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Melting Temperature of Nickel by a Pulse Heating Technique

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The melting temperature of 99.98+ % pure nickel was measured by means of a subsecond duration pulse heating technique. The results, based on IPTS-68, yield a value of 1729 K for the melting temperature with an estimated maximum uncertainty of ±4 K.

KEY WORDS: high temperature; melting temperature; nickel; pulse heating technique.

1. INTRODUCTION

The melting temperature of nickel is listed as a secondary reference point on the International Practical Temperature Scale of 1968 (IPTS-68) [1] with a value of 1728 K. The listed value is based on a conversion to the IPTS-68 of selected experimental determinations obtained prior to 1950 by visual optical pyrometry. In the present work, an independent redetermination of the melting temperature of nickel has been carried out by a rapid pulse heating technique in which temperature is measured with a high-speed photoelectric pyrometer. The short heating time (less than 1 s) of the present technique tends to minimize the problems associated with the quasi-steady-state experiments at high temperatures, in particular, specimen contamination and evaporation.

The method is based on rapid resistive self-heating of the specimen from room temperature to its melting temperature in less than 1 s by the passage of an electrical current pulse through it. The specimen temperature

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is measured at the rate of 1200 times per second with a high-speed photoelectric pyrometer [2]. The data are recorded with a digital data acquisition system, which has a time resolution of 0.4 ms and a full-scale signal resolution of about one part in 8000. Details regarding the construction and operation of the measurement system and other pertinent information, such as error analysis, etc., are given in earlier publications [3, 4].

2. MEASUREMENTS

The measurements were performed on two tubular specimens fabricated by an electroerosion technique from cylindrical rods. Nominal dimensions of the tubes were: length, 75 mm; outside diameter, 6.4 mm; wall thickness, 0.5 mm. The pyrometric temperature measurements were made by sighting through a small rectangular hole (1 × 0.5 mm) in the wall at the middle of the specimen, thereby approximating blackbody conditions. In order to compensate for the cross-sectional nonuniformity created by the

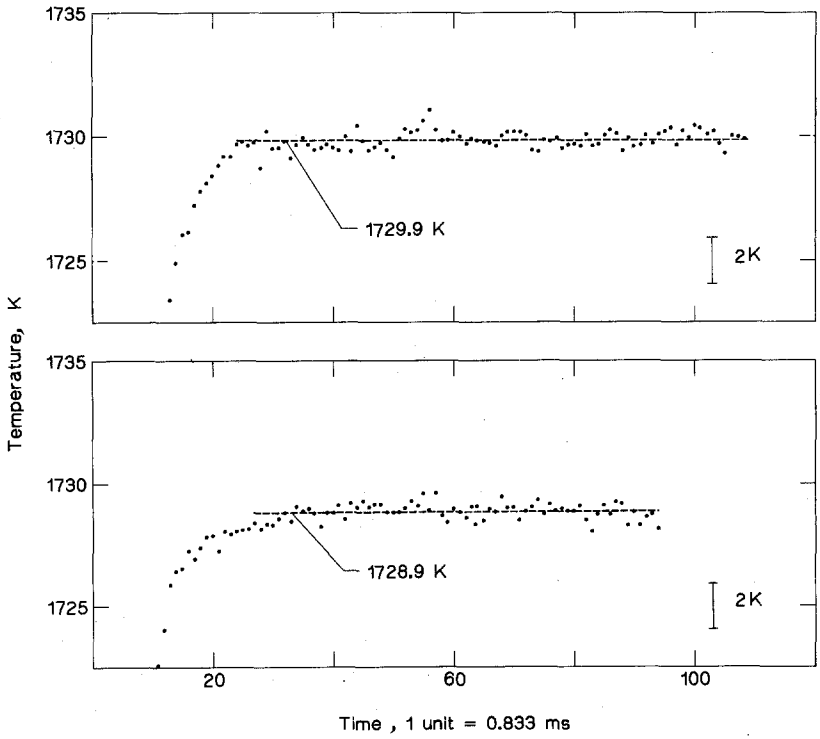

Fig. 1. Variation of the temperature of specimen 1 (upper) and specimen 2 (lower) near and at the melting temperature of nickel.

Table I. Experimental Results for the Melting Temperature of Nickel

Specimen number	Heating rate[a] ($K \cdot s^{-1}$)	Number of temperatures at plateau	Melting temperature (K)	Standard deviation (K)
1	2570	84	1729.9	0.4
2	2560	68	1728.9	0.4

[a] Derivative of the temperature versus time functions (at approximately 30 K below the melting plateau) obtained by fitting the temperature data before melting to a quadratic function in time with the least squares method.

hole, a portion of the specimen was removed by grinding a flat along the length of the tube, excluding the 1 mm length of the hole. The sighting hole was fabricated 0.8 mm off center from the tube axis to improve the blackbody quality. The heat loss due to thermal radiation was reduced by polishing the outer surface of each specimen. The results of a typical analysis furnished by the manufacturer indicated that the material was 99.98^{+} % pure with the following impurities in ppm by mass: Fe, 30; O, 20; N, 13; C, 8; Sn, < 15, In, < 6; Sr, < 5; Cl, Ge, Nd, S, Se, Sm, Ta, Te, < 3 each; the total amount of all other detected elements was less than 20 ppm, each element being below 1 ppm limit.

The experiments were conducted with the specimens in an argon environment at about 0.2 MPa (~2 atm). The specimens were heated from room temperature to their melting temperature in about 600 ms. The heating rate near the melting temperature was about 2500 $K \cdot s^{-1}$. Upon completion of the experiments, the high-speed pyrometer was calibrated against a tungsten filament reference lamp that in turn had been calibrated against the NBS Photoelectric Pyrometer by the Radiometric Physics Division at NBS. All temperatures reported in this work, except where explicitly noted otherwise, are based on IPTS-68.

Temperature of the specimens was measured near and during the melting period. The results are shown in Fig. 1. The plateau in temperature indicates the region of solid and liquid equilibrium. The melting temperature for each specimen was obtained by averaging the temperatures at the plateau. A summary of the results for the two specimens is given in Table I. The average melting temperature for the two specimens is 1729.4 K with an absolute deviation of 0.5 K. It may be concluded that the melting temperature of nickel is 1729 K.

3. ESTIMATE OF ERRORS

A detailed analysis of the sources and magnitudes of errors involved in the measurement of specimen temperature with the NBS pulse heating

Table II. Sources and Magnitudes of Errors in the Measurement of Temperature in the Range 1700–1800 K

Source	Error (K)
Reference lamp	
Calibration	2
Drift between calibrations	1
Pyrometer	
Alignment	1
Calibration	1
Specimen	
Temperature nonuniformity	3
Blackbody quality	1
Total error in temperature[a]	4

[a] Square root of the sum of squares of the individual errors.

system has been given in an earlier publication [3]. More recently, errors in temperature measurements with the same system were discussed in connection with the measurement of the melting temperature of palladium [5], which is about 100 K above that of nickel. The error contributions to the measurement of temperature in the present work arising from the reference lamp, the pyrometer, and the specimen are summarized in Table II. Assuming that the various error contributions are uncorrelated, it is concluded that the maximum error (uncertainty) in the measured melting temperature of nickel is ± 4 K.

4. DISCUSSION

Results for the melting temperature of nickel as reported in the literature are presented in Table III, along with corresponding values based on IPTS-68 for comparison with the present work. Prior to the gas-thermometer work by Day and Sosman [6], most of the early determinations of the nickel melting temperature were made by means of thermocouples (for a summary see Refs. 7 and 8). All subsequent investigations, with the exception of our work, have utilized visual optical pyrometry for the measurement of temperature.

In the experiments by Wensel and Roeser [7] and by Van Dusen and Dahl [8], melting temperatures were determined by measuring the relative brightness of blackbodies at the melting temperatures of gold and nickel, and then calculating the upper temperatures by means of Planck's law (based on accepted values for the gold point and the second radiation constant). Their results form the basis for the secondary reference point

Table III. Values for the Melting Temperature of Nickel Reported in the Literature

Investigators	Ref.	Year	Specimen purity (%)	Constants[a]		Melting point (K)	
				T_{Au} (K)	c_2 (cm · K)	As reported[b]	On IPTS-68
Day and Sosman	6	1911	99.84	—	—	1725	—
Wensel and Roeser	7	1930	99.94	1336	1.432	1728 ± 1	1728[c]
Van Dusen and Dahl	8	1947	99.84	1336	1.432	1728 ± 1	1728[c]
Schofield and Bacon	10	1953	99.86	1336.15	1.438	1724 ± 10	1726
Oriani and Jones	11	1954	99.999	1336.15	1.438	1725 ± 4	1727
Present work			99.98+	1337.58	1.4388	1729 ± 4	1729

[a] Values of T_{Au} (gold point) and c_2 (second radiation constant) used by the investigator.
[b] Except for the melting temperature reported by Day and Sosman [6], which was obtained by gas thermometry, all others were obtained by pyrometry based on the given values of T_{Au} and c_2.
[c] Basis for the secondary reference point value of 1728 K given in the extended list of IPTS-68 [9].

value of 1728 K assigned to the melting temperature of nickel [1, 9]. In subsequent work by Schofield and Bacon [10] and by Oriani and Jones [11], the radiation temperature measurements were referenced indirectly to the gold point by means of calibrated tungsten-filament lamps.

As may be seen in Table III, these investigations yield somewhat lower melting temperature values than the present result, 1729 K. However, the differences are within the measurement uncertainties reported by the investigators. With the exception of the work by Oriani and Jones, the specimens used in the earlier measurements were less pure than those used in the present study, which may account partially for the lower melting temperatures obtained earlier. The depression of the melting temperature due to the premelting effects of impurities in the present specimens is estimated to be not more than 1 K.

Of the 17 secondary reference points above the freezing point of gold given in a recent extended list [9], seven are based solely on measurements by Cezairliyan and co-workers using a rapid pulse heating technique. The agreement of the present result with the assigned value for the nickel melting temperature (1728 K), which is based on quasi-steady-state experiments, strengthens the foundation for secondary reference points at higher temperatures determined by dynamic techniques.

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