

Figure 6:Optical image, nanograph and micro-fibril angle distribution map of a wood slice (by courtesy of J. Keckes)

#### Example 3 – sliced wood sample

Wood represents a complex hierarchical nanocomposite with helical structure. At the higher hierarchical levels, the wood morphology is dominated by annular rings and branches growing from the stem. Position-resolved SAXS/WAXS (Figure 6) on wood slices (8 cm x 4.5 cm; resolution of 0.5 mm) was used to characterize structural properties of the wood at the cross section of a branch and the stem. The position-resolved WAXS data show a varying texture of cellulose nano-fibrils, documenting gradients of helical micro-fibril angle (MFA) across the sample. Since the high MFA correspond to higher flexibility and toughness, the position-resolved measurements reveal also the mechanical function of various wood features. The 2-D MFA distribution nicely illustrates that the stem centers and branch are optimized for higher strains.

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Figure 1: X-ray Nanography set-up

## General

X-ray Nanography, or two-dimensional scanning Small Angle X-Ray Scattering (2-D scanning SAXS), is a powerful method for displaying and investigating the microscopic structure of specimens on the µm-scale. For instance, Nanography shows inhomogeneities such as different chemical compositions or varying density within a sample. In this way, Nanography combines the nanoworld with the microworld. A Nanography investigation requires a specimen to be mounted on a motor-driven XY stage, in order to automatically scan the sample through the X-ray beam.

Each individual point within the nanograph represents the integral SAXS/WAXS intensity collected by the 2-D detector at this position. The intensity distribution is displayed by means of a color-coded contour plot.



Figure 2: Principle of Nanography

Nanographs can be collected in two ways:

- The first method uses glassy carbon, which is a strong isotropic scatterer in the forward direction, behind the sample (Figure 2, transmission geometry). The displayed intensities are a measure for the density variations in the sample.
- The second method does not use any additional standard. In this case, the intensity at a certain position can be interpreted as a measure of scattering centers within the sample.

The resulting nanographs obtained with both methods are different but complementary, as can be seen in Figure 3.

With the NANOSTAR, the user can select several spots of interest on the nanograph via mouse-click and run detailed SAXS measurements automatically. These subsequent SAXS measurements can be done without removing the sample.

Up to 18 samples can be positioned on the NANOSTAR's large XY stage and scanned sequentially using measurement routines that are optimized for the individual samples.



Figure 3: Vertebra of an 18-year old woman. Left: transmission; right: scattering intensity (by courtesy of S. Rinnerthaler)

# **Applications**

## Example 1 – sliced bone sample

Figure 4 shows a detailed scan on a sliced bone sample (rat femur). Although the beam diameter on the sample is about 200  $\mu$ m, a smaller step size of 50  $\mu$ m was chosen. Selecting

a smaller step size may be advantageous for scanning very inhomogeneous samples since it may give smoother pictures.



Figure 4: Detailed scan of sliced rat femur (sample provided by A.Valenta)

## Example 2 – homogeneous samples

Also in the case of homogeneous samples showing a non-uniform thickness, e.g. a powder sample mounted between two foils, Nanography is often useful. From theory it follows that the ideal sample thickness for obtaining maximum scattering intensity is the reciprocal value of the sample's linear absorption coefficient, i.e. the transmission is 1/e  $\approx$  0.37. Hence, Nanography can be used to select a position with optimum transmission, prior to the actual SAXS measurement. Figure 5 shows nanographs of two such cases. The transmission is displayed in different colors (e.g. white – transparent, black – opaque)



Figure 5: Nanographs of homogeneous samples