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“Incompressible” pore effect on the mechanical behavior of Low-K dielectric films

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ABSTRACT

New Low-K dielectric constant materials development is underway. Introducing porosity is one of the ways to lower the dielectric constant. Those are typically spin coated organic filled glasses. The pore size is on the order of a several nanometers and pore introduction compromises mechanical properties of low-K thin films, especially fracture toughness, as these materials are typically brittle.

In our previous studies we have evaluated different low-K dielectric constant materials in terms of their mechanical properties using nanoindentation. It was interesting to see that for a large range of various porous low-K materials the modulus-to-hardness ratio was constant. It was also found that the indenter contact is mostly elastic, as the loading and unloading portions of the load-displacement curve did not show any hysteresis, following indentation depth to the 3/2 power load dependence. Based on these results current analysis explains the observed constant modulus-to-hardness ratio.

The paper also describes the “incompressible” pore effect. As a particle gets smaller, the yield stress increases due to the Hall-Petch effect, but for the nanometer-size particles there are also high surface energy contributions that prevent is from deforming plastically. The same approach can be applied for a nanometer size pore elastic deformation, thus we call it an “incompressible” pore concept.

INTRODUCTION

Organo-silicate glass (OSG) is one of the novel low-K ($k<4$) interlayer dielectrics (ILD) to replace SiO_2 in modern microelectronic devices. One of the ways to reduce dielectric constant is by introducing porosity (Figure 1a). OSG is a nanoporous material with up to 50% porosity, and an average pore size on the order of nanometers. While decreasing dielectric constant, increasing porosity compromises films mechanical reliability. The four interrelated properties responsible for the mechanical reliability are fracture toughness, elastic modulus, hardness, and interfacial adhesion. In our previous studies we have evaluated different low-K dielectric materials in terms of their mechanical properties using nanoindentation [1-3]. It was observed that the elastic modulus correlates with hardness (Figure 1b) [2, 3]. Based on a classical porous media behavior [4], it was also proposed that nanoindentation elastic modulus measurements could be used for estimating film porosity [2]. This paper attempts to explain the observed constant modulus-to-hardness ratio measured by nanoindetation, and also considers the surface energy contributions to the nanoporous media elastic deformation during nanoindentation.

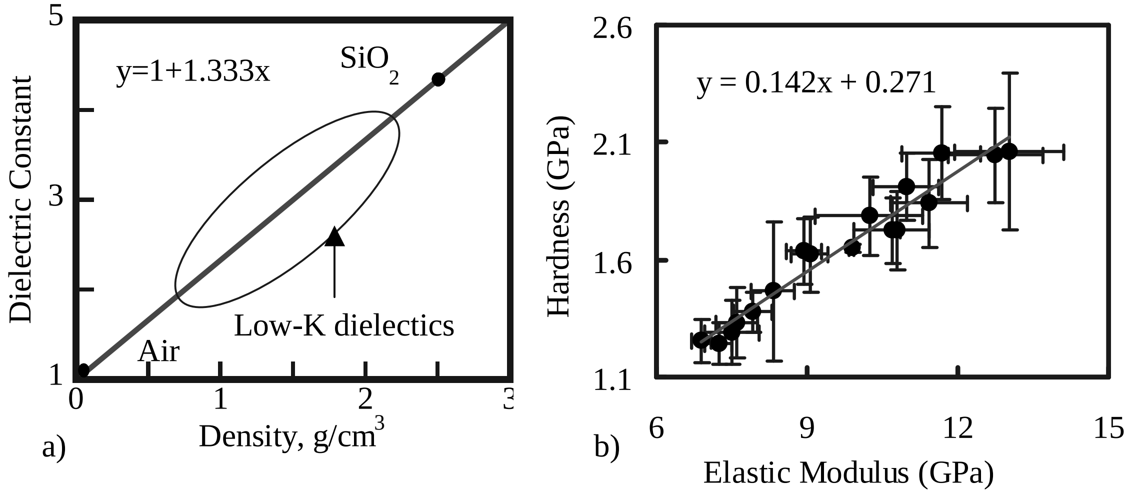


Figure 1. a) Dielectric constant as a function of low-K density; b) Elastic modulus and hardness for a family of low-K films.

DISCUSSION

As seen in Figure 2a, there is almost no hysteresis between the loading an unloading portions of the load-displacement curve, which indicates an elastic contact, which can be fit with a Hertzian-like $h^{1.5}$ power law load dependence (Figure 2b).

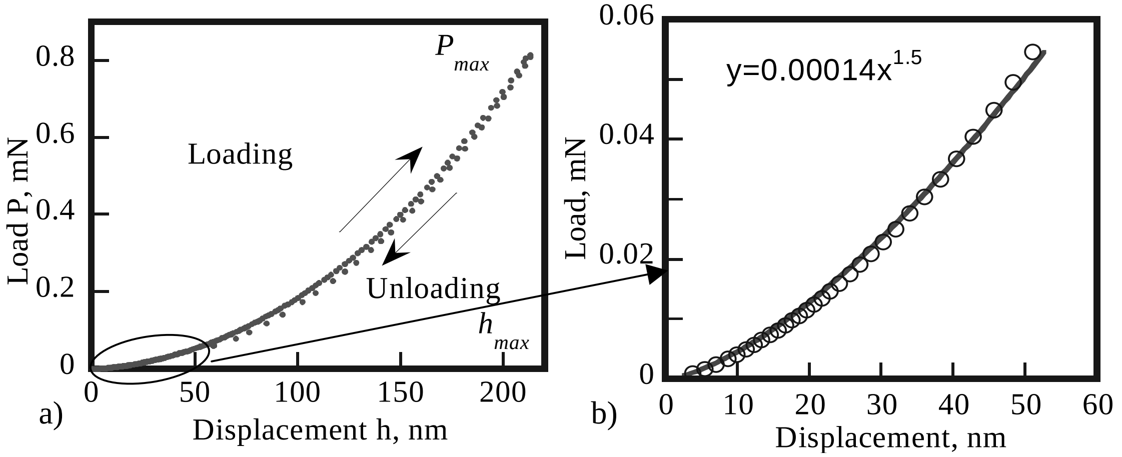


Figure 2. a) Load-displacement curve for a 1 μm thick OSG film; b) Power law fit to a shallow indentation depth data in Figure 2a.

The reduced elastic modulus is determined from the unloading stiffness *dP*/*dh*, and the knowledge of the indenter tip function, *A*:

$$E_r = \frac{\sqrt{\pi}}{2}\frac{dP}{dh}\frac{1}{\sqrt{A}} \qquad (1).$$

The measurement can be performed by either using the Olive and Pharr analysis [5] employing the unloading slope, or by using the tip oscillation continuous stiffness method. Hardness is simply the maximum indentation load divided by the contact area:

$$H = \frac{P_{\max}}{A_{projected}} \tag{2}$$

To first order the tip function for projected contact area, A, is related to the indentation depth, h, as A=24.5h^2, so one can write:

$$H = \frac{P_{\max}}{24.5 \cdot h_{\max}^2} \text{ and } E_r = \frac{\sqrt{\pi}}{2}\frac{dP}{dh}\frac{1}{\sqrt{24.5}h} \tag{3}$$

This assumes a perfectly sharp tip and for tips with finite radius of curvature, a simple correction can be made [6]. It appears that for both loading and unloading portions of the curve load is proportional to $h^{3/2}$ (Figure 2). If the unloading stiffness then can be treated as Sneddon's for a circular punch [7], one would get:

$$E_r = \frac{\sqrt{\pi}}{2}\frac{dP}{dh}\frac{1}{\sqrt{24.5}h} = const \cdot \frac{dP}{dh}\frac{1}{h} = \frac{const}{\sqrt{h}} \tag{4}$$

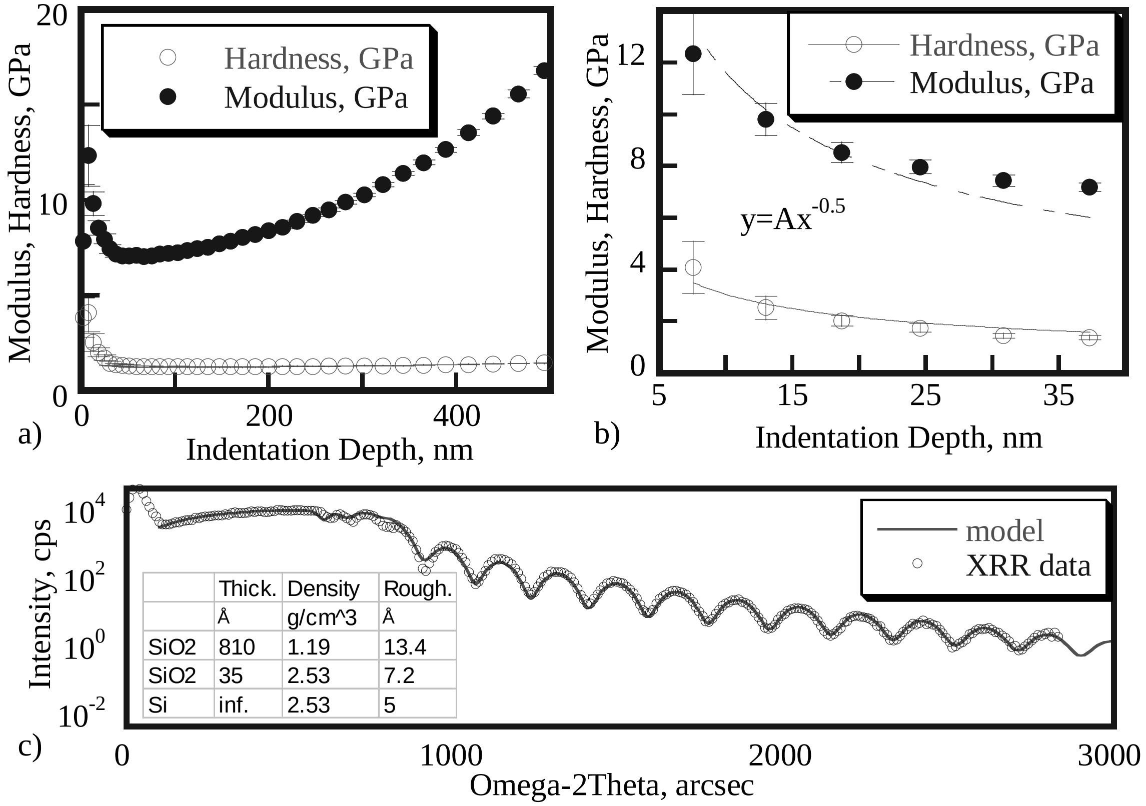
Taking hardness as $H = \frac{P}{const \cdot h^2} = \frac{const}{\sqrt{h}}$, we see that the resulting ratio E/H=constant for the load-displacement profiles following the elastic $P \sim h^{3/2}$ law. This however arrives at the unacceptable result that the modulus is a function of the film thickness. Typically there is an increase in the measured elastic modulus at the film surface attributed to the difficulty of determining the point of contact and the tip function at shallow indentation depth (Figure 3a). At greater depth the modulus reaches the minima, and then the tip starts feeling the presence of a higher modulus substrate, so the measured modulus increases. It turns out that in addition to the surface effects, one would have to consider the modulus decrease with indentation depth as depicted by equation 4, and Sneddon's analysis might not be appropriate for the films exhibiting elastic $P \sim h^{3/2}$ behavior. Figure 3b shows the shallow indentation depth modulus and hardness data from Figure 3a along with the $h^{-0.5}$ fits, proving the concept.

When almost elastic contact is observed using a Berkovich tip with materials exhibiting little or no plasticity, such as low-K dielectrics in this case, one can use a spherical tip with Hertzian analysis [8]. For elastic Hertzian contact indentation load is related to the indentation depth as:

$$P = \frac{4}{3}E_r\sqrt{Rh^3} \tag{5}$$

Now the reduced film modulus can be readily extracted from equation 5 if the spherical tip radius, R is known and the loading and unloading curves are elastic as in Figure 2.

We have attempted to measure low-K film density using X-Ray reflectivity (Figure 3b) [9]. The best fit was obtained by using a thin intermediate SiO_2 layer, and the fitting parameters are shown in Figure 3c [10]. The thickness measurement of 820 Å was confirmed with spectroscopic ellipsometry, and the 50% density of SiO_2 was confirmed with RBS measurements.



	Thick.	Density	Rough.
	Å	g/cm^3	Å
SiO2	810	1.19	13.4
SiO2	35	2.53	7.2
Si	inf.	2.53	5

Figure 3. a) Elastic modulus and hardness as a function of the indentation depth for a 1 μm thick low-K film; c) Shallow depth data from a); c) X-Ray reflectivity data along with a model fit obtained from an 80 nm thick low-K film.

Now we discuss the "incompressible" pore concept as applied to the nanoporous low-K dielectric films. Extra work is done during the deformation process due to the pore surface deformation. The current analysis takes into account the surface energy contributions from the pores to the low-K materials deformation during indentation process. Consider a porous periodic structure shown in Figure 4a, where λ_S is an average distance between the pores. The basis for such simplification is high resolution TEM work presented in [2, 3]. For the volume fraction of the pores, f_V, the total number of pores, N_S, would be:

$$N_S = \frac{1}{\lambda_S^3} = \frac{f_V}{\frac{4}{3}\pi r_S^3} \qquad (6),$$

where r_S is the pore radius. With the known surface energy of the low-K materials, γ_S, we can find the surface work contribution:

$$W_S = \gamma_S 4\pi r_S^2 \cdot \frac{f_V}{\frac{4}{3}\pi r_S^3} = \frac{3\gamma_S f_V}{r_S} \qquad (7).$$

The contribution of the matrix in a porous material would be:

$$W_{matrix} = \frac{E_0\varepsilon_0}{2}(1 - f_V) \qquad (8)$$

Since $W_{\mathrm{Measured}} = W_{\mathrm{Matrix}} + W_{\mathrm{PoreSurface}}$, we can write a work balance equation (per unit volume):

$$\frac{E\varepsilon^2}{2} = \frac{E_0\varepsilon^2}{2}(1 - f_V) + \frac{3\gamma_S f_V}{r_S} \qquad (9),$$

so

$$E = E_0(1 - f_V) + \frac{6\gamma_S f_V}{\varepsilon^2 r_S} \qquad (10).$$

For a sharp Berkovich indenter the strains induced by indentation can be quite high, on the order of 10% [11-13]. Figure 4b shows the surface work contribution to the low-K elastic modulus (second term in equation 10). Pore surface deformation contributions can add as much as a 100 GPa to the low-K elastic modulus for a several nanometer pore size, and 0.3 J/m^2 surface energy of the pore material. The effect is more pronounced for the smaller pore size and larger pore volume fraction (Figure 4b). This model assumes that all pores contribute to the effect, and although the increase in low-K elastic modulus over 72 GPa (dense SiO$_2$) does not seem physically realistic, the effect of surface work contribution on the elastic modulus should not be completely ignored.

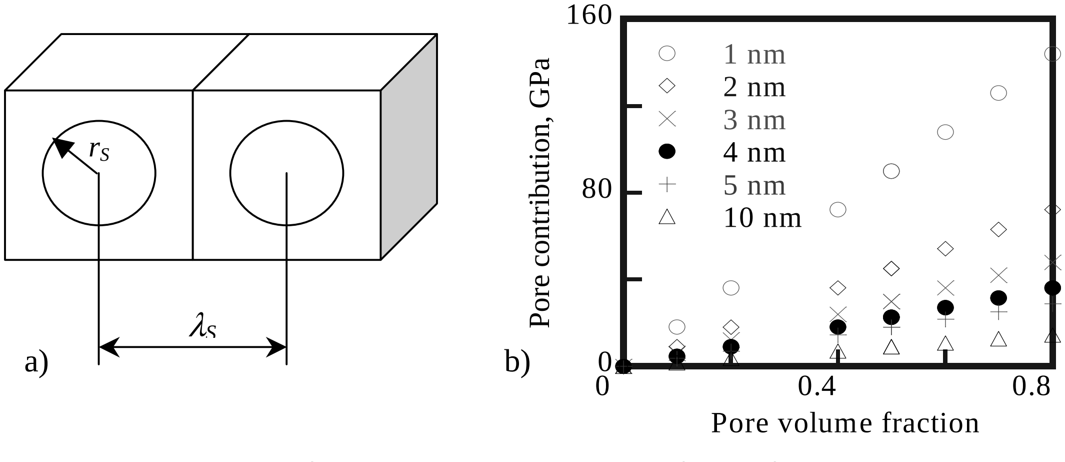


Figure 4. a) Schematic of a porous media; b) Pore surface deformation contribution to the elastic modulus (second term in equation 10).

CONCLUSIONS

A constant modulus to hardness ratio measured by nanoindentation is explained for elastic contact in low-K dielectric constant materials. Hertzian elastic analysis with a spherical tip indentation is proposed instead of sharp Berkovich indentation for measuring elastic modulus of low-K dielectrics exhibiting mostly elastic indentation contact. A simple model, “incompressible” pore effect, that considers surface energy contribution to the elastic deformation of nanoporous media is presented.

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