Annual Report

Funding Programme:	**Helmholtz Joint Research Groups**
Project ID No.:	HRSF 0004
Project Title:	Compton X-ray microscopy of biological specimens
Principal Investigator:	Heinz Graafsma
Report Period (=Calendar Year):	01/2018-12/2018

1) Group Structure

Please report briefly on the structure and personnel development of your group.

The group consists of 3 contributing subgroups, 2 on the German side, headed by Henry Chapman and Heinz Graafsma, respectively and one on the Russian side headed by Oleg Tolbanov.

The subgroups consist on many people involved in the project to a small degree (as outlined in the proposal). The proposal called for the addition of a PostDoc and a PhD student on the German side, both of which we were unable to recruit. Recruitment efforts are still ongoing.

2) Network/ Meetings

Please describe how the group works together. Have there been any international meetings organized by or attended by the group? What is the contribution of the group to the networking of international partners and the Helmholtz Centre?

The partners have met a total of 6 times, once before the official project start. The Russian partners joined by means of a video conference.

The meetings were focused on the preparation of a joined beamtime at PETRA III, as well as data evaluation and preparation of a follow up beam time in 2019 implementing lessons learned from the first experiments.

The Compton X-ray microscopy approach and project was presented by Pablo Villanueva-Perez at the XRM2018 in Saskatoon, Canada in August 2018

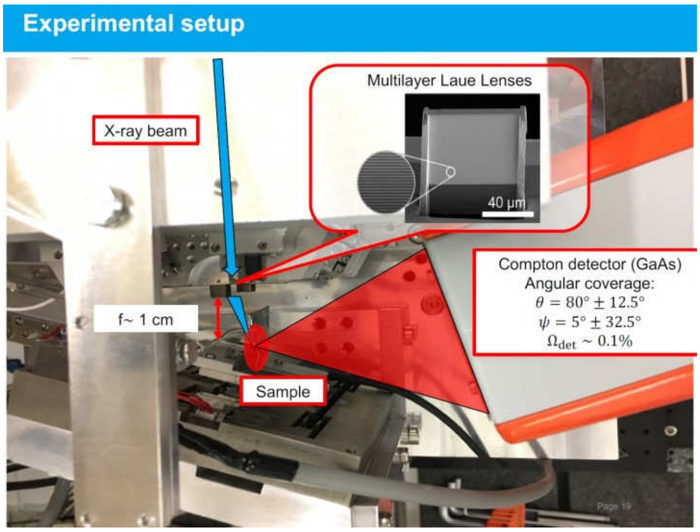
3) Scientific Progress / Milestones

How has your work plan progressed? What important milestones could be achieved during the report period? Is the progress of your work in accordance with original planning or has the work plan been changed?

Most of the first year was spend in PHASE 1: Exploration. A theoretical study outlining the project idea was published and a pilot experiment testing the MLLs and detectors was performed at the PETRA P10 beamline.

The paper by Villanueva-Perez, Pablo, Sasa Bajt, and H. N. Chapman. "Dose efficient Compton X-ray microscopy." *Optica* 5.4 (2018): 450-457 outlines the core idea of our project and was published in April 2018. It is concluded that a beam energy of 64 keV is optimal for Compton microscopy.

The accompanying experiment at Petra P11 at an energy of 28keV and a GaAs detector consisting of two 2x3 sensor tiles were used to detect the Compton scattered photons. An annotated photograph of the experimental setup is shown to the right.



The beamtime was a success in that the images could be successfully measured and showed an improved contrast of the absorption measurement. The experiment further showed possible avenues for future improvement. First of all: the beam energy was not optimal, a follow up experiment at P07 is scheduled for May 2018. In this next phase we will use an energy of approximately 65 keV.

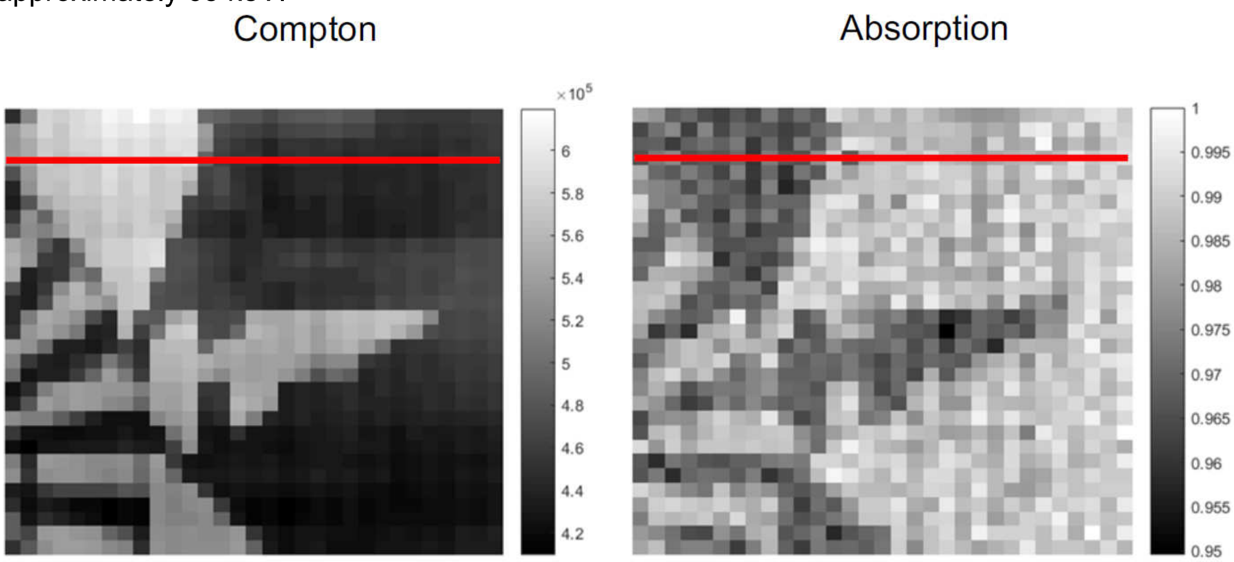
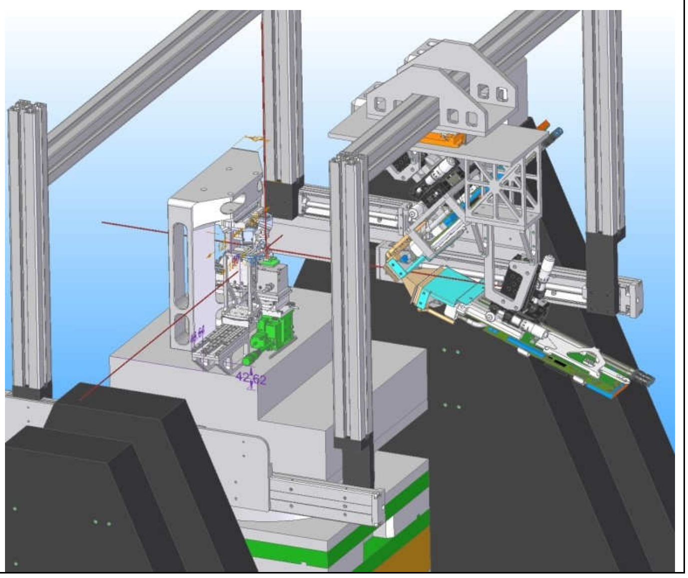


Figure: Comparison of the image obtained with the new method (left) to the traditional method (right). Please note that due to the way the data is obtained, the images are inverted, i.e., a light color in one corresponds to a dark color in the other.

For the next experiment improvements in the experimental setup (shown below), detectors and the MLLs were identified. The next generation experiment will feature more GaAs detector tiles to collect more of the scattered photons.



The currently used thickness of 500 µm was found to be sufficient of the experiment, even if the beam energy is increased further. Most advantageous are the increase in area coverage (more tiles) and the increase in sensor response uniformity.

Efforts to develop tiles larger than 2x3 are still ongoing. Initial tests with 2x6 modules failed due to the CTE mismatch of GaAs and Silicon that causes stresses in the material during the current flip chip bonding process, as a high temperature step in involved in the solder reflow. A pilot run for bonding with a room temperature process was initiated with an external partner and we are hopeful to see results of this in the coming year.

The technology of “X-ray transparent” backside contact has been created for pixel HR GaAs:Cr sensors in order to reduce X-ray absorption in the contact material. It was shown by calculation that the optimal X-ray transparent contact, ensuring good adhesion to the HR GaAs:Cr surface, is a multilayer contact based on VNi/Al films with a total thickness of 1 μm. The contact provides transmission of at least 98% in the range of X-ray energy from 10 keV and above.

The technology of pixel contacts manufacturing of HR GaAs: Cr pixel sensors has been optimized. The advantage of the optimized technology includes reduction of the UBM size down to 25 microns. The reduction of UBM size allows to increase the height of bumps, that in turn leads to the increase of flip-chip bonded assemblies of matrix sensors with primary electronics chips (ASIC).

It is shown that the resistivity distribution and values across the wafers are determined by the profile and doping level of the donor impurity, as well as the thermal stability of the initial n-GaAs wafers. It has been established that the control of the initial n-GaAs wafers characteristics and the selection of the optimal chromium compensation mode, allows to obtain HR GaAs:Cr wafers with a diameter of 4 inches characterized with a non-uniform resistivity distribution not exceeding 30%;

It is shown that the typical pattern of the count rate distribution over the sensor area is determined by the presence of a dislocation grid, formed due to the growth technology of the initial n-GaAs crystals. The origin of dislocations is connected with the presence of mechanical stresses due to the temperature gradient during the growth of a GaAs crystal by the Czochralski method. It is established that the dislocation grid is detected in transmitted near-infrared radiation (900–930 nm);

In summary the project is proceeding broadly according to the anticipated schedule, some deviations in details of the work packages have occurred, e.g. problems with the bonding of larger sensors, but these are expected be remedied within the proposed work plan.

4) Financial Plan / Time Schedule

Can you comply with the financial plan and time schedule or do you see a need for adjustment?

Due to the failed recruitment of the two positions on this grant we are significantly underspending on personnel compared to the financial plan.

In addition, a large fraction on the anticipated material cost has been committed by ordering the requested materials and/or services, but not been spend, as the contractors have yet to invoice their work.

We do not see a need for adjustment at the given time, as underspending and delays will be remedied once the open positions are hired and the people will contribute to the project full time instead of their originally anticipated fractional contribution.

5) Publications of the Group

Villanueva-Perez, Pablo, Sasa Bajt, and H. N. Chapman. "Dose efficient Compton X-ray microscopy." *Optica* 5.4 (2018): 450-457

Villanueva-Perez, P., Bajt, S., & Chapman, H. (2018). Scanning Compton X-ray Microscopy. *Microscopy and Microanalysis, 24*(S2), 180-181. doi:10.1017/S1431927618013259

I. Chsherbakov et al. “The influence of contact material and its fabrication on X-ray HR-GaAs:Cr sensor noise characteristics” was published in January 2019, DOI: 10.1088/1748-0221/14/01/C01026

6) External Funding

No additional external or third party funding has been applied for.

7) Patent Applications
No. of pending/granted patents

No patents have been applied for.

8) Awards received by Group Members

None so far