	Experiment title: **Texture measurements on ultrathin Ni-based germanosilicide in bottom of trenches**	**Experiment number**: 02-02-814
Beamline: BM02	**Date of experiment**: from: June, 18th 2014 to: June, 23rd 2014	**Date of report**: September, 1st
Shifts: 15	**Local contact(s)**: Tra Nguyen Thanh, Nathalie Boudet	*Received at ESRF:*

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Report:
Introduction

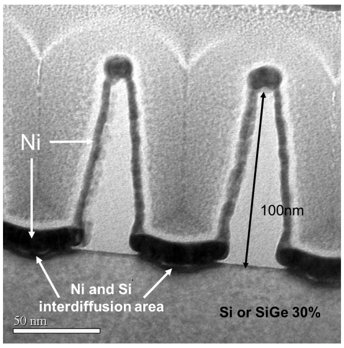

Fig.1 : TEM cross-section of 11nm thick Ni deposited at the bottom a 1/3 aspect ratio trenches with 90nm in pitch.

In microelectronics, silicides are used to decrease access resistance and increase the speed of complementary metal–oxide–semiconductor (CMOS) transistor [1]. Silicidation process arises from sequential metal/semiconductor reactions. Indeed, under two thermal budgets separated by unreacted metal removal, metal diffuses into semiconductor to form successively metal rich silicide and monosilicide. Monosilicide features depend on metal rich silicide. Thus, growth mechanisms must be understood in terms of kinetics, phase sequence, element incorporation or distribution and texture evolution in order to accurately define silicide process [2,3]. Our work fits into the industrial development of the next Ultra Thin Box and Buried Silicon-On-Isolator (UTBB SOI) CMOS generations for the 14 nm and 10 nm nodes. Such aggressive dimensions require the use of original materials and very low pitch patterns, which imply new challenges and new unknowns to overcome. FDSOI architecture is the best candidate to improve significantly electrostatic gate control. $Si_{1-x}Ge_x$ source and drain will replace Si to strain the channel and thus improve significantly carriers mobility [4]. Thus, Ni based germanosilicidation will be carried out in the bottom of 1/3 aspect ratio trenches (Fig. 1). For the first time, innovative $Ni_{0.85}Pt_{0.15}$ alloy will be used for germanosilicidation. The main aim of our study is the evaluation of the confinement impact on phase texture and thermal stability as function of Pt content. The final interest is to determine how germanosilicidation with Ni-based alloys acts on CMOS performances through stress measurement, phase and texture determination in high aspect ratio trenches.

A first campain in June 2013 was dedicated to the characterization of Ni rich germanosilicide formed after the first rapid thermal anneal (Fig. 2). We demonstrated that the pitch size changes the phase sequence

[5]. To complete this work, we dedicated the second campaign, occurred between June, 18th and 23rd, to the study of the mono-germanosilicide texture after the application of a second thermal budget (390 °C – 60 sec) as explained in Fig. 3.

Samples description

We chose the D2AM beam line for its large capability to explore samples in every cristallographic directions by XRD thanks to the newly upgraded optics, the 6-circles goniometer coupled with an XPAD detector. During this campaign, we focused the characterizations on the texture of the Ni based germanosilicide in the bottom of very tight trenches. To succeed this objective, we were able to product very tight patterned trenches thanks to e-beam lithography, as shown in Fig. 1. The most aggressive grating trenches measure 64 nm in pitch, 32 nm in line width and 100 nm in depth in 600*600 µm² areas. With the latest 300 mm RF-PVD generation equipment, ultrathin films with high bottom coverage were deposited into the trenches. Thus, 7 nm thick Ni or NiPt(15 at.%) alloy capped with 7 nm of TiN layer were deposited. As explained in Fig. 2, silicidation was performed using a RTA annealing system at 280 °C. During this thermal process, the solid state reaction occurred only between Ni alloy and $Si_{0.7}Ge_{0.3}$. The germano-silicide is only present in bottom trenches. Finally, a hot chemical selective etch have been carried out with a 300 mm state of the art equipment. This process aims to remove the TiN capping layer and the unreacted Ni-based metal selectively with respect of Ni rich germano-silicide compound. Finally, a second RTA at 390 °C during 60 sec was applied to transform the Ni rich phases into mono-germanosilicide.

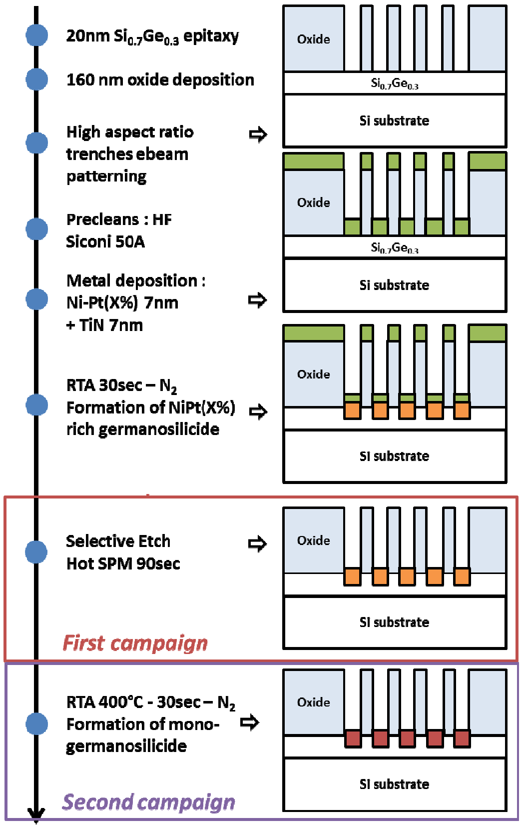

Fig.2 : Germanosilicidation process into bottom trenches

In order to extract a trend of the Ni germanosilicide texture as a function of pitch, pitch size was ranged from 1 µm to 64 nm. Ni and NiPt(15 at.%) were used to extract the impact of Pt on germanosilicidation. It is noteworthy that such samples are therefore representative of what occurred on CMOS source and drain. The pitch sizes addressed here belong to several generation nodes, scaled down to the FDSOI 10 nm agressiveness.

Experimental setup and method

The D2AM beam line was equipped with new optics, a new 6-circles goniometer (Kappa geometry) and a new 2D pixel detector (XPAD). A fluorescence detector was added for both sample alignement and sample analysis. For this experiment, the beam energy was set at 8.2 keV. Beam size has beem optimized at 100 x 150 µm² to fit in the beam footprint with the die shortness . Mesurements were carried out in reflection, at 20° in incident angle. The detector was fixed at 40 ° and at 11 cm from the sample (> 20 ° aperture range). To explore a larger reciprocal space, sample was tilted at 0° and 45° in χ angle and Φ angle rotates from 180 ° to - 180 ° around the normal to the surface. In this configuration, several Debye-Scherrer rings were recorded on the same picture with a 30 sec exposure time. Thus, the acquisition time is considerably reduced compared to standard θ - 2θ measurement.

On the whole, 2 days were dedicated to the configuration setup adjustements and to the troubleshooting linked to the measurement setup, the alignement method and the samples themselves. The main issue appeared to be the sample alignment. Indeed, a very accurate alignment between the very small die, the center of the goniometer and the X-ray beam was requiered during the Φ rotation at χ = 0° and 45°. Eventually, we were able to launch macros to fairly increase measurement number.

Results

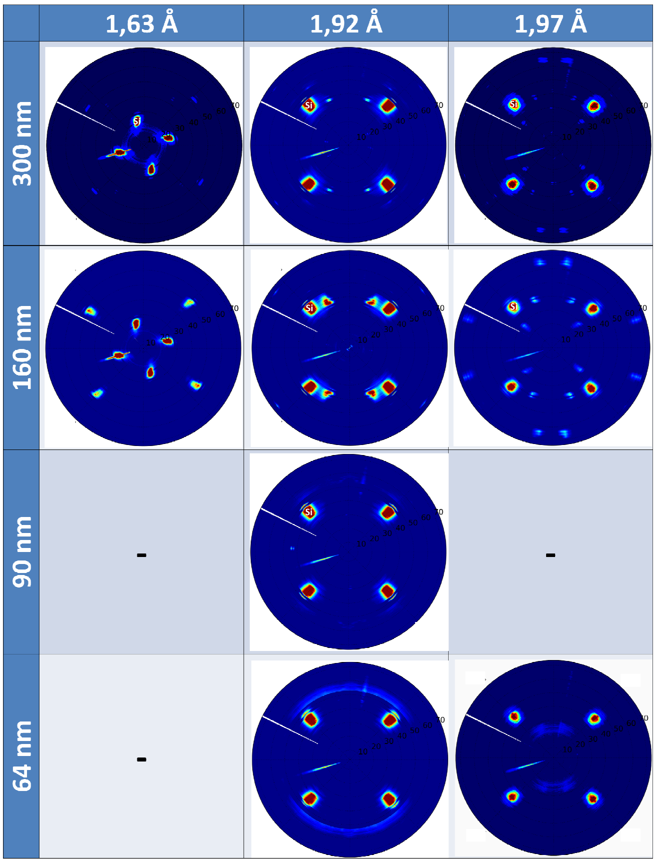

Fig.3 : Pole figures of Ni based samples, annealed at 390 °C during 60 sec, plotted as a function of interplanar spacing and pitch size.

This first campaign was very profitable. With the help of Tra Nguyen, postdoctoral fellow on the beam line, we were able to plot pole figures for all of our samples with a home made software Deva (D2AM EDF images Visualisation and Analysis). This software written by Tra Nguyen is serving for online analysis of data acquiered from area detectors (XPAD) available at the BM02 beamline.

One of the main results is presented in Fig. 3. Pole figures of Ni based samples annealed after the second RTA at 390 °C are plotted as a function of pitch. For all pitches, the monogermanosilicide is formed as indicated by interplanar spacing. But, the texture clearly evoluates. Indeed, for larger pitch, the monogermanosilicide grows in epitaxy with the substrate. Whereas a fiber texture is observed for the narrowest pitch. These results demonstrates the influence of pitch size on the texture of mono-germanosilicide and are in adequation with the first campaign results.

Perspectives and conclusions

This campaign were dedicated to the characterization of Ni germanosilicide formed after the first thermal budget to complete previous results obtained after the first RTA. The investigations were particularly fruitful since we demonstrated the impact of pitch size on the growth of mono-germanosilicide. Indeed, texture changes with the pitch size which could impact the agglomeration process. BM2/D2AM revealed to be perfectly well suited for this kind of experiment with such samples.

As perspectives, we will investigate the morphology of these mono-germanosilicides by TEM scheduled in CEA-LETI, as well as atom probe tomography (APT) performed in IM2NP. Indeed, atom diffusion and redistribution in nanoscale samples could be analysed with this additional technique that is able to determine the 3D composition at the atomic scale and is particularly adapted to silicide analysis [6].

This experiment is innovative and unique in terms of studied materials, thicknesses, and patterns. Thanks to these studies, we expect to appreciate the confinement and additive element on the agglomeration of the germanosilicide. By comparing our data with atom probe and TEM results, the role of alloying elements should be clarified. With these results, the mechanisms of formation and degradation of the germano-silicide will be better understood and will help us to control germano-silicidation in its industrial integration. These results were or will be published in :

- E. Bourjot, T. Nguyen Thanh, et al., “Synchrotron texture analysis on ultrathin Ni-based germanosilicide in bottom of trenches: application to pMOS 14nm UTBB SOI”, MRS Spring 2014, (Oral presentation)
- E. Bourjot, La germanosiliciuration à base de Ni et NiPt(15 at.%), PhD report (to be published)
- T. Nguyen Thanh, E. Bourjot, F. Nemouchi et al.: “Strain state investigation of SiGe nanolayers in trenched patterns after germano-silicidation for sub-14 nm FDSOI applications” – XTOP 2014 (poster).

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