Thermochimica Acta, 134 (1988) 401–406
Elsevier Science Publishers B.V., Amsterdam

COMPARISON OF TEMPERATURE RESPONSE WITHIN AND BETWEEN POWER COMPENSATED AND DIFFERENTIAL TEMPERATURE DSC INSTRUMENTS

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ABSTRACT

Experiments to determine melting points are conducted in which paired indium samples have been placed in the sample and reference cells in power compensated and differential temperature DSC instruments. In some cases, it has been found that the peak temperatures for the two cells differ within an instrument, contrary to general expectations.

INTRODUCTION

Three different types of DSC/DTA instruments are utilized to compare the temperature responses of the reference and sample cells when using paired indium samples. In the DTA DELTATHERM V system with Model D8600 contollers and D8100 DTA/DSC module, the sample and reference materials are placed in aluminum cups on separate nickel platforms. The temperatures are detected by separate chromel-alumel thermocouples welded to the nickel platforms which are located in the heater block [1]. This instrument is a true DTA.

A Perkin-Elmer 7 Series DSC is also used. This is a power-compensated instrument which plots the difference in rate of energy supplied to the sample and reference materials in their separate cells. These cells are thermally insulated from each other and have separate heaters [2].

The third instrument used is the DuPont Cell Model 910 with the Du Pont Thermal Analyzer and Model 1090 Control console. This is a heat-flux DSC. The sample and reference pans are placed on platforms on the constantan thermoelectric disc which is the major path of heat transfer for the sample. A chromel-constantan differential temperature monitoring system is completed by connecting a chromel wire to each platform, both within a single silver heating block [3].

For the purposes of this experiment it is not considered necessary to recalibrate the instruments since only the peak temperatures differences are of interest.

Thermal Analysis Proc. 9th ICTA Congress, Jerusalem, Israel, 21–25 Aug. 1988
0040-6031/88/$03.50

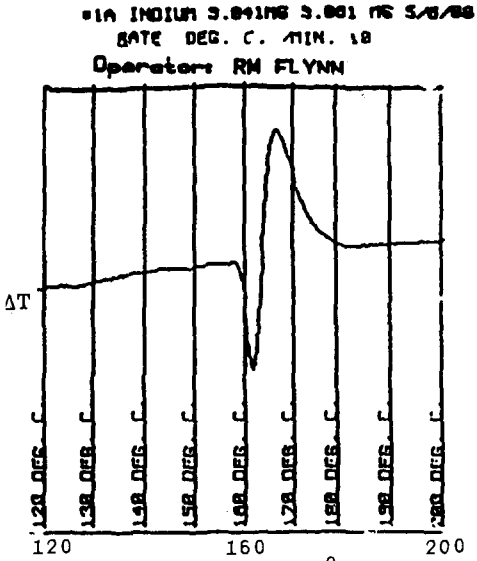

Fig. 1. Melting peaks for 2 indium samples heated at 10 degrees C/min. (DELTATHERM DSC/DTA)

DTA/DSC

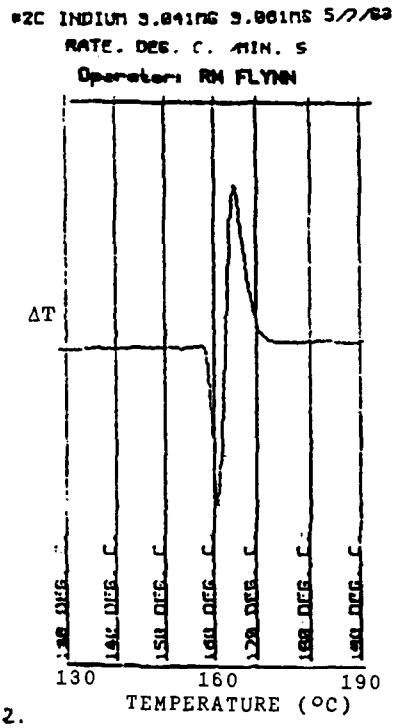

Fig. 2. Melting peaks for 2 indium samples heated at 5 degrees C/min. (DELTATHERM DSC/DTA)

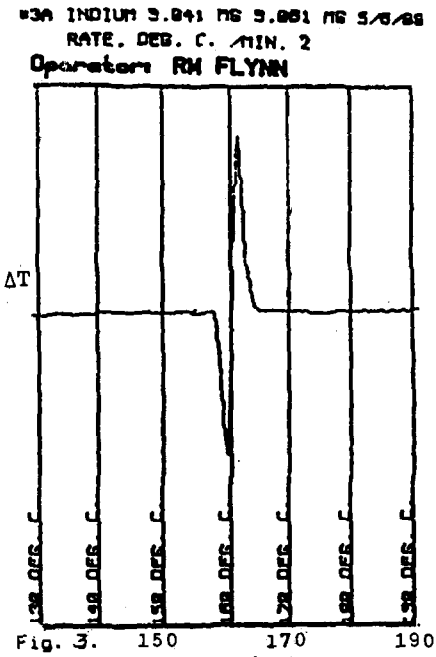

Fig. 3. Melting peaks for 2 indium samples heated at 2 degrees C/min. (DELTATHERM DSC/DTA)

Deltatherm V system
Model D8600 controllers
with D8100 DTA/DSC

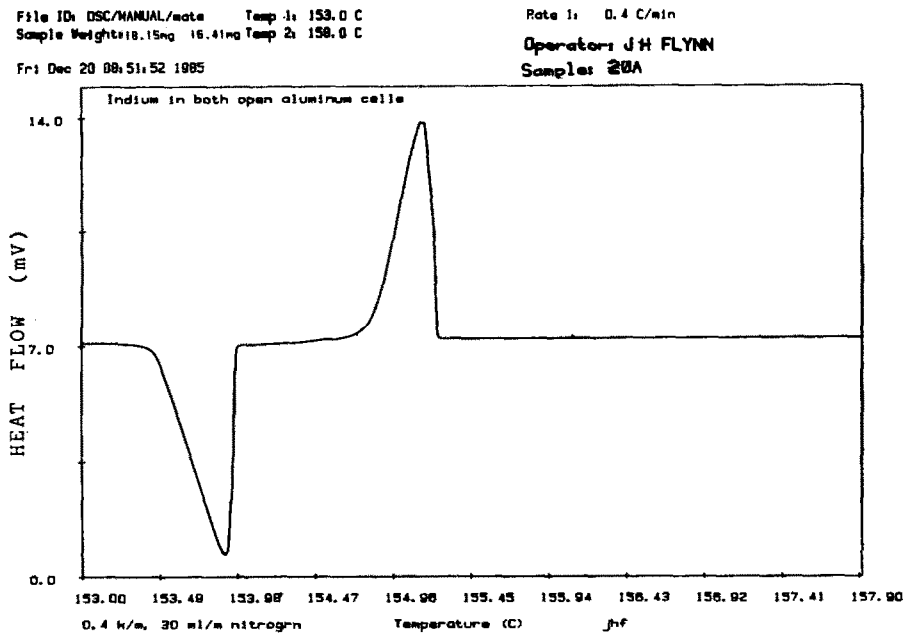

Fig. 4. Melting peaks for 2 indium samples heated at 0.4 degrees C/min. (PERKIN-ELMER 7 Series DSC)

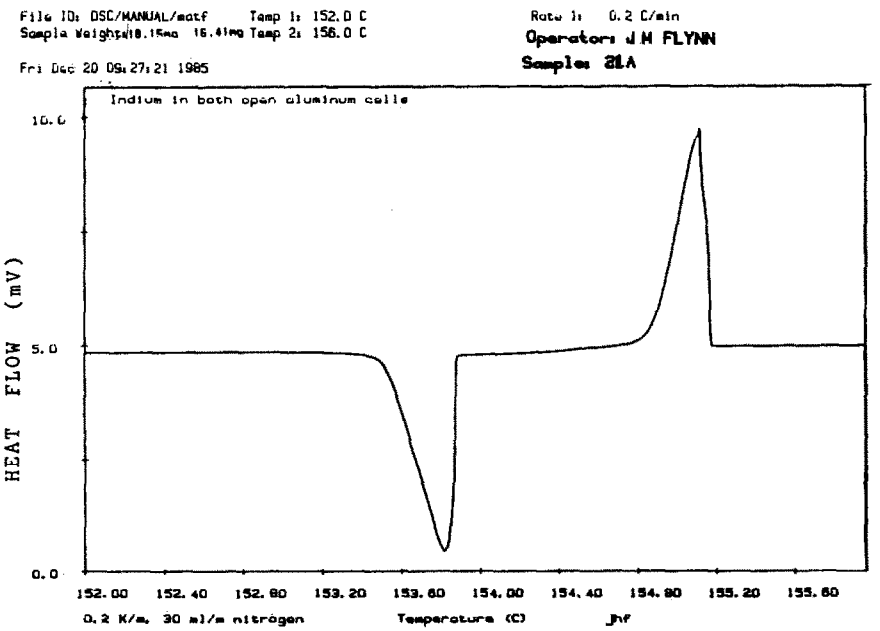

Fig. 5. Melting peaks for 2 indium samples heated at 0.2 degrees C/min. (PERKIN-ELMER 7 Series DSC)

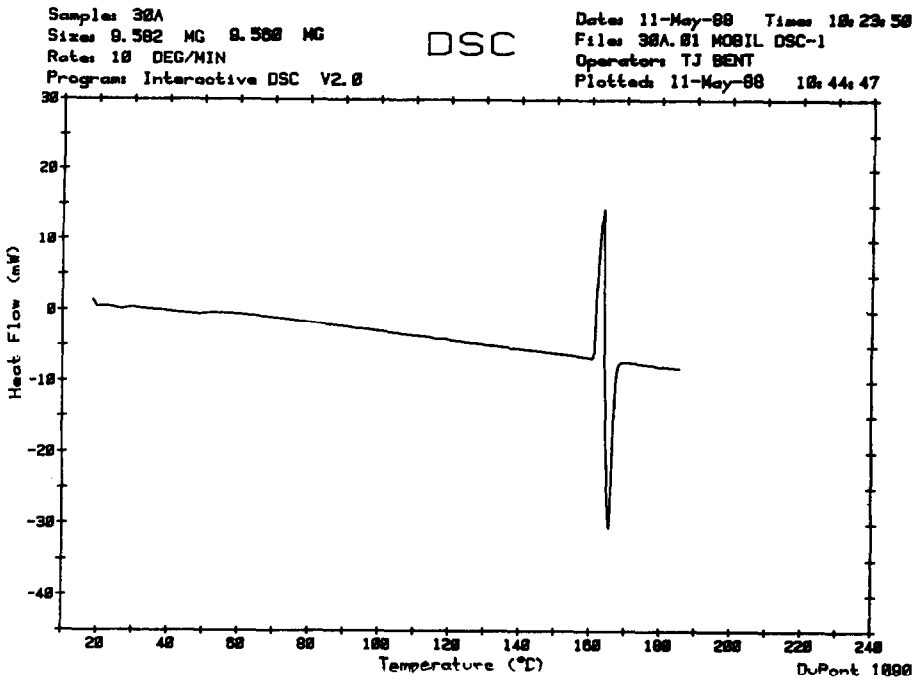

Fig. 6. Melting peaks of 2 indium samples heated at 10 degree C/min. (DUPONT 1090 DSC)

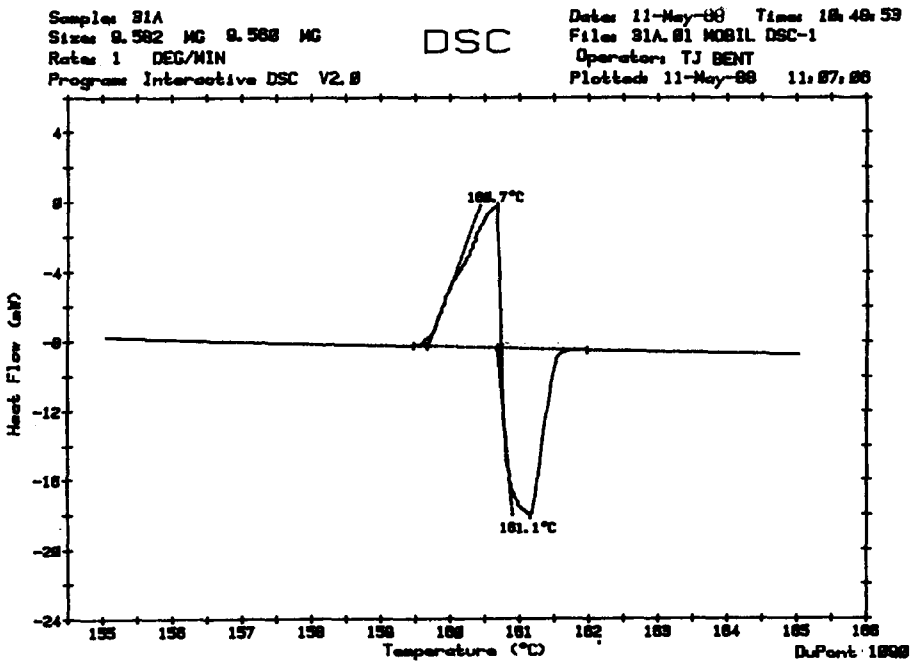

Fig. 7. Melting peaks for 2 indium samples heated at 1 degrees C/min. (DUPONT 1090 DSC)

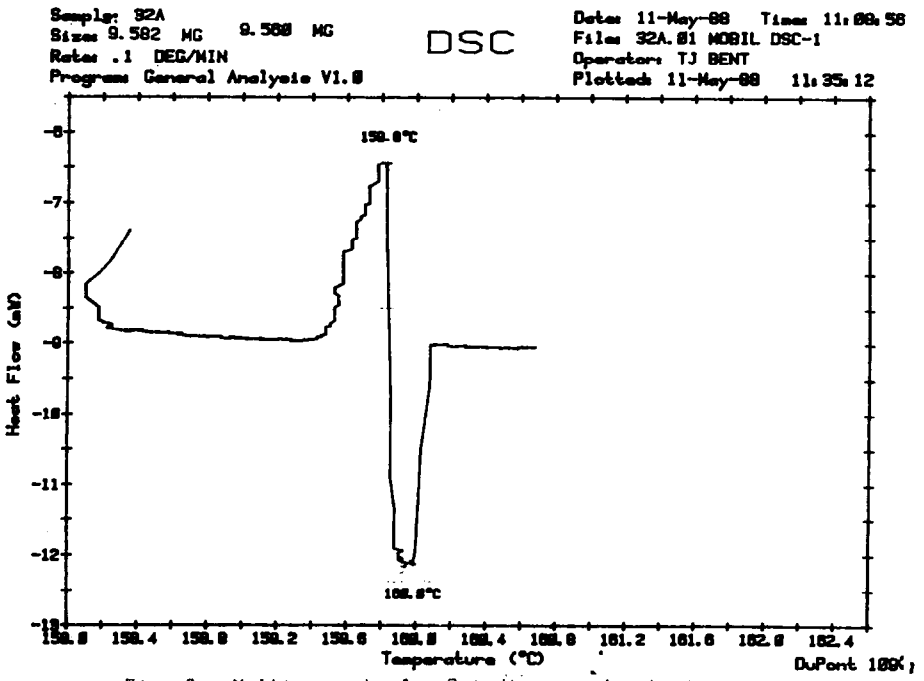

Fig. 8. Melting peaks for 2 indium samples heated at 0.1 degrees C/min. (DUPONT 1090 DSC)

TABLE 1

Heating rates and melting peak temperatures for pairs of indium samples in three different instruments.

Heating Rate	DSC or DTA Instrument	Peak 1 °C	Peak 2 °C	△Peak °C
10 °C/min	DELTATHERM	162	168	6
5 °C/min	DELTATHERM	161	163	2
2 °C/min	DELTATHERM	159	161	2
0.4 °C/min	P-E 7 Series	153.90	155.12	1.12
0.2 °C/min	P-E 7 Series	153.98	155.09	1.01
0.1 °C/min	P-E 7 Series	153.78	155.07	1.29
10 °C/min	DUPONT 1090	163.7	165.3	1.4
1 °C/min	DUPONT 1090	160.7	161.1	0.4
0.1 °C/min	DUPONT 1090	159.8	160.0	0.2

EXPERIMENTAL

The indium samples used are of 99.999% purity. The weights of the paired samples used in the Deltatherm instrument are 9.041 mg. and 9.061 mg., respectively. Runs were carried out at heating rates of 10 deg. C/min., 5 deg. C/min. and 2 deg.C/min. The samples used in the Perkin-Elmer instrument have weights of 16.41 mg. and 18.15 mg. The heating rates are 0.4 deg.C/min., 0.2 deg.C/min and 0.1 deg. C/min. The DuPont instrument was used for paired samples of 9.582 mg. and 9.560 mg. The heating rates used are 10 deg.C/min., 1 deg. C/min. and 0.1 deg.C/min. (vide Figures 1-8)

RESULTS AND DISCUSSION

The overall results are shown in Table 1. In all runs a temperature differential was found between the peak temperature for the indium in the "sample" holder and that in the reference holder. In the case of the Deltatherm and DuPont instruments, the difference seems to be heating rate dependent with smaller differences at lower heating rates. The heating rate dependence is not unexpected in a heat flow type of DSC.

The Perkin-Elmer DSC responses, on the contrary, display an almost constant peak temperature difference over the three heating rates used. In Model 1B and earlier, it was possible to adjust this temperature difference by use of an external control knob, but this flexibility has been lost in more advanced models.

CONCLUSIONS

Although a difference in peak temperature between paired indium samples was found for each of the three instruments used, it must be noted that these represent a very limited number of cases, and only one instrument from each manufacturer was used. Many more runs must be carried out with different instruments to validate these results. If this temperature differential is found to be inherent in an instrument, it must be taken into account in experiments involving two thermally active substances with one in the sample holder and the other in the reference holder.

REFERENCES

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