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Thermophysical Measurements on Iron Above 1500 K Using a Transient (Subsecond) Technique

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Simultaneous measurements of heat capacity, electrical resistivity and hemispherical total emittance of iron (99.9% pure) in the temperature range 1500 to 1800 K, and the melting point of iron by a subsecond duration, transient technique are described. The measurements indicate increases in heat capacity and electrical resistivity as the result of the solid-solid phase transformation ($\gamma \rightarrow \delta$) in iron. The measured value of the hemispherical total emittance at 1720 K is 0.33. The average of the results of two experiments yield a value of 1808 K for the melting point of iron. Estimated inaccuracies of measured properties are: 3 percent for heat capacity and emittance, 1 percent for electrical resistivity, and 5 K for the melting point.

Key words: Electrical resistivity; emittance; heat capacity; high-speed measurements; high temperature; iron; melting point; phase transformation; thermodynamics; thermophysical properties.

1. Introduction

Most of the reliable measurements of thermophysical and related properties of iron reported in the literature were performed at temperatures below 1500 K. Even at these temperatures, considerable disagreements exist between the results of various investigations. To a large extent, these disagreements may be attributed to difficulties associated with the magnetic and allotropic transformations in iron. The objective of the present work is to measure selected properties (heat capacity, electrical resistivity, hemispherical total emittance, and melting point) of iron above 1500 K using a transient technique.

The method is based on rapid resistive self-heating of the specimen from room temperature to high temperatures in less than one second by the passage of an electrical current pulse through it; and on measuring with millisecond resolution, such experimental quantities as current through the specimen, potential drop accross the specimen, and specimen temperature. Details regarding the construction and operation of the measurement system, the formulation of relations for the properties, the methods of measuring experimental quantities, and other pertinent information are given in earlier publications [1, 2] [1].

2. Measurements

Specimens: The specimens were tubes fabricated from rods[2] by removing the center portion using an electro-erosion technique. The nominal dimensions of the specimens were: length, 102 mm; outside diameter, 6.3 mm; and wall thickness, 0.5 mm. The outer surfaces of the specimens were polished to reduce heat loss due to thermal radiation.

Procedure: Because of the $\gamma \rightarrow \delta$ phase transformation in iron, the measurements were performed in the following two ranges: low (1500–1660 K), and high (1700–1800 K). Four experiments were conducted on two specimens; two experiments in the low range and two experiments in the high range. The duration of the current pulses in the experiments ranged from 500 to 900 ms, and the heating rate ranged from 2000 to 3500 $K \cdot s^{-1}$. Radiative heat loss from the specimen was, in all cases, less than 2 percent at 1500 K and less than 4 percent at 1800 K of the input power. All the experiments were conducted with the specimen in a vacuum environment of approximately 10^{-5} torr.

3. Experimental Results

The properties reported in this paper are based on the International Practical Temperature Scale of 1968 [3]. In all computations, the geometrical quantities are based on their room temperature (298 K) dimensions. The experimental results for the heat capacity and the electrical resistivity of iron (for each phase) are presented as linear functions in temperature. These functions were obtained by least squares approximation of the individual points corresponding to the measurements on the two specimens. The final values of the properties at 20 degree temperature intervals computed from these functions are presented in table 1. The results of individual experiments are given in the appendix.

[1] Figures in brackets indicate the literature references at the end of this paper.
[2] The specimens in rod form were furnished by the Office of Standard Reference Materials (OSRM) of NBS (Standard Reference Material 734–S, Electrolytic Iron, 99.9 % pure). The details regarding the impurities are documented by OSRM.

TABLE 1. *Heat capacity and electrical resistivity of iron*

Phase	Temperature (K)	$c_p(\mathrm{J \cdot mol^{-1} \cdot K^{-1}})$	$\rho(10^{-8}\Omega \cdot \mathrm{m})$
γ-iron	1500	36.12	120.66
	1520	36.37	121.10
	1540	36.61	121.54
	1560	36.86	121.98
	1580	37.11	122.42
	1600	37.35	122.86
	1620	37.60	123.29
	1640	37.84	123.73
	1660	38.09	124.17
δ-iron	1700	41.46	125.35
	1720	42.09	125.65
	1740	42.72	125.96
	1760	43.35	126.26
	1780	43.99	126.57
	1800	44.62	126.87

Heat Capacity: Heat capacity was computed from data taken during the heating period. A correction for power loss due to thermal radiation was made using the single measured value of hemispherical total emittance in conjunction with the slope of emittance versus temperature function obtained from the literature [4].

The functions for heat capacity are:
In the temperature range 1500 to 1660 K (γ-iron),

$$c_p = 17.64 + 1.232 \times 10^{-2} T \tag{1}$$

In the temperature range 1700 to 1800 K (δ-iron),

$$c_p = -12.28 + 3.161 \times 10^{-2} T \tag{2}$$

where T is in K, and c_p is in $\mathrm{J \cdot mol^{-1} \cdot K^{-1}}$. The standard deviation of experimental data from equations (1) and (2) is 0.4 percent. In the computations of heat capacity, the atomic weight of iron was taken as 55.85.

Electrical Resistivity: The electrical resistivity was determined from the same experiments that were used to calculate the heat capacity.

The functions for electrical resistivity are:
In the temperature range 1500 to 1660 K (γ-iron),

$$\rho = 87.72 + 2.196 \times 10^{-2} T \tag{3}$$

In the temperature range 1700 to 1800 K (δ-iron),

$$\rho = 99.37 + 1.528 \times 10^{-2} T \tag{4}$$

where T is in K and ρ is in $10^{-8}\Omega \cdot \mathrm{m}$. The standard deviation of experimental data from equations (3) and (4) is 0.5 percent. The electrical resistivity of one of the specimens measured at 293 K using a Kelvin bridge was $10.2 \times 10^{-8}\Omega \cdot \mathrm{m}$.

Hemispherical Total Emittance: In one of the experiments in the low temperature range, data were taken during the initial cooling period of the specimen which followed the heating period. The results yielded a value of 0.33 for the hemispherical total emittance at 1720 K.

Melting Point: The melting point of a specimen is manifested by a plateau in the temperature versus time function (fig. 1). The melting point of the specimen was determined by averaging the temperature points at the plateau. The results for two specimens (fig. 2) yielded the values 1807.5 K and 1808.6 K, with standard deviations of 0.2 K and 0.4 K, respectively. It may be concluded that the melting point of iron is 1808 K.

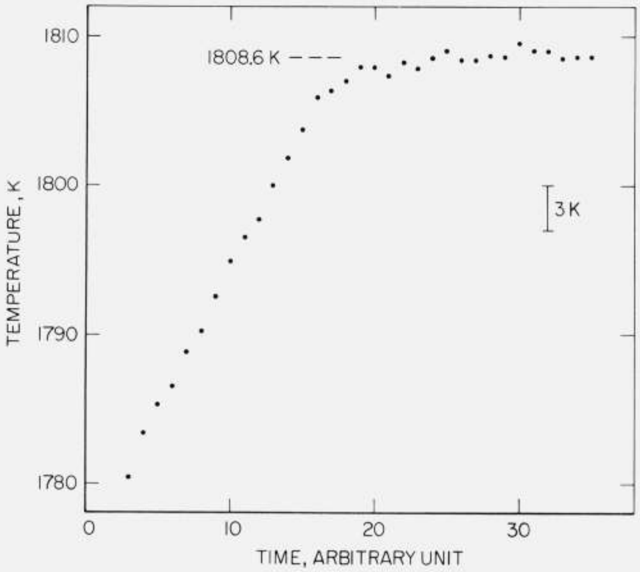

FIGURE 1. *Variation of temperature of iron (specimen 2) as a function of time near and at the melting point (1 time unit= 0.833 ms).*

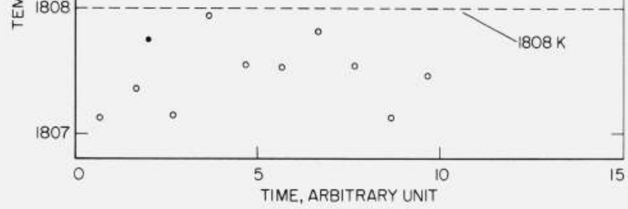

FIGURE 2. *Variation of temperatures of two iron specimens as a function of time at the melting point (1 time unit=0.833 ms).*

Estimate of Errors: The details for estimating errors in measured and computed quantities using the present measurement system are given in an earlier publication [2]. In this paper, the specific items were recomputed whenever the present conditions differed from those in the earlier publication.

The results for imprecision[3] and inaccuracy[4] in the properties are: 0.6 percent and 3 percent for heat capacity, 0.5 percent and 1 percent for electrical resistivity, 0.5 K and 5 K for the melting point. The inaccuracy in the hemispherical total emittance is estimated to be 3 percent.

4. Discussion

The heat capacity and electrical resistivity results of this work are compared graphically with those in the literature in figures 3 and 4, respectively.

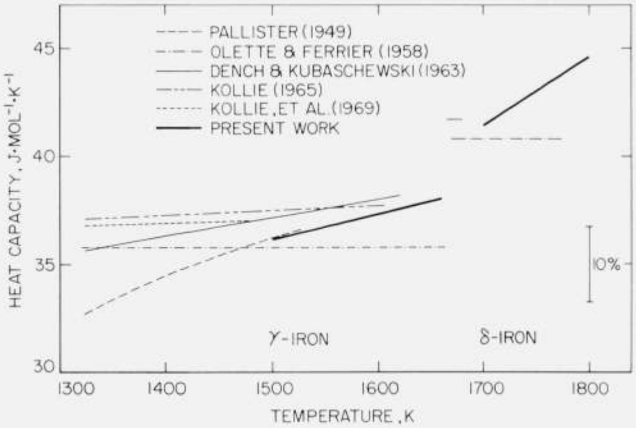

FIGURE 3. *Heat capacity of iron reported in the literature.*

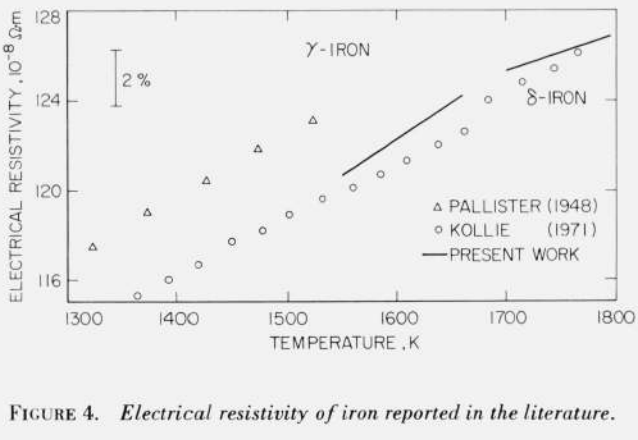

FIGURE 4. *Electrical resistivity of iron reported in the literature.*

[3] Imprecision refers to the standard deviation of an individual point as computed from the difference between measured value and that from the smooth function obtained by the least squares method.

[4] Inaccuracy refers to the estimated total error (random and systematic).

From figure 3 it may be seen that at 1300 K considerable disagreements (up to approximately 10%) exist in the heat capacity values reported in the literature; however, immediately below the $\gamma \rightarrow \delta$ transformation the disagreements (especially between the results of recent investigations) are reduced to a value less than 3 percent. In contrast to the constant values for the heat capacity of δ-iron reported in the literature, the results of the present work show that heat capacity increases with temperature as the melting point is approached.

Figures 3 and 4 show that the heat capacity and electrical resistivity are discontinuous at the transformation point. Extrapolation of the properties to the transformation temperature (1683 K, based on preliminary measurements in this laboratory) indicates increases of approximately 7 percent in the heat capacity and 0.3 percent in the electrical resistivity during the $\gamma \rightarrow \delta$ transformation. Since the time resolution of the experiments was longer than the time required for the transformation, it is not likely that the high values for heat capacity above the transformation point were due to any residual transformation energy. The actual increase in the electrical resistivity is likely to be larger than the value given above because the expansion that occurs in iron during the $\gamma \rightarrow \delta$ transformation was not included in the electrical resistivity computations.

The melting point of iron reported in the literature is given in table 2. All the tabulated values are within 6 K of each other. The maximum difference of the present value from the values reported by other investigators is 4 K.

TABLE 2. *Melting point of iron*

Investigator	Reference	Year	Melting point* (K)
Bristow	11	1939	1806±5
Chipman & Marshall	12	1940	1807
Roeser & Wensel	13	1942	1812±1
Schofield & Bacon	14	1953	1810±10
Present work	...	...	1808±5

*All temperatures are corrected to the IPTS 1968.

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5. Appendix

TABLE A-1. *Experimental results on heat capacity and electrical resistivity of iron**

Phase	Temperature K	Specimen 1		Specimen 2	
		c_p	ρ	c_p	ρ
γ-iron	1500	35.87	121.12	36.38	120.13
	1520	36.12	121.59	36.62	120.59
	1540	36.37	122.05	36.85	121.04
	1560	36.62	122.51	37.08	121.49
	1580	36.88	122.95	37.31	121.92
	1600	37.14	123.39	37.54	122.35
	1620	37.40	123.82	37.78	122.76
	1640	37.67	124.25	38.01	123.17
	1660	37.94	124.66	38.27	123.56
δ-iron	1700	41.37	125.80	41.66	124.87
	1720	41.95	126.13	42.22	125.22
	1740	42.58	126.45	42.83	125.54
	1760	43.18	126.71	43.48	125.83
	1780	43.81	127.07	44.13	126.08
	1800	44.51	127.38	44.86	126.30

*Heat capacity in $J \cdot mol^{-1} \cdot K^{-1}$, electrical resistivity in $10^{-8}\ \Omega \cdot m$.

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