

## Experiment Report Form

**The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.**

Once completed, the report should be submitted electronically to the User Office via the User Portal:

<https://www.esrf.fr/misapps/SMISWebClient/protected/welcome.do>

### ***Reports supporting requests for additional beam time***

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

### ***Reports on experiments relating to long term projects***

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

### ***Published papers***

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

### **Deadlines for submission of Experimental Reports**

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

### **Instructions for preparing your Report**

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

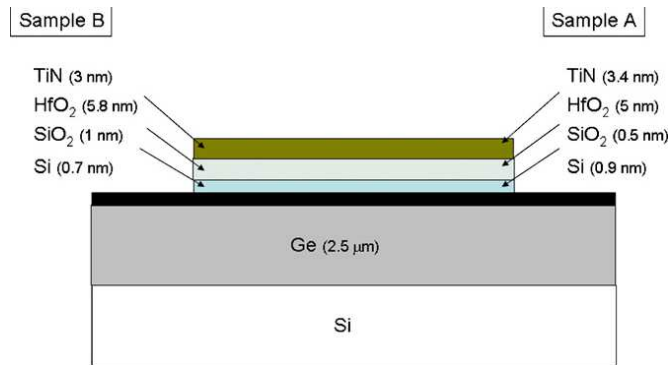


	<b>Experiment title: HAXPES to investigate the impact of buried interfaces chemical properties on advanced metal gate/high-k stacks</b>	<b>Experiment number:</b> 25-02-647
<b>Beamline:</b>	<b>Date of experiment:</b> from: 29/10/2008 to: 04/11/2008	<b>Date of report:</b> 27/02/2013
<b>Shifts:</b>	<b>Local contact(s): J. Rubio-Zuazo</b>	<i>Received at ESRF:</i>
<b>Names and affiliations of applicants (* indicates experimentalists):</b>  <b>E. Martinez, CEA-LETI, Minatec campus, 17 rue des Martyrs, 38054 Grenoble, france</b> <b>P. Batude, CEA-LETI, Minatec campus, 17 rue des Martyrs, 38054 Grenoble, france</b>		

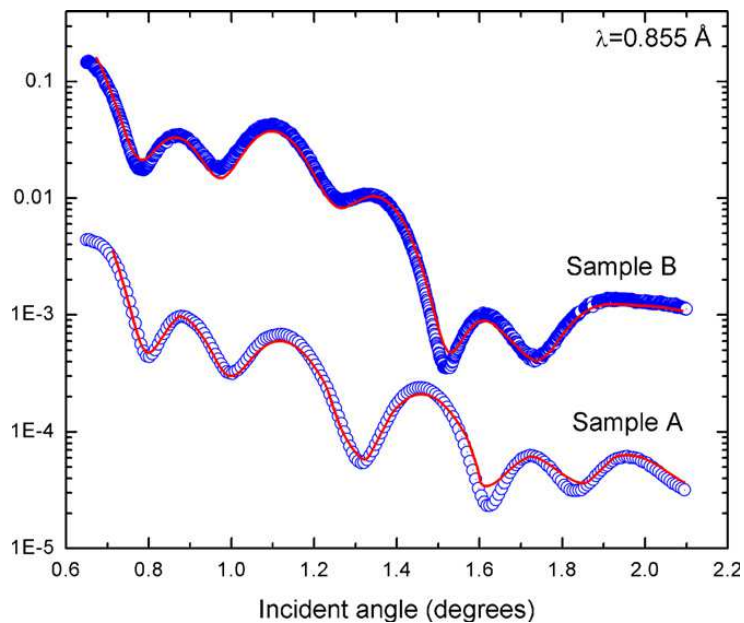
## Report:

The goal of the experiment was to perform accurate, non-destructive determination of depth concentration profiles with HAXPES (2-12 keV) on advanced CMOS structures deposited on high mobility substrates such as Ge. During the beamtime, two samples having the structure described in Fig. 1 (Ge/Si/SiO<sub>2</sub>/HfO<sub>2</sub>/TiN stack) have been studied. The use of electrons with kinetic energies from few eV up to 15 keV enables to tune the information depth being able to analyze the desired interface in a non-destructive way. XRR enables the determination of the exact layer thickness and density (see Fig. 2).

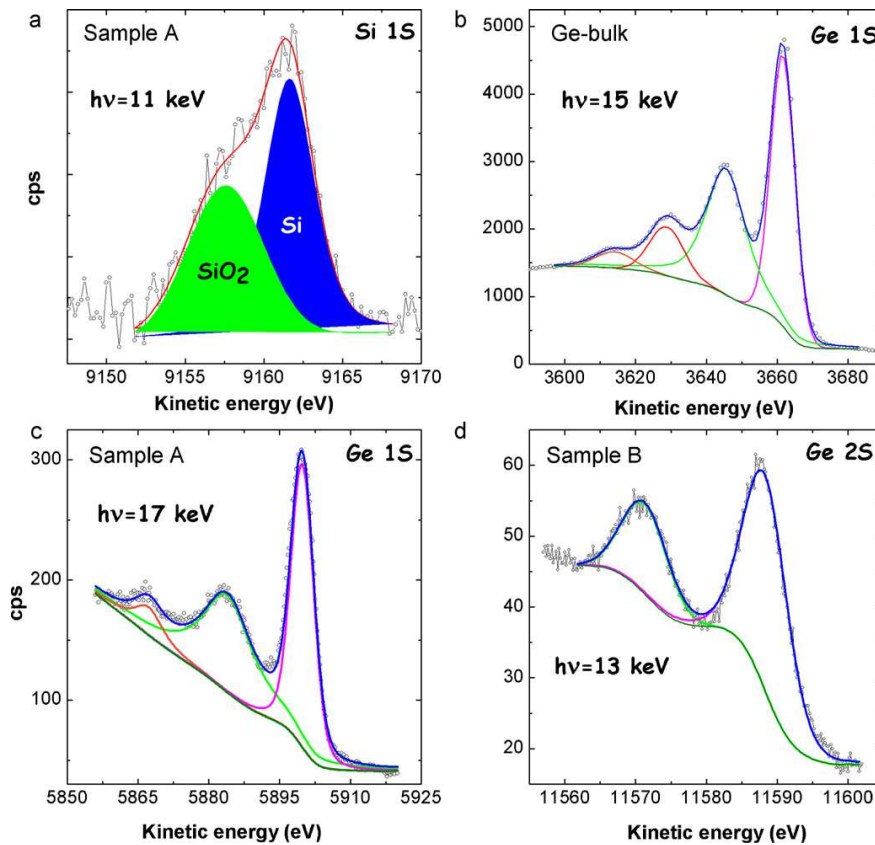
The results suggest that the Si interlayer prevents the Ge oxidation (see Fig. 3). Depth profiles of the electronic structure have been obtained for both samples by following the evolution of the photoemission signal from the Hf 2p<sub>3/2</sub> core level as a function of the photoelectron kinetic energy (see Fig. 4). The depth profile of the electronic structure reveals the presence of a chemical shift of the Hf 2p<sub>3/2</sub> core level, which is related to an interfacial bonding state. Our results demonstrate the excellent capability of HAXPES to study buried interfaces in a non-destructive way.



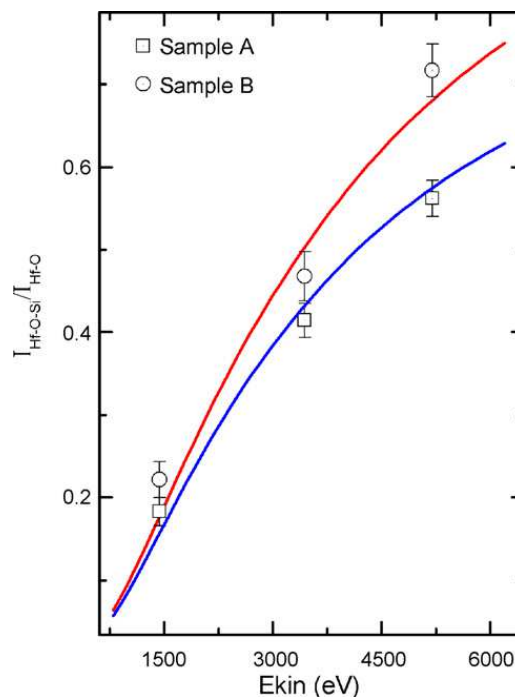
**Fig. 1.** Schema of the analyzed CMOS. The final structures of interest are composed of a stack of thin layers in which the relevant interfaces are buried by a several nanometers thick overlayer. The two samples studied in the present work are composed of a Ge/SiO<sub>2</sub>/HfO<sub>2</sub>/TiN stack with thickness of 2500, 0.9, 0.5, 5.3.4 nm and 2500, 0.7, 1, 5.8, 3 nm, respectively, as obtained from the X-ray reflectivity measurements.



**Fig. 2.** X-ray reflectivity measurements on sample A and sample B. The experimental data are represented by the open circles while the solid lines represent the corresponding fit. The exact thickness and roughness of each layer forming the stack are obtained from the fit to the Kiessig fringes. An error function has been used for the roughness determination.



**Fig. 3.** Representative HAXPES spectra for sample A, sample B and a Ge-bulk sample. (a) Si 1s spectra for sample A taken at a photon energy of 11 keV, which corresponds to an electron kinetic energy of 9 keV. The extremely high sensitivity of HAXPES enables the discrimination between Si and from a SiO<sub>2</sub> monolayer buried at depths 8.5nm and 8 nm, respectively. Representative Ge 1s and 2s spectra taken at a photon energy between 11 keV and 17 keV, which corresponds to an electron kinetic energy range between 3.5 keV and 11.5 keV, are given for Ge-bulk (b), sample A (c) and sample B (d). The large penetration depth of high kinetic energy electrons enables the analysis of the Ge substrate buried by 10nm thick layers for sample A and sample B. No trace of Ge oxide is present on the spectra.



**Fig. 4.** Ratio between the peak areas of the Hf- O- Si and Hf- O components of the Hf 2p<sub>3/2</sub> photoemission spectra (Fig. 5) for sample A (squares) and sample B (circles). The solid line represents a fit to the experimental data using the model depicted in the following reference: J. Rubio-Zuazo at al., Appl. Surf. Sci. 257, 3007 (2011).