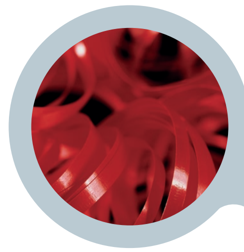
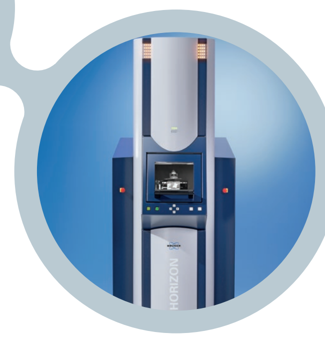


Polymers



N8 HORIZON

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N8 HORIZON: SAXS on Polymer Clay Nanocomposites

Small Angle X-ray Scattering (SAXS) is a truly non-destructive method for characterization of nanostructures, complementary to other methods e.g. electron microscopy or atomic force microscopy.

SAXS provides global, statistically relevant insight into the 3-dimensional nanostructural properties of the entire system in its native environment. The sample is positioned in the X-ray beam as is, without any cumbersome preparation that may alter the sample.

Polymer Clay Composites are very attractive materials for aerospace, automotive, and other engineering applications for their outstanding properties as stiffness, strength-to-weight ratio, fatigue and corrosion resistance …

Depending on the interaction between the nanoclays and polymer matrix different dispersion regimes are possible. Either the polymer just enters between the clay platelets (intercalation), or the polymer separates the clay platelets so that they don't interact anymore with each other (exfoliation), Figure 1. In practice, the exfoliated structure is preferred for most applications. SAXS provides this structural information right away.

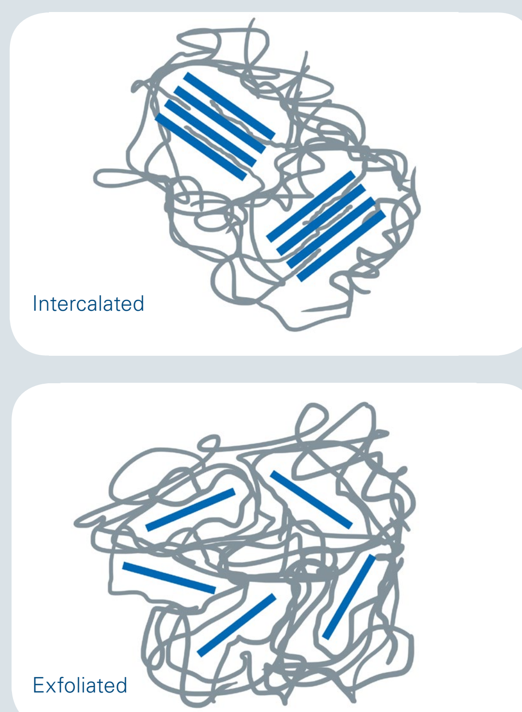

Figure 1: Intercalated and exfoliated structures

Measurement

A PP/SEBS/clay nano-composite sample was measured with the N8 HORIZON. Styrene-Ethylene-Butylene-Styrene (SEBS) block copolymer is added as compatibilizer to improve the dispersion of the clay particles in the polypropylene (PP) matrix.

The sample could be mounted directly to the sample holder frame without any further preparation. A 2D frame of the sample was collected for 1000s, as well as of the background, which was in this case the empty chamber (Figure 2). Due to the sample thickness, the sample transmission was extremely low (only 0.3%). Still data quality was by far sufficient for data evaluation. Extending the data collection time only improved statistics, but did not change the results. All measurements were done at room temperature.

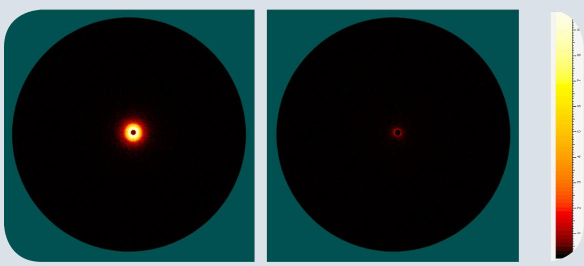

Figure 2: 2D Frames, both shown with the same color code for the intensity

Results

Data processing, including radial integration and background subtraction, and subsequent data analysis were all done with the comprehensive DIFFRAC.SAXS evaluation suite.

The radially integrated data are shown in Figure 3. At low angles the characteristic SAXS profile of the lamellar spacing of the polymer chains can be identified. This is even more clearly seen in a Kratky plot (Figure 4). The interlayer distance d in the lamellar polymer structure can be calculated from the position of the main peak at $q = 0.024 Å^{-1}$ via:

$$D = 2\pi / q_{peak}$$

The resulting lamellar thickness for this example is about 260Å.

The absence of any intensity increase in the Kratky plot towards high q indicates that for this example the structure is almost completely exfoliated.

Instrument configuration	
X-ray Souce	Air-cooled microfocus X-ray source (IµS)
Optics	MONTEL mirror
Collimation system	SCATEX pinholes, 2x 550µm
Beamstop	3.15mm
Detector	2D VÅNTEC-500, SDD 650mm

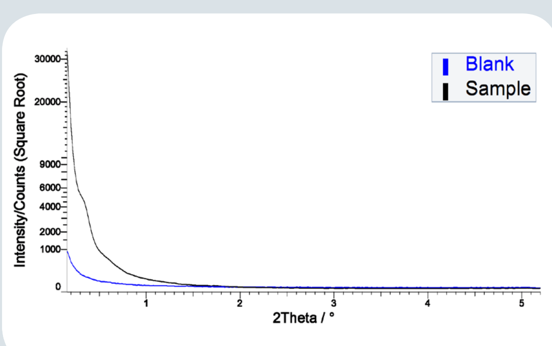

Figure 3: 1D data sets as a result of the radial integration

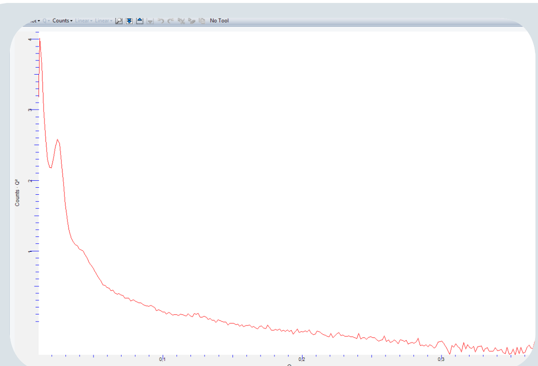

Figure 4: Kratky plot of background-subtracted data

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