ESRF	**Experiment title:** Microfocus GISAXS - a new tool for the investigation of heterogeneous surfaces and interfaces	**Experiment number**: SC-1205
Beamline: ID13	**Date of experiment**: from: 21.02.2003 to: 25.02.2003	**Date of report**: 24/02/2004
Shifts: 12	**Local contact(s)**: S.V. Roth, M. Burghammer	*Received at ESRF:*

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Report:

Microbeam grazing incidence small-angle x-ray scattering (µGISAXS) is a novel technique developed at the beamline ID13 of ESRF. It combines the high surface sensitivity of GISAXS with the spatial resolution of the 5µm collimated microfocused beam of ID13. This unique combination offers the advantage of a two orders of magnitude reduced footprint in both dimensions, the footprint being 5x300µm^2 (y/x with x being the beam direction). The first pioneering experiments at ID13 [1,2,3,4] have clearly shown the feasibility of µGISAXS as an advanced scattering technique for the investigation of gradient noble metal nanocluster/ polymer multilayers and two-dimensionally nanostructured polymeric surfaces.

In the current experiment our aim was two-fold. Firstly, from the instrumental point of view we investigated the utilizability of Beryllium compound reflective lenses (BCRL) [5] as microfocusing optical elements in GISAXS geometry. Their high transmitted flux and low divergence at comparable beam sizes (on the order of 10µm) make them ideal optical elements for high-resolution µGISAXS and µSAXS studies [6]. Secondly from the sample point of view we investigated ex-situ prepared gradient noble metal nano-cluster/polymer multilayer structures using different preparation methods and metals. The noble metals chosen were gold (Au) and silver (Ag).

In fig. 1 we present two typical out-of-plane scans a double gradient multilayer sample [7]. It consists of a layer of orientationally disordered prism-shaped Ag nano-clusters on top of a polystyrene (PS) layer on a Si-substrate. The Ag nanocluster layer was prepared by deposition from a solution on part of PS covered Si-substrate and subsequent drying. Due to capillary forces the concentration profile in the drying solution becomes position-dependant. As a consequence a double gradient layer structure emerges. From out-of-plane scans the most prominent in-plane length scale ξ_{max} can be deduced, see fig. 2. As can clearly be seen the sample is dominated by two cross over lengths. The first one at zero stems from the transition region pure PS-layer to Ag-covered layer. The second cross-over emerges when the influence of the initial liquid boundaries of the nano-cluster solution vanishes after some millimeters [7].

This experiment successfully demonstrated the novel combination of BCRL and µGISAXS [6,7]. Due to the high transmitted flux the measuring times could effectively be lowered and due to the low divergence the minimal detectable q-value q_{min} is reduced. Calculating a maximal most prominent in-plane length scale via $\xi_{limit}=2\pi/q_{min}$, we estimate $\xi\approx 1\mu m$, i.e. now on the same order as the lateral beam size of 5µm.

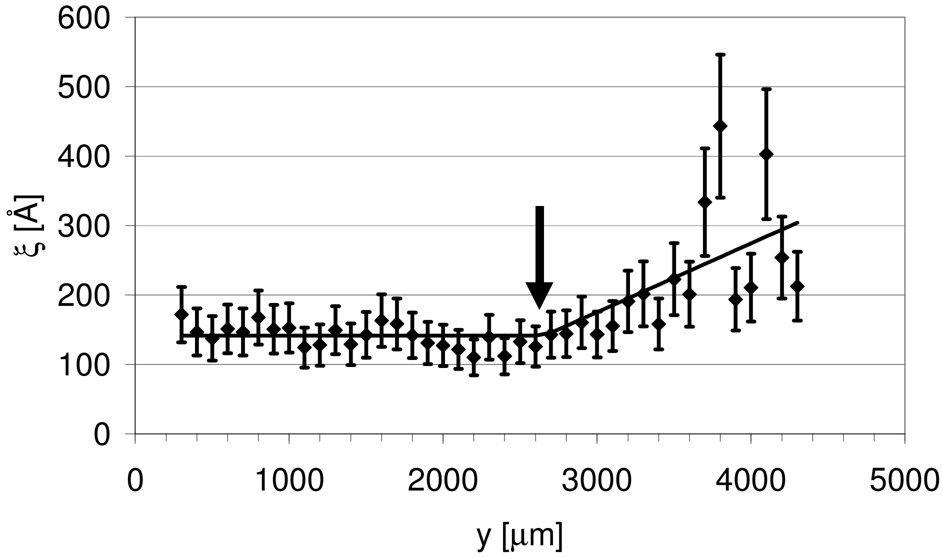

Fig. 1. Most-prominent in-plane length ξ_{max} as a function of the scan position. Note the double gradient structure: PS->Ag-layer with ξ_{max} =142Å and the second increase around y=2600µm indicating the end of the influence of the liquid-air-polystyrene boundary conditions.

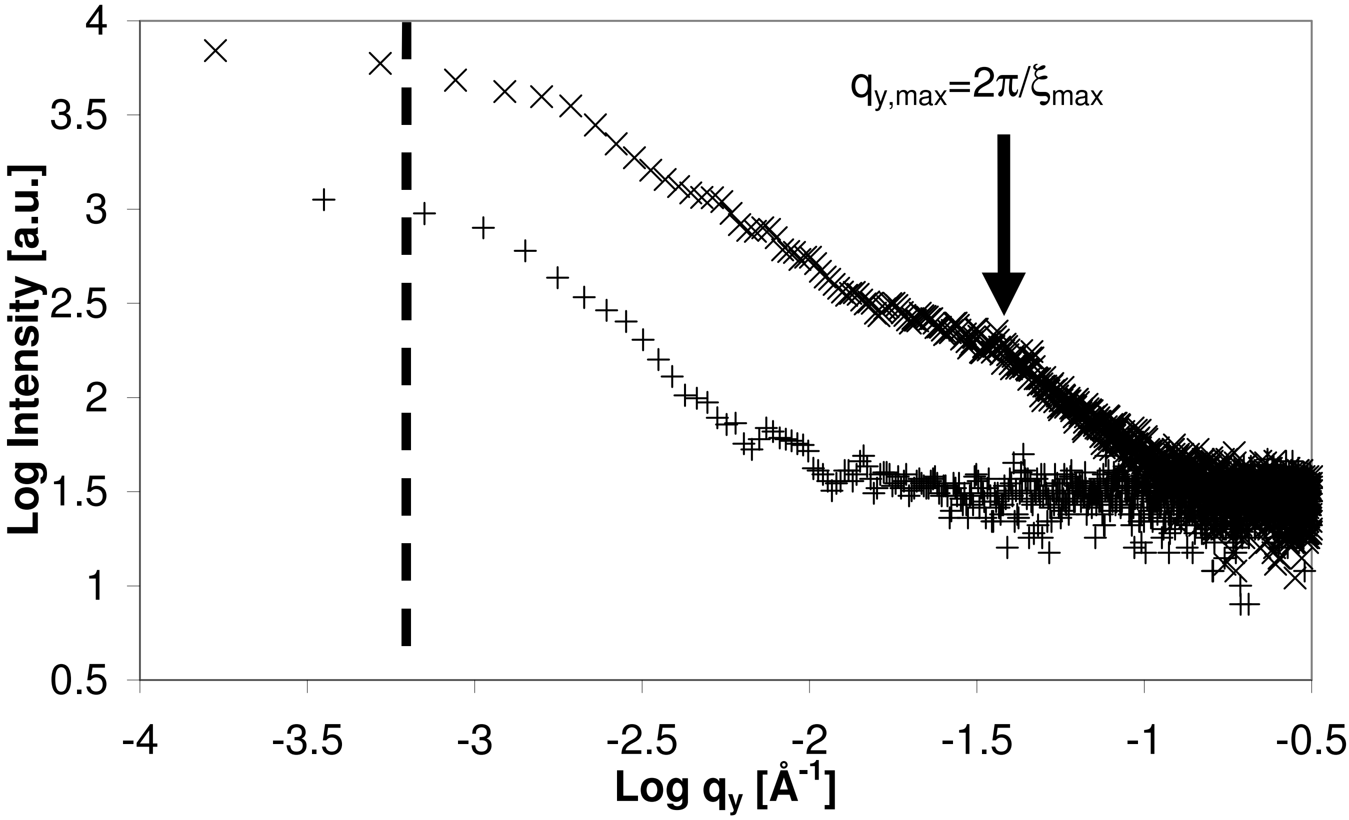

Fig. 2. Out-of-plane scans at y=1700µm (pure PS-film) and y=3100µm (Ag-layer) for q_z=0.102Å^{-1}. The side maxima at $q_{y,max}$ indicates the most-prominent in-plane length via $\xi_{max}=2\pi/ q_{y,max}$ as a result of the build-up of the Ag-cluster layers. The dashed line marks the resolution of the µGISAXS experiment.

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