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Analysis of Pore Structures in Ultra Low-k Dielectrics

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1. Introduction

As the research and development of interlayer dielectrics come to the "ultra" low-k regime, analysis of "pore" structures is getting more and more important. In the study we report here, complementary approaches were employed to analyze pore structures in ultra low-k dielectrics in an effort to obtain their complete picture through analysis of experimental results.

2. Porosity Determination

The porosity x is an important pore information. Porosity can be calculated from density ρ obtained from X-ray reflectance (XRR), refractive index n from spectroscopic ellipsometry (SE), or from dielectric constant k from capacitance (C-V) measurement of porous low-k dielectrics in the case when the corresponding values ρ_0, n_0, or k_0 for non-porous but otherwise the same dielectrics are known as:

$$x = 1 - r / r_0 \quad , \qquad (1)$$
$$x = 1 - [(n^2 - 1) / (n^2 + 2)] / [(n_0^2 - 1) / (n_0^2 + 2)], \qquad (2)$$
$$x = 1 - [(k - 1) / (k + 2)] / [(k_0 - 1) / (k_0 + 2)], \qquad (3)$$

respectively. It should be noted that the skeletal density of porous silica film we experimentally determined by XRR was 1.5 g cm^{-3}, only 68 % of the density 2.2 g cm^{-3} of thermal SiO_2 film. Density of a porous low-k dielectric determined by XRR (Fig. 1) of 0.81 g cm^{-3} gives the porosity x = 0.46 from eq. (1). The results from SE and C-V as well as from XRR are summarized in Table I.

On the other hand, nitrogen gas adsorption (GA) at 77 K provides the total volume of gas molecules which

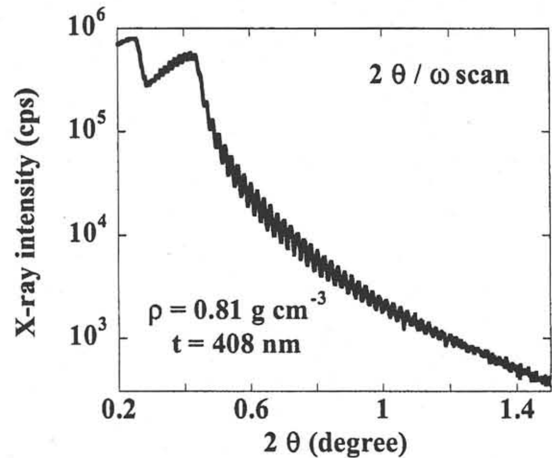

Fig. 1 X-ray reflectance from porous low-k.

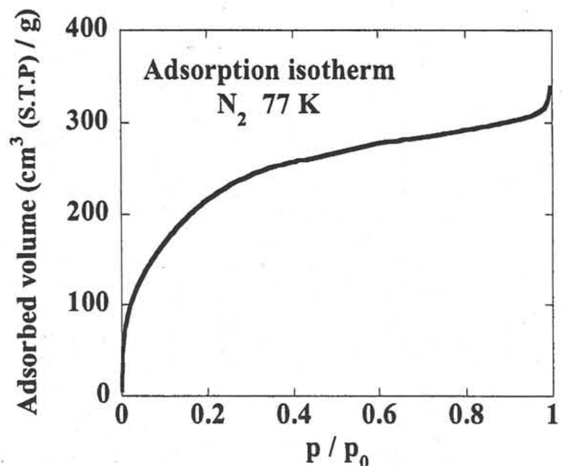

Fig. 2 Adsorption isotherm of porous low-k.

Table I Measured physical parameters, analysis techniques, measured values from porous low-k and non-porous reference films, and calculated porosities are compared for five techniques

Technique	Parameter	Analysis	Value	Reference	Porosity
XRR	Denisty	Eq. (1)	0.81 g cm^{-3}	1.5 g cm^{-3}	46 %
SE	Ref. Index	Eq. (2)	1.25	1.44	40 %
C-V	Diel. Const.	Eq. (3)	2.1	3.7	43 %
GA	Pore Volume	(Kelvin eq.)	0.47 cm^3 g^{-1}	none	38 %
Model	Pore Size	Guinier Plot	0.71 nm	none	48 %

is necessary for porosity determination. The porosity is given by multiplying the adsorbed gas volume per unit adsorbed into pores directly (Fig. 2), so that the information about non-porous counterpart is not sample weight by the density of the sample which we can determine by XRR. The result is also shown in Table I.

Focused ion beam thinning of porous low-k dielectric film down to 50 – 200 nm followed by observation by scanning electron microscope (STEM) resulted in an image of a pore structure [1]. The STEM image showed a cross section of hexagonally ordered structure which is asymmetrically compressed toward the thickness direction. The degree of this asymmetric compression is controllable (reducible) through preparation conditions [2]. Diffraction and scattering measurements of X-ray (XRD and XRS) provide the inter-pore spacing d and pore radius r, respectively (Fig. 3). The porosity x is then geometrically calculated from d and r, the result of which is in the last row of Table I. The porosity x determined from the five different experimental techniques in Table I is in the range of 43 ± 5 %. Possible origin of lower x from GA is now under investigation.

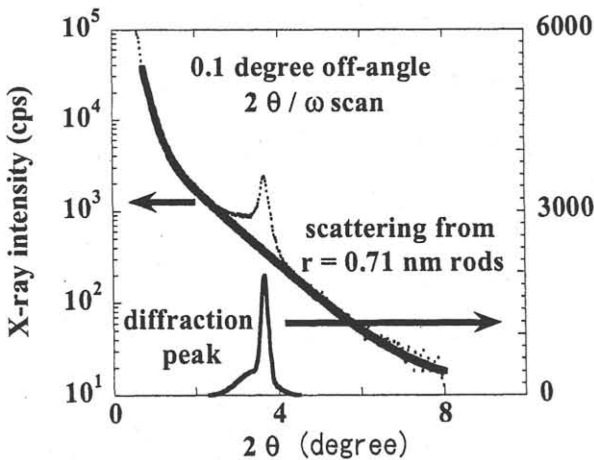

Fig. 3 X-ray diffraction and scattering.

3. Pore Size Distribution and Discussion

Pore size distribution (psd) is also important for low-k dielectric technology. That is because it will affect the compatibility of the material to integration processes such as chemical mechanical polishing, plasma deposition and etching, and wet cleaning processes. Dependence of relative pressure p / p_0 on the meniscus curvature (radius) described by the Kelvin equation was used to obtain psd from GA results [3], while best fitting of XRS data, after subtraction of diffraction component, with model psd functions was used for the analysis of XRS results. The psd from GA and XRS are shown in Fig. 4.

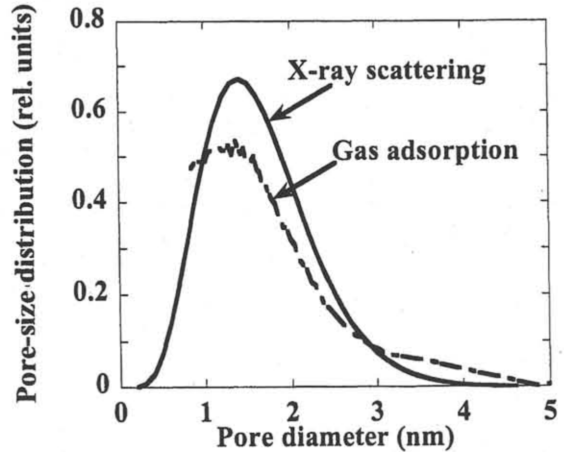

Fig. 4 Pore-size distributions from XRS and GA.

Infiltration of gas or liquid molecules into pore structures in ultra low-k dielectric is considered to be responsible for damages of low-k dielectric during integration processes. The damages include changes in dielectric constant, thickness, refractive index, mechanical hardness, or electric insulation performance upon wet or dry processes. Gas adsorption technique in which molecules infiltrating into pore structures are used as the probe potentially provides essential information regarding that important issue, while the combination of XRR, XRD, XRS, and other techniques has given a complete picture of the pore structure in ultra low-k dielectrics.

4. Summary

To summarize, seven measurement techniques of XRR, SE, C-V, GA, STEM, XRD, XRS have been employed to analyze pore structures in ultra low-k dielectrics, and the results on porosity and pore size distribution have been compared and discussed. It has been demonstrated that the combination of the techniques provides complete picture of the pore structure of ultra low-k dielectrics.

Acknowledgments

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