SiO_2-Aerogel Low-k Films: Improved Mechanical Properties as Pre-condition for Successful Integration

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Introduction

Since Cu-Damascene technique has been introduced in IC-production and thus the dielectric is patterned instead of the metal, mechanical loads on the layer stacks caused by Cu-CMP are considerable. Mesoporous SiO_2-Aerogel low-k films do not display superior mechanical properties because of their high porosity. While k-value decreases with increasing porosity the elastic modulus decreases also (see figure1).

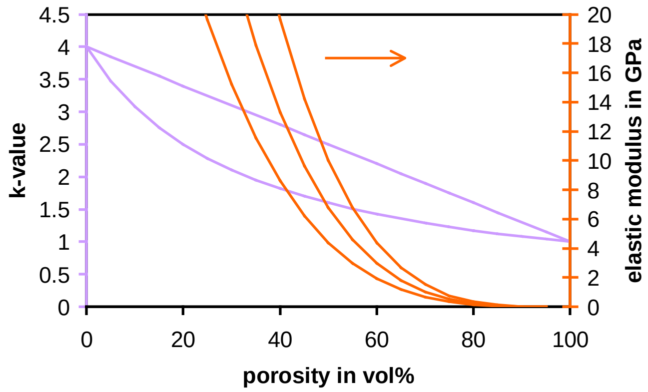


Figure 1 *Elastic modulus and k value vs porosity of silicon dioxide, for estimation of elasticity dependence on porosity a power law was assumed $E{\sim}\rho^{3.7}$ (for assumed SiO_2 skeleton densities of ρ=1.6, 1.8, 2.0 g/cm³), estimation of k-value is based on parallel and series model arrangement and k=4 for SiO_2*

A low elastic modulus of the dielectric film leads to a strong elastic deformation during polishing resulting in inhomogeneously polished surface. Other mechanical properties like strength and toughness are linked with elastic behaviour closely. Small elastic modulus is an evidence for predisposition to inelastic deformation and failure at small loads. Investigations regarding optimization of elastic modulus paying respect to the dielectric constant contain both, the experimental variation of the SiO_2-Aerogel films structure and the adaptation of the testing procedure to the low-modulus film on stiff silicon substrate.

Stiffening of SiO_2-Aerogel low-k films

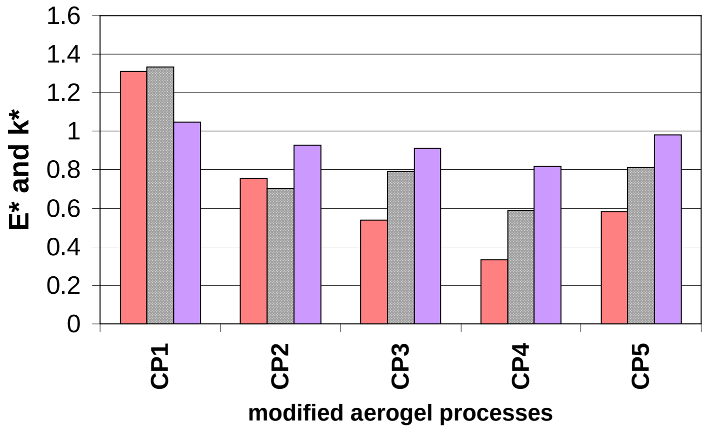
Possibilities to reinforce the porous SiO_2-Aerogel structure are limited. First the stiffening of the SiO_2-network has been investigated. After spin-on deposition the films has been exposed to a solvent ambient to allow gelling. Through this ambient chemical composition of the film can be influenced by adds to enforce cross-linking and finally gelling. The state of the gel before drying is decisive for formation of effective porosity due to more or less reduced SiO_2-network collapse. Gelation/ aging conditions were modified and properties of the obtained films were compared with properties of a film prepared in pure solvent atmosphere without adds. Best results yield the addition of water to the solvent ambient (see table 1).

Table1 *Properties of aerogel films prepared by addition of water to the gelation ambient*

Process	Water content vol%	Porosity vol%	Film thickness nm	Dielectric constant	Elastic modulus GPa
reference	0	50%	500	2.1	3.7
Ia	7	58%	655	1.8	2.8
Ib	14	-	635	2.0	2.9
Ic	28	-	688	1.9	-

The effect of water addition is provable immediately by thickness measurement. Thickness was determined at a step by TENCOR surface profilometer to avoid the impact of a changed refractive index. The SiO_2-network did not collapse in the same extent like the reference film. This explains also the raised porosity of these modified films (obtained from refractive index). The lower effective elastic modulus as well as the lower dielectric constants result from the higher pore volume fraction. Thus the stiffening of the SiO_2-network before drying was successful, but the effective elastic modulus is lower due to higher porosity. Next step consisted in optimization of exposure time in order to find out, if elastic modulus of the stiffened SiO_2-network is also higher at the same k-value like the reference film, that means at the same porosity level. Therefore the exposure time was varied. The preliminary results are displayed in figure 2. Five process variations were selected to demonstrate the ratio of elastic modulus to k-value for aerogel films relative to the reference film.

Figure 2 *Normalised elastic modulus and k-value of aerogel films prepared in modified gelation/aging ambient*



relative to properties of aerogel film gelled in pure solvent ambient,
elastic modulus (red: LSAW, patterned: Nanoindentation) and k-value (purple)

Aerogel films prepared under process conditions CP1 showed the most promising results. The elastic modulus of these films was about 30% higher than the reference (for details see [1]). Although the elastic modulus could be increased, the absolute effective values of the films are (slightly) below 5GPa. It is expected that also further optimization of the modified processes will not bring a significant jump to values much higher than 5GPa at k<2.2. Compared to investigations described in literature it is obvious, that integration of these materials will be challenging and requires an adaptation of all subsequent integration steps. Especially Cu-CMP (and diffusion barrier polishing) has to be carried out with smallest as possible down-forces and abrasive slurries to minimize mechanical load to the layer stack. Additionally, intrinsic stresses of all layers of the stack have to be tuned to each other to avoid extreme stress values under outer loads especially near interfaces.
The differences between both measured elastic moduli for every film (figure 3) come from different testing methods, Nanoindentation and Laser-generated surface acoustic waves analysis (LSAW). Some remarks regarding this issue are added below.

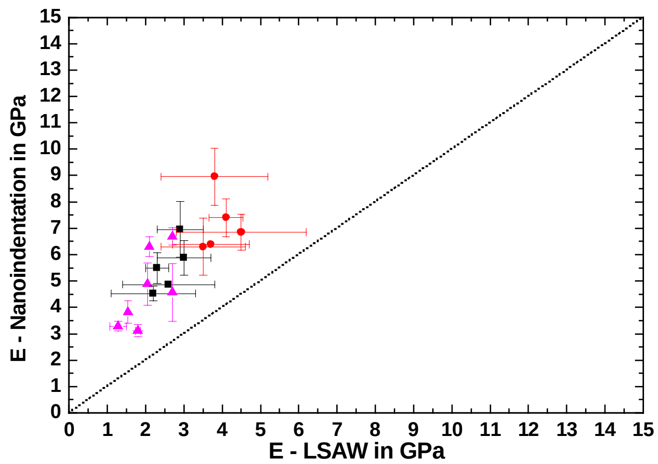


Figure 3 *Comparison of elastic moduli for different aerogel samples determined by Nanoindentation and by LSAW-analysis showing assumed overestimation of Nanoindentation values*

Determination of elastic modulus

Nanoindentation and LSAW are applied as testing methods to characterize the elastic behavior of SiO_2-Aerogel films. For the measurements we could use the Nanoindentation station UMIS2000 at the Department of Physics (Prof. F. Richter) and the LSAW-measurements were carried out by Dr. D. Schneider at the Fraunhofer Institute for Material and Beam Technology IWS Dresden. Figure 3 contains a comparison of elastic moduli for different aerogel samples determined by Nanoindentation and by LSAW-analysis showing expected overestimation of Nanoindentation values. Detailed investigations have shown, that data obtained from Nanoindentation measurement of low-modulus films with high porosity like aerogels contain a few crucial errors [2]. The applied indenter loads are very small (<1mN), but they cause a high penetration depth resulting in increased substrate influence. Therefore no clear plateau can be find in diagrams of elastic modulus vs. penetration depth. Thus no load-independent value for elastic modulus of the aerogel films can be obtained. We have developed a procedure to estimate elastic modulus by extrapolating fitted data obtained over a wide load region to the y-axis (see figure 4). The use of a spherical indenter instead of a Berkovich pyramid is now tested to avoid high penetration depth and inhomogenous stress fields under the indenter tip.

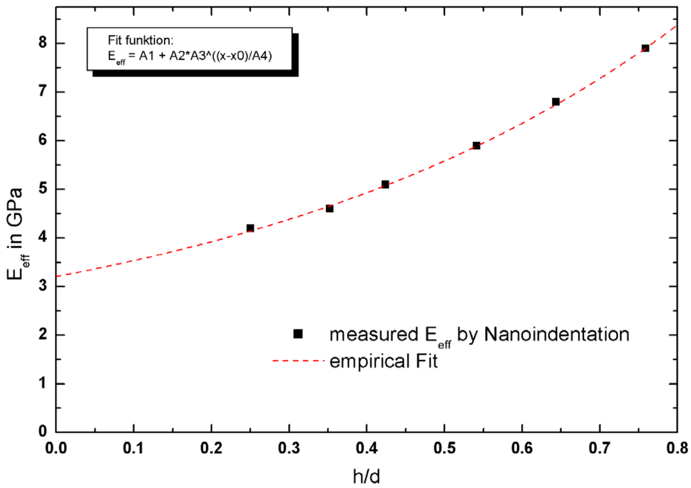


***Figure 4** Extrapolation of an empirical fit function to the y-axis in order to estimate elastic modulus of an aerogel film*

Secondly, the scattering of the data increases with decreasing indenter loads due to high sensitivity of the measurement station and a lot of disturbing environment effects are detected. More attention has to be directed also to deviations of indenter shape, which are generally compensated by correction functions. A third issue regards the contact between indenter tip and film surface. In the very low load region (<0.1mN) attraction and repulsion forces between both partners can cause an error in zero point detection. To clear up these items a lot of scientific work concerning testing methods will be necessary in the future.

Acknowledgement

We thank C.C. Himcinschi and Prof. D.R.T. Zahn for fruitful co-operation and methodological as well as technical support with respect to ellipsometric spectroscopy, I. Hermann and Prof. F. Richter with respect to Nanoindentation (Institute of Physics, TU Chemnitz) and Dr. D. Schneider for measurements of aerogel films by LSAW-analysis (Fraunhofer IWS Dresden).

References

[1] Frühauf, S.; Streiter, I.; Puschmann, R.; Schulz, S.E.; Himcinschi, C.C.; Flannery, C. M.; Gessner, T.; Zahn, D.R.T.: “Modified silica xerogel as a low-k dielectric with improved mechanical properties”, Proc. of the “Advanced Metallization Conference 2002” (AMC), MRS (2003), in press.

[2] Puschmann, R., Diploma, TU Chemnitz, 2002