ESRF	**Experiment title:** **In situ kinetic study of the solid-state reaction between an ultrathin NiCo film and an InGaAs/InP substrate**	**Experiment number**: **02-02- 827**
Beamline: BM02	**Date of experiment**: from: 26-11-2015 to: 30-11-2015	**Date of report**: 10/02/2016
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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 26th of November to the 30th of November and it is the third 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 $In_{0.53}Ga_{0.47}As$. 20 nm of Nickel Cobalt thin film 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.

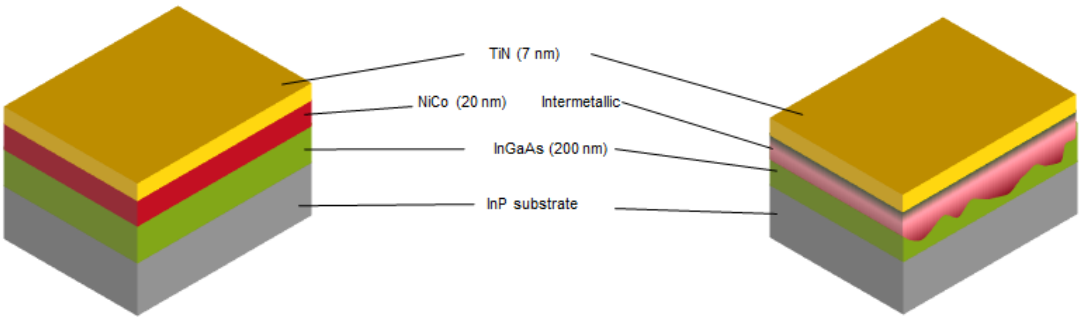

Figure 1. (a) Scheme of the as-deposited stack: TiN/NiCo(20 nm)/InGaAs/InP (b) Scheme of the sample annealed at T>250 °C

Experimental setup and methodolgy:

The intermetallic exhibits a strong monocristalline texture. Thus, a global 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 beam line equipped with a 6-circles goniometer (Kappa geometry) and a 2D pixel detector (CdTe based 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 followed by N_2 injection to evacuate residual air in it. The detector was fixed at 30° 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 (Spec) controlling the goniometer.

Due to our previous experience with Ni-based intermetallics we choose to do measurements only over a 90° range of φ angle positions . This is choice was justified by the hexagonal symmetry of the intermetallic which can be deduced from only ¼ 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. 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 LaB_6 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.

Several isothermal annealing temperature were put in place in order to extract the activation energy for the formation of the intermetallic (typically 205 °C, 210 °C, 215 °C and 220 °C).

First day has been dedicated to the furnace setup, annealing and sample/beam alignements. We spent the next 3 days doing the annealing isothermal measurements.

Preliminary results:

While we found multiple phase transitions according to annealing temperature after a ramp annealing that has been done during our last campaign for the same sample stack (see Figure 2), the isothermal annealing of these same samples gave rise to only one phase in the case of isothermal annealing (see Figure 3).

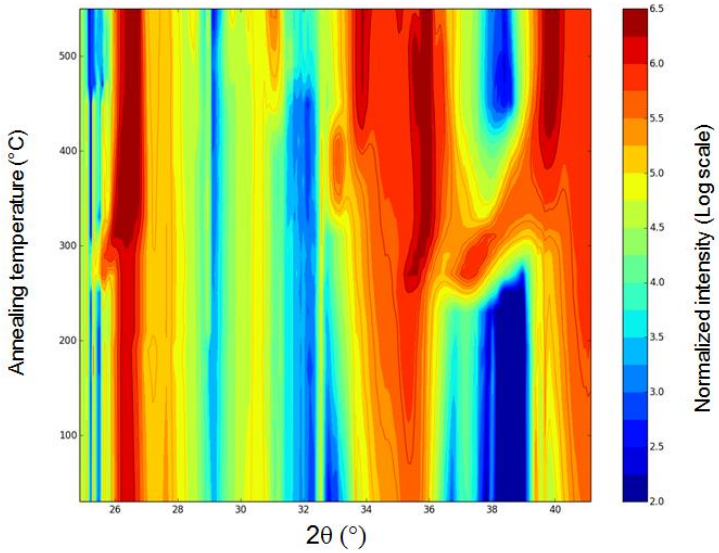

Figure 2. "Detextured" temperature vs 2θ map of a NiCo/InGaAs sample annealed by ramps of 20°

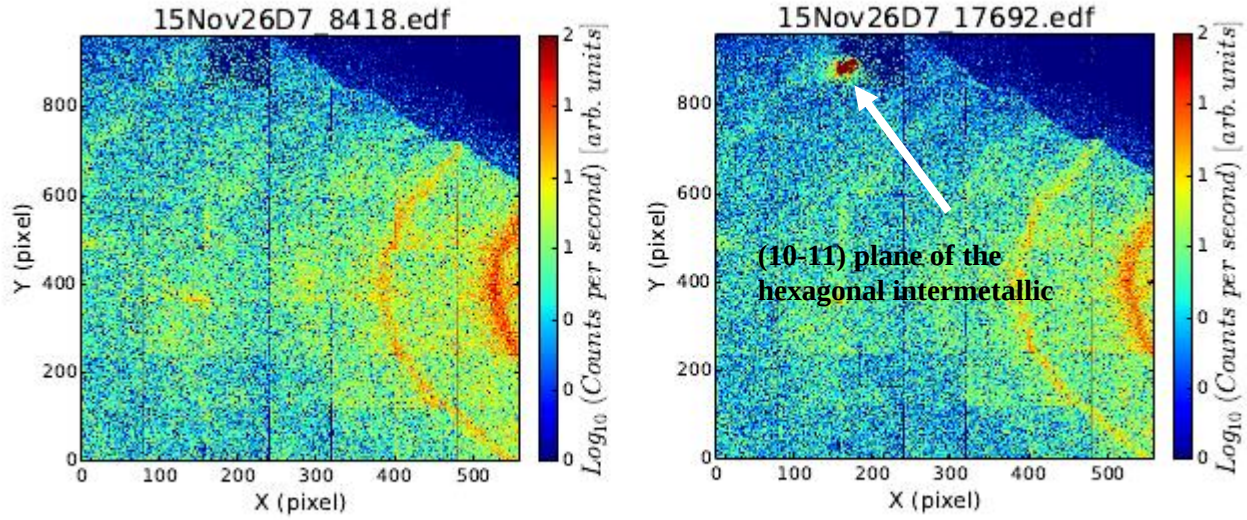

Figure 3. (a) Raw diffraction image of the sample before annealing at φ=-197° (b) Raw diffraction image of the sample after 1 hour of annealing at 215 °C

Based on the position (χ,φ) of the diffraction spot of the (10-11) plane, we conclude that this unique phase exhibits the following orientation :

$(10\text{-}11)_{NiCo\text{-}InGaAs}//(100)_{InGaAs}$ and $[0001]_{NiCo\text{-}InGaAs}//[0\text{-}11]_{InGaAs}$

The complete image correction and treatement will ultimately lead us to extract the activation energy for the formation of the NiCo-InGaAs intermetallic.

Perspectives:

Throughout the last campaigns, we were able to perform structural and texture analysis on intermetallics formed by solid-state reaction of Ni or NiCo on $In_{0.53}Ga_{0.47}As$. These investigations have shown that the texture, structure and composition of these intermetallic depend on the annealing temperature, on the substrate and on the metal stoechiometry (Ni vs Ni(Co)). Moreover, the intermetallic orientation and lattice parameter depend on the lattice parameter of the $In_{0.53}Ga_{0.47}As$ substrate (work to be published promptly). Therefore, it is interesting to investigate different Indium composition in the substrate in order to monitor the intermetallic orientation evolution and the possibility to control it.

References:

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