## Morphological and dimensional studies of organized nanostructures by small angle x-ray scattering

## **Guillaume FREYCHET**

Supervisors: Mireille Maret and Patrice Gergaud

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Salle Palladium (1st floor) - Maison MINATEC

**Abstract:** The semiconductor industry now faces significant challenges in terms of characterization. Indeed, the reduction of feature size and separation distance pushes the in-line metrology techniques such as microscopy (SEM) and ellipsometry (OCD) to their resolution limits. So in order to cope with demand of the industry (a precise control of the uniformity and defects) some needs in terms of metrology are appearing.

In this context, the capabilities of the X-ray techniques were evaluated. To detect 1-100 nm sized objects X-ray scattering at small angle (SAXS), using incident angles ranging from 0.1 to 10° was used. This technique consists in sending X-rays on a sample and collecting thanks to a 2D detector the diffracted photons through the sample (study in transmission). This technique is sensitive to the electronic density contrast in the sample and is nondestructive. Moreover, the statistical information over large areas (several mm2) obtained from SAXS is complementary with the local and direct information obtained by SEM. A second technique called GISAXS (Grazing-incidence SAXS) was also tested. The X-ray beam hints the sample under grazing incidence and the diffused beam is recorded in reflection. GISAXS is more suited for the study of nano-objects supported or embedded not far from the surface. The aim of my work was to implement the SAXS and GISAXS techniques on the nano-characterization platform at the CEA-LETI and to demonstrate the ability of these techniques to control the size and morphology of nanostructures present in samples coming from the micro-electronics industry. This work was focused on two different materials: line gratings and films of self-assembled block copolymer.

For the study of line gratings, we showed the capabilities of CD-SAXS to extract the period, the line width and the line profile (height and sidewall angle) with a sub-nanometer resolution. Moreover, the line roughness was also studied, showing promising results in the case of periodic roughness with sub-nanometer amplitude. The results obtained by CD-SAXS coupled with microscopy techniques lead to a precise description of lines both on a local scale and on large areas (statistical information given by CD-SAXS).

The GISAXS studies were focused on block copolymer films, studied for patterning applications with sub-20nm pitch sizes. First, the mostly used block copolymer PS-b-PMMA with perpendicular PMMA cylinders was studied using hard x-rays to probe the capabilities of different treatments (UV irradiation , chemical treatment and coupling of these two methods) to remove totally the PMMA blocks. Then, taking advantage of the enhancement of the electronic contrast between PS and PMMA blocks at the carbon-K edge (soft x-rays), measurements were carried out to characterize PS-b-PMMA films with spherical and parallel cylinder morphology in which PMMA blocks cannot be removed. For high-  $\chi$  copolymer films, (characterized by higher repulsion between

blocks), GISAXS measurements using hard x-rays led to a precise description of the reduced size patterns owing to a marked electronic contrast between the two blocks. Finally the new contact hole shrink approach developed at CEA was studied by GISAXS and CD-SAXS. A preliminary analysis of results has shown the better suitability of CD-SAXS for studying deep holes. The multiple scattering effects occurring in complex layer stacking and measured by GISAXS make difficult the extraction of the hole sizes.