Fast Scanning Calorimetry (50 000 K/s cooling and heating)

Alexander Minakov, Christoph Schick

University of Rostock, Institute of Physics, 18051 Rostock, Germany christoph.schick@uni-rostock.de

Utilizing a thin film gas pressure sensor as a fast calorimeter we are able to extend the scanning rate range of commercial DSC's (μ K/s to 8 K/s) to rates as high as 50 000 K/s. With these sensors we are able to measure at controlled cooling at the same high rates as on heating. Because of the fast equilibration time isothermal experiments can be performed after scanning at several thousand Kelvin per second. The dead time after such a quench is in the order of 10 ms and the time resolution is in the order of milliseconds.

These ultra fast calorimeters allow us to study the kinetics of extremely fast processes. For example, we are able to follow isothermal crystallization in the whole temperature range between melting and glass transition of ε -Polycaprolactone (PCL) and isotactic polypropylene (iPP). At the maximum crystallization rate crystallization half time is 200 ms for PCL and 50 ms for iPP, respectively. The crystallization process can be easily followed in an isothermal crystallization experiment after cooling at 2000 K/s in order to produce amorphous samples.

On the other hand the high heating and cooling rates allow us to study reorganization of semicrystalline polymers on heating. For PET crystallized at 130 °C reorganization needs less than 40 ms between 150 °C and 200 °C. At heating rates of 1300 K/s, where reorganization is prevented, the sample melts in the temperature range 150 °C to 220 °C. The "main" melting peak at about 250 °C, as always seen in DSC curves, disappears totally. This clearly shows that **all** crystals formed at 130 °C melt below 220 °C.

We will report about very fast reorganization of nanostructures in semicrystalline materials on heating as well as on cooling or isothermally utilizing the new calorimetric technique. Additionally results will be presented for temperature modulated experiments at frequencies up to kHz for nm thin films.

References

Adamovsky, S. A.; Minakov, A. A.; Schick, C., *Scanning microcalorimetry at high cooling rate,* Thermochim. Acta 403 (2003)1, 55 – 63

Adamovsky, S.; Schick, C., Ultra-fast isothermal calorimetry using thin film sensors, Thermochim. Acta 415 (2004) 1-7

Minakov, A.; Mordvintsev, D.; Schick, C., Melting and reorganization of poly(ethylene terephthalate) on fast heating (1000 K/s), Polymer 45 (2004) 3755-3763

Minakov, A. A.; Mordvintsev, D. A.; Schick, C., Isothermal reorganization of poly(ethylene terephthalate) revealed by fast calorimetry (1000 K/s-1; 5 ms), Faraday Discuss 128 (2005) 261

Minakov, A.A.; Adamovsky, S.A.; Schick, C., Non adiabatic thin-film (chip) nanocalorimetry, Thermochim. Acta 432/2 (2005) 177 – 185

Huth, H.; Minakov, A.; Schick, C., High Sensitive Differential AC-Chip Calorimeter for Nanogram Samples, Netsu Sokutei 32/2 (2005) 70 - 76

Lupascu, V.; Huth, H.; Schick, C.; Wübbenhorst, M., *Specific heat and dielectric relaxations in ultra-thin PS layers,* Thermochim. Acta 432/2 (2005) 222 – 228

Serghei, A.; Huth, H.; Schellenberger, M.; Schick, C.; Kremer, F., *Pattern formation in thin PS films induced by an enhanced mobility in ambient air*, Phys. Rev. E 71/1 (2005) 061801-1 - 4