

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 aims 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. This project will perform trials of the developments 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).

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.

Objectives

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

The specific objectives are 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 will focus on improving this aspect. They are typically made of 2 different metal alloy wires.

Objective 1 Novel, low drift temperature sensors

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 is advancing the state of the art by developing optical-based sensors using single-crystal sapphire light guides. Metrological characterisation of the sapphire tube thermometer will be performed.

Objective 2 Ultra-low drift sensors

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 will be implemented. The improved Pt-Rh thermocouples described above for objective 1 are also a good candidate for this.

Objective 3 Traceable surface temperature measurement methods

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, are being developed, and trialled in-process, for traceably calibrating contact surface temperature probes. Phosphor thermometry has been selected for robust metrological assessment and a industrially relevant method is being 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 has been 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 have also been commissioned. The portable standard flame is currently being 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

Seven different Pt-Rh alloy two-wire thermocouples were constructed and measured to find the most stable combination, this identified Pt-6%Rh vs. Pt-40%Rh as an 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 were started and are running presently at NPL, PTB, CEM and KRISS, the Korean NMI, a collaborator on the project.

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. Further investigation of multiple wavelengths and/or a redesign of the tungsten filament will be carried out.

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 are being performed.

Objective 2 Ultra-low drift sensors

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*, stability/robustness tests are on-going. Licensing opportunities are currently being realised.

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

Objective 3 Traceable surface temperature measurement methods

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 is undergoing evaluation.

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.

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 in due course. Additionally, a UV flame thermometry system has been developed at DTU, the instrument will provide a novel method of flame thermometry and species determination.

UOXF have developed a laser induced grating scattering (LIGS) combustion thermometer and demonstrated successful thermometry in a non-laboratory flame with an uncertainty of 3.5 %. With a significant fraction of this uncertainty ascribed to flame flicker, it is expected that this will be significantly reduced when measuring the portable standard flame. More recently, a single-mode laser system for LIGS in H₂O at 3 μm has been constructed and tested. The portable standard flame has now been installed at UOXF and experiments using the standard flame using H₂O LIGS will begin soon.

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 will be assessed. Currently two papers are ready for submission regarding the portable standard flame performance and a further publication is planned describing trials.

Impact

A significant effort has been given to interact with relevant members of the temperature measurement industry. As a result the stakeholder community interacting with the project has grown to 118 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.

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. 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.

Impact on relevant standards

Immediate impact on standards will be 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 widely 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.

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.
- 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 an industrial stakeholder.
- 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 is being trialled.

As detailed in the results section above many end-users are being 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 118 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 are being achieved and disseminated, more commercialisation and licensing opportunities are becoming 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.

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.

List of publications

1. J.V. Pearce, F. Edler, C.J. Elliott, L. Rosso, G. Sutton, S. MacKenzie, G. Machin, EMPRESS – A European Project to enhance process efficiency through improved temperature measurement, Proc. 17th International Congress of Metrology (2015), EDP Sciences, DOI: 10.1051/metrology/20150008001 (2015)
2. M.A. Rodríguez-Conejo, Gas remote sensing through Fourier transform hyperspectral imaging in the mid-infrared range, PhD Dissertation (awaiting publication)
3. G. Sutton, The NPL Standard Flame: Operating Instructions, NPL Report ENG 65, ISSN 1754-2987 (2016)
4. 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)

5. 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
6. 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)
7. J.V. Pearce, F. Edler, C.J. Elliott, L. Rosso, G. Sutton, R. Zante, G. Machin, EMPRESS: A European Project to Enhance Process Control Through Improved Temperature Measurement, Int. J. Thermophys. **38**, 118 (2017)
8. C.J. Elliott, A. Greenen, D. Tucker, T. Ford, J.V. Pearce, A Slim-line Integrated Self-validating Thermocouple – Initial Results, Int. J. Thermophys. **38**, 141 (2017)
9. J.V. Pearce, Extra points for thermometry, Nature Physics Vol. **13**, January 2017, p. 104

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		https://www.strath.ac.uk/research/advancedformingresearchcentre/ourwork/projects/empressproject/
Internal Funded Partners:	External Funded Partners:	Unfunded Partners:
1 NPL, United Kingdom	10 Elkem, Norway	17 BAE, United Kingdom
2 BRML, Romania	11 GF, Italy	18 CCPI, United Kingdom
3 CEM, Spain	12 MUT, Germany	
4 CMI, Czech Republic	13 STRATH, United Kingdom	
5 DTI, Denmark	14 UC3M, Spain	
6 DTU, Denmark	15 UCAM, United Kingdom	
7 INRIM, Italy	16 UOXF, United Kingdom	
8 JV, Norway		
9 PTB, Germany		