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# APPLICATIONS OF A NEW LOW TEMPERATURE DIFFERENTIAL SCANNING CALORIMETER

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A new low temperature differential scanning calorimeter is described which operates over the temperature range −150 °C to 700 °C. The equipment features a chromel heat flux DSC sensor plate located in a cobalt alloy heating chamber, enabling work to be carried out in oxidizing atmospheres to above 600 °C. Full data processing and programme control facilities are provided by an IBM compatible computer system. The performance of the instrument is illustrated by experiments on indium and poly (vinyl chloride).

The DSC 700 is the most recent addition to the Stanton Redcroft range of differential scanning calorimeters. It operates over the range from −150° to 700° and is designed to combine the reliability and ease of use required for quality control work with the performance and versatility necessary for R and D studies.

## Description of the apparatus

### *DSC head and furnace assembly*

A close-up of the head assembly is shown in Fig. 1. The sample and reference materials are contained in 6 mm diameter crucibles (*C*), 2 mm high. Crucibles 4 mm high are also available, and materials include aluminium, platinum, alumina and quartz. Crimped an large volume encapsulation crucibles are also offered and the design of the latter enables samples to be crimped before encapsulation. The crucibles are supported on a heat flux plate (*P*), constructed from chromel and forming part of a chromel–alumel detection system. The detector plates are isolated from the main plate by slots and feature integral ears (*I*), for the positive crucible location required for quantitative measurements.

A cross section of the head and furnace assembly is shown in Fig. 2. The DSC head (*D*), is mounted on a four bore alumina rod (*R*). The head assembly is designed so that it can be removed for replacement if accidentally damaged. The head is enclosed within a cobalt alloy block (*B*), which is

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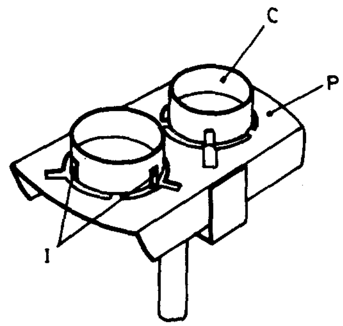


**Fig. 1** DSC 700 heat flux head

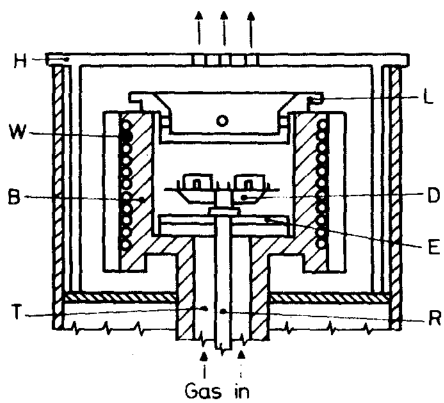


**Fig. 2** Cross-section of DSC 700 furnace assembly

heated by means of the mineral insulated winding (*W*). Independent rotation of the head and asymmetric disc assembly (*E*), within the block enables temperature dependent baseline adjustment to be achieved at manufacture.

Full atmosphere control is obtained by passing the desired gas directly through the sample cell via the inlet tube (*T*). The inlet design ensures that the gas is pre-heated to sample chamber temperature and enters the sample chamber in such a way that the dynamic equilibrium is not disturbed. Typical flow rates are in the range 10–50 ml $min^{-1}$. The gas leaves the cell via the exit holes in the inner lid (*L*), and the heat shield (*H*). The complete furnace assembly is mounted in a gas tight stainless steel chamber. Access to this chamber is via a removable outer lid which is clamped against an '0' ring seal to provide a completely closed system. The materials used in the construction of the head and sample chamber assembly allow work to be carried out in oxidising atmospheres to above 600°. A photograph of the DSC module, showing the sample chamber and the inner and outer lids, is given Fig. 3.

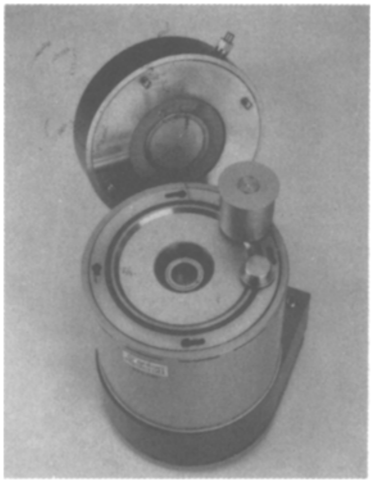

**Fig. 3** Photograph of DSC 700 module

A liquid nitrogen cooling attachment is available which is inserted through the outer lid of the DSC. This enables programmed heating or cooling experiments to be carried out over the temperature range −150° to 400°.

*Control and processing modules*

Full data-processing and programme control facilities are provided for the DSC 700 by the Trace range of IBM compatible computer systems. Options range through monochrome and colour systems for single instrument operation to colour systems which enable up to three instruments to be controlled simultaneously, with full foreground-background facilities. A photograph of the DSC 700 and a Trace 2 system based on an Olivetti M24 microcomputer is shown in Fig. 4. All systems feature extensive and easy to use menu-driven software, which includes a wide range of application packages covering areas such as purity, kinetics and specific heat.

For those wishing to work with a chart recorder, a microprocessor-based lineariser module is available and in conjunction with the low noise DC amplifier of the DSC 700 provides a constant sensitivity DSC signal over the operating range of the instrument. Sensitivities range from 1 mW to 100 mW and above for full scale deflection.

The DSC 700 uses a microprocessor-based temperature programmer to give scan rates from 0.1 to 100 deg/min. Facilities for multi-stage operation and automatic gas switching are incorporated as standard.

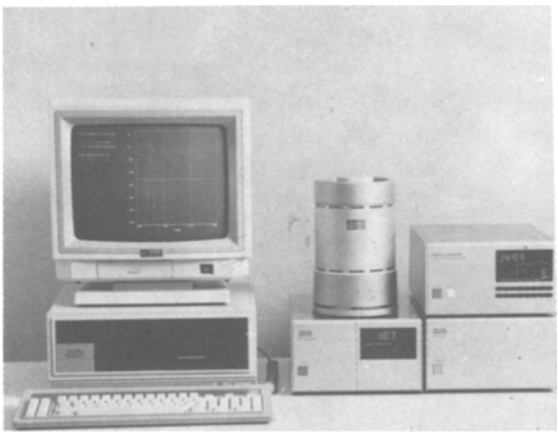

**Fig. 4** Photograph of DSC 700 and Trace 2 systems

## Instrument performance

Experiments using the fusion endotherms of pure metals have shown the calorimetric precision of the unit to be better than 1%. The large dynamic range of the instrument is illustrated by experiments on indium which show a linear relationship between peak area and sample weight over the range 5–100 mg (Fig. 5). Peak area measurements were found to be independent of heating rate and gas flow rate over the normal operating range of the instrument.

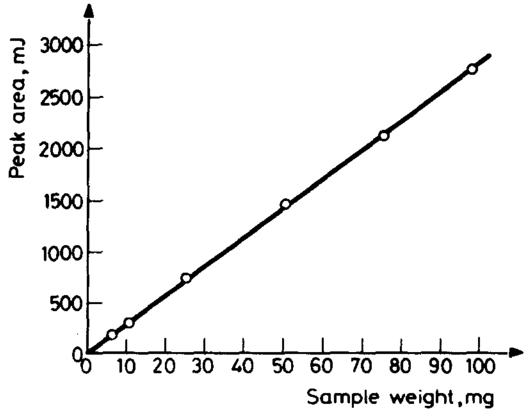


Fig. 5 Peak area *vs.* sample weight plot for indium (5 deg min$^{-1}$, nitrogen, 20 ml min$^{-1}$)

The ability of the instrument to work to high temperatures in oxidising atmospheres with materials that produce corrosive products, is illustrated by runs on poly(vinyl chloride) and a vinyl chloride/vinyl acetate copolymer (Fig. 6).

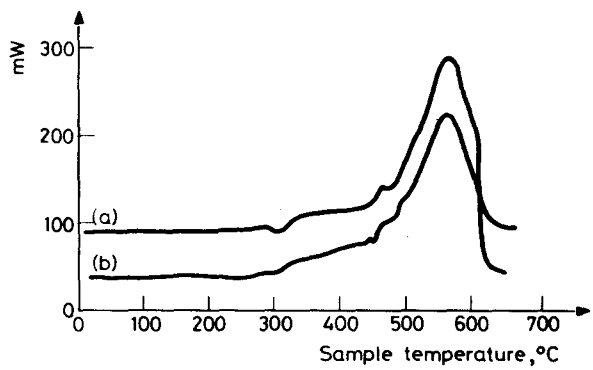


Fig. 6 DSC curves for (a) poly(vinyl chloride) and (b) vinyl chloride/vinyl acetate copolymer (5 mg, 20 deg min$^{-1}$, air, 20 ml min$^{-1}$)

**Zusammenfassung** — Ein neues Tieftemperatur-Differential–Scanning-Kalorimeter für den Bereich von −120 °C bis +700 °C wird beschrieben. Das Gerät enthält eine Wärmefluss-DSC-Sensorplatte aus Chromel in einer Heizkammer aus einer Kobaltlegierung, was Arbeiten in oxidierender Atmosphäre bis über 600 °C ermöglicht. Ein IBM-kompatibles Computersystem bietet alle Möglichkeiten der Datenverarbeitung und Programmregelung. Die Leistungsfähigkeit des Geräts wird an Hand von Experimenten mit Indium und Polyvinylchlorid demonstriert.

**РЕЗЮМЕ** — Описан новый низкотемпературный дифференциальный сканирующий калориметр, работающий в интервале температур от −150° до 700°. Характерной деталью ДСК аппаратуры является чувствительная к тепловому потоку пластинка из хромеля, находящаяся в кобальтовом сплаве камеры нагрева и дающая возможность проводить измерения в окислительной атмосфере выше 600°. Полная обработка данных и программированный контроль осуществляется с помощью IBM компьютерной системы. Действие аппаратуры показано на экспериментальных примерах с индием и поливинилхлоридом.