Journal of Thermal Analysis and Calorimetry, Vol. 72 (2003) 1077–1079

Workshop on Sample Controlled Thermal Analysis

O. T. Sørensen[*]

Ceramic Materials Consultancy, Denmark

Synopsis

The 6-year-old term 'Sample Controlled Thermal Analysis' (SCTA) designates the branch of thermal analysis where a feed-back from the sample is used to control its heating or cooling. In this method , it is therefore the sample itself which determines its own heating and cooling conditions as the name implies. This approach can be considered as a real breakthrough in the field of thermal analysis. It has indeed the advantage, as compared with conventional thermal analysis, of providing: elimination of uncertainties due to thermal effects in the sample container, improved resolution, accurate determination of reaction temperatures and accurate kinetic data. SCTA has since its introduction in the early seventies been used in many studies both on inorganic and to a certain extent organic (polymers) compounds with the aim to study the temperature, the type and the kinetics of reactions taking place during heating and cooling: in the case of ceramics and adsorbents SCTA has even been used in synthesis of materials with specific properties. These techniques are now also available in commercial TA instruments.

A new book in the series Hot Topics in Thermal Analysis and Calorimetry (edited by Judit Simon, Hungary) was announced at the workshop. The title is: Sample Controlled Thermal Analysis: Origin, Goals, Multiple Forms, Applications and Future. The editors are Ole Toft Sørensen (Denmark) and Jean Rouquerol (France). This book is expected to be published by Kluwer during the spring 2003.

Programme

1) Welcome and spirit of SCTA, O. T. Sørensen, Denmark
2) Basic methods and techniques:
- Rate-Controlled SCTA, J. Rouquerol, France
- Stepwise and Forced Stepwise SCTA, O. T. Sørensen, Denmark
- Proportional – and Peak Slope heating SCTA, G. Parkes, UK
- Sample Controlled Thermomicroscopy, E. L. Charsley, UK
- Large scaled sample controlled TG, J. O. Hill, Australia

3) General discussion on basic techniques
4) Applications:
- Kinetic studies, J. M. Criado, Spain

* Author for correspondence: E-mail: otsdk@yahoo.dk

1388–6150/2003/ $ 20.00
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- Ceramics, O. T. Sørensen, Denmark
- Adsorbents, P. Llewellyn, France
- Catalysts, P. Barnes, UK
- Polymers, T. Ozawa, Japan
- Synthesis, J. M. Criado, Spain

5) General discussion on SCTA applications
6) General discussion on future developments

O. T. Sørensen
Workshop Chairman

Spirit and Basic Techniques of SCTA

O. T. Sørensen[**]

Ceramic Materials Consultancy, Denmark

As the name sample controlled thermal analysis implies, the characteristic feature of this technique is that the sample itself is controlling the conditions under which it will be analysed, which can be the temperature, the heating or cooling rate, but it can also be more general conditions like the atmosphere. This of course is very different from conventional thermal analysis, where the sample is submitted to the conditions fixed by the analyst. As described in the synopsis for this workshop, this technique therefore generally gives a considerable improvement in accuracy and resolution compared to conventional thermal analysis. It has therefore been used in many studies, which was amply demonstrated in this workshop.

Generally several parameters can be used to control the measuring conditions in SCTA. As illustrated in the following Fig. 1, these can for instance be the mass (thermogravimetry), the pressure and/or the composition of gases released during a decomposition or the physcal dimension (dilatometry).

As demonstrated in this figure, the sample gives, depending on the type of measurement, a feedback to the controlling system as a mass signal (TG), length signal (dilatometry), or as a pressure/composition signal (Evolved gas detection system). These signals are collected by the data acquisition system and treated in an algorithm in the computer, which controls the heating/cooling system of the thermoanalyser.

Generally a SCTA measurement is performed in the following way: when the sample during heating at constant heating rate (conventional TA) reaches a transformation temperature, the change in the controlling parameter – weight, length, pressure or compostion of a specific gas – will via the controlling algorithm (in the computer) trigger a change in the measuring conditions, which will be maintained during the transformation. At the end of the transformation this algorithm will again change the setting such that the heating at constant rate is resumed. The heating between the

** Author for correspondence: E-mail: otsdk@yahoo.dk

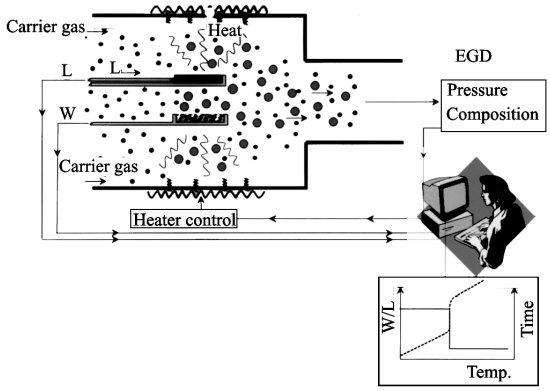

Fig. 1 Reaction controlled thermal analysis (RCTA)

transformations can of course be carried out manually using the transformation temperatures determined in a preliminary run.

SCTA can be performed using different techniques to control the conditions during a transition. To mention a few:

- Quasi-Isothermal Quasi-Isobaric Thermogravimetry (Paulik and Paulik)
- Constant Rate TG (Rouquerol)
- Constant Changing Rate TG (Criado)
- Stepwise Isothermal Analysis (SIA) (Sørensen)
- Forced Stepwise Isothermal Analysis (FSIA) (Sørensen)
- Proportional Heating (PH) (Parkes, Barnes and Charsley)
- Maximum Resolution (Max Res) (Schenker and Riesen)
- Peak Slope Heating (Parkes, Barnes and Charsley)
- Rate-Jump TA
- Dynamic Heating (Opferman)

Examples of applications using many of these techniques were described and discussed during the workshop.

References

1 O. T. Sørensen, J. Therm. Anal. Cal., 56 (1999) 17.