Proposal title : In-situ study of the formation of Ni-InGaAs intermetallics on GaAs/Si substrates : towards device applications		**Proposal Number** : 02-02-840
Beamline : D2AM/ ESRF BM02	**Date(s) of experiment** : from 10/11/2016 to 14/11/2016	**Date of report** : 08/02/2017
Nb Shifts : 12	**Local contact(s)**: Nils Blanc, Nathalie Boudet	**Date of submission** : 15/02/2017

Objective and expected results (less than 10 lines):

The objective of the present experiment was to investigate the kinetic parameters, phase sequence and textures of the formed intermetallic after a solid-state reaction (SSR) between Ni and InGaAs. This goal was achieved by performing *in-situ* 3D reciprocal space maps measurements at the D2AM/BM02 beamline of the ESRF. This campaign was conducted from 10-14 November, 2016 and it is the third 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. These experiments were very interesting mainly because we observed the texture of the intermetallic to be completely different according to the ramp-up speed during annealing.

Results and the conclusions of the study (main part):

1) Sample description :

Our experiments mainly consisted of 2 different parts. In the first part of this experiment, the original stack which consisted of an InP substrate on which a layer of 150 nm of $In_{0.53}Ga_{0.47}As$ was deposited. 20nm of Ni thin film was then deposited by RF-PVD. Finally, a thin film (7nm) of TiN was deposited on the stack in order to prevent sample contamination. In the second part of the experiment, the same samples which were pre-annealed at 4 different temperatures *i.e.*, 200°C, 250°C, 300°C and 450°C respectively were used for the further investigation of the intermetallic.

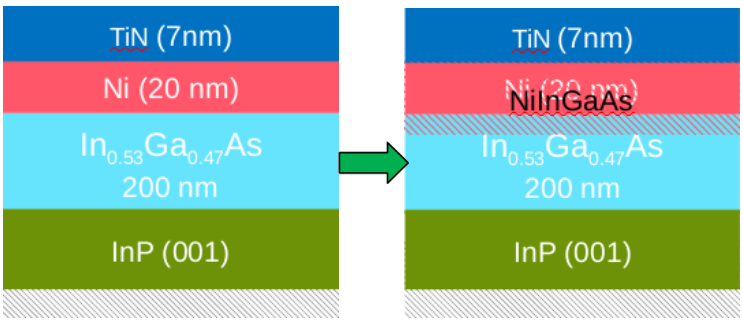

Fig. 1 (a) : Scheme of the as-deposited stacks TiN/Ni (20nm)/InGaAs/InP (b) Formation of the eventual intermetallic with the annealing process

2) Experimental setup and methodology:

The intermetallic exhibits a strong monocrystalline texture. Thus, we retain our method allowing to collect information along a large area of the reciprocal space is mandatory in order to keep track of all diffracting signals from the intermetallic. The experiments were carried at the BM02/D2AM beamline equipped with a 6-circle goniometer (kappa geometry) and a 2D pixel detector (FReLoN CCD camera). The energy was set to 10 keV which is relevant in order to prevent Ga and As fluorescence. A furnace (Anton Paar DHS 1100) compatible with the goniometer was set up in front of the X-ray beam in order to perform the annealing. To prevent atmosphere contamination during the annealing, we covered them with a thermostable dome made of Graphite. The annealing was performed under constant highly pure N_2 flow. Before each measurement, the sample environment under the protective dome has undergone several cycles of pumping to primary vacuum followed by N_2 injection to evacuate residual air in it. The

detector was fixed at 25° and at 16 cm from the samples. Finally, the experiments were performed at 2 different χ positions of 20° and 40°. Temperature was monitored throughout the main Spec software controlling the goniometer.

Due to our previous experience with Ni-based intermetallics, we chose to do measurements only over a 180° of φ angle positions. This choice was justified by the hexagonal symmetry of the intermetallic which can be deduced even with only half of a pole figure. Moreover, by doing so, we insure a more rapid and correct monitoring of the evolution of the intermetallic during the isothermal annealing. In order to calibrate the setup, a LaB_6 sample was measured as well.

Data is being treated by using an *in-house* developed software developed in collaboration with the D2AM team [1] specifically for image and data processing on this beamline. In this configuration, several Debye-Scherrer rings were recorded on the same picture with just 1 second exposure time. Thus, the acquisition time is considerably reduced compared to standard θ-2θ measurements. Isothermal annealing was performed as well in order to extract the activation energy for the formation of the intermetallic. Setup of the furnace and the sample/beam alignments were performed pretty fast in half a day thanks to the expertise of the D2AM team and the rest of the experimental shifts were utilized to perform the measurements.

3) Preliminary results:

We have demonstrated, thanks to our previous ESRF experiment (No. 02-02-827), that the texture of the intermetallic is completely different according to the ramp-up speed during annealing (Rapid Thermal Annealing vs. slow annealing). However, it is yet not clear which force drives the texture formation and thus responsible for such difference between two similar as-deposited samples. To better understand the texture formation, we used in the first part of the experiments, Ni-InGaAs on InP substrate samples which were pre-annealed at 4 different temperatures *i.e.*, 200°C, 250°C, 300°C and 450°C respectively. We then performed *in-situ* annealing on these samples to continue the intermetallic growth that has already started. Such procedure is very interesting since it begins with pre-annealed samples at different formation stages (*i.e.,* incomplete nucleation, incomplete Ni consumption or incomplete metal/semiconductor interface formation) and allow us to follow the growth evolution and texture formation with slow annealing (work to be published soon). From Fig. 2, we can see the so-formed intermetallic which presents

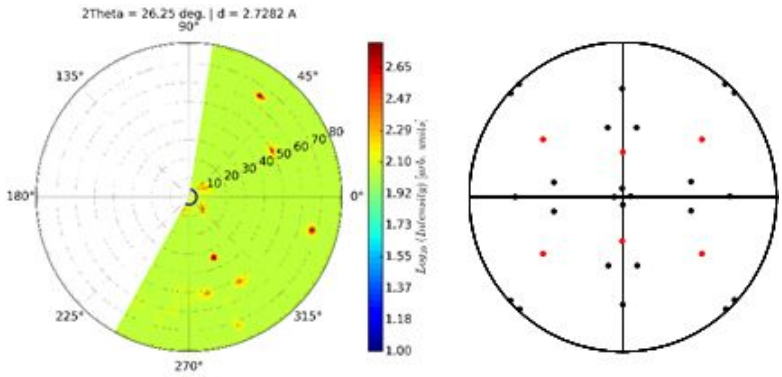

Fig. 2 (a): Pole figure of the Ni-InGaAs (at 500°C) which was preannealed at 300°C where we can see the hexagonal structure on the {10-11} intermetallic reflection and (b) corresponding stereographic projection

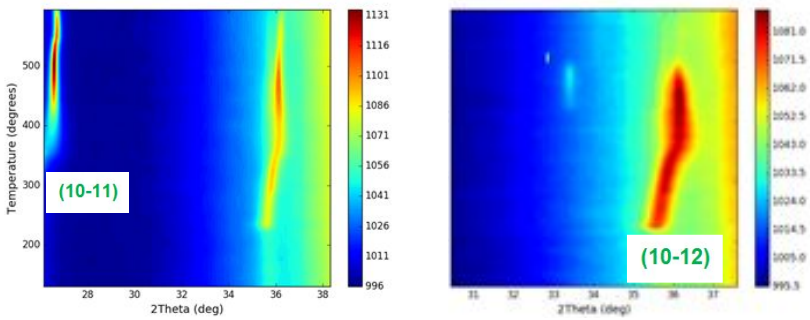

Fig. 3 : Color map of the evolution of intensities of (a) 10-11 (b)10-12 intermetallic according to 2θ angle and annealing temperature of the ramp 10 °C/min for Ni(20 nm)/InGaAs/InP sample

different orientations (more information in [2][3]). The 3D-RSM measurements were mandatory since they allowed us to get accurate kinetic evolution thanks to the reduced time of measurements coupled simultaneously with texture information and micro-structural parameters acquisition. In the second part of the campaign, the as-deposited samples were analyzed during annealing up to 550 °C and using a Kissinger methodology with several ramps approximately between 1°C/min and 15°C/min. Fig. 3 shows the color map of the evolution of the intensities of (a) 10-11 (b) 10-12 intermetallic according to 2θ angle and annealing temperature. All these fruitful results will be soon presented in the MAM 2017 conference (March 2017) and consequently in a peer-reviewed publication.

4) Perspectives:

Thanks to this campaign and the previous campaigns, we were able to perform structural and texture analysis on intermetallics formed by solid-state reaction of Ni (and NiCo) on $In_{0.53}Ga_{0.47}As$. These investigations have shown that the texture, structure and composition of these intermetallics depending on the annealing temperature, on the substrate and on the metal stoichiometry. Moreover, the intermetallic orientation and lattice parameter depend on the lattice parameter of the $In_{0.53}Ga_{0.47}As$ substrate. With the help of much faster scans, we might be able to accelerate the whole process and observe the phase changes more precisely. In the future campaigns, we aim at going to thinner samples and with gratings which have many industrial benefits.

5) References:

[1] T. T. Nguyen, et al.(IITC/MAM), 2015 IEEE International. IEEE, 2015.
[2] Zhiou, S., et al. Journal of Applied Physics 120.13 (2016): 135304.
[3] Zhiou, S., et al. (IITC/MAM), 2015 IEEE International. IEEE, 2015.

Justification and comments about the use of beam time (5 lines max.):

The use of synchrotron source is necessary to observe the diffraction signals on these ultra-thin samples which are otherwise difficult to measure on laboratory diffractometers. More importantly, we were able to perform *in-situ* study of the SSR of Ni on InGaAs by using furnace which was adapted to the Kappa goniometer of the synchrotron.