SIMULTANEOUS MEASUREMENTS OF THERMAL AND DIELECTRIC PROPERTIES AS WELL AS SAXS/WAXS EXPERIMENTS DURING POLYMER CRYSTALLIZATION

Andreas Wurm
Institut für Physik. Universität Rostock

Polymer composites based on nano sized fillers such as those of montmorillonite (clay) type or carbon nanotubes (CNT) have attracted much interest during the last years. Composites based on CNT are very promising due to its high conductivity and the improved mechanical properties, even when containing only small amounts of carbon nanotubes. Furthermore there is evidence that the crystallization behavior is influenced by nano additives also. For example in [1] it is shown that clay particles act as nucleating agent for poly(epsilon-caprolactone) (PCL).

We have studied crystallization, cold crystallization behavior and isothermal crystallization behavior of iPP-CNT and PCL-CNT composites with different amounts of filler. Using DSC, HyperDSC and ultra-fast calorimetry scanning rates during heating and cooling in the range between 0.01 K/s and 100,000 K/s have been applied. The possibility of fast controlled cooling allows studying isothermal crystallization in the whole range between melting temperature and glass transition.

In order to understand the nucleation and crystallization in CNT-polymer composites simultaneous experiments with a combination of different methods are extremely useful. Due to different sample geometry and thermal conditions it is usually extremely difficult to compare the results of individually performed crystallisation experiments. As an important supplement to the classical techniques for crystallization (SAXS/WAXS or DSC), measurements of the AC conductivity and permittivity are of large interest for CNT-polymer composites. These experiments allow the investigation of the changes in the conductive percolation structure by crystallization. Two effects are expected to influence conductivity and dielectric properties: (i) crystallisation near the CNT and at the CNT-polymer-CNT contacts and (ii) crystallization in the bulk.

Therefore a microcalorimeter was designed, which allows to measure thermal properties like heat capacity and thermal conductivity, which can be used in a X-ray beam of a synchrotron, where simultaneously SAXS and WAXS patterns on the same sample volume can be recorded. On the other hand the microcalorimeter was extended for additional dielectric relaxation spectroscopy, again at the same time on the same sample volume.

REFERENCES

1. E. Di Maio, S. Iannace, L. Sorrentino, L. Nicolais, Polymer, 45, 26, 8893(2004)

2. A. Wurm, R. Soliman, J.G.P. Goossens, W. Bras, C. Schick, J Non-Crys. Solids, 351, 2773 (2005)

3. A. Wurm, R. Soliman, C. Schick, Polymer, 44, 7467 (2003)