Thermochimica Acta, 92 (1985) 411-414
Elsevier Science Publishers B.V., Amsterdam

THERMAL ANALYSIS CONTROLLED BY MICROCOMPUTER

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ABSTRACT

A microcomputer system for coltrolling and evaluating thermal analysis is described. The system enables automation of measuremet according the given demands of the experimentalist. Breaks on the cooling and heating curves are searched by means of the regression analysis.
The error of measurement is approx. ± 0.2 K in the temperature region of 300 - 1100 °C.

THE PRINCIPLE OF MEASUREMENT

The measurement is carried out by direct thermal analysis. The cooling and heating curves of specimen of 20 g weight at defined cooling and heating speeds are recorded. The operation of the electrically heated oven is controlled by a microcomputer. The sample temperature is tranduced by a thermocouple PtRh10-Pt with a diameter of wires of 0.5 mm. The cold joint of the thermocouple is in an ultrathermostate at the 28 + 0.1 C temperature.
The analogue signal of the thermocouple voltage is an input to the digital voltmeter MT-100. Thus digitalized data are with the chosen time periods read in by the computer and stored. After finishing the measurement the breaks on the curves are searched by the method of regression analysis.

THE MICROCOMPUTER SYSTEM

It is composed of two relatively independent microcomputers. The first one is used for controlling the experiment, the second one for evaluating the meausured data.
The controlling microcomputer
Hardware: INTEL 8085, 20 kB EPROM, 20 kB RWM, the alphanume-

Proceedings of JCTA 85, Bratislava

rical display 64 characters 16 lines, attached are: recorder, paper tape peripheries, real time clock, digital voltmeter MT-100, power regulator 15 A/220V, communication channel (HANDSHAKE).

Software: operational system (EPROM): contains a monitor, ASSEMBLER, BASIC. The essential user's subroutine are programed in ASSEMBLER, stored in EPROM. They are as follows: real time clock service (INTERRUPT), oven service (INTERRUPT 100 times per sec. - 200 levels of the oven power), read in the data from MT-100 (both external and internal start). The fundamental measurement program is itself written in BASIC. The essential subroutines: data input from MT-100, in the chosen time interval, testing its reliability, turning into the temperature, storing; adjusting both the oven temperature and the time to the chosen value; establishing both the cooling and heating regime in the adjusted temperature region with the given temperature change speed.

By means of these programs it is possible to adjust any oven regime with multirepeating respectively. After finishing the measurement the data are either recorded on a punched tape or transferred to the evaluating computer.

The evaluating computer

Hardware: TESLA 8080, 96 kB RWM (max. 512 kB), 32 kB EPROM, graphic display 512 points x 256 lines, 80 character keybord attached are: a cassette unit, paper tape peripheries, typewriter, plotter. Communication channels: parallel, serial. This is a modular system with a possibility of simple modification.

Software: the operational system contains a monitor, ASSEMBLER, graphic BASIC. The program evaluation is created in BASIC. It can be started after having read in measured data from the cotrol ling computer or punched tape. It contains subroutines for filtration of error points, smoothing the curves by the Whittaker - Robinson method, choosing the demanded part of the curve for evaluation. The break parameter on the curves can be established by three basic ways:

1. the temperature of the break point is directly read from the curve
2. the measured curve is approximated by a suitable function (polynomical of 1-6 degree) and the break value is determined from the function value of the given point
3. the break value is determined numerically as a cross point of the two approximating functions; the first one in the region before, the second one in the region after the break.

MEASUREMENT

The measurement of the beforehand prepared samples is fully automatized: the sample is placed into the oven. The control program starts. It assures the demanded time temperature course in the oven, storing the data in the chosen temterature regions. The fundamental working cycle is following: heating-cooling-heating. Measurement of the nonprepared samples is as follows: the oven temperature is program adjusted to the value by 20 - 30 K higher than the melting temperature of the sample. After melting the sample is blended and and the process proceeds as in the case of the prepared sample.

EVALUATION OF THE MEASUREMENT

After the data have been read in by the evaluating computer they are drawn on to the display. Occasional errors are removed. In case of need it is always possible to smooth the curve. A suitable part of the curve to be evaluated is chosen - this is the working region. Here the breaks are searched by the three methods mentioned above. In the case of need the measured and evaluated curve can be drawn by a plotter.

PRECISION

Several parameters of the system have been tested:

1. its competence to hold the temperature on the adjusted level. The maximum regulation deviation has been ± ? K in the whole temperature region.

2. competence to hold the linear temperature change
   The working cooling and heating speed was 2 K/min. Non-linearities appeared on the curves but they did not cause artefacts on the curves. Heating and cooling speeds fluctuated in the region of 1.5 - 2.5 K/min.
3. Precision of determining the break temperature
   This is the most important parameter of the whole system. 50 cooling and heating curve measurements of the sample currently used for thermocouple calibration were carried out. All measurements were repeated min. 5 times (in one case 10 times). It was found that the reproducibility of measurement in the region of 600 - 1400 K was better than ± 0.2 K at cooling and ± 0.3 K at heating.