ESRF	**Experiment title:** GISAXS structural determination of ultra lowk porous dielectrics obtained through PECVD processes	**Experiment number**: MA-687
Beamline:	**Date of experiment**: from: Dec. 11th, 2008 to: Dec. 14th, 2008	**Date of report**: Feb 6th; 2009
Shifts: 12 (16b)	**Local contact(s): J.P. Simon*, M. Maret*, SIMaP, UMR 5266 CNRS-Grenoble-INP &UJF**	*Received at ESRF:*

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Frame work of the project :

Integrated circuits with improved functionalities and performances in a smaller package are studied in the research and development laboratories; one of the actual limitations is the cross-talk between wires through the dielectric barrier. In current devices, bulk SiOCH materials have dielectric constant (k) higher than 3. Pores may be introduced into the dielectric matrix to decrease k (Ultra Low K materials, ULK),but these nanometric pores have to be homogeneously distributed, both in size and position. The actual attempts with a pore volume fraction ~ 30% reach k values close to 2, but these interconnected and disordered ULK are not compatible with integration process in term of chemical absoption and mechanical properties.
The idea is to structure the porosity, both to achieve the best mechanical properties and to have closed pores. A Contract with the "Agence Nationale de la Recherche; Department of Nanomaterials" has been obtained in partnership between laboratories, LETI for the process and macroscopic characterisations and coordination of the project, IEM of Montpellier for the screening of the promising "silicium"-based polymer precursors and first characterisations and the SIMAP both for GISAXS and the simulation of the pore pattern together with nanoindentation and simulation of mechanical properties with the pore pattern.

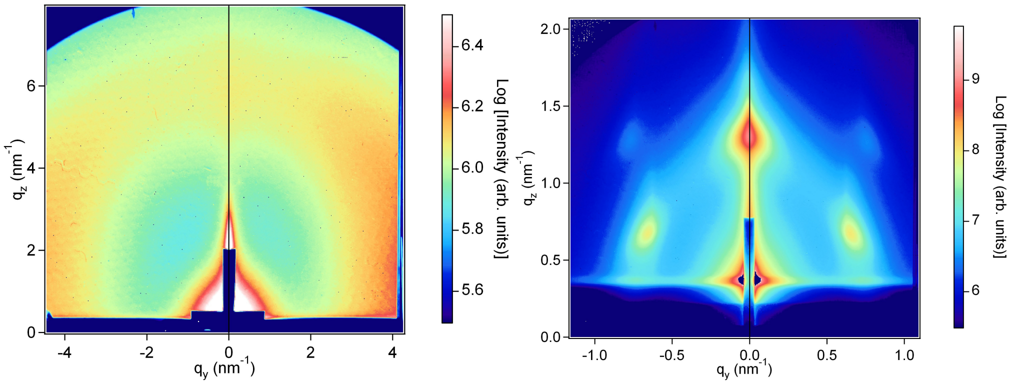
Experimental method and strategy:

The whole camera is under vacuum; two sample-detector distances have been used at 8.95keV, allowing to measure with the 2d-CCD @Princeton 16 bits camera a q range up to $8nm^{-1}$ at 350mm (SG) and in the case of the Long Geometry (LG=1350mm), down to $0.05nm^{-1}$, in the horizontal range. 95% of the beam flux is focused within a heigth (<0.1mm): This focus together with long samples (80mm) are such that the footprint of the beam remains well inside the sample and allow a comparative measurement of (integrated) intensities: using different grazing angle α_i above the critical angle of the Si buffer (0.2°), it was checked that the GISAXS intensity was proportional to the probed volume, ~$1/\sin(\alpha_i)$, i.e. to the footprint length of the beam.

First, we studied models systems such as mesoporous silica with well known pattern of pores (cylinders, honeycomb, monodisperse spheres in a regular pattern, ...), which is the "Skill" of the IEM Laboratory.
A TEOS silica gel was compared with three already studied pore patterns: disordered percolating micropores of size smaller then the nm, mesopores of several nm and submicronic macropores. Then two families of organosilates were studied BTSE, a blend of MTSE and TEOS as precursors in different amounts.. At this step, the samples were prepared by spin coating and three stages were selected, the as-prepared blend, the dried blend at 150°C and the calcinated skelet at 400°C.

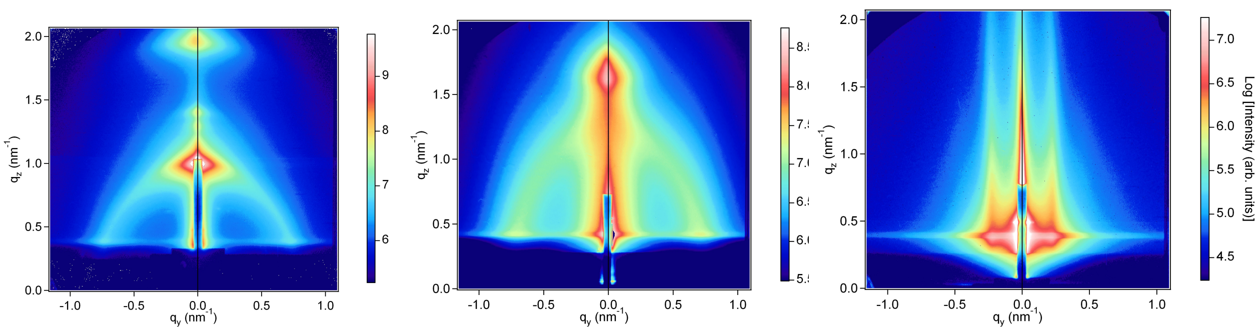
Results and preliminary analysis:

TEOS (silice):



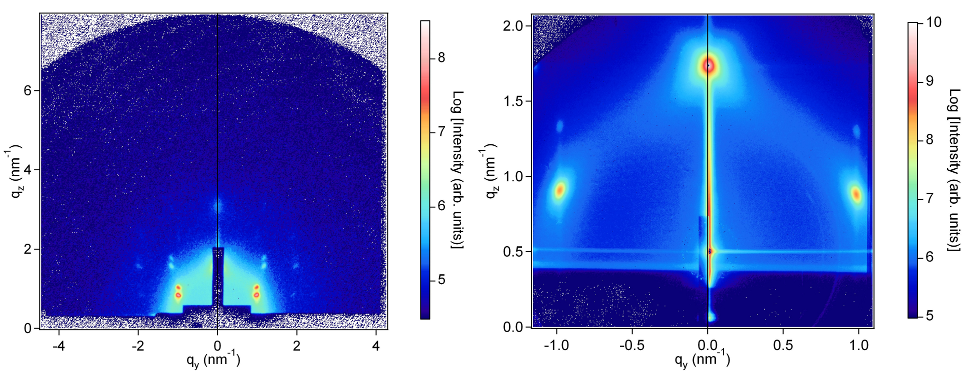
Left, micropores (a fit is possible with pores of 0.6nm of diameter with correlations with a mean distance of ~1nm). Rigth, mesopores (two lattices a square one at $0.7nm^{-1}$, i.e. d_{sq}=9nm, a rectangular one, $q_h=0.8nm^{-1}$, $q_v=1.25nm^{-1}$; i.e. d_{01}= 5nm; d_{10}=7.85nm)

Precursors: from left to rigth ***BTSE***:, as-prepared, calcined and ***MTSE-O*** calcined (Long Geometry)



In BTSE, there are pores in strates which interdistance decreases from 6.3nm to 4.9nm and a ring which looks like the correlation ring of amorphous patterns: a fit is possible with sheroids correlated with a volume fraction of 40% and an shrinkage of the layer (both ellipsoids and their interdistance) from 0.8 to 0.6. In MTSE-O, the pattern looks like pores well organized in the horizontal plane.

MTES 0.4/**TEOS** 0.6: (dried at 150°C, but all images from deposition to calcinated states are similar)



The left image has been recorded at a grazing angle in between the critical angle of the layer and the one of the buffer: the whole diffraction pattern is doubled, the two patterns being shifted by the angle between the incident beam and the one reflected on the buffer. More intense spots are on an "hexagonal" array, but there superimposes a rectangular spot array. The image does not depend on a rotation around the normal to the layer: the sample selects the domains which are in diffraction conditions with a mosaicity around the normal to the surface. The analysis has been undertaken.