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Experiment Report Form

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

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Reports on experiments relating to long term projects

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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;
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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 reflectivity and grazing incidence diffraction	**Experiment number**: **MA-15**
Beamline: BM05	**Date of experiment**: from: 16 June 2006 to: 20 June 2006	**Date of report**: 12/01/2007
Shifts: 12	**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

In a former experiment at ESRF on BM05 (see report n°30785-A), several XRR in-situ measurements including isotherm and ramp annealings of Ni thin films on Si(100) have given interesting results concerning the first stages of reaction [7]. Unfortunately the stability of the experimental setup was not sufficient to fully analyse the silicide formation and especially the roughness.

Since these experiments, a counterweight was installed on the 2 theta arm and this was efficient to improve the stability of the detector. However, despite different tests, it was not possible to record simultaneously x-ray diffraction and reflectivity.

The samples were pure Ni or Ni(Pt) thin films with thickness ranging from 10 to 30 nm deposited on Si(100) 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

Isotherm annealing were performed on Ni/Si(100). These *in situ* reflectivity measurements were analysed with a program developed during the last months at L2MP. This program allows to treat the high quantity of files resulting from the necessity to record short scans during a long period of time (about one scan per minute during few hours). Each file was treated by a fast Fourier Transform (FFT) to deconvolute the reflectivity curve. This allows to obtain the thickness corresponding to the different layers that appear or disappear during the annealing. However the layers should not be too numerous and have distinct thickness.

For 10 nm of Ni deposited on Si(100), the simulation of the reflectivity spectra have shown that during the isotherm annealing, Ni is consumed when Ni_2Si phase grows as expected [7]. They have also shown that an oxygen containing layer is present at Ni/Ni_2Si interface. The presence of the oxygen enriched layer was later confirmed by TEM.

For 30 nm of Ni deposited on Si(100), the simulation shows a resulting stack is $Ni/Ni_2Si/NiSi/Si$ with eventually an oxide layer between Ni and Ni_2Si for temperature below 350°C. This shows that Ni_2Si and NiSi are formed during the annealing below 350°C.

For most of the experiments, Ni did not react with the Si substrate and a Ni oxide was formed: this was due to a poor vacuum level induced by a leak in the chamber. Because of the presence of the layer with oxygen, it is difficult to conclude about the kinetics of formation of Ni_2Si.

Conclusion

We were able to follow "in situ" the reaction between a thin film of metal (few nanometers) and a Si(100) substrate by X-ray reflectivity thanks to the ESRF beam. An intense X-ray synchrotron beam is essential for such a small amount of matter and to be able to follow the reaction kinetics. It has been shown that thin films up to 10 nm could be studied by XRR. We have also shown that the results of in situ experiments could be treated by FFT in order to follow the consumption of the deposited metal (Ni or Ni with Pt) and the growth of the expected silicides.

The stability of the alignment has been greatly improved. However an unexpected oxidation has been found and have complicated the interpretation of the results. The vacuum chamber has to be improved to prevent oxidation on our samples.

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