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Experiment Report Form

	Experiment title: Improvement of neutron multilayer optics: x-ray scattering during Ni thin film growth and ion erosion.	**Experiment number**: MI-893
Beamline:	**Date of experiment**: from: 21/11/07 to: 26/11/07	**Date of report**: 14/02/08
Shifts:	**Local contact(s)**: Dr. Eric ZIEGLER	*Received at ESRF:*

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Preliminary report (data analysis still in progress):

Part of the experiment was performed as expected, but some problems occured which made it impossible to perform the whole experimental scheme detailed in the proposal. First, the pulsed power supply to allow for pulsed DC magnetron sputtering could not work for technical reasons, thus this part of the experiment could not be performed. Second, measuring the scattering from the surface roughness of the superpolished Silicon substrate that was used as a starting surface happenned to be challenging, due to the very good surface quality and hence very weak scattering signal. Thus, in addition to the usual time allocated to setting up the experiment (potisionning of elements, alignment, growth calibrations, etc), a noticeable part of the beamtime was used to get very clean conditions for the scattering measurement, mainly by aligning several slit sets at different distances before the sample. This can be considered as an extension of the beamline commissionning, and it was essential for several user experiments that came just afterwards and that used the same setup. A flat field calibration of the CCD-

detector and the measurement of reference scans with the scintillation counter, which can be considered the same way, were also performed during this experiment. The part of the experiment concerning surface modification with ion irradiation could not be performed in good conditions, since the ion gun could not work properly during the time of the experiment. Some measurements were taken during ion processing, but the data appear to be nearly impossible to use. Last, the experiment suffered from serious beamline Front End problems, which also caused a significant time loss

Despite these points, part of the initial program could be performed. The incident beam energy was calibrated and set to 17.5keV. The growth rates of the Ni layer for two conditions, namely sputtering with Argon and sputtering with a mixture of Argon and air with the same total gas flow rate, were calibrated thanks to the real-time reflectivity measured with the ion chamber (see Figure 1).

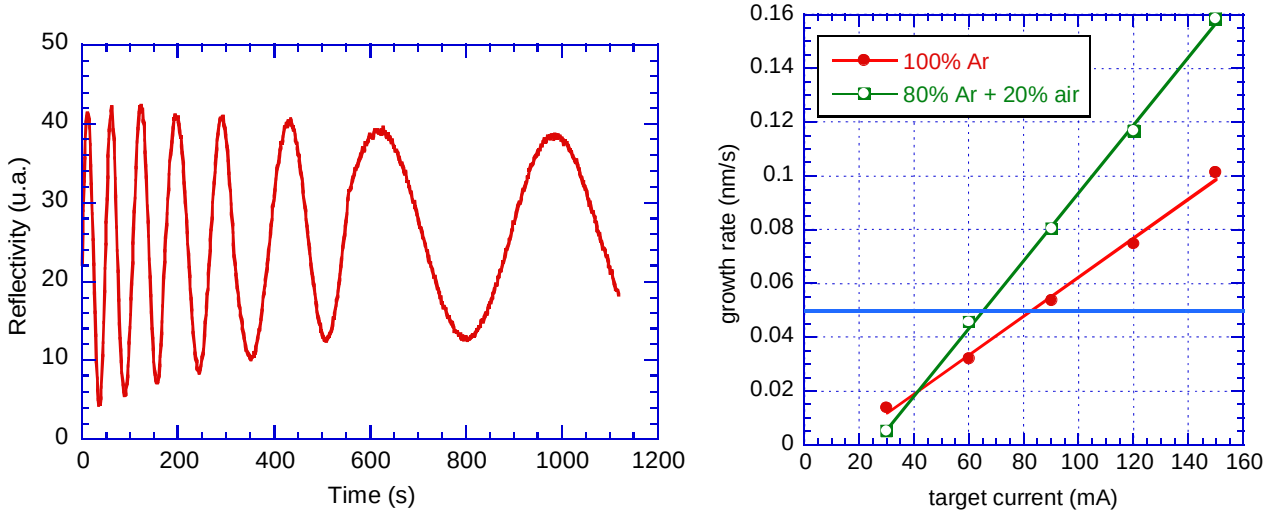

Figure 1: Left: in-situ real time reflectivity measured during the growth of Ni with Argon, changing the target current from 150 to 30 mA. Right: growth rate calibration deduced from the in-situ reflectivity for the two types of growth conditions. The thick line indicates the growth rate finally used for in-situ scattering experiments.

These were used to grow the two types of Ni layers up to about 50 nm, starting each time from a fresh Si surface, with a rate of 0.05 nm/s. This rate was chosen to get closer to the conditions used in the actual production of neutron supermirrors, but still making the in-situ scattering measurement possible.

In each case, complete reflectivity (Figure 2 (a) and (c)) and scattering scans from the Si surface were measured accurately before starting the deposition. Then the scattering diagrams were recorded in real time during the two types of deposition with the CCD detector, at the incidence angle $\theta=0.14°$ ($> \theta_c^{Ni}$). Reflectivity scans were measured again after deposition (Figure 2 (b) and (d)). Some differences already appear on the reflectivity curves measured on the layers deposited in the two types of conditions, while the reflectivity from the starting Si surface look very similar, suggesting that some distinct processes took place during growth in the two cases. Some qualitative differences can also be seen between the two cases on the scattering data (not shown), but this is only preliminary since some extended analysis, which is in progress, is necessary to properly interpret these data.

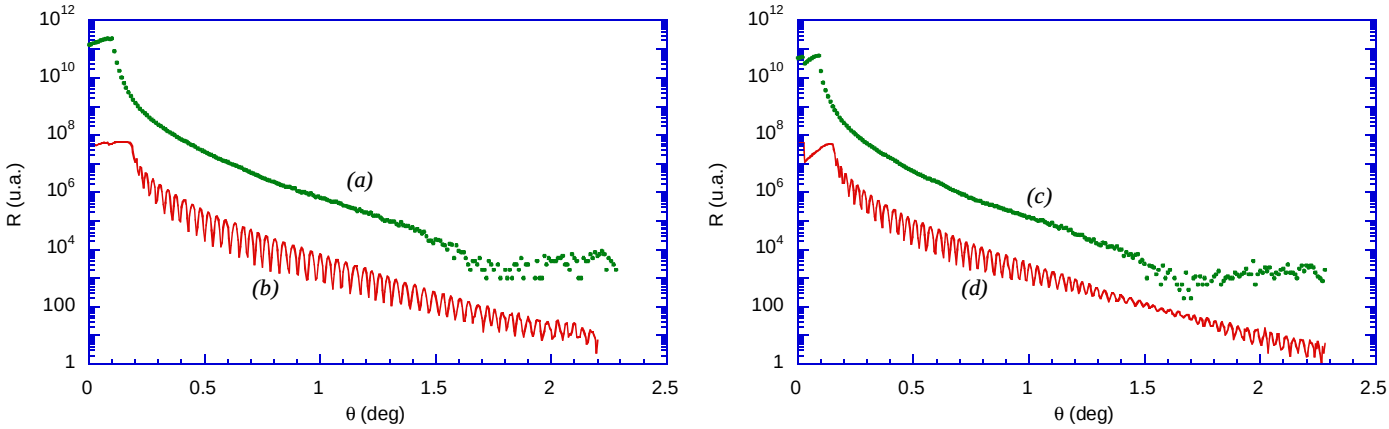

Figure 2 : Reflectivity scans measured before and after depositing about 50nm of Ni in different conditions on a superpolished Si surface: before (a) and after (b) deposition with 100% Argon; before (c) and after (d) deposition with 80% Argon and 20% air. The curves are shifted vertically for clarity.