UC3M indicates that the system is still within its calibration uncertainty. Following return of the system from UOXF, and later DTU, the long-term stability and reproducibility of the system has been assessed. Currently two papers are ready for submission regarding the portable standard flame performance and a further publication is planned describing trials.

Impact

Significant effort has been put into to interacting with relevant members of the temperature measurement industry. As a result the stakeholder community interacting with the project has grown to 126 members and several companies have enquired about the results of the project and ways in which to commercially exploit them; this has been detailed below. A workshop for the EMPRESS project stakeholder community was held at the AFRC (Advanced Forming Research Centre at STRATH) in Glasgow in March 2017 and attracted around 70 delegates. This workshop was primarily used to bring together scientists and engineers from academia, research institutes and industrial establishments to present and discuss both the latest developments in the field of traceable temperature measurement for process control and end-users' requirements and challenges. This event was very successful and created a significant promotion of the EMPRESS project, with very positive feedback received from the attendees. A second equally successful workshop was held at NPL in April 2018.

An introductory paper on the project was presented at 'Metrologie', in Paris in September 2015; a summary paper has been published in Measurement + Control. The project has been presented at high profile meetings including the Rolls-Royce Measurement Partners Seminar and the 7th EVI-GTI Gas Turbine Instrumentation Conference 2015. Project results were presented at the XIII Symposium on Temperature and Thermal Measurements in Industry and Science held in Poland (June 2016). A workshop for thermocouple users was held at NPL in November 2016 to disseminate project outputs, which has turned into a new series of one-day workshops entitled "Thermocouple Users and Manufacturers Conference"; the next one in the series is the 3rd workshop on 4 December 2018.

This dissemination and publicity for the project has enabled the stakeholder community to grow and should facilitate the uptake of the exploitable results from the project. Importantly there is an increasingly vibrant LinkedIn group "Thermocouples in Industry" which has also spun out of the project.

Impact on relevant standards

Immediate impact on standards will come as a result of the establishment of a framework for implementing a new, optimised Pt-Rh thermocouple for the IEC standard 60584-1, which will address the gap between 1500 °C and 1800 °C, where no readily available stable sensors exist. This is being ensured by participation of the project partners in the IEC TC 65/SC 65B, the project results have been presented to the group, this is the leading international standardisation group in temperature sensors and interaction with them ensures uptake of the projects results into wisely used standards. It will also clarify some ambiguities associated with the ASTM standard E1751-09, which covers non-letter designated thermocouples. Compliance with regulatory requirements such as the Aerospace Material Specification standard AMS2750E will be facilitated.

For welding, the project will have an impact on EN 13445, ASME VIII and PD 5500 in increasing confidence that surface temperature measurements accurately represent the pre- and post-weld heat treatment temperature and are compliant with ISO 15614-1 (2004). There are currently no standards governing calibration of contact surface temperature probes; this project will set out the necessary technical framework and BSI will be approached to partner in the development of a standard.

There are currently no standards specific to flame or combustion thermometry and this project will lay the groundwork needed to initiate a discussion with standards bodies on improving this situation. This will help with a wide range of applications necessary for manufacture of aircraft and space vehicles which require compliance with relevant standards, in particular ISO 2685.

UCAM/CCPI presented the double wall MI thermocouple results at the SAE International Aerospace Electrical Interconnect Systems Symposium (AEISS) meeting to AMEC committee (responsible for the AMS2750E aerospace heat treatment standard) in October 2016. In addition, CCPI presented the double wall MI thermocouple developments to the ASTM May 2018 Committee Week in San Diego, 20-24 May 2018, specifically the E20 committee responsible for ASTM E 585/E 585M – 04 on MI thermocouple cable (the American equivalent to IEC 61515).

Impact on industrial and other user communities

Immediate industrial impact will be as a result of the direct involvement of industrial project partners. In addition, establishing a complete traceability link from industrial temperature measurements through to the SI will flow to end users via a number of routes, all of which have been progressed during the project:

- Commercialisation of new Pt-Rh, carbon and new format Type K/N thermocouples and sapphire based contact sensors via project partners, or other stakeholders, will enable end-users to realise the benefits of ultra-low drift thermocouples over a wide range of temperatures. External collaborators are involved in testing the newly developed thermocouples. During the project, partners CCPI and UCAM embarked on a commercial collaboration, with CCPI licensing the ground-breaking double wall MI thermocouple technology from UCAM.
- Commercialisation of self-validating thermocouples will allow the user to realise in-situ calibration of thermocouples in-process. Early trials of this have garnered interest for licensing of the technology from CCPI; NPL and CCPI signed a licensing agreement with NPL in May 2018 to allow CCPI, as a large thermocouple manufacturer, to pursue commercial exploitation of the self-validating thermocouples. A central ISO 17025 calibration facility for users' contact surface temperature probes, or apparatus, using fluorescence thermometry for traceable determination of the actual temperature of the surface.
- A portable standard flame for traceable calibration of users' flame/combustion thermometry systems, has been developed and cross-validated using several sophisticated flame thermometry measurement systems. The resulting validated standard flame is now available at NPL as an artefact available to end users for validation of their flame/combustion thermometry systems.
- The UC3M/CEM Hyperspectral Imaging System has been validated by the cross-comparison of the standard flame against other methods. Following on from this work, UC3M/CEM will develop a practical imaging spectrometer for industrial use in EMPRESS2

As detailed in the results section above, many end-users have been involved in different objectives of the project by providing samples, facilitating industrial tests or training their staff on the new technology.

As a result of the widespread project dissemination efforts, the Stakeholder Community associated with the project has been growing, with membership currently standing at 126 parties across government, academia, and industry, including Imperial College, Rolls-Royce, NASA and KRISS. Stakeholder community newsletters have been sent to the members approximately every 6 months to update members on current results and upcoming events. As results of the project have been achieved and disseminated, more commercialisation and licensing opportunities have become apparent.

Impact on the metrological and scientific communities

The consortium has representation on the Task Group 'Guides on Thermometry' of the BIPM Consultative Committee for Thermometry (CCT), who are responsible for promoting good thermometry practice and traceability to the SI, with a focus on secondary (i.e. practical) thermometry; and the EURAMET Technical Committee on Thermometry (TC-T). The representation are ensuring that CCT members are fully aware of the project outputs and that project outputs are widely disseminated via EURAMET TC-T activities.

The latest phosphor thermometry results were given to the EURAMET TC-T at a workshop in February 2016, and have been instrumental in the formation of the Inaugural International Conference on Phosphor Thermometry (ICPT-2018), July 2018 at the University of Strathclyde.

The new developed sensors will make a substantial impact on the ability of NMIs to effectively disseminate the temperature scale to users up to at least 1800 °C, with a concomitant improvement in the Calibration and Measurement Capability (CMC), as recorded on the BIPM key comparisons database, boosting the technical and economic competitiveness of European NMIs and their customers.

Longer-term economic, social and environmental impacts

The project has, and will have, impact on a broad front. Through improved facilities at NMIs and the resulting propagation of lower uncertainties, as well as through new products which are in the process of being commercialised, better efficiency and consistency across a wide range of processes will be achievable. The efficiency of almost every process depends on its accurate temperature control; furthermore, improving energy efficiency is inextricably linked to reduced carbon emissions. Sectors that are vulnerable to rising energy prices and EU policies aimed at reducing greenhouse gases, with the resulting threat to international competitiveness, will benefit from improved process control. As sensors become more reliable in the longer term, safety will also increase. Shifting to a more energy efficient economy will accelerate the spread of technological innovations and boost economic growth, creating high quality jobs. Finally, the population of the EU is increasingly affected by climate change; energy efficiency is a key factor in the reduction of the causative greenhouse gases.

List of publications

1. Extra points for thermometry, J.V. Pearce, Nature Physics Vol. 13, January 2017, p. 104
2. EMPRESS: A pan-European project to enhance manufacturing process efficiency through improved temperature control, J.V. Pearce, Measurement and Control, Vol. 49 No. 8 pp. 252-255, October 2016
3. Optimising Pt-Rh thermocouple wire composition to minimise composition change due to evaporation of oxides, J.V. Pearce, Johnson Matthey Technology Review 60(4) 238-242 (2016)
4. EMPRESS – A European Project to enhance process efficiency through improved temperature measurement, J.V. Pearce, F. Edler, C.J. Elliott, L. Rosso, G. Sutton, S. MacKenzie, G. Machin, Proc. 17th International Congress of Metrologie (2015), EDP Sciences, DOI: 10.1051/metrology/20150008001 (2015)
5. Major Step Forward in Type K and Type N Thermocouple Performance, T. Ford, Industrial Heating, 8 January 2018, https://www.industrialheating.com/articles/93965-major-step-forward-in-type-k-and-n-thermocouple-performance
6. Thermoelemente auf Graphitbasis - Möglichkeiten und Grenzen; Graphite-based thermocouples – possibilities and limitation, F. Edler, S. Haupt, tm – Technisaches Messen, Volume 84, Issue 12, Pages 779–786 (2017) doi.org/10.1515/teme-2017-0073
7. The NPL Standard Flame: operating instructions, NPL Report ENG 65, November 2016, ISSN 1754-2987 http://www.npl.co.uk/content/ConPublication/7281

Project start date and duration:		01 May 2015 (36 months)
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Internal Funded Partners: 1 NPL, United Kingdom 2 BRML, Romania 3 CEM, Spain 4 CMI, Czech Republic 5 DTI, Denmark 6 DTU, Denmark 7 INRIM, Italy 8 JV, Norway 9 PTB, Germany	External Funded Partners: 10 Elkem, Norway 11 GF, Italy 12 MUT, Germany 13 STRATH, United Kingdom 14 UC3M, Spain 15 UCAM, United Kingdom 16 UOXF, United Kingdom	Unfunded Partners: 17 BAE, United Kingdom 18 CCPI, United Kingdom
RMG1: BIM, Bulgaria (Employing organisation); CMI, Czech Republic (Guestworking organisation)		