ESRF	**Experiment title:** Time-resolved X-ray multi-projection imaging for materials science	**Experiment number**: MI-1449
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Report:

Overview: The experiment aimed to demonstrate time-resolved X-ray multi-projection imaging (XMPI), employing a novel beam-splitting scheme[1], for the acquisition of 3D movies of processes that cannot be recorded with state-of-the-art time-resolved 3D imaging techniques, such as time-resolved tomography. The performance of such techniques is based on fast rotations, which impose mechanical constraints on the setup and samples (which may limit the achievable resolution or the experimental conditions) and can introduce shear forces (which may alter the studied processes). XMPI is developed[2] to overcome these limitations. It has two cores; the experimental apparatus and the data reconstruction. The main focus of this experiment was to demonstrate the instrumentation of the experimental apparatus for the first time and its application for the study of two material science samples governed by fast dynamics: granular systems and metallic foams.

Experimental apparatus: XMPI provides multiple projections by splitting the main beam into secondary beams using crystals oriented in diffraction conditions. They are placed in the beam's path sequentially, each diffracting a portion of the energy spectrum and transmitting the non-diffracted or absorbed part. Each crystal is mounted on a motor assembly with three linear translations and three angular degrees of freedom for their positioning and orientation in diffraction conditions for a specific part of the spectrum. The arrangement of the crystals is such that the diffracted beams intersect at a common point, where the sample is placed and can be

[1] Vagovic, P., Bellucci, V., Villanueva-Perez, P., Yashiro, W.: Hard X-ray Multi-Projection Imaging system for SASE beams (patent pending, application number: EP21200564.9, 1. 10. 2021)

[2] This work received funding by 1) HORIZON-EIC-2021-PATHFINDEROPEN-01-01, MHz--TOMOSCOPY project, Grant agreement: 101046448, 2) ERC-STG-2020, 3DX-FLASH (948426), and 3) INVISION RAC project, BMBF-05K18XXA, VR 2017-06719.

imaged simultaneously by the beams. Detectors[3] are aligned with respect to the diffracted beams and placed around the sample. They provided a field-of-view and pixel size of 4×4 mm^2 and 4 µm, respectively.

XMPI Commissioning: First, a bent C-333 crystal was used, as an energy-dispersive element, to assess and monitor the beam's spectrum and characterize beam-splitting crystal candidates in the conditions of ID19. The characterization was done with respect to their spectral response during diffraction by monitoring the dips on the bent crystal spectrum (Fig.1a)[4]. The central energy was estimated to be 17.25 keV. The Si-111, Si-220, and Ge-400 crystals were identified as optimal for the setup and were used for the final configuration. The Ge-400 was very thick (1 mm), and a FLIR A7600sc thermal camera was added to the setup to monitor its thermal load (Fig.1b) due to its higher absorbance. The final configuration allowed synchronous recording up to 3kHz with the three cameras. This constituted an important milestone in the project. It serves as an experimental proof of concept for the commissioned setup and marks the starting point for further improvements.

Measurements: Both samples' processes were studied with 1 kHz acquisition rate to match their expected dynamics while maximizing the recordings' exposure times. Interesting kinematics were recorded (one example is shown in Fig.1c). More precise results will be obtained with the completion of the data 3D reconstruction. Preliminary results from the thermal camera indicate that the Ge crystal can withstand the monitored interaction conditions for 15 min. without permanent damage.

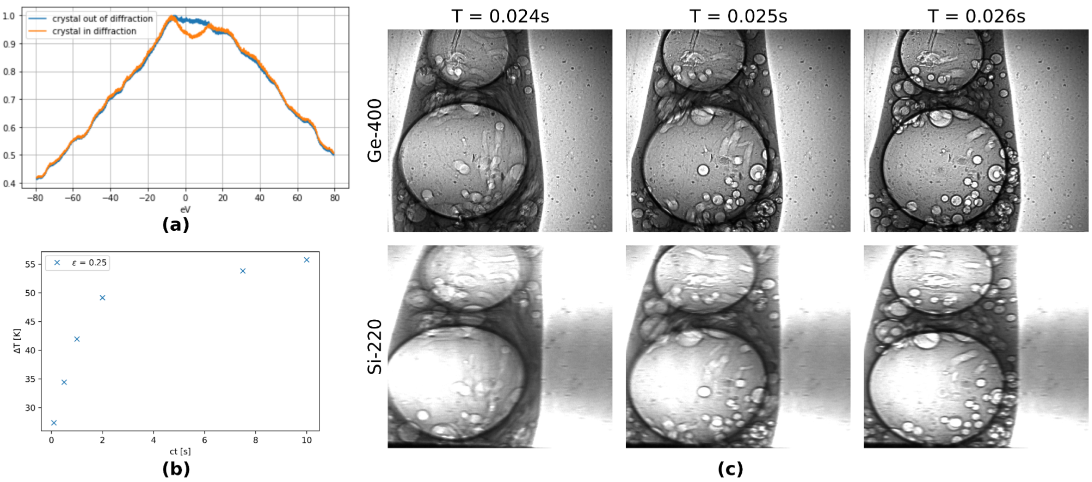

Fig.1a The spectrometer interacts with 160 eV of the beam spectrum. The blue distribution is a spectrum measurement when the Si-220 crystal is in the beam path but very far off the Bragg condition. The orange distribution is a spectrum measurement when Si-220 is in Laue condition and a portion of the beam is diffracted.
Fig.1b Dependence of maximum temperature change (ΔT) on the time the shutter remains open (ct). The calculations assume a relative emissivity coefficient of Germanium $\epsilon = 0.25$. The plotted results are part of an ongoing analysis.
Fig.1c Frames from one of the metallic foam samples. An instance of bubble movement can be seen. The projections from the diffracted beam of Ge-400 and Si-220 are shown in the top and bottom row respectively. The columns correspond to the relevant timestamps when the event took place.

Discussion and Outlook: The experiment was an important step towards establishing XMPI as a user technique that can record volumetric information without rotating the sample, enabling the possibility to image processes that could not be explored before. Exploiting the unique capabilities of ID19, we have managed to reach 3 kHz recording speed, using almost the full dynamical range of the detector system, and have reached the targeted goals for this experiment. We were also able to identify weak points in the beam-splitting crystals. Si-111 was experiencing intense vibrations, and it is expected that only projections from Si-220 and Ge-400 will be used for the data reconstruction. Based on this experience, we are already working on updated fabrication and mounting procedures to address the encountered challenges. We have also designed a new beam-splitting scheme tailored to the beam characteristics of ID19 that will enable at least three projections with a wider angular span between the diffracted beams. The increased angular spacing will benefit further the reconstruction and enable the tackling of more complex processes. The results and an extended summary of the work, including the capabilities of XMPI at ESRF, will be submitted for publication. A new proposal is being drafted for the continuation of the experiments at ID19 to establish XMPI. The beamline conditions are considered optimal for the commissioning of XMPI's full potential. The proposal will target to extend the recording speeds to 10 kHz and will be deployed for the study of fiber-reinforced composites under loading conditions, with a spatial resolution of 10 µm. These systems are governed by fast dynamics, with events of interest happening at unmapped timeframes; they occur at different times during the process and at varying rates (sub-millisecond to a fraction of a second).

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[3] An indirect detecting scheme was used, composed of i) an efficient scintillator, ii) a 90° mirror that redirects the visible light to a motorised microscope configuration which focuses the light on the camera sensor, iii) a magnification system (visible microscope), and iv) fast cameras optimised for kHz and beyond acquisitions (Photron Nova S16 models).

[4] The data were recorded with a 1x indirect detector with a PCO.edge.5.5 camera, provided by the beamline.