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CONSTRUCTION OF A DIFFERENTIAL CALORIMETER FOR THE DETERMINATION OF HEATS OF ALLOYING AND TRANSFORMATION BETWEEN 800 K AND 1600 K

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ABSTRACT

The "Tandem" calorimeter, the principle of which consists in measuring temperature differences between an "active" and a metallic reference specimen, differs from conventional calorimeters above all in its simple construction, its greater mechanical stability and its significantly lower cost. At the same time, good reproducibility of the results can be achieved with a satisfactory accuracy of measurement.

INTRODUCTION

As part of a continuing program of calorimetric work being carried out at Aachen, numerous measurements of molar heats, heats of formation and transformation have been and are being made by means of an adiabatic calorimeter [e. g. [1)]] . However, minor repairs to this delicate and sophisticated apparatus are frequently required. Consequently, a more robust re-construction has been found desirable and is now under way [2,3)]. At the same time, an alternative calorimeter of simpler design has been built. This novel design is mechanically more stable, requires less expenditure and nearly attains the accuracy and maximum temperature of measurement of the adiabatic calorimeter. It may be termed "Tandem" calorimeter. Its principle of operation consists in measuring the temperature differences between an "active" and a reference metal specimen.

EXPERIMENTAL

The cylindrical specimens have a diameter of ca. 20 mm and a length of ca. 30 mm. These dimensions represent sufficient mass to produce significant thermal effects and a reasonably small heat exchange with the environment. The weight of the two specimens is such that their heat contents are similar at the temperature of measurement. In the centre of each specimen is a junction of the Ni/NiCr differential thermocouple - see Fig. 1. A second thermocouple records the actual temperature in the test specimen. The

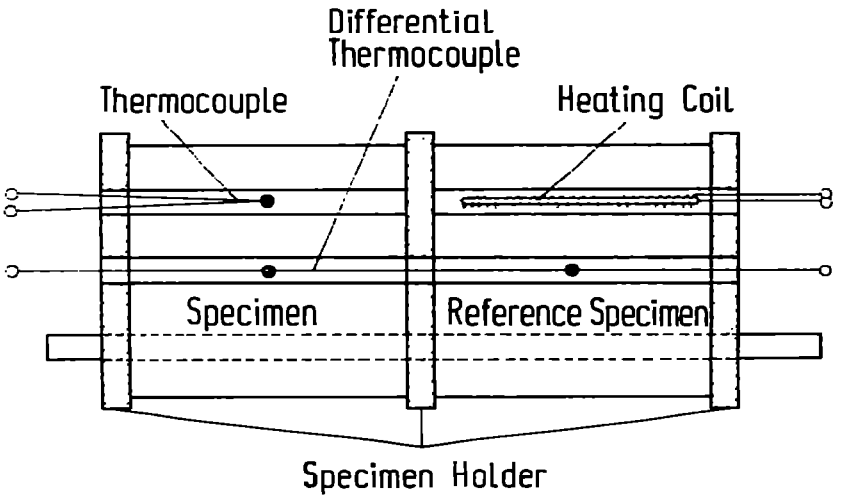

Fig. 1. Experimental Arrangement

assembly is situated in a furnace with a sufficiently long zone of uniform temperature. To prevent oxidation, a flow of gas consisting of 50 % argon and 50 % hydrogen is passed through the furnace tube.

Exothermic or endothermic temperature effects in the specimen under investigation are registered as a function of time by a recorder (Fig. 2). For the calibration of the corresponding $\triangle T \sim \triangle t$ areas in terms of Joules, the reference specimen contains

a heating coil made of tungsten wire (Fig. 1), by means of which a quantitatively defined amount of electrical energy can be supplied (Fig. 2). In practice, the heating current and time are chosen so that the areas under the measurement and calibration curves are roughly equal. In each experiment, calibration is carried out in the same temperature interval as that in which the actual effect is recorded.

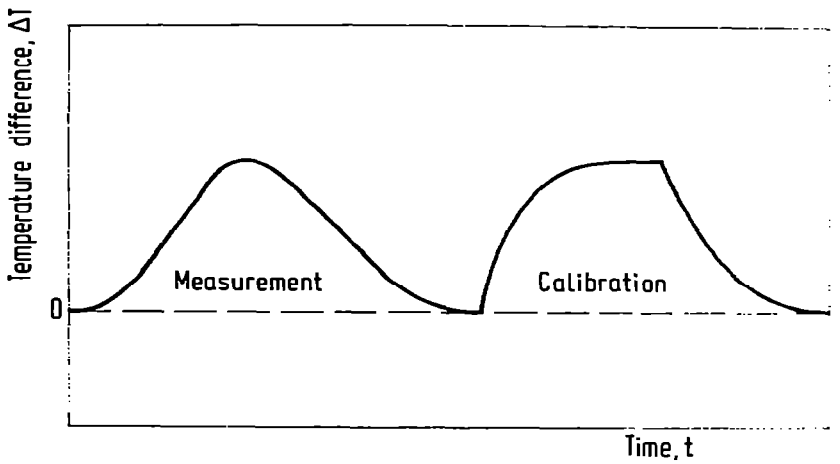

Fig. 2. Temperature difference-Time-Diagram

RESULTS

Measurements have been made on pure iron and its alloys with cobalt, nickel and molybdenum. The observed heats of transformation agree with accepted values to within 60 J mol^{-1}. Heats of formation have also been determined. Here again, the agreement with values obtained by the adiabatic calorimeter mentioned earlier is good - the reproducibility in the temperature range 800 K to 1600 K being within 5 %.

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References

1) Kubaschewski, O., and Grundmann, J.,
Ber.d.Bunsenges., 81 (1977) 1239

2) Hack, K.,
Dissertation, RWTH Aachen (1978)

3) Nüssler, D.,
Ph.D. work Aachen, Private Communication