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Abstract. EMPRESS 2 is a European project to enhance the efficiency of high value manufacturing processes by improving temperature measurement and control capability. This project seeks to address four contemporary thermometry challenges in this sector, and new developments from this and its predecessor project, EMPRESS, will be described:

- Below 1000 °C many industrial processes require reliable surface thermometry e.g. welding, coating, forging and forming. Conventional non-contact surface thermometry techniques e.g. thermal imaging are prone to large errors (tens of degrees) due to reflected thermal radiation and unknown emissivity. Contact thermometry approaches are prone to similarly large errors. Traceable imaging phosphor thermometry is being developed to overcome these difficulties, and is being combined with quantitative thermography to determine emissivity for thermometry over wide fields of view.
- Above 1300 °C sensor drift is a significant unaddressed issue for casting, forging and heat treatment, causing large errors. There is a need for more stable sensors and standardisation of at least one new thermocouple type to fill the gap from 1300 °C to 1800 °C. This is being addressed through improved Pt-Rh thermocouples and optimisation of double-walled mineral insulated, metal sheathed thermocouples by mitigating insulation breakdown and drift effects.
- Combustion temperature measurement is very challenging and traceability is almost non-existent; for example, thermocouple measurements of flame temperatures can be in error by hundreds of degrees. A ‘standard flame’ that can be transported to users’ sites has been developed, and is being deployed in several high value manufacturing and industrial applications to a) demonstrate the possibility of reducing flame temperature uncertainties by at least an order of magnitude and b) for the first time to demonstrate in-situ traceability to the International Temperature Scale of 1990 (ITS-90).
- Many processes are not amenable to any conventional thermometry techniques due to inaccessibility, ionising radiation, electromagnetic interference, and contamination; here methods based on optical fibres are ideal but there are no traceable calibration techniques for such sensors currently available. A suite of different fibre-optic thermometers and calibration techniques is being developed to address this. In some cases (ionising radiation) darkening of the fibre is a problem, and this is being

overcome by the development of novel thermometry approaches based on practical 'hollow core' fibres.

1. Introduction
Manufacturing in the European Union (EU) currently accounts for about 40 % of EU exports, and the lion's share of company R&D takes place in high value manufacturing [1]. However, the proportion of manufacturing has been in decline as services account for an increasing share of exports. The EU has strategic ambitions to restore a sustainable level of manufacturing and achieve or retain global leadership in sectors such as automotive, aeronautics, engineering, space, chemicals and pharmaceutical industries [2]. EU companies cannot compete on low price, low quality products. They must turn to innovation and productivity, energy and resource efficiency (and minimising waste), and high value-added to compete in global markets [3]. In practical terms this means enhancing efficiency – that is, energy efficiency and improved product consistency, which are strongly correlated – in industrial processes, which means improving process control, i.e. temperature measurement.

There is a practical imperative for European organisations: the EU's climate and energy package mandates a 20 % increase in energy efficiency across Europe by 2020 [4]. This is considered a Grand Challenge in EURAMET's Strategic Research Agenda for Metrology in Europe 2016 [5], which also identified the improvement of the efficiency of power generation and other industrial processes through improved metrology of critical monitoring and control parameters as a research priority [5]. Improved *in-situ* monitoring and control for enhancing process efficiency through improved instrumentation is identified in [5] as a key outcome. All energy efficiency gains are accompanied by a corresponding reduction in carbon emissions [6]. An energy efficiency improvement target of 20 % is also the aim of the European Directive 2012/27EU [7] on energy efficiency, which has recently been updated with an even more ambitious target of 30 % by 2030 in COM(2016) 860 [8] with a proposal for legislation in COM(2016) 761 [9]. This signals the intent of the EU to bring energy efficiency to the forefront of its strategy. The target of 30 % looks set to be formalised by a revision to Directive 2012/27/EU [9].

There are a number of key challenges in high value manufacturing associated with improving efficiency (i.e. both energy efficiency and improved productivity) and reducing greenhouse gas emissions. Surface temperature measurement is a common challenge in advanced manufacturing. Measurement of the temperature of billets during forming, forging and heat treatment up to 1000 °C remains problematic because contact probes cannot be inserted or placed in contact with the surface due to contamination and heat flow effects (which cause errors). Non-contact thermometry methods are beset by the problem of unknown emissivity and reflected thermal radiation. Heat treatment of steel structures before and after welding is a critical part of large scale manufacturing, and a rapid, practical means of measuring the surface temperature is urgently needed due to the very high rejection rates and poor product consistency associated with existing methodologies. Measurement of the surface temperature of rapidly moving parts e.g. automotive brake disks, which can reach up to 1000 °C, remains challenging due to the difficulty of ensuring that conventional contact probes are in adequate thermal contact with the surface. The use of phosphors has facilitated improvements in this area because they are effectively a coating which is in almost perfect thermal contact with the surface and it is non-perturbative, but they provide only a point measurement; what is really needed is an imaging technique to provide information on the spatial temperature distribution. Here traceable 2D phosphor thermometry will be developed, both with scanning techniques and by combination with quantitative thermography to solve the emissivity problem with conventional radiance based thermometry approaches.

The imminent introduction of a new double-walled MI thermocouple by UCAM and CCPI [10] will result in the commercial availability of a new ultra-stable thermocouple for use up to 1300 °C. However, the new double-walled format makes it challenging to meet the dimensional requirements set out in IEC 61515 [11] because the thicker protective outer wall reduces the amount of space inside leading to the thermocouple wires having a smaller diameter which in many cases falls below the minimum permitted by the standard. Similar limitations apply to industry standards such as AMS 2750E [12] (heavily used in aerospace manufacturing), which may limit uptake. Drift tests of the new thermocouples alongside conventional MI thermocouples are required to provide the documentary evidence needed to revise IEC 61515. To maximise the benefits to end users, further optimisation is required, in particular the optimisation of the ratio of the two outer wall thicknesses. Furthermore, now that these sensors exhibit substantially improved stability up to 1300 °C, a new measurement error has appeared, due to the decreased resistance of the insulating ceramic material between the thermocouple wires [13]. Also above about 1000 °C Pt-Rh thermocouples are increasingly widely used. In EMPRESS a systematic investigation identified the Pt-40%Rh vs. Pt-6%Rh thermocouple as being optimal in terms of stability; and a series of measurements are required to progress the standardisation of these developments. Essential underpinning standardisation measurements are needed.

Fire resistance testing and standardisation in aerospace applications remains a significant issue due to the extreme difficulty of measuring reliable and traceable flame temperatures; this generally requires the two dimensional measurement of flame temperature. Flame and combustion temperature in R&D production settings is often performed with sophisticated laser diagnostic apparatus; but, despite the sophistication, temperature uncertainties are typically of the order of 10 % and are not traceable. Measurements are often used to validate combustion models; more accurate modelling requires better measurements. A number of European companies are developing combined heat and power (CHP) plants and waste incinerators for both domestic and industrial use; widespread uptake of CHP is identified as a priority by the European Commission [14]. For small electricity/heat production units such as, for example, waste incinerators and/or district power plants, selective non-catalytic reduction (SNCR) technology is commonly used to reduce NO_x emissions at around 1000 °C. A 2D temperature profile measurement of the combustion process will enable better control of the temperature in the NO_x SNRC process, to optimise NO_x reduction and consumption of NH_3/NH_4OH. A portable standard flame is needed to introduce traceability, to improve process control accuracy, and to enhance efficiency.

There are many processes (e.g. brake pad production/testing, forging) which strongly depend on temperature and which are not amenable to monitoring with conventional sensors such as thermocouples (contamination/transmutation/electromagnetic fields) or thermography (unknown emissivity/no line-of-sight). Examples include plasma-based processes, silicon processing, ionising radiation, particularly the gamma rays used for radiotracers or weld inspection, and high temperature industrial furnaces e.g. induction furnaces. Fibre-optic thermometers have become widespread since their inception in the 1960s, but few, if any, currently offer the possibility of traceable measurements. Different types of fibre-optic thermometers need to be developed offering immunity to different types of harsh environment, including point sensors and distributed sensors. Development of each type of sensor needs to be accompanied by the development of a traceable calibration methodology, and an *in-situ* demonstration in process environments in order to demonstrate the utility of the new metrological framework for fibre-optic thermometry.

This paper is laid out as follows. Firstly the EMPRESS2 consortium is introduced with a description of the partners' activities and synergies. The technical activities are then summarised, with four work packages devoted to phosphor thermometry, thermocouples, combustion thermometry and fibre optic thermometry respectively. The paper concludes with a brief summary.

2. The consortium

The consortium comprises 25 partners across Europe including 9 NMIs, 1 DI, 4 universities, 2 research institutes, and 9 companies. The partners are summarised in Table 1; this also shows the strong complementarity of the specialisms.

Table 1. Project partners and their associated specialisms.

Participant Type	Short Name	Organisation legal full name	Country	Specialism
NMI, Project Coordinator	NPL	National Physical Laboratory	United Kingdom	Thermocouples, fibre optics, phosphor, combustion
NMI	CEM	Centro Español de Metrología	Spain	Thermocouples
NMI	CMI	Český Metrologický Institut Brno	Czech Republic	Thermocouples
NMI	DTI	Teknologisk Institut	Denmark	Surface temperature
NMI	INRIM	Istituto Nazionale di Ricerca Metrologica	Italy	Phosphor thermometry

NMI	JV	Justervesenet	Norway	Optics, blackbodies
NMI	PTB	Physikalisch-Technische Bundesanstalt	Germany	Thermocouples, photonics
NMI	TUBITAK	Turkiye Bilimsel ve Teknolojik Arastirma Kurumu	Turkey	Thermocouples
NMI	UL	Univerza v Ljubljani	Slovenia	Thermocouples
DI	DTU	Danmarks Tekniske Universitet	Denmark	IR & UV spectroscopy
Research institute	CNR	Consiglio Nazionale delle Ricerche	Italy	Tribology
Research institute	IPHT	Leibniz-Institut für Photonische Technologien eV	Germany	Fibre optics, lasers
Company	Elkem	Elkem AS	Norway	Silicon processing
Company	ITT	ITT	Italy	Braking systems
Company	MUT	MUT Advanced Heating GmbH	Germany	Furnace manufacturing
Company	ACERINOX	Acerinox SA	Spain	Steel manufacturing
Company	BAE	BAE Systems Marine Limited	United Kingdom	Shipbuilding
Company	B&W Volund	Babcock & Wilcox Vølund A/S	Denmark	Waste incineration
Company	CCPI	CCPI Europe Ltd	United Kingdom	Thermocouples
Company	JM	Johnson Matthey	United Kingdom	Precious metals
Company	Sensia	Sensia Solutions	Spain	IR imaging devices
University	UoS	University of Southampton	United Kingdom	Hollow core fibres
University	STRATH	University of Strathclyde (includes Advanced Forming Research Centre, AFRC)	United Kingdom	Phosphor thermometry & forming/forging
University	UC3M	Universidad Carlos III de Madrid	Spain	Optics & IR instruments
University	UCAM	The Chancellor, Masters and Scholars of the University of Cambridge	United Kingdom	Thermocouples

Table 2. Grouping of specialisms to illustrate the collaborative activities.

Activity	**Participant**	**Specialism**
Phosphor thermometry (decay-time)	INRIM	Phosphor thermometry development
	CNR	Tribology
	ITT	Manufacture of braking systems

Phosphor thermometry (intensity ratio)	NPL	Phosphor thermometry development, traceable calibration techniques
	STRATH	Phosphor thermometry development
	DTI	Phosphor thermometry development
	BAE	Provide access to marine manufacturing for trials
Thermocouple thermometry	CMI, TUBITAK, UL, NPL, PTB, CEM, DTI	Traceable calibration facilities
	JM, CCPI, UCAM	Supply of thermocouple wire
Combustion thermometry	NPL	Supply, calibrate portable standard flame
	DTU	Development of IR, UV spectroscopy *in-situ*/on-line measurement techniques
	B&W VOLUND	Provide access to waste incineration facilities for trials
	SENSIA	Development of commercial IR imaging devices
	UC3M	Development of optics and IR instrumentation
Fibre-optic thermometry (hybrid BB/FBG)	IPHT	Development of laser and fibre optic techniques
	JV	Development of sapphire based sensors; traceable calibration techniques
	PTB	Development of FBG fibre optic sensors; traceable calibration techniques
	MUT	Provide access to industrial furnace manufacturing for trials
	Elkem	Provide access to silicon processing for trials
Fibre-optic thermometry (hollow-core/bundles)	UoS	Optical fibre development, instrumentation
	NPL	Development of traceable calibration techniques
Fibre-optic thermometry (distributed)		
	ACERINOX	Provide access to stainless steel manufacturing for trials
	CEM	Development of traceable calibration techniques

3. The work packages

Good progress was made in the first EMPRESS project [15,16], 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. In particular, each of the key developments was tested in at least one real-world setting. The activities are grouped into four work packages devoted to phosphor thermometry, thermocouples, combustion thermometry and fibre optic thermometry respectively.

3.1 WP1: Accurate methods for phosphor thermometry

In the new project, EMPRESS 2, phosphor thermometry will be further developed to facilitate reliable surface temperature mapping for the first time, so that parts undergoing forging, forming, welding, or heat treatment can be monitored with respect to the entire surface rather than a point. Secondly, the technique will be combined with quantitative thermography to enable real-time determination of emissivity, with a target uncertainty of better than 3 °C up to 1000 °C. Multiple partners, bringing complementary techniques, will enable validation of these techniques and implementation in-process in a suite of manufacturing environments.

In EMPRESS, traceable phosphor thermometry was established on a routine basis, with the phosphor coating being applied to a surface and a spot measurement made with uncertainty of about 1 °C. With this, surface temperatures up to 500 °C can be performed. The concept is being taken forward in EMPRESS2 with three aims in mind, namely to extend to 1000 °C, to extend to two dimensions, i.e. phosphor imaging, and to combine with quantitative thermography to enable *in situ* determination of emissivity.

The aim of this activity is to develop, validate and test a suite of phosphor thermometry sensing techniques for accurate and traceable surface temperature measurement. The outputs will be trialled in high-value manufacturing processes, such as welding, coating, forging and forming, to provide traceable surface temperature measurements. The systems will be developed for use up to 1000 °C.

There are three key types of phosphor thermometry systems to be developed, each with a specific type of application in mind:

1. The first is a 2D intensity ratio phosphor thermometer, employing a traceably calibrated phosphor. This will be combined with quantitative thermography, enabling the production of emissivity maps.
2. The second will be optimised for the coating of billets to enable both online monitoring of billet temperature over the entire piece during heat treatment, and 'offline' monitoring whereby the phosphor records the temperature during the heat treatment for subsequent determination. The latter approach is useful when the billet cannot be viewed.
3. The third is a fibre-optic system for remote interrogation of the phosphor with a specific application in automotive brake pad/disk temperature determination. The target uncertainty of the techniques is less than 3 °C up to 1000 °C.

The following is a summary of the six key tasks.

3.1.1 Task 1.1 Development of a phosphor thermometer from 500 °C to 1000 °C

The aim of this task is to develop a phosphor thermometry capability up to 1000 °C and to demonstrate its performance in a number of trials. The accuracy of non-contact surface thermometry critically depends on knowledge of the surface emissivity. Additionally, background thermal radiation reflected from the surface can introduce significant measurement errors. Phosphor thermometry involves the interrogation of a thin phosphor coating (previously applied to the surface) following excitation from UV radiation. By measuring either the decay in the subsequent fluorescence with time or measuring the ratio of two emission bands, the temperature can be found that is independent of surface emissivity, background radiation and moderate levels of participating media between the surface and the instrument (i.e. windows, smoke etc.). By combining phosphor thermometry and established thermal imaging, it will be possible to independently determine the surface emissivity and to significantly reduce thermal imaging uncertainties (to an uncertainty better than 3 °C).

The requirements and adaptations of phosphor thermometry will mostly depend on the applications and their specific conditions. In the last two decades, this technique has been increasingly applied to a myriad of processes such as turbine engine diagnostics, monitoring of thermal barrier coatings [41], and more recently for temperature measurement for medical applications on the nanoscale [42,43].

For surface thermometry for forming and forging processes, good adhesion to the metal substrate is required. This is often achieved by testing numerous ceramic binders, sintering recipes and coating processes in order to match the thermal expansion coefficient of the coating and the metal substrate over the whole temperature range to avoid delamination. Additionally, this is also affected by the environmental conditions i.e. furnace atmosphere (vacuum, gas fired or electrical furnace), and presence of graphite based lubricant during the forging process. For example, a preform or billet with a phosphor coating patch submitted to a long heat treatment inside a gas fired furnace (oxidative atmosphere with high water partial pressure) could accelerate the coating delamination due to metal surface oxidation. Pressure effect on the luminescent lifetime of various phosphors has also been studied recently and this is particularly relevant for phosphors inside combustors and gas turbines [44].

As already mentioned, NPL have successfully applied phosphor coating to very diverse metal substrates and observed good integrity and durability of the coating for mild process conditions. In terms of thermal history coatings, these rely on the irreversible oxidation of the rare earth element within the phosphor to indicate the highest temperature achieved. For this reason, they are either used in their amorphous state or they need to be sintered in either a reducing atmosphere or a vacuum. Moreover, in order to withstand longer exposure time in harsh environment and improve coating robustness, atmospheric plasma spray can be used to apply the phosphor powder onto the intricate component to be monitored.

The following is a summary of the six key tasks.

Progress: NPL has developed a prototype intensity ratio imaging phosphor thermometer that can measure from 20 °C to 450 °C [17]. This has been applied to a real-world measurement problem, namely a 3 mm x 1 mm x 20 mm steel coupon electrically heated in the NPL electro-thermal mechanical testing (ETMT) machine. Although the coupon is behind a Perspex cover, unlike thermography (thermal imaging), the phosphor thermometry is unperturbed by the cover, and, also unlike thermography, information on the emissivity is not needed. Figure 1 shows a typical 2D image of the coupon made with the new system. Significant impact has been demonstrated in the application of this device to characterizing photovoltaic cells [28].

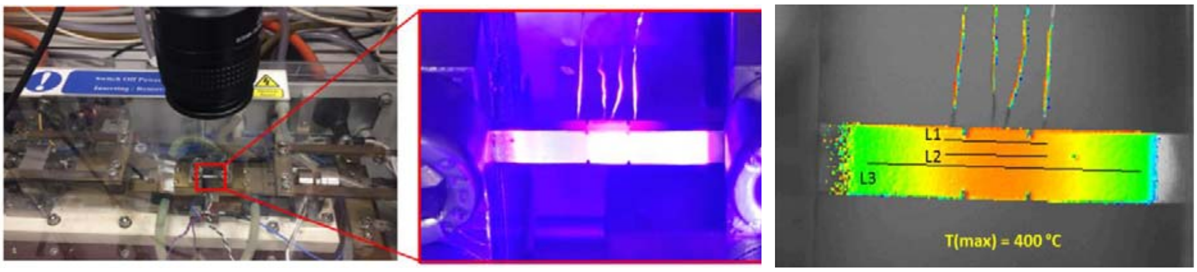

Figure 1. Phosphor thermometry applied to a coupon undergoing mechanical testing. Left: the testing apparatus, with the coupon shown in the box. Centre and right: temperature distribution (shown by colour) of the coupon.

The phosphor coatings are capable of being sprayed with thicknesses of around 35 μm while remaining durable and homogeneous. Additionally, if larger signal intensities are required the coatings can be applied thicker. Point measurements are currently achievable reliably, as the work carried out in EMPRESS demonstrated [15], although this was carried out at lower temperatures from about 20 °C to 200 °C. For the high temperature phosphor, three calibration cycles, from 20 °C to 750 °C, have been completed for the intensity ratio imaging phosphor thermometer using Mg_4FGeO_6:Mn phosphor and 643-2 binder. This has demonstrated a measurement uncertainty of less than 3 °C to 10 °C for temperatures below 600 °C and between 600 °C and 750 °C respectively. In addition to a high-temperature furnace system, an additional small box furnace with sapphire window has been developed and tested up to 1000 °C. This new facility provides the capability for rapid thermal cycling and *in situ* calibration of the phosphor thermometry system.

AFRC have supplied six different metal samples to NPL for testing, that would cover a very wide range of applications/sectors (specifically aerospace, oil and gas, and automotive). The materials supplied are H13 tool steel, Inconel 718+ (nickel based super alloy), C42 MOD (micro alloyed steel), Ti64 (titanium alloy) Ti-10-2-3 and Nimonic 105 (nickel-cobalt-chromium-base alloy). NPL has sent back flat square samples cut from these materials (20 mm by 20 mm) for emissivity measurement trials from 300 °C to 900 °C.

A new coating recipe supplied by a specialist coating company in the UK has solved a number of challenges associated with adhesion of the phosphor to various surfaces, with coatings remaining adhered and still retaining the previously mentioned calibration uncertainties after seven cycles between ambient temperature and about 750 °C. Measurements of the different metal samples are ongoing and trials of the phosphor thermometry to determine emissivity *in situ* at AFRC are ongoing.

NPL, DTI and STRATH are also currently in the process of intercomparing their phosphor thermometry instrumentation using a set of coupons of different metals coated with calibrated phosphor by the three participants to assess comparability of phosphor application and the instrumentation itself.

3.1.2 Task 1.2 Development of phosphor sensors for surface temperature monitoring on forging tools

The aim of this task is to devise a phosphor sensor that is suitable for monitoring surface temperatures on the surfaces of forging tools up to 1000 °C. Tool temperature is critical for the quality of the components produced, but it is difficult to monitor due to the extreme conditions at the tool surface (high temperature, high mechanical loads and the presence of lubricants) and intermittent access (optical access can only be gained when the press is open between operations, whilst the temperatures experienced during the pressing process are the most critical). Phosphors operate at high temperatures, offer good signal to noise ratio and emissivity independent measurement, so they have the potential to meet the needs of this extreme environment. Two sensors can be envisaged. One for tool temperature monitoring on a continuous basis between every pressing operation and one for use during the setup of the apparatus, prior to actual processing, to record the surface temperature during the process when the tool is closed. The former can be achieved using an online sensor and the latter by an offline 'memory' type sensor. The task will seek to identify and characterise a suitable phosphor for each type of application.

Progress: STRATH has identified suitable phosphors with measurement capability in the relevant temperature range experienced by tools and work pieces in representative forging processes. This includes those phosphors with both on-line (i.e. live measurements) and off-line (i.e. inaccessible during the process – data is recovered afterwards) response modes. Using a selected phosphor a coating-type sensor design has been devised for on-line surface temperature mapping, and prototypes have been manufactured. Currently terbium doped yttrium orthosilicate, YSO:Tb is being assessed; terbium doped phosphors have been shown to have sufficient photoluminescent intensity at temperatures above 500 °C, and exhibit decay lifetimes in millisecond timescales. A sample of 5 % terbium doped yttrium orthosilicate has been produced using the sol-gel process and is currently being assessed. A measurement system based on 2D lifetime decay mapping has been designed and is under development. Appropriate measurement systems including detectors and signal processing routines are also under development. Ultimately both systems will be tested and calibrated to determine the dynamic range and measurement uncertainty (target 3 °C). A three-gate technique has been identified that allows for the use of a single camera for surface phosphor thermometry and data analysis methods are under development.

3.1.3 Task 1.3 Fibre-optic based measurement system for temperatures of up to 1000 °C in a braking system

The aim of this task is to exploit a phosphor-based approach to develop a novel remote fibre-optic thermometer system, in order to provide traceable measurements in selected applications in the automotive industry for braking system development. The temperature at the contact surface of the disk and the brake pad during operation impacts significantly on brake performance, yet its measurement is very difficult. It can range between 400 °C and 1000 °C depending on the brake features. Temperature measurements, generally performed by using thermocouples inserted in the brake itself, are very complex and exhibit poor reliability. In contrast, analytical or numerical mathematical methods, often used to predict the brake temperature in the contact zone of the friction pair, typically requires numerous simplifications and restrictions in order to offer solutions to the observed problem. The phosphor-based fibre-optic thermometer system will address these issues.

Progress: INRIM has performed a traceable calibration of the selected phosphor in an isothermal environment by measuring its decay time at a series of known temperatures (at least two) over the whole working temperature range [18]. A standalone system, including an electro-optical unit, signal processing and software for measuring the fluorescence decay time as a function of temperature has been developed at INRIM.

Different parameters have been considered such as functionality temperature range, fluorescence lifetime, thermal sensitivity, signal to noise ratio and optical features. Two phosphors were eventually selected: Chromium-doped $YAlO_3$ (Cr:YAP) and Cr-doped gadolinium aluminium perovskite (Cr:GAP). Cr:YAP exhibits a high temperature sensitivity over a wide working range (i.e. from 10 ms / °C to almost 100 ms / °C from room temperature to more than 700 °C), a high intensity of the fluorescence signal and a long fluorescence lifetime.

Chromium-doped gadolinium aluminium perovsike (Cr:GAP) is a phosphor that combines the ultra-bright luminescence of transition metal dopants with long decay-times even at high-temperatures; it is basically suitable for temperature measurements higher than 250 °C, but is more sensitive at temperatures above about 750 °C. A suitable high temperature ceramic binder, to be mixed with the phosphor powder, was also selected and several tests with different phosphor/binder ratio have been performed on the different type of surfaces in order to obtain adequate adhesion and uniformity of the phosphor coating.

At INRIM the characterization of the two selected phosphors, Cr:YAP and Cr:GAP, has continued in order to assess its suitability under conditions similar to that of automotive braking systems (e.g. chemical stability, interaction between the phosphor and the brake pad, etc.). A phosphor layer, mixed with a suitable ceramic binder, has been coated on a brake pad sample. This was then placed in an oven and exposed to temperatures up to about 400 °C. De-activation of the phosphor luminescence was observed after a long exposure to temperatures around 400 °C, which is thought to be due to the contamination by gases emitted by the hot brake pad material into the phosphor layer. In addition, hysteresis of the phosphor was observed. An effective solution was to bake the brake pad prior to application of the phosphor in order to outgas the volatile contaminants, then, after application of the phosphor, to carry out a high temperature annealing for at least an hour to stabilize the phosphor.

3.1.4 Task 1.4 In-process tests of a phosphor thermometer

The aim of this task is to deploy the instrumentation developed in Task 1.1 (fibre-optic phosphor thermometer) under real operating conditions at STRATH (AFRC, heat treatment) and BAE (welding pre-heat treatment).

NPL will demonstrate precision phosphor thermometry of billets during heat treatment at STRATH (AFRC) and will compare the results with conventional thermal imaging techniques. NPL will demonstrate and test the 2D intensity ratio phosphor thermometry system in a number of marine welding applications in-process at BAE. NPL will demonstrate the phosphor thermometer/thermography combination in an industrial environment e.g. glass manufacturing at collaborator AGH. DTI will facilitate access to the collaborator's site.

3.1.5 Task 1.5 Testing phosphor thermometry in a forging process

The aim of this task is to test the phosphor thermometer systems developed at STRATH under real operating conditions in one of the heat treatment facilities at STRATH (AFRC). STRATH will modify at least two selected forging tools to incorporate at least one of the selected phosphor sensors devised by STRATH. Multiple tool sets will be used to enable both online and offline sensors to be tested.

Progress: STRATH has developed emissivity-stable thermographic phosphor coatings for a dual phosphor/pyrometer thermometry system capable of providing reference temperature measurements to the dynamic correction of surface emissivity. Emissivity measurements carried out at AFRC have demonstrated the high stability of these phosphor coatings in the temperature range from 50 °C to 550 °C, bound to the stainless steel substrate. Currently the three-gate phosphor thermometer measurement system constructed at STRATH is limited to 550 °C due to the decreasing lifetime of the phosphor MFG:Mn. To increase the temperature range, experiments are ongoing to increase the ratio of phosphor to binder, while retaining the emissivity characteristics.

Other issues with the three-gate phosphor thermometer system have been overcome, including excessive bleed-through excitation (addressed by adding an additional optical filter) and coating degradation causing a slow reduction in the luminosity of the phosphor over several hours (a result of substrate-binder interaction, solved by using a different substrate).

The system is now able to perform continuous 'online' measurements during processes such as forming and forging at the AFRC, although this requires postprocessing to extract temperature data. Further development is underway to enable real-time measurements.

3.1.6 Task 1.6 Phosphor thermometry applied to brake pads

The aim of this task is to study the influence of temperature, as measured at the interface of an automotive brake pad and disk by phosphor thermometry, on braking system properties. Good performance of a braking system is strictly dependent on the formation of a so-called friction layer or third body, which forms when a disk and pad come into contact during braking. The heat generation during braking affects the topology of the friction layer. This in turn induces phenomena such as material deformation that changes the contact pressure, whose uneven distribution is the main reason for uneven wear. The measurement of the temperature at the interface is therefore an aspect of paramount importance when forecasting the whole performance of a braking system.

INRIM will develop an appropriate binding mechanism for good adhesion of a thick layer of the phosphor on at least one of the types of brake pad that is used in industrial applications (supplied by ITT). CNR will advise on binder property constraints. The phosphor thermometer developed by INRIM will be applied to the measurement of surface temperature in the brake testing rig at ITT's industrial facilities.

The surface of the pads and/or disks tested will be analysed by CNR, in order to evaluate the friction layer formed, if any, and any changes in terms of composition, leading to potential material degradation. As the thickness of tribologically induced films is usually of the order of 1 μm or thinner, surface techniques will be preferred. The analysis will either be carried out on both the pad and the disk or only on the disk, depending on the complexity of the pad composition chosen by ITT. SEM-EDS will be used by CNR to determine the morphological and elemental composition, on both the surface and cross section of the worn materials.

Progress: At INRIM the technique for preparing the phosphor coating has been refined by improving the phosphor/binder ratio, the thickness, and the curing process. A series of laboratory measurements were then performed to validate the phosphor temperature measurements on brake pads using the INRIM reference hot plate. A selected brake pad was machined: three bores with different depths were drilled and a Cr:YAP phosphor coating was applied to the bottom of each bore. The aim ws to obtain, using phosphor thermometry, the temperature profile across the brake pad and consequently the temperature at the interface. The brake pad was positioned on the reference surface of the hot plate and pressed against it by applying a combination of weights through a mechanical mechanism to ensure a reproducible contact force. A phosphor spot was also directly applied to the metal surface of the hot plate to provide a phosphor thermometry reference temperature. At several temperatures in the range from ambient to 200 °C, the reference temperature generated by the hot plate apparatus was compared to the phosphor reference temperature and to the extrapolated phosphor temperature (i.e. the temperature obtained by linear extrapolation of the three temperature readings across the brake pad). A linear relationship between the phosphor reference temperature and the hot plate reference temperature was observed, consistent with a thermal contact resistance which depends on the applied load on the brake pad.

CNR have performed Scanning Electron Microscopy using the Energy Dispersion Spectroscopy probe. A new brake pad was analysed and the main constituents were observed (polymer as matrix, iron oxide, barite, aluminium oxide, carbon, steel, various sulphides and oxides, and others). Brake pads having the same composition as the new one, but having been subjected to two braking cycles at the facilities of ITT, were then analysed to assess the friction layer formed and changes in composition. The main changes consisted of surface oxidation and flattening/compression of some constituents at the surface. This information will be used to inform further development of the phosphor application methodology. Analysis of the opposite face (usually a grey iron disc) is ongoing.

3.2 WP2: Low-drift thermocouples

In EMPRESS, a systematic investigation of a number of different Pt-Rh thermocouples showed that the most stable thermocouple consistent with readily available Pt-Rh wire compositions, at least in the temperature range 1324 °C to 1492 °C, is the Pt-40%Rh versus Pt-6%Rh thermocouple [19-21]. This is due to the combined effects of thermoelectric drift due to vaporisation of Pt and Rh oxides [34,35,39] A preliminary reference function was also drafted (Figure 2) [19]. In EMPRESS2, to facilitate uptake of the new thermocouple type, NMIs will systematically determine its reference function. This builds on the development of high temperature fixed points [22] in FP5 project G6RD-CT-2001-00610 HIMERT [23], EMRP JRP IND01 HiTeMS [24,45] and EMPIR JRP 14IND04 EMPRESS [25]. The utility of the optimised thermocouple will be demonstrated by trials in industrial furnace manufacturing, steel manufacturing, and other trials at collaborators' sites e.g. float glass manufacturing applications.

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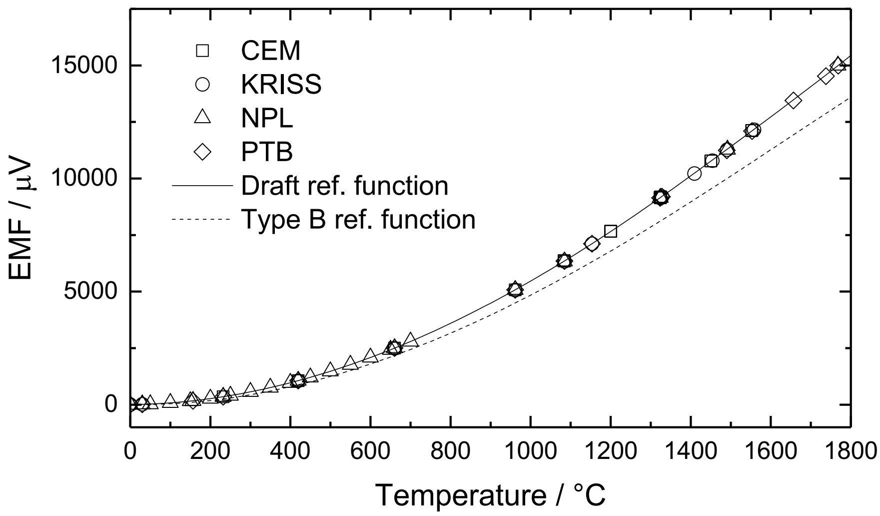

Figure 2. Preliminary reference function for the Pt-40%Rh vs. Pt-6%Rh thermocouple showing aggregated calibration data arranged by participant. The solid line shows the preliminary reference function; dashed line shows the Type B thermocouple reference function for comparison.

A second part of the thermocouple activities concerns the double-walled mineral insulated, metal sheathed (MI) thermocouple developed by UCAM [10]. This is currently the subject of a collaboration between UCAM and CCPI aimed at commercialising the new MI thermocouple type, which exhibits thermoelectric stability approaching that of noble metal thermocouples, at least up to about 1200 °C [10]. Some validation of the drift performance of Type K and Type N thermocouples with this format was performed by UCAM (Figure 3), and in an industrial vacuum furnace at AFRC in EMPRESS (Figure 4); further validation is urgently needed and will be undertaken by several NMIs. This will include a systematic optimisation of the wall geometry, specifically a determination of the optimum ratio of the two wall thicknesses. Finally, the progressive breakdown of the insulation resistance above 600 °C, as the ceramic becomes conductive, will also be studied; this is a potential problem for end-users since the new MI thermocouple is more stable and hence can be expected to be used at higher temperatures more regularly.

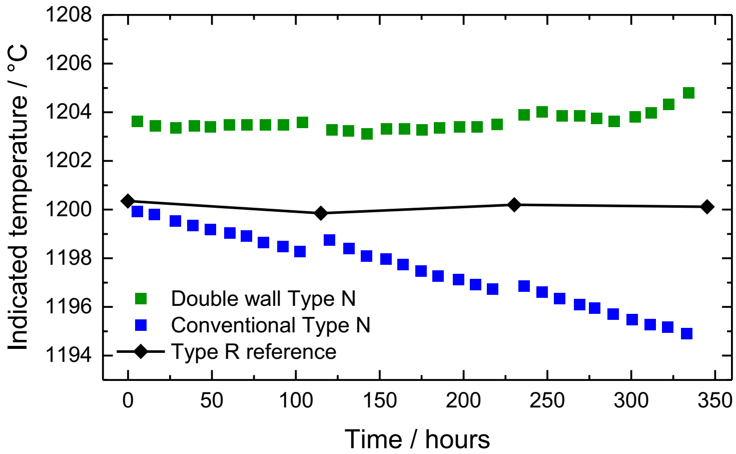

Figure 3. Comparison between the drift of 3 mm double wall Inconel 600 sheathed Type N thermocouples and the conventional equivalent during cycling between room temperature and 1200 °C.

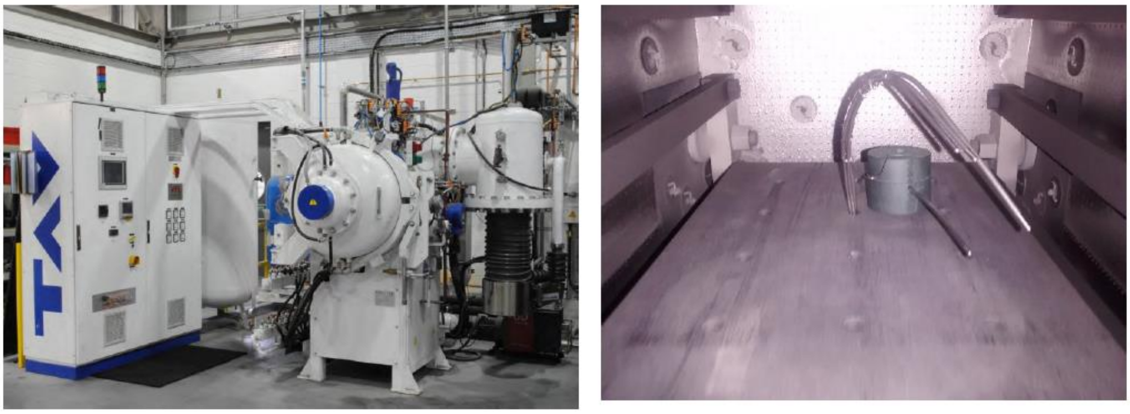

Figure 4. The industrial vacuum furnace at AFRC used for trials of the double-walled MI thermocouples (left) and the thermocouples installed in the furnace (right).

The objective is to characterise these novel thermocouples metrologically with in-process, traceable uncertainties of better than 3 °C for the new Pt-40%Rh vs. Pt-6%Rh thermocouples at temperatures up to 1769 °C and for the double-walled MI thermocouples at temperatures up to 1200 °C, as well as the insulation resistance breakdown. There are three key tasks:

3.2.1 Task 2.1 Standardisation of Pt-40%Rh vs. Pt-6%Rh thermocouples

The aim of this task is to establish an emf-temperature reference function for Pt-40%Rh vs. Pt-6%Rh thermocouples in the temperature range between 0 °C and 1769 °C in air. This will be based on a set of at least 5 (of 11 investigated) exceptionally thermoelectrically stable and homogeneous similar thermocouples or on one or two 'master' thermocouples. All required emf measurements will be traceable to the International Temperature Scale of 1990 (ITS-90) [26], based on measurements of the emf at fixed points and in comparison. The thermocouples will be constructed by the partners. Additionally, the post-assembling heat treatment will be optimised to reach the most stable thermoelectric conditions of the Pt-40%Rh vs. Pt-6%Rh thermocouples.

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The aim is to reach an expanded uncertainty of the emf-temperature reference function of less than a temperature equivalent of 0.5 °C up to 960 °C, rising smoothly to approximately 2 °C at 1769 °C.

Eleven Pt-40%Rh vs. Pt-6%Rh thermocouples have been constructed by PTB, NPL, CEM, CMI and TUBITAK by using thermoelements which have been provided by JM, and additional thermocouples provided to UL and DTI. These are now being used in the reference function determination. In particular, an agreed post-assembly heat treatment has been performed by PTB, CEM, CMI, NPL and TUBITAK to achieve a unique and thermoelectrically stable condition for all thermocouples. PTB and NPL have jointly prepared a measurement protocol to unify the pan-European measurement effort. A by-product of this activity was a revised EURAMET best practice guide on thermocouple calibration, incorporating new, documented information on typical thermoelectric homogeneity values for a range of thermocouples [40].

Using the facilities of PTB, NPL, CEM, CMI, DTI, TUBITAK and UL traceable measurements to ITS-90 are being performed at, where available, all ITS-90 fixed points and high temperature metal-carbon eutectic fixed points, and by applying comparison methods in the temperature range between 0 °C and 1769 °C in order to yield a large number of emf-temperature pairs. During these measurements, tests of the thermoelectric stability and homogeneity will be performed repeatedly according to the agreed measurement protocol.

Progress: The measurements to determine the reference function for Pt-40%Rh vs. Pt-6%Rh thermocouples were completed and an evaluation of the data was carried out. Ten of the eleven thermocouples met the specified stability requirements (drift rate at the Cu point (1084.62 °C) less than 0.5 µV within 50 hours annealing at 1350 °C). The following general principles were applied for the reference function determination:

- Comparison measurements provide the most data points were therefore considered as the key task for the determination of the reference function
- Fixed point measurements were performed to check the thermoelectric stability of the Pt-40%Rh/Pt-6%Rh thermocouples during the measurements by comparison, but were also used to determine the reference function
- Repeated homogeneity tests of the Pt-40%Rh/Pt-6%Rh thermocouples were performed

Detailed information on the measurement conditions (temperature ranges of the comparison measurements, apparatus, standard thermometers and fixed points used are described in [32].

For each of the ten Pt-40%Rh vs. Pt-6%Rh thermocouples different numbers of temperature / emf pairs were obtained. The data of the thermocouples CEM-2018-1 and CEM-2018-2 covered the widest temperature range and the difference between them was smaller than the expanded uncertainty. Furthermore, they showed good thermoelectric homogeneity and stability. One condition to perform a least squares fitting to determine the emf-temperature relationship of a thermocouple is that all data are from one thermocouple or the data are from a set of statistically indistinguishable thermocouples. Therefore, based on all data measured, the data of the CEM thermocouples were chosen for the construction of the reference function.

The resulting reference function was subject to considerable analysis, and arrived at a final solution of two polynomials, one from 0 °C to 660.323 °C (the freezing point of aluminium), and another one above 660.323 °C. The reference function relates the thermoelectric voltage emf in units of µV to the temperature t in units of °C:

$$emf = \sum_{i=0}^{n} a_i \cdot t^i$$

The polynomial coefficients are shown in Table 3. The uncertainty associated with the reference function was also carefully analysed, and is shown in Figure 5. Full results are presented in [32].

Table 3: Reference function coefficients.

coefficients	below Al	above Al
a_0	0	71435.3813
a_1	0.137315647	-568.1009491
a_2	0.006333794	1.941456131
a_3	-3.65645E-06	-0.003686384
a_4	8.80882E-09	4.29132E-06
a_5	-1.13502E-11	-3.12896E-09
a_6	5.40932E-15	1.39612E-12
a_7		-3.48828E-16
a_8		3.73963E-20

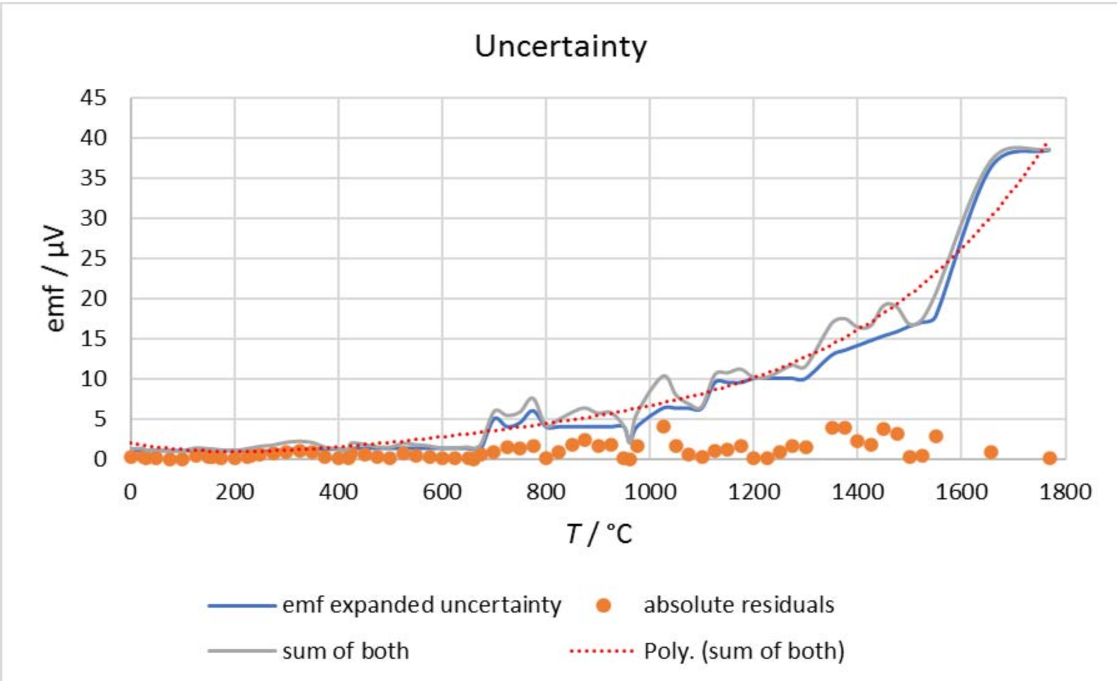

Figure 5: Uncertainty of the reference function.

A number of trials of the Pt-40%Rh vs. Pt-6%Rh thermocouples in industrial conditions are currently underway e.g. float glass manufacturing to 1550 °C in Denmark and quartz glass manufacturing in the UK. PTB are leading trials in industrial furnace manufacturing at MUT, and CMI will lead trials in a steel manufacturing facility to demonstrate the achieved stability of the thermocouples and the validity of the new emf-temperature reference function.

3.2.2 Task 2.2 Optimisation of the stability of double-walled MI thermocouples up to 1200 °C

The aim of this task is to optimise the design of double-walled MI thermocouples and to establish the basis for the standardisation of the double-walled MI thermocouples of types K and N. Comparison tests will be executed using double-walled MI thermocouples to find an optimal inner to outer wall thickness ratio. 60 further double-walled MI thermocouples, with the most promising inner to outer wall ratio, will be constructed and investigated in comparison with conventional MI thermocouples of the same type. The results will provide the basis for approaching standardisation bodies which will open up the opportunity to use the sensors widely in industrial applications.

Firstly, CCPI, with UCAM's cooperation, will manufacture a cohort of double-walled MI thermocouples with 3 different inner to outer wall thickness ratios, including types K and N, with Inconel 600 sheaths and an outer diameter of 3 mm. NPL and UL will manufacture two Fe-C high temperature fixed point cells (1153 °C) to be used for drift testing. A series of drift tests on a wide selection of the cohort will be performed by PTB, CEM, CMI, NPL, TUBITAK and UL. Furthermore the influence of different electrical and magnetic fields on the operation of the double-walled MI thermocouples will be investigated by UL on at least two thermocouples, simulating everyday industrial use.

Progress: All the Type N and Type K cables (conventional, dual wall 'standard geometry' and alternative geometry) have been manufactured. The conventional thermocouples and dual wall 'standard geometry' thermocouples of Type N have been distributed to the partners for isothermal drift tests at 1200 °C (3 partners) and for thermal cycling tests between 300 °C and 1150 °C (3 partners).

Tests on Type N thermocouples using a Fe-C fixed point cell were undertaken before and after a drift test in thermal cyclic condition at 1150 °C for conventional thermocouples and dual wall thermocouples (both standard and alternative geometries). Further drift tests are currently running on conventional and dual wall (standard geometry) Type K thermocouples in isothermal conditions at 1150 °C and 1200 °C which resulted in total number of hours of tests (both Type N and Type K thermocouples) of more than 1100 hours.

The comparison measurements using Type N thermocouples by applying isothermal drift tests at 1200 °C and by applying tests under thermal cycling conditions (300 °C to 1150 °C) according to the agreed protocol have been completed. The same investigation and tests using the Type K thermocouples have been completed too or will be completed shortly at the remaining NMIs.

3.2.3 Task 2.3 Assessment of the insulation resistance breakdown of MI thermocouples up to 1200 °C

The aim of this task is to investigate the issue of insulation breakdown for MI thermocouples and to perform measurements of the insulation leakage of double-walled and conventional MI thermocouples of types K and N. There is evidence that, under particular conditions, conventional thermocouples experience insulation resistance breakdown at high temperatures. With operation at higher temperatures, less insulation between the thermoelements, and for the longer times that double-walled thermocouples allow, it is envisaged that the insulation material in the double walled thermocouples could experience resistance breakdown even more frequently. Measurement schemes will be devised to improve the characterisation of this effect by UCAM, CCPI, NPL and CEM.

Progress: A cohort of 12 thermocouples (6 Type K and 6 Type N) from two manufacturers but with three different diameters (1 mm, 2 mm, 3 mm) each are now being investigated. Furthermore, the upper temperature limit of the investigation was increased to 1250 °C. A jointly agreed measurement setup and a measurement protocol with a two-stage measurement procedure were discussed and agreed among the partners involved. Some measurements are now complete, and these will be analysed to gain some insight into the effect on measurement uncertainty.

3.2.4 IEC 61515

A key task is to provide evidence to the IEC committee TC 65/SC 65B/WG5 responsible for the MI thermocouple standard IEC 61515 [11] that the stability of double-walled MI thermocouples is superior to that of conventional single-walled thermocouples. A further difficulty with compliance of the double-walled MI cables with the IEC 61515 standard is the reduced cross-sectional area within the thermocouple due to the thicker wall. This means there is less space for the thermoelements which impedes compliance with the geometrical specifications. Evidence of superior stability, even with reduced thermoelement size and spacing, is needed before modifications to the geometrical requirements of IEC 61515 can be proposed.

3.3 WP3: Demonstration of a validated in-situ combustion reference standard

In EMPRESS a portable standard flame was developed. By having a very well characterised gas composition and a judiciously designed geometry, the flame has a stable, spatially uniform temperature profile, and can be transported to end-users' sites to validate flame and combustion thermometry apparatus [33].

The flame system has been fully commissioned and is available as a measurement service to external customers. Using laser Rayleigh scattering, it has been possible to determine the post-flame temperature with an uncertainty of less than 0.5 % of temperature – this is a factor of two less than the original target uncertainty of 1 % of temperature. The flame and laser interrogation system are shown in Figure 6. Additionally, the system can provide a number of fixed and reproducible temperatures and species concentrations for propane/air

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equivalence ratios from ϕ = 0.8 (lean flame) to ϕ = 1.4 (rich flame). The range of (fixed) highly uniform temperatures that can be attained (dependent on ϕ) is between 2050 K and 2250 K. This provides a robust mechanism to not only validate third party optical techniques but also assess their linearity. Measurements made by UC3M/CEM using a hyperspectral imager show excellent agreement with the standard portable flame (Figure 7), validating the technique and leading the way for development of a low-cost instrument in EMPRESS2. Measurements made by DTU using IR/UV spectroscopy are equally impressive, showing outstanding agreement with the NPL measurements. Additionally, comparison of the measured and modelled IR emission spectra by DTU allowed further improvement of the temperature profile characterisation performed by NPL. This demonstrates the value of collaborating on this type of activity. Figure 7 also shows the reproducibility of the flame temperature as measured after each journey to the partners' laboratories. Figure 8 shows the FTIR hyperspectral imaging system results at UC3M, which yields both temperature and density of individual species.

The idea of using the NPL portable standard flame is 1) to have a high temperature calibration source which can be used for absolute calibration of e.g. thermal imaging or sweeping systems and 2) to have a tool for optimisation and fine tuning of the above mentioned systems, so uncertainties in temperature retrievals with use of the standard flame are less than 5 %. In general, by providing an ideal, well characterised, uniform region of temperature, given by the standard flame, it is possible to calibrate other industrial combustion thermometers (e.g. pyrometers) under similar conditions.

Practical flames of end users (e.g. an industrial scale combustion facility) usually have high temporal and spatial variations in temperature (and therefore in gas emissivity) because of e.g. turbulence, poor gas/fuel mixing and flame source (burner) instabilities. Therefore, retrieved gas temperature will usually show high temporal fluctuations if the measurements are taken with sufficient time resolution (e.g. a few seconds). Those fluctuations are significantly higher than 5 % uncertainty in the standard flame measurements and reflect the nature of the real process. Knowing that optimized thermal imaging or sweeping systems can guarantee overall uncertainty in the gas temperature retrievals of 5 % or less, all above mentioned temperature fluctuations can be entirely attributed to the process and not to the measurements, the system used, or the data processing. This will provide a valuable tool for end users because they can rely on the given time-dependent temperature data. Temperature fluctuations above 5 % will show how the real flame differs from the standard flame.

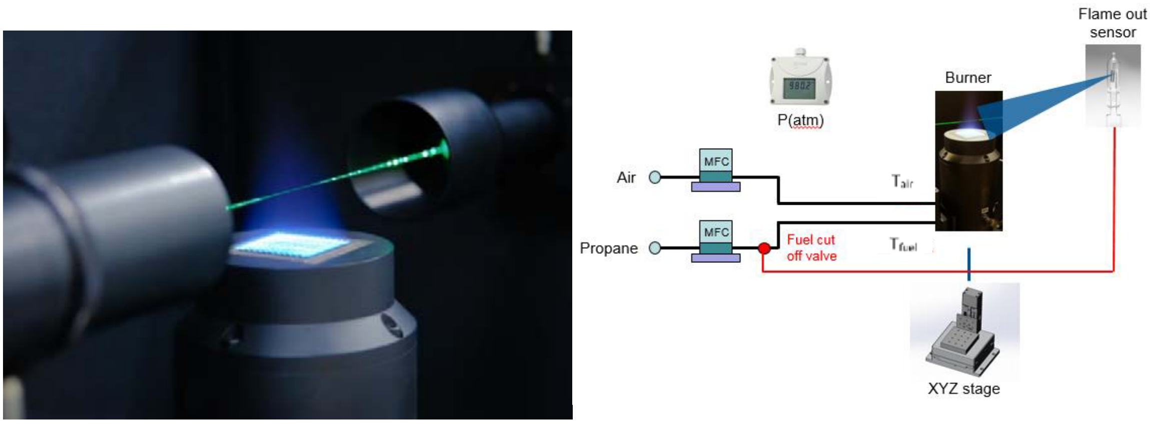

Figure 6. Left: Portable standard flame being interrogated by the laser Rayleigh scattering apparatus. Right: The gas metering and burner control system.

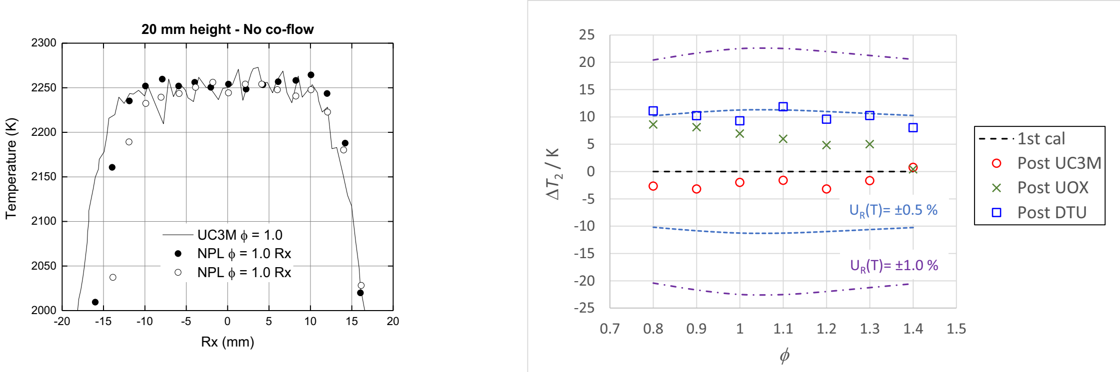

Figure 7. Left: Comparison of the spatial flame temperatures measured by NPL (Rayleigh scattering) and UC3M/CEM (hyperspectral imaging). Right: Shift in flame temperature as a function of ϕ following measurement campaigns at UC3M, UOXF and DTU.

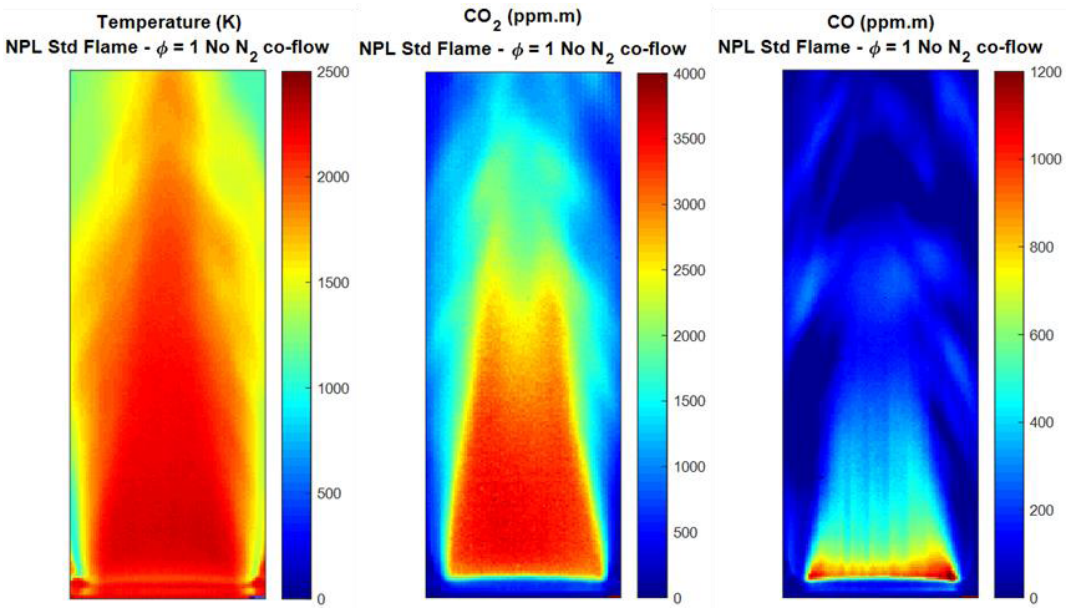

Figure 8. Temperature (left) and column density maps (middle – CO_2, right – CO) for the standard flame under stoichiometric conditions (ϕ = 1.0), measured with the FTIR hyperspectral imaging apparatus.

In EMPRESS2, the flame will be used to facilitate the development of two new practical flame thermometers. The first, a low-cost (i.e. thousands of euros rather than tens of thousands) multi-spectral imaging system for flames, will be developed and calibrated against the standard flame, then demonstrated in industrial applications e.g. fire resistance testing and natural gas production. The second device is an infrared on-sight/sweeping IR emission measurement system that can measure the 2D temperature profiles in flames or hot flue gases. This will then be calibrated against the standard flame, and demonstrated in a NO_x selective non-catalytic reduction process. There are three key tasks:

3.3.1 Task 3.1 Supply and coordinate circulation, and the calibration of a portable standard flame

The aim of this task is to supply the partners (DTU and UC3M) with NPL's (portable) flame reference standard for use in their facilities. Before and after each journey away from NPL the flame is checked and re-calibrated using the Rayleigh scattering thermometry system [27] to ensure consistent performance.

Progress: The flame flowmeters and reference platinum resistance thermometer (PRT) have been re-calibrated successfully. No evidence of drift outside the uncertainty of calibration was found. The standard flame system, as a whole, has also been re-calibrated with the NPL Rayleigh scattering thermometry system for propane/air equivalence ratios of 0.8 to 1.4, in steps of 0.1. The measured temperatures were all within the standard uncertainty of 0.5 %. This process was repeated prior to each deployment of the NPL portable standard flame at partners' facilities.

3.3.2 Task 3.2 Development of a thermal imaging system

The aim of this task is to develop a multispectral thermal imaging system that is suitable for temperature measurements in various industrial applications with overall uncertainties better than 0.5 %. The system will use a multi-filter based concept incorporated with an IR imaging camera.

DTU and UC3M will take a low spectral resolution (i.e. wavenumber resolution of 1 cm^{-1}) approach for temperature measurements. High resolution experimental IR emission spectra obtained on the portable standard flame will be used to simulate of low-resolution (band-) spectra, in order to investigate the influence of spectral resolution on the accuracy of the temperature calculated from the IR emission spectra. Additionally, the optimal centre wavelengths and spectral bandwidths for use in the temperature measurement of flames will be defined.

Progress: CEM have calibrated at least two of UC3M's IR cameras at CEM using a range of at least two different blackbodies up to 3300 K to provide traceability for UC3M's measurements in industrial applications.

The low spectral resolution approach for temperature measurements has been tested. Interferograms obtained on the NPL standard flame in the EMPRESS project [16] have been cropped around the zero path difference point in order to simulate low resolution spectra and to retrieve temperature values from them. On a central point about 20 mm above the burner, the measurement accuracy is quite good for resolutions of 1 cm^{-1} and 2 cm^{-1} (around 1 %). Theoretical emission spectra have been generated at high spectral resolution (0.5 cm^{-1}) for a variety of temperatures (T) and CO_2 column densities (Q) in the medium infrared region. Spectra have been integrated over bands of different spectral widths to simulate measurements from a multispectral system.

Quadratic functions have been used to fit T and Q as a function of the integrated radiances, and accuracy has been assessed for different numbers of bands and centre wavelengths. Fitting has been performed using principal components as independent variables, because they provide the optimal use of information and make it possible to compare also results based on high resolution spectra. It has been found that centre wavelengths are not critical as long as they cover the emission band of CO_2, and that the most influential factor in the accuracy of temperature recovery is the range of temperatures used to train the fitting function. For six bands in the region from 4 μm to 5 μm, the root mean square value for T is less than 1 K if the training range is between 1500 K and 2000 K. However, if it extends from 500 to 2300 K, root mean square values are larger than 30 K, so an alternative approach was sought.

Two of the originally selected band-pass filters were subsequently replaced with new ones and the system was calibrated with an in-house blackbody taking into account CO_2 in the atmosphere (about 400 ppm) which absorbs part of the radiation emitted by the flame. The filters provided satisfactory measurements, so the filter set and analysis procedure was considered to be optimised.

The multispectral imaging system with the new filter set has been tested using the NPL portable standard flame. A multispectral measurement consists of a vector of six values representing six incident radiances defined by each filter. A radiance spectrum incident on the multispectral system can be calculated knowing the flame temperature, CO_2 density column in the flame, CO_2 concentration in the ambient air, and the distance between the measuring system and the flame [36,38]. A simulated multispectral measurement vector of the measuring system is obtained using calculated radiance spectra and the transmittances of the six filters.

In order to estimate temperature and column density in the flame, a set of simulated multispectral measurement vectors is generated for a range of temperatures and CO_2 column densities in the NPL portable standard flame. The absolute error between the measured spectra and simulated multispectral measurement vectors is calculated for the whole set. The values of the temperature and density column that yield the smallest error are considered to be the retrieved values. The use of a fitting function, investigated in the earlier work, was abandoned.

Preliminary results look promising [31]: an agreement of better than 5 % was seen between temperatures retrieved from the multispectral measurements and those obtained with the hyperspectral approach using an

FTIR spectrometer that was developed in the first EMPRESS project. However, work is ongoing to improve the consistency of the measurements over the whole temperature range from 450 °C to 1150 °C.

3.3.3 Task 3.3 Development of a sweeping emission measurement system

The aim of this task is to develop a FTIR-based system prototype for on-sight sweeping IR emission measurements. An optimal temperature profile retrieval algorithm will be selected from low/high-resolution FTIR emission spectra using e.g. line-by-line models or the selected bands approach. The system performance and temperature retrieval algorithm will be investigated using the portable standard flame, targeting an overall uncertainty of 0.5 %. The sweeping system will be used for measurements of 2D temperature profiles in the first pass (900 °C to 1100 °C) of a waste incinerator. The results will be used for NOx selective non-catalytic reduction (SNRC) process optimisation and for the validation of CFD modelling by the end-user, B&W Vølund.

DTU and UC3M will select an optimal temperature profile retrieval algorithm from the high/low resolution IR emission measurements developed above. DTU will develop an FTIR on-sight sweeping/emission measurement system prototype for 2D profiles using the portable standard flame as a reference. DTU will test and calibrate the FTIR system against the portable standard flame. DTU will perform in-situ, on-sight, 2D temperature profile measurements for the optimisation of NOx SNCR processes at B&W Vølund, and for the validation of the CFD modelling (of B&W Vølund) of a waste incinerator.

Progress: An existing fast IR CEDIP camera with an InSb detector array has been serviced and upgraded for faster data acquisition. Two new IR gratings optimised in the 1800 cm^{-1} to 2900 cm^{-1} and 2800 cm^{-1} to 4500 cm^{-1} spectral ranges have been ordered and purchased. The camera was tested in combination with a small IR-grating spectrometer (with two new gratings) on the NPL portable standard flame and the results were compared with those obtained in the EMPRESS project with the high-end FTIR spectrometer. Excellent agreement in the overall spectra measurements was found. It was concluded that a more compact grating spectrometer with the CEDIP camera system is a good alternative to the FTIR-based one, giving good spectral coverage, fast acquisition time and having no moving parts. It has been shown that a machine learning approach to retrieve the gas temperature for normal and lean combustion cases (which are of most interest for practical applications) yields accuracies of 0.23 % and 0.21 % respectively [30].

DTU and B&W Vølund, with support from the Filborna WeT plant (Swedish waste incineration plant) have made preliminary measurements using a high-end FTIR spectrometer and a manually driven sweeping probe at the Filborna WeT plant. The measurements were made along a path close to the NH_3 injection[1] point (cross-section 10 m by 4.8 m, temperature about 950 °C). An existing access port was used. The measurements have been made at various line of sight angles including -90°, -45°, 0°, 45° and 90° (0° is the horizontal cross-stack direction, 90° is parallel to the vertical wall). Measurements were made at high and low plant operation loads. Two spectral resolutions (2 cm^{-1} and 8 cm^{-1}) with the fast scanning FTIR mode (100 kHz) were used for IR emission measurements in the spectral range 1000 cm^{-1} to 6000 cm^{-1}. The system (FTIR spectrometer and probe) was calibrated using a calibrated portable blackbody at 875 °C. The sweeping measurements were supplied by on-site (permanently installed) thermocouples and IR pyrometers.

The results showed variations in the effective gas/particle temperatures for different lines of sight. Gas IR emission spectra consist primarily of CO_2 and H_2O emission bands that make data analysis possible with the use of the latest version of the HITEMP spectral database [29]. Modelling of H_2O spectral emission (1000 cm^{-1} to 6000 cm^{-1}) for various temperature profiles and over long distances show that effective H_2O emissivity and temperature depend on the temperature profile uniformity. Thus a constant temperature profile gives exactly the same emissivity (i.e. 1) and temperature. Conversely, a non-uniform temperature profile gives lower effective emissivity (<1) and temperature. The effective emissivity of H_2O can be used to evaluate the uniformity of the temperature profile. Another conclusion is that low spectral resolution (from 8 cm^{-1} and lower) and high sampling rate are preferable because of turbulence effects in the gas, especially at low plant loading.

During a service stop in the summer of 2019, B&W Vølund modified two existing access ports for cross-stack measurements at the Filborna WeT plant. These ports enable simultaneous IR emission measurements with the sweeping system and cross-stack temperature profile mapping. A new measurement campaign at Filborna WeT plant has been completed in August 2021 and analysis of the acquired data is ongoing. Figure 9 shows simultaneous line-of-sight fast IR emission measurements with use of the FTIR spectrometer and IR

[1] Part of the selective non-catalytic reduction process employed by the plant.

grating spectrometer equipped with the CEDIP IR camera. Figure 10 shows visible images of the highly non-uniform hot gas taken an interval of 4 s apart from the right side of the boiler. The patterns are quite stable over that short period of time.

Figure 9. Parallel fast IR emission measurements with IR spectrometer and CEDIP IR camera (left panel: left boiler side) and FTIR spectrometer with sweeping probe (right panel: right boiler side). Both systems are viewing the same axis.

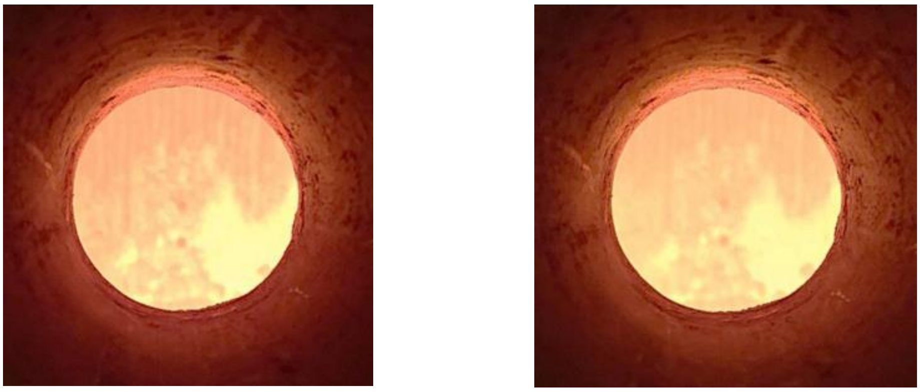

Figure 10. Two visible photographs of hot gas from the right side of the boiler (FTIR spectrometer side). The two photos are taken 4 s apart. Two darker dots near the centre are access ports on the opposite side of the boiler. The gas is highly non-uniform in temperature, although there is no significant pattern change in the field of view over a duration of 4 s.

3.4 WP4: Traceable fibre-optic thermometry

Completely new to EMPRESS2, a suite of new fibre-optic techniques will be developed with the aim of making traceable temperature measurements in harsh environments possible. Phosphor-based fibre-optic thermometry will be developed using a traceably calibrated phosphor to more than 600 °C using a new type of coating for the fibre. For ionising radiation environments, a hollow-core fibre-optic thermometer will be developed, which is suitable for high gamma radiation environments, as the hollow geometry renders the fibre potentially immune to the darkening effects. A distributed fibre-optic temperature sensor will be developed, alongside a traceable calibration method. Its performance will be investigated with a range of coatings. For high temperatures, a hybrid thermometer based on a Fibre-Bragg Grating (FBG) at the tip of the sapphire fibre and a blackbody will be developed which exploits the redundancy of the two complementary measurements, and which can be traceably calibrated. There are four key tasks:

3.4.1 Task 4.1 Development of traceable phosphor-based contact thermometers to 650 °C

The aim of this task is to develop, calibrate and test novel phosphor tipped fibre-optic thermometers up to 650 °C that are immune to electromagnetic interference. This will include field trials in suitably harsh environments to demonstrate their performance. NPL and DTI will each develop a phosphor-based fibre-optic thermometer to 650 °C and cross-validate. They will then jointly establish traceable calibration methods for the new fibre optic

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thermometers using fixed points and liquid baths from room temperature to 650 °C. The thermometers will then be trialled in a plasma storm of charged particles at DTI and a large magnetic field e.g. 1 Tesla (at a collaborator's site e.g. Danfysik A/S).

Progress: NPL have selected a phosphor (Mg_4FGeO_6:Mn) and binder (Ceramabind 643-1) and suitable high temperature gold coated fibres have been identified. The thermometer has been built with the MFG:Mn phosphor powder encapsulated in a ceramic tube that is bonded to the cleaved end of the fibre (Figure 11). The instrumentation has a blue (420 nm) LED coupled to the fibre that excites the phosphor. A dichroic beam splitter separates the emitted radiance from the excitation and the emitted radiance is further divided into wavelength bands at 630 nm and 660 nm, again with a dichroic beam splitter. The signal at each wavelength band is detected with a new design of high sensitivity photodiodes (Hamamatsu S2387 1010) with NPL designed and buit transimpedance amplifiers that have gain between 107 and 109 V/A. A National Instruments USB-6356 data acquisition unit records the amplified signals. The ratio of the signals at the two wavelengths is temperature dependent and the system has been tested from -90 °C to 660 °C. Below room temperature a stirred bath was used with its temperature given by a calibrated 25 Ω standard platinum resistance thermometer and F700 resistance bridge, and above room temperature a dry block calibrator with its temperature given by a calibrated Type R thermocouple. The effect of signal excitation level, a know influencing factor, has been assessed as well as other uncertainty contributions. The target of 5 °C up to 500 °C has been achieved, but there are issues with step changes that may be due to physical movement between the fibre and the potted phosphor powder associated with temperature cycling. Now the technique has been proven, a more robust thermometer is being manufactured.

Figure 11. Fibre-optic 'pig-tail' traceably calibrated phosphor thermometer. Excitation (blue light) yields phosphor emission (red light), giving a purple appearance.

DTI has established the hardware and software for a fibre-optic based phosphor thermometer based on the lifetime method up to approximately 400 °C. Two separate aluminium-coated optical fibres are used in the thermometer, one to transmit light from the 415 nm LED to the phosphor and the other to collect light for the lifetime determination. There are challenges related to the degradation of the phosphor at higher temperatures, which result in a smaller signal. This degradation is a challenge for the lifetime method and may limit the maximum temperature and long-term calibration drift performance. The calibration of the finished sensor and optical system is ongoing.

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3.4.2 Task 4.2 Development of novel fibre-based thermometers for harsh environments

The aim of this task is to develop and test two novel fibre-optic based thermometers. The first, incorporating hollow core fibres will operate over a modest temperature range (20 °C to 150 °C) but it will have the potential for immunity to high gamma fluxes. The second, comprising a flexible, ordered fibre bundle, will provide a thermal imaging capability for the remote inspection of hard to reach hostile environments. Both instruments will be tested in harsh environments.

Progress: UoS has investigated various types of hollow-core optical fibres (which offer higher immunity to gamma fluxes) for a phosphor-tipped fibre-optic thermometer, and have concluded that for phosphor excitation at 420 nm and emission at 660 nm, antiresonant-type fibres are the most promising options. The new design is able to deliver the excitation signal to the phosphor at the distal end and collect and guide the emitted signal back to the proximal end for analysis. Two types of anti-resonant hollow-core fibre samples, the 'tubular' and 'nested antiresonant' fibre have been fabricated and a significant effort deployed to interface them with standard optical components.

NPL has assessed these fibres for gamma radiation immunity in NPL's irradiation facility. The transmission losses at 420 nm and 630 nm were approximately 16 dB/m and 12.3 dB/m respectively. The change in transmission following an exposure of 400 Gy was less than 2 %. The change in transmission ratio of two 20 nm bands at 630 nm and 661 nm (potential device specification) was less than 1 %. This demonstrates that it should be possible to operate an intensity ratio phosphor thermometer with high gamma immunity using hollow-core fibres. However, UoS identified excitation of optical modes guided in the glass cladding a potential issue and have redesigned both fibre and interconnection scheme to further reduce this by one to two orders. A dedicated setup for characterising hollow-core fibres in the visible region (400 nm to 700 nm) where phosphors operate has been built at UoS to investigate further. For reliable operation, an 'all-fibre' approach is being taken, with all free space components replaced by fibre equivalents which are spliced together. This will guarantee high efficiency coupling of lasers at the excitation and emission wavelengths. In addition, optimum hollow-core fibre designs for operation at the desired wavelengths have been investigated, identifying the optimum thicknesses in the anti-resonant fibre design to allow low-loss guidance of both excitation and emission wavelength. The theoretical work is now focusing on modifying the structure of the fibre to maximise the collection efficiency when the phosphor is attached to the distal end of the fibre for temperature measurements.

3.4.3 Task 4.3 Development of distributed temperature sensors using Brillouin scattering up to 650 °C

The aim of this task is to develop a fibre-optic distributed temperature sensor (DTS) using Brillouin scattering for use in harsh environments, namely stainless steel manufacturing up to 650 °C, and to evaluate its performance.

CEM will assess the facilities of ACERINOX in El Campo de Gibraltar and will decide on the most suitable locations to test the fibre-optic DTS. At least one fibre-optic DTS will be employed, with variants having different coatings of aluminium and copper to suit specific applications. These will be optically characterised to assess the Brillouin frequency shifts, ageing, and attenuation of at least two different single-mode optical fibres. The response of the two different fibre-optic coatings to a range of different temperatures up to 650 °C will also be assessed. CEM will perform the calibration of at least three of the differently coated fibre-optic DTS up to 650 °C. Finally, ACERINOX will deploy the fibre-optic DTS to compare their measurements with those sensors used in their routine measurements.

Progress: The design of the fibre-optic DTS based on Brillouin scattering is completed, and the fibre-optics have been procured. The system has been traceably calibrated up to 600 °C. Analysis of the measurements is ongoing.

3.4.4 Task 4.4 Deployment of hybrid fibre-optic based high temperature sensors to 1500 °C

The aim of this task is to develop novel fibre-optic based temperature sensors that will be capable of operation up to 1500 °C and to demonstrate that, with field trials in three extreme industrial environments. Specifically, Fibre-Bragg Grating (FBG) thermometers offer great potential for high temperature applications. However, silica-based fibres are currently limited to around 1000 °C and they are prone to grating degradation at higher temperatures. This task will develop Bragg and blackbody cavity fibre-optic sensors based on sapphire fibres with the aim of combining them in a single hybrid sensor. By doing this, confidence in the measurement will be greatly improved as the measurement uncertainty is expected to be halved.

IPHT will develop optoelectronics and signal processing instrumentation and prepare sapphire FBGs in test fibres. PTB will develop signal processing algorithms for the temperature dependence of the asymmetric resonance peak of the FBGs in relation to the stability, repeatability and resolution of the measurements. JV will advise on the compatibility of the sapphire FBG with the blackbody part of the sensor. The output will be a functioning sapphire FBG with associated read-out instrumentation.

Progress: IPHT and PTB have agreed on the design of the sapphire FBG thermometers. PTB has designed, purchased and assembled a measurement setup for the high resolution spectral analysis of FBGs, which is based on a tuneable laser source. The software integration of all components is complete, and the characterisation of the thermometers provided by IPHT is underway.

IPHT have developed inscribed Fibre Bragg gratings with an operating wavelength of about 1540 nm in several fibres with a length >80 cm, so that the probe will fit into the calibration furnaces at PTB. IPHT packaged sapphire fibres with protection tubes and conventional optical fibres, and in the first series of measurements no ageing effects were observed. Different signal processing algorithms for the asymmetric peak detection were investigated on a prototype device. An ITS-90 traceable calibration of a FBG thermometer based on sapphire fibre will be performed up to 1500 °C at PTB.

IPHT have also established a test facility to provide temperature environments up to 1500 °C. The effect of self-radiation on the blackbody radiation was investigated. IPHT recorded spectrally resolved data in immersion tests in a furnace. Analysis performed by JV and IPHT showed a substantial self-radiation issue. A possible mitigation strategy is to use multiple wavelengths to deduce the temperature, but it is likely that substantial scattering on the sapphire surface also contributes to the signal, and this requires a more sophisticated model, which is under development.

PTB has advanced the laser-based setup with SI traceable gas cells to provide a wavelength reference. Several datasets on the spectral reflection coefficient at different temperatures have been acquired, as measured using the tuneable diode laser setup. The data shows interference effects from the multimode transmission in the sapphire fibre. While the interference patterns are stable and repeatable, they pose a signal processing challenge and may limit the resolution of the FBG thermometer; the development of mitigation approaches is ongoing.

The hybrid thermometer

These two techniques (FBG and blackbody cavity) will then be merged to create a hybrid thermometer. IPHT will adapt signal/data processing techniques to improve robustness against spurious effects e.g. thermal expansion, and will fabricate a sapphire hybrid thermometer packaged for laboratory calibration. JV, PTB and IPHT will investigate the effect of strain and thermal expansion on the measured signal, and will adapt signal processing with respect to fibre self-radiation and sensor position.

Following the manufacture, testing and validation will be performed to identify robust ways to merge the data from the two sources. Thermal cycling between room temperature and 1500 °C will be performed to assess the stability, and a validation with ITS-90 fixed points will be carried out over this temperature range. The hybrid sensor will be demonstrated in harsh industrial environments in the NPL gamma-ray facility, in silicon processing at Elkem, and in industrial furnaces at MUT.

4. Conclusion

A new European project, EMPRESS2, has been described, and the progress in the first quarter (nine months) presented. The aim is to enhance the efficiency of high value manufacturing and industrial processes by improving temperature measurement and control capability. This project seeks to address four contemporary thermometry challenges in this sector, and new developments from this and its predecessor project, EMPRESS, have been described. The key developments are in the areas of phosphor thermometry for surface temperature measurement, characterisation and improvement of ultra-stable thermocouples, application of a portable standard flame for developing new commercial flame imaging and spectroscopy-based instrumentation, and a suite of new fibre optic thermometers. The theme running through all the activities is the establishment of in-process traceability to the ITS-90, and the demonstration of the new thermometry techniques in-process by application to contemporary process control challenges.

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