Thermochimica Acta, 96 (1985) 223–225
Elsevier Science Publishers B.V., Amsterdam – Printed in The Netherlands

Note

CONTINUOUS THERMOANALYSIS: THE ESSENCE OF THE METHOD AND EQUIPMENT

Ch.M. KASHKAI

The Institute of Geology, Academy of Sciences, Azerbaijan S.S.R., Prospect Narimanova 29 A, 370143 Baku (U.S.S.R.)

(Received 20 May 1985)

The development of differential thermal analysis is characterized by the difference in the methods and forms of its realization. This is due to the tendency to by-pass the problems not solved, yet, connected with inhomogeneity and non-controllability of the heat field both around a sample and inside it, during the process of programming the temperature influence. These questions are considered in detail in the literature where an estimation of the role and influence of different factors on the quality of thermal investigations is made.

METHOD AND EQUIPMENT

The analysis of the factors imposing errors results in the conclusion that the quality and character of thermal investigations can be changed radically in the case where two main conditions are satisfied: (1) by using a one-dimensional configuration of a sample; (2) by using a thermostatic gradient temperature field. An attempt to realize these conditions gave rise to the creation of the method of continuous thermal analysis (CTA).

The method is based on the continuous and uniform renewal of a sample under conditions of thermostatic gradient temperature field and separate measurement of the thermal characteristics of every section of the sample. A diagram illustrating the principal of the method of realization is shown in Fig. 1.

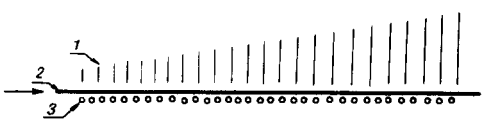

Fig. 1. Diagram illustrating the principal and the equipment for the method of continuous thermal analysis. (1) Temperature field with an assigned gradient along the length; (2) uniformly moving stream of a sample; (3) a line of thermocouples (or one "running" thermocouple).

0040-6031/85/$03.30

According to this method, the temperature field can be distinguished only by the length, along which the temperature gradient is assigned. At the same time, in any section, the temperature field must be maintained constant with time. Practically all the constructive elements inside the thermostat, including the thermocouples, are initially brought into stable thermal equilibrium with their immediate environment.

A one-dimensional configuration is given to the sample under study, i.e., the mass of the sample is uniformly distributed along the length in the minimum possible section. This is realized by the creation of a uniform and continuous stream of the sample. In this case, uniform movement of the sample stream through the temperature field results in the fact that every microsection of the sample is warmed according to the programmed gradient and, on the whole, the whole sample stream, that is, within the limits of temperature field, acquires the same gradient along its full length.

Consequently, against the background of the equilibrium temperature field, any thermal deviations are connected with the thermal characteristic of the sample itself (the thermal contribution of the sample carriers (crucibles) has a constant value). Therefore, the application of a reference material loses every meaning, but a differential measurement is realized by using each thermocouple with reference to the difference of two values: the temperature

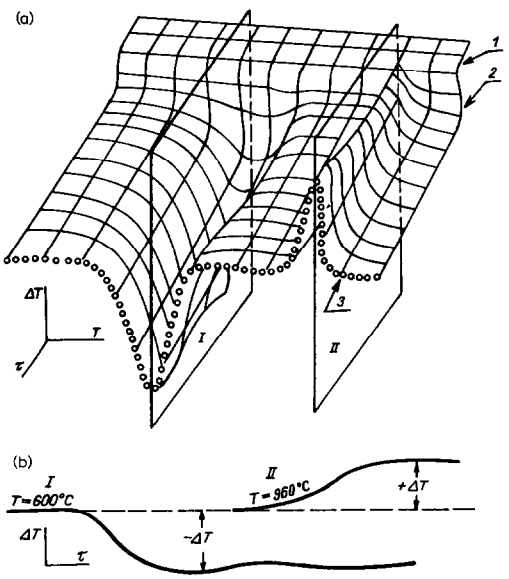

Fig. 2. The view of thermograms obtained using CTA. (a) Volumetric recording of kaolinite thermal characteristics: (1) the background plane corresponding to the thermal contribution of empty crucibles; (2) displacement of background plane in proportion to the sample mass. (b) Isothermal scanning of thermal characteristics for various sample sections (3).

of the thermostated section without a sample and the temperature of the same section with a uniformly moving sample.

Thanks to the stability of the thermal surroundings around the sample and parallel orientation of the temperature gradient and sample mass it is possible to eliminate a lot of uncontrollable heat fluctuations and to decrease a number of values thereby allowing more correct theoretical substantiation and interpretation of the analysis results.

On the other hand, the method of continuous thermal analysis introduces a new, very important parameter—thermal scanning of the sample mass, that is, expressed in the successive measurement of thermal characteristics of various sections of the sample in any temperature interval. It should also be stressed that one more significant feature peculiar to this method is the independence of the temperature of time. Thus, in continuous thermoanalysis the investigations are carried out in three coordinates: temperature difference (ΔT), temperature (T), time (τ), and any two-coordinate cross-sections can also be recorded (see Fig. 2). As a sample moves with uniform rate, a time coordinate also reflects the location of any sample section at every moment in time within the temperature field.

CONCLUSIONS

Besides traditional investigations, the method of continuous thermal analysis creates new opportunities for studying the thermal characteristics of substances. In particular, due to the continuous and uniform renewal of a sample, non-dying-out thermoeffects are produced that allow the study of thermal non-uniformity of a sample making it possible, in a single experiment, to record a lot of thermal parameters for various sample sections and, by means of statistical treatment, to get more reliable data for each sample. As the sample is simultaneously contained in the whole temperature interval, its total thermal characteristics can be recorded, experimentally, almost instantly and repeatedly. In this case thermoinert sections also convey considerable information, allowing the measurement of thermal capacity and heat conductivity of a sample and the calibration of the deviations of the background line and its dependence on the sample mass. At the same time, the constancy of experimental conditions, mainly the stability of the temperature influence, provide high reproducibility and steadiness of calibration parameters. This feature allows one to carry out the phase analysis of mixtures. Owing to the continuous stream of a sample and consequent high productivity of the measurements, the method of continuous thermal analysis, apart from its use in research, can be successfully applied in industrial laboratories, for example, for the control of ore quality or of some other thermoactive raw material.