Advanced Techniques in Temperature Modulation DSC

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Temperature modulated DSC (TMDSC) techniques consist of a temperature program with a linear temperature ramp and a superimposed small temperature perturbation. For this modulation different signal shapes are used:

- step-wise temperature changes followed by isothermal segments [1],
- single frequency sinusoidal modulations [2] and
- multi-frequency periodic modulations by superposition of multiple sinusoidal functions [3] or the use of non-sinusoidal modulation functions [4].

TMDSC is used for heat capacity measurements, determination of the kinetic contribution to the DSC signal, separation of different components in the heat flow signal and the measurement of the frequency dependence of the heat capacity. One advantage of temperature modulated DSC in general is the possibility to measure heat capacity changes also during ongoing excess heat production processes (reaction, crystallization, evaporation etc.). This is achieved by separation of the measured sample response signal (heat flow) into the so called "reversing" and "non-reversing" heat flow components.

TOPEM® is a novel temperature modulated DSC technique where a non-periodic stochastic temperature perturbation is superimposed to a conventional DSC temperature program. Using an advanced evaluation procedure both, the quasi static material properties as well as the frequency dependency of thermal processes can be simultaneously analyzed in one single measurement. Using quasi-static properties improves the separation possibilities of temperature modulated DSC considerably. The frequency dependence of thermal processes can be used to get more insight in molecular dynamics and allows an easier identification of thermal events. These possibilities are shown on typical examples.

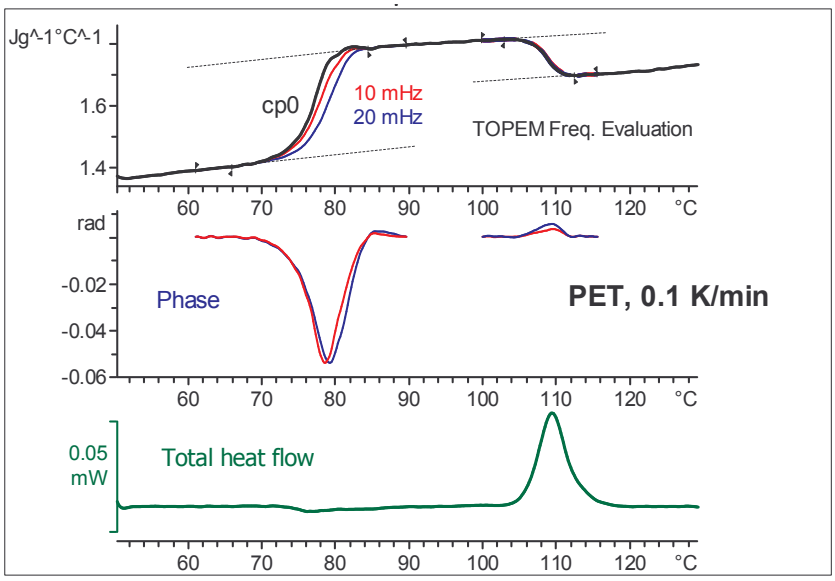

Figure: Example of one TOPEM® measurement of amorphous PET resulting in several curves. The glass transition step is shifted to higher temperatures with increasing frequency, whereas in the crystallization region the frequency curves are identical. The underlying heating rate is 0.1 K/min and therefore the crystallization peak has a hight of approx. 40 µW only.

Literature

[1] S.C. Mraw and D.F. Naas, J. Chem. Thermodyn. 11 (1979) 567.
[2] H. Gobrecht, K. Hamann, G. Willers, J. Phys. E: Sci. Instrum., 4 (1971) 21.
[3] B. Wunderlich, R. Androsch, M. Pyda and Y.K. Kwon, Thermochim. Acta, 348 (2000) 181.
[4] M. Merzlyakov and C. Schick, Thermochim. Acta, 377 (2001) 193.