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State of the Art Instrumentation and Trends in Development of Hard- and Software for Thermal Analysis

W. -D. Emmerich, **E. Kaisersberger**, J. Opfermann
NETZSCH-Gerätebau GmbH, Wittelsbacherstr. 42, D-95100 Selb/Germany

The development of Thermal Analysis techniques, seen in a commercial scale, doesn't seem to be a continuous process. Periods with apparent stagnation are followed by relative fast and multiform progresses. The advances often concentrate on just a single or a few techniques. The following is a trial to describe state of the art methods, techniques, and actual trends in Thermal Analysis.

To start with hardware of the most widely used method, DSC, the relative stagnation in instrument technique is past: new sensors, new data acquisition, new temperature programming are introduced. The compromise of sensitivity and signal time constant, intrinsic properties of DSC systems, is brought to extremes in one direction or the other by multiple-junction thermocouple sensors, or by semiconductor sensors based on space technology [1]. The limitations set by the above mentioned competing properties are attacked by the combination of the heat flux and the power compensation principle within one sensor [2], respectively in one instrument. The use of high resolution A/D converters accompanies the improvements in sensor technology to show detection limits and signal noise of DSC's in the sub-µW range, near to the resolution of traditional micro-calorimeters, however still keeping the faster response time of the DSC. The application variety has profited from the modulated temperature technique, and improvements in understanding of overlapping phenomena and in quantitative specific heat results are often achieved [3, 4].

The combination of heat flux DSC with TG in true simultaneous instruments (STA) is extended for broad temperature ranges, covering most of all organic and inorganic applications.

Also the TG shows actual improvements in sample holder technology with respect to sample temperature measurement. From a standard TG qualitative or semi-quantitative information about caloric events can be determined by SDTA or c-DTA, i.e. calculated 'DTA' signals from the comparison of the measured sample temperature with a fictive, linear temperature at the sample position. Temperature calibration of TG became extremely simple and accurate through these new techniques, as existing DSC calibration standards can be applied also for the TG now. The function of different temperature control modes including rate-controlled modes and high-resolution techniques is improved as a consequence of the better temperature measurement. New balance techniques lead to higher long term stability of thermobalances together with higher weight capacity and weighing ranges.

Far from just being a modern trend, coupling of gas analysis techniques plays an important role in Thermal Analysis of today [5]. The coupling techniques become more and more improved [6] and the combined gas analysis e.g. by mass spectrometry and by Fourier-transform infrared spectroscopy is nowadays simpler to use through software integration, but additionally also more sensitive. Quantitative and selective work became possible in routine applications through the introduction of the Pulse Thermal Analysis technique [7].

Dilatometry now supplies accurate thermal expansion and volume-change (density) data in the range 10 K to >3000 K, an important contribution to thermophysical properties databases. New furnaces, sample holder materials and temperature sensors had to be matched for the extension of the temperature limit to 2800 °C [8]. Dynamic mechanical methods (DMA), dielectric methods (DEA, TSC) are used for characterization of relaxation phenomena in the field of polymers and composites and are covering broader frequency and load ranges to come nearer to the practical application conditions of the analyzed materials.

General trends in Thermal Analysis are found in macro and micro techniques as well.

The macro techniques are approached by scaling-up of TG's and STA's and couplings, micro techniques, however, are more based on originally non Thermal Analysis techniques, like modified atomic force microscopy [9]. Visualization of thermally induced processes seems to be a periodic trend of development in instrumentation and instrument combinations.

Automatic sample handling, automatic evaluation and documentation of results are both influenced by hard- and software development and profit from progress in computer capacity. Precise atmosphere control in Thermal Analysis instruments is often discussed but seldom consequently realized. However modern instrumentation can cover the range from high vacuum (10^{-6} mbar) up to high pressure (150 bar), an extremely wide range of pressures and possibilities for specific sample conditions. Temperature programmed reduction (TPR), temperature programmed desorption (TPD) are examples especially in catalyst research for making use of precise atmosphere control in thermobalances [10].

The general and extremely fast development of computer technology stimulated also the development of software for Thermal Analysis. Common user interfaces displaced the special solutions of earlier stages and integrate Thermal Analysis to be now one standard application of the uncountable applications available. The common acceptance of the software is under no discussion anymore, and functionality, reliability and graphical presentation of results reached a quite high standard. The drastically increased capacity and speed of modern computers allow extensive application of advanced software, including statistical evaluations and numerical calculation methods, e.g. in kinetics, multicomponent analysis, thermal simulations, etc. [11].

High demands in material research and numerous quality control applications are the basis for a steadily growing interest in Thermal Analysis techniques. The commercial instrument market has to fulfill these requirements with adequate development of methods and instrument technology.

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