

## Small Angle Scattering I Tools and Techniques

**MS14.01.01 THE DEVELOPMENT OF INSTRUMENTS FOR SMALL ANGLE SCATTERING OF NEUTRONS DURING THE LAST DECADES.** T. Springer, B. Alefeld and D. Schwahn, Forschungszentrum Jülich GmbH, Institut für Festkörperforschung, D-52425 Jülich, Germany.

Neutron small angle scattering is an efficient method to investigate mesoscopic structures in condensed matter physics, material science and biology, for dimensions between 1 and  $10^3$  nm, or Q-values between  $10^{-2}$  and  $10^{-5}$  Å<sup>-1</sup>. The most common instrument is the slit hole camera [1] with very long distances between entrance slit, sample and detector, e.g. up to 80 m for the well-known D11 at the ILL in Grenoble. For very high resolution, the *Bonse Hart camera* covers a Q-range between  $10^{-4}$  and  $10^{-5}$  Å<sup>-1</sup> using a pair of parallel ideal silicon crystals as collimator [2]. By multi-slit crystals the analyser can be multiplexed. Recently, we succeeded to build a prototype of a *focusing camera* [3] with a 4 m long copper-covered glass mirror of very high quality; the entrance slit is imaged in the detector plane. For the first time, Q-values down to a few  $10^{-4}$  Å<sup>-1</sup> were reached, with a very low parasitic background. This instrument is well suited for pulsed sources because the detector is off the primary beam, and the length is relatively small.

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**MS14.01.02 SIMULTANEOUS SAXS AND LIGHT SCATTERING MEASUREMENTS OF POLYMER CRYSTALLIZATION AND PHASE TRANSITIONS** Richard S. Stein and Yvonne Akpalu, University of Massachusetts; H. G. Zachmann, J. Cronauer, and S. Groth, University of Hamburg, Germany

Simultaneous small-angle x-ray (SAXS) and light scattering (SALS) and wide-angle x-ray diffraction (WAXD) measurements were made during the melting and crystallization of blends of linear and branched polyethylene.

The degrees of crystallinity were obtained from the WAXD, the invariants and identity periods from the SAXS, and the spherulite sizes and light scattering invariants from the SALS using H<sub>v</sub> and V<sub>v</sub> polarization. The V<sub>v</sub> SALS invariant exhibited a maximum when samples were about half filled with spherulites, whereas the H<sub>v</sub> invariant monotonically increased. The SALS intensity continued to increase after the spherulites became volume filling demonstrating secondary crystallization within previously formed spherulites. The identity period decreased in later stages of crystallization as the branched component crystallized.

Measurements were made during one-stage crystallization as well as during two-stage, where in the first stage, the temperature was first held below the melting point of the linear component but above that of the branched, and then in the second stage, it was held below the melting points of both. While differences were observed during crystallization for miscible and immiscible melts, the striking difference occurred during melting, where a scattering maximum occurred for the immiscible melt but not for the miscible.

**MS14.01.03 DEVELOPMENT AND APPLICATIONS OF MICROBEAM SMALL-ANGLE SCATTERING USING SYNCHROTRON RADIATION.** Christian Riekkel, ESRF, B.P.220, F-38043 Grenoble Cedex.

The brilliance of third generation synchrotron sources -such as ESRF, APS or Spring-8- allows new approaches in the development of microbeam small-angle Xray scattering cameras. Such cameras are particularly of interest for scanning SAXS applications. Bent monochromator/bent mirror optics has allowed to reach a line focus at the sample position of  $=150(h)*15(v)$  μm<sup>2</sup> at 0.09 nm for a flux of about  $10^{10}$  photons/sec[1]. A symmetric beam size of 2-3 μm diameter can be obtained by Bragg-Fresnel[2] or glass-capillary optics[3]. The status of both types of optics for low-angle experiments is discussed and exemplified by selected experiments in materials research.

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**MS14.01.04 IMAGING OF SMALL-ANGLE SCATTERING FOR ENHANCED RADIOGRAPHY.** Thomas Rieker, University of New Mexico, Advanced Materials Laboratory, 1001 University Blvd. S.E., Albuquerque, NM 87106

Small-angle x-ray or neutron scattering, which arises from differences in scattering contrast, can be imaged to produce radiographs of objects. This is unlike standard radiography which relies on absorption contrast. Since the fundamental mechanisms of scattering and absorption differ, new information can be gleaned. Of particular interest is the shifting of contrast between regions within an object by changing the q at which the small-angle image is produced. Small-angle radiographs make it possible to study objects with little or no absorption contrast. Scattering and absorption radiographs of a variety of objects using both neutrons and x-rays will be compared and the instrumentation used will be discussed.

**MS14.01.05 ORNL DOUBLE-CRYSTAL ULTRA SMALL-ANGLE NEUTRON SCATTERING FACILITY.** M. Agamalian, R. Triolo\*, G. D. Wignall, Oak Ridge National Laboratory, Oak Ridge, TN 37831

Conventional small-angle neutron scattering instruments (SANS) are widely used for structural studies in the resolution range  $10^3$  -  $10^4$  Å. The upper resolution limit can be extended by over two orders of magnitude (to  $\sim 2*10^{-5}$  Å) by using the Double Crystal Diffractometer (DCD) with the Bonse-Hart collimation technique. The ultimate resolution of the DCD is determined by the tails of the Bragg reflection curves, which may be minimized by multiple reflections in channel-cut crystals.

A new Double-Crystal Diffractometer for Ultra Small-Angle Neutron Scattering (USANS) has been recently built at High Flux Isotope Reactor, Oak Ridge National Laboratory using the Si(111) triple bounce channel-cut single crystals for both the monochromator and analyzer. The half-width of the rocking curve is about  $\pm 1.0$  arcsec, and the wavelength of the primary neutron beam is 2.59 Å.

The DCD has been tested by standard monodisperse polystyrene latex with the radius of  $2.5*10^4$  Å, as determined by optical microscopy. This radius determined by the DCD is  $2.48*10^4$  Å, in good agreement with microscopy. The minimum measurable scattering vector is  $Q_{min} = 3.5*10^{-5}$  Å<sup>-1</sup> which corresponds to the distance in real space  $2\pi/Q_{min} = 1.8*10^5$  Å

The USANS technique has also been used to study a dilute solution of flexible rodlike micelles in D<sub>2</sub>O. The radius of gyration obtained in the experiment is  $R_g = 1.9*10^4$  Å, which gives the length of the rodlike micelles  $L = 6.5*10^4$  Å, assuming a rigid rod model.

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