EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

ESRF

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Reports supporting requests for additional beam time

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** X-Ray beam characterisation of high-precision silicon dosimeters with integrated tissue-equivalent materials fabricated with 3D technology by means of 2D focused beam scans	**Experiment number**: MI-1286
Beamline: ID21	**Date of experiment**: from: 10/05/2017 to: 16/05/2017	**Date of report**: 12.07.2017
Shifts: 18	**Local contact(s)**: Murielle Salomé	*Received at ESRF:*

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Report:

Aim of the experiment:

The aim of this experiment was to characterise our latest implementation of silicon micro-dosimeters fabricated using 3D sensor technology. The devices under test now include an integrated tissue-equivalent polymer. The tests are carried out at device level, in photoelectric mode, using a focused X-ray beam. This experiment has the main objective of finalising our understanding of the fabrication technology that was developed in the past 4 years within the framework of the Si-3DMiMic project, funded by the Norwegian Council for Research. The tests focused mostly on charge collection dynamics and timing response studies.

Sample description, experimental techniques and results:

The Devices Under Test (DUT) are solid-state micro-dosimeters fabricated on high-resistivity, p-type silicon (~10kΩcm). The starting material was a 10µm thick Silicon On Insulator (SOI) wafer. Each device has an active area of 2.4x2.4 mm^2 (physical size 4.8x4.8 mm^2). The active area is composed of 1056 independent sensitive volumes (configured in a 33x32 matrix). The sensitive cells are comparable in size to human cells and two radiuses were implemented (13.5 and 19 µm). The sensor layout is arranged in odd and even columns, readout at opposite sides of the device. Two cells are shown in Fig.1(a), they features a planar (n+) readout electrode in the middle (core) and a cylindrical 3D trench (p+) as biasing electrode. This configuration ensures that the Sensitive Volume (SV) within each cell, is physically separed from the rest of the substrate, thus achieving a perfect definintion of the charge collection region. The pitch between neighboring cells is equal to 75 µm. The excess silicon outside the cylindrical cells is removed and replaced with a tissue-equivalent polymer (polyimide, Fig.1(b)) necessary to mimic the interaction of radiation with the human body. Each die is glued and wire bonded to custom designed PCBs that fit the ID21 sample support (Fig.1(c)), ensuring ease of use in the Scanning X-ray Microscope (SXM). The signals are readout using micro-coaxial cables from the back-side of the PCB. The connection to the instruments outside of the vacuum chamber is performed through a flange featuring hermetic SMA connectors (the inside of the vacuum chamber is shown in Fig.1(d)).

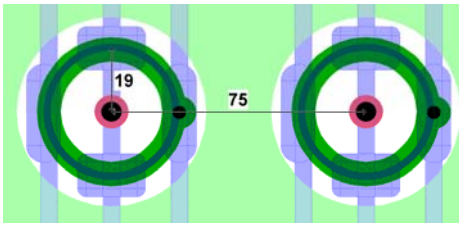

Fig. 1(a). Sensor layout. Two cells.

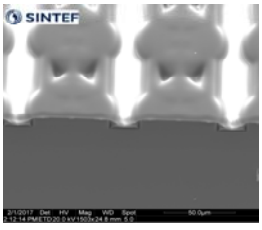

Fig. 1(b). Polymer

Fig. 1(c). Sample

Fig. 1(d) SXM

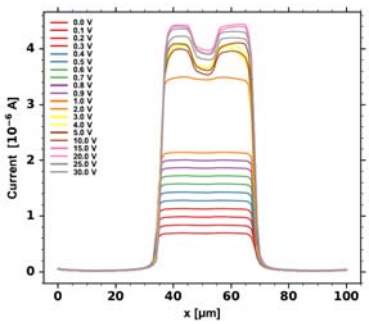

Fig. 2(a). Core signal 1D.

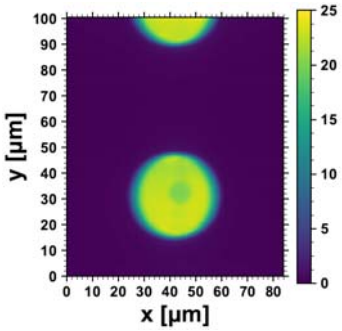

Fig. 2(b). Core signal 2D

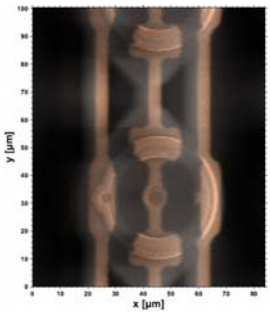

Fig. 2(c). Al and Si fluorescence

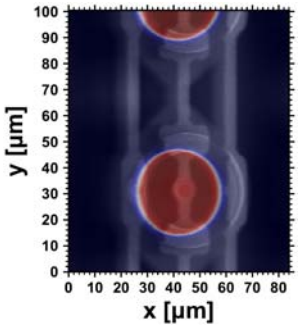

Fig. 2(d). Fluorescence signal

Two measurement modes where used: (i) with the use of Keithley 458 electrometers, the sensor response was recorded as a function of beam position and, (ii) with the use of 2.3GHz wide band preamplifiers and a Digital Sampling Oscilloscope (DSO) the time response of the sensors was acquired as function of time and beam position. Both 1D and 2D spatial scans were performed. An X-ray energy of 7.2 keV was used. The focused X-ray beam had a size of about 1.1x0.4 μm^2 and hit the sensors with a 30° tilt angle. Additional features of the SXM, such as X-ray fluorescence, allowed precise visualisation of different materials and elements in the sensor (e.g. the metal lines). The up- and down-stream beam intensity monitors were used to normalize the acquired data.

Results obtained using electrometer readout are reported in Fig.2. A 1D scan is shown in Fig.2(a) for a wide range of bias voltages. The signal increases as more voltage is applied to the sensor, saturation is achieved at as low as 3V of bias. The FWHM of the signal was found to be between 35 and 40 µm, in accordance with the designed diameter of the cell. A decrease in signal amplitude in the middle of the cell can be noticed at higher voltages. This effect is caused by the presence of the deep n+ core implant and is accentuated at higher voltages. A 2D scan at a bias of 30V is shown in Fig.2.(b) (plotted in arbitrary units). The uniformity and FWHM of the response are confirmed and no signal is observed in any of the regions outside the p+ deep trench, demonstrating its effectiveness in creating perfectly defined sensitive volumes. No bi-products of the interaction between X-rays and the tissue-equivalent polymer are detected. Fig.2(c) shows the overlay of the fluorescence signals for silicon (gray) and aluminium (orange). This measurement is important because it allows to appreciate the severe topography of the top sensor layer. This is an excellent, non-invasive diagnostic tool, and it highlighted some issues with the metal layer in these devices (broken metal lines were detected using this method, task that was difficult even using SEM imaging). The flurescence images and the data from a 2D scan are overlayed in Fig.2(d). This image shows how the 30 degree tilt of the beam results in a slight parallax effect, but it also shows that the signal is readout from the expected regions.

Both 1D and 2D scans were repeated using a transimpedence amplifier connected to a DSO. The data acquisition was triggered using a signal sent by the synchrotron at each bunch. A current pulse function of time was acquired for each scan position. The data were then processed using a python script. Each pulse was integrated using the trapzoidal method within a rolling window moved in steps of 0.5 ns. The result of the integration is representative of the charge collected in each scan point. By plotting the carge collected in each interval covered by the rolling window, it was possible to show the collected charge as function of time (both in 1D and 2D). Fig.3(a) shows the integration results over a 2D scan for a time of 2.5ns after the trigger, were the signal is at its maximum (Vbias=30V). After just 3ns (Fig.3(b)) most of the charge is already collected (signal decrease of more than 60%). This is also visible by observing the pulses extracted from the center of the cell (Fig.3(c)). For higher bias voltages, the signal exhibits a very rapid rise time and most of the charge is collected in the 5ns following the peak. Fig.3(c) allows to appreciate the effect of the bias voltage in increasing the response time of the sensor. These results demonstrate that the latest version of these solid-state microdosimeters is exceeding the performance of our benchmark technology (tested in the past years at ID21) in terms of sensitive volume definition and speed. These sensors are the most advanced solid-state micro-dosimeters and will now be tested in medical radiation facilities with protons and carbon ions.

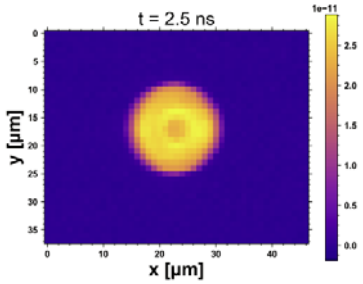

Fig. 3(a). Peak signal (2.5ns)

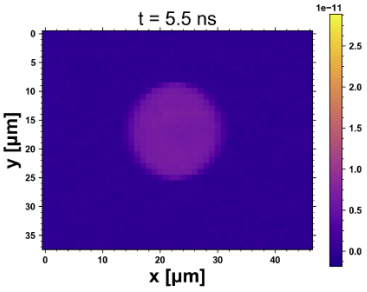

Fig. 3(b). Signal after 3ns

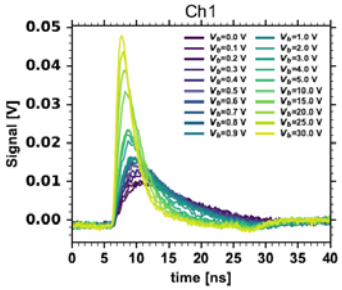

Fig. 3(c). Signal pulses