	Experiment title: Real- time X-ray imaging of ultrafast fracture phenomena in silicon	**Experiment number**: MA 3231
Beamline: ID 19	**Date of experiment**: from: 09 Sept. 2016 to: 12. Sept. 2016	**Date of report**: Feb. 2017
Shifts: 9	**Local contact(s)**: Dr. Alexander Rack	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Dr. RACK Alexander Oliver, ESRF;
Dr ATRASH Fouad, Laboratory Jordan Valley Semiconductors Ltd R&D,
Dr. SHERMAN Dov, Laboratory Technion - Israel Institute of Technology Department of Materials Engineering,
Dr. SCHEEL Mario, Laboratory Synchrotron Soleil L Orme des Merisiers

Report:

1. Experimental Setup

The experiments were performed during 4-bunch mode at 25 - 31 mA. The silicon samples were prepared with a 1mm notch at the edge. Fig. 1 shows the Si-sample glued inside the aluminium frame. By heating from the bottom from 21 °C (RT) to about 100 °C, thermal expansion causes strain to initiate a crack. Slits were opened by 3.5 x 2.0 mm^2 to image about 0.5 mm from the tip of the precrack. The 2D detector systems were placed with the pco.Dimax in the direct beam for the fast radiography and the image intensifier with a P46 (YAG:Ce) phosphor screen and the 2nd pco.Dimax at 24° to collect the 220 Si reflection to image the related strain. The camera systems operated with exposure times of 1.28 ms at a max. acquisition rate of 35 504 images per second.

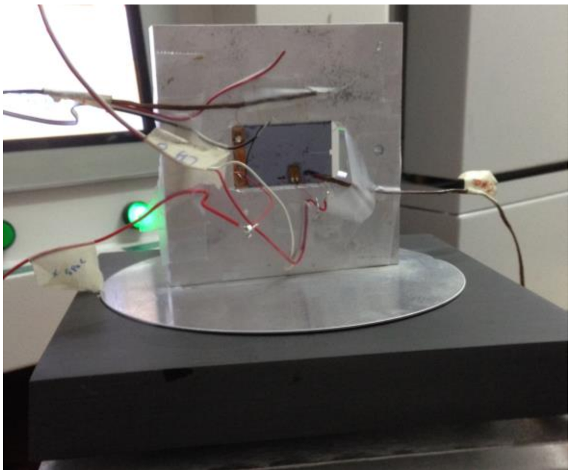

Fig. 1: Si-sample in Al-frame is heated from the bottom. Thermocouples for temperature control and contacts for camera trigger are visible.

2. Results

Fig. 2 shows by example the sample no.2 before (fig. 2a -b) and after (fig. 2c -d) the crack appeared in diffraction and direct images respectively. The crack becomes clearly visible in the radiography as well as in the diffraction image. The long-range strain field at the notch tip vanished to a great extend. Concerning the resolution, the set-up was decent. Problems arise with the trigger signal and the frame rate.

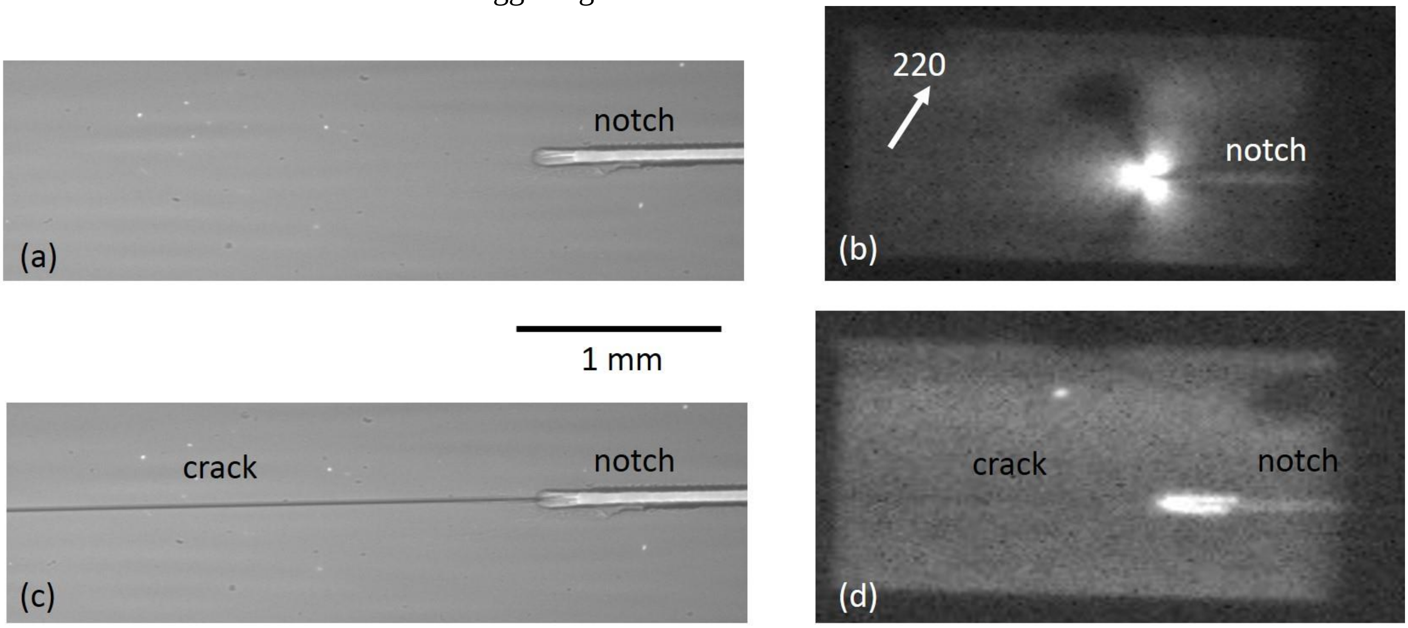

Fig. 2: Si sample before crack initiation (a) direct image (radiography), (b) diffraction image with huge strain fields as white and black contrasts at the tip of the notch, (c) direct image with crack, (d) diffraction image, with strain relaxation due to crack formation

The electrical signal worked not reliable enough to stop the camera and most of the data were overwritten. Finally the crack was faster than expected [Rack 2016] and appeared between 2 frames. But the strain field can be simulated by finite element methods which is still ongoing. From this data the energy will be calculated, which is needed to start the crack tip propagation.

In another successful experiment with wider field of view, the jumps of the crack tip could be clearly detected. The crack tip is pinned for 2-10 frames at the same position before doing a jump with higher speed, as already described in [Rack 2016]. After a jump, the strain at the crack tip increases up to critical value, which initiates the next jump. To make this strain visible, a difference picture from two diffraction images was calculated that way, that the first image after previous jump was subtracted from last image before next jump. The accumulated strain is indicated in Fig. 3 with an arrow. Again by ongoing numerical simulations the strain can be simulated to find the critical values.

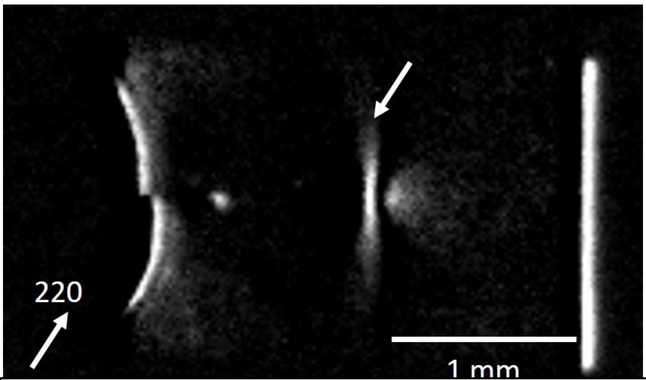

Fig. 3: Difference picture from 2 diffraction images between 2 jumps of the crack tip shows the accumulated strain to initiate the following jump is marked by an arrow. Crack propagation from left to the right.

3. Consequences

The trigger signal for the start and stop of the cameras has to be optimised as well as the frame rate. At least one camera operating at about 120,000 Hz would be needed. High power laser shots seem to be the adequate method as proofed already in cooperation of Dr. J. Grenzer/HZDR [Olbinado 2018].

References

Rack, A., Scheel, M., Danilewsky, A.N. (2016) “In situ fracture dynamics in silicon wafers under thermal stress revealed in real-time by direct and diffraction X-ray imaging” (2016), IUCrJ,3(2), 108-114.

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