MI-1398: Imaging of ultrafast wavefront from compressed Ni Micro-pillars using Tele-ptychography.

Angel Rodriguez-Fernandez[1], Mariana Verezhak[2], Ana Diaz[2], Magnus Hornqvist colliander[3] and Dina Carbone[4]

[1]*Eu XFEL GmbH, DE-22869 Schenefeld, Germany*
[2]*PSI, CH-5232 Villigen PSI, Switzerland*
[3]*Chalmers University of Technology,, SE-412 96 Gothenburg, Sweden.*
[4]*MAX IV, Lund Univeristy, SE-224 84 Lund, Sweden*

A) Overview

We wanted to obtain a quantitative map of the strain field in compressed Ni micro-pillars by the analysis of dynamical diffraction (DD) effects, so called echoes, that consists of finely structured intensity strongly dependent on the crystalline properties of the sample. To this aim we used a combination of nanobeam and inversion of coherent data described in our previous work [1-4]. In our previous experiment [2] the DD from a Si strained wafer was measured in the forward direction, where absorption effects compete with the transmitted beam in intensity ratio. We exploited the diffraction direction in this case for studying both a Si wafer to compare with the results in the forward direction and a series of 5 μm thick pillars FIB into a perfect Ni crystal. The echoes in this Ni micropillars have a mutual distance of less than 0.5 μm. Therefore, a proper measurement of this signal can only be done using a nano-focused beam in combination with a high-resolution technique as tele-ptychography [3,4]. This permits to reconstruct the wavefront at the focus with tens of nm resolution [2]. This experiment is part of a larger project that aims at demonstrating that DD, which can be dominant even in diffraction from microscopic crystals, not only cannot be disregarded in the analysis of diffraction data, but can even be exploited for a better understanding of their structure under static and dynamical deformation.

As presented in Fig.1, a number of square-shape micro-pillars with dimensions of 5x5x15 μm^3 will be manufactured at the edge of a Ni (100) single crystal using FIB milling. Some of these Ni micro-pillars will be compressed to different degrees using a SEM compatible nano-indenter. The compression along the (001) allows easy activation of the (111) family of slip planes. The samples preparation and pre-characterization is done at Chalmers University of Technology [5].

B) Quality of measurement/data

The sample was mounted in the center of rotation of the goniometer, in the focal plane of the FZP (with a foci of 76 nm at 8.3 keV) on the PI x-y piezo stage. A pinhole with diameter of 3 µm was mounted on a Npoint stage (provided by the users) to scan the pinhole in the plane perpendicular to the diffracted beam, at few mm beyond the sample. The Eiger detector was placed 5.3 m downstream the pinhole in the diffraction direction, 2θ~43.16° to collect the DD signal [2.

The Ni micro-pillar were aligned to the asymmetric (111) LD reflection. A scanning diffraction map of the sample, taken without the pinhole, was performed to localize the pillars and select areas to investigate. The selected areas from the map were measured using a pinhole scan over an area of 10x10 μm^2, enough to collect the echoes produced.

We have several problems during the experiment as the new control software was implemented during that week. Also the detector Eiger2M (ESRF-PSI collaboration) due to some errors limited our time of collection. Everything was solved and the team of ID01 also gave extra inhouse beamtime to us to recover for the inconvenience. The support of ID01 was really good also, as due to COVID a lot of members of our group were not able to attend to the experiment.

C) Status and progress of evaluation

The part of the data is analysed a manuscript has been written for submission in Optica in relation to the Si wafer studies. The Ni data is still under analysis.

D) Results

As presented in the Figure 2, we first repeated the experiment from [2], in the diffraction direction to be convinced that the reconstruction algorithm will work for the diffraction direction. Also in that we were able to improve our knowledge of the technique and optimized parameters for the scan of the pinhole during the ptychographic scans. We studied the same sample as in our previous work [2], in this study we observed the reflection (220) of Si at the energy of 8.345 keV in Laue geometry. As it is possible to observe from Figure 2 (a) the signal is more than 80 μm wide. If we propagate the beam to the focus as shown in Figure 2 (b), we can observe that all the maxima. It is possible to appreciate that the resolution needed for observing the echoes located in the shoulders of the signal is really high. In Figure 2(c) we present the projection of a section along the x direction of scanning. The results fit to the simulations and a manuscript has been composed and is ready for submission.

We would like to also add the information from the strain observed in this study to a mayor publication together with extra data collected at NanoMAX in the forward direction, explaining better the technique and the simulations performed.

In the case of the Ni pillars, we observe the echoes signal as proposed, that is observed in Figure 3 (a), where it is possible to see that the broadening of the beam at focus is of the order of 5 μm, while in Figure 3(b) the beam size at focus is of the order of 76 nm as expected due to the FZP focus. In Figure 3(c) is presented the amplitude of the reconstruction that is a signal of around 5 μm, in the propagation to the focus (Figure 3.d) we can observed that the beam size in y keeps of nm size while the signal in the x due to the dynamical diffraction is broader to 5 μm. The study was performed about and below the absorption edge of Ni, presenting different depended for the echoes intensity and positions as proposed.

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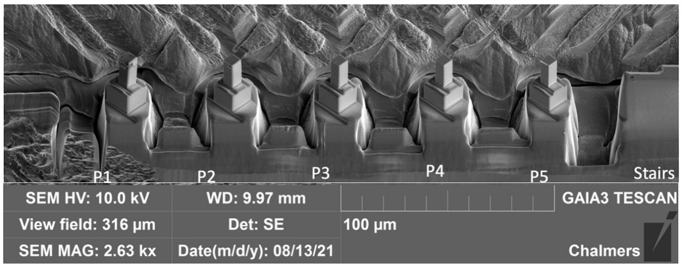

Figure 1. SEM image of the Ni pillars performed at Chalmers University of Technology using a Focus Ion Beam. Pillar P5 was not indented, while P1 to P4 had different indentation loads.

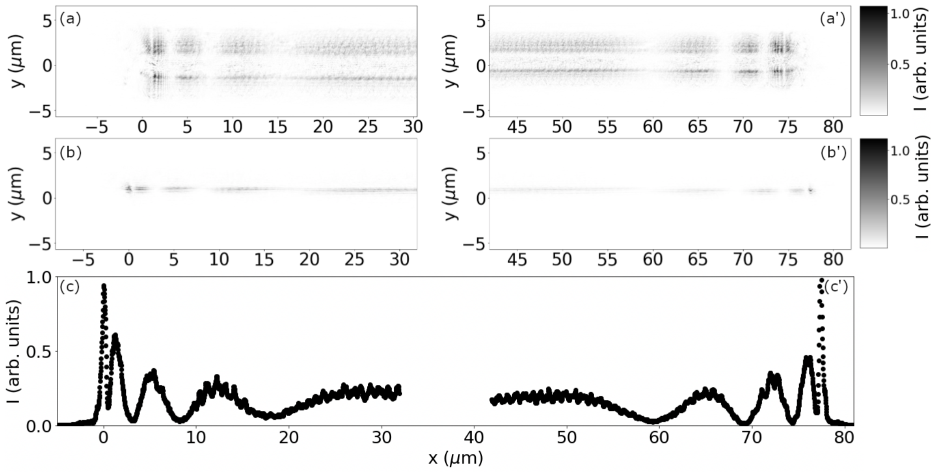

Figure 2. Reconstruction and propagation of the dynamical diffracted echoes for a Si wafer, the reflection under study was the Si (220) at 8.346 keV. (a) reconstruction, (b) propagation to the focus and (c) projection of a section of the propagation at the focus.

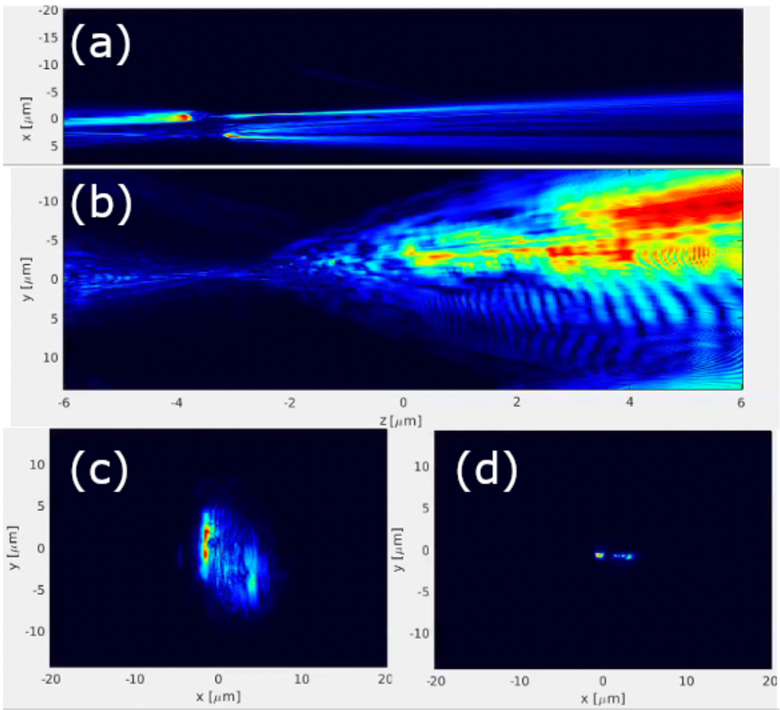

Fig 3. First reconstruction showing echoes in Ni pillars. (a) Propagation of the amplitude of the wavefront along the z-x plane. (b) Propagation of the amplitude of the wavefront along the z-y plane. (c) Amplitude reconstructed at the pinhole plane and (d) Amplitude propagated to the focal plane of the FZP, where the sample was located.