Characterisation of the NPL Thermal Conductivity Reference Material Inconel 600

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Abstract

Due to the depletion of the thermal conductivity reference materials, the National Physical Laboratory (NPL) has certified a new thermal conductivity reference material, NPL code 2I09 that is based on Inconel 600. This batch of material has been thermally and electrically characterised over the temperature range 100 °C to 500 °C. The primary thermal conductivity measurements used to generate the certified values were carried out in the NPL Axial Heat Flow apparatus, which is a UK national standard measurement facility. It is based on a steady-state absolute technique and is suitable for measuring specimens with thermal conductivities in the range 10 W·m^{-1}·K^{-1} to 240 W·m^{-1}·K^{-1}. Comparisons were made between the reference thermal conductivity values and the derived thermal conductivity values obtained from two indirect methods based upon electrical resistivity and thermal diffusivity measurements, respectively. The reference thermal conductivity values of NPL 2I09 compare with those calculated from the measurements of electrical resistivity within 2 % and those calculated from the measurements of thermal diffusivity, specific heat capacity, density and thermal expansion better than 4 %. In addition, the certified thermal conductivity values of the new batch of Inconel 600, the NPL code 2I09, were compared with the representative reference thermal conductivity values of Inconel 600 reported by J Clark and R Tye, and the agreement is within 2 %. The new reference material NPL 2I09 is available from NPL and can be used to calibrate or check apparatus that provides measurements of thermal conductivity which includes the range 14 W·m^{-1}·K^{-1} to 22 W·m^{-1}·K^{-1} covering the temperature range from 100 °C to 500 °C. The overall uncertainty on the certified values is estimated to be within ± 4.8 %, based on a standard uncertainty multiplied by a coverage factor $k = 2$, providing a level of confidence of approximately 95 %.

Keywords Axial heat flow apparatus · Electrical resistivity · Inconel 600 · Reference material · Specific heat · Thermal conductivity · Thermal diffusivity

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1 Introduction

Test laboratories accredited under the ISO 17025 [1] need to meet five requirements: the general, structural, resource, process, and management system requirements. Within the resource requirements, certified reference materials are important means for establishing metrological traceability. For thermal conductivity test methods based on comparative principles [2, 3], the use of thermal conductivity reference materials is a necessity for calibration of measurement devices. For rapid techniques such as the flash method and similar transient techniques [4–6], although they are absolute methods, the actual execution of the measurement itself is subject to random and systematic errors. Therefore, it is important to verify the performance of a device, for example, using reference materials.

In the past, the National Physical Laboratory (NPL) in the UK produced a series of thermal conductivity reference materials with different thermal conductivity and temperature ranges using the NPL standard long-bar apparatus and the NPL short-sample apparatus. For example, there were reference specimens of Stainless Steel 310, Inconel 600, Nimonic 75 and Stainless Steel 430 [7] spanning a range of thermal conductivities commonly associated with industrial applications. However, the previous stocks of the reference materials were depleted, and the two sets of NPL apparatus are no longer available.

Therefore, in the past decade, NPL has designed and validated a new absolute Axial Heat Flow (AHF) apparatus [8] as the UK's National Standard apparatus. It has been used as the primary method in the development of new thermal conductivity reference materials that replace the previous stocks. The details of the development of the Stainless Steel 304-based NPL thermal conductivity reference material, 2S09 have been previously discussed and published [9].

This paper presents the development of a new Inconel 600-based NPL thermal conductivity reference material, identified as NPL 2I09, over the temperature range 100 °C to 500 °C using the NPL AHF apparatus as the primary method. It also includes the cross-checks made between the reference thermal conductivity values obtained using the primary method, and the thermal conductivity values derived indirectly from: (i) the measurements of thermal diffusivity, specific heat capacity, density and thermal expansion; (ii) the measurements of electrical resistivity via the Wiedemann–Franz relationship.

The new thermal conductivity reference material, NPL 2I09, replaces the previous stocks. It is available from NPL and can be used to calibrate or validate thermal conductivity measurement apparatuses that operate in the temperature range 100 °C to 500 °C. The overall uncertainty on the certified values of NPL 2I09 is estimated to be within ± 4.8 %, based on a standard uncertainty multiplied by a coverage factor $k = 2$, providing a level of confidence of approximately 95 %.

Table 1 The composition and heat treatment of the Inconel 600

Material	Inconel 600								
Composition weight (%)	C	Si	Mn	S	Co	Cr	Cu	Fe	Ni
	0.07	0.45	0.2	0.003	0.18	15.28	0.34	8.71	74.76
Heat treatment	In vacuum, heat up to 1150 °C and held for two hours, afterwards rapid nitrogen cooled								
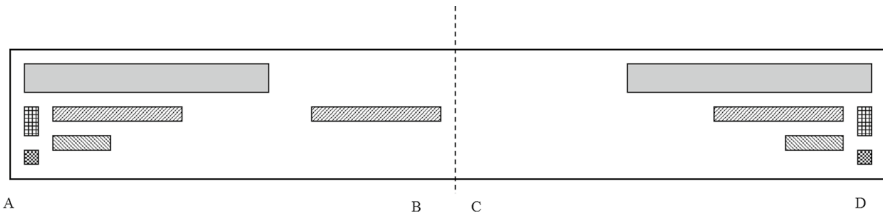
Fig. 1 The specimen cutting plan for the Inconel 600 bar

2 Material and Preparation of Specimens

2.1 Material Composition and Heat Treatment

A stock of Inconel 600 material was acquired in the form of a solid cylindrical bar having a nominal diameter of 60 mm and 1000 mm long. Details of the composition and heat treatment are given in Table 1.

2.2 Preparation of Specimens

The 1000-mm long cylindrical bar was divided into two equal lengths, as shown in Fig. 1 and each end was labelled, with the starting end of the original bar marked as "A" end, then "B", "C" and the finishing end of the original bar marked as "D" end.

Afterwards, various specimens were machined from different locations of the bar for characterising the Inconel 600. The details of the prepared specimens are listed in Table 2.

3 Measurement Methods

3.1 Thermal Conductivity

The thermal conductivity of the new Inconel 600 reference material has been characterised over the temperature range 100 °C to 500 °C using NPL's newly designed absolute Axial Heat Flow (AHF) apparatus [8] as a primary method. The NPL

Table 2 Details of the Inconel 600 specimens prepared for the characterisation (unit: mm)

End of bar from which the specimen is cut	Thermal diffusivity specimen	Specific heat specimen	Electrical resistivity specimen	Thermal conductivity specimen	Thermal expansion and density specimen
A	Ø12.5×2	Ø5.0×1.5	Ø6.0×80	Ø20×160	Ø6.0×25
B	–	–	Ø6.0×80	–	–
C	–	–	–	–	–
D	Ø12.5×2	Ø5.0×1.5	Ø6.0×80	Ø20×160	Ø6.0×25

AHF apparatus is based on an absolute method of measurement and is most suitable for measurements on specimens with thermal conductivities in the range of 10 W·m⁻¹·K⁻¹ to 240 W·m⁻¹·K⁻¹ and at temperatures between 50 °C and 500 °C, with relative expanded measurement uncertainty typically ± 3 % to ± 4 % (coverage factor $k = 2$) [8]. Its use is normally restricted to thermally homogeneous specimens with an overall length of 160 mm and a uniform diameter of 20 mm. The schematic views of the NPL Axial Heat Flow apparatus are shown in Fig. 2. In this apparatus, a linear heat flow is induced in a cylindrical bar-shaped specimen by using a guarded heater to apply heat at a known rate at one end and extracting the heat at the other end using an isothermal cooling unit. The heat is constrained to flow axially through the specimen with minimal loss or gain using a linear edge-guard system.
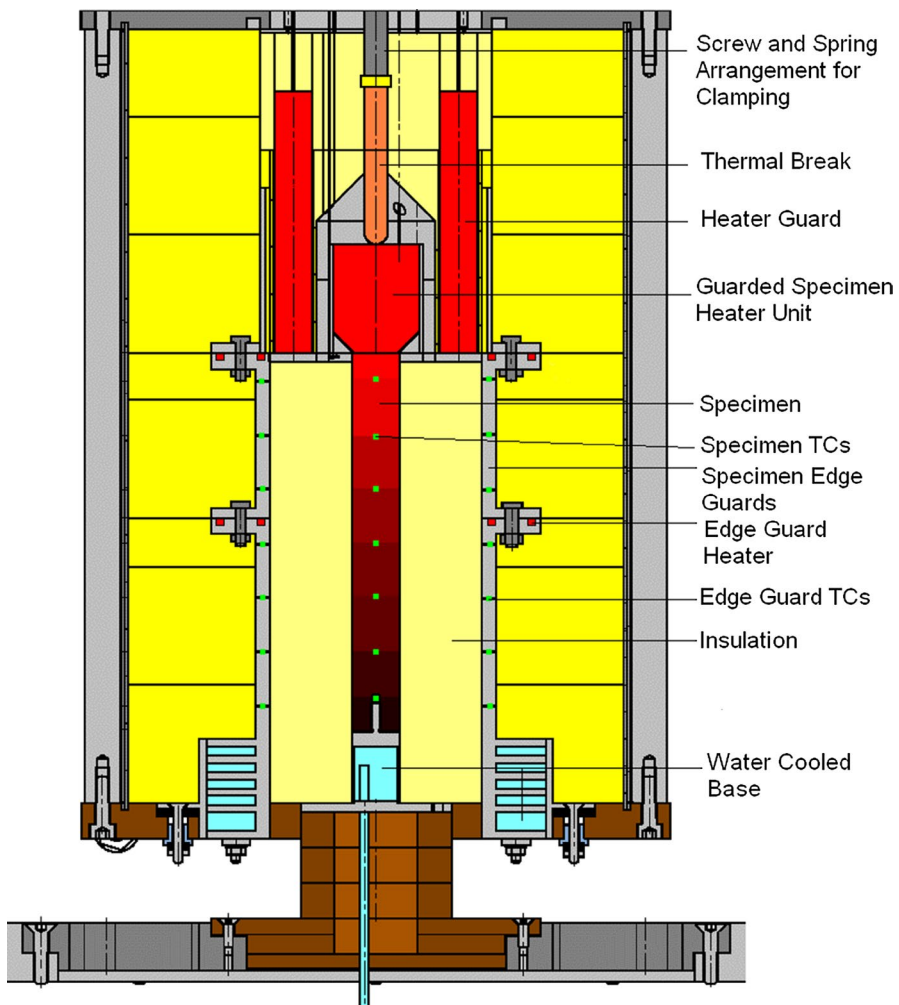

Fig. 2 Schematic diagram of the NPL Axial Heat Flow Apparatus (note: TCs – thermocouples)

The temperatures on the linear edge guard is maintained to match closely to the temperatures of the specimen at the same height level. The temperatures along the specimen are measured using thermocouples, and the heat flux is determined by the amount of electrical power used to heat the main specimen heater. The electrical power is maintained at a fixed value to obtain an appropriate steady-state temperature gradient along the specimen. Then knowing the specimen's length and cross-sectional area, the thermal conductivity of the specimen can be calculated. This simple calculation given by the following equation is based on Fourier's law:

$$\lambda(t) = \frac{(Q/A)}{(\Delta T/\Delta x)}, \tag{1}$$

where $\lambda(T)$ is the thermal conductivity of the test specimen (W·m^{-1}·K^{-1}) at the mean specimen temperature, T (°C), Q is the amount of heat passing through the length of a specimen (W), A is the cross-sectional area of the test specimen (m^2), ΔT is the temperature difference between two thermocouple positions along the test specimen (K), and Δx is the distance between two thermocouple positions along the test specimen (m). The mean specimen temperature, T, is the average of the readings of the two thermocouples for each specimen zone.

3.2 Thermal Diffusivity

The Laser-Flash System, NETZSCH LFA 427 at NPL, was used to measure the thermal diffusivity of the Inconel 600 specimens. During measurements, laser light is pulsed onto one side of a plane parallel sample, and the temperature response on the other side of the sample is measured. From this response, the thermal diffusivity of the sample can be calculated [4, 5]. Each specimen for thermal diffusivity measurements is a disc that is 12.5 mm in diameter and approximately 2 mm thick, and the surfaces of the disc are coated with graphite to improve the absorption on the heated face and increase the thermal radiation from the rear temperature sensing face. Each of the specimens was heated (or cooled) to a measuring temperature in a graphite heated furnace under argon environment and stabilised with respect to temperature for at least 10 min prior to making a thermal diffusivity measurement. According to the NPL procedure for measurement of thermal diffusivity of solids using LFA, the relative expanded uncertainty of the thermal diffusivity measurements is typically ± 4 % ($k = 2$).

3.3 Specific Heat Capacity

The specific heat capacity of each of the Inconel 600 specimens was measured using a twin-crucible differential scanning calorimeter, NETZSCH DSC 404 apparatus at NPL. Each specimen is heated at a rate of 5 K min^{-1} over the temperature range 40 °C to 800 °C in an argon environment. The test specimen was a disc 5.0 mm in diameter and 1.5 mm thick. The relative expanded measurement uncertainty is estimated to be within ± 4 % ($k = 2$) [9].

3.4 Thermal Expansion

Thermal expansion measurements were made using a twin push-rod dilatometer, Netzsch 402 ED apparatus at NPL on test specimens approximately 25 mm in length and 6 mm in diameter. In the test apparatus, the specimens were heated to 800 °C under argon environment at a heating rate of 5 K min^{-1} and the test was repeated three times. The thermal expansion data were used to correct the specimen dimensions at elevated temperatures. The relative expanded measurement uncertainty is estimated to be within ± 4 % ($k = 2$) [7].

3.5 Density

Density measurements were carried out at room temperature, 20 °C by weighing specimens in air and distilled water. The true density, d_{20} (kg m^{-3}), was obtained by use of the equation:

$$d_{20} = \frac{d_w M_a - d_a M_w}{M_a - M_w},$$ (2)

where M_a (kg) is the measured mass in air at room temperature, M_w (kg) is the measured mass immersed in water at room temperature, d_w (kg m^{-3}) is the density of water and d_a (kg m^{-3}) is the density of air. The relative expanded uncertainty is typically ± 0.1 % ($k = 2$) [9].

3.6 Electrical Resistivity

The electrical resistivity measurements were made by the conventional four-probe contact method on Inconel 600 specimens. The details of the method can be found in the reference [10]. In this method, a constant current, I (A), is passed through the test specimen. Two central probes are used to measure the voltage drop, U (V). The resistivity, ρ ($\Omega \cdot$m), is calculated from Eq. 3:

$$\rho = \frac{(U/I)A}{L}.$$ (3)

The measured thermal expansion coefficient was used to correct the specimen cross-sectional area, A (m^2) and the distance between the voltage probes, L (m) at a measurement temperature.

The electrical resistivity at each temperature is measured for three values of current, 5, 10, and 15 amps, to check that no resistance heating occurs in the tests. Also, the voltage is taken as the average of two readings with and without reversal of the current, to eliminate parasitic thermal contact potentials due to the voltage probes. The experimental uncertainty is less than 0.5 % [7].

3.7 Indirect Methods—Derivation of Thermal Conductivity

3.7.1 Indirect Method 1—Derivation of Thermal Conductivity from Electrical Resistivity

As one of the indirect methods, the measured electrical resistivity can be used to calculate the thermal conductivity, using an empirically modified Wiedemann–Franz relationship proposed by Smith and Palmer [11], Eq. 4:

$$\lambda(T) = L_{sp}\left(\frac{T + 273.15}{\rho}\right) + C_{sp}. \tag{4}$$

For Inconel 600, a Nickel–chromium alloy, the modified Lorenz number for the electronic component L_{sp} is 2.2×10^{-8} (V K^{-1})^2, and the constant represents all contributions other than those due to electrons, C_{sp} is 6.0 (W·m^{-1}·K^{-1}) [7].

3.7.2 Derivation of Thermal Conductivity from Thermal Diffusivity, Specific Heat Capacity, Density and Thermal Expansion

As another indirect method, the measured thermal diffusivity, specific heat capacity, density and thermal expansion can also be used to calculate the thermal conductivity, see Eq. 5:

$$\lambda(T) = a(T)d(T)C_p(T) \tag{5}$$

where $a(T)$ is thermal diffusivity (m^2·s^{-1}), $C_p(T)$ is specific heat capacity (J·kg^{-1}·K^{-1}) and $d(T)$ is density of a material (kg·m^{-3}).

Since the sample thickness $L(T)$ and density $d(T)$ were measured at room temperature and noted as L_{20} and d_{20}, thermal expansion $\Delta L(T)$ needs to be considered in the calculation of thermal conductivity. In the following calculations, we have assumed the samples are isotropic.

For thermal diffusivity measurements using the laser-flash technique, the effect of thermal expansion of the test specimen at temperature, T, can be corrected using Eq. 6:

$$a(T) = a_{measured}(T)\left[1 + \frac{\Delta L(T)}{L_{20}}\right]^2. \tag{6}$$

The corrected density at temperature, T, can be calculated from Eq. 7:

$$d(T) = d_{20}\left[1 + \frac{\Delta L(T)}{L_{20}}\right]^{-3}. \tag{7}$$

Therefore, thermal conductivity at temperature, T, can be calculated using Eq. 8:

$$\lambda(T) = a_{measured}(T)d_{20}(T)C_p(T)\left[1 + \frac{\Delta L(T)}{L_{20}}\right]^{-1}. \qquad (8)$$

4 Characterisation of the NPL Thermal Conductivity Reference Material, NPL 2I09

4.1 Uniformity in Terms of Electrical Resistivity

The initial assessment of the uniformity of the new NPL thermal conductivity reference material NPL 2I09 was carried out using electrical resistivity measurements on three test specimens. These specimens were machined from the two ends and the middle of the metal bar and were labelled as 2I-A2 (from "A" end), 2I-B2 (from middle) and 2I-D2 (from "D" end).

The measured electrical resistivity values of the three specimens are shown in Fig. 3(a), and the relative difference of the electrical resistivity values between each specimen and the mean values is shown in Fig. 3(b). The difference is within ± 0.5 %, *i.e.,* within the measurement uncertainty of the electrical resistivity. The initial assessment has shown that the material is uniform in terms of electrical resistivity.

4.2 Uniformity in Terms of Thermal Conductivity

The assessment of material uniformity in terms of thermal conductivity was carried out by measurements using the primary method, the NPL Axial Heat Flow apparatus on two specimens machined from both ends of the metal bar, one was labelled as 2I-A2 and another was labelled as 2I-D2.
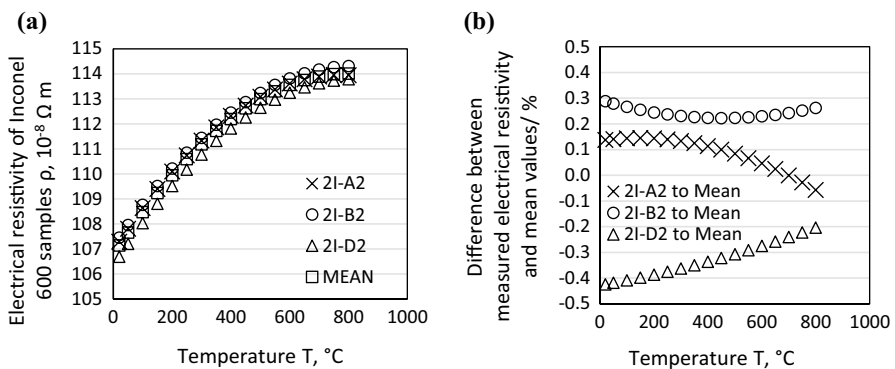

Fig. 3 Initial uniformity assessment in terms of electrical resistivity (a) measured electrical resistivity of the three specimens; (b) difference between the measured electrical resistivity values of the three specimens and their mean value

The measured thermal conductivity values of the two specimens are shown in Fig. 4(a), and the difference between the two specimens is shown in Fig. 4(b). The difference is well within the measurement uncertainty of the NPL Axial Heat Flow apparatus, which is typically 3 % to 4 % over the temperature range 100 °C to 500 °C based on a standard uncertainty multiplied by a coverage factor $k=2$, providing a level of confidence of approximately 95 % [8]. The assessment has shown that the material is uniform in terms of thermal conductivity.

4.3 Stability in Terms of Thermal Conductivity

The assessment of the stability of the new NPL thermal conductivity reference material 2I09 was carried out on both the specimen 2I-A2 and 2I-D2 using the NPL Axial Heat Flow apparatus. After the thermal conductivity measurements under the steady-state condition with the specimen heater maintained at the maximum temperature 600 °C, the measurements were then repeated sixteen times with half an hour interval between each repeat. Figure 5 shows an example of the stability assessment on the specimen 2I-D2 in terms of thermal conductivity. It shows that the repeated measurements at the mean specimen temperatures of 461.1 °C and 524.6 °C are stable within 0.5 %, well within the measurement uncertainty of the NPL Axial Heat Flow apparatus. The two mean specimen temperatures were calculated from two different zones/segments along the test specimen while the specimen heater was maintained at 600 °C.

4.4 Uncertainty Evaluation

The expanded relative measurement uncertainty of the thermal conductivity values of the NPL reference material 2I09 is estimated to be ± 4.8 % ($k=2$) providing a level of confidence of approximately 95 %. The uncertainty evaluation has included the relative uncertainty of thermal conductivity measurements using the NPL AHF apparatus (u_c), stability, reproducibility, and uniformity assessment
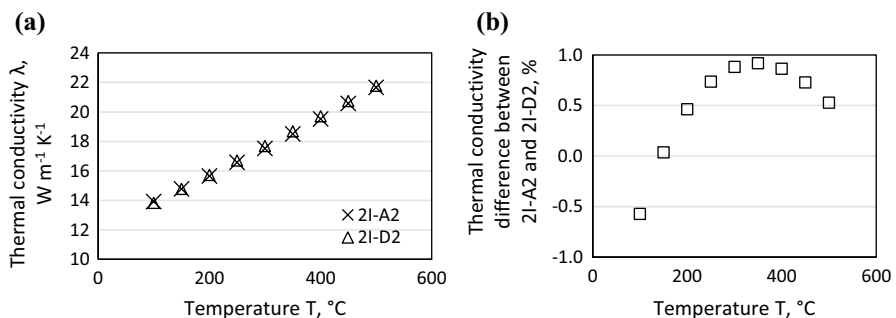

Fig. 4 Uniformity assessment in terms of thermal conductivity (a) measured thermal conductivity values of the two specimens 2I-A2 and 2I-D2; (b) the difference between the measured thermal conductivity values of the two specimens
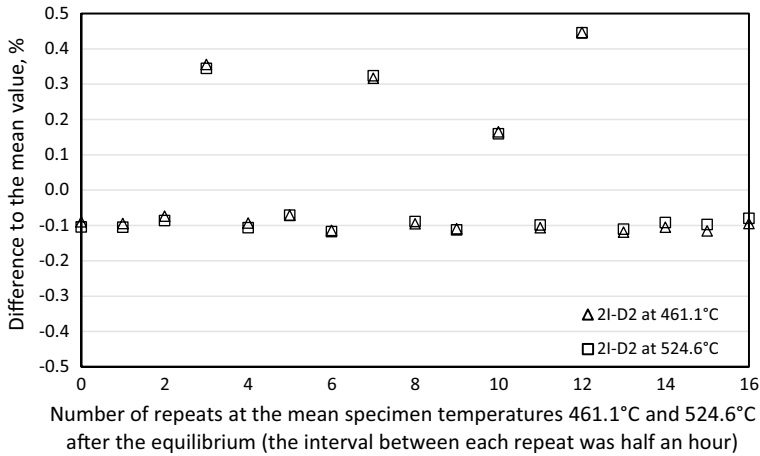
Fig. 5 An example of stability assessment on the specimen 2I-D2 in terms of thermal conductivity

results, and a small uncertainty of curve fitting of the relation between the thermal conductivity and temperature.

The details of the sources of uncertainty contributing to u_c is given below. Based on the differentiation of Eq. 1, the relative uncertainty of thermal conductivity measurements using the NPL AHF apparatus, u_c, is

$$u_c = \sqrt{u_Q^2 + u_A^2 + u_{\Delta T}^2 + u_{\Delta x}^2}, \tag{9}$$

where u_Q is the relative uncertainty of the heat passing through the length of a test specimen; u_A is the relative uncertainty of the cross-sectional area of the test specimen; $u_{\Delta T}$ is the relative uncertainty of the temperature difference between two thermocouple positions along the test specimen and $u_{\Delta x}$ is the relative uncertainty of the distance measurements between two thermocouple positions along the test specimen.

The sources of uncertainty contributing to $u_{\Delta x}$ include the calibration uncertainty and resolution of the travelling microscope that is used for measuring the distance between two thermocouple positions along the test specimen; the misalignment during the measurement of distance; the tolerance between the outer diameter of the thermocouple and the diameter of the thermocouple hole and the uncertainty of the correction of thermal expansion (which is negligible).

The sources of uncertainty contributing to $u_{\Delta T}$ include the calibration uncertainty and resolution of the digital voltmeter; the calibration uncertainty of the thermocouples; the batch agreement between the thermocouples; and the uncertainty of the curve fitting of temperature and thermocouple emf output.

The sources of uncertainty contributing to u_A include the calibration uncertainty and resolution of the calliper that is used for measuring the diameter of the test specimen; the misalignment during the measurement of diameter; and the variation of the specimen diameter that is measured at nine positions. The

coefficient between u_A and the relative measurement uncertainty of specimen diameter is 2.

The sources of uncertainty contributing to u_Q include the calibration uncertainty and resolution of the Digital Voltmeter; the calibration uncertainty of the standard resistor that is used for determining the electrical current through the specimen heater; the heat loss/gain due to the temperature imbalance between the specimen heater and the edge guard of the specimen heater; and the heat loss/gain due to the temperature imbalance between the test specimen and the edge guard of the test specimen.

The stability and reproducibility of the whole measurement system are assessed separately. In addition, the relative uncertainty of the measurement of mean specimen temperature also contributes to the thermal conductivity measurements via the temperature dependence of thermal conductivity of the NPL reference material 2I09.

5 Discussion

Although the UK's National Standard, the NPL Axial Heat Flow apparatus, was used as the primary method to characterise the thermal conductivity reference material NPL 2I09, additional cross-checks were performed by comparing the measured values with the derived thermal conductivity values using the other two indirect methods mentioned in Sect. 3.7 above. The results of the cross-checking are shown in Fig. 6. The reference thermal conductivity values and those calculated from the measurements of electrical resistivity agreed within 2 %. Those values calculated
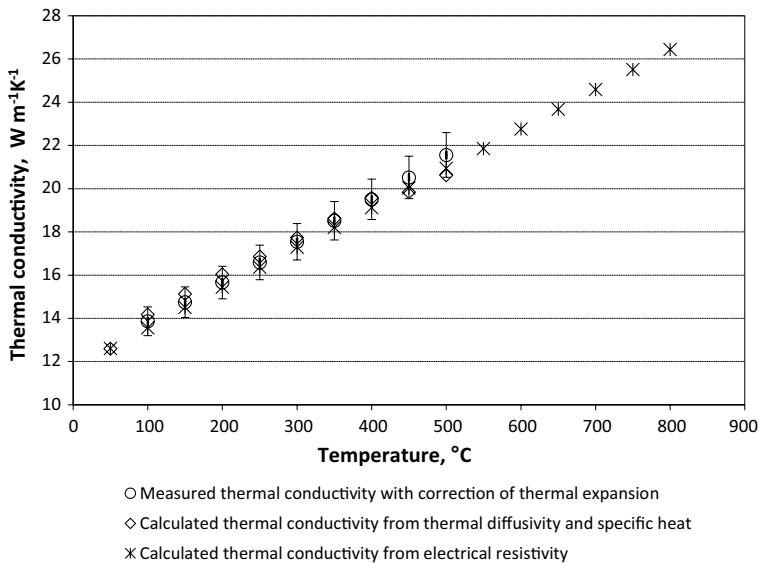

○ Measured thermal conductivity with correction of thermal expansion

◇ Calculated thermal conductivity from thermal diffusivity and specific heat

✕ Calculated thermal conductivity from electrical resistivity

Fig. 6 Comparison between the measured thermal conductivity values of 2I09 using the primary method, NPL AHF apparatus and the derived values using the two indirect methods mentioned in Sect. 3.7

from the measurements of thermal diffusivity, specific heat capacity, density and thermal expansion agreed with the reference values within 4 %. The agreements were within the uncertainty of the new NPL thermal conductivity reference material 2I09 (see the uncertainty bars shown in Fig. 6).

After cross-checking with the other two indirect methods available at NPL, further checks were performed by comparing the thermal conductivity values of the new NPL reference material 2I09 with the representative reference thermal conductivity values of Inconel 600 reported in the Table 13 of paper [7]. The representative reference values reported in their paper were based on the thermal conductivity measurements of the previous stocks of Inconel 600 reference materials, which were certified using the NPL standard long-bar apparatus and the NPL short-sample apparatus. However, the previous stocks of the NPL Inconel 600 reference materials have been depleted, and the two NPL apparatus sets are no longer available. The results presented in Fig. 7 show that the agreement is within 2 %, *i.e.,* less than half of the uncertainty of the NPL thermal conductivity reference material 2I09.

6 Summary

This paper presents the development of a new stock of NPL thermal conductivity reference material 2I09 that is based on Inconel 600, as the previous stocks were depleted. The new NPL reference material 2I09 has been characterised over the temperature range 100 °C to 500 °C using the primary method, NPL's newly designed absolute Axial Heat Flow apparatus. The material is uniform in terms of electrical resistivity and thermal conductivity and it is stable. The certified thermal conductivity values of the new NPL reference material 2I09 is summarised in Table 3, and the dependence of its thermal conductivity on temperature is shown in Eq. 10. The expanded uncertainty of the new thermal conductivity reference material NPL 2I09 is 4.8 % ($k = 2$) providing a level of confidence of approximately 95 %. It includes
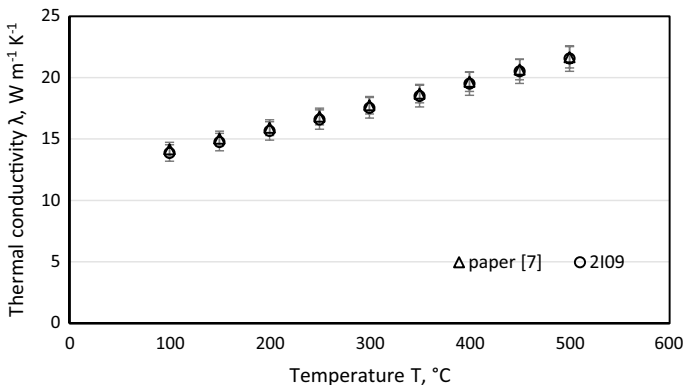

Fig. 7 Comparison between the thermal conductivity values of NPL 2I09 and the representative reference thermal conductivity values of Inconel 600 reported in the Table 13 of paper [7]

Table 3 Certified thermal conductivity values of the new NPL reference material 2I09

Temperature T	Thermal conductivity λ
°C	W·m^{-1}·K^{-1}
100	13.9
150	14.8
200	15.7
250	16.6
300	17.5
350	18.5
400	19.5
450	20.5
500	21.6

the measurement uncertainty, uniformity, stability, and a small uncertainty of curve fitting.

$$\lambda = 4.3208 \times 10^{-6}T^2 + 1.6638 \times 10^{-2}T + 12.158, \tag{10}$$

where the unit of the temperature, T is °C.

Cross-checks were performed between the reference thermal conductivity values of NPL 2I09 and the derived thermal conductivity values from the two indirect methods, and further checked with the representative reference thermal conductivity values of Inconel 600 reported in the Table 13 of paper [7]. The reference thermal conductivity values and those calculated from the measurements of electrical resistivity agreed within 2 %. Those values calculated from the measurements of thermal diffusivity, specific heat capacity, density and thermal expansion agreed with the reference values within 4 %. In addition, the reference thermal conductivity values of the NPL 2I09 agreed within 2 % with the representative reference values of Inconel 600 [7].

The new stock of Inconel 600-based thermal conductivity reference material, NPL 2I09, replaces the previous stocks which were depleted. It is available from NPL and can be used to calibrate or validate thermal conductivity measurement apparatus that operates in the temperature range 100 °C to 500 °C.

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