ESRF	**Experiment title:** Test of an appropriate setup for Grazing Incidence X-ray Scattering (GISAXS) during the 3D heteroepitaxial growth performed in situ, in UHV, by molecular beam epitaxy, application to the Pd/MgO(001) system.	**Experiment number:** MI-257
Beamline: ID32	**Date of experiment:** from: 22-01-99 to: 02-02-99	**Date of report:** 23 – 08 - 99
Shifts: 24	**Local contact(s):** Roberto Felici	*Received at ESRF:* - 2 MAR. 2000

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Report:

A fully dedicated experimental set-up was been built on the ID32 beamline in order to perform GISAXS *in situ*, in UHV, during molecular beam deposition, without any window before the sample, thus avoiding any unwanted background scattering. Two pairs of motorised slits working in secondary vacuum were developed and installed respectively 5 meter and 1 meter upstream the sample. The first pair of slits was used to define the beam size (0.2 mm V x 0.05 mm H), and the second pair was used to remove scattering by the primary slits. A fast (1 ms of closure time) beam shutter was installed just before the secondary slits, followed by a hollow diode to monitor the primary beam intensity by measuring the beam vertically scattered by the primary slits. All this beam-defining assembly was pumped by several ions pump, enabling to reach a vacuum of ~5 x 10^{-8} Torr. A 1-meter long differential pumping pipe pumped by a large ion pump, and allowing a differential pressure of more than 4 orders of magnitude between both ends, was installed between this beam-defining line and the UHV chamber (base pressure 10^{-10} mbar) mounted on a six circle diffractometer of " 2+2 " type, which is usually used to perform grazing incidence x-ray scattering measurements in UHV. The usual beryllium window assembly of the UHV chamber was replaced by a more standard part hooked to the entrance line by a CF38 bellow, allowing for a small rotation of the chamber around the vertical axis, used to define the angle of incidence of the x-ray beam with respect to the surface of the sample, which was kept vertical. A 0.8 meter-long cone was connected to an exit pipe through a CF38 bellow, and ended by a 100 mm diameter beryllium window placed just in front of a 16-bit x-ray CCD detector. The cone and the detector were hooked to the goniometer detector arm, thus allowing precise vertical and horizontal alignment. A motorised tantalum beam-stop with a T-shape was introduced between the exit Be window and the 2D detector. Pd and Pt were evaporated from electron-beam-heated rod (99.99% purity) using commercial evaporators (Omicron Instruments EFM4, Germany), and Ag was deposited using an effusion cell (from Meca-2000). The deposition rates, ~1 Å/min, were calibrated with a quartz microbalance prior and after the x-ray measurements. Most of the measurements were performed on cumulative metal deposits, the growth being interrupted during CCD acquisition, but some were made in real-time, without interruption of the growth.

A special preparation of the MgO(001) substrates led to MgO(001) surfaces that are very flat and of high crystalline quality, free from any impurity, with in-plane domain size larger than 1 μm, average terrace size of 6000 Å, and a rms. roughness of 2.4 Å.

The x-ray beam energy was set at 11 keV, below the L and K adsorption edges of the three metallic species, in order to avoid fluorescence background. Most measurements were performed slightly above the critical angle for total external reflection of MgO. In addition, some situations were measured for different incident angles between 0 and 0.5° in 0.02° steps, in order to precisely characterise the influence of the incident angle. The incident beam was unfocused; its size was reduced to 35-μm (H) x 200-μm (V) by several pre-sample slits.

The experiment was very successful, the background was indeed very low, allowing to record easily the GISAXS from as low as 0.5 ML of deposit. The following depositions were characterised: Ag on MgO(001) at 300 K, up to 100 Å; at 600 K, up to 30 Å, at 423 K, up to 50 Å, and at 300 K under O_2 partial pressure, up to 50 Å; Pd on MgO(001) at 300 K up to 13 Å, at 500 K, up to 30 Å, and at 700 K, up to 10 Å; finally Pt on MgO at 800 K, up to 30 Å. All the last deposits were carbon coated, allowing HRTEM plane views to be made for comparison. The average island size, separation and height deduced from the GISAXS and HRTEM data were found perfectly consistent.

An example of the data is given in the figure below, in the case of the growth of Ag on MgO(001) at room temperature. The GISAXS pictures are shown for an equivalent amount of Ag deposited of 1 ML (upper left), 5 ML (upper right), 18 ML (lower left, note the second order of scattering perpendicular to the surface), and 50 ML (lower right; note that, even at 50 ML, large correlated islands are still present)

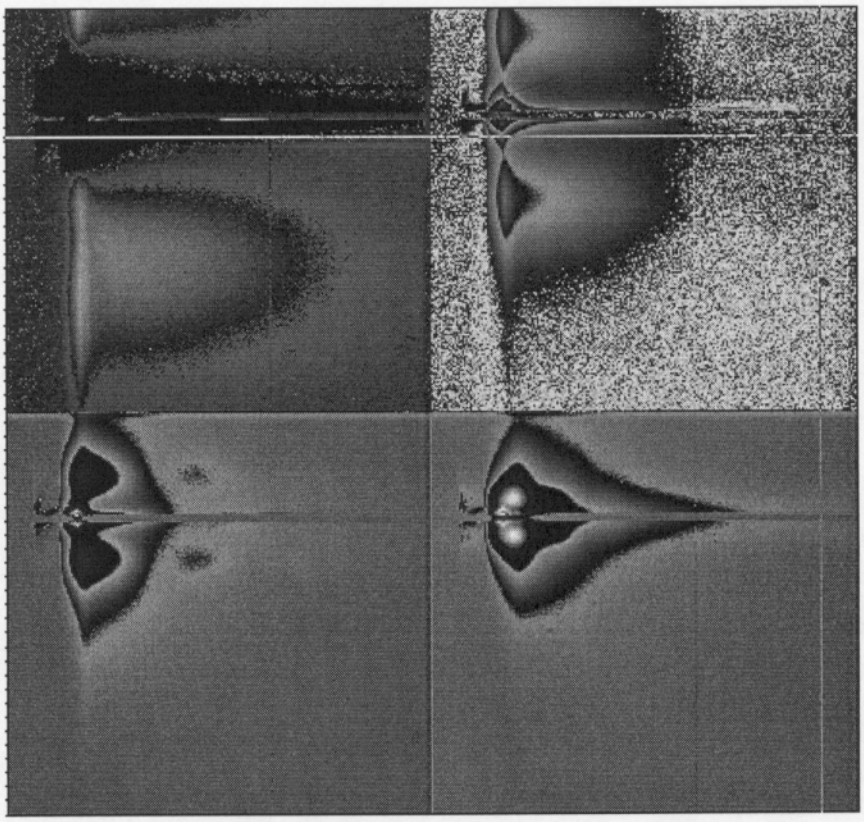

Figure: GISAXS data for 1, 5, 18 and 50 ML of Ag in situ deposited on MgO(001) at room temperature. The vertical is parallel to the sample surface; the horizontal is perpendicular. The evolution is typical of a process of nucleation, growth and coalescence of correlated islands.

Because the beam could not be focused, recording a deposit of ~1 ML could take as much as 10 min. We hope that the implementation of double focusing will decrease this acquisition time, thus helping recording dynamic growth processes.