

ESRF  
experimental  
report

**Experiment title:**  
Morphological study of Deposited clusters by Grazing  
Incidence Small Angle X-ray Scattering.

**Experiment  
number:**  
HC-198

**Beamline used:**  
ID02

**Date and time of experiment:**  
from: 06-Feb-96  
to: 08-Feb-96

**Local contact(s):**  
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September 1, 1996

**28 FEB. 1997**

*received  
(completed by  
ESRF):*

## Report:

### Principle and experimental set-up:

The Grazing Incidence Small Angle X-ray Scattering (GISAXS) is a new surface analysis technique which allows to characterize morphologically aggregates deposited on a substrate or embedded in a matrix near the surface. So, the experiments are carried out at / or near the critical angle of the substrate or the matrix.

We have decided to study the first growth stages of the gold thin films (according to the Volmer Weber mechanism), especially when the deposition is, assisted by ion implantation (Ion Beam Assisted Deposition or Dynamic Ion Mixing). The gold samples were obtained by sputtering and deposited on Si substrates coated with 25 nm of amorphous carbon sublayer. The deposition rate was  $0.02 \text{ nm.s}^{-1}$  and the equivalent average thickness varied from 0.8 nm to 2 nm. When the Dynamic Ion Mixing was used, Argon ions with an energy of 120 KeV impinged the sample. The assistance degree  $Q$  is defined as the number of ions arriving on the sample divided by the number of the gold atoms deposited on the surface. Three different values of  $Q$  were considered,  $Q_1 = 0.05 \%$ ,  $Q_2 = 0.5 \%$  and  $Q_3 = 2.5 \%$  and also the non-assisted case ( $Q = 0$ ), in order to check the influence of the ion implantation.

Concerning the experimental set-up, the wavelength used on ID02 beam line was chosen at  $\lambda = 0.144 \text{ nm}$ . Horizontal and vertical slits, located between the double crystal Si(111) monochromator and the mirror defined a beam cross-section of  $100 \times 100 \mu\text{m}^2$ . Translation and rotation of the sample-holder were possible inside the scattering chamber and allowed the adjustment, under vacuum of the surface sample at grazing incidence. To obtain the whole information in the scattering plane, a 2D detector was used.

### Results

Figure 1 shows a typical scattering pattern obtained from gold sample with 3.1 nm of average thickness without assistance. In this recording plane, the main characteristic is the presence of two scattering streaks symmetrical around the beam-stop which masks the reflected beam. This indicates an interference effect between gold aggregates parallel to the surface substrate. In order to describe all the  $q$  space, we decompose the reciprocal vector as following:  $\mathbf{q} = \mathbf{q}_y + \mathbf{q}_z$ .

with:  $\mathbf{q}_y$  axis is parallel to the sample surface and  $\mathbf{q}_z$  axis is perpendicular to the sample surface

Cross-sections along  $\mathbf{q}_y$  and  $\mathbf{q}_z$  are shown in figure 2 and 3 respectively. If we consider gold aggregates with a cylindrical shape, from the first cross-section we determine the correlation distance and the mean diameter of gold aggregates, From the  $\mathbf{q}_z$  direction, the average height can be determined with a good accuracy because the scattering patterns of all the samples studied at ESRF showed the presence of the first scattering order implying a very narrow height distribution.

**Experiment report** (If this work has been published, please give reference and abstract):

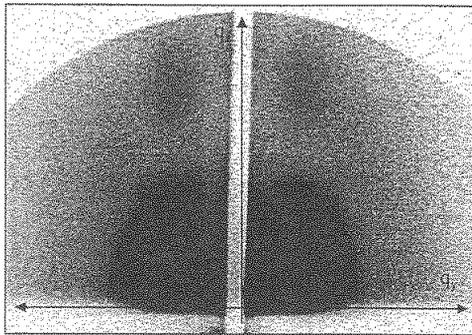
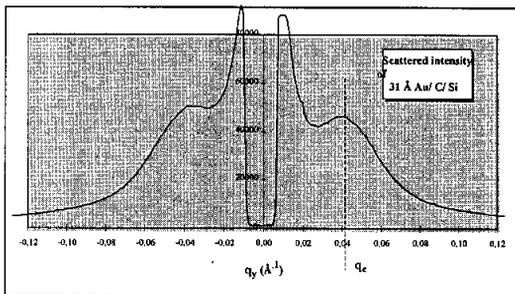


fig. 1 : Scattering pattern of 3.1 nm Au / C / Si



g.2: Cross-section along  $q_y$

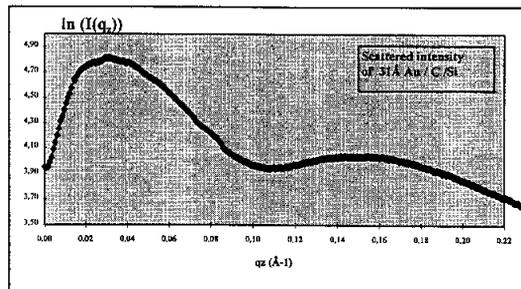


fig. 3: Cross-section along  $q_z$  and for  $I(q_z)$

From these parameters, we can determine the density of aggregates, the average surface and the mean volume of the aggregates in order to study the evolution of the kinetic growth. The evolution of the aggregate density is displayed on figure 4, for different sample assisted ( $Q = 2.5\%$ ) or not versus the total number of deposited gold atoms.

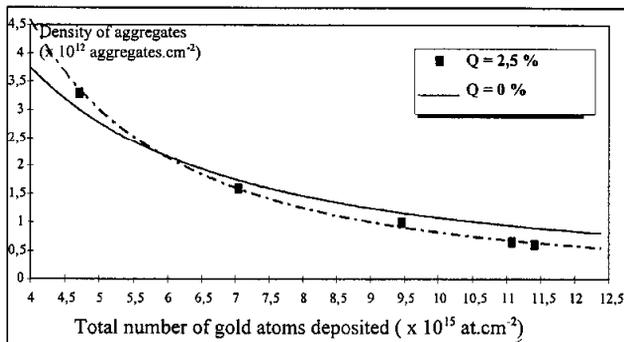


fig. 4: Evolution of the aggregates density in function of number of gold atoms deposited

The influence of ion implantation are the following :

When the assistance degree is equal to  $Q = 0.05\%$  and  $Q = 0.5\%$ , there is no significant effect on the growth of gold thin films in comparison to  $Q = 0\%$ . In fact, the number of high energy impinging ions is too low to **break** the growing islands or to increase the surface diffusion.

For  $Q = 2.5\%$ , we can define two ranges : when the total number of gold atoms is below  $6.10^{15} \text{ at.cm}^{-2}$ , the density is higher than for  $Q = 0\%$ . In this case, the nucleation sites produced during the ion bombardment prevent the surface diffusion of the gold atoms. The opposite scheme takes place when the number of gold atoms is above  $6.10^{15} \text{ at.cm}^{-2}$ . A detailed analysis is in progress.