Traceable thermometry for high value manufacturing: some case studies

Jonathan Pearce
Introduction

- Better efficiency implies better temperature control
- Better stability, lower uncertainty
- Traceability to the SI

EMPRESS 2
- Phosphor thermometry
- Thermocouples
- Combustion/flame thermometry
- Fibre-optic thermometry
- Practical Johnson noise thermometry
IS THERE A REPRODUCIBILITY CRISIS?

A Nature survey lifts the lid on how researchers view the 'crisis' rocking science and what they think will help.

BY MONTA BAKER

52% Yes, a significant crisis
38% Yes, a slight crisis
7% Don’t know
3% No, there is no crisis

1,476 RESEARCHERS SURVEYED

HAVE YOU FAILED TO REPRODUCE AN EXPERIMENT?

Most scientists have experienced failure to reproduce results.

- Someone else’s
- My own

Chemistry
Biology
Physics and engineering
Medicine
Earth and environment
Other

Metrology is key to reproducing results

Scientists of all stripes must work with measurement experts so that studies can be compared, urge Martyn Sené, Ian Gilmore and Jan-Theo Broersen.

Imagine you are a policy maker who needs to know how much carbon is stored in the South American forest. On the ground data in this area are thin. So when you cross two recently published maps of surface biomass, both made using the same satellite data, you think it’s your lucky day. Unfortunately, these maps differ in their estimates of biomass by about 20% across the continent, and by even more on a local level. Which map, if either, can you trust?

Many column inches have been dedicated to discussing this 'reproducibility crisis' in scientific research. Researchers are新たにincentiviated to try to replicate results, and when they do, these results often don’t match.

Little attention has been paid to these discussions on how metrology can help.

Metrology is the science of measurement: practices to develop internationally agreed reference points so that measures — of anything from length or mass to radiation — can be compared worldwide.

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I measured the temperature of the antenna to be 148.4 K. Is that correct? Wasn't it 168 K on NPL-SATI?

We can only answer questions like this if we can trace the measurement back to universal reference standards.

In the case of temperature, this means traceability to the SI unit, the kelvin.
EMPRESS 2

- Phosphor thermometry
- Thermocouples
- Combustion/flame thermometry
- Fibre-optic thermometry

Practical Johnson noise thermometry
EMPRESS 2

- Solve a suite of specific, documented process control problems in high value manufacturing
- By establishing in-process traceability to ITS-90

- WP1: Surface temperature: phosphor thermometry (STRATH)  Extend to 2D
  Extend to 1000 °C
  Combine with thermal imaging

- WP2: Standardising high stability thermocouples (PTB)
  Towards standardising Pt-40%Rh vs. Pt-6%Rh

- WP3: Combustion thermometry (DTU)
  Demonstrating its use in-process

- WP4: Harsh environments: fibre-optics (NPL)
  Completely new – introducing traceability

- WP5: Impact (AFRC)
Activity & specialism groupings

**Phosphor thermometry (decay-time)**
- Phosphor thermometry development
- Tribology
- Manufacture of brake pads

**Phosphor thermometry (intensity ratio)**
- Phosphor thermometry development, traceable calibration techniques
- Development of phosphor thermometer for online/offline monitoring; AFRC provide access to industrial processes
- Development of phosphor thermometer
- Provide access to marine manufacturing for trials

**Fibre-optic thermometry (hybrid BB/FBG)**
- Development of laser and fibre-optic technologies
- Development of sapphire based sensors; traceable calibration techniques
- Development of FBG fibre-optic sensors
- Provide access to industrial furnace manufacturing for trials
- Provide access to silicon processing for trials

**Fibre-optic thermometry (hollow-core/bundles)**
- Optical fibre development, instrumentation
- Development of traceable calibration techniques

**Fibre-optic thermometry (distributed)**
- Development, manufacture and testing of DTS fibre-optic thermometers
- Supply of stainless steel
- Development of traceable calibration techniques

**Combustion thermometry**
- Supply, calibrate portable standard flame
- Develop IR, UV spectroscopy
- Provide access to waste incineration facilities for trials
- Development of IR imaging devices
- Development of optics and IR instrumentation

**Thermocouple thermometry**
- Traceable calibration facilities
- Supply of thermocouple wire
• 11 NMIs
• 4 universities
• 11 companies (6 unfunded)
• 67 letters of support
• 142 members of the stakeholder community
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- Phosphor thermometry
- Thermocouples
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- Fibre-optic thermometry

Practical Johnson noise thermometry
“Impossible” reliable surface temperatures

- Contact sensors, slow, extract heat from surface, time consuming, also have large unquantified errors $>> 10$ °C
- Radiance based methods, emissivity, reflected radiation can lead to large unquantified errors $>> 10$ °C
Non-contact non-radiance traceable surface thermometry

- Apply specific phosphor to surface and activate
- Either – decay time of emitted light
- Or – 2 line ratio method
**WP1 Phosphor thermometry**

**NPL**
- Develop traceable 2D intensity ratio phosphor thermometer

**NPL**
- Combine with thermal imaging

**NPL, STRAT**
- Determine emissivity of test samples with spot pyrometer, compare with phosphor thermometer

**NPL, STRAT**
- Testing of phosphor applied to billets during heat treatment

**NPL, STRAT**
- Testing of phosphor applied to welding applications

**NPL**
- Testing of phosphor applied to welding applications

**NPL, DTI, AGH**
- Testing of phosphor applied to welding applications

**NPL, INRIM**
- Validate at NPL, or locally, using either
  - ITS-90 fixed points
  - Comparison calibration
  - Phosphor thermometer-based surface calibrator

**STRAF, NPL, DTI**
- Develop phosphor thermometry for temperature mapping of forging tool

**INRIM, ITT, CNR**
- Develop phosphor thermometer to 1000 °C in conjunction with on-site fibre optic probing and sensing

**INPUT FROM WP4**

**DTI**
- Revise EURAMET best practice guide on surface temperature measurement (developed in EMPRESS) to include phosphor thermometry techniques

**Sensors available for exploitation**
- 2D intensity ratio phosphor thermometer to 1000 °C
- Combined intensity ratio phosphor thermometer/thermography system to 1000 °C
Phosphor thermometry

- Meet standards for pre- and post-welding heat treatment
- BS EN 13445, ASME VIII, PD5500, ISO 15614-1
- ISO 8502-4:2000 for coating


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- Phosphor thermometry
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WP2 Standardising high stability thermocouples

**Standardisation of Pt-40%Rh/Pt-6%Rh thermocouple**

- **PTB, CCPI, NPL, CEM, CMI, DTI, TUBITAK, UL, JM**
- Construct thermocouples according to agreed procedure, and perform calibration with all available facilities
- UL, PTB, NPL, CEM, CMI, DTI and TUBITAK
- Provide draft reference function to IEC committee

- **NPL, DTI (collaborator e.g. Ardagh Glass Holmegaard)**
- Trial in e.g. glass manufacturing to 1700 °C as a demonstration of achieved stability, and of the reference function
- **PTB, MUT**
- Trial the Pt-Rh thermocouples in industrial furnace manufacturing
- **UL (collaborator e.g. Kambic)**
- Trial the Pt-Rh thermocouples in industrial furnace manufacturing
- **CMI (collaborator e.g. Trinecke Zelezarny)**
- Trial the Pt-Rh thermocouples in steel manufacturing facility

**Optimisation of double-walled MI thermocouple stability up to 1250 °C**

- **CCPI, UCAM**
- Manufacture and supply double-walled and single-walled MI thermocouples

- **UCAM, CCPI**
- Optimise inner to outer wall thickness ratio with respect to drift rate
- **UCAM, CCPI**
- Metallurgical analysis of selected DW MI cables
- **PTB, CEM, CMI, NPL, TUBITAK, UL**
- Assess stability of double-walled MI cable of selected types (K,N) and cable diameters and compare with conventional cable
- **UL**
- Assess influence of electrical and magnetic fields on operation of the DW MI cables

- **NPL, UCAM, CEM, CCPI**
- Develop technique for quantifying insulation resistance of MI thermocouples as a function of temperature
- **NPL, CEM, UCAM, CCPI**
- Develop mitigation for insulation resistance breakdown of MI thermocouples

- **NPL, PTB, CCPI, CEM, CMI, TUBITAK, UCAM, UL**
- Prepare joint peer-reviewed publication on the performance of DW MI thermocouples.
- Provide evidence to IEC61515 committee that stability, insulation resistance, and time response of DW MI thermocouples are comparable to, or better than, conventional MI thermocouples.
Pt-Rh thermocouples

- Systematic evaluation of stability of a large number of different Pt-Rh thermocouples using multi-wire thermocouple and HTFPs (NPL, PTB)
- Optimum Pt-40%Rh/Pt-6%Rh
- Preliminary reference function (NPL, PTB, CEM, KRISS)
- IEC TC 65/SC 65B/WG5
- EMPRESS 2: 7 European NMI participants
Double-walled MI thermocouples

- Stability
- Optimal ratio of wall thicknesses
- Insulation resistance breakdown
- Lay framework for standards e.g. relax dimensional requirements of IEC 61515:2016, AMS2750E
- Presented to SAE (Nadcap), IEC TC 65/SC/65B/WG5
Self-validating thermocouples

- Even noble metal thermocouples can drift by as much as tens of degrees
- No visible sign in-process of this happening
- Self-validation for in-process calibration/traceability
- Develop miniature fixed points
- Same format as conventional sensors
- Robust

In-situ thermocouple trials
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- Phosphor thermometry
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Practical Johnson noise thermometry
WP3 Combustion thermometry

NPL
Supply and coordinate circulation (and calibration) of portable standard flame

UC3M, DTU
Use high res measured emission spectra to simulate low res spectra; explore relation between spectral resolution and T accuracy; design filters for optimal centre wavelengths and spectral bandwidths

CEM
Calibrate IR cameras to provide traceability of UC3M measurements

UC3M, SENSIA, CEM 3.2
Develop multispectral imaging device and calibrate

UC3M, NPL
Calibrate against NPL standard flame and other flame sources

UC3M, SENSIA
Trials on various flame sources

DTU
Selection of optimal T retrieval algorithm from previous task

DTU, UC3M
Develop FTIR on-sight/sweeping emission measurement system or 2D profiles using portable standard flame as a reference

DTU, NPL
Testing and calibration of developed FTIR against NPL standard flame; target uncertainty 0.5%

DTU, B&W Volund
Perform in-situ 2D temperature profile measurements for optimisation of NO_xSNCR processes, and validation of CFD modelling of a waste incinerator

DTU, CEM, NPL, SENSIA, UC3M, VOLUND
Write papers and trade journal articles to outline findings and demonstrate linkage between portable standard flame & improved process efficiency

Sensors available for exploitation
- Low resolution, economical multispectral imaging flame thermometer
- FTIR sweeping emission flame thermometer system

Develop low-cost thermal imaging system

FTIR on-sight/sweeping
DFWM/LIGS

Hyper-spectral imaging

UV spectrometry

Fully validated portable standard flame
NOx SNCR

- NOx SNCR process: NH3/urea injection optimisation
- Very narrow band of temperatures for optimal NOx reduction
- NOx, CFD and radiative heat transfer modelling

Goals:
- Process optimisation through *in-situ* temperature control
- Improved boiler design, more efficient process

Images: Alex Fateev, DTU
EMPRESS 2
- Phosphor thermometry
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WP4 Harsh environments: fibre-optics

**Phosphor-based fibre optics**
- NPL, DTI: Select and traceably calibrate phosphor to 1000 °C
- NPL, DTI: Develop phosphor-based fibre-optic thermometer to 660 °C

**Hollow-core fibre thermometer**
- SOTON, NPL: Develop hollow core fibre phosphor tipped thermometer (including instrumentation) immune to gamma radiation
- SOTON, NPL: Develop mid-IR thermal imaging fibre bundle for remote inspection to 660 °C suitable for harsh environments e.g. ionising radiation, magnetic fields

**Brillouin scattering DTS thermometer**
- CSIC, CEM, FOCUS: Design and optically characterise the Brillouin Scattering distributed sensor
- CSIC, CEM: Develop calibration procedure and perform system calibration to provide ITS-90 traceability
- CEM, CSIC: Perform in-situ trials of the TDS in the facilities of ACERINOX – stainless steel manufacturing
- JV, PTB, IPHT: Develop metallic or ceramic cavity to create BB for fibre tip
- JV, PTB, NPL, IPHT, MUT, ELKEM: Trial in process at ELKEM, MUT, and NPL’s gamma-ray facility

**Fibre-optic and BB-based thermometer**
- JV, PTB, IPHT: Develop instrumentation on system-wide level (optoelectronics, signal processing) for traceable FBG thermometer based on sapphire fibre to 1500 °C

**Sensors available for exploitation**
- Two phosphor-based fibre-optic thermometers with separately developed instrumentation, cross-validated, to 650 °C
- Hollow-core phosphor-tipped fibre-optic thermometer suited to harsh environments e.g. ionising radiation, magnetic fields to 1000 °C
- Thermal imaging fibre bundle system for remote inspection to 1000 °C
- Distributed fibre-optic temperature sensor based on Brillouin scattering to 650 °C
- Hybrid blackbody/FBG fibre-optic thermometer to 1500 °C

**Also as input to WP1**

- Trial in plasma storm of charged particles and large magnetic fields at collaborator e.g. Danfysik
- Trial in gamma ray environment at NPL or e.g. Sellafield
- Trial in forging/forming process at AFRC
- Trial in forging/forming process at AFRC
Fibre-optic phosphor thermometer

- Regular & hollow-core phosphor tipped
- Traceable calibration
- Hollow core fibre exposed to gamma radiation
Hybrid fibre-optic based sensor to 1500 °C

- Sapphire fibre Bragg grating
- Temperature dependence of spectral reflectivity
- Project objective: Characterisation and ITS-90 traceable calibration up to 1500 °C
- Long-term objective: very precise T measurement between 1600 °C and 1900 °C
- Stephan Krenek (PTB)
Workshop (x2)

~60 delegates
~40 organisations

Next one on 5 May 2020 at AFRC, Glasgow
Speakers from:
• Land Instruments,
• Otto-von-Guericke-Universität Magdeburg
• Tata Steel
• Heraeus Conamic UK
• CCPI Europe
• Metrosol
• Oxsensis
• University of Southampton
• University of Strathclyde
• Danmarks Tekniske Universitet
• Physikalisch-Technische Bundesanstalt
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Johnson noise thermometry

\[ \overline{V_T^2} = 4kTR\Delta f \]

- \( V_T \): Johnson noise voltage
- \( k \): Boltzmann constant
- \( T \): temperature
- \( R \): resistance of sensor
- \( \Delta f \): frequency bandwidth

Same over all frequencies – white noise
History

- Small signal, wide frequency band effect
- Some excellent JNTs out there – but sub-mK accuracy, not practical
- Brixy (Forschungszentrum Jülich) – successful, but slow and not commercialised
- Oak Ridge National Laboratory – didn’t really work (but some really good ideas, and extremely well documented)
- Two main problems:
  - Very small signals
  - Difficult in determining the bandwidth

None have been commercialised

There are currently no other industrial thermometers based on Johnson noise
Background

- Metrosol & NPL
- Proof of concept
- Measure net voltage due to thermal movement of electrons – PRIMARY
- Linked to T through fundamental physics
- Accuracy ± 1 °C
- Doesn’t need calibration
- All things that can change in harsh environments are measured
- So, no calibration drift
- Tiny voltage

\[ V_T^2 = 4kTR\Delta f \]

Prior art: high cost, room sized experiments operating in a screened room.

**Importantly, the temperature measurement is unaffected by changes in the property of the sensor (except the resistance which we can measure simultaneously with the temperature and apply compensation), and is independent of any calibration.**

\( V_T \): Johnson noise voltage
\( k \): Boltzmann constant
\( T \): temperature
\( R \): resistance of sensor
\( \Delta f \): frequency bandwidth
The key problems

- Correlation (amplifiers etc. introduce noise)
- In practice, can't measure bandwidth because frequency response is not rectangular: use Nyquist equation in ratio form (‘substitution’)
- Need a reference
- Need to switch between sense & ref resistor

- Measurement time – statistical effect
- Component non-linearity – frequency dependent attenuation of ‘white’ Johnson noise: need to match bandwidths
- Have to limit bandwidth so that measurement is on flat part of the frequency response
- Limits resistor to 100 Ω
- Limits the size of the Johnson noise signal
- Dependent on condition of cables – the very problem we want to avoid

\[ V_T^2 = 4kTRΔf \]
**The topology: JNT1**

- Replace reference with pseudo-random noise source with calibration tones
- Requires no switching
- No need to match time constant of the two arms (there's only one arm) – better accuracy
- Tolerant of non-flat frequency response, since the two signals experience the same frequency response
- Can operate at much higher resistance (5000 Ω c.f. 100 Ω)
- And much higher bandwidth (1MHz c.f. 100 kHz)
- **Factor of 1000 improvement in signal over previous attempts**

The measurement time of Metrosol JNT1 is about a factor of 20 faster than in previous attempts by others, at developing a practical Johnson noise thermometer.
Early results

- Passed industrial EMC testing
- Radiated Field Immunity test to EN61000-4-3, 10 V m⁻¹ 80-1000 MHz
- By a comfortable margin

This problem has completely stopped previous efforts
- Cabling
- Grounding
- Shielding
- Op-amps
- Full tri-axial probe connections

This level of EMC immunity had not previously been achieved and indeed this was one of the main reasons why previous attempts by others to produce a commercial JNT have not materialised.
Early results

- Standard deviation about 0.241 °C
- Target uncertainty about 1 °C over about 7 seconds
- Excellent EMC compatibility/immunity
- Aim to start commercialising in 2020/21
Towards commercialisation – JNT 2

- More compact
- Superior EMI immunity
- On-board DSP
- FPGAs – available unlocked for specific applications (with suitable IP protection)
- On-board ADC identified

The second, more compact prototype JNT 2 is currently in development (programme runs to Q3 2020) to produce a JNT that is close to commercialisation.

http://www.johnson-noise-thermometer.com
EMPRESS 2

- Phosphor thermometry
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Practical Johnson noise thermometry
Thank you!

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