	Experiment title: **Texture measurements on ultrathin Ni-based germanosilicide in bottom of trenches**	**Experiment number**: 02-02 805
Beamline:	**Date of experiment**: from: June, 26th 2013 to: July, 1st. 2013	**Date of report**: August, 27th
Shifts:	**Local contact(s)**: Nils Blanc, Nathalie Boudet	*Received at ESRF:*

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Report:

Introduction

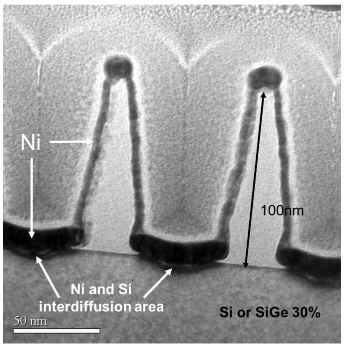

Fig.1 : TEM cross-section of 11nm thick Ni deposited at the bottom a 1/3 aspect ratio trenches with 90nm in pitch.

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. 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, kinetics of phase formation 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.

This campain, occurred between June, 26th and July, 1st 2013, is the first part of the series which will tackle with this large topic. 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.

Samples description

During this campaign, we focused the characterizations on the texture of the Ni-rich 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 μm^2 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 NiPt(X%) alloy capped with 7 nm of TiN layer were deposited. (X = 0, 10 and 15%). As explained in Fig. 2, silicidation was performed using a RTA annealing system at two different temperatures : 220°C and 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.

In order to extract a trend of the Ni rich germanosilicide texture as a function of Pt content and pitch, Pt content in the deposited alloy has been ranged from 0 % to 15 % and pitch from 1 µm to 64 nm. 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.

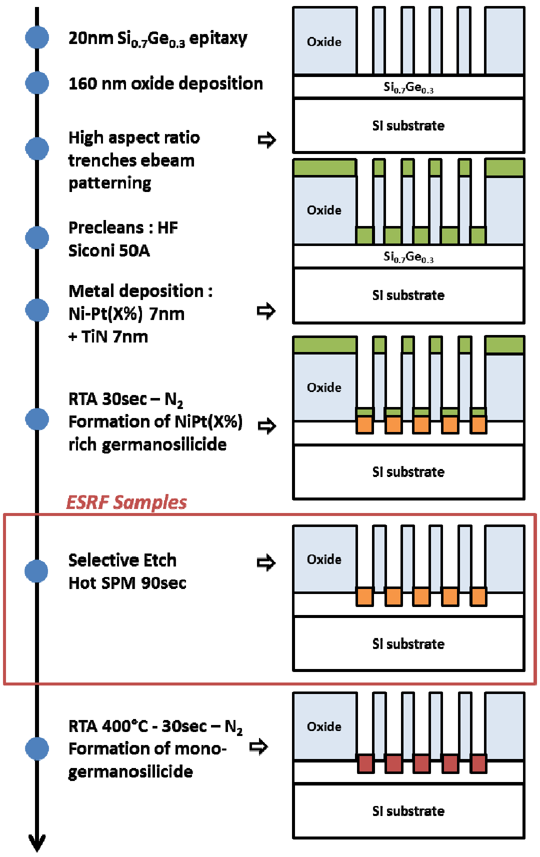

Fig.2 : Germanosilicidation process into bottom trenches

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 μm^2 to fit in the beam footprint with the die shortness . Mesurements were carried out in reflection, at two incident (7 ° and 20 °). The detector was fixed at 40 ° and at 11 cm from the sample (> 20 ° aperture range). 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. To explore a large part a reciprocal lattice, Φ angle rotates from 180 ° to - 180 ° around the normal to the surface.

On the whole, 2.5 days were dedicated to the configuration setup adjustements and to the troubleshooting linked to the measurement setup, the alignement method and the samples themselves. Firstly, the main issue appeared to be the reduction of noise. It has been improved by adding lead covers. The second difficulty was the need of a very accurate alignment between the very small die, the center of the goniometer and the X-ray beam during the Φ rotation. As Ni is only present in the trenches, the beam energy has been tuned at 8.4 keV to record Ni fluorescence. Thus, a precise alignment method was developped and systematically executed before the analysis. Eventually, we were able to launch macros to fairly increase measurement number in the last days of the campaign.

Results

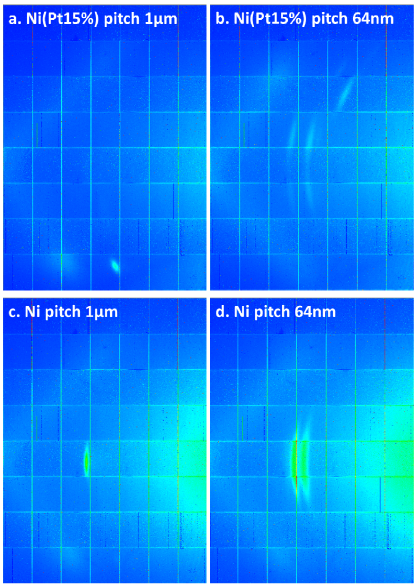

Fig.3 : Changes in particuliar textures observed with NiPt(15%) sample in pitch 1 µm (a.) and 64 nm (b.) respectively and with Ni sample in pitch 1 µm (c.) and 64 nm (d.)

This first campaign was very profitable. Despite 220 °C reflowed samples did not diffracted, because of their very thin thickness, 280°C annealed samples gave publishable results. Indeed, for the first time with these materials in such tiny pitch trenches, a significant change in texture has been highlighted as the function of pitch and Pt content, as shown in Fig. 3. From an epitaxial texture in the largest pitch, the Ni rich germanosilicide film switches to axiotaxy [5] in the narrowest pitch. The Pt content promotes the epitaxial texture in small patterns, but the effect seems to be lost with pitch size reduction. At this moment, results are still under further exploitation. Phase identification and pole figures will be extracted for a coming publication. BM2/D2AM revealed to be perfectly well suited for this kind of experiment with such samples.

The last day, we tried fluorescence measurement on a die to quantify the content of Ge, Ni and Pt. Indeed, in the litterature [6,7], the incorporated amount of Pt into silicide during the first steps of the process is still under discussion. For this first try, energy beam was tuned at the Pt threashold (11.7 keV) to record Ge (Kα), Ni (Kα) and Pt (Lα) emission ray at 9.886 keV, 7.478 keV and 9.442 keV respectively, showed in Fig.4. This 3 peaks are well defined and the method is easy to achieve. This item could be adressed in the next proposal to clarify the redistribution of Pt whithin the mono-germanosilicide.

Perspectives and conclusions

This first campaign were dedicated to the characterization of Ni rich germanosilicide formed after the first thermal budget. To complete our work, we would like to study the mono-germanosilicide texture formed from the Ni rich germanosilicide by applying a second thermal budget (400 °C – 30 sec), as showned in Fig.2. We are particularly interested in exploring the impact of Ni rich germanosilicide texture on the mono-germanosilicide formation. Then, we will focus on phases and texture in degraded germanosilicides (after high thermal budget) in order to investigate the relation between texture and film agglomeration. A second item should be related to the quantification of Pt content incorporated in the germanosilicide by fluorescence. The results of some samples will be compared to TEM scheduled in CEA-LETI, as weel 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 [8].

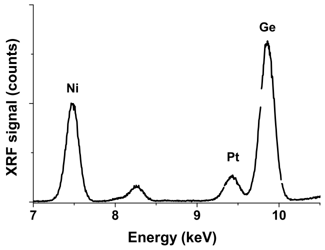

Fig.4 : X-ray spectrum of NiPt15%-germanosilicide in 1µm pitched trenches.

This experiment is innovative and unique in terms of studied materials, thicknesses, and patterns. Moreover, it gave publishable results. 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.

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