Anisotropic Thermal Conductivity of Nanoporous Silica Film

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***Abstract*—In this paper, thermal conductivity of porous silica film with porosity from 21 to 64% was studied comprehensively. The corresponded dielectric constant is from 2.5 to 1.5. It is observed that the porous silica material has strong anisotropic characteristic. A serial-parallel hybrid model is proposed to explain the correlation between porosity and thermal conductivity in both in-plane and cross-plane components. The pores in the higher porosity silica film tend to distribute horizontally. This distribution of the pores in the dielectric film is the main factor that induces the anisotropic characteristic. The nonuniform distribution of pores also makes the conventional two-dimensional model of 3ù method inappropriate for extracting the in-plane thermal conductivity. A new method based on the hybrid model was proposed to extract the in-plane thermal conductivity successfully. The anisotropic characteristic of the thermal conductivity may be accompanied by the anisotropic dielectric constant, which will greatly complicate the thermal management and resistance-capacitance delay simulation of the circuits and should be avoided. The proposed model would be helpful on evaluation of new porous low dielectric constant materials.**

***Index Terms*—Dielectric constant, porosity, porous silica, thermal conductivity.**

I. INTRODUCTION

WITH THE progress of IC process technology, the device density increases and performance improves continuously. However, signal propagating along metal interconnects is delayed by the resistance (R) of the metal lines and the capacitance (C) between adjacent conductors. When metal interconnects are designed in a higher packing density, resistance-capacitance (RC) delay of interconnect becomes a major factor of limiting circuit performance [1]–[3]. To reduce *R*, the conventional Al–Cu alloy had been replaced by Cu, which is the second lowest resistivity material. The only solution to further improve RC delay is to replace conventional dielectric, dense SiO_2, with other dielectrics with lower dielectric constant (low-K). The implementation of low-K dielectrics reduces the capacitance between adjacent conductors in the multi-level interconnect structure and, therefore, reduces the signal propagation delay [1]. Power dissipation is also reduced during switching because the use of low-K materials increases the ac impedance of interconnects [4].

One important problem associated with using the low-K material is the lower thermal conductivity of low-K materials compared to conventional dielectric of SiO_2. Thermal energy is a byproduct of circuit operation. Thermal energy density and its spatial gradients govern the temperature distribution and can strongly influence both the electrical currents and the circuit lifetime [5]–[8]. The low thermal conductivity of low-K materials will cause very large temperature rises and temperature gradient in interconnects and devices, which will then increase electromigration-induced interconnect failure and reduce the lifetime of devices. Furthermore, the large temperature rise will induce the voluminal expansion of low-K material which will greatly increase the mechanical stress in a multilevel interconnect structure.

One effective approach to achieve lower dielectric constant is to incorporate pores of microscopic dimensions into the dielectric material [9]. Porous material has a broad pore size distribution like porous silica. The dominant scattering mechanism would be from the pores of the smallest sizes and the maximum phonon mean free path would be limited to the largest pore size [1]. The thermal conduction issue should be worse in porous materials than in condensed materials. If the distribution of pores in the porous material is uniform, the dielectric constant is known to decrease approximately linearly with porosity [11]. However, the effect of porosity on thermal conductivity of thin dielectric films has not been clearly quantified.

The thermal conductivity of dielectric is not always isotropic. In addition to the intrinsic mechanisms present in bulk materials, the observed anisotropy may be caused by grain or molecular structures unique to thin films, and by the size effect associated with boundary or interface scatterings of phonons [3], [11]. The spin-on process used in the deposition of low-K film may cause the partial alignment of molecular chains. Because the weak Van der Waals interaction between the neighboring chains restrains the transport of atomic vibration energy, the thermal conduction between chains has a relatively large thermal resistance [5]. On the other hand, the strong coupling of atomic vibrations along the chains enhances energy transport and leads to a more effective thermal transport in that direction. If a porous material is with the anisotropic thermal conductivity characteristic, the effect of porosity on thermal conductivity is more complicated. Therefore, understanding thermal conductivity of low-K films is critical in material properties, the improvement of composing technique, and the thermal management of integrated circuit.

In this paper, we study the thermal conductivity of porous silica with various porosities. The 3ω method was employed.

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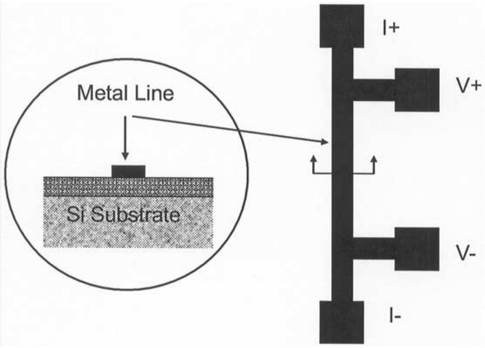

Fig. 1. Schematic drawing of the test structure used for the 3ω method.

The system implementation and detailed experimental procedure are explained in Section II. In Section III, we show that the porous silica material has strong anisotropic characteristic in thermal conductivity and show that the 3ω method is inappropriate to extract the in-plane thermal conductivity. In Section IV, a serial-parallel hybrid model is proposed to explain the correlation between porosity in both in-plane and cross-plane components. A new method, based on the hybrid model, is also proposed to extract the in-plane thermal conductivity and dielectric constant of such films.

II. EXPERIMENTAL PROCEDURE

A. System Implementation

The 3ω method uses a harmonic electrical current in the metal bridge to generate heating temperature fluctuations [11]. Fig. 1 presents the schematic drawing of the test structure. The sinusoidal current at the frequency ω generates a 2ω heating source and a corresponding 2ω temperature rise in the sample. Since the resistance of the heater depends linearly on temperature, there is also a 2ω resistance variation which, combined with the 1ω heating current, induces a 3ω component in the voltage drop across the metal bridge. This 3ω component can be detected by a lock-in amplifier and can be used to extract the thermal conductivity. Hence, this measuring technique is often referred to as the 3ω method. Several physical models, including a one-dimensional (1-D) model [12], [13], a two-dimensional (2-D) model [14], and a multilayer model [15] had been proposed to correlate the 3ω component to the thermal conductivity of homogeneous and isotropic dielectric materials. The errors of the 3ω method due to heat loss through metal pad, radiation, and gas convection have been discussed in literatures [11], [12], [16].

As mentioned, the 3ω measurement system should provide the first harmonic current flowing through specimen to generate the second harmonic oscillation of resistance. The system can also measure the induced third harmonic voltage oscillation across the metal line. We choose a digital lock-in amplifier of model SR850 produced by Stanford Research Systems to measure the 3ω signal. A block diagram of our measurement system is presented in Fig. 2. The 1ω voltage from the sine out of SR850 is boosted into an ac current by a simple electronic circuit served as a fixed current source. The feedback resistor R1 should be nearly temperature independent to avoid generating a 3ω component by the 1ω current. The output voltage buffer of current source must be carefully chosen to provide a sufficient current to generate enough heat and temperature rise in the specimen. The capacitor C should provide a sufficiently large impedance to block ac current from flowing through the measurement frequency range. The output current of the simple current source is limited by the voltage buffer. The voltage buffer we used, Buf04, can provide a stable harmonic current up to 28 mA, which is sufficient for the low-K materials with thermal conductivity poorer than silicon dioxide. The frequency range (1ω signal) is 20 Hz to 1 KHz.

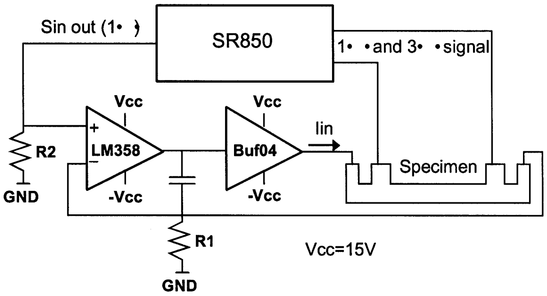

Fig. 2. Block diagram of the 3ω-measurement system used in this work.

B. Sample Preparation and Characterization

The dielectric used in our experiment is a spincoated aromatic hydrocarbon thermal setting polymer—porous silica. The porous silica is cured at 400 °C and is a composite of SiO_2 and pores. The porosity is controlled by adjusting the amount of solvent—the alcohol. The test structure is a single metal line which serves simultaneously as heater and thermometer. In order to measure the accurate 3ω voltage component, a four-terminal cross bridge structure is adopted. The heater/thermometer is fabricated with 200-nm-thick Al. It is deposited in a thermal evaporator system and is patterned by lift-off process. Most low-K materials are sensitive to the chemicals used in the photolithography process. Thus, a passivation layer, 30-nm-thick silicon nitride, is deposited on low-K film before photolithography process. The thermal conductivity of the silicon nitride layer is also measured using the 3ω method and multilayer model is used to correct its effect on the low-K samples. Fig. 3 shows the layout of test structure with 4-μm-wide heater.

Besides the thermal conductivity, some fundamental characteristics of dielectrics are also measured or inspected. Ellipsometry was used to determine the film thickness, the refraction index, and the porosity. The measured results are listed in Table I. Porosity was calculated from refractive indices using the first-order rule of mixtures [17], [18]. The spectrophotometer (N&K analyzer 1200, Nikon) is another instrument for doublechecking the film thickness and refraction index. Fourier-transformed infrared (FTIR) was employed to characterize the chemical bonds of the dielectrics.

Computer-controlled measurement system with precision impedance meter of model Agilent 4284A was used to obtain high frequency capacitance–voltage (*C*–*V*) at 100 KHz. The metal electrode of the capacitor is the same one used in the 3ω measurement. Dielectric constant was calculated from the capacitance at accumulation mode with film thickness given by Scanning Electron Microscopy (SEM) or ellipsometry.

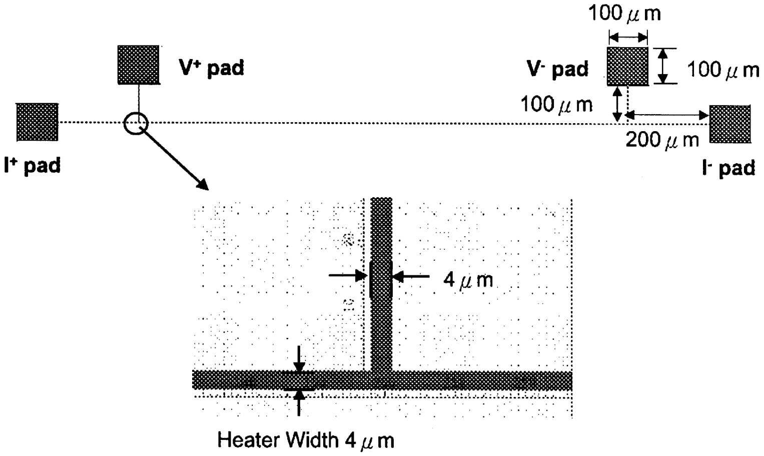

Fig. 3. Layout of test structure with 4-μm-wide heater. For the test structure with 15-μm-wide heater, only the line width is widened from 4 to 15 μm and the other layout parameters are fixed.

TABLE I
FUNDAMENTAL CHARACTERISTICS OF DIELECTRICS MEASURED BY ELLIPSOMETRY METHOD

Porosity (%)	21.2%	28.8%	37.7%	42.1%	56.7	64.2%
Thickness (nm)	314.7	555.9	673.9	673.7	670.4	671.5
Refraction Index	1.362	1.329	1.285	1.268	1.199	1.170

TABLE II
EXTRACTED THERMAL CONDUCTIVITY OF ALL OF THE SILICA FILMS BASED ON THE 1-D MODEL OF 3ω METHOD [12], [13]

Thermal conductivity (W/mK)	Porosity					
	21.2%	28.8%	37.7%	42.1%	56.7	64.2%
4μm Metal Bridge	0.2611	0.2443	0.1862	0.1836	0.1756	0.1529
15μm Metal Bridge	0.2591	0.2170	0.1495	0.1455	0.1368	0.1126

III. RESULT AND DISCUSSION

A. Correlation Between Porosity and Thermal Conductivity

The porosity of porous silica films studied ranges from 21.2% to 64.2%. The silica film with larger porosity has more nanopores, impurities (unreacted chemicals), and hanging and loose-ended silica chains, all of which contribute to significant phonon scattering and limit the mean free path of phonon. Table II lists the extracted thermal conductivity of all the silica films based on the 1-D model. It is expected that the thermal conductivity from both 4 μm and 15 μm wide heaters decreases with the increasing porosity. At the same time, the difference between thermal conductivity extracted from 4 μm wide heater and 15 μm wide heater increases with the increase of porosity. This phenomenon results from the contribution of in-plane thermal conduction. Since the silica film thickness is about one sixth of the 4 μm wide heater, the in-plane thermal conduction does not affect the extracted cross-plane thermal conductivity greatly and only a 35% increment is observed at 64% porosity.

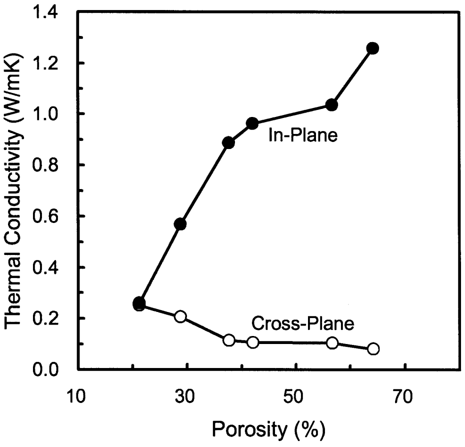

Fig. 4. In-plane and cross-plane thermal conductivity of silica film extracted from the 2-D model of 3ω method [14].

The in-plane and cross-plane thermal conductivity of each silica film were extracted using the 2-D model at first and the results are shown in Fig. 4. About 3–5 data were extracted for every silica films and the range of data is around 10%. Fig. 4 shows the average values. It is observed that the cross-plane thermal conductivity decreases while the in-plane thermal conductivity increases greatly with the increase of porosity. The factors inducing the anisotropic characteristic are the anisotropy of the crystalline domains, the ordering of the crystalline domains or the molecular chains in the surrounding matrix. However, it is unreasonable that these factors lead to an increase in in-plane thermal conductivity as the porosity increases from 21.1% to 64.2%. If the in-plane thermal conductivity is dubious, the relative cross-plane thermal conductivity is also suspicious. The overestimation of in-plane thermal conductivity results in underestimation of cross-plane thermal conductivity. It means that the 2-D model cannot be applied to the porous silica film studied here. As the width of metal heater is much larger than the thickness of silica film, the thermal conductivity extracted from 15-μm-wide heater and 1-D model is close to the actual value of the cross-plane thermal conductivity than that extracted from the 2-D model.

The reason why 2-D model does not work properly is brief explained. The 2-D model assumes one phase system. As the pores distribute uniformly, the 2-D model can be adapted. However, as the pores distribute horizontally, the dielectric becomes laminated with two phase. In this case, the multilayers model should be used to describe the thermal conduction. Unfortunately, how many layers should be assumed is unknown. Furthermore, as the pores distributed vertically, the published multilayer model still does not work. Therefore, new model must be developed to extract the actual thermal conductivity of the dielectric.

B. Conventional Anisotropic Models

Thermal conduction in a two-phase system has been a longstanding theoretical issue. Several models have been proposed, such as the parallel model, the serial model, the dilute particle model, and the dilute fluid model [9], [19]. The concept of the parallel model and serial model treats the thermal conduction paths in the medium and through the pores. The dilute particle model and the dilute fluid model are more sophisticated for they treat the dilute minor phase as noncontacted inclusions in the matrix of another phase. These four models are typically referred to as the limits of possible thermal conductivity. In addition, semi-empirical models or models dealing with details of the shape and the contact of particles in the solid state were also proposed [19]. Chuan Hu *et al.* for example, proposed two semi-empirical models, porosity weighted simple medium (PWSM) model and porosity weighted dilute medium (PWDM) model (see Fig. 5) [9]. The PWSM model is a combination of the parallel and serial model

$$K_{eff} = K_s \frac{PK_f + (1-P)K_s}{K_s}(1-P^x) + K_f \frac{K_s}{\varepsilon K_s + (1-P)K_f} P^x. \quad (1)$$

On the other hand, the PWDM model is a combination of the dilute particle and dilute fluid model

$$K_{eff} = K_s \frac{2(1-P)K_s + (1-2P)K_f}{(2+P)K_s + (1-P)K_f}(1-P^x) + K_f \frac{(3-2P)K_s + 2PK_f}{PK_s + (3-P)K_f} P^x \quad (2)$$

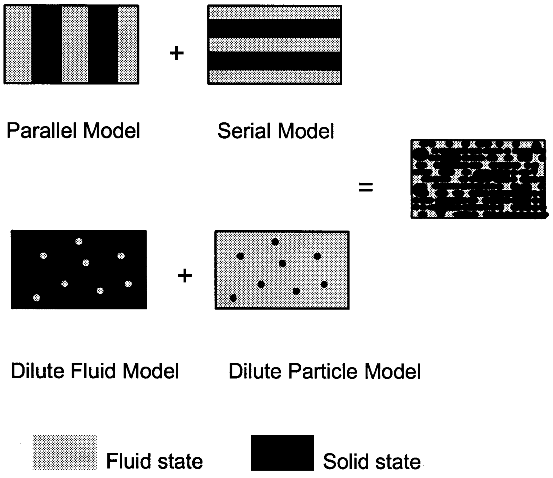

Fig. 5. Schematic illustration of the previously proposed two semi-empirical models: porosity weighted simple medium (PWSM) model and porosity weighted dilute medium (PWDM) model.

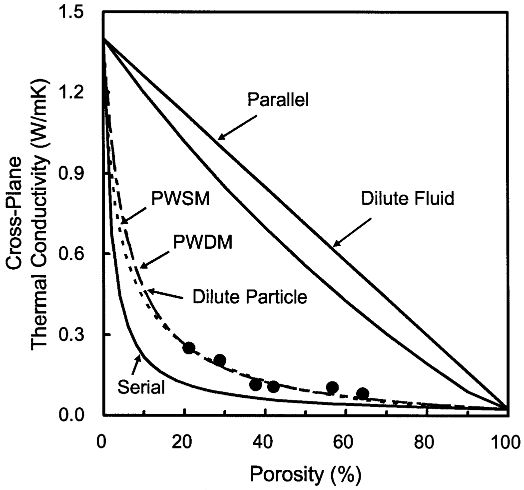

Fig. 6. Comparison of the measured cross-plane thermal conductivity with the previously proposed six models. The experimental results are best fitted with the PWSM model with $x = 0.099$.

where K_s is the thermal conductivity of the wall material (solid state, SiO_2, etc.), K_f is the thermal conductivity of the pore material (fluid state, air, etc.), P is the porosity, and x is the fitting parameter. The fitting parameter x, ranging from 0 to ∞, is introduced to account for the combined effect of pore shape, pore size, and other parameters on thermal conductivity. In fact, x is essentially the contact between good conducting solid-state materials and cannot represent the realistic porosity in the porous film. The first term in (1), (2) represents the contribution of the parallel medium (dilute fluid) and the second term in (1), (2) represents the contribution of the serial medium (dilute particle). The parallel medium means that the paths for cross-plane heat transport are parallel in the vertical direction and the serial medium means that the paths for cross-plane heat transport are serial in the vertical direction. In other words, the parallel medium is the pore distributed in the vertical direction and the serial medium is the pore distributed in the horizontal direction.

Our data of the silica films and the results of the above-mentioned six models are presented in Fig. 6. The parameter x is optimized in the PWSM and PWDM model. The best fitted value

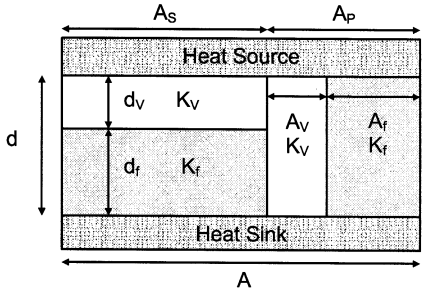

Fig. 7. Schematic illustration of the newly proposed serial-parallel hybrid model in the cross-plane direction. The parallel component and serial component are connected in parallel for the cross-plane direction.

of x is 0.099 for the PWSM model, which represents the small contribution of the parallel medium. The best fitted value of x is almost zero for the PWDM model and, thus, the PWDM model is identical to the dilute particle model. However, the contribution of the decreasing thermal conductivity almost due to the dilute particle model is impractical because $x \sim 0$ means the silica particles served as the inclusions in the matrix of pores. Therefore, the only suitable model that describes the porosity dependence of the thermal conductivity is the PWSM model with $x = 0.099$. From the previous discussions, we can conclude that the main factor attributing to the anisotropic characteristic of porous silica films is the pores distributed in the horizontal direction.

It is assumed that the heat flow in the 2-D model radially transports from the heater to the film and the substrate. However, we know the pores are distributed horizontally within the film, as we just discussed. The heat flow in the realistic porous silica film is different from the assumption of the 2-D model. Thus, the increase of in-plane thermal conductivity with the increase of porosity, which appears unreasonable, is a result of a faulty assumption.

Using a wide metal bridge and adopting 1-D model, one can obtain nearly valid cross-plane thermal conductivity. However, the parameter x in (1) and (2) only represents the relative value in the PWSM model or the PWDM model. Therefore, the previous mentioned models can not be used to extract in-plane thermal conductivity as the nano-pores being distributed nonuniformly. In Section III-C, a serial-parallel hybrid model is proposed to solve the problem.

C. Parallel-Serial Hybrid Model

Fig. 7 shows the schematic drawing of our serial-parallel hybrid model to predict the thermal conductivity of porous material with different porosities. Heat is generated in the top electrode and is transport toward the bottom electrode. As the width of top electrode is much wider than the thickness of dielectric, the fringing heat transport can be ignored. It is assumed that the dielectric can be divided into two parts : the parallel part—where pores are distributed in the vertical direction, and the serial part—where pores are distributed in the horizontal direction. The thermal conductance of the serial part (G_s) is expressed as

$$\frac{1}{G_s} = \frac{d_v}{A_s k_v} + \frac{d_f}{A_s k_f} \tag{3}$$

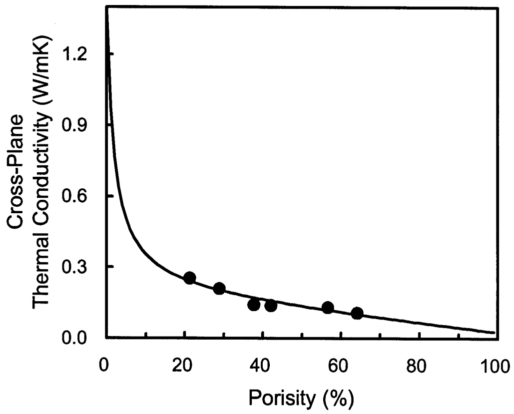

Fig. 8. Comparison of the measured cross-plane thermal conductivity with the serial-parallel hybrid model. The measured results are best fitted with $x = 0.119$.

and the thermal conductance of the parallel part (G_p) is expressed as

$$G_p = \frac{k_v A_v}{d} + \frac{k_f A_f}{d} \tag{4}$$

where d_v is the thickness of the inclusion medium in the serial part, d_f is the thickness of the host medium in the serial component, d is the thickness of the dielectric and is equal to $d_v + d_f$, K_v is the thermal conductivity of the inclusion medium, K_f is the thermal conductivity of the host medium, A_s is the area of the serial component, A_v is the area of the inclusion medium in the parallel component, and A_f is the area of the host medium in the parallel component. The total thermal conductance is the sum of G_s and G_p: i.e.,

$$\begin{aligned} G &= A_s \left(\frac{d_v}{k_v} + \frac{d_f}{k_f} \right)^{-1} + \left(\frac{k_v A_v}{d} + \frac{k_f A_f}{d} \right) \\ &= \frac{k_{eff} \left(A_s + A_p \right)}{d} \end{aligned} \tag{5}$$

where A_p is the area of the parallel component and is equal to $A_v + A_f$. Thus, the effective thermal conductivity is

$$\begin{aligned} k_{eff} &= \frac{d A_s \left(\frac{d_v}{k_v} + \frac{d_f}{k_f} \right)^{-1} + \left(k_v A_v + k_f A_f \right)}{A} \\ &= \frac{A_s}{A} \left(\frac{d_v}{d k_v} + \frac{d_f}{d k_f} \right)^{-1} + \frac{k_v A_v + k_f A_f}{A} \\ &= \frac{A_s}{A} \left(\frac{P}{k_v} + \frac{P}{k_f} \right)^{-1} \\ &\quad + \frac{k_v A_v - k_f A_v + k_f A_v + k_f A_f}{A} \\ &= (1 - x) \left(\frac{P}{k_v} + \frac{P}{k_f} \right)^{-1} + \left(k_v - k_f \right) P x + k_f x \end{aligned} \tag{6}$$

where $A = A_s + A_p$ is the total area, $x = A_p / A$ is the ratio of the parallel component area to the total area, and $A_v / A_p = d_v / d = P$ is the porosity of the dielectric. Fig. 8 shows the measured data and the calculation results. The best fitted value

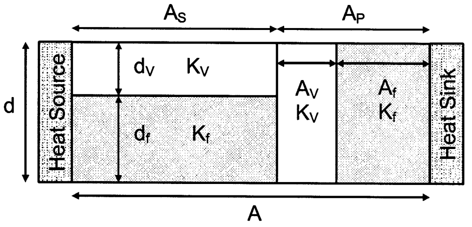

Fig. 9. Schematic illustration of the newly proposed serial-parallel hybrid model in the cross-plane direction. The parallel component and serial component are now connected in serial for the in-plane direction.

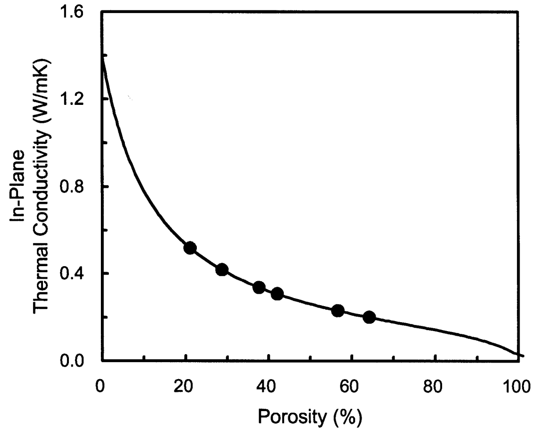

Fig. 10. Extracted in-plane thermal conductivity from the serial-parallel hybrid model as a function of porosity.

of the ratio x, which is 0.13, implies that the serial component contributes more to the effective dielectric constant.

Now, we use this model to extract the in-plane component of thermal conductivity. As shown in Fig. 9, the parallel part and serial part are now connected in serial from the in-plane direction. The in-plane thermal conductance is now expressed as

$$G_s = \frac{k_v d_v}{A_s} + \frac{k_f d_f}{A_s} \tag{7}$$

$$\frac{1}{G_p} = \frac{A_v}{k_v d} + \frac{A_f}{k_f d} \tag{8}$$

and

$$\begin{aligned} k_{eff} &= \frac{1}{\frac{A_s}{A}\left(\frac{k_v d_v}{d} + \frac{k_f d_f}{d}\right)^{-1} + \left(\frac{A_v}{Ak_v} + \frac{A_f}{Ak_f}\right)} \\ &= \frac{1}{(1-x)\left(k_v P + k_f(1-P)\right)^{-1} + \frac{Px}{k_v} + \frac{(1-P)x}{k_f}}. \end{aligned} \tag{9}$$

The meanings of all symbols are identical to those in the cross-plane case (3)–(6). The parameter x, the ratio of the parallel part area to the total area, can be extracted from the cross-plane equations and is then applied to the in-plane equations. After getting x, we can calculate the in-plane thermal conductivity as a function of porosity. The calculated values of in-plane thermal conductivity at various porosities are shown in Fig. 10. Similar to the cross-plane thermal conductivity, the in-plane thermal conductivity decreases almost linearly with the increase in porosity, but is always higher than the cross-plane thermal conductivity.

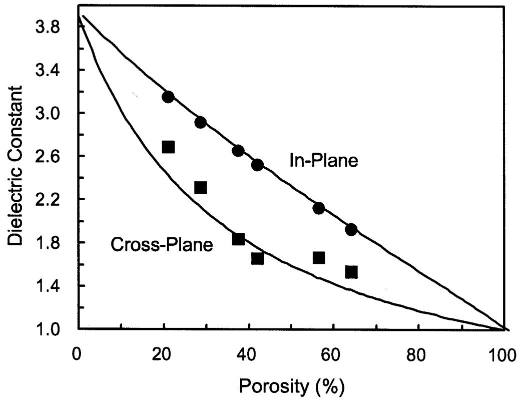

Fig. 11. Comparison of the measured cross-plane dielectric constant and the extracted in-plane dielectric constant as a function of porosity. The measured results are best fitted with $x = 0.13$.

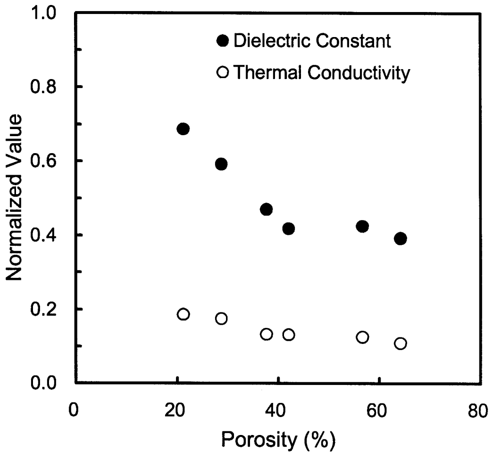

Fig. 12. Comparison of the impact of porosity on the cross-plane thermal conductivity and dielectric constant.

The proposed hybrid model only considers the effect of pores on the thermal conductivity, regardless the influences of the possibly different molecular chains in the high porosity silica film or the more phonon scattering induced by the higher pore inclusions. However, the model is still sufficient for evaluating the anisotropic characteristic of the thermal conductivity.

By replacing thermal conductivity with dielectric constant and thermal conductance with capacitance, this model can be used to evaluate the anisotropic dielectric constant. Fig. 11 compares the measured cross-plane dielectric constant and the extracted in-plane dielectric constant. The best fitted parameter x is 0.119 in this case. This value is very close to that for thermal conductivity, which confirms the validity of the proposed hybrid model. It is clear that the nonuniform distributed nanopores also results in anisotropic dielectric constant of the porous silica film. Because of the horizontally distribution of pores, the cross-plane component decreases with the increase of porosity nonlinearly while the in-plane component decreases almost linearly. Furthermore, the cross-plane component is lower than the in-plane component.

Fig. 12 compares the impact of porosity on the cross-plane thermal conductivity and dielectric constant. The values had been normalized to the values of pure SiO_2, i.e., thermal conductivity $= 1.4$ W/mK and dielectric constant$= 3.9$. It is observed that the thermal conductivity decreases faster than the dielectric constant. The silica film with 64.2% porosity lowers the dielectric constant of SiO_2 by roughly a factor of 2.5. In contrast, the thermal conductivity is about 13.5 times lower than that of SiO_2. The cross-plane thermal conductivity and in-plane the thermal conductivity decrease from 0.25 to 0.11 and from 0.52 to 0.20, respectively, as the porosity increases from 21% to 64%. Thus, a tradeoff between thermal and electrical properties has to be optimized for ultra-large-scale integration with porous low-K materials.

IV. CONCLUSIONS

Porous silica is one of the candidates to achieve dielectric constant lower than 2.0. However, as the nano-pores distribute nonuniformly, the porous silica material show strong anisotropic characteristic in thermal conductivity. We proposed a serial-parallel Hhybrid model to explain the correlation between porosity and thermal conductivity in both in-plane and cross-plane components. The pores in the higher porosity silica film tend to distribute horizontally. This distribution of the pores in the dielectric film is the main factor that induces the anisotropic characteristic in both thermal conductivity and dielectric constant. The nonuniform distribution of pores also renders the 3ω method inappropriate. A novel method based on the hybrid model was proposed to extract the in-plane thermal conductivity and dielectric constant of such film.

It is expected that the newly proposed hybrid model can be applied to porous materials with uniformly distributed pores, too. The major restriction may be the need of films with various porosities but the same pore distribution. New porous material may be synthesized in the future. If the microstructure is quite different from the silica film used in this work, modification of the model may be necessary. The porous silica films studied in this work is close pore system. For the open pore system, since the pores of open pore system open at the top or bottom surface, it is supposed that the value of fitting parameter x is close to 1. That is the parallel part dominant.

The anisotropic characteristic of the thermal conductivity may be accompanied by the anisotropic dielectric constant, which is a greatly complicated problem for the RC delay simulation of the circuits. Efficient thermal and structural design of future generation IC with porous low-K materials should take these factors into consideration.

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