ESRF	**Experiment title:** **In -situ study of the Solid State Reaction between an ultrathin metallic film and an InGaAs substrate**	**Experiment number:** **MA-2455**
Beamline: BM02	**Date of experiment:** from: 13-11-2014 to: 17-11-2014	**Date of report:** 10/02/2015
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Introduction :

III-V alloys are an adequate alternative for silicon in the advanced CMOS industry and are able to help continuing the down-scaling of MOSFET transistors. Nevertheless, ultra-low contacts are mandatory in order to achieve high-performance logic operation. The mainstream is to use solid-state reaction in order to form an intermetallic compound (metal/III-V) contact.

Thus, the aim of the present experiment was to probe the kinetic parameters, phase sequence and crystallographic structures of the formed intermetallic after a solid-state reaction (SSR) between Ni and InGaAs. This goal was achieved by means of 3D Reciprocal Space Mapping. This campaign was conducted from 13th of November to the 17th of November and it is the first of a series of campaigns that aim to describe novel materials dedicated to form the contact materials for the advanced sub-7nm MOSFETS based on III-V materials.

Sample Description:

We used 2 types of samples. The original stack is common for these samples and consisted of an InP substrate on which we deposited a layer of 150 nm of $In_{0.53}Ga_{0.47}As$. Nickel deposition of two film thicknesses (7 nm and 20 nm) followed next and was performed by means of RF-PVD. Finally a thin film (7 nm) of TiN was deposited on the stack in order to prevent sample contamination. Full description of both samples is given in Figure 1.

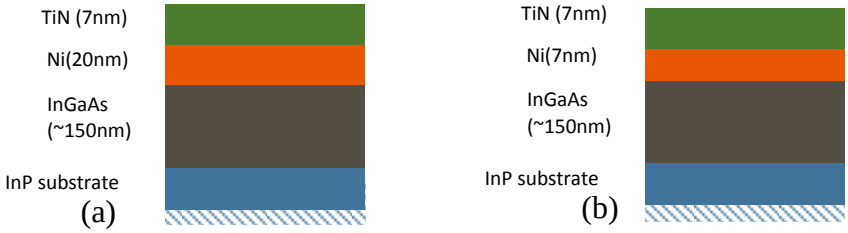

Figure 1. Scheme of the as-deposited stacks. (a) TiN/Ni(20 nm)/inGaAs/inP, (b) TiN/Ni(7nm)/inGaAs/inP

Experimental setup and methodolgy:

The experiments were carried at the BM02/D2AM beam line equipped with a 6-circles goniometer (Kappa geometry) and a 2D pixel detector (XPAD). The energy was set to 10 keV which is relevant in order to prevent Ga and As fluorescence. A furnace compatible with the goniometer (Anton Paar DHS 1100) was set up in front of the X-ray beam in order to perform the annealings. To prevent atmosphere contamination during the annealing, we covered them with a thermostable dome made of Polyether ether ketone (PEEK). The annealings were done under constant highly pure N_2 flow. Before each measurement, the sample environement under the protective dome has undergone several cycles of pumping to primary vaccum followeed by N_2 injection to evacuate residual air in it. The detector was fixed at 25° and at 20 cm from the samples (which corresponds to roughly 20° of aperture range). Finally, χ angle of 50° was set, which gives a maximum range going from 0° to 70° depending on the 2θ position. Temperature was monitored throughout the main software controlling the goniometer. Measures were done for several φ angle positions (on 360°) in order to cover a large part of the reciprocal space and reconstruct full pole figures over 360°. As the 2D camera has some dead rows and columns, we did each acquisition at ν=0° and 2° (in-plane rotation of the detector). In order to calibrate the setup, a LaB6 powder was mesured as well.

Data was treated using the DEVA software developped by our team in collaboration with the D2AM team [1] [2] specifically for image and data processing on this beam line. In this configuration, several Debye-Scherrer rings were recorded on the same picture with a 10 s exposure time. Thus, the acquisition time is considerably reduced compared to standard θ-2θ measurements.

Two variants of annealing were put in place. First of all, we annealed the samples by ramps starting from a temperature of 150 °C up to 550 °C with steps of 20 °C. At each step full 3D RSM was performed. This type of measurement permits to follow the intermetallic formation steps and eventually its degradation. Secondly, we performed isothermal measurements at one given temperature (185 °C) for more than 16 hours in order to extract the kinetics' parameters that corresponds to the intermetallic formation. These measures were carried out for both Ni-thicknesses.

First day has been dedicated to the furnace setup, annealing and sample/beam alignements. We spent the next 2 days doing the annealing ramps measurements. The last 2 days were dedicated to isothermal measurements.

Results:

This campaign was very profitable as we identified several (hkl) planes of the hexagonal intermetallic as described by the litterature [3][4]. The intermetallic formation was monitored either in the case of ramps or isothermals. For instance, in Figure 2 the evolution of the {101} plane pole figures of the intermetallic at different temperatures. We observe that the intermetallic is formed at 210 °C and begin to deteriorate at a temperature of 410 °C. A new phase is observed at 510°C. Thus, the so-formed intermetallics with hexagonal structure seem to degrade at higher temperatures and a new phase, yet to be identified, is formed along with this degradation.

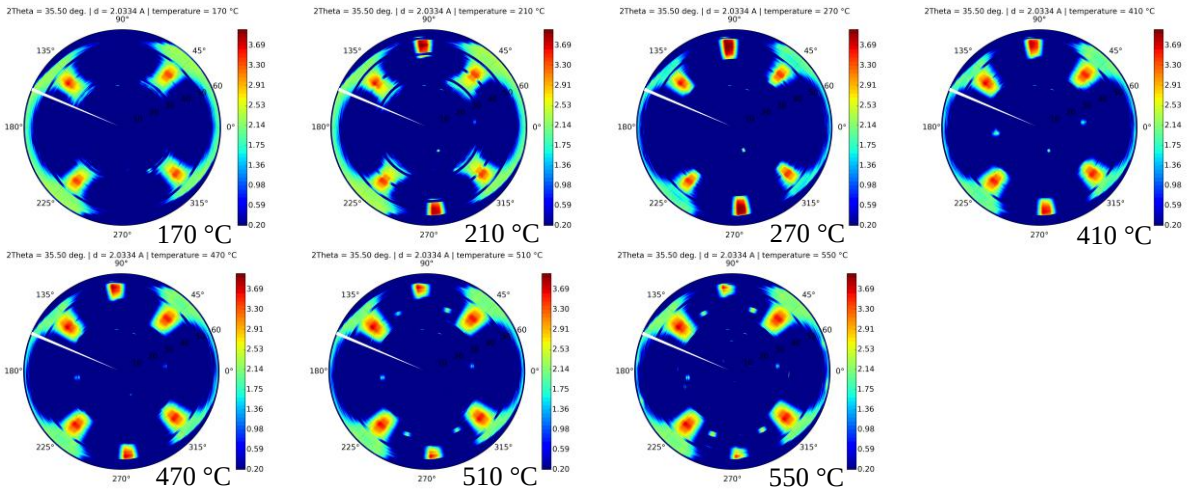

Figure 2. X-ray diffraction pole figures of Ni/InGaAs/InP sample (at different temperatures). The fixed angle was 29.75° corresponding to the intermetallic (101) reflection.

Unlike our ex-situ measures [2], we observed only one orientation of the intermetallic in place of 4. This observed orientation is the following : $(100)_{NiInGaAs}\| (001)_{InGaAs;}$ $[001]_{NiInGaAs} \| [0\text{-}1\text{-}1]_{InGaAs}$

We are still running data analysis in order to provide full crystallographic and kinetic descripton of the so-formed Ni-InGaAs intermetallic. These results are innovative enough for publication. We thus already submitted an abstract to the IITC/MAM 2015 conference (May 2015) which will be held in Grenoble. A peer-reviewed paper will be also submitted very soon.

Perspectives:

Co was already studied as a contact material [5] but the corresponding intermetallic yielded a very non-uniform interface with the semiconductor although it exhibits a relatively good stability at higher temperatures. Recent electrical measurements on Ni-Co alloys seem prove that this alloy could be a promising option in order to stabilize the intermetallic orientation at temperatures greater than 400°C. As a matter of fact, we have already observed through ex-situ experiments unusual textures compared to the Ni based intermetallics, which could explain this enhanced behavior as suggested by S. Gaudet for equivalent silicides [6]. These differences can be explained by lattice parameter mismatch/match, phase kinetics, etc… A further study on theses alloys is vital in order to enhance thermal stability and electrical properties.

References:

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