

# Structure Characterization of Porous Interlevel Dielectric Films



To extend the dielectric constant of interlevel dielectrics below a value of ~2.6 seen in today's IC chips, porous low-k material has been evaluated as a viable candidate by industries. In this paper, the current status and the future need in metrologies for characterizing porous structure including porosity and pore size distribution are discussed.

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With continued miniaturization or rescaling of the integrated chip (IC), interconnects have become a potential bottleneck in chip integration and performance. Line resistance and interline capacitance control interconnect performance in terms of device speed, power consumption, and signal-to-noise ratio. The noise issue is expected to become more of a concern in the future as the chip voltage declines from 5 to 3.5 to 1.2. To lower the interline capacitance at nodes beyond 65 nm, and especially the 45 nm node, the consensus among experts is that porous interlevel dielectrics (ILD) are needed to replace CVD organosilicates used in current 90 nm node. Introducing porosity into ILD will lower its dielectric constant, hence the interline capacitance. However, it also weakens its mechanical strength and makes the integration a formidable task. In addition, porosity adds many other complications in the plasma etch, ashing, and barrier layer construction processes. In general, the porous structure tends to densify upon exposure to the etching plasma, and the chemical composition can be altered as well, often by loss of hydrogen and carbon atoms.

To generate sub-100 nm features in porous ILD with acceptable yield, one must have exacting control over the porous film structure. The first step toward achieving this control, before addressing material concerns, is to develop metrologies that quantitatively characterize the pore structure with accuracy in terms of porosity, average pore size, pore size distribution, and so on. The purpose of this note is to briefly review the state-of-the-art metrologies in measuring porous structure in ILD candidate materials. Most metrologies were developed within the last five years, and

they have been used offline by material suppliers, fabrication line equipment suppliers, and IC manufacturers. The ongoing work in searching for inline technique, especially the use of X-rays will also be discussed.

Structure characterization in bulk porous materials is a mature field. By measuring the pressure dependence of liquid or gas infiltration into the porous material one can determine both total porosity and pore size distribution.[1] There are several techniques, including gas adsorption, mercury intrusion, and mass uptake, capable of characterizing pores significantly smaller than 100 nm. However, these traditional methods lack the sensitivity to quantify porosity in thin porous ILD, which is typically a few hundred nanometers thick. The sample mass of typical films will be less than a few mg, meaning that the usual observables (i.e., pressure drop in a gas adsorption experiment or mass in an gravimetric experiment) exhibit exceedingly small changes as the pores are filled with the condensate. Thin ILD films require techniques with extraordinary sensitivity.

There are techniques suitable for the on-wafer characterization of the pore structures in low-k ILD films. These include scattering-based techniques utilizing either neutrons or X-rays,[2] positronium annihilation lifetime spectroscopy (PALS)[3,4] and ellipsometric porosimetry (EP).[5] Each of these techniques comes with strengths and weaknesses. PALS is well-suited for exceedingly small pores (1 nm to 20 nm in diameter) and capable of quantifying closed pores not connected to the surface. However, a PALS result is difficult to quantify in terms of an absolute pore volume. EP measures the refractive index and thickness of the film before and after being filled by condensate. To deduce porosity from

the refractive index, approximations such as additive polarizabilities are required. These two techniques also have deficiencies in characterizing samples with ordered porous structures, such as stratified or 3-D ordered ILD synthesized by mesophase template technique.[6] The X-ray and neutron scattering technique can be powerful; it applies a combination of specular reflectivity and scattering measurement and can address thin films with skin layers, stratified structure, and 3-D ordered ILD. However, it is limited by the access to neutron scattering facilities or a synchrotron X-ray source with high energy beam.

On the other hand, X-ray reflectivity (XR) can be routinely conducted using a laboratory X-ray source to determine film density profile, not just the average density, as well as the film thickness. Recently an XR-based porosimetry technique (XRP) has been developed[7] to measure pore size distribution and porosity without invoking the approximations used in EP. XR directly measures the film density profile, provided the film composition is known. During a gradually increasing or decreasing of the partial pressure of condensate vapors such as toluene in the presence of the film, the change in both film density profile and thickness are monitored using XR. The change in film density can be directly related to the amount of adsorbed condensate, and thus porosity, if the density of the condensed fluid is known or assumed to be bulk-like. Monitoring the amount of adsorbed condensate as a function of the partial pressure defines a physisorption isotherm, the basic starting point for any number of analytical interpretations in the well-known field of porosimetry. It is important to realize that XRP – assuming only that the density of the condense fluid is bulk-like – directly reveals the average porosity, average film density, and average wall density of the material separating the pores. By further invoking a model of condensation (for example, the Kelvin equation for capillary condensation), it is straightforward to calculate the average pore size and pore size distribution from the physisorption isotherm.

None of the aforementioned techniques can be applied as the processing monitoring tool in a fabrication line or inline. PALS requires high vacuum; EL and XRP invoke the infiltration of liquid into ILD; and X-ray and neutron scattering technique call for a massive beam source at the fabrication site. Recently, grazing incident small-angle X-ray scattering (GISAXS) has been explored as a viable technique for inline measurement. By aligning the incident X-ray beam a few tenths

of a degree from the ILD surface, the sampling volume increases by several hundred times in comparison to a normal incidence condition. This provides sufficient signal to be detected near the reflected beam, even with a laboratory X-ray source. Recent results demonstrate that GISAXS signals can be correlated semi-quantitatively with film porosity and pore size. This observation opens the possibility that GISAXS could be used as an inline quality control tool in the future, but many obstacles still remain. They include the difficulties in decoupling the surface roughness contribution from porous structure scattering and in delineating the complex evanescent wave existed in layered or ordered structure. Current theory in GISAXS is not yet adequate to address the aforementioned challenges.

All the above discussions have been focused on the characterization of blank or unpatterned porous ILD films. Upon plasma processing for pattern etching or photoresist ashing the porous structure will often be modified; in most cases densification occurs. For some cases, a limited and controlled densification at the sidewall of the patterns is desirable since it may facilitate the deposition of barrier layer on the sidewall. This points to the need of additional metrology to quantify the extent of sidewall densification. Work is in progress within NIST to develop metrology to address this type of porous structure measurement need.

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