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Measurement of Selected Thermophysical Properties of the NPL Certified Reference Material Stainless Steel 310

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Flash methods 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 checked 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, Stainless Steel 310. Tests were carried out between −125 and 1000°C.

KEY WORDS: certified reference material; laser flash method; stainless steel 310; thermal conductivity; thermal diffusivity.

1. INTRODUCTION

Stainless Steel 310 combines excellent high-temperature properties with good ductility and weldability and is designed for high-temperature services. It resists oxidation in continuous service at temperatures up to

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Table I. Composition of the Stainless Steel 310 Standard Material

Material	Fe	Cr	Ni	Mn	Si	Others
(mass%)	52.3	25.1	20.4	1.5	0.6	0.1

1150°C. Like other austenitic steels, stainless steel has excellent toughness, even down to cryogenic temperatures. Stainless Steel 310 specimens analyzed in this work were supplied by the National Physical Laboratories (NPL), Teddington, United Kingdom, as a certified thermal conductivity reference material. The chemical composition of the material is listed in Table I.

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 [1]. The room-temperature bulk density of the material is $7.830\,\mathrm{g \cdot cm^{-3}}$ (measured by characterization of mass and volume of a larger block) at room temperature. Stainless Steel 310 material was thermally treated at 1050°C for 1 h prior to certification at NPL and the tests carried out in this work.

A DIL 402 C pushrod dilatometer was employed to determine the thermal expansion and density change ρ as a function of temperature T 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. From the measured data, the thermal conductivity λ of the material was determined according to the following equation:

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

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.

2. EXPERIMENTAL

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

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 measurements on the stainless steel 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 $3\,K \cdot min^{-1}$. 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 two times 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 Stainless Steel 310 was measured using NETZSCH Models DSC 404 C Pegasus and DSC 204 Phoenix differential scanning calorimeters. The DSC 204 was used for tests between −125 and 100°C. The DSC 404 C was used for 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 an inert gas atmosphere during heating at heating rates between 10 and $20\,K \cdot min^{-1}$. Evaluation of the specific heat was carried out employing the ratio method. Technical details regarding the instruments and evaluation technique can be found elsewhere [2].

The thermal diffusivity was measured employing a NETZSCH model LFA 457 MicroFlash laser-flash apparatus [3]. 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°C while the system was equipped with the liquid-nitrogen cooled low-temperature furnace. The tests between room temperature and 1000°C were made in steps of 50°C. 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 Eq. (1). The resulting thermal conductivity was compared to values from the certificate.

3. RESULTS AND DISCUSSION

Presented in Fig. 1 are the mean thermal expansion results for stainless steel 310 as well as the deviation from the mean values of the individual runs. It can be seen that the material expands with a slightly increasing

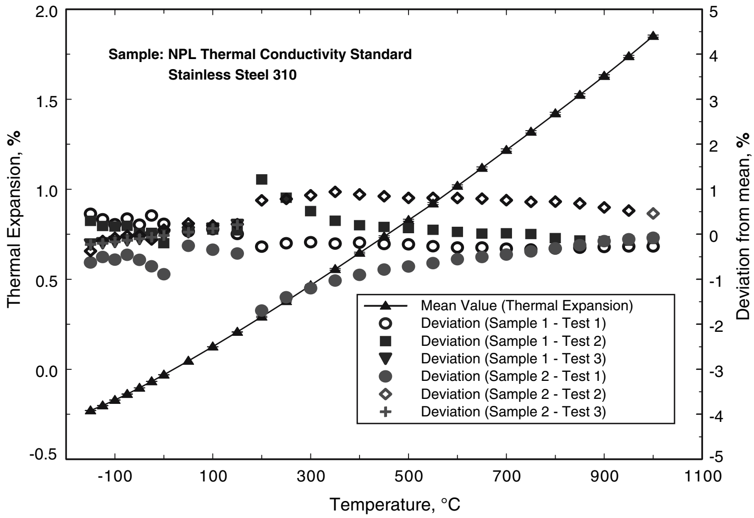

Fig. 1. Thermal expansion of Stainless Steel 310 (mean values) and deviations of the individual results from the mean values.

rate of expansion versus temperature. The repeatability for the different test runs on the same sample was within the uncertainty of the instrument (generally within ±1%). No significant differences were obtained between the results of the two samples tested.

Presented in Fig. 2 are the volumetric expansion and density change of the Stainless Steel 310 material. The volumetric expansion was calculated using the mean values of the thermal expansion results according to the following equation

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

The density was calculated using a room-temperature bulk density of $7.830\,\text{g}\cdot\text{cm}^{-3}$ 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 $7.878\,\text{g}\cdot\text{cm}^{-3}$ at −125°C to $7.411\,\text{g}\cdot\text{cm}^{-3}$ at 1000°C.

Shown in Fig. 3 is the measured mean specific heat of Stainless Steel 310 between −125 and 1000°C. As can be seen in the plot, the differences between the individual runs are generally in the range of ±2%, which corresponds to the typical accuracy of the DSC systems used for the tests. In the low-temperature range, a strong increase in the specific heat results

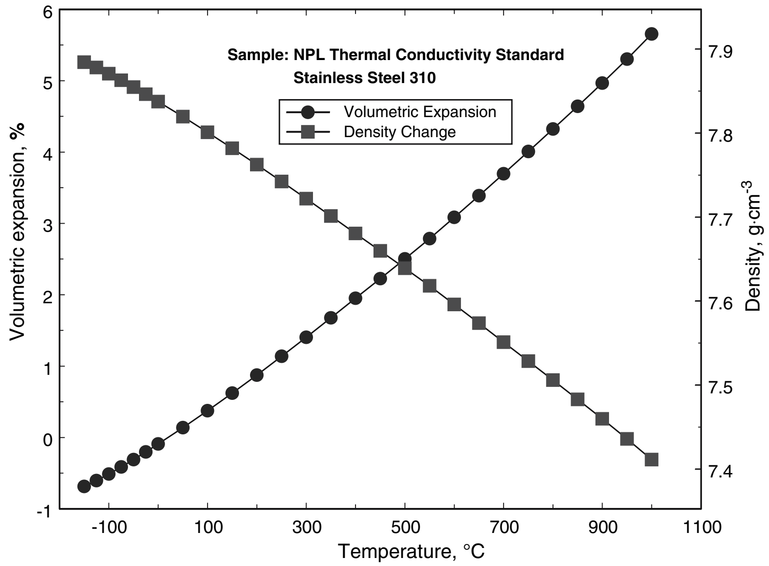

Fig. 2. Volumetric expansion and density of Stainless Steel 310.

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 is probably due to the dissolution of Ni_3Cr clusters causing an additional contribution to the specific heat [4]. It must 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.

Presented in Fig. 4 are the thermal-diffusivity results (mean values and deviations from the mean values) of the Stainless Steel 310 material between −125 and 1000°C measured with the laser flash method. Again, only slight differences (±2%) were obtained between the different runs and the different samples. From −125 to −100°C, a slight decrease in the results was measured. Around −100°C, a minimum was measured in the test data. Above −100°C, the thermal diffusivity increases with temperature. Between 600 and 700°C, an overlapping maximum was obtained in the results. The reason for this effect can again be found in the dissolution of Ni_3Cr clusters.

Presented in Fig. 5 are the thermal conductivity values calculated by multiplying the density, specific heat, and thermal diffusivity. Additionally shown are the certified values from NPL [1] between 50 and 500°C. The error bars on the certified values shown in the curves represent the

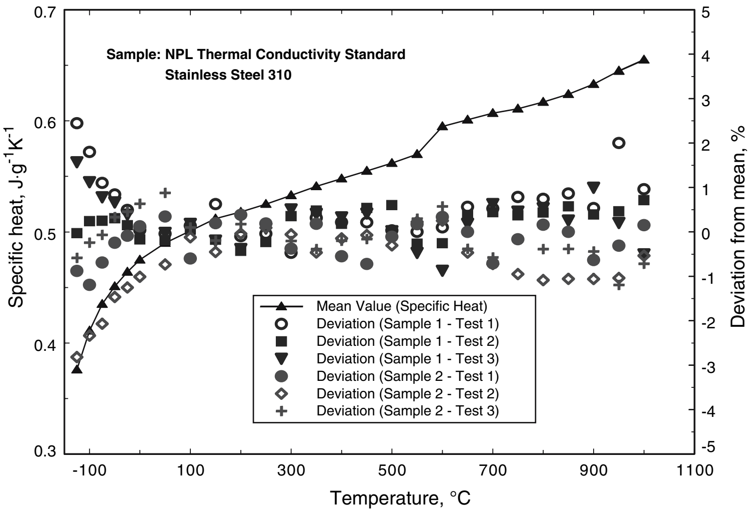

Fig. 3. Apparent specific heat of Stainless Steel 310 (mean values) and deviations of the individual runs from the mean value.

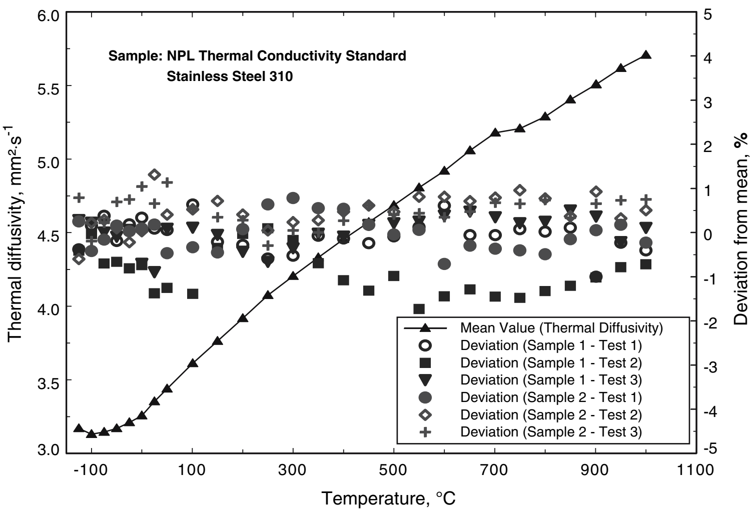

Fig. 4. Thermal diffusivity of Stainless Steel 310 (mean values and deviations of the individual results from the mean values).

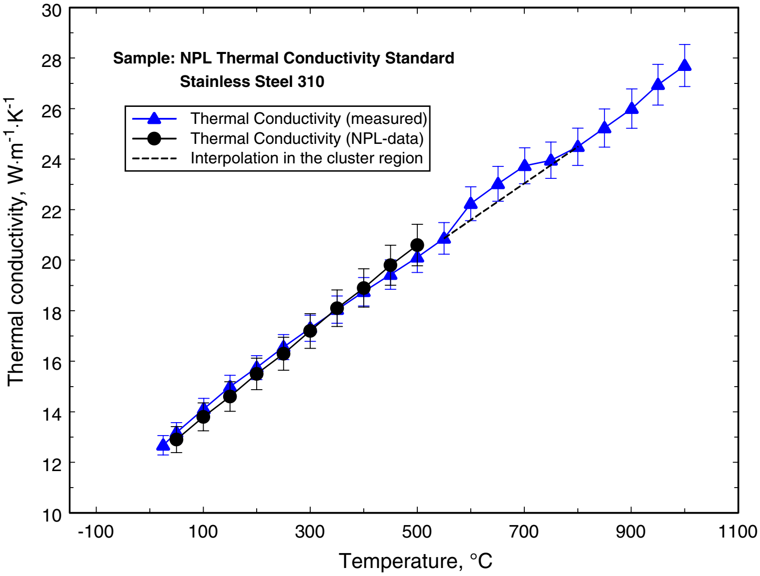

Fig. 5. Thermal conductivity of Stainless Steel 310 (comparison of the calculated results and NPL reference data).

uncertainty mentioned in the NPL certificate (±4%) and the uncertainty of the tests carried out in this work (approx. ±3%). 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 an overlapping 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 overlapping 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 Fig. 5. The values of the different measured thermophysical properties and the calculated thermal conductivity are summarized in Table II.

Presented in Table III 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

Table II. Thermophysical Properties of Stainless Steel 310 as Measured and Calculated in this Work

Temperature (°C)	Thermal Diffusivity ($mm^2 \cdot s^{-1}$)	Specific Heat ($J \cdot g^{-1} \cdot K^{-1}$)	Density ($g \cdot cm^{-3}$)	Thermal Conductivity ($W \cdot m^{-1} \cdot K^{-1}$)
−125	3.170	0.376	7.878	9.39
−100	3.130	0.411	7.871	10.12
−75	3.145	0.435	7.863	10.76
−50	3.170	0.451	7.855	11.23
−25	3.210	0.464	7.846	11.69
0	3.256	0.475	7.838	12.12
25	3.352	0.483	7.829	12.67
50	3.439	0.490	7.820	13.18
101	3.611	0.501	7.801	14.11
150	3.763	0.512	7.782	14.99
200	3.917	0.518	7.762	15.75
250	4.075	0.525	7.742	16.56
300	4.205	0.533	7.722	17.31
350	4.331	0.541	7.701	18.04
400	4.455	0.548	7.681	18.75
450	4.571	0.555	7.660	19.43
500	4.686	0.562	7.639	20.12
550	4.806	0.570	7.618	20.86
600	4.920	0.595	7.596	22.24
651	5.058	0.601	7.574	23.02
701	5.179	0.607	7.551	23.74
750	5.207	0.611	7.529	23.95
800	5.288	0.617	7.506	24.49
850	5.404	0.624	7.483	25.23
901	5.506	0.633	7.460	26.00
950	5.618	0.645	7.436	26.94
1000	5.707	0.655	7.411	27.70

values was 2.7%, which is within the stated uncertainty of the certified standard material.

4. CONCLUSION

Selected thermophysical properties of Stainless Steel 310 such as density, specific heat, and thermal diffusivity were measured using pushrod dilatometry, differential scanning calorimetry and the laser flash technique, respectively. The product of the different measured thermophysical properties allowed the determination of the thermal conductivity. Comparisons

Table III. Comparison of the Thermal Conductivity of Stainless Steel 310 (NPL reference data and calculated results from this work)

Temperature (°C)	Thermal Conductivity (NPL) $(W \cdot m^{-1} \cdot K^{-1})$	Thermal Conductivity (This Work) $(W \cdot m^{-1} \cdot K^{-1})$	Deviation (%)
50	12.90	13.18	2.16
100	13.80	14.11	2.27
150	14.60	14.99	2.69
200	15.50	15.75	1.61
250	16.30	16.56	1.61
300	17.20	17.31	0.62
350	18.10	18.04	−0.31
400	18.90	18.75	−0.78
450	19.80	19.43	−1.85
500	20.60	20.12	−2.34

of the results with the data of the corresponding NPL certificate give 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. 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.

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