# More stable paper packaging in variable environments

BillerudKörsnas, a Swedish pulp and paper manufacturer, has been looking at moisture-induced mechanical failure of paper. Together with the Research Institute of Sweden (RISE) and DESY, BillerudKörsnas conducted synchrotron radiation-based X-ray scattering experiments at DESY's PETRA III facility to observe the so-called mechano-sorptive creep (MSC) mechanism in situ. MSC is the increased creep that occurs under conditions of varying humidity and is not yet properly understood. Understanding MSC should provide solutions to improve the robustness of paper products against moisture with the ultimate goal to design a sustainable alternative to plastic products.

## CHALLENGE

Studies already exist on the effect of MSC on paper, but an adequate explanation of the underlying mechanism has yet to be found. Part of the reason for this is the complex microstructure of paper fibrils containing crystalline cellulose and non-crystalline cellulose. To get a more complete picture of MSC, the structural changes of a single crystal and of the fibrils, composed of many crystals, have to be examined at different humidity levels.
Wide-angle X-ray scattering (WAXS) and small-angle X-ray scattering (SAXS) can be applied at these length scales, but standard lab devices struggle with unordered samples such as a sheet of paper. Synchrotron radiation delivers higher quality data for unordered samples while allowing the dynamics of MSC to be studied by incorporating experiments on mechanical properties, such as stress-strain and creep tests, during time-resolved SAXS and WAXS measurements. As a high-brilliance and high-flux X-ray source, PETRA III allows rapid data collection together with higher quality data for unordered samples and therefore provides the experimental backbone for this study.

## METHOD

In a first step, a setup was built by RISE and positioned inside the beamline which allowed a tensile tester to rest inside a climate-controlled chamber to control and modify the relative humidity in the course of the experiment. At the PETRA III beamline P03 Micro- and Nano-focus X-ray Scattering (MiNaXs) WAXS and SAXS were used to identify the crystal structure at different relative humidity levels. Simultaneously macroscopic mechanical properties of the paper samples were examined using standard methods such as stress-strain tests, creep tests and mechano-sorptive creep measurements. Paper samples with high fiber orientation and a high cellulose content were provided by the industrial partner BillerudKörsnas.

Corrugated paper boxes are frequently used as lightweight but fairly robust storage containers. Nonetheless, changes in humidity can lead to structural failure, causing damage to fresh and sensitive produce.

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## INSIGTHS AND ANALYSIS

The WAXS measurements revealed a change in the cellulose unit crystal for varying relative humidity. Cellulose crystals were strained in the longitudinal direction and compressed along the transverse crystal direction with increasing humidity. If the humidity is reduced again, the straining and compression diminish again, showing that the mechanism involved in MSC is in fact reversible. At first sight, the changes in size seem contradictory, but the scientists were able to formulate a reasonable hypothesis about MSC from the scattering data. The non-crystalline cellulose molecules on the surface of the fibrils swell in a lateral and longitudinal direction upon absorbing water, resulting in lateral compressive forces and axial extensive forces on the underlying crystalline regions, which ultimately result in structural failure if the forces on the fibers become too large.

SAXS measurements were also able to track changes in the fibrils during the different mechanical tests, such as stress experiments over time and mechano-sorptive creep tests. Like the WAXS experiments, SAXS showed that both mechanical forces and humidity lead to an immediate but reversible change in the orientation of the fibrils.

## BENEFITS

The high-flux X-rays from the synchrotron source PETRA III allowed X-ray scattering experiments and mechanical tests to be performed on paper at the same time. Despite the fact that the microstructure of paper is naturally unordered, the high brilliance of PETRA III allowed experiments to produce high-quality data and experiments to be conducted with the necessary temporal resolution. Using WAXS and SAXS measurements a deeper understanding of the influence of humidity on paper products and extension on existing theories about MSC with new data on the nanometer scale was gained.

The new insights gained at PETRA III have brought the paper manufacturer BillerudKörsnas a step closer to developing paper products that are more durable under humid conditions. The improved knowledge of the underlying mechanism of MSC on the nanometer scale will help the design and development of new paper products. Humidity-resistant paper products could in future become an alternative renewable resource and reduce the demand for oil-based plastic products. In the meantime, synchrotron radiation from PETRA III will continue to help tackle complicated and dynamic problems.

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