Experiment Report MA 3332

In situ imaging of platelets and micro-cracks formation and growth in bonded, hydrogen-implanted Si wafers using Full Field X-ray Diffraction Microscopy

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In this experiment, we used the newly developed Full Field Diffraction X-ray Microscope on beamline ID01 to study in situ buried micro-cracks in hydrogen-implanted silicon substrates relevant to the SmartCut process.

The sample we studied consisted in H-implanted Si wafers bonded on Si wafers. A pre-annealing of 2 hrs at 350 C led to the precipitation of the implanted H atoms in a shallow layer, near the bonding interface, resulting in a layer of micro-cracks that were preliminarly observed using infra-red laser confocal microscopy at the CEA-LETI.

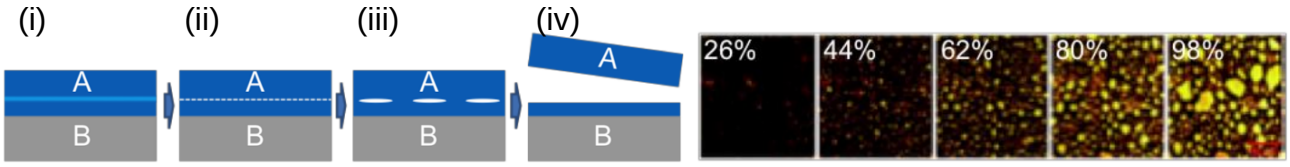

Fig. 1: *(left)* Schematic view of the SmartCut process: (i) upon annealing the implanted layer precipitates in (ii) platelets which then ripen into (iii) microcracks leading to (iv) the fracture. *(right)* IR transmission optical images of microcracks growth in implanted silicon as a function of the percentage of the fracture budget. (Image size: 1.2mm)

Initially we had planned to study the micro-cracks from the very early stages (step (i) to (ii) in Fig.1). We started by the observations in pre-annealed samples where micro-cracks had already been evidenced using infra-red microscopy. These samples consisted in patterned (32 µm square cavities, sample “AP1”) and plain Si wafer bonded to the H-implanted Si wafers (sample “AP2”) (Fig. 2).

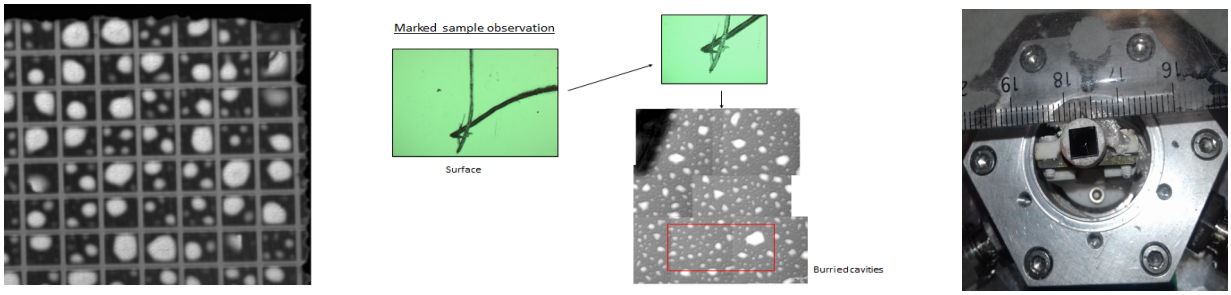

Fig. 2: *(left)* Infra-red laser confocal microscopy of sample “AP1” showing the micro-cracks buried 250 µm below the surface. The square cavities are about 32 µm long. *(middle)* Optical and IR microscopy in sample “AP2”, showing also the microcracks buried 250 µm below the surface. The red rectangle is about 100 µm x 200 µm and corresponds roughly to the field of view of the FFDXM instrument for the 008 reflection. *(right)* Sample “AP2” mounted on the button furnace for *in* situ observation of the evolution of the microcracks.

Using this technique quite analogous to dark-field transmission electron microscopy, we imaged the 008 and 228 Bragg reflections in real space using a set of polymer compound refractive lenses (CRL) between the sample and the detector. Given the focal lengths ratio, we expected a

magnification factor of about x60, *i.e.* the real space 6 µm pixels of the detector correspond to about 100 nm at the sample position. We chose these reflection to have the largest emergence angle (to decrease the projection of the field of view) while keeping the incidence angle high enough to minimize the path length in the Si wafer (and minimize the attenuation). The largest scattering angle in the horizontal geometry used was also limited by the polarization factor of the horizontally polarized beam. The incidence energy was set to 19.7 keV, which is the 3rd harmonic of the undulator at ID01. This energy was well-suited for the CRL and for the diffraction geometry. We finally used a double crystal monochromator instead of a multilayer monochromator to achieve the largest energy resolution at the cost of incoming flux. High energy/angular resolution is needed in this study to avoid the wash out out the ineresting signal by the tails of the very intense Bragg peak of the Si substrate.

Typical imaging scans were performed at constant temperature, using either rocking curves ("phi" angle of the sample) or radial scans across the Bragg reflection. We were able to observe the micro-cracks as well as the cavities in the sample "AP1" simply by imaging near the Bragg peaks of either the implanted or the patterned crystal, taking advantage of the fact that the crystals are slightly misaligned (~0.1 to 0.2 degree in tilt and twist), as shown in Fig. 2 and Fig. 3.

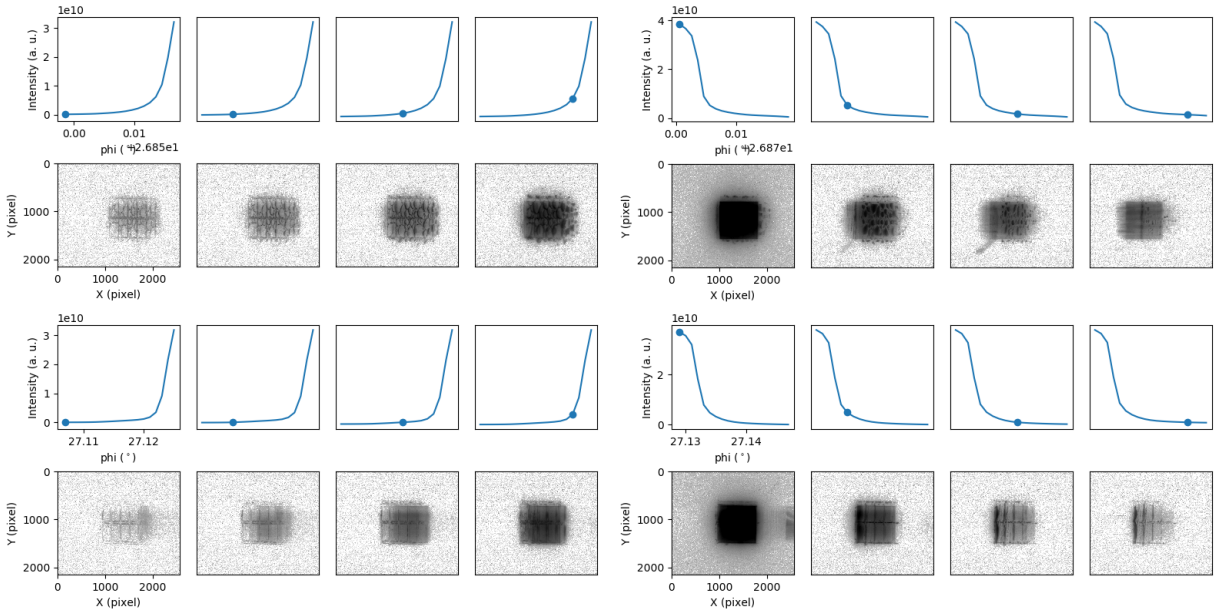

Fig. 2: *(top left)* Radial scan imaging on the left and *(top right)* right side of the 008 Bragg reflection of the implanted crystal and *(bottom left)* similar radial scan imaging on the left and *(top right)* right side of the 008 Bragg reflection of the patterned and bonded crystal in sample "AP1". The projection of the specular 008 reflection results in a distorted image along X, *e.g.* the square cavities appear rectangular.

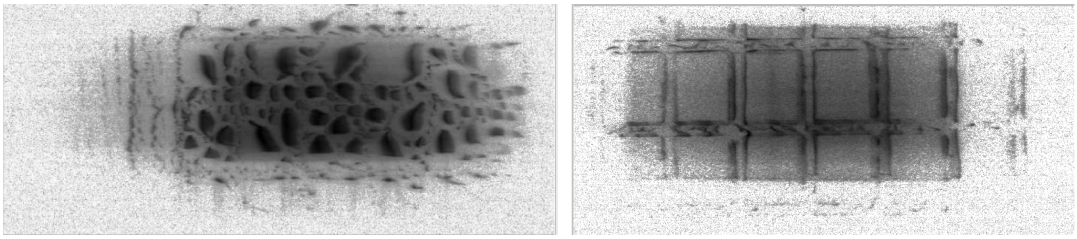

Fig. 3: *(left)* Projection-corrected images from near the 008 Bragg reflection from the implanted crystal and (righ) from the patterned, bonded crystal in sample "AP1".

Using the button furnace, we could also perform the imaging while annealing the sample "AP2" *in situ*, as shown in Fig. 4. In particular, we could observe single coalescence events in the real-space images.

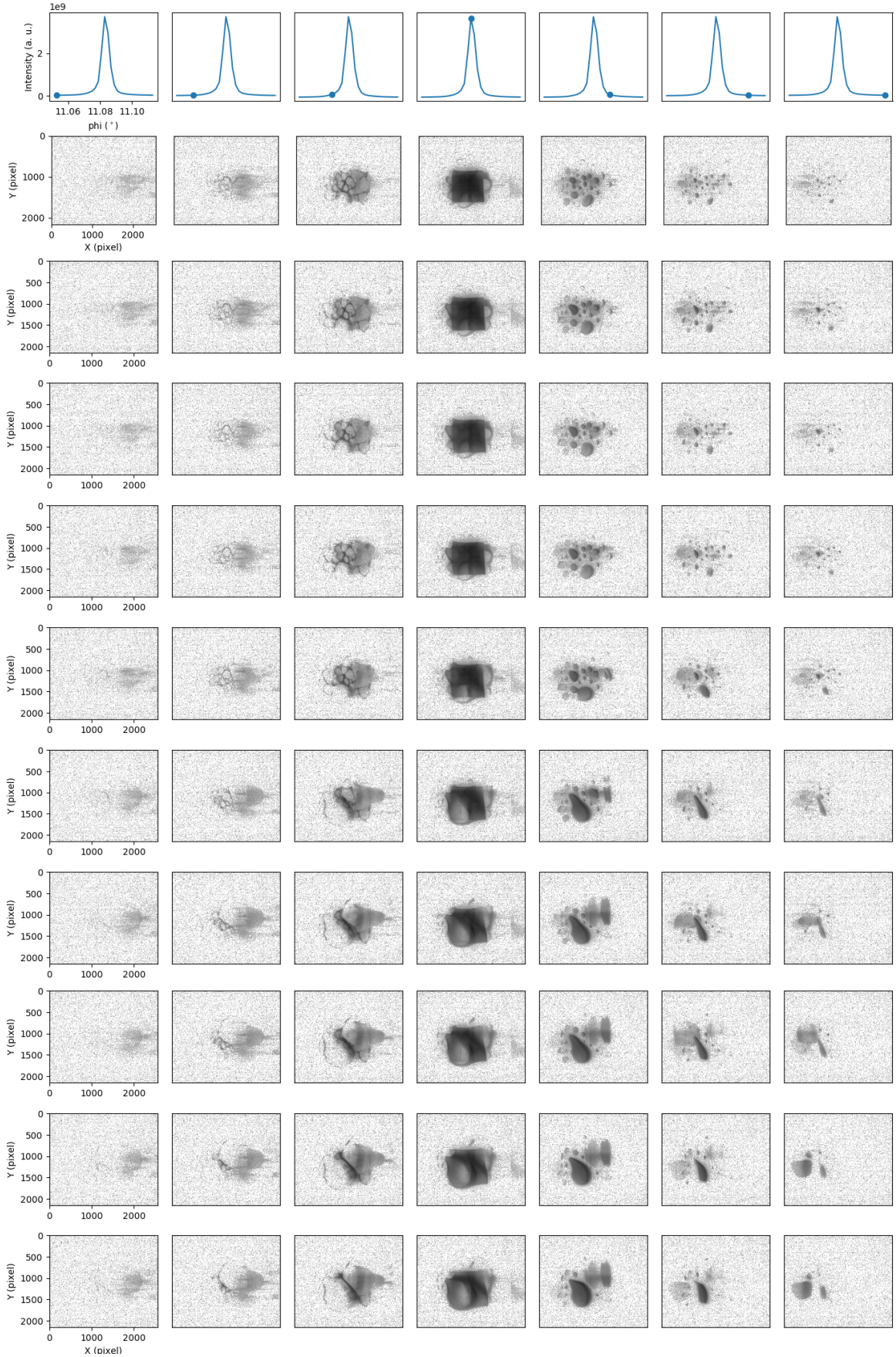

Fig. 4: *(top line)* Position on the radial scan across the 228 Bragg reflection of the implanted crystal in sample “AP2”. *(following lines)* FFDXM images from successive radial scans taken at 360C. Time goes from top to bottom and there is about 18 mins between two successive scans.

Early conclusions and outlooks:

Experiment MA3332 was very successful: we could image for the very first time the buried displacement fields due to micro-cracks in implanted and bonded Si wafers. We were further able to perform the measurements during *in situ* annealing and observe micro-cracks coalescence events.

Data analysis is still ongoing to extract quantitative information on the displacement fields of the micro-cracks, including the tilt and strain components.