Experimental Report

15/05/2014

Proposal: **DIR-122** **Council:** 10/2012

Title: Influence of plasma treatment on Low K materials

This proposal is a new proposal

Researh Area: Materials

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Samples: SiOCH
Cu
Ta
TaN
Toluene

Instrument	Req. Days	All. Days	From	To
D17	1	1	06/08/2013	07/08/2013

Abstract:

Experiment DIR-122

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_3 de-oxidation, and restoration processes. The changes on the composition and/or porosity of the low-k dielectric material are small and very difficult to trace and quantify by using lab-based techniques. These small changes can be hidden by the wafers' curvature induced broadening of the reflectivity. This is what could happen in the experiment DIR-118 and the reason why that experiment worth to be repeated.

The second interest from STM for this experiment was related with another subject. They wanted to quantify the adhesion between an oxide layer and a carbon layer beneath it. No alternative methods using by using lab-based techniques are known for the reproducible quantification of the adhesion between a very thin oxide layer and the carbon one. The scotch test is widely used, but it is not reproducible and does allow a systematic comparison among a set of samples having slightly different adhesion characteristics.

1.1.2 Large Scale Facility characterization proposal

Specificity and high flux of neutrons at the ILL could allow the neutron reflectometry instruments to trace and quantify such small changes. Neutron reflectometry can measure the small changes in the density, thickness and roughness of layers in a heterostructure. Furthermore, thanks to the high sensitivity of neutrons to the presence of hydrogen, it is a very efficient technique to trace the solvents diffusion through porous layers. It is then possible to correlate changes in the diffusion velocity with changes in the material porosity.

Concerning the quantification of the adhesion between the two layers, neutron reflectometry allows studying the interface between the two layers. Any change in a particular characteristic of the interface (roughness, thickness and/or density of the transition region, etc.) and its strength, could be correlated with the adhesion between the layers. If this is possible, the ILL could define a systematic procedure to measure and quantify the adhesion between these two layers.

1.1.3 Samples description and preparation

The set of low-k porous dielectric layers characterized was exactly the same as the one used in the experiment DIR-118. On the other hand, the set of samples characterised for the adhesion subject correspond to carbon on Si substrates, 50-100 nm thick, with an oxide layer on top (1-10 nm thick) without any additional treatment. The original 300 mm diameter wafers were cut on pieces of 50x50 mm for their characterization.

The samples underwent a specific preparation process before the LSF experiments, as it was concluded necessary after the fail of the Exp. DIR-118 due to the wafers curvature. The wafers curvature was measured using a Fizeau interferometer, and then a specific sample holder was prepared for samples straightening. This must be done in order to guarantee a flat sample surface providing high quality reflectometry data

1.1.4 Reflectometry Characterization

The reflectometry experiment was performed using the ILL instrument D17. 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 on low-k porous dielectric layers, 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). For the experiment on layers adhesion, high resolution (2 angles) reflectometry data were acquired from dry samples. The experiment addressing these two subjects lasts for 24 hrs.

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

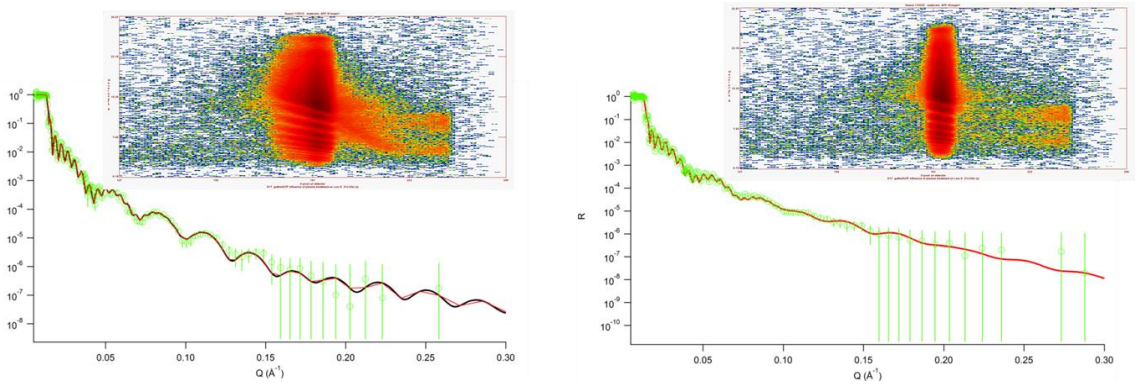

Figure 1. Reflectivity curves of the two samples studied to quantify the adhesion between the two layers.

1.1.5 Main conclusions

In the case of low-k porous dielectric layers, no differences were observed among the full set of samples characterised. This would indicate that this technique is not able to trace the small changes induced by the treatments on the porous layers analyzed.

In the case of the adhesion study, the results are quite promising. Clear differences were observed between the two samples analyzed. It was observed the appearance of a transition layer in between the oxide and the carbon layer. The characteristics (thickness, roughness, density) of this intermediate layer are quite different for the two samples studied. This means that the intermediate layer is correlated with the adhesion between the two layers.

1.1.6 Recommendation(s) for future experiments

- Precise measurement of the wafers curvature before the experiment.
- Perform a systematic study: increase the statistics and the number of oxidation processes analyzed. Include samples with different delamination percentage in qualitative tests like the scotch test, in order to quantitatively correlate the properties of the intermediate layer (thickness, roughness, or density) with the adhesion/delamination of the layers.