Proceedings of the Iowa Academy of Science

Volume 64 | Annual Issue Article 51

1957

Measurements of Specific Heats by a Pulse Method

R. C. Strittmater
Iowa State College

G. J. Pearson
Iowa State College

G. C. Danielson
Iowa State College

Measurements of Specific Heats by a Pulse Method[1]

By R. C. Strittmater, G. J. Pearson and G. C. Danielson

Introduction

Calorimetric methods of measuring specific heats at high temperatures are slow and often involve laborious precautions to eliminate heat losses. Pulse heating methods are rapid and heat losses can be made negligible. The accuracy of the calorimetric methods, however, has been greater than that of the pulse heating methods, which need further development. The purpose of the present investigation was to develop further and to evaluate again one of the more promising pulse heating methods.

Kurrelmeyer, Mais and Green (1) suggested in 1941 the use of pulse heating. Baxter (2) described in 1944 a pulse method in which the current and voltage across a thin wire sample were recorded simultaneously. This information, combined with knowledge of the resistance as a function of temperature, gave the specific heat. The principle of Baxter's method has been used in preliminary measurements by Khotkovitch and Bagrov (3) and by Nathahn (4), and is also used in this investigation.

Procedure

If a sample, consisting of the central segment of a fine wire, is heated in vacuum by an electric current of high density, it can be shown that it is possible to neglect all heat losses without affecting the specific heat determination by as much as one per cent. The specific heat of the sample at constant pressure, in calories/gram-degree C, is

$$C_p = (eidt)/(Jmd\theta) = (ei)/(Jm) \quad (d\theta/dt),$$

where e is the voltage across the sample during the time interval dt, i is the current through the sample during this same time interval, m is the mass of the sample, and $d\theta$ is the temperature rise produced by the energy input eidt. If e is in volts, i in amperes, t in seconds, n in grams, and θ in degrees Centigrade, the constant J equals 4.186 joules per calorie.

In order to determine $d\theta/dt$, the resistance of the sample as a function of temperature must be measured by an auxiliary experi-

[1]This work was performed in the Ames Laboratory of the Atomic Energy Commission.

ment. During the pulse heating, e and i are measured independently as functions of time. The resistance, r = e/i, is then known as a function of time and, from the resistance versus temperature curve, the temperature can also be found as a function of time. The slope, $d\theta/dt$, of the temperature versus time curve may thus be measured and tabulated for each instant of time (and hence for each corre-

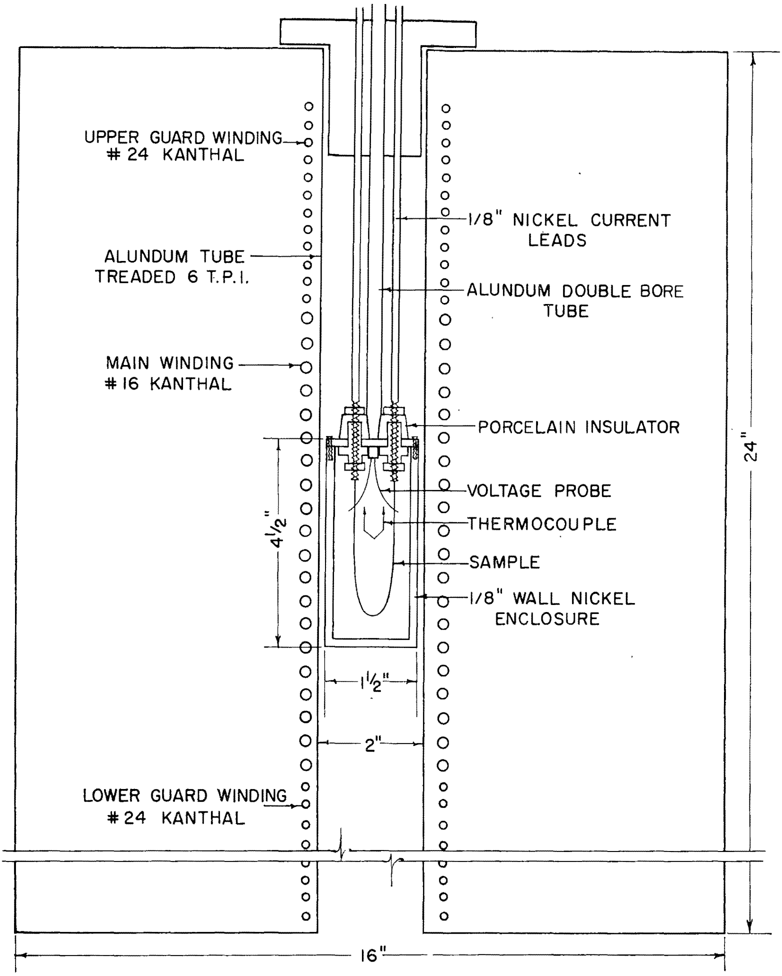

Figure 1. Furnace for the Measurement of Resistance Versus Temperature.

sponding temperature) at which the voltage and current are determined.

The resistance as a function of temperature was measured by means of the furnace shown in Fig. 1. Each resistance value was obtained from the voltage across the sample, which was given directly by a Rubicon type potentiometer, divided by the corresponding current through the sample. The current was obtained from a measurement of the voltage across a one ohm standard resistor in series with the sample. The chromel-alumel thermocouple at the center of the sample loop measured the temperature to within 0.3° C.

In order to avoid heat losses by air conduction, the sample was placed in a vacuum chamber for pulse heating. The temperature was increased from 20° C. to 450° C. in 50 milliseconds for the platinum sample and in 150 milliseconds for the nickel sample. The pulse was provided by a 12 volt storage battery switched on and off by a mercury contact relay. The voltage, e, across the sample and a voltage proportional to the current, i, through the sample were recorded on a dual beam oscilloscope and the two traces photographed on film driven past the scope face at about 20 ft./sec. Time markers were provided from a frequency standard at one millisecond intervals.

The samples were about 0.005 inch in diameter and seven inches long. After sufficient data had been taken on a sample, the wire was cut under a microscope at the center of each voltage probe and the mass, m, determined by means of a microbalance.

Results

The specific heat of platinum was found to vary from about 0.003 cal/gm° C. near room temperature to 0.038 cal/gm° C. at 450° C. Comparison of our results with calorimetric measurements by White (5) indicates errors in our values of five to ten per cent.

The resistance of our nickel sample increased from 0.7 ohms at 20° C. to 3.0 ohms at 450° C. with a conspicuous decrease in slope at the Curie temperature near 360° C. The specific heat versus temperature for our nickel sample is shown in Fig. 2. The calorimetric determination by Sykes and Wilkinson (6) is shown for comparison purposes. The errors in our values are again about five to ten per cent. From theoretical considerations one would expect that the curve should drop very steeply at the Curie temperature. The drop in our curve is faster than the drop in the curves obtained by most other investigators. This observation suggests to us that the pulse method may be superior to the calorimetric methods in the determination of specific heats at such phase transitions.

In order to reduce errors to less than two per cent, the following improvements have been undertaken by Wallace and Sidles (7): (a) the sample will be mounted in a vacuum furnace so that the temperature range over which the sample is heated during one pulse may be reduced; (b) the resistance of the sample during pulse heating will

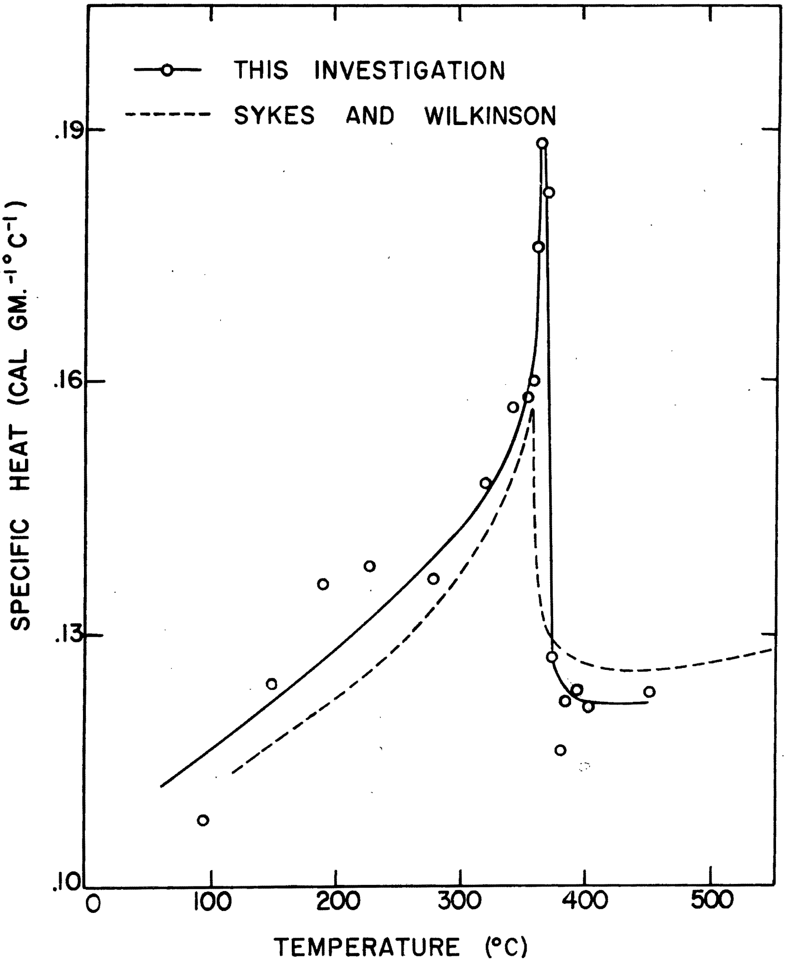

Figure 2. Specific Heat of Nickel.

be measured directly by the unbalance in a Kelvin bridge; and (c) the potential probes will be eliminated and correction made for the very small heat losses to the end electrodes.

Conclusion

A pulse heating method for measuring the specific heats of metals at high temperatures has been developed. Preliminary results have been obtained for platinum and nickel in the temperature range 20° C. to 450° C. With further development, the pulse heating method seems to show considerable promise of becoming a superior method for the measurement of specific heats at high temperatures and particularly useful in the study of phase transitions.

Literature Cited

1. B. Kurrelmeyer, W. H. Mais and E. H. Green. Phys. Rev. 59, 921 (1941).
2. H. Baxter. Nature 153, 316 (1944).
3. V. I. Khotkovitch and N. K. Bagrov. Doklady Akademii Nauk S.S.S.R. 81 (6), 1055 (1951).
4. A. M. Nathan. J. Appl. Phys. 22, 234 (1951).
5. W. P. White. Phys. Rev. 12, 436 (1918).
6. C. Sykes and H. Wilkinson. Proc. Phys. Soc. 50, 834 (1938).
7. D. C. Wallace and P. H. Sidles (Iowa State College). Private Communication.

Institute for Atomic Research
and Department of Physics
Iowa State College
Ames, Iowa