ESRF	**Experiment title:** In situ X-ray scattering study of layer growth and ion erosion of thin films	**Experiment number**: SI1007
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Shifts:	**Local contact(s)**: Luca PEVERINI	

Names and affiliations of applicants (* indicates experimentalists):

Eric Ziegler - ESRF, BP 220 - 38043, Grenoble Cedex, France
Thierry Bigault - Institute Laue Langevin, BP 156 - 38042 Grenoble Cedex 9 - France
Igor Kozhevnikov - Crystallography Institute, Leninsky prospect 59, Moscow 119333, Russia

Report:

During the experiment SI-1007 we have studied the processes of sputter deposition and ion bombardment of W thin films using grazing incidence X-ray scattering. The objective of the experiment was the determination of the roughness spectrum evolution in term of scattering diagrams recorded in real time.

The setup used for the experiment is shown in Figure 1. It consist of a vacuum chamber with ultimate pressure 10^{-7} hPa, a magnetron sputter source, an ECR ion source, and a liquid-nitrogen cooled CCD camera (1024x256 pixels, Princeton Instruments) coupled to an image intensifier from Proxitronic. The scattering geometry, and the spatial resolution of the detector (LSF = 170 µm) placed at 1 m from the sample, enabled us to obtain information about the surface with spatial wavelengths spanning from 0.15 μm^{-1} up to 50 μm^{-1}. As the exposure time necessary for accumulating high quality XRS data (see Fig. 1) was in the order of the time needed to grow (or remove) a monolayer of material (0.2- 1 nm/min), real-time monitoring of the processes has been possible.

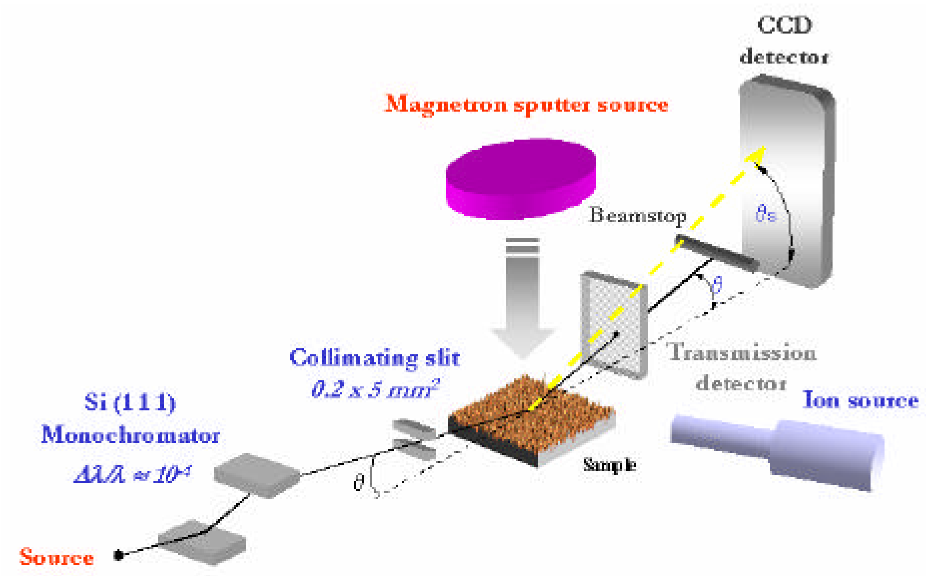

Figure 1 Setup of the experiment

Two experiments in situ during growth and erosion tungsten film were performed subsequently at different grazing angle of probing beam, namely θ_0 = 0.125 deg and 0.5 deg, so that the beam fell onto sample either in the total external reflection (TER) region or out of it (OTER) (the critical angle at a working energy of 17.5 keV is about 0.25 deg for tungsten). Tungsten films were thus deposited onto silicon substrates under identical technological conditions and the diffuse scattered intensity distribution was recorded in situ as a function of the grazing scattering angle. Using an ionization chamber placed downstream of the sample the intensity specularly reflected was recorded in parallel. Immediately after the deposition process the film was irradiated with 1 keV Ar ions and a set of measurements were performed following the same method. The measurements of the scattering diagram were performed after each 30 s of deposition, and integration time of CCD detector was 0.1 s or 30 s for measurement in TER or OTER, respectively.

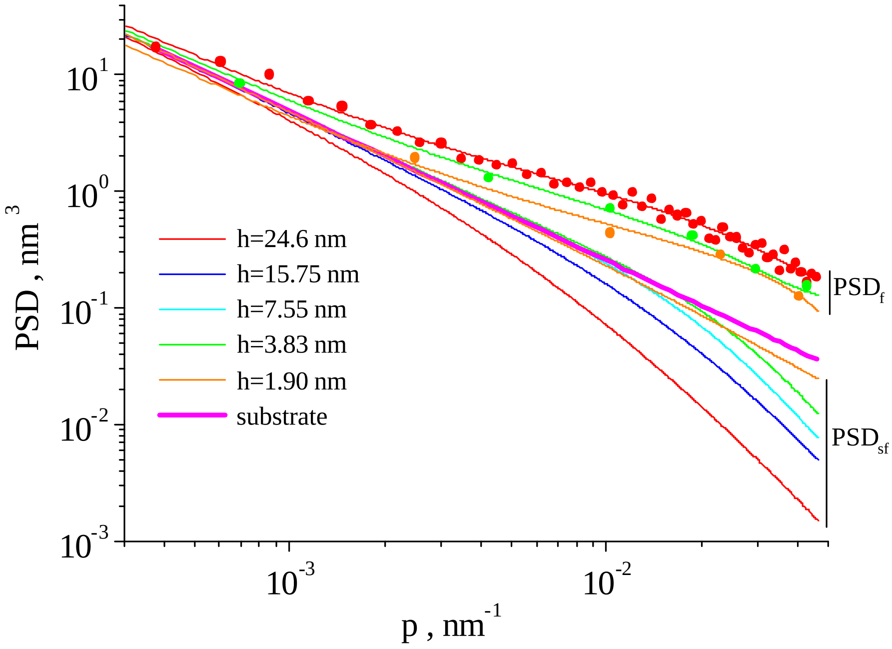

Figure-2: PSDs calculated (PSD_f) from a sequence of scattering diagrams recorded at different film thicknesses and mutual correlation between film and substrate topography(PSD_{sf}). The solids lines shows the smoothened PSDs extracted using a logarithmic fit of the measured data.

As an example, figure 2 shows PSDs of the external surface calculated[1,2] from the diffuse scattering diagrams during the growth process and the PSDs representing the mutual correlation between film and substrate topography. In parallel, the total reflectivity signal was used to extract information about film density and thickness variation with time during the experiment necessary for the extraction of the PSD function.

As one can see, the PSD-functions of the external film surface increase with the film thickness due to the development of intrinsic film roughness. Moreover, the variation of the PSD-function is more pronounced at high spatial frequencies, as also predicted from the existing theories on film growth[3,4]. In the measured range of spatial frequency this increase in the PSD corresponds to a slight increase of the rms roughness, from 0.18 nm for the bare substrate up to 0.23 nm after deposition of a 24.6 nm thick film. The analysis of the PSDs presented on figure 2, enabled us to obtain the expected information: 1) the evolution of the cross correlation between film topography and substrate of the study processes [5] 2) the scaling behavior of the film during both growth and erosion of tungsten film. The scaling exponents characteristic of the two processes were finally obtained through a data collapse of the found in roughness spectra.

These results are novel and proved the suitability of the method to quantitatively analyze growth processes in alternative to conventional scanning probe techniques, e.g., (AFM or STM). The experiment is thus considered successful.

References:

1. I. V. Kozhevnikov, Journal of X-Ray Science and Technology **8,** 253-275 (2000).
2. V. E. Asadchikov, I. V. Kozhevnikov, and Y. S. Krivonosov, Crystallography Reports **48,** 836-850 (2003).
3. A.-L. Barabâasi and H. E. Stanley, *Fractal concepts in surface growth* (Cambridge University Press, Cambridge, 1995).
4. D. G. Stearns, Applied Physics Letters **62,** 1745-1747 (1993).
5. L. Peverini, E. Ziegler, T. Bigault, and I. Kozhevnikov, Physical Review B (Condensed Matter and Materials Physics) **72,** 045445 (2005).