


Experiment title:

Anomalous GISAXS study of magnetic alloy nanostructures grown on van der Waals surfaces

Experiment number:

02-01-639

Beamline:
Date of experiment:

from: 15/07/04 to: 19/07/04

Date of report:
Shifts:

Local contact(s): Jean Paul Simon

Received at ESRF:

Names and affiliations of applicants (* indicates experimentalists):

*M. Maret *, J.P. Simon*, V. Chamard*, B. Gilles**

LTPCM,ENSEEG Domaine universitaire, BP75, 38402 Saint Martin d'Hères, France.

B. Riedlinger, U. Mazur**

Department of Physics, University of Konstanz, PO Box M621, 78457 Konstanz, Germany

Report:

GISAXS measurements on the small angle x-ray scattering setup of the CRG-BM02 beamline were performed in submonolayer deposits of CrPt₃, FePt and CoPt grown on WSe₂(0001) surface and in deposits of fcc metals (Cu, Pt) and FePt grown on GaSe terminated Si(111). These samples were previously prepared in the molecular beam epitaxy chambers at the CEA-Grenoble and at the University of Konstanz. They were then transferred on BM02 using a vacuum valise and placed inside a chamber under 10⁻⁵ torr, in order to avoid radiation damage. The alloy samples were obtained under ultra high vacuum by the co-deposition of two atomic fluxes emitted from two separated e-gun sources. The three alloys grow as 3D islands on WSe₂(0001) as observed by RHEED. In contrast, the 3D growth mode of the FePt alloy on Si:GaSe is not observable, due either to a chemical reactivity between Fe and Si occurring likely at the deposition temperatures (300 and 500°C) or to a 2D-growth of bcc Fe at the early stages favored by a small misfit between bcc Fe(111) and the Ge topmost hexagonal dense plane and by a segregation of Pt atoms at the top surface. Therefore we have deposited fcc metals on Si(111):GaSe, which is more favorable to a 3D growth, without however the epitaxial relationships observed for the alloy deposits on WSe₂(0001). The growth temperatures varied between 20-500°C and the coverages between 0.5-1Å. Due to the thinness of the WSe₂ substrate (100µm) the samples were stucked on STM plates using Ag lake.

The GISAXS patterns were recorded with a CCD camera (1340x1300 pixels) located at 640mm from sample. The pixel size is 50x50µm². A photon energy of 7keV ($\lambda=0.177\text{nm}$) was chosen and the size of the beam after focussing was 0.1*0.5mm²(VxH). The scattered intensities were thus recorded for horizontal q_y and vertical q_z scattering vectors up to 1.8 and 3 nm⁻¹, respectively. A vertical beam stop was used to protect the CCD from the strong intensity scattered in the specular plane. Due to the small size and the waveness of the substrates, particular care was taken in order to align the sample in the beam: i) the use of a photomultiplier monitor between the sample and the camera allowed us to optimize the sample position by measuring the total external reflectivity plateau; ii) without beam stop the CCD was used to monitor the shape and the intensity of the specular spot. A refined position was finally obtained by emphasizing the GISAXS signal expected from the nanostructures and minimizing the diffuse one from the STM plate. GISAXS patterns were recorded at different incidence angles α_i between 0.4 and 0.8 deg (the critical angle of WSe₂ being of 0.455deg, $q_{z,c}=0.56\text{nm}^{-1}$). Figure 1 shows typical GISAXS patterns measured for $\alpha_i=0.4\text{deg}$.

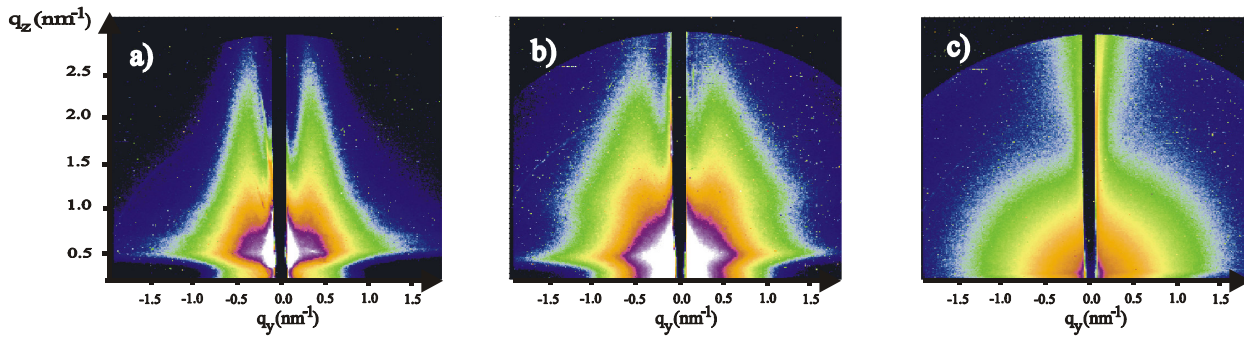


Figure 1: GISAXS patterns: a) 0.5 Å deposit of CrPt₃ grown at 500°C on WSe₂(0001); b) 0.5 Å of CoPt at 300°C on WSe₂; c) Pt grown at 300°C on Si(111):GaSe.

The enhancement of the intensity along $q_{z,c}=0.56\text{nm}^{-1}$ is due to dynamical effects at the critical angle. The side maxima on both parts of the q_z direction are interference peaks, which are the signature of a typical distance between the nanostructures grown on WSe₂. Indeed, when the size of WSe₂ substrate was too small (typically smaller than $3\times 3\text{ mm}^2$) the diffuse signal coming from the STM plate prevailed with a typical flame like shape. Then, for Cu and FePt deposits on Si(111):GaSe the triangular shape of the GISAXS patterns suggests that the surface roughness is dominant, while for the Pt deposit (fig.1c) the isotropic GISAXS signal can be attributed to Pt nanostructures.

For the 0.5 Å-thick alloy deposits on WSe₂(0001), preliminary radial analyses of the GISAXS intensity distribution in the vertical and horizontal directions lead to the following results.

When the co-deposition temperature increases from 300 to 500°C, i) for the CrPt₃ alloy, the lateral sizes of the nanostructures increases from 4 to 6 nm, with almost no change in the height, and consequently the interparticle distance increases, ii) while for the FePt and CoPt alloys no significant increase in lateral size is measured, a small increase in height from 1.3 to 1.45 would be reliable. For the latter ones, these parameters are in good agreement with the STM measurements. For the CrPt₃ samples, STM measurements have not yet been performed.

We think that these different behaviours are related to the structure of the ordered phases grown along the [111] direction, which is favored by the high deposition temperatures between 300 and 500°C: the fcc L1₂ structure for the CrPt₃ nanostructures and the tetragonal L1₀ structure with the three possible variants for the CoPt and FePt nanostructures. *In situ* GISAXS experiments during the growth of the two structurally different nanostructures would be well appropriated to confirm the dependence of the morphology with the co-deposition temperature. Finally, during the experiments on BM02 no anomalous study was attempted due to the weakness of the GISAXS signals.