Combined μXRD and μXRF mapping of hierarchical materials – atomic structure, crystallographic texture and chemical mapping over large areas.

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It is textbook knowledge that a material's atomic structure determines its properties. However, nature and humans have developed hierarchical materials, where structures and properties at one length scale influence those at higher length scales [1]. Studying the structure of these complex materials often requires both traditional crystallographic methods and other methods in combination. “Conventional” powder diffraction provides accurate information on the average atomic structure of the probed powder. However, to properly study hierarchical materials, we require spatial resolution while maintaining accurate information on the atomic structure, and preferably in combination with other modalities.

The low-emittance MAX IV (Lund, Sweden), the world’s first 4th-generation synchrotron [2], allows us to easily and quickly focus the X-ray beam on the micron scale. At the DanMAX beamline, we have implemented fast linear scanning stages, hardware detector triggering, and optimized scanning strategies. Thus, it is possible to raster-scan macroscopic samples of thousands of square millimeters at micrometer resolution.

A photon-counting CdTe hybrid pixel array detector captures structural data for the crystalline components and texture contrast from the azimuthal variation of the diffraction rings. An X-ray fluorescence detector simultaneously captures local spectroscopic data to map the sample's elemental composition. Diffraction mapping techniques produce large amounts of data, not uncommonly hundreds of thousands or even millions of diffraction patterns. Efficient data pipelines at MAX IV allow for live azimuthal integration of the collected area detector images [3], and the efficient data structure at DanMAX enables data analysis immediately following the data collection [4, 5]. A wide range of Python-based Jupyter notebooks makes it easy and quick to perform qualitative analysis of the large mapping datasets.

The combination of spatially resolved structural data (XRD), texture data, and spectroscopic data (XRF), can e.g. be used to map element deposition during growth, hierarchical crystallization, and crystallite orientations, as seen in e.g., bones [6], shells, and teeth. With texture mapping, we can follow, e.g., domain switching under high voltage [7] or template-induced preferred crystallite orientations or in additively manufactured ceramics, aiding in the understanding of these highly specialized piezoelectric components.

Here we will showcase examples of how the system has been used to study, e.g, battery degradation in aged pouch cell batteries, induced texture in 3D printed piezoelectric, growth of cow bones [6], and texture in crystallized elements in lizard skin [8, 9]. Common to the previously mentioned materials is their hierarchical nature.

[1] R. Lakes, Nature, (1993). 361, 511-515.
[2] P. F. Tavares, E. Al-Dmour, A. Andersson, et al., J. Synchrotron Rad. (2018). 25, 1291–1316.
[3] A. B. Jensen, T. E. K. Christensen, C. Weninger, H. Birkedal, J. Synchrotron Rad. (2022). 29, 1420–1428.
[4] https://manual.nexusformat.org/classes/applications/NXazint1d.html (accessed 13/3-2026)
[5] https://github.com/fgjorup/plaid (accessed 13/3-2026)
[6] A. Rodriguez-Palomo, P. A. S. Vibe, M. R. V. Jørgensen, H. Birkedal, Faraday Discuss. (2025) 261, 116-131.
[7] A. Pramanick, C. Babori, F. Albertini, et al., Adv. Eng. Mater. (2025). 27, 2402104.
[8] A. Rodriguez-Palomo, M. Didziokas, M. S. B. Jacobsen, et al., Adv. Eng. Mater. (2025). 27, e26169.
[9] A. Rodriguez-Palomo, M. S. Jacobsen, T. E. K. Christensen, et al., Acta Biomaterialia. (2025), 204, 457-469.