# **Optimizing Heat Treatment of Gas Turbine Blades with a Co–C Fixed Point for Improved In-service Thermocouples**

**J. V. Pearce · G. Machin · T. Ford · S. Wardle**

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**Abstract** Improvement of energy efficiency of jet aircraft is achieved by operating gas turbine engines at higher temperatures. To facilitate this, gas turbine engine manufacturers are continuously developing new alloys for hot-zone turbine blades that will withstand the increased in-service temperatures. A critical part of the manufacture of these blades is heat treatment to ensure that they attain the necessary metallurgical characteristics. Current heat-treatment temperature-control requirements are at the limit of what is achievable with conventional thermocouple calibrations. A project that will allow thermocouple manufacturer CCPI Europe Ltd. to realize uncertainties of  $\pm 1$ <sup>o</sup>C, or better, in the calibration of its noble metal thermocouples is described. This will be realized through implementing a Co–C eutectic fixed point in CCPI's calibration chain. As this melts at 1,324◦C, very close to the heat-treatment temperatures required, low uncertainties will be obtained. This should yield an increase in effectiveness of the heat-treatment process performed by Bodycote Heat Treatments Ltd., allowing them to respond effectively to the increasingly stringent demands of engine manufacturers. Outside the current project, there is a strong requirement by industry for lower uncertainties at and above 1,300◦C. Successful implementation of the current fixed point in an industrial setting is likely to result in rapid take-up by other companies, probably through the supply of ultra-low uncertainty thermocouples, looking to improve their high-temperature processes.

National Physical Laboratory, Hampton Road, Teddington TW11 0LW, UK e-mail: jonathan.pearce@npl.co.uk

T. Ford

CCPI Europe, Units 3, 4 & 5, Norwood Ind. Est., Rotherham Close, Killamarsh S21 2JU, UK

### S. Wardle Bodycote Heat Treatments Ltd., Wilmore Road, Derby DE24 9HZ, UK

J. V. Pearce (B) · G. Machin

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## **1 Introduction**

The increasing popularity of air travel is placing demands on airlines to reduce the effect of the resulting carbon dioxide emissions on the environment. At the same time, as the cost of fuel increases, one of the biggest challenges facing airlines today is to reduce fuel consumption. The next generation of commercial aircraft will be required to operate with much higher fuel efficiency than at present. The simplest way to enable this is to operate the engines at higher temperatures. This requires new alloys for the turbine blades, with more exacting heat-treatment requirements to ensure that the necessary metallurgical properties are attained, while at the same time reducing the permissible tolerances on the temperature control within the heat-treatment furnaces.

The next generation of turbine blades will undergo heat treatment at over  $1,310°C$ to attain the required properties. In the project described here, the heat treatment, which is controlled by noble metal thermocouples, is performed at Bodycote Heat Treatments, Derby, UK. The process has exacting control requirements  $(\pm 3^{\circ}C)$  at the limits of the lowest accredited uncertainty currently available from thermocouple manufacturers. The heat treatment is therefore limited by available uncertainties, and additional detailed inspection is required to verify the process efficacy. Ideally, Bodycote would like to attain  $\pm 1$ <sup>°</sup>C temperature uncertainty in process, but this is currently not possible because even the best accredited uncertainties attainable for noble metal thermocouple calibration (by NMIs) at these temperatures are around  $\pm 1$ <sup>°</sup>C.

The principal method of control and measurement within the heat-treatment furnaces involves measurement of ambient temperature with thermocouples. Thus, the immediate way of addressing the problem of reduced tolerances on temperature measurement and control is to reduce the calibration uncertainty of the thermocouples in the temperature range of the heat-treatment process, here in the region of 1,300◦C.

Thermocouples are conventionally calibrated by measuring the voltage generated at fixed points on the ITS-90 which are relevant to the temperature range of use expected for the thermocouple. A calibration curve is then produced which relates the output of the thermocouple to the ITS-90. However, the highest conventional fixed point is copper at  $1,084.62°$ C. The next highest is the palladium wire bridge at  $1,554.8°$ C. In between, there are no calibration points, so the thermocouple calibration must be interpolated over a wide temperature range for measurements in the region of interest for the current application, which gives rise to a relatively large uncertainty. Clearly, what is required is a fixed point in the region of  $1,300\degree C$ , which makes the cobalt–carbon eutectic fixed point a clear choice.

The main goals of the project are:

- Reduce the calibration uncertainty of Type S thermocouples to  $\lt 1$ °C in the temperature region around 1,300◦C.
- Avoid destruction of the thermocouple hot junction during calibration (as in the wire bridge method).
- Increase confidence in the calibration by having a fully assessed fixed-point cell rather than relying on a piece of palladium wire of uncertain purity.

This article describes the first steps on the road to realization of these aims, with the construction, characterization and implementation of a cobalt–carbon eutectic fixed point, with a melting temperature of 1,324◦C, in the calibration laboratory of a major UK thermocouple manufacturer, CCPI Europe.

## **2 Project Overview**

The project is a joint collaboration between NPL and two UK industrial partners: CCPI Europe (P1) and the multi-national Bodycote Heat Treatments Ltd. (P2). Bodycote Derby is their only UK facility that can offer vacuum heat treatment and hot isostatic pressing at the same location. They specialize in the processing of aerospace gas turbine components. The heat treatments are performed in five vacuum furnaces of dimensions  $1 \text{ m} \times 1.5 \text{ m} \times 0.8 \text{ m}$  with a payload of up to 1,000 kg and a temperature range of 500–1,400◦C. They have attained national, international and customer-specific quality accreditations, including ISO 9001:2000, AS/EN 9100, TS 157, and NADCAP accreditation.

The other partner, CCPI Europe, is a multi-national organization specializing in a wide range of temperature-sensing equipment and services. They are one of the UK's largest manufacturers of thermocouples, and are accredited by UKAS for calibrations. They have a sophisticated calibration laboratory, and provide calibrated Type S thermocouples for the furnaces at Bodycote. These thermocouples are calibrated at several temperatures including the Au fixed point (crucible) and the Pd fixed point (wire bridge), and have an uncertainty of  $\pm 3^{\circ}$ C at around 1,300°C.

For the process under consideration, the turbine blades are placed on pallets in the furnace at P2 and the heat treatment is performed over a pre-determined temperature/ time sequence, with each heat-treatment run lasting 2–3 days. The thermocouples and furnaces are serviced every few months to ensure that optimum performance is continually attained. The temperature of the furnace is controlled and monitored with several Type S thermocouples placed around the furnace volume.

Turbine blades for the next generation of jet engines require heat treatment with a tolerance of +2/−3◦C, which is depicted by the grey zones in Fig. [1.](#page-3-0) This tolerance is at the limit of what is achievable with thermocouple calibrations at P1. More than 2<sup>°</sup>C above or 3<sup>°</sup>C below the nominal process temperature can result in improper heat treatment, shown by the white zones in Fig. [1.](#page-3-0) Figure [1](#page-3-0) shows that the current thermocouple calibration uncertainties at P1 (black point), while acceptable for existing technologies, are at the limit of what is acceptable for the new heat treatment. The current project will result in thermocouple calibration uncertainties, a factor of between two and three times lower, depicted by the white point in Fig. [1.](#page-3-0)

The scheme is as follows. The Co–C eutectic fixed point for thermocouple calibration has been constructed and evaluated at NPL. P1, assisted by NPL, has implemented a high-temperature furnace capability, and installed the fixed point in the furnace to achieve a practical calibration facility at the Co–C fixed point. NPL, P1 and P2 are working to develop a calibration strategy. P1 will perform calibrations of Type S thermocouples, to provide calibrated thermocouples for use by P2 for evaluation in a process furnace under real-world conditions. For the best evaluation, and to avoid



<span id="page-3-0"></span>**Fig. 1** Illustration showing the current temperature measurement uncertainty (*black*) and that arising from calibration with a Co–C fixed point (*white*), in relation to the tolerances required for heat treatment of the next generation of turbine blades.  $T$  is the measured temperature, and  $T_{\text{process}}$  is the ideal process temperature

disruption of operations at P2, as well as accreditation issues, a dual calibration strategy will be followed: the test thermocouples are calibrated using both old and new methods, so that the effect of the new calibration could be directly evaluated.

# **3 Eutectic Fixed Point**

The advent of metal–carbon eutectic fixed points is revolutionizing high-temperature metrology [\[1](#page-8-0)[–4](#page-8-1)], offering a series of high-quality, robust fixed points from the copper point (1,084.62◦C) to above 3,000◦C. For industries that rely on thermocouples for temperature measurement and process control, the main advantages of metal–carbon eutectics are:

- The opportunity to dispense with the wire-bridge calibration at the melting point of palladium, which involves destruction of the hot junction and use of palladium of uncertain purity, and enables instead the use of a well-characterized fixed-point crucible.
- Ability to reduce the adverse effects of interpolation over the large temperature range between the copper point and the palladium wire point.
- Ultimately, as higher-temperature M–C eutectic fixed points for thermocouple calibration become available as secondary reference points traceable to ITS-90 [\[5](#page-8-2)], the ability to dispense with extrapolation at temperatures above the palladium or platinum wire point.

The key fixed-point material for the current application is the Co–C eutectic alloy, which melts at nominally 1,324℃, and is already sufficiently characterized and repeatable for this application  $[2,6]$  $[2,6]$  $[2,6]$ , thereby dramatically reducing the interpolation uncertainty involved in the measurement of process temperatures at around  $1,300\degree C$ , mid-way between the previous calibration points of copper  $(1,084.62°\text{C})$  and palladium (1,554.8◦C).

#### **4 Implementation of Co–C Fixed Point**

Care must be taken with the furnace setup when implementing the Co–C fixed point for contact thermometry, with particular regard to ensuring a safe, inert-gas environment and convenient insertion/removal of the crucible. The experimental arrangement in a three-zone tube furnace is shown in Fig. [2.](#page-5-0) Adequate immersion of the thermocouple is ensured with judicious use of insulating graphite felt interspersed with conducting graphite disks. The crucible itself sits inside a hollow graphite cylinder, and mechanically isolated from it by ensuring that there is a gap between the crucible and cylinder except at the bottom, where the cylinder sits on a graphite-felt pad. This ensures a high degree of temperature uniformity, critical for high performance of the fixed point [\[7](#page-8-5)]. Experimental details can be found in Ref. [\[9](#page-8-6)].

The furnace arrangement shown in Fig. [2](#page-5-0) was set up in the calibration laboratory of P1, and tests were performed to verify that the temperature uniformity was sufficient for reliable operation of the fixed point. By adjusting the end-zone set points, it was possible to establish a temperature gradient of 0.25 K over the length of the crucible. However, to prevent possible mechanical problems with the crucible, a temperature difference of 0.7 K between the top and bottom of the crucible was established to ensure that melting starts at the top of the ingot where there is free space for expansion [\[4](#page-8-1)[,7](#page-8-5)]. The temperature gradient is shown in Fig. [3.](#page-6-0) The high quality and long duration of the melt, as measured at P1 following installation, is shown in Fig. [4.](#page-6-1)

The thermodynamic temperature of melting of the Co–C point has been considered by Sadli et al. [\[5\]](#page-8-2) to be  $(1324.0\pm0.6) °C$  ( $k = 2$ ). More recent measurements by Anhalt et al. [\[8](#page-8-7)] are consistent with this determination but have a smaller uncertainty: NMIJ cell—(1324.10 ± 0.22)°C, NPL cell— (1323.96 ± 0.23)°C, and LNE-INM/CNAM cell—(1324.03  $\pm$  0.21)<sup>°</sup>C ( $k = 2$ ). To assign a temperature to the current fixed-point crucible, a small non-contact crucible will be constructed, employing Co and C from the same batch as used for the contact thermometry, for determination of the melting temperature using non-contact thermometry at NPL. The advantage of this is that the cell temperature will be directly traceable to ITS-90. Thus, pending this determination, for this application the Co–C melting point is assigned a temperature of  $(1324.0 \pm 0.6)$ °C [\[5](#page-8-2)] with respect to ITS-90. This uncertainty of  $0.6$ °C ( $k = 2$ ) in the assignment of a melting temperature using non-contact thermometry is conservative. It incorporates effects including furnace temperature uniformity, material purity and the dependence of the melting temperature on the prior freezing rate. We expect it to be revised downwards by around 25–50% on completion of the non-contact thermometry measurements.

To evaluate the effectiveness of the calibration with the Co–C fixed point, the thermocouples will also be calibrated with existing methods in addition to the new method. Effectively, the Co–C fixed point is simply added to the list of calibration fixed points (including Au and Pd) and two deviation curves are generated; one with Co–C point, and one without. This has the benefit of direct visualization of the effect of the new <span id="page-5-0"></span>**Fig. 2** Schematic of experimental setup for a Co–C fixed point. The furnace work tube volume is subject to a pressure of approximately 1.1 bar. The system is optimized with respect to temperature uniformity in the crucible volume, easy removal of the crucible during filling, and effective thermalization of the thermocouple. The graphite equalizer is designed to minimize any remaining temperature gradient along the crucible volume



calibration methodology, while at the same time making no impact on the commercial processing performed at Bodycote. A simple uncertainty budget for a Type S thermocouple calibrated at the Co–C fixed point is shown in Table [1.](#page-7-0) When the non-contact thermometry measurements are performed as mentioned above, the uncertainty of the ingot temperature will fall to approximately  $0.25\degree C$  ( $k = 2$ ). The overall expanded uncertainty for calibration for this example is  $\lt 1^\circ$ C. Note that the largest contribution to the calibration uncertainty at the fixed-point temperature is the thermocouple homogeneity; further work in this area, such as the development of in situ calibration methods, is likely to further reduce the measurement uncertainty in this temperature region.



<span id="page-6-0"></span>**Fig. 3** Temperature distribution over the crucible volume, illustrating the effect of changing end-zone set points.  $\delta T$  is the difference in temperature from the position at the bottom of the fixed-point crucible. The best temperature uniformity was obtained when the top heating zone of the three-zone furnace was set to 6 K below the centre zone, but a 5 K setting was used for the measurements to be sure that the crucible is slightly hotter at the top as described in the text. For comparison, a schematic diagram of the crucible is shown to illustrate the location of the temperature gradient in relation to the crucible. While the smaller temperature gradient gives better uniformity, the higher gradient was selected to better facilitate melting at the top, as crucible durability is a priority on an equal footing with melt quality and duration



<span id="page-6-1"></span>**Fig. 4** Melting and freezing curves showing thermocouple output voltage (emf) as a function of time, for the Co–C eutectic fixed point, measured at CCPI with a Type S thermocouple following installation. Here the furnace setpoint is approximately 3 K above the melting temperature for the melt and approximately 15 K below the freezing temperature for the freeze

## **5 Conclusions**

A project has been described with the aim of reducing thermocouple calibration uncertainties to below 1◦C in the UKAS-accredited laboratory of CCPI Europe. This will

<span id="page-7-0"></span>**Table 1** Uncertainty budget for Type S thermocouple at the Co–C fixed point. There are two dominant contributions. The first arises from the thermocouple inhomogeneity which has been extensively characterized at NPL and which is determined at, e.g., the freezing point of silver by measuring the difference in thermocouple voltage between its value at the thermowell bottom of a fixed-point crucible and on raising it by 10 cm. The second contribution arises from the determination of the melting temperature using noncontact thermometry. The uncertainty estimate here is conservative because it is that assigned for general use to comply with UKAS accreditation

Components	Uncertainty	Probability distribution	Sensitivity coefficient	Uncertainty contribution $u_i$	Unit
Plateau reproduc- ibility	0.50	Normal	1.00	0.50	$\mu$ V
Determination of inflection point	0.12	Rectangular	1.00	0.12	$\mu$ V
Heat flux effect	0.50	Rectangular	1.00	0.50	μV
Voltage determi- nation with digital voltmeter	0.50	Normal	1.00	0.50	μV
Cold junction	0.01	Rectangular	6.00	0.04	μV
Inhomogeneity	4.30	Rectangular	1.00	4.30	μV
Drift	0.50	Normal	1.00	0.50	$\mu$ V
Combined uncer- tainty $(k = 2)$				8.83	μV
Combined uncer- tainty $(k = 2)$				0.68	$\circ$ C
T assignment uncertainty $(k = 2)$				0.60	$\circ$ C
Overall expanded uncertainty $(k=2)$ / $\degree$ C				0.91	$\circ$ C

enable the supply of thermocouples to Bodycote Derby for improved heat-treatment process control to meet the required tolerances for the next generation of turbine blades for jet engines. The performance of the thermocouples will be assessed in situ during heat treatment of turbine blades at Bodycote. So far, the principal aim of the project, to achieve thermocouple calibration uncertainties of better than 1◦C at CCPI by implementing a Co–C fixed point, has been achieved.

Future follow-on projects could include the implementation of commercial Pt/Pd thermocouples, development of Ni-C, Fe-C and Pd-C eutectic fixed points for commercial thermocouple calibrations and development of in situ calibration of thermocouples, possibly with miniature fixed points to facilitate self-calibrating thermocouples. These last two options would result in further dramatic reductions in measurement uncertainty, since they would effectively remove one of the largest contributions to the uncertainty, namely the inhomogeneity of the thermocouple.

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