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Measurement of the Thermal Conductivity of Stainless Steel AISI 304L up to 550 K

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New measurements of the thermal conductivity of stainless steel AISI 304L over the temperature range 300 to 550 K are reported. To perform the measurements, the transient hot-wire technique was employed, with a new wire sensor. The sensor makes use of a soft silicone paste material and of two thin polyimide films, between the hot wires of the apparatus and the stainless steel specimen. The transient temperature rise of the wire sensor is measured in response to an electrical heating step over a period of 40 μs to 2 s, allowing an absolute determination of the thermal conductivity of the solid, as well as of the polyimide film and the silicone paste. The method is based on a full theoretical model with equations solved by a two-dimensional finite-element method applied to the exact geometry. At the 95% confidence level, the standard deviation of the thermal conductivity measurements is 0.6%, while the standard uncertainty of the technique is less than 1.5%.

KEY WORDS: AISI 304L; stainless steel; thermal conductivity; transient hot-wire.

1. INTRODUCTION

In 1983, Bogaard [1] presented a review paper on the thermal conductivity of AISI 304L between 100 and 1707 K. His paper was a compilation and critical analysis of 20 sets of data from 15 references. His "recommended" values, however, included a physically unexplained, slight inflection in the temporal change of the thermal conductivity, in contrast to the previous compilation of Chu and Ho [2], which showed no such inflection. Chu and Ho [2] had access to the same sets of data as Bogaard [1], but rejected the low data values obtained by three laboratories in the temperature range

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300 to 600 K and produced a smooth curve for the thermal conductivity of AISI 304L. Graves et al. [3] in 1991, in an attempt to investigate the anomalous behavior reported by Bogaard, measured the thermal conductivity of AISI 304L steel and found no such behavior. Since then, to our knowledge, no other measurements of the thermal conductivity of stainless steel AISI 304L have been reported.

In this work, the thermal conductivity of a piece of AISI 304L, supplied by Anter Corporation Ltd., as a Standard Reference Material traceable to NIST via SRM 1460, was measured. The technique employed for the measurements was the transient hot-wire technique, with a modified wire sensor. The temperature range covered was from 300 to 550 K, and at the 95% confidence level, the standard deviation of the thermal conductivity measurements and of the product (density × heat capacity), ρC_p, is 0.6%. The standard uncertainty [4] of the technique is better than 1.5% for the measurement of the thermal conductivity and better than 5% for the measurement of the product (ρC_p).

2. EXPERIMENTAL

The actual instrument employed for the measurement of the thermal conductivity of solids at elevated temperatures is described elsewhere [5]. In order to measure the thermal conductivity of steel, however, a new two-wire sensor was developed.

The two wires of the technique, made out of 25-μm-diameter tantalum wire of 2 and 5 cm length, placed one after the other, are spot-welded to flattened 0.5 mm diameter tantalum wires. These, in turn are spot-welded to thick metal-sheathed Chromel wires, as shown in Fig. 1. The wires are subsequently placed in a flattened silicone paste layer (high-temperature red silicone paste, BORO 650, VersaChem U.S.A.), which is sandwiched between two 25-μm-thick polyimide films (Kapton HN polyimide film, DuPont de Nemours). The whole assembly is then placed between the two pieces of AISI 304L of dimensions $10 \times 5 \times 2\ \mathrm{cm}^3$, each.

The advantages of employing a soft silicone layer were discussed in a previous publication [5, 6]. In this case, the polyimide film insured that no electrical contact existed between the wires and the steel. Furthermore, its great adhesive power to the metal produced a sensor that had no air gaps in its interface with the steel, while at the same time can easily be removed and reused. To check the absence of air gaps, the thermal conductivity of Pyroceram 9606 was measured and our previously reported values were reproduced [6].

The introduction of the 25-μm-thick polyimide film results in one more heat transfer equation to be solved, together with the previously

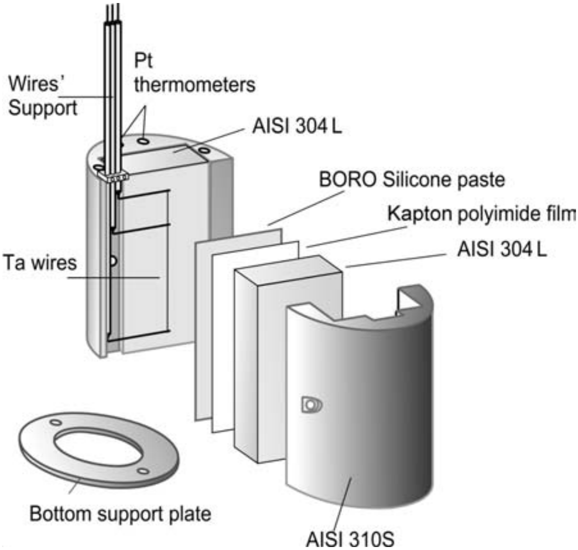

Fig. 1. Wire sensor arrangement.

described ones [5]. Hence, the full set of equations refers to the heat transfer (a) in the wire, (b) in the silicone paste, (c) in the polyimide film, and (d) in the solid, with equivalent initial and boundary conditions. This set, as described before [5], was solved by a finite-element method for the exact geometry of the sensor.

The wire sensor arrangement with the two AISI 304L steel blocks, is held together in two semi-cylinder parts made of AISI 310S steel (see Fig. 1). The whole arrangement is consequently placed in the center of an accurate, vertical three-zone tubular furnace (Model TVS 12, Carbolite), and two Class-1 calibrated platinum-resistance thermometers embedded on the top and bottom of the half cylinder are used to record the temperature.

The wires are heated over a period of 40 μs to 2 s by electrical current, and the thermal conductivity is determined in an absolute way from the transient temperature rise of the wire. In order to heat the wires and measure their resistance at the same time, a computer-controlled Wheatstone bridge is employed [5]. The characteristics of the two intermediate layers (silicone paste and polyimide film) are evaluated from measurements at short times (typically: $t < 0.4$ s for the silicone paste and $0.4 < t < 0.8$ s for the polyimide), whereas those of the metal are consequently derived essentially independently, from measurements at longer times (typically: $t > 0.8$ s). Hence, the thermal conductivity, λ, and the product (ρC_p), of the metal and the two intermediate layers, as well as the thickness of the silicone layer are uniquely determined from the five hundred measurements of the temperature rise accumulated during one run. Temperature rises employed are between 3 to 4 K over a maximum period of 2 s.

3. MEASUREMENTS

3.1. Validation of Technique

The standard uncertainty of the measurement of the resistance of the wires is a function of the uncertainties of the time intervals and the associated voltage applied [5]. Time intervals are measured with a precision of ±1 μs, while voltages are registered with a precision of 1 μV. The final result is also influenced by the standard uncertainty of the platinum resistance thermometers. These have been calibrated with a standard uncertainty of ±20 mK. Accounting for a number of other small errors, such as the measurements of the wire lengths and the temperature coefficient of resistance of tantalum, as well as errors associated with the finite-element analysis employed, it is estimated that the technique has a standard uncertainty of better than 1.5% in the measurement of the thermal conductivity, and better than 5% in the measurement of the product (ρC_p).

An important advantage of the proposed configuration is that it can also be employed to measure the thermal conductivity of fluids. So, the wires in their support, before being placed in the silicone layer, were placed in toluene at 295.15 K and the thermal conductivity, λ, and the product (ρC_p) obtained, were in excellent agreement with the literature values. Liquid toluene has been proposed by the Subcommittee on Transport Properties of the International Union of Pure and Applied Chemistry as a standard with an uncertainty of 0.5% [7].

3.2. Results and Discussion

The two blocks of AISI 304L were provided by Anter Corporation, U.S.A. Table I lists the chemical composition of the specimens, as provided by Anter Corporation U.S.A. In the same table, the composition of the specimen employed by Graves et al. [3] is also shown. It can be seen that the two specimens are of very similar composition.

In Fig. 2 the excellent agreement of the experimental results and the values predicted by FEM curves, indicative of the accurate modeling of the surfaces involved and the absence of any other layer, such as an air gap, is shown.

Table I. Chemical Composition (mass %) of Various Steels

Element	AISI 304L typical composition	AISI 304L measured by Graves et al. [2]	AISI 304L measured in this work
C	0.03 max	0.022	0.02
Si	1.0	0.41	0.40
Mn	2.0	1.81	1.73
P	0.045	0.025	0.027
S	0.03	0.008	0.029
Ni	8–12	9.20	9.03
Cr	18–20	18.10	18.22
Mo			0.14
Cu			0.47
N			0.04

As has already been discussed, the proposed technique makes use of a soft silicone paste layer and two polyimide films. The results for the thermal conductivity (λ) and the product (ρC_p) of the metal and of the two intermediate layers (silicone paste and polyimide) are shown in Table II. Measurements were performed up to 550 K, which is the starting temperature for the solidification of the paste. Values in brackets of the product

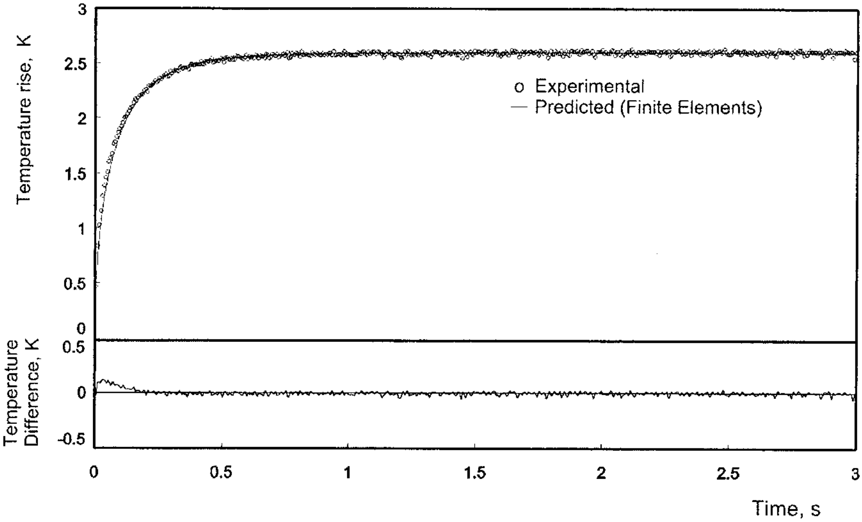

Fig. 2. Experimental and predicted temperature rise in AISI 304L at 536.73 K.

Table II. Measured Properties of AISI 304L, of Silicone Paste, and of Polyimide Film as a Function of Temperature

T (K)	λ ($W \cdot m^{-1} \cdot K^{-1}$)	$100(\lambda_{exp} - \lambda_{fit})/\lambda_{fit}$ (%)	(ρC_p) ($kJ \cdot m^{-3} \cdot K^{-1}$)	λ ($W \cdot m^{-1} \cdot K^{-1}$)	(ρC_p) ($kJ \cdot m^{-3} \cdot K^{-1}$)	λ ($W \cdot m^{-1} \cdot K^{-1}$)
		AISI 304L		Red Silicone Paste		Polyimide
306.834	14.34	−0.55	3672	0.1820	10660	0.1200
325.896	14.94	0.64	3712	0.1825	11490	0.1215
364.493	15.66	−0.03	3767	0.1827	13780	0.1225
374.189	15.84	−0.13	3799	0.1829	14820	0.1230
398.415	16.40	0.38	(3822)	0.1831	(15704)	0.1240
422.824	16.74	−0.33	(3861)	0.1862	(16900)	0.1255
452.205	17.32	0.03	3909	0.1869	17680	0.1265
481.939	17.78	−0.15	4001	0.1875	18150	0.1268
509.320	18.23	−0.02	(4055)	0.1882	(18400)	0.1271
536.730	18.60	−0.16	4140	0.1884	19030	0.1272
545.573	18.79	0.20	4174	–	–	0.1273

(ρC_p) are of slightly higher uncertainty due to electrical noise. In Table II only the thermal conductivity of the polyimide film is reported, since the product of the (ρC_p) was found constant with temperature and equal to 1548 $kJ \cdot m^{-3} \cdot K^{-1}$ [8].

The measured thermal conductivity values of AISI 304L, λ ($W \cdot m^{-1} \cdot K^{-1}$), shown in Table II, were fitted as a function of the absolute temperature T (K) to a second-order equation as

$$\lambda = \lambda(298.15\ \mathrm{K}) \sum_i a_i \left(\frac{T}{298.15} \right)^i, \tag{1}$$

where the coefficients a_i and the values of λ (298.15 K) are shown in Table III. The maximum deviation of the experimental points presented in Table I, from the above equation, is 0.64%. At the 95% confidence level, the standard deviation of the thermal conductivity measurements of AISI 304L is

Table III. Coefficients of Eq. (1)

λ (298.15 K) ($W \cdot m^{-1} \cdot K^{-1}$)	14.22
a_0 (–)	0.3989
a_1 (–)	0.7200
a_2 (–)	−0.1188

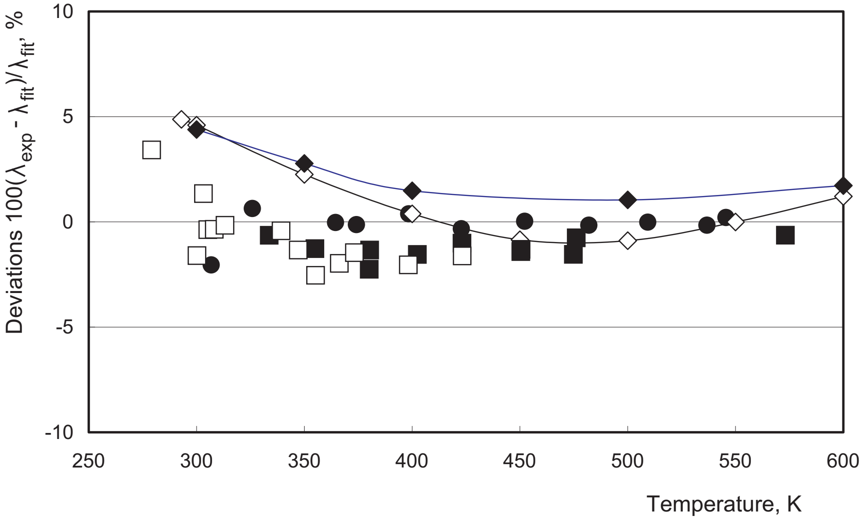

Fig. 3. Percentage deviations of the thermal conductivity measurements of AISI 304L as a function of temperature, from the values calculated by Eq. (1). (●) Present work; (◇) Bogaard [1]; (◆) Chu and Ho [2]; Graves et al. [3]: (☐) Springfields Laboratory values; (■) Oak Ridge National Laboratory values.

0.6%, and of the product (ρC_p) is also 0.6%, which are well within the standard uncertainties of the technique.

In Fig. 3, the deviations of the present data, as well as those of other investigators, from the values calculated by Eq. (1), are shown:

(a) The AISI 304L thermal conductivity recommended values by Bogaard [1], based on an average over all the experimental data from 15 references, and a quoted uncertainty of 4% (no confidence level is specified), show good agreement with the present set. There is, however, a distinct difference of slopes between the two data sets.

(b) The values reported by Chu and Ho [2] with an average uncertainty of 5% (no confidence level is specified) are also shown in the same figure. As already discussed, Chu and Ho [2] had access to the same sets of data as Bogaard [1], but rejected the low data values obtained by three laboratories in the temperature range 300 to 600 K and produced a smooth curve for the thermal conductivity of AISI 304L. The present set of measurements is in excellent agreement with these values.

(c) As already mentioned, Graves et al. [3], in order to investigate the anomalous slope behavior proposed by Bogaard [1], performed two sets of measurements on a sample of AISI 304L (composition shown in Table I):

- In the Oak Ridge National Laboratory a high-temperature longitudinal apparatus was employed to measure the thermal conductivity between 300 and 1000 K.
- In the Springfields Laboratory, a laser flash apparatus was used to measure the thermal diffusivity, between 300 and 420 K.

The thermal conductivity and diffusivity measurements, reported by Graves et al. [3] with quoted uncertainties of 1.5% and 2%, respectively (no confidence level is specified), are also in excellent agreement with the present set of measurements. Furthermore, the anomalous behavior reported by Bogaard [1] was not observed.

From the above presentation it is apparent that the present set of thermal-conductivity values agree well with the four previous sets of measurements.

4. CONCLUSIONS

A novel application of the transient hot-wire technique for measurements of the thermal conductivity of electrically conducting solids up to 550 K, has been described. The tantalum wires of the technique were placed, firstly in a soft silicone layer, then sandwiched between two polyimide films, and finally between the metal blocks. This arrangement allows, at very small times, the calculation of the unknown properties of the soft paste and of the polyimide film and with these values, at larger times, the calculation of the thermal conductivity and the product (ρC_p) of the stainless steel. The method is based on a full theoretical model with equations solved by finite elements for the exact geometry. At the 95% confidence level, the standard deviation of the thermal conductivity measurements as well as of the product (ρC_p) is 0.6%. As already discussed, the technique has a standard uncertainty of better than 1.5% in the measurement of the thermal conductivity and better than 5% in the measurement of the product (ρC_p).

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