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ESRF

Experiment Report Form

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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.

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- 1st March for experiments carried out up until June of the previous year;
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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:** **In-situ study of the growth kinetic and interfacial roughness during the first stages of silicide formation using grazing incidence diffraction and reflectivity**	**Experiment number**: **ME1003**
Beamline: BM5	**Date of experiment**: from: 09/03/05 to: 14/03/05	**Date of report**: 11/01/2006
Shifts:	**Local contact(s)**: E. Ziegler	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

Introduction:

The aims of this experiment were to investigate the first stages of the silicides 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 because of the following advantages: low resistivity and low Si consumption, formation controlled by Ni diffusion, adequate work function for metal gate.

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 analyzed extensively [2,3]: for relatively large thickness 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 thickness,

the growth should also be controlled by the interfacial reaction [6]. Other phenomena like nucleation, lateral growth, stress should certainly play an important role.

Experimental method:

Pure Ni or Ni(Pt) thin films with thickness ranging from 10 to 50 nm were deposited by sputtering on a-Si and (100)Si 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 specific vacuum chamber was used. A thermocouple was in contact with the sample and another thermocouple was used to control the heating element. A maximum of 20°C/min heating rate was used.

Results:

Figure 1 shows *in situ* reflectivity curves at different temperatures during a heating ramp (5°C/min) of a 50 nm Ni(5%Pt) film on (100)Si. During this experiment, the beam alignment has been readjusted several time to compensate for the loss of alignment. Despite the difficulty of the measurement, figure 1 shows the change in the modulation spacing and intensity associated to the formation of the silicides and the development of roughness linked to this formation.

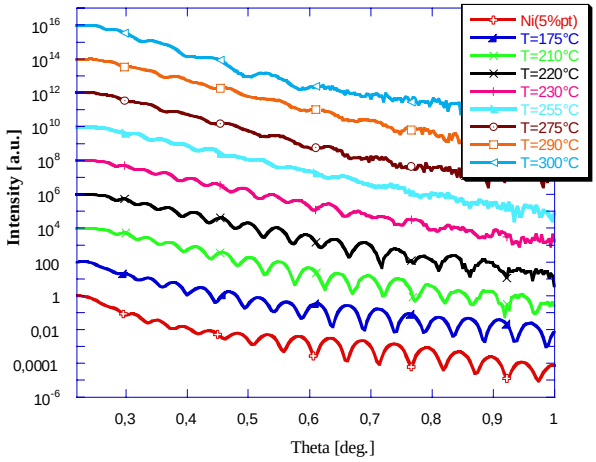

Figure 1 : X ray reflectivity spectra for a 50 nm Ni (5%Pt) film on (100)Si during heating (5°C/min)

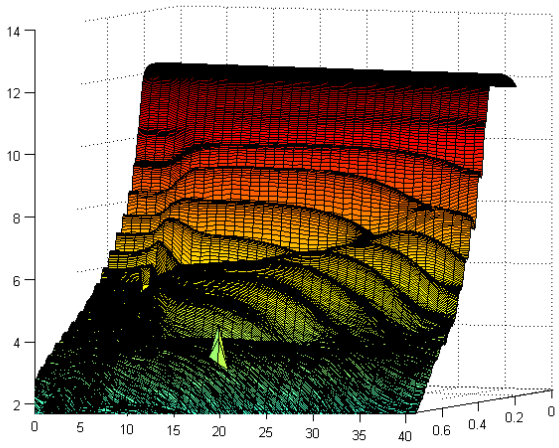

Figure 2 : X ray reflectivity spectra 50 nm Ni film on (100)Si during *in situ* annealing

Figure 2 shows the reflectivity spectra as a function of time during a heat treatment (20°C/min up to 150°C followed by 1°C/min up to 300°C) of a 50 nm Ni film on (100)Si.
Several oscillations might be seen but it is difficult to directly conclude on the phase formation from these curves. In order to interpret the results, *ex situ* X-ray diffraction were performed at L2MP after the different heat treatment. The simulation of the X-ray reflectivity spectra combined with X-ray diffraction results have shown that the change in the X-ray reflectivity spectra corresponds to the growth of Ni_2Si at the expense of Ni [7]. Further analysis are under investigation.

Some *ex situ* measurements were also performed on samples annealed by rapid thermal process. Figure 3 shows the reflectivity spectra for a 9 nm Ni film on (100)Si doped with As annealed by rapid thermal anneal during 30 s at temperatures between 400°C and 650°C. One can see that even for a very small thickness of Ni and relatively high annealing temperature, the reflectivity spectra allow to determine the thickness and the roughness of the silicide. Figure 3 shows also that even after the formation of NiSi that follows the one of Ni_2Si (T=400°C), the roughness is sufficiently low to obtain a nice reflectivity curve. The increase in roughness for higher annealing temperature is due to the agglomeration of the NiSi layer that was confirmed by atomic force microscopy, resistivity measurement, and scanning electron microscopy [8].

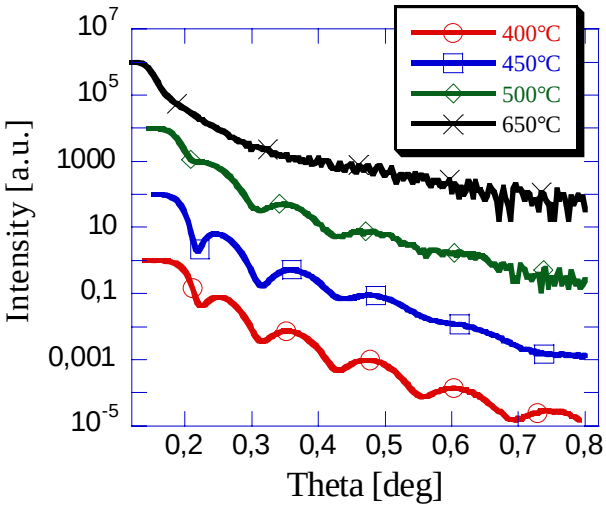

Figure 4 : Reflectivity spectra for 9 nm of Ni on (100)Si doped with As.

Conclusion:

In situ and real time X-ray reflectivity measurements during heat treatment have been performed to study the formation of Ni silicide and the roughness linked to this formation. The heat treatment were performed with constant heating rates and during isotherms at different temperatures below 500°C in a vacuum chamber. These first measurements are very promising and show that it is possible to follow *in situ* and in real time the silicide formation and the development of the roughness.
The *ex situ* spectra obtained on very thin films of NiSi show that it is possible to analyze weak quantities of silicide and to characterize the agglomeration phenomenon of NiSi.

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[8] K. Hoummada, D. Mangelinck, C. Perrin, P. Gas, V. Carron, F. Laugier, and E. Ziegler, abstract sent for the MAM workshop (Grenoble, 2006), to be published in Microelectronics Engineering.