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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:**

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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.

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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:** Solid state reactions at nanoscale: In-situ and real time study of the silicide formation, nanostructuration, and interfacial roughness by combined X-ray diffraction, X-ray reflectivity, sheet resistance and laser reflectometry	**Experiment number:** MA-126
Beamline: BM05	**Date of experiment:** from: 29 November 2006 to: 5 December 2006	**Date of report:** 12/01/2007
Shifts: 18	**Local contact(s):** Dr. Eric Ziegler	*Received at ESRF:*

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Report:

The aims of this experiment were to investigate the first stages of the silicide formation and the development of the roughness linked to this formation.

Metal silicides are widely used as contacts and interconnections in very large-scale integrated (VLSI) circuits because they can decrease the contact resistance and thus increase the speed of the devices. The Ni monosilicide (NiSi) is the most promising candidate for future CMOS. With the continuous scaling down of devices, the silicide thickness should be decreased and it becomes important to understand and control the first stages of the silicide formation. Furthermore, very shallow junctions are needed for the future transistors and it is more and more critical to control the roughness of the silicide/silicon interface. Indeed the roughness of this interface can lead to unacceptable leakage current in CMOS [1].

The fundamental mechanisms of the silicide formation are still not fully understood. Due to the practical interest of silicides in the microelectronic industry, the solid-state reaction between a thin metal film and Si has been analysed extensively [2], [3]: for relatively large thicknesses of silicides (several tens of nanometers), the formation is sequential and usually controlled by diffusion with a parabolic growth rate [4]. However, recent synchrotron experiments [5] have shown the formation of transient phases ($Ni_{31}Si_{12}$, Ni_3Si_2) with very short lifetime (~30 s) that is not in accordance with the usual sequence of phase formation. For low thicknesses, the growth should also be controlled by the interfacial reaction and other phenomena like nucleation, lateral growth, and stress should certainly play an important role [6].

Experimental method

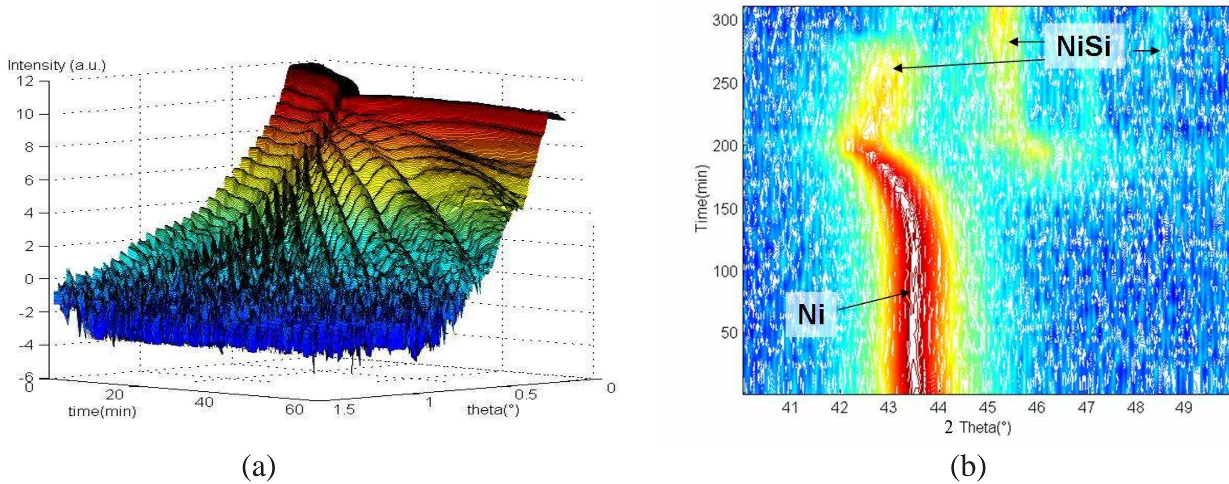
We have reported from former experiments at ESRF on BM05 (see report n°30785-A and the report for MA-15), that for technical reasons, the detector movement was not stable during the fast scans. It was also not possible to record simultaneously x-ray diffraction and reflectivity. In June 2006, the stability of the detector was improved using a counterweight, but the vacuum set-up was not enough efficient and most of

our samples have been oxidized. Between June and December, we have fabricated a new cover, able to content the BM05 furnace and to prevent from vacuum leakages.

Ni, Pd and Ni(Pt) thin films with thickness ranging from 10 to 50 nm were deposited on Si(100) and Si(111) at L2MP. *In situ* reflectivity measurements were performed with constant heating rates and during isotherms at different temperatures below 500°C in a vacuum chamber. A new specific vacuum chamber was used and the vacuum measured in the chamber was at least 10^{-6} mbar. A thermocouple was used to control the heating element and another thermocouple was in contact with the sample. The contact between the thermocouple and the sample has also been improved. A maximum of 20°C/min heating rate was used.

Results

Isotherm annealings were performed on Ni/Si(100), Ni(Pt)/Si(100) and Pd/Si(111).



(a) (b)

Figure 1: In-situ experiments on 40 nm Ni(13%Pt) deposited on Si(100) **1.a)** RRX curves obtained on BM05 during an isotherm heat treatment at 260°C. One scan takes 1 minute. The first scan is as-deposited, after 12 min, the temperature is maintained at 260°C.**1.b)** XRD curves recorded at the L2MP, during a temperature ramping between 100°C and 400°C with a step of 10°C. One scan takes 5 minutes.

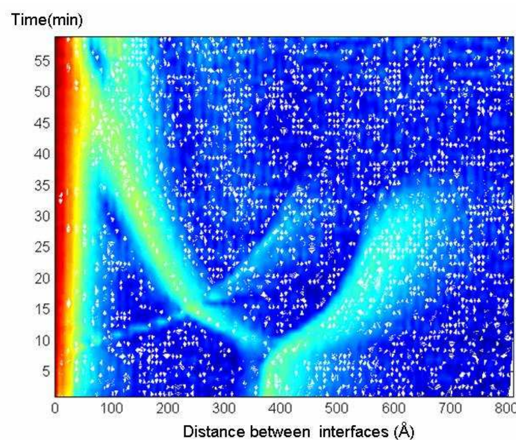


Figure 2: Inverse FFT of the RRX curves of figure 1.a; the peak around 400 Å corresponds to the total stack thickness ; the two other peaks correspond to the Ni film that decreases, and to the Ni_2Si phase that increases.

As a first result, we have shown in Fig.1 (a), the XRR curves obtained on BM05 during an isotherm treatment at 260°C for 40 nm of Ni (13%Pt) deposited on Si(100). The evolution of the curves is not obvious to interpret. To characterise the present phases, XRD are needed. In situ XRD experiments recorded at the L2MP on the same sample during a temperature ramping between 100°C and 400°C with a step of 10°C are shown in Fig. 1(b). The evolution of the intensity of the peaks, characteristic of the phases is given by the levels of the colors (from red, highest instensity, to blue, lowest intensity). We can follow with temperature, the consumption of Ni, with the accumulation of Pt in the Ni that has not been consumed (the peak moves towards lowest angles) and the appearance of the NiSi phase. Thanks to the program we have developped to treat the reflectivity curves by fast Fourier transform, we have obtained the results shown in fig. 2. We can clearly follow the evolution of the thicknesses of the different layers:

- around 400 Å, the peak corresponds to the total stack thickness
- after 10 min, the Ni film decreases (from 400 Å to 0) and to the Ni_2Si phase increases.

Conclusion

These experiments are very encouraging and have given nice results that are analyzed at the present time. When rugosity is not too high, when the vacuum set-up is enough sufficient, the reflectivity is a very efficient tool to determine the formation kinetics of thin films.

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2. Nicolet, M.-A., in *VSLI Electronics, Microstructure Science*, N.G. Einspruch and G.B. Larrabee, Editors. 1983, Academic: New-York. p. 330.
3. Gas, P. and F.M. d'Heurle, Applied Surface Science, 1993. 73: p. 153-161.
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5. Lavoie, C., *et al*, *Towards implementation of a nickel silicide process for CMOS technologies.* Microelectronic Engineering Materials for Advanced Metallization 2003, 2003. 70(2-4): p. 144-157.
6. Nemouchi, F. *et al*, Applied Physics Letters, 2005. 86: p. 41903.