Measurement of the Thermophysical Properties of an NPL Thermal Conductivity Standard Inconel 600

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Abstract

Flash methods (Parker et al., 1961) have become one of the most commonly used techniques for measuring the thermal diffusivity and thermal conductivity of various kinds of solids and liquids such as metals, carbon materials, ceramics and polymers. Easy sample preparation, small sample dimensions, fast measurement times and high accuracy are only some of the advantages of this non-destructive measurement technique.

However, the accuracy of measurement and level of uncertainty of the resulting data are becoming increasingly important for countless industrial applications. Instruments must be analyzed to determine the uncertainty of the system at different temperature and application ranges.

One way of checking the accuracy of the results is to cross-check the unit with certified reference materials. However, there is a lack of standard materials for thermal diffusivity/thermal conductivity all over the world. Furthermore, for some available standards, the thermophysical properties are known only over a limited temperature range.

Presented in this work are thermophysical property measurements on a certified thermal conductivity standard, Inconel 600 (Redgrove, 2003). Tests were carried out between -125 and 1000°C. A DIL 402 C pushrod dilatometer was employed to determine the thermal expansion and density change ρ of the material. The specific heat c_p was measured using differential scanning calorimetry. The thermal diffusivity a was measured employing the laser flash technique. Using the measured data, the thermal conductivity λ of the material was determined according to the following equation:

$$\lambda(T) = \rho(T) \cdot c_p(T) \cdot a(T) \quad . \tag{1}$$

Introduction

Inconel 600 is a nonmagnetic, nickel-based high-temperature alloy with high mechanical strength, hot and cold workability and resistance to corrosion. This alloy also displays freedom from aging or stress corrosion throughout the annealed to heavily cold worked condition range. It can be used up to 1000°C without irreversible changes. The Inconel 600 specimens analysed in this work were supplied by the National Physical Laboratories (NPL), UK, as a certified thermal conductivity reference material. The composition of the material is listed in table 1.

Table 1. Composition of the Inconel 600 standard material

Material	Ni	Cr	Fe	Si	Mn	Others
Weight-%	74.4	16.0	8.2	0.29	0.2	0.9

The thermal conductivity is certified between 50 and 500°C. However, the material can be used from the low-temperature range all the way up to 1000°C (Redgrove, 2003). The room-temperature bulk density of the material was 8.340 g/cm³ at room temperature. The Inconel material was thermally treated at 1120°C for 2 hours prior to certification at NPL and the tests carried out later in this work.

Measurement of different thermophysical properties such as thermal expansion and density change, specific heat and thermal diffusivity allows a detailed insight into the material's behavior under thermal treatment and determination of the thermal conductivity. Therefore, intercomparison of the measured results for the thermal conductivity and NPL values was possible. Furthermore, the measurements were carried out down to -150°C and up to 1000°C to check if the material can be used as a standard material over an extended temperature range.

Experimental

From a cylinder block, 50 mm in diameter and 50 mm high, different samples were prepared for the various tests techniques. For each measurement method, two samples were prepared and tested several times. Therefore, it was possible to check the thermal stability and homogeneity of the material but also to determine the reproducibility of the test results.

For the thermal expansion measurements, a NETZSCH DIL 402 pushrod dilatometer was employed. The system can be equipped with different furnaces allowing measurements from sub-ambient temperatures up to 2000°C. For the measurements on the Inconel samples, a low-temperature furnace was used for tests between -150 and 50°C. Using an SiC furnace, the same samples were tested between 50 and 1000°C. All tests were carried out in an inert atmosphere (helium) at a heating rate of 3 K/min. The samples measured with the dilatometer were 25 mm long and had a diameter of 6 mm. Each sample was measured three times in the low- and in the high-temperature furnace. The systems were calibrated with a platinum standard prior to the tests. From the measured thermal expansion, the volumetric expansion and density change were determined.

The specific heat of Inconel 600 was measured using the NETZSCH models DSC 404 C Pegasus and DSC 204 Phoenix differential scanning calorimeter. The DSC 204 was used for tests between -125 and 100°C. The DSC 404 C was used for the tests between 100 and 1000°C. The samples tested with the DSCs were 5 mm in diameter and 1 mm high. All tests were carried out in inert gas at heating rates between 10 and 20 K/min. Evaluation of the specific heat was carried out employing the ratio-method. Technical details regarding the instruments and evaluation technique can be found elsewhere (Blumm and Kaisersberger, 2001).

The thermal diffusivity was measured employing a NETZSCH Model LFA 457 MicroFlash laser flash apparatus. The system allows measurements between -125 and 1100°C (using two exchangeable furnaces). The tests were carried out between -125 and 25°C in steps of 25 K while the system was equipped with the liquid-nitrogen cooled low-temperature furnace. The tests between room temperature and 1000°C were done in steps of 50 K. Again, two different samples were measured three times in each temperature range. All tests were carried out in inert atmospheres (helium and argon).

From the measurement results, the thermal conductivity was calculated according to equation 1. The resulting thermal conductivity was compared to the values from the certificate. The resulting deviations were less than 4% which is within the stated uncertainty level provided by NPL (Redgrove, 2003).

Results and discussion

Presented in figure 1 are the thermal expansion results for Inconel 600 for all test runs. Additionally shown is the mean value of all test runs. It can be seen that the material expands with a slightly increasing rate of expansion versus temperature. Furthermore, no influences of phase transitions were detected in these test runs. The repeatability for the different test runs on the same sample was within the uncertainty of the instrument (approx. 0.5%). No differences were obtained between the results of the two samples tested.

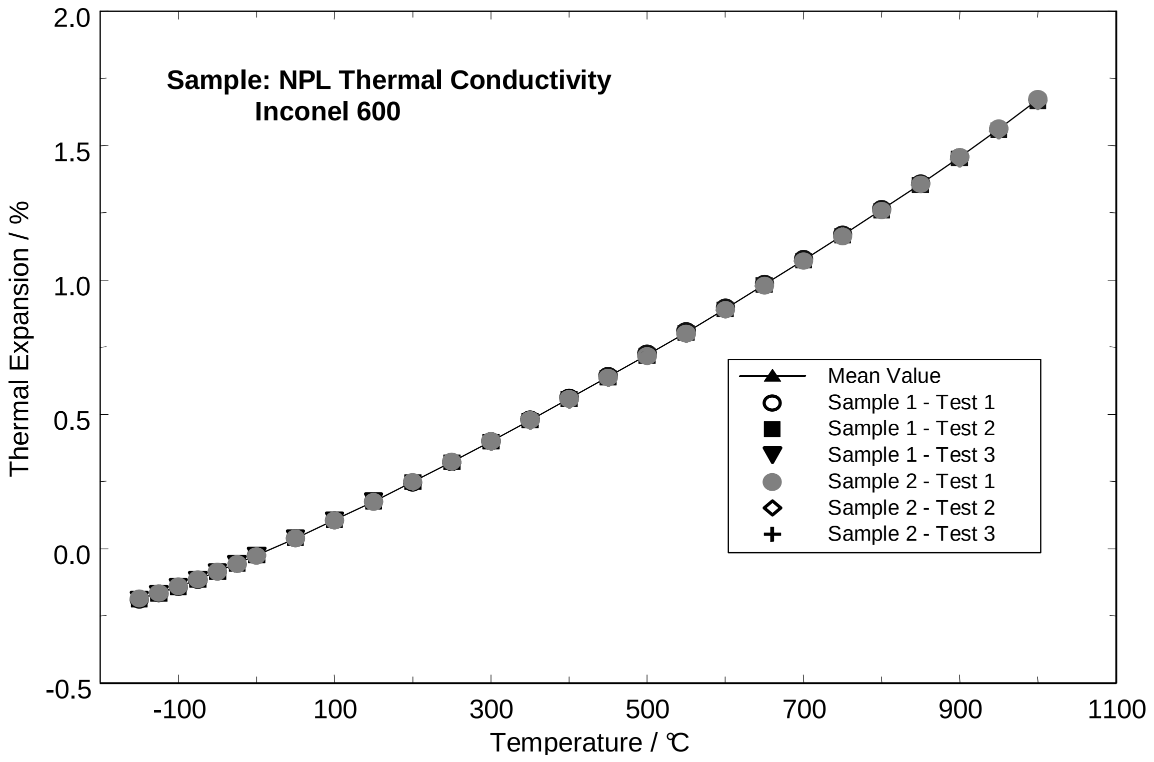

Figure 1. Thermal Expansion of two samples of Inconel 600 material and calculated mean value.

Presented in figure 2 are the volumetric expansion and density change of the Inconel 600 material. The volumetric expansion was calculated using the mean values of the thermal expansion results according to equation 2:

$$\frac{\Delta V}{V_0}(T) \quad = \quad 3 \cdot \left(\frac{\Delta L(T)}{L_0} \right) + 3 \cdot \left(\frac{\Delta L(T)}{L_0} \right)^2 + \left(\frac{\Delta L(T)}{L_0} \right)^3 \quad . \qquad (2)$$

The density was calculated using a room-temperature bulk density of 8.341 g/cm³ and the volumetric expansion data. It can be seen that the volume change of the sample is more than 6% between –150 and 1000°C. Therefore, the density decreases versus temperature from 8.383 g/cm³ at –125°C to 7.937 g/cm³ at 1000°C.

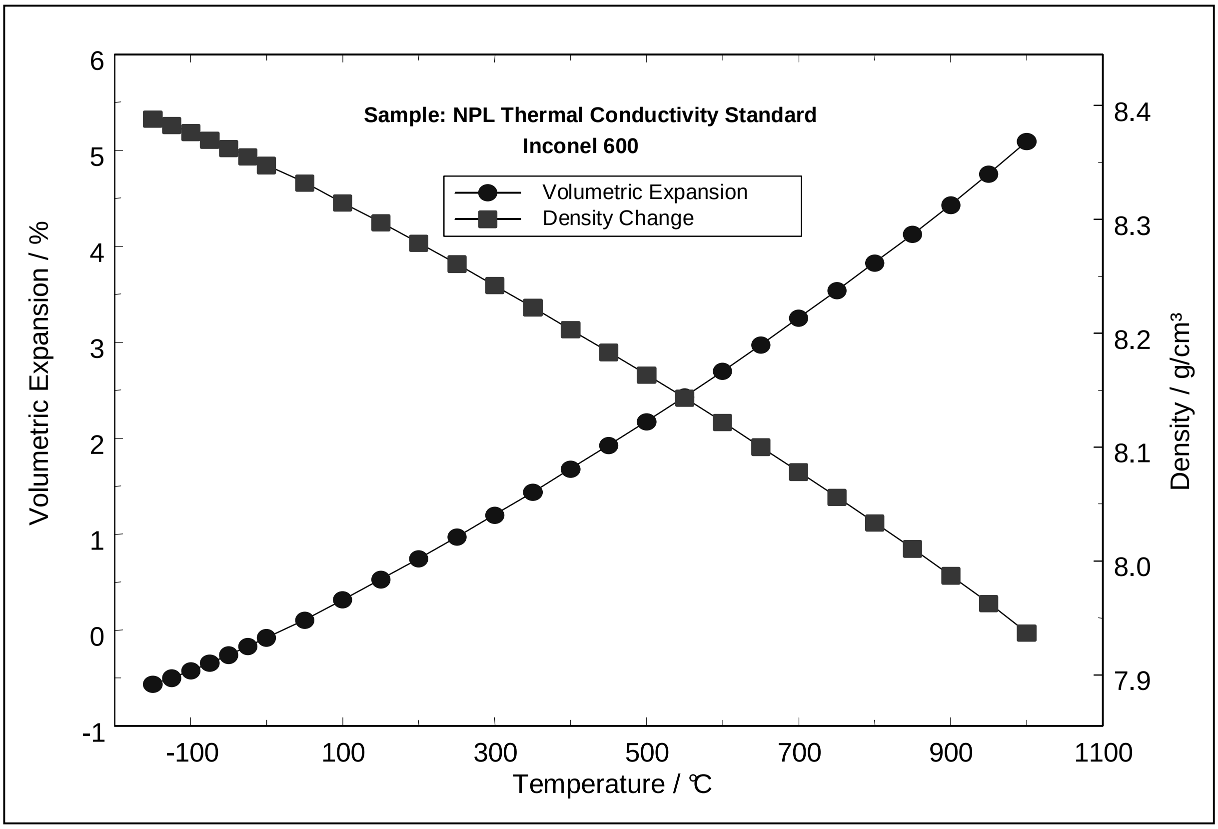

Figure 2. Volumetric Expansion and Density of Inconel 600.

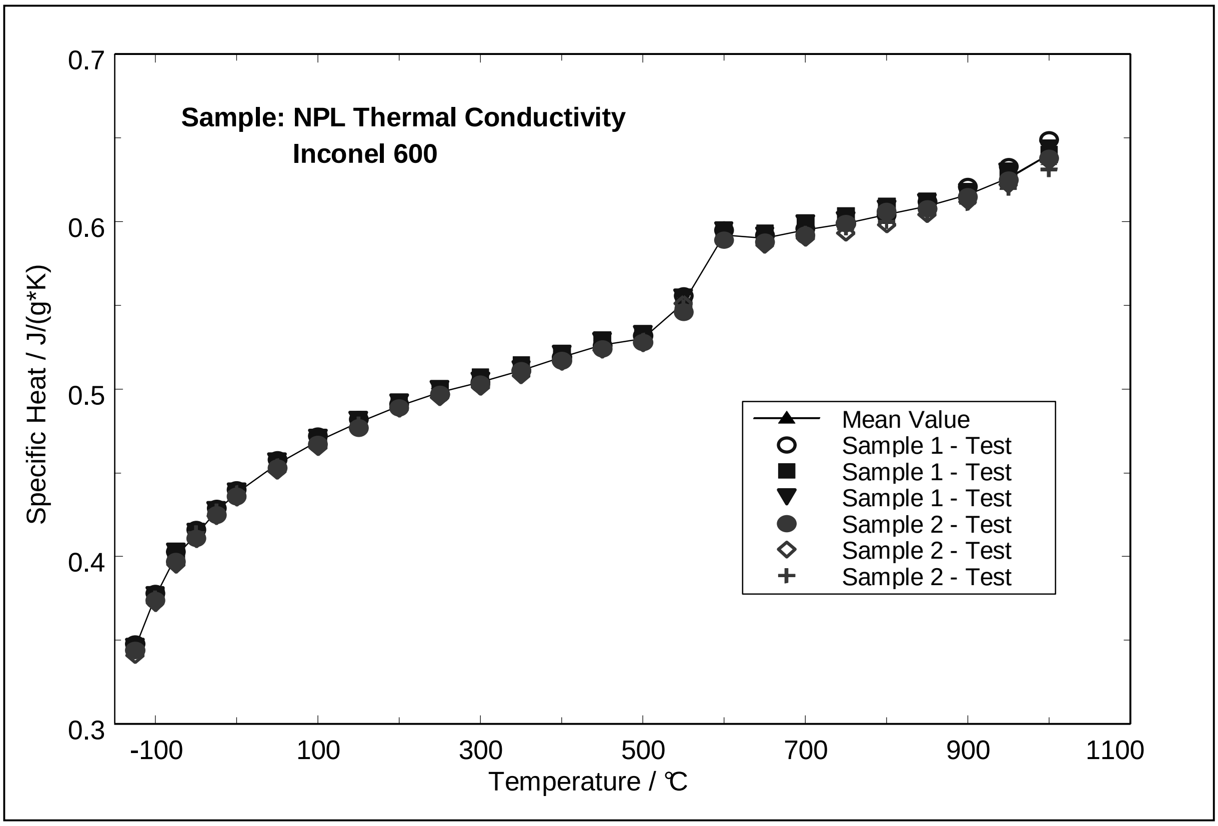

Figure 3. Apparent specific heat of Inconel 600.

Shown in figure 3 is the measured specific heat of Inconel 600 between –125 and 1000°C. The differences between the individual runs are in the range of ±2% which corresponds to the typical accuracy of the unit. In the low-temperature range, a strong increase in the specific heat results can be seen, as expected from the Debye theory. Between 550 and 700°C, an endothermal step can be seen in the measured specific heat. This step can be explained by the formation of Ni_3Cr clusters causing an additional contribution to the specific heat (Richter and Born, 2004). It has to be pointed out that it is critical to separate the true specific heat from a possible enthalpy change caused by the phase change. Therefore, the measured data represents the apparent specific heat in this temperature range.

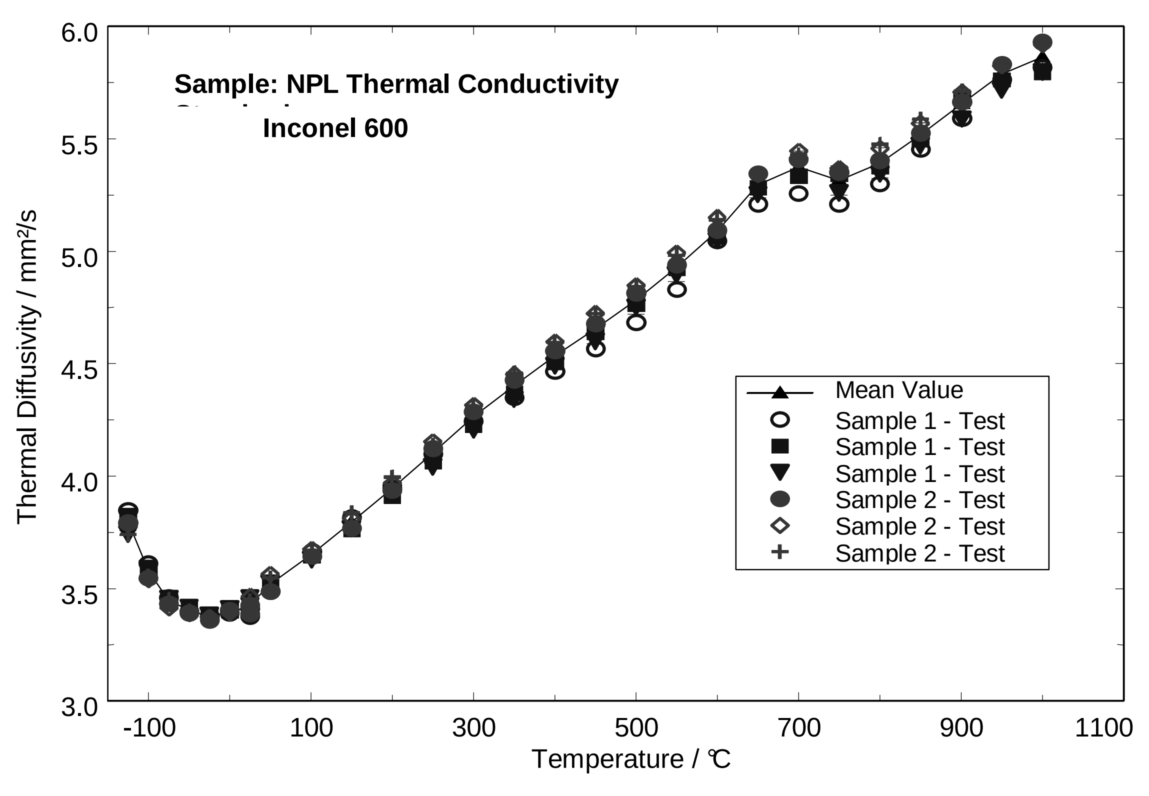

Figure 4. Thermal diffusivity of Inconel 600.

Presented in figure 4 are the thermal diffusivity results of the Inconel 600 material between –125 and 1000°C measured with the laser flash method. Again, only slight differences (±2.5%) were obtained between the different runs and the different samples. From –125 to –25°C, a decrease in the results was measured. Around –25°C, a minimum was measured in the test data. Above room temperature, the thermal diffusivity increases versus temperature. Between 550 and 700°C, an overlapping maximum was obtained in the results. The reason for this effect can again be found in the formation of Ni_3Cr clusters.

Presented in figure 5 are the thermal conductivity values calculated by multiplying density, specific heat and thermal diffusivity. Additionally shown are the certified values from NPL (Redgrove, 2003) between 50 and 500°C. The error bars on the certified values shown in the curves represent the uncertainty mentioned in the NPL certificate (±4%) and the uncertainty of the tests carried out in this work (approx. 3.5%). This uncertainty was determined on the basis of the typical uncertainty of the instruments used for the measurements. Within the uncertainty of the standard material and the accuracy of the tests, both values agree quite well in the overlapping temperature range. The critical range for the thermal conductivity determination was the temperature range between 550 and 700°C. Here, the calculated thermal conductivity shows on overlapped effect. Due to the fact that the cluster formation occurs in this temperature range, the results represent only the apparent thermal conductivity. Not considering a possible overlapped phase transition enthalpy in the specific heat, the true thermal conductivity might follow a nearly linear increase in this temperature range as indicated as a dashed line in figure 5. The values of the different measured thermophysical properties and the calculated thermal conductivity are summarized in table 2.

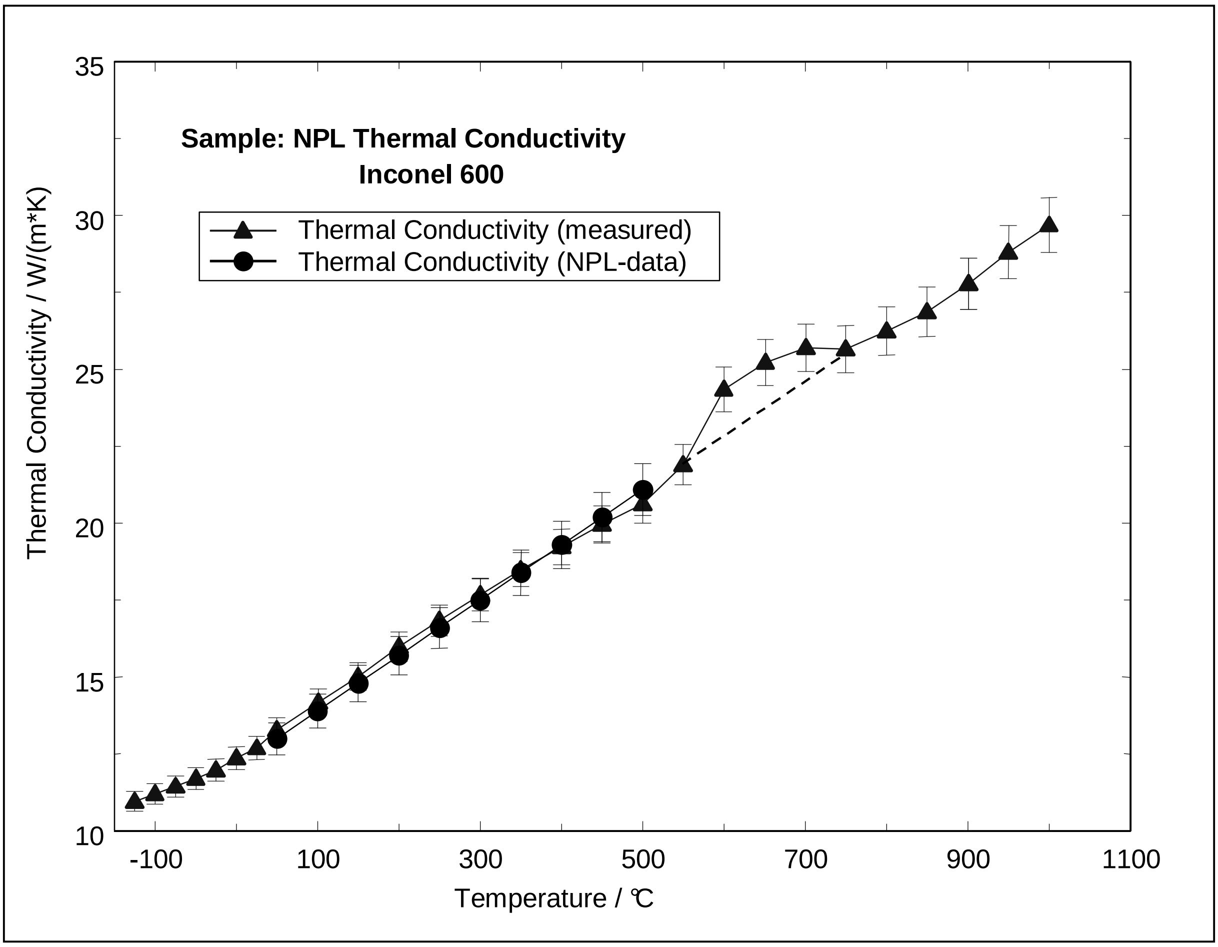

Figure 5. Thermal conductivity of Inconel 600 (comparison of the test results and NPL data).

Table 2. Thermophysical Properties of Inconel 600.

Temperature / °C	Thermal Diffusivity / mm²/s	Specific Heat / J/(g*K)	Density / g/cm³	Thermal Conductivity / W/(m*K)
-125	3.801	0.344	8.383	10.961
-100	3.577	0.374	8.377	11.207
-50	3.405	0.411	8.363	11.704
0	3.397	0.436	8.348	12.364
25	3.428	0.444	8.340	12.694
50	3.519	0.453	8.332	13.283
100	3.653	0.467	8.315	14.184
150	3.794	0.477	8.297	15.016
200	3.947	0.489	8.279	15.980
250	4.102	0.497	8.261	16.842
300	4.264	0.503	8.242	17.678
350	4.400	0.511	8.223	18.488
400	4.534	0.517	8.203	19.229
450	4.654	0.524	8.183	19.957
500	4.783	0.528	8.164	20.617
550	4.926	0.546	8.143	21.902
600	5.090	0.589	8.122	24.350
650	5.295	0.588	8.100	25.220
700	5.375	0.592	8.078	25.705
750	5.315	0.599	8.056	25.648
800	5.392	0.606	8.034	26.250
850	5.517	0.608	8.011	26.871
900	5.657	0.615	7.987	27.788
950	5.788	0.625	7.963	28.805
1000	5.863	0.638	7.937	29.689

Table 3. Comparison of the Thermal Conductivity of Inconel 600 (NPL data and this work)

Temperature / °C	Thermal Conductivity (NPL) / W/(m*K)	Thermal Conductivity (This Work) / W/(m*K)	Deviation / %
50	13.000	13.282	2.172
100	13.900	14.184	2.046
150	14.800	15.016	1.459
200	15.700	15.980	1.784
250	16.600	16.842	1.456
300	17.500	17.678	1.015
350	18.400	18.488	0.478
400	19.300	19.229	-0.368
450	20.200	19.957	-1.203
500	21.100	20.617	-2.291

Presented in table 3 is a comparison of the thermal conductivity values determined from the measurement of the room-temperature bulk density, thermal expansion, specific heat and thermal diffusivity and the values presented in the NPL certificate. As can be seen, the maximum deviation between the mean values of the test results and the certified values was 2.3%.

Conclusion

Different thermophysical properties of Inconel 600 such as density, specific heat and thermal diffusivity were measured using pushrod dilatometry, differential scanning calorimetry and the laser flash technique. Multiplying the different thermophysical properties allowed determination of the thermal conductivity. Comparing the results with the data of the corresponding NPL certificate gives an insight into the accuracy of the techniques employed. The resulting data can be the basis for extension of the temperature range for this thermal conductivity standard and for using it as a multiply properties standard material. However, the results clearly show that the temperature range between 550 and 700°C is critical for this kind of material and should therefore not be considered for cross checks or calibration processes.

References

Parker W J, Jenkins R J, Butler C P and Abbott G L, 1961, *J. Appl. Phys.* **32** 1679-1684

Redgrove J, 2003, *Certificate of Calibration – Thermal Conductivity of NPL Reference Materials Inconel 600*, National Physical Laboratory, Teddington

Blumm J, Kaisersberger E, 2001, *J. Therm. Anal. Cal.* **64** 385-391

Richter F, Born L, 2004, Materialwissenschaft und Werkstofftechnik, **17**, 11 233-237