

Time- and Spatially-Resolved X-ray Diffraction Using the Timepix4 Pixel Detector for Steel Lattice Observations

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Abstract

Time-resolved X-ray diffraction (TR-XRD) in fan-beam geometry, enables simultaneous spatial and temporal mapping of structural evolution in materials. The data-driven readout, based on the Timepix4 hybrid pixel detector, delivers per-photon energy resolution (~ 1 keV) and time-of-arrival (≤ 200 ps) information at rates up to 600 MHit/s, allowing real-time reconstruction of scattering angles from charge-shared clusters across multiple energy bins. This approach transforms spatially distributed diffraction patterns into position-sensitive diffractograms, revealing local phase kinetics along the beam path. Applied to advanced heat treatments of high-strength steels, the technique captures spatially resolved austenite decomposition, carbon partitioning, and texture development during Quenching and Partitioning (Q-P) cycles. By correlating processing gradients with microstructural heterogeneity in bulk samples, TR-XRD accelerates alloy optimization for lightweight, high-performance automotive and structural applications.

Instrumentation

For the measurement, a data acquisition chain based on the newly developed Timetpix4 was used. This setup allows measurement in data-driven mode, where each hit pixel is accompanied by information about the time of arrival and energy. The processing chain was developed at the University of West Bohemia and the Institute of Experimental and Applied Physics (CTU). [1]

Timepix4

Timepix4, developed by the Medipix collaboration, offers a sensitive area of 7 cm^2 , composed of 512×448 pixels, each $55 \mu\text{m} \times 55 \mu\text{m}$ in size. It supports both traditional frame-based acquisition and a data-driven mode, where each pixel hit is read out independently. In this mode, the detector delivers energy measurements with a resolution of approximately one keV, along with time-of-arrival information with a precision of up to 200 picoseconds.

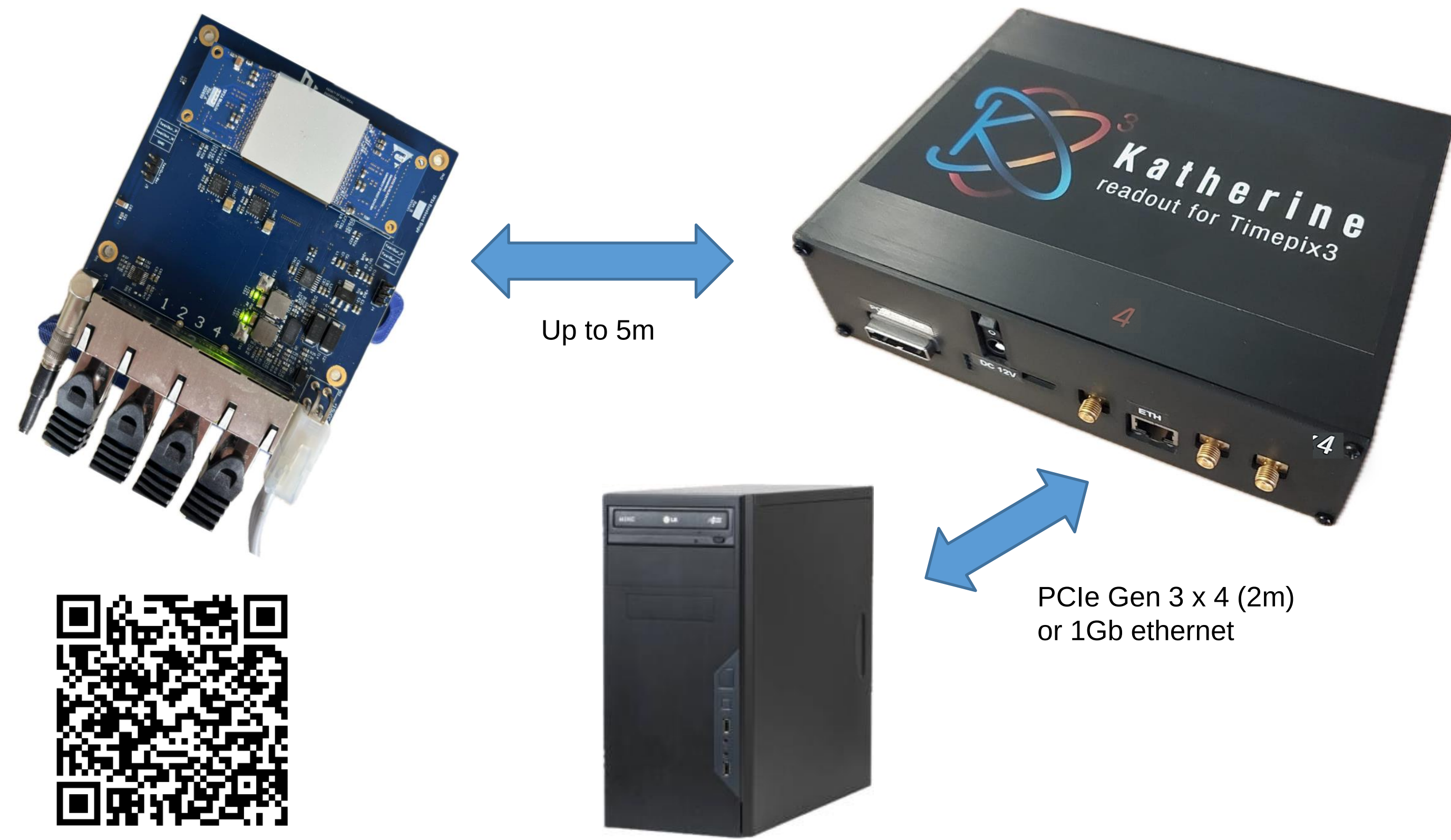


Fig. 1 Measurement setup with Timepix4 chipboard and Katherine readout. The detector module is connected to the readout using 5x UTP cables. The interconnection with a PC can be established either via 1Gb Ethernet for smaller data rates or PCIe Gen 3 in cases of high-throughput demand.

The chipboard consists of two main parts: a sensor module and a mainboard, designed for flexibility, performance, and measurement accuracy. The sensor module is based on the Timepix4 chip, optimized for high-speed data transfer (up to 40 Gbps, ~ 600 MHit/s) and reliable operation without the need for additional active components. Its modular design allows easy integration of different sensors and large-area detector configurations. The mainboard provides power and signal interfacing, using a dual-stage low-noise supply for stable operation and supporting data transfer rates up to 5 Gbps per differential pair. It connects to the readout electronics via four RJ45 ports, enabling versatile setup options. A Peltier module is used to stabilize the detector's temperature at the required value.

Kathrine readout

The readout ecosystem "Katherine Readout" for Timepix4-based detectors is built around a modular "generic detector platform" chassis and plug-in interconnect board, featuring an Intel Arria 10 SoC (dual-core Cortex-A9 + FPGA) with 2 GB DDR4, integrated bias voltage (± 1 kV with leakage measurement), sensor power (12 V), and flexible I/O (GPIO, Gigabit Ethernet UDP, PCIe Gen3 $\times 4$). Data streams (ToT/ToA) are pre-decoded and relayed via eight high-speed input lines (2.56 Gbps) and an external-triggered TDC (50 ps bins) before transmission. Software support includes the BURDAMAN and Track Lab suites for control, calibration (DAC scans, threshold equalization), and data acquisition, with multi-day acquisitions possible thanks to extended ToA bit length and logical pixel addressing. Initial performance shows ~ 11 Mhit/s via Gigabit Ethernet and 30 Mhit/s via PCIe implementation. (Goal of ~ 350 Mhit/s and multi-chip support are under development).

Fan beam XRD

X-ray diffraction (XRD) is an analytical technique used to investigate the structural properties of crystalline materials. When a monochromatic beam of X-rays interacts with a periodic crystal lattice, it is diffracted according to Bragg's law, producing characteristic patterns that reveal the atomic arrangement, lattice parameters, and crystallographic orientation. XRD is widely used across materials science, physics, and chemistry to identify phases, determine crystallite size and strain, and study structural changes under varying environmental conditions such as temperature, pressure, or applied stress.[2]

Conventionally, XRD measurements usually use a pencil beam. This measurement example is shown in Figure 3. It can be observed that the sample examined in this experiment does not produce typical Debye-Scherrer rings, but instead clusters, due to its large grain size. Data obtained using this method are easy to evaluate, but provide information about a single spot only.

Fan-beam XRD employs a narrow, divergent sheet of X-rays that illuminates an extended region of the sample simultaneously, enabling both spatially and time-resolved diffraction measurements. By combining the fan-beam geometry with a position-sensitive 2D detector, it becomes possible to capture diffraction information from multiple spatial locations simultaneously and monitor dynamic structural changes in real-time. This approach significantly enhances temporal resolution, allowing for the mapping of inhomogeneous samples, phase transitions, or mechanical responses with both spatial and temporal precision—making it particularly valuable for in situ and operando experiments.

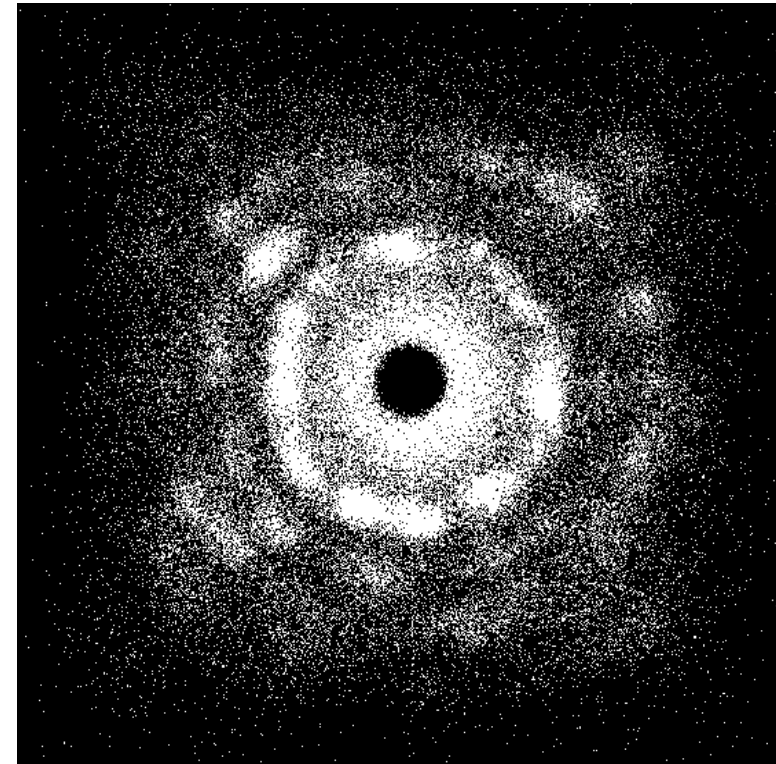


Fig. 2 Pencil beam measurement example.

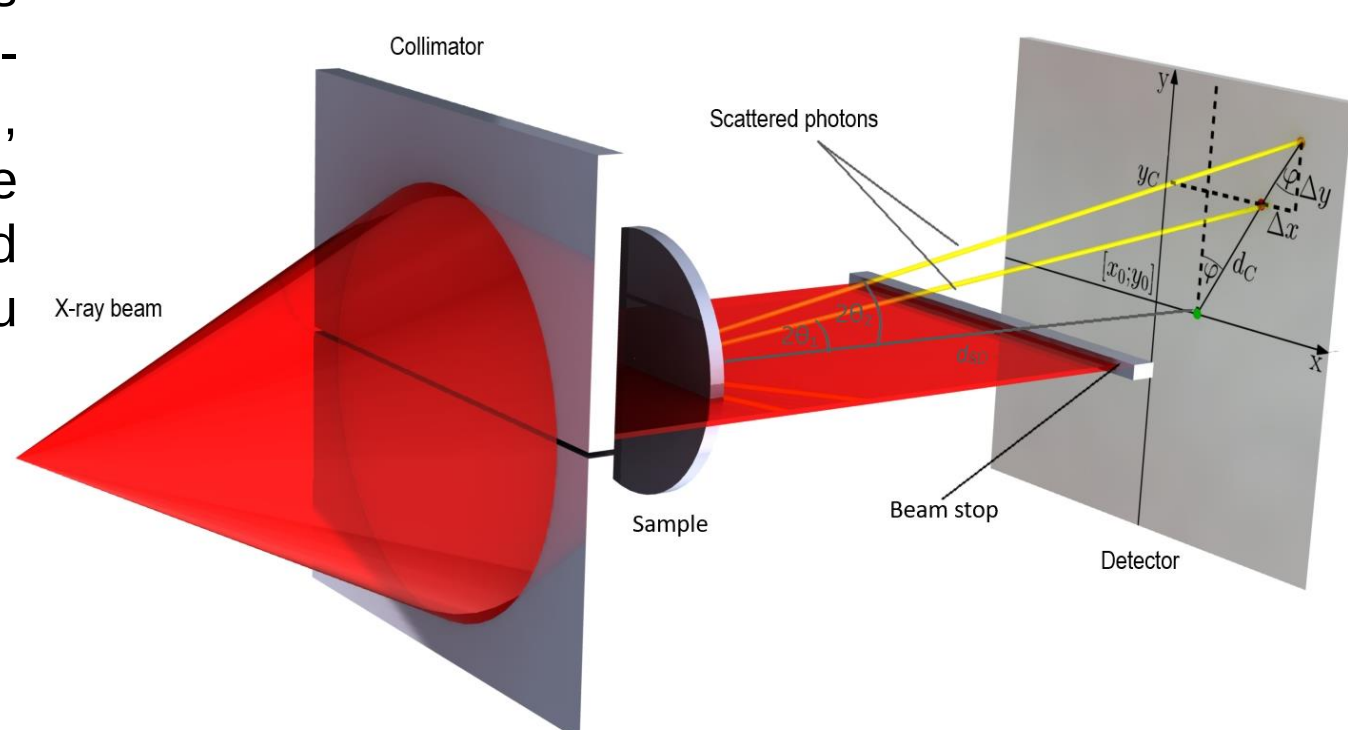


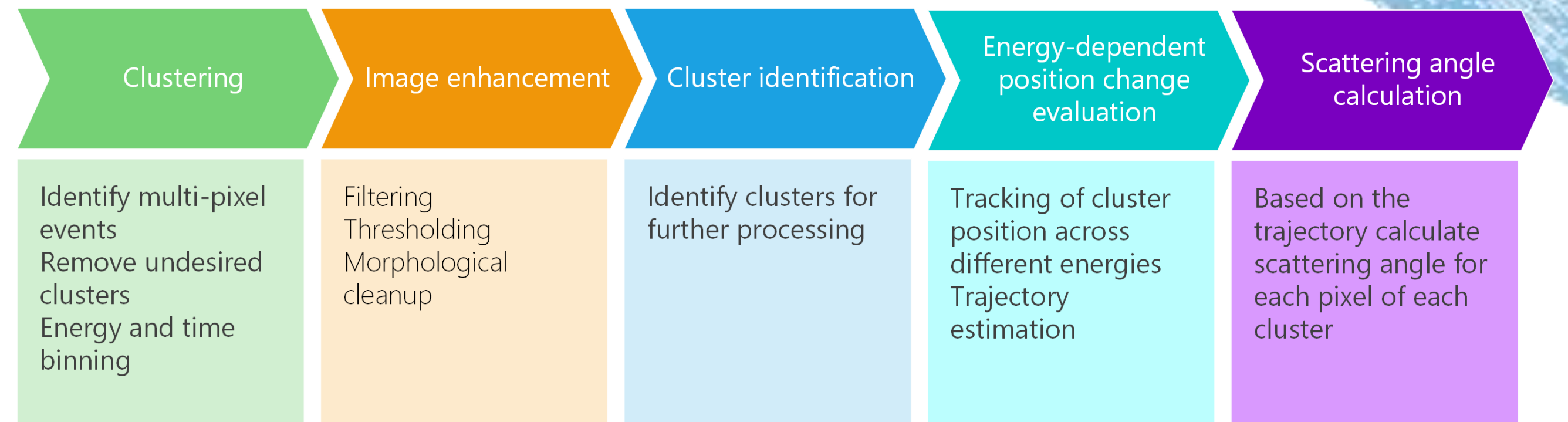
Fig. 3 Setup example for fan-beam XRD.

Data processing

The data processing chain exploits the time and energy information of each pixel. The first step is clustering. In this step, the stream of pixels is analyzed to identify clusters created by charge-sharing between neighboring pixels. These clusters are then filtered to reject hits caused by other particles but x-rays (e.g. alpha particles).

Then the energy and time information is used to create a 4D array: a series of pixel-hit-based images for each energy and time bin. This step already allows the user to select a specific time window to analyze.

Since the fan-beam geometry produces spatially distributed patterns rather than standard Debye-Scherrer rings, advanced processing is required. In this process, several steps are needed to retrieve the patterns (clusters) from the image. This must be performed for multiple energy bins, allowing the cluster shift direction to be observed, so that the origin of the cluster and then the scattering angle can be reconstructed.



The real example of measured data for a specific energy and time bins is shown below. One of the clusters is highlighted, allowing the position change for various energies to be visible.

Data were collected using a setup described earlier and a tabletop X-ray tube (Amptek Mini-X2) at 50kV.

The sample selected for the measurement was a $400 \mu\text{m}$ thick austenitic steel plate.

This sample was thermally treated to allow growth of large grains (visible in the images below).

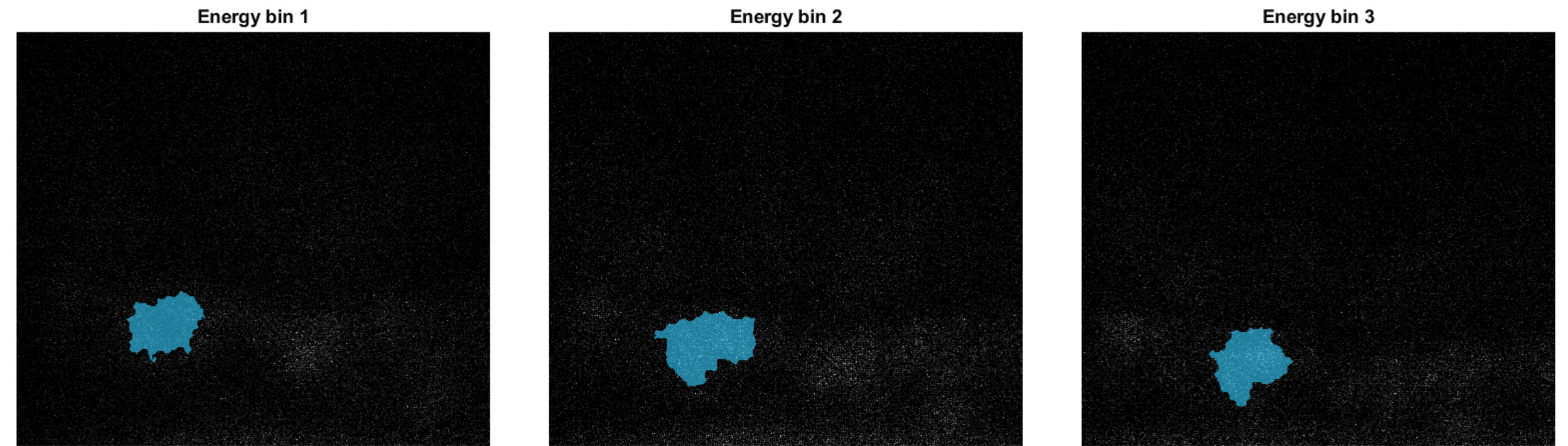


Fig. 4 Process of the cluster matching, tracking, and restoration of its origin

Application in novel steel development

Time-resolved X-ray diffraction (TR-XRD) has proven to be an excellent tool in the development of novel steel alloys, enabling real-time monitoring of phase transformations, lattice parameter evolution, and microstructural changes during advanced heat treatments, such as Quenching and Partitioning (Q-P). [4]

The utilization of a fan-beam promises to enhance its capabilities by providing a spatial resolution along the fan line. Providing information about both the location and kinetics of austenite-to-martensite or bainite transitions, carbon partitioning dynamics, and retained austenite stability under controlled heating, isothermal holding, and rapid quenching cycles. This information is crucial for optimizing the mechanical properties of the alloys, including strength, ductility, and toughness.[5]

By observing the variations in the diffracted pattern and its peak shifts in situ, processing parameters can be optimized to achieve the desired phase fractions and texture development, as well as residual stress states in bulk samples, thereby accelerating alloy design iterations and reducing reliance on post-mortem analysis.

This capability has great potential for accelerating the development of next-generation steels tailored for lightweight automotive structures, energy-efficient manufacturing, and high-performance engineering applications.

This potential is further enhanced by the simplicity of setup, which utilizes a polychromatic beam, eliminating the need for complex geometry elements such as monochromators.

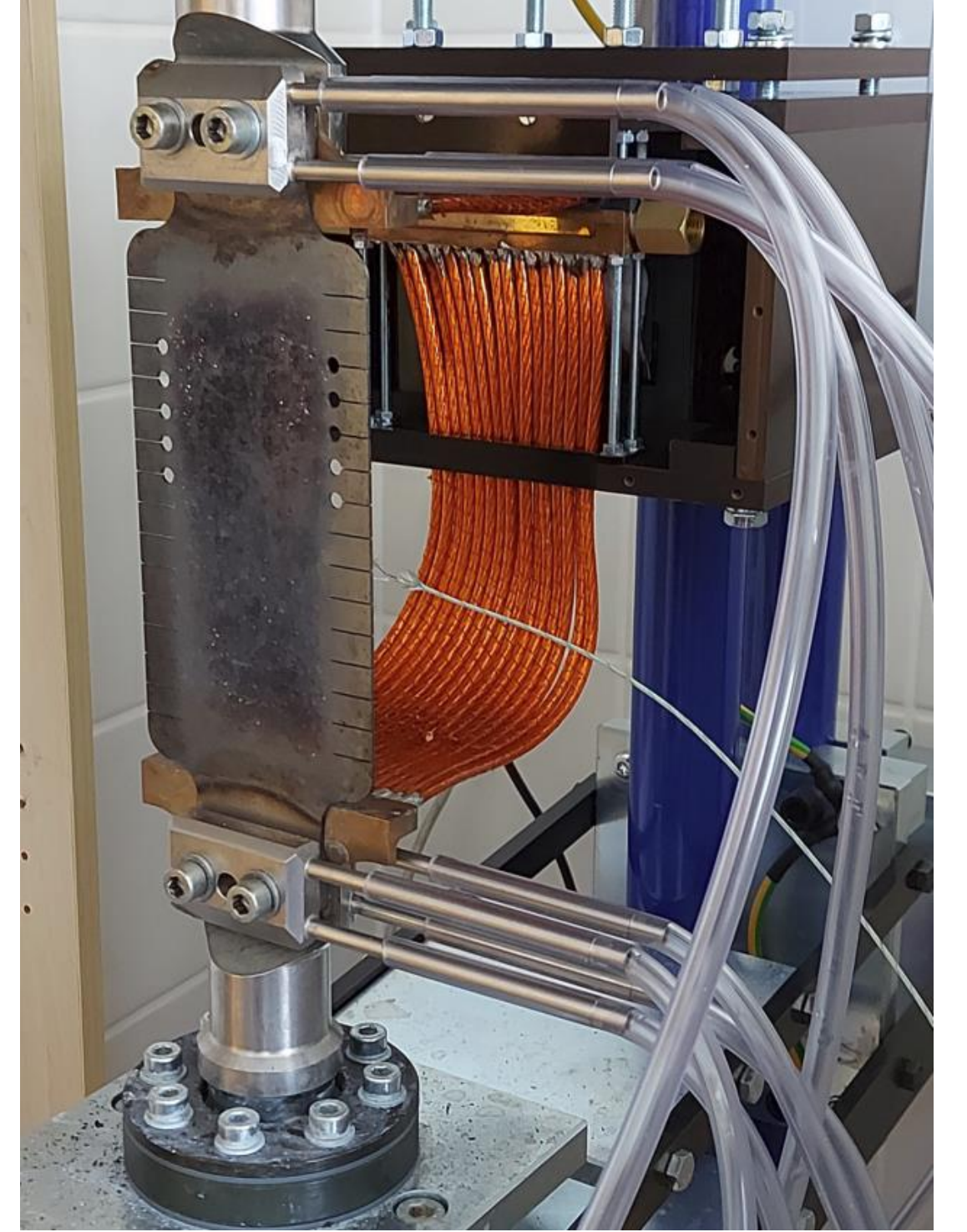


Fig. 5 Example of a thermomechanical simulator with a steel alloy sample.

CONCLUSIONS

The integration of Timepix4 with fan-beam XRD establishes a laboratory-scale platform for spatially and temporally resolved diffraction studies of dynamic processes in metals. Real-time cluster tracking and energy-selective reconstruction yield scattering-angle maps with sub-millimeter spatial resolution along the fan line and second-scale temporal precision, even with low-flux tabletop sources. In novel steel development, this capability directly visualizes location-specific phase transformation rates, retained austenite stability, and residual stress gradients under realistic thermomechanical conditions. The method significantly reduces design iteration cycles by replacing post-mortem characterization with in-operando feedback, paving the way for the rapid tailoring of next-generation AHSS and UHSS alloys with a superior strength-ductility balance for energy-efficient and sustainable engineering solutions.

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