	Experiment title: Aerotaxy-an exploratory study.	Experiment number: MA2061
Beamline: BM26	Date of experiment: from: 31/01/2014 to: 03/02/2014	Date of report: 01/03/2014
Shifts: 9	Local contact(s): Giuseppe Portale	<i>Received at ESRF:</i>
Names and affiliations of applicants (* indicates experimentalists): Main Proposer: Edvin Lundgren ¹ Co-proposers: Anders Mikkelsen ^{1*} , Knut Deppert ² Additional Participants: Sarah McKibbin ^{1*} , Olivier Balmes ^{3*} , Simon Tågerud ^{2*} 1 - Division of Synchrotron Radiation Research, Department of Physics, Lund University, Box 118, 22100 Lund, Sweden. 2 - Solid State Physics, Department of Physics, Lund University, Box 118, 22100 Lund, Sweden. 3 - MAX-lab, Lund University, Box 118, 22100 Lund, Sweden.		

It has recently been demonstrated that III-V nanowires can be grown without a supporting substrate by flowing Au nanoparticles through furnaces containing MOCVD precursor molecules [1]. Catalyzed by gold seed particles, the molecules crack in the gas and crystallize to form GaAs crystals, resulting in nanowires. This growth method Aerotaxy may have important implications for high efficiency photovoltaics using nanostructured surfaces [2] and light emitting diode applications [3]. Both of these applications would benefit from a transition away from batch grown nanowires on expensive wafers to a flow process method to reduce the cost of producing large volumes of nanowires and furthermore, problems associated with lattice mismatching for LEDs could be eliminated [3, 4]. To optimise this aerotaxy process for such industrial applications it is important to gain fundamental understanding of the aerotaxy growth process, which can be done with an in-situ SAXS/WAXS/XRD experiment to observe the growth process. We have performed an exploratory experiment, to detect nanoparticles in a gas flow by diffraction and scattering based methods which will provide crystallographic and structural information in real time.

We successfully detected in-situ metallic nanoparticles as they flow in a nitrogen carrier gas which opens up future experiments to investigate Aerotaxy nanowire growth. We were able to detect unfiltered Au (the seeding agent for GaAs Aerotaxy nanowires) and Pt nanoparticles within a quartz flow chamber with polyimide windows for x-ray exposure and detection. Typical SAXS traces for the Au particles are shown in Fig. 1 a). From the SAXS measurements, the average size of unfiltered, sintered Au particles was 16 nm with 41% polydispersity (with a log normal distribution from 5–100 nm) and spherical in shape. In contrast, the unfiltered and unsintered Au particles, were determined to have an average size of 6 nm with mass fractal aggregation. These results confirm the expected output of the aerosol system and using different spark electrodes in the generator, similar results were obtained for measured in-situ, unfiltered Pt nanoparticles (Fig. 1 b)). Furthermore we were able to immediately observe loss of the signal when spark generation (and thus nanoparticle production) was turned off eliminating the possibility the increase in signal intensity originated from particle build-up on the x-ray windows.

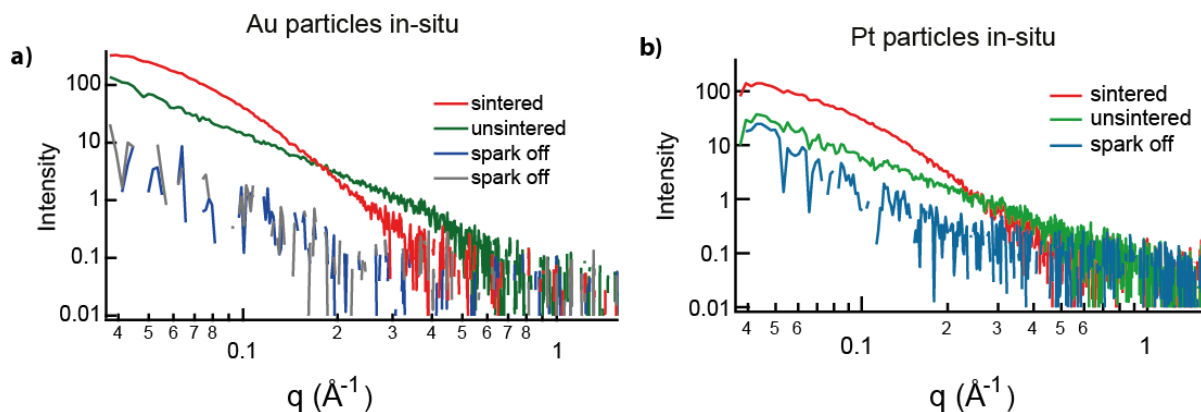


Fig. 1 Recent results from BM26 MA2061 Feb 2014. **a)** In-situ SAXS detection of unfiltered Au nanoparticles. **b)** In-situ SAXS detection of unfiltered Pt nanoparticles.

We also measured many pre-prepared samples containing nanoparticles or nanowires on polyimide film. We obtained clear SAXS/WAXS signals for 60 nm Au nanoparticles with densities between 25 - 100 particles/ μm^2 (a typical measurement comparing Au samples of different densities is shown in Fig. 2 a)), as well as sintered and unsintered Pt particles of all sizes. Fig. 2 b) shows the SAXS and an example WAXS measurement of Aerotaxy and wafer grown GaAs nanowires.

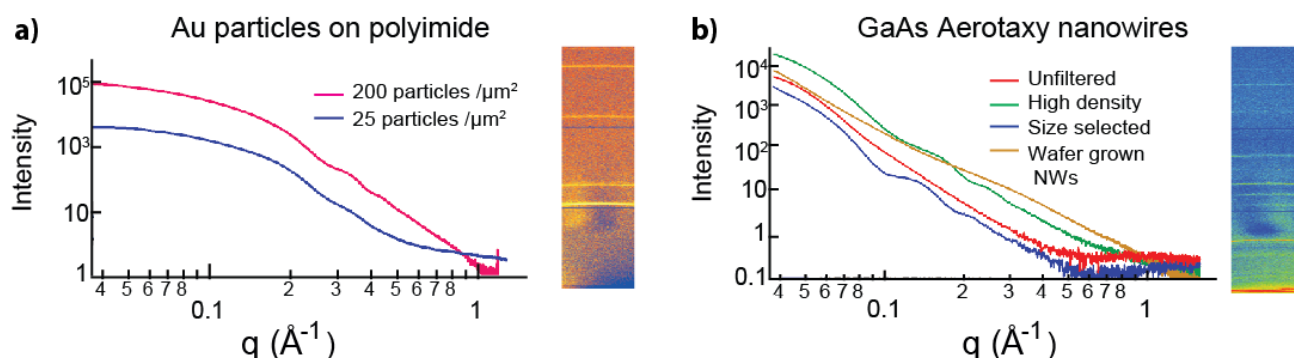


Fig. 2. Recent results from BM26 MA2061 Feb 2014. SAXS and WAXS of pre-prepared samples containing nanoparticles or nanowires deposited on polyimide film **a)** 60nm wide Au nanoparticles on polyimide substrate of different densities and **b)** GaAs nanowires grown by Aerotaxy or via the conventional wafer grown method.

We have successfully detected in-situ nanoparticles by maximising output of particles and measuring the entire size distribution produced by aerosol spark generation. In future experiments we will supply III-V materials for in-situ observations of Aerotaxy nanowire growth with a large spread of diameters. Using the current setup at BM26 we were unable to detect size selected nanoparticles, which would improve the quality of the data for publication and eventual control of Aerotaxy growth. Differential mobility analysers in the aerosol generator can select the nanoparticle size with an accuracy of ± 5 nm but has the drawback of dramatically reducing the particle density in the gas flow, which resulted in a signal equal to or under the noise level of BM26. The densities considered ($1-2 \times 10^6$ particles/ cm^3) may not be an issue at ID02, and so we plan to test size selection detection using that beamline. We also plan to now employ sheath flow aerosol focusing lenses to increase the density of particles to a small stream in the x-ray beam. This will improve both the SAXS and WAXS signals, the latter being particularly important to obtain crystallographic information on nanowire growth.

References

- [1] M. Heurlin, M. H. Magnusson, D. Lindgren, M. Ek, L. R. Wallenberg, K. Deppert and L. Samuelson, *Nature* 492 (2012) 90.
- [2] J. Wallentin, et al. *Science* 339, 1057 (2013);
- [2] T. Borgström, et al. *IEEE J. Sel. Top. Quantum Electron.* 17, (2011) 1050.
- [3] F. Qian, S. Gradečak, Y. Li, C.-Y. Wen, and C. M. Lieber, *Nano Lett.* 5 (2005) 2287