# Experimental Report

Proposal:	<b>DIR-118 Council:</b> 10/2012
Title:	nfluence of plasma treatment on low K materials within the framework of the IRT nanoelectronics
This proposal is Researh Area:	new proposal
Main proposer:	LEPINAY MATTHIEU
Experimental To	m: LEPINAY MATTHIEU IMBERT GREGORY SEGURA RUIZ Jaime Alberto
Local Contact:	GUTFREUND Philipp
Samples:	SiOCH Cu Ta TaN
Instrument	Req. Days All. Days From To
FIGARO	1 1 12/06/2013 13/06/2013
Abstract:	

## **Experiment DIR-118**

### 1.1.1 Description of the industrial request

ST Microelectronics has an active research in low-k porous dielectric materials. They were interested in characterizing the porosity and composition changes suffered by low-k porous dielectric materials during the plasma etching, chemical-mechanical polishing (CMP), NH<sub>3</sub> de-oxidation, and restoration processes. The changes on the composition and/or porosity of the low-k dielectric material are small and impossible to trace and quantify by using lab-based techniques.

### 1.1.2 Samples description and preparation

The set of samples characterized correspond to porous low-k dielectric layers on Si substrates, 50-100 nm thick, exposed to plasma etching, CMP, NH3 de-oxidation, and different restoration treatments. The original 300 mm diameter wafers were cut on pieces of 50x50 mm for their characterization.

Eigth samples were measured during this experiment: reference; with SiCN plasma etching; with SiCN plasma etching+ dHF; with SiCN plasma etching + CH4, with SiCN plasma etching + glyco; with CMP+NH3; with CMP+NH3 + restoration process No. 1); (with CMP+NH3 + restoration process No. 2. The samples did not undergo any sample preparation process before the experiments, as it was concluded unnecessary after the discussion with the R&D people from ST Microelectronics involved in this experiment.

### 1.1.3 Reflectometry Characterization

The reflectometry experiment was performed using the ILL instrument FIGARO. On the sample stage, a vacuum chamber with a good control of the pressure allowing the change of the sample atmosphere was installed. During the experiment, high resolution (2 angles) reflectometry data were acquired from dry and saturated samples, and fast (5-20 s) intermediate resolution (1 angle) reflectometry data were acquired during the incorporation of deuterated-solvents in the layers (kinetics). The experiment was done in 24 hrs.



Figure 1. Representative 2D reflectivity image.

After the experiment, data reduction and fitting of the reflectivity curves was done using LAMP-Cosmos and Motofit packages, respectively.

### 1.1.4 Main results and conclusions

There was a problem with the measurements due to the unexpected large curvature of the wafers. Due to this curvature, the 2D specular reflection was very broad, making difficult to extract the reflectivity information.

No differences were observed among the full set of samples characterized. This can be due mainly to two different possibilities. The technique is not able to trace the small changes induced on the porous material by the treatments analyzed. The second possibility is that due to the unexpected curvature of the wafers, the broadening of the reflection could hide the small changes arising from the processes the layers have undergone. Therefore, samples' straightening is mandatory.

#### 1.1.5 Recommendation(s) for future experiments

• Precise measurement of the wafers curvature before the experiment.

• Preparation of specific samples avoiding the wafer bending (porous layer deposited on both sides for instance. This possibility has been rejected by STM, as they are not allowed to change the standard processes done in the production lines.

• Preparation of a specific sample holder for samples straightening. This must be done in order to guarantee a flat sample surface in order to get high quality reflectometry data.