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Measurements of Specific Heat and Electrical Resistivity of Austenitic Stainless Steel (St. 1.4970) in the Range 300–1500 K by Pulse Calorimetry[1]

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A direct heating pulse calorimetric technique has been applied for the measurements of specific heat, electrical resistivity, and hemispherical total emissivity of austenitic stainless steel (St. 1.4970), a candidate for thermal conductivity standard reference material. The specific heat and electrical resistivity were measured in the range 300 to 1500 K, and the hemispherical total emissivity was measured in the range 1300 to 1500 K. The maximum measurement uncertainties were estimated to be 3% for specific heat, 1% for electrical resistivity, and 5% for emissivity.

KEY WORDS: austenitic stainless steel; electrical resistivity; hemispherical total emissivity; pulse heating calorimetry; specific heat.

1. INTRODUCTION

Austenitic stainless steel (St. 1.4970) was among one of the first materials selected by the Thermophysics Working Group of the German Ceramic Society as candidates for thermophysical property standard reference materials. This material was extensively studied within the cooperative measurements program organized by the group. The results of this study were evaluated by Binkele [1]. The differences between the results of six participants in the measurement of specific heat stimulated additional measurements at the Boris Kidrich Institute using direct heating pulse calorimetry.

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2. MEASUREMENTS

The austenitic stainless steel (St. 1.4970) specimens were supplied by Fa. Roechling, Voelkingen/Saar. All the specimens used for cooperative measurements were taken from the same batch of material, whose chemical composition fully conformed with Deutsche Industrie Normal (DIN) standards. Table I presents both the DIN specification and the results of chemical analysis of the batch (manufacturer's denomination 53790/ Roechling), from which all specimens for cooperative studies were machined [1].

Our measurements were performed on the specimen which was previously subjected to specific heat measurements at Physikalisch-Technische Bundesanstalt in Braunschweig (PTB) by Hanitzsch [2]. The specimen diameter was 2.97 mm, and the total length 379.5 mm. Our pulse measurements were performed on the specimen as received from PTB, without any additional thermal treatment.

The direct pulse heating calorimetric technique developed at the Boris Kidrich Institute [3] was used for the measurements. This method enables simultaneous measurements of specific heat and electrical resistivity in the temperature range from 300 to 1900 K. In addition, this technique enables determination of hemispherical total emissivity above 1000 K. Evaluation of the method, including detailed analysis of measurement uncertainties is given in an earlier publication [4].

A total of five pulse heating experiments was performed on the St. 1.4970 specimen, covering different temperature ranges. The basic information on these experiments is given in Table II.

Table I. Chemical Composition of Austenitic Stainless Steel (St. 1.4970)

Element	DIN specification (% by weight)	Batch 53790 (% by weight)
C	0.08– 0.12	0.09
Si	0.25– 0.45	0.45
Mn	1.60– 2.00	1.7
P	⩽0.03	0.003
S	⩽0.015	0.004
Cr	14.5 –15.5	14.6
Ni	15.0 –16.0	15.0
Mo	1.05– 1.25	1.25
Ti	0.35– 0.55	0.46
Cu		0.07
B		0.0045
Al		<0.006
Fe	Balance to 100%	

Table II. Summary of Pulse Heating Experiments

Run	Heating period (ms)	Temperature range (K)
ST1	1200	300–530
ST4	2000	300–1020
ST6	3000	300–1330
ST7	3000	460–1450
ST10	3000	600–1520

3. EXPERIMENTAL RESULTS

3.1. Specific Heat

The final averaged values of specific heat were obtained in the 300–1500 K range. Deviation of the individual C_p curves from the smoothed curve obtained by spline fitting was generally within $\pm 2\%$. The maximum uncertainty in the specific heat results was estimated to be 3%. Our final results as well as the results of six participants in the previous cooperative measurements—Shulz, Brandt, Neumann, Richter, Emmerich [1], and Hanitzsch [1, 2]—are presented in Fig. 1. Our results agree best with those obtained by Hanitzsch [2] except in the vicinity of the anomaly, around 950 K. In our measurements, the anomaly always appears at temperatures approximately 50 K higher than the above value.

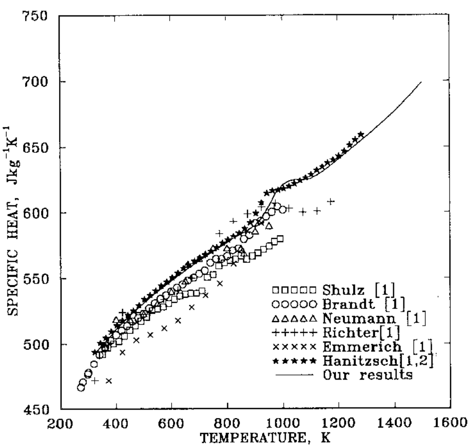

Fig. 1. Specific heat of austenitic stainless steel (St. 1.4970).

3.2. Electrical Resistivity

The final electrical resistivity values were obtained from the same set of five experiments shown in Table II. Deviation of individual results from the smoothed curve did not exceed 0.7%. The maximum measurement uncertainty was estimated to be 1%. Our final results, as well as the results of Hanitzsch obtained on the same specimen [2], are presented in Fig. 2. The difference between the two sets of results is less than 2% in the entire range.

After completion of the pulse heating experiments, additional electrical resistivity measurements were made at room temperature, using the stationary four-probe technique with current reversal. The maximum uncertainty of these measurements was estimated to be 0.5%. At 20°C, a resistivity value of $\rho_0 = 78.39 \times 10^{-8}\ \Omega \cdot \mathrm{m}$ was obtained, which agreed well with extrapolation of the results of the pulse heating experiments (deviation less than 0.5%). Subsequently, the specimen was thermally treated at 1060°C for 0.5 h and cooled in water, for comparison with the original Binkele data obtained under the same conditions [1]. The new value for electrical resistivity was $\rho_0 = 77.42 \times 10^{-8}\ \Omega \cdot \mathrm{m}$, compared with the value of $76.53 \times 10^{-8}\ \Omega \cdot \mathrm{m}$ quoted by Binkele [1].

The specific heat and electrical resistivity results for St. 1.4970 are summarized in Table III.

3.3. Hemispherical Total Emissivity

Hemispherical total emissivity was determined from the data of pulse heating experiments at temperatures above 1300 K. The results are presen-

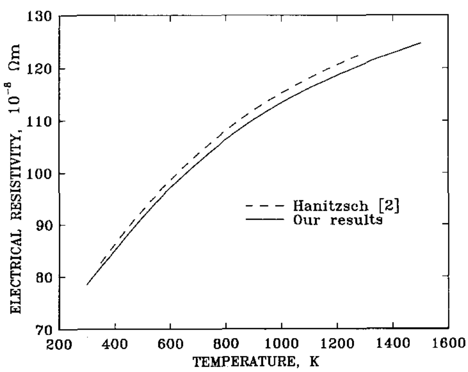

Fig. 2. Electrical resistivity of austenitic stainless steel (St. 1.4970).

Table III. Specific Heat and Electrical Resistivity of Austenitic Stainless Steel (St. 1.4970)

T (K)	C_p ($J \cdot kg^{-1} \cdot K^{-1}$)	ρ ($10^{-8}\,\Omega \cdot m$)
300	—	78.50
325	493	80.17
350	499	81.83
375	505	83.50
400	511	85.15
425	517	86.78
450	522	88.39
475	527	89.96
500	531	91.49
525	536	92.96
550	540	94.40
575	544	95.79
600	548	97.13
625	552	98.42
650	556	99.68
675	560	100.88
700	563	102.04
725	567	103.16
750	570	104.26
775	574	105.32
800	577	106.34
825	580	107.34
850	584	108.31
875	587	109.24
900	590	110.14
925	596	111.00
950	605	111.82
975	615	112.60
1000	621	113.36
1025	624	114.08
1050	625	114.78
1075	625	115.46
1100	626	116.12
1125	629	116.76
1150	632	117.37
1175	636	117.97
1200	640	118.56
1225	645	119.14
1250	649	119.71
1275	653	120.27
1300	658	120.81
1325	662	121.34
1350	667	121.84
1375	671	122.33
1400	676	122.81
1425	682	123.27
1450	687	123.73
1475	693	124.19
1500	699	124.64

ted in Table IV, which are in good agreement with the values obtained from the function proposed by Hanitzsch [2]:

$$\varepsilon = 0.1341 + 1.6659 \times 10^{-4} t - 1.8030 \times 10^{-8} t^2 \tag{1}$$

where t is temperature in °C. The maximum uncertainty in our hemispherical total emissivity results is estimated to be 5% [4].

4. DISCUSSION

Our specific heat results are in good agreement with the results of Hanitzsch [2], whose measurements preceded ours on the same specimen. They are approximately 1% above the recommended values given by Binkele [1]. The updated report by Binkele [5], which was received in the final stage of the preparation of our paper, contains new recommended functions for C_p based on additional experimental results. These functions are as follows:

$50 < t < 600°C$,

$$C_p = 0.4820 + 2.3077 \times 10^{-4} t - 9.5870 \times 10^{-8} t^2 \tag{2}$$

$600 < t < 700°C$,

$$C_p = 12.0362 - 5.47199 \times 10^{-2} t + 8.65981 \times 10^{-5} t^2 - 4.5341 \times 10^{-8} t^3 \tag{3}$$

$700 < t < 1020°C$,

$$C_p = 0.6806 - 2.5910 \times 10^{-4} t + 2.3305 \times 10^{-7} t^2 \tag{4}$$

where C_p is in $J \cdot g^{-1} \cdot K^{-1}$ and t is in °C.

The values obtained from these functions virtually overlap our results. The only significant difference is in the position of the anomaly expressed

Table IV. Hemispherical Total Emissivity of Austenitic Stainless Steel (St. 1.4970)

T (K)	ε
1320	0.295
1410	0.292
1440	0.292
1460	0.299
1490	0.309

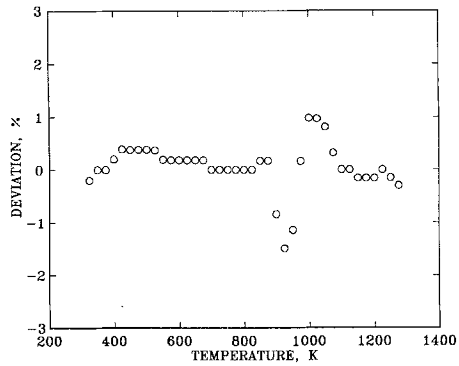

Fig. 3. Deviation of our specific heat results from those expressed by Eqs. (2), (3), and (4).

as the wide peak on the C_p curve in the 900–1000 K range. The peak in our work is shifted toward higher temperatures by approximately 50 K, probably due to the nature of our dynamic measurement technique. The complex processes in the structure of this high-alloy steel could be affected by the heating rates used in our measurements. Deviations of our results from the recommended functions [Eqs. (2)–(4)] are shown in Fig. 3. Compared with the cooperative measurements, our results have provided additional information on specific heat in the 1293–1500 K range.

The only available electrical resistivity results for St. 1.4970 over a wide temperature range were those obtained by Hanitzsch [2] for the same specimen. Comparison of the two sets in the range 350–1270 K shows that both results follow a similar temperature dependence. Our results are 1 % lower at the lowest temperature; the difference increases to 1.7 % at the highest temperature. As indicated in the foregoing section, our room temperature electrical resistivity value was 2.4 % higher than the corresponding value quoted by Binkele [1]; the difference decreased to 1.2 % after thermal treatment of the specimen.

The hemispherical total emissivity measurements were not a part of the primary objective of this experimental investigation. Experimental data obtained in the pulse heating experiments enabled determination of hemispherical total emissivity above 1300 K, which confirmed the results obtained by Hanitzsch [1, 2].

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