EUROPEAN SYNCHROTRON RADIATION FACILITY

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Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

Reports supporting requests for additional beam time

Reports can now be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** Grazing-incidence diffraction from polycrystalline multilayers	**Experiment number**: SI-1306
Beamline: ID01	**Date of experiment**: from: 10. 5. 2006 to: 16. 5. 2006	**Date of report**: 18. .8. 2006
Shifts: 15	**Local contact(s)**: Till Hartmut Metzger	*Received at ESRF:*

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Report:

X-ray diffraction from periodic polycrystalline multilayers in grazing-incidence geometry is a new method making possible to determine the parameters of polycrystalline layers and their changes across the multilayer stack [1]. The method uses the fact that the primary beam irradiating the sample under a small incidence angle reflects specularly at the multilayer interfaces and the interference of the transmitted beam with the reflected beams creates a standing wave, the period of which equals the multilayer period. The grains of the polycrystalline material in the multilayer are irradiated by this standing wave, so that the intensity of the wave diffracted from these grains is modulated by the standing-wave intensity distribution. Similarly, after diffraction from the grains, the wave is reflected and transmitted through the multilayer interfaces and another standing wave results. Changing the angle of incidence α_i or the angle of exit α_f of the measured x-ray beam, the measured diffracted intensity $I_{\boldsymbol{h}}(\alpha_i, \alpha_f, 2\Theta)$ exhibits sharp maxima or minima corresponding to multilayer satellite maxima in the reflectivity curve $R(\alpha_i)$. Here we have denoted 2Θ the scattering angle in the azimu-thal direction (see Fig. 1). The position of the antinodes of a standing wave created by the interference of the beams transmitted and reflected from a periodic multilayer depends on the incidence angle and it can be sensitively shifted by changing α_i. Therefore, the shape of the diffraction curve $I_{\boldsymbol{h}}(\alpha_i, \alpha_f, 2\Theta=\text{const})$ depends sensitively on the position of the diffracting layer in the multilayer period; a diffraction maximum is observed, if the diffracting layer lies in an antinode of the standing wave.

Preliminary measurements using a laboratory set-up [1] demonstrated the feasibility of the concept and the beamtime SI1306 was devoted to thorough tests of the method. We have measured a series of 4 Ni/C multilayers deposited by magnetron rf sputtering on glass substrates. The period $D = T_{Ni} + T_C = 20$ nm of the multilayer was the same in all samples, the ratio T_{Ni}/D changed from ¼ (sample H1) to 4 (sample H4) . The measurements were carried out using two energies – 8.2 keV and 8.33 keV, the latter is close to the NiK absorption edge. For given incidence angle α_i we measured the intensity distribution $I_{\boldsymbol{h}}(\alpha_i = \text{const}, \alpha_f, 2\Theta)$ using a linear detector perpendicular to the sample surface and changing the azimuthal scattering angle 2Θ.

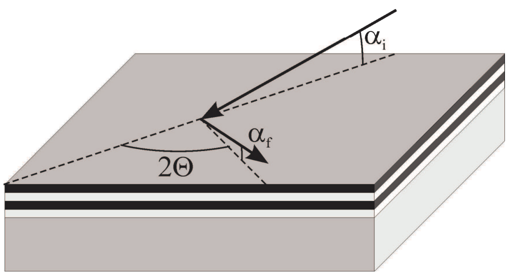

Fig. 1. Sketch of the scattering geometry

Figure 2 shows the reflectivity curve of sample H1, and in Fig 3. we present the 2Θ scan of the same sample measured for α_i = 1 deg. The peaks in the curve were identified to highly textured pure Ni and nearly not textured Ni_3C phases. In Fig. 4 we have plotted, an example of the measured intensity map in the angular (α_f,2Θ) space for α_i = 0.45 deg; this value corresponds to the intensity minimum between two satellite maxima at the reflectivity curve. The map represents a part of the Debye-Scherer ring; the sharp modulation along the α_f axis is clearly visible.

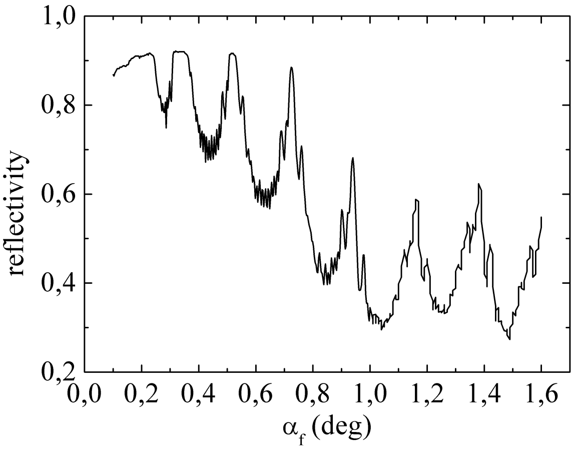

Fig. 2. Reflectivity of sample H1, 8.2 keV

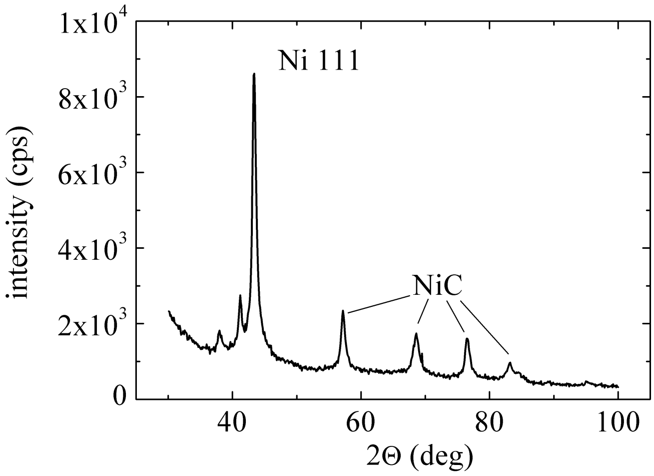

Fig. 3. 2Θ scan of sample H1, 8.2 keV

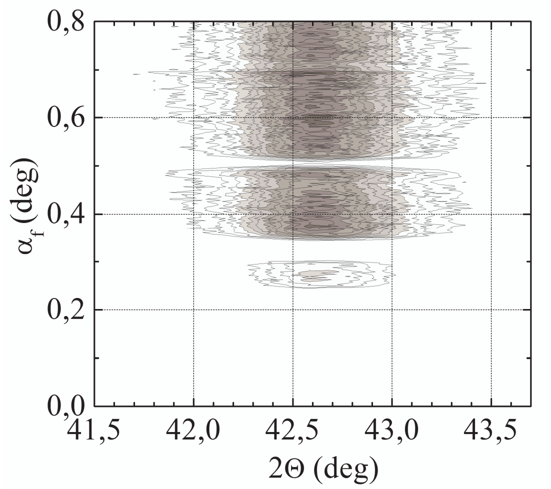

Fig 4. (2Θ,α_f) intensity map of sample H1, 8.2 keV, α_i = 0.45 deg

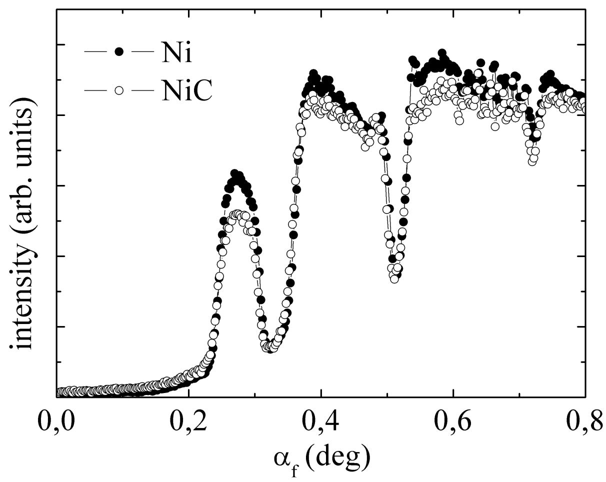

Fig. 5 2Θ-integrated intensities, 2Θ = 42.5 deg (Ni) and 2Θ = 57 deg (NiC)

Figure 5 shows the main result of the measurements – the comparison of the 2Θ-integrated intensities measured around 42.5 deg (Ni 111 diffraction) and 57 deg (diffraction maximum of NiC). The curves differ especially close to the standing-wave modulation at α_f = 0.5 deg; the difference can be ascribed to the fact that the Ni and NiC layers have differfent positions in the period of the Ni/C multilayer. The NiC layers occur at the Ni-C interfaces due to an interdiffusion and subsequent chemical reaction.

From the comparison of the measured curves with numerical simulations it will be possible to determine exactly the position of the NiC layers in the multilayer and to study the interdiffusion process in detail. This will be the subject of further investigations.

[1] P. F. Fewster, N. L. Andrew, V. Holy, and K. Barmak, Phys. Rev. B **72**, 174105 (2005).