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ANALYSIS OF INDENTATION DEPTH DEPENDENCE OF ELASTIC PROPERTIES OF INTER-LAYER DIELECTRIC FILMS ON SILICON

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Abstract – Analysis of indentation depth dependence of elastic modulus of soft films on silicon substrate measured by nanoindentation was studied. The experimental results were compared with two existing models that describe the effects of the substrate on elastic modulus of layered solid. Saha-Nix model that is the recent modification of King model agreed well with the experimental results to a certain limit of the indentation depth. It was found that the depth limit which Saha-Nix model is valid depended almost linearly on the relative elastic modulus of the film to the substrate E_f/E_s. We further introduced an additional scaling parameter that is a linear function of E_f/E_s in Saha-Nix model. The improved model was applied to porous silica low-k films. The model predicted a constant intrinsic elastic modulus of the film up to a depth of 30 % of the film thickness.

Keywords : Nanoindentation, Low-k, Elastic modulus

1. INTRODUCTION

Recently, the mechanical properties of inter-layer dielectric (ILD) thin film materials, low-k in particular, have been of great importance in the semiconductor device industry. Nanoindentation technique is now essential for the mechanical characterization of such materials.

In general, the elastic modulus of thin film on substrate measured by nanoindentation has indentation depth dependence which is caused by the elastic deformation of substrate materials. In order to obtain intrinsic elastic modulus of very thin film from larger indentation, an appropriate method for dealing with the substrate effect has been required.

In this study, we made an attempt to develop a method for extracting the ‘film-only’ elastic modulus of the soft ILD films on silicon substrates from nanoindentation measurements.

2. EXPERIMENT

2.1. Thin film samples

The elastic moduli of the following ILD films on silicon (100) substrates have been studied: thermally oxidized silica, PE-CVD SiCN, PE-CVD SiOC, spin-on deposited (SOD) TEOS (tetraethoxysilane) glass, plasma co-polymerized (PCP) organic low-k [1] and spin-on deposited porous silica low-k [2] of four different porosities. The thicknesses of these films were measured using a spectroscopic ellipsometry. These are listed in Table I.

TABLE I. ILD samples used for the investigation of elastic modulus

Material	Method of preparation	Thickness (nm)
Silica	Thermal oxidation	400
SiCN	PE-CVD	50,100, 200, 300
SiOC	PE-CVD	95, 50, 100,198, 300
TEOS	SOD	223
Organic Low-k	PCP	320
Porous Silica Low-k	SOD	260, 300, 304, 330

2.2. Nanoindentation

The mechanical properties of the film/substrate systems were characterized using a Nano Indenter DCM^{TM} (MTS Nano Innovation Center, Oak Ridge, TN) with a Berkovich diamond indenter tip. The indentations were made up to a depth of the film thickness with a constant strain rate of 0.01 s^{-1}. Both composite elastic modulus of the film/substrate system and contact area were recorded as functions of indentation depth by the continuous stiffness measurement (CSM) technique. Nine indentations were made in each sample and the results presented are an average of these indentations.

3. MODELS FOR THE ELASTIC PROPERTIES OF THIN FILMS ON SUBSTRATES

3.1 Doerner-Nix model

Doerner and Nix [3] first proposed that the effective composite elastic modulus, E_{comp}, in a layered solid can be described as a function of plane strain Young’s modulus of the relevant materials:

$$\frac{1}{E_{comp}} = \frac{1-\nu_f^2}{E_f}\left(1-e^{-\frac{\beta t}{h}}\right) + \frac{1-\nu_s^2}{E_s}\left(e^{-\frac{\beta t}{h}}\right), \qquad (1)$$

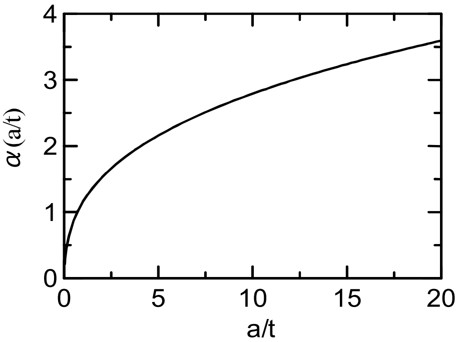


Fig. 1 Plot of α as a function of normalized punch size, *a*/*t*.

where β is a scaling parameter, t is the thickness of the film, h is the depth of indentation, E is the Young's modulus and ν is Poisson's ratio. Subscript f and s refer to the film and substrate properties, respectively. They determined the scaling parameter β empirically based on measurements of tungsten films on silicon substrates. Hence their model was valid only for their specific film/substrate composite systems.

3.2 King model

King [4] modified the Doerner-Nix equation for the composite modulus of layered solid that is applicable to arbitrary combination of film and substrate and defined it as

$$\frac{1}{E_{comp}} = \frac{1-\nu_f^2}{E_f}\left(1-e^{-\frac{\alpha t}{a}}\right)+\frac{1-\nu_s^2}{E_s}\left(e^{-\frac{\alpha t}{a}}\right), \quad (2)$$

where a is the square root of the projected contact area of a flat punch, t is the thickness of the film and α is a numerically obtained scaling parameter that is a function of a/t. The scaling parameter α for the case of a triangular punch is shown in Fig. 1. It is noted that King's analysis was based on the infinitesimal indentation, or namely, the contact of a flat punch. So King's equation does not include the indentation depth h.

3.3 Saha-Nix model

Recently, Saha and Nix [5] further extended the King equation for more realistic case of finite indentation depth with Berkovich indenter. They assumed the triangular flat punch to be located at the tip of the Berkovich indenter as shown in Fig.2. Hence in their analysis, the effective film thickness below the flat punch is (t-h) instead of the actual film thickness t. The modified equation is then written as

$$\frac{1}{E_{comp}} = \frac{1-\nu_f^2}{E_f}\left(1-e^{-\frac{\alpha(t-h)}{a}}\right)+\frac{1-\nu_s^2}{E_s}\left(e^{-\frac{\alpha(t-h)}{a}}\right). \quad (3)$$

The scaling parameter α is naturally the same as that of King model.

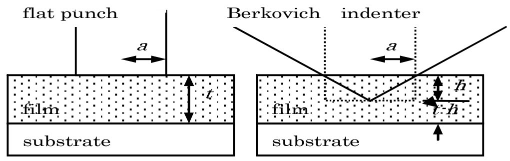


Fig. 2 (a) A flat punch indents a film of thickness t in King model. (b) A flat punch indents the effective film thickness of (t-h) in Saha-Nix model.

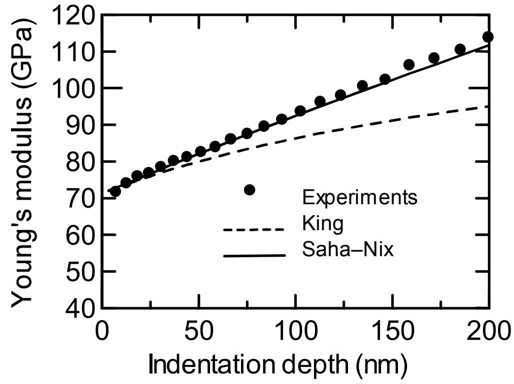


Fig.3 Composite Young's modulus of thermally oxidized silica of thickness of 400nm on silicon as a function of indentation depth.

4. COMPARISON BETWEEN MODELS AND EXPERIMENTS

The indentation depth dependence of experimental value of composite Young's modulus of the film/substrate systems and model calculation of (2) and (3) were compared. Young's modulus of the film/substrate systems were converted from measured composite modulus assuming the value of Poisson's ratio of 0.2 throughout the experiments for simplicity. The measured value of Young's modulus at the indentation depth of 5 nm was used as the 'film-only' Young's modulus E_f in (2) and (3) for ILD films taking account of uncertainty arising from surface roughness and errors in the contact point and the area function of the indenter. Young' modulus of silicon (100) substrate E_s was assumed to be 170 GPa.

4.1 Silica layer

Figure 3 shows the composite Young's modulus of 400nm thermally oxidized silica film on silicon substrate. Young's modulus of silica and silicon film/substrate composite system tends to 72 GPa of that of bulk fused silica at extremely small indentation depth and then it increases with increasing depth of indentation. The intrinsic Young's modulus of the silica film on the substrate can be obtained by taking the limit of the value of composite modulus at the surface of the film. Compared the experimental results to the model predictions, Saha-Nix model described well the indentation depth dependence of composite modulus of this film substrate system up to 200nm, while King model was valid only for quite small indentation depth less than a tenth of the thickness of silica layer.

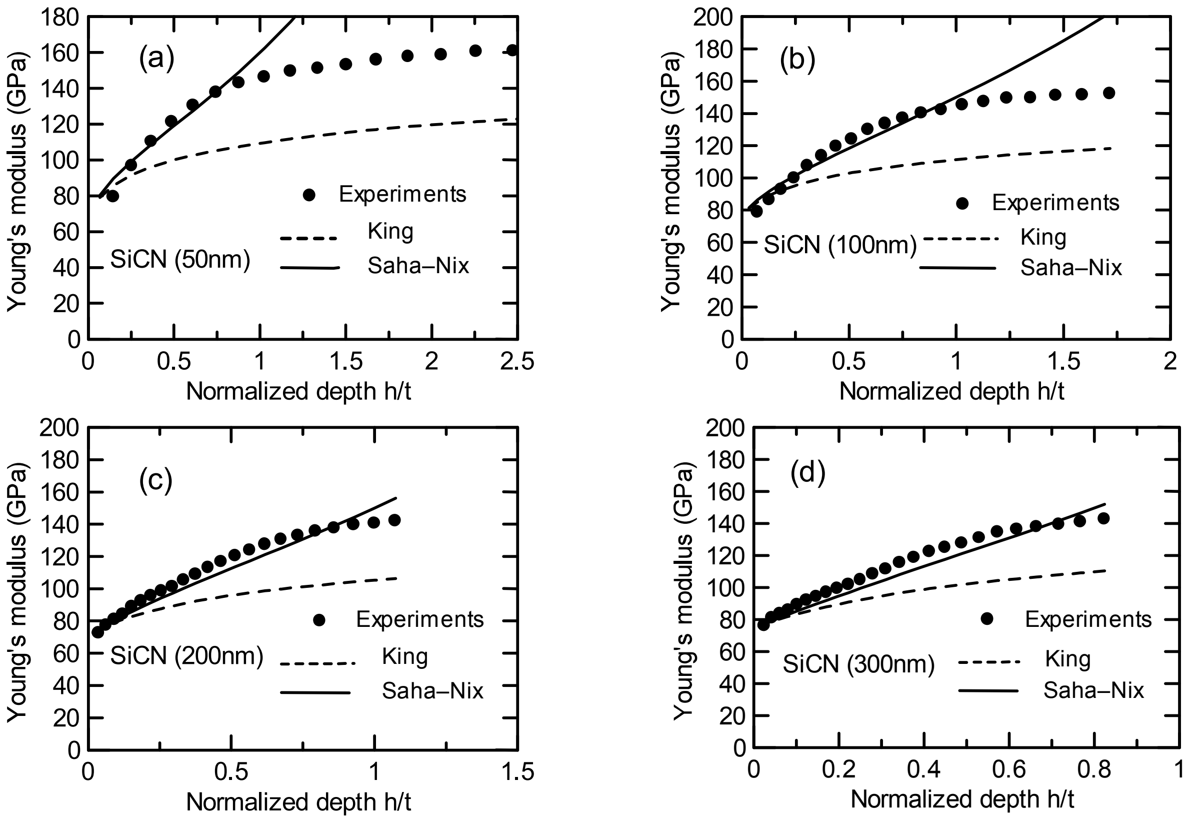


Fig. 4 Composite Young's moduli of SiCN films of thicknesses of (a) 50, (b) 100, (c) 200 and (d) 300nm.

4.2 SiCN films

Figures 4 (a) (b), (c) and (d) show the indentation depth dependences of Young's modulus of SiCN/Si systems that the film thicknesses are ranging from 50 to 300nm. The indentation depth *h* was normalized by the film thickness *t* for ease of comparison of the results. The measurement value at the film surface gives the intrinsic SiCN film modulus of 70 GPa for every sample. Obviously King model get failing even at extremely small indentation depth as the case of silica. Saha-Nix model agreed with the measured values for every SiCN film sample to a certain extent of indentation depth. It is noted that the relative depth limits with respect to the film thickness that Saha-Nix model was valid were nearly equal to 0.9 for every SiCN film in spite of the great difference in thickness.

4.3 TEOS glass film

Composite Young's modulus of TEOS glass on silicon substrate of thickness of 223 nm is appeared in Fig. 5. The 'film-only' Young's modulus of this film was estimated as 48 GPa. The modulus of TEOS glass is smaller than that of thermally oxidized silica layer, owing to the imperfection of silica bonding network arising from residual ethoxy groups. Calculation of Saha-Nix model and experimental values showed in good agreement with the indentation depth ranging up to 0.8 of the film thickness for TEOS derived sol-gel glass film.

4.3 SiOC films

Figures 6(a) to (e) show Young's moduli of SiOC/Si systems as functions of the relative indentation depth h/t. SiOC of thickness of 95 nm was deposited on the condition so as to have higher strength than others. The intrinsic Young's modulus of SiOC (95nm) was obtained as 23 GPa. For SiOC (95nm), Saha-Nix model worked well to the relative depth of h/t = 0.4. With regard to remaining four SiOC films deposited on the same condition, the intrinsic Young's moduli were equally found to be 11 GPa. And we see that the prediction of Saha-Nix model matches experimental values up to about h/t = 0.25 that is approximately independent from the film thickness as found in SiCN films.

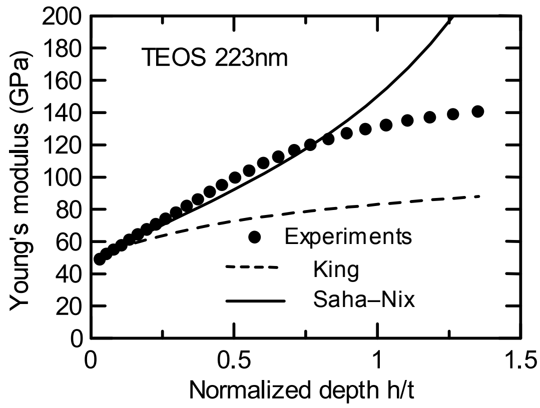
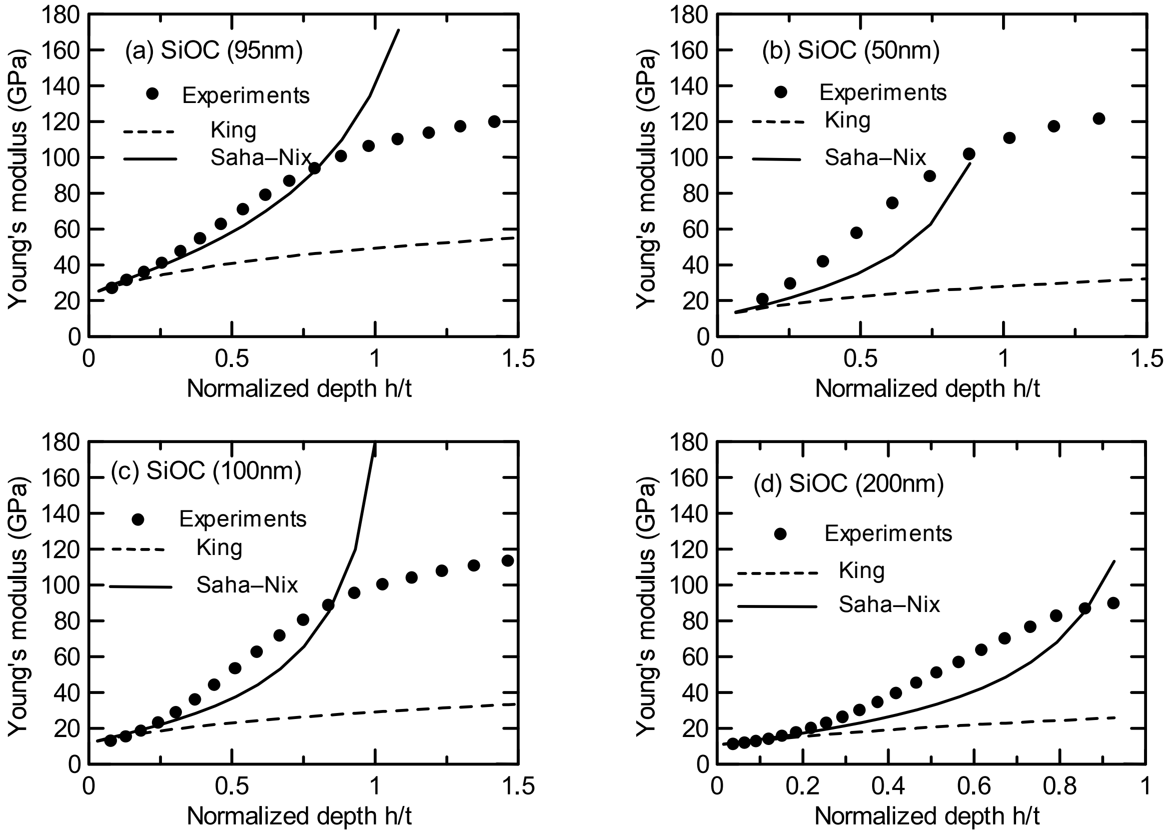


Fig. 5. Composite Young's modulus of TEOS glass film on silicon substrate.



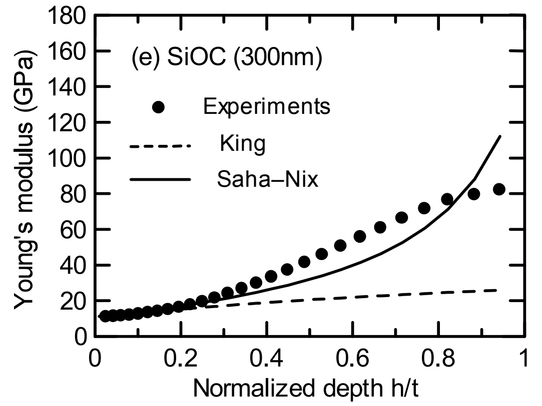


Fig. 6. Composite Young's moduli of SiOC films on silicon substrates. (a) SiOC (95 nm) was deposited on the condition so as to be harder than others.

4.4 PCP organic low-k film

The relative indentation depth dependence of composite modulus of PCP organic low-k on silicon substrates are shown in Fig. 7. Saha-Nix model is effective to the relative depth of 0.18 for the PCP organic low-k having the intrinsic film modulus of 3.2 GPa.

4.5 Summary on the experimental results

The composite Young's modulus of film/substrate system was more significantly affected by substrate stiffness when the difference of elastic modulus between the soft ILD film material and the silicon substrate was greater. The value of composite modulus in the vicinity of a film surface gives the intrinsic Young's modulus of the film as shown in the cases of thermally oxidized silica, SiCN and SiOC films.

The King model could not predict the indentation depth dependence of the composite elastic modulus of film/substrate systems. Saha-Nix model which is the extension of King's analysis showed fairly good agreement with our experimental results to a certain extent of indentation depth. We observed that the relative depth limit with respect to film thickness which their model showed its validity was depended on the elastic modulus of the ILD film rather than film thickness as seen in the cases of SiCN

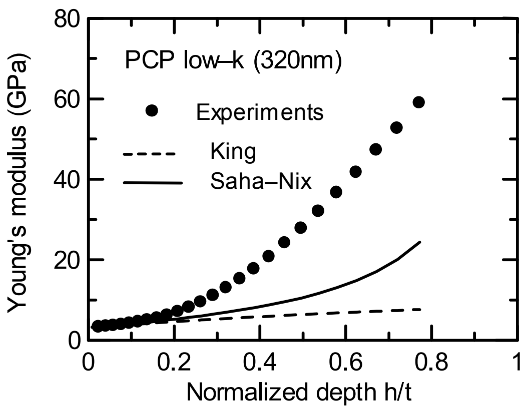


Fig. 7. Normalized indentation depth dependence of composite Young's modulus of plasma co-polymerized organic low-k film on silicon substrate.

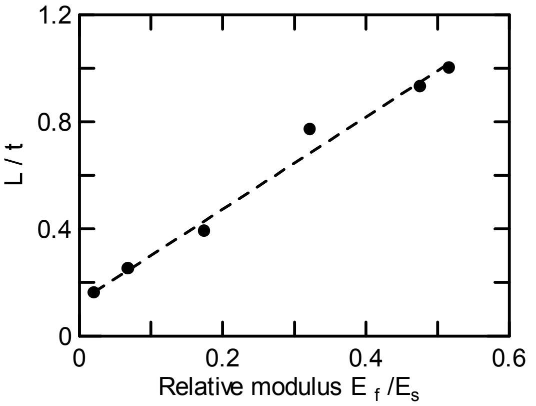


Fig. 8. The depth limit L that Saha-Nix model matches the experimental composite Young's modulus within an error of 10 % normalize by film thickness t as a function of relative modulus E_f/E_s.

and SiOC films, while there were great differences in thickness in the samples. It seems that the relative depth limit monotonically decreases with decreasing in film modulus from 72 to 3 GPa. On the basis of this fact, let us examine the possibility of an extension of Saha-Nix model so as to be applicable to larger indentation depth for extremely soft ILD films in the following section.

5. FURTHER EXTENSION OF SAHA-NIX MODEL

5.1 Introduction of an additional scaling parameter

The depth limit did not depend on film thickness but on the relative elastic modulus E_f/E_s. We now define the depth limit L as an indentation depth that the difference between measured value and that of Saha-Nix model falls within an error of 10 %. Figure 8 shows the plot of L normalized by t as a function of the E_f/E_s. We can see that L/t almost linearly varies on E_f/E_s. This fact suggests that the indentation depth should be scaled by a linear function of the relative modulus of the film with respect to the modulus of silicon substrate in an improved model of the substrate effect. We further extended Saha-Nix model so as to include the relative modulus of the film E_f/E_s as a scaling parameter of indentation depth γ as well as the square root of the projected contact area a. And the scaling parameter γ is assumed to be a linear function of the relative modulus E_f/E_s. Thus, new equation that can describe the substrate effect on indentation modulus of film/substrate system is written as

$$\frac{1}{E_{comp}} = \frac{1-\nu_f^2}{E_f}\left(1-e^{-\frac{\alpha\left(t-h/\gamma\right)}{a}}\right)+\frac{1-\nu_s^2}{E_s}\left(e^{-\frac{\alpha\left(t-h/\gamma\right)}{a}}\right), \quad (4a)$$

$$\gamma = c - d\cdot\left(E_f/E_s\right), \quad (4b)$$

where c and d are the coefficients to be experimentally determined for the relevant film material.

5.2 Application to porous silica low-k films

The further modified substrate effect model (4a) and (4b) was examined its application to porous silica low-k films. The scaling function γ was determined first by means of a linear regression of the relative moduli E_f/E_s of three of four porous silica low-k films with different film modulus resulted from difference in porosity.

For convenience of calculation, we rewrite (4a) as

$$E_f(h_k) = \frac{\left(1-\nu_f^2\right)\cdot\frac{E_s}{\left(1-\nu_s^2\right)}\cdot E_{comp}(h_k)\cdot\left(1-e^{-\alpha_k\frac{\left(t-h_k/\gamma\right)}{a_k}}\right)}{\frac{E_s}{\left(1-\nu_s^2\right)}-E_{comp}(h_k)\cdot\left(e^{-\alpha_k\frac{\left(t-h_k/\gamma\right)}{a_k}}\right)} \quad (5)$$

$$(k = 0, 1, 2, \ldots N).$$

In (5), h_k (k = 0, 1, 2,…N) are the data points of the experimental indentation depth, $E_{comp}(h_k)$, the measured value of composite film/substrate modulus at indentation depth h_k, a_k and α_k are the corresponding square root of contact area and scaling function of King model $\alpha(a_k/t)$, respectively.

The procedure of determination of the scaling function γ is as follows:

(A) Input the Young's modulus of silicon substrate of 170 GPa and the measured value at the indentation depth of $h_0 \approx$ 5-8 nm as the intrinsic Young' modulus of the film $E_f(h_0)$ in (5) as well as film thickness t.
(B) Input the initial guess of the scaling parameter γ.
(C) Calculate $E_f(h_k)$ by (5) with γ for the data points of the indentation depth h_k., substituting the measured $E_{comp}(h_k)$.
(D) Calculate the sum of the square of variance of E_f between neighbouring indentation depths, $[E_f(h_{k+1})-E_f(h_k)]^2$.
(E) Find the value of γ that minimize the sum of the square of variance.
(F) Perform the procedure (A) to (E) for three porous silica film samples. Then γ_i (i=1, 2, 3) can be obtained for three samples.
(G) Determine the coefficients c and d in γ of (4b) by means of a linear regression of γ_is.

This calculation of a 'nonlinear' least square fitting was performed by using the solver tool of Microsoft EXCEL. The results of the calculation are shown in Fig. 9.

The correction of indentation depth dependence of Young's modulus was subsequently performed for remaining one porous silica sample in terms of our model. We solved the nonlinear equation (5) combined with (4b) for $E_f(h)$ by using the experimental value of $E_{comp}(h)$ as an input, assuming only the Young's modulus of silicon substrate E_s of 170 GPa. The solution showed good convergence within an error of 10^{-5}, even though the initial value of E_f of 15 GPa corresponding to the experimental value at the indentation depth of 20 % of the film thickness was assumed. As shown in Fig. 10, the result was in good

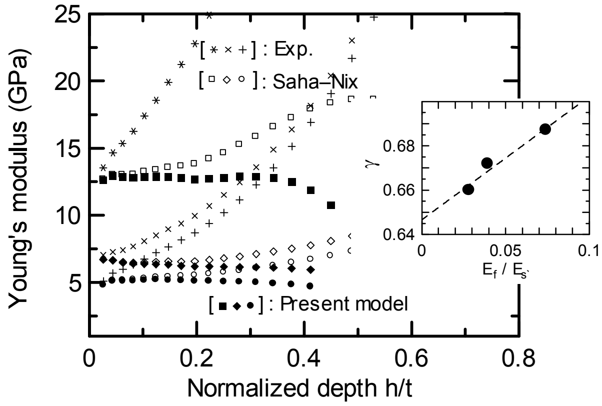


Fig. 9. Plot of Young's moduli as functions of normalized depth. Crossed symbols indicate the experimental composite Young's modulus used in the calculation, open symbols represent corrections by Saha-Nix model and filled symbols show estimation by the present model. Inset shows the scaling function γ in the present model as a function of relative modulus E_f/E_s obtained by a least square method.

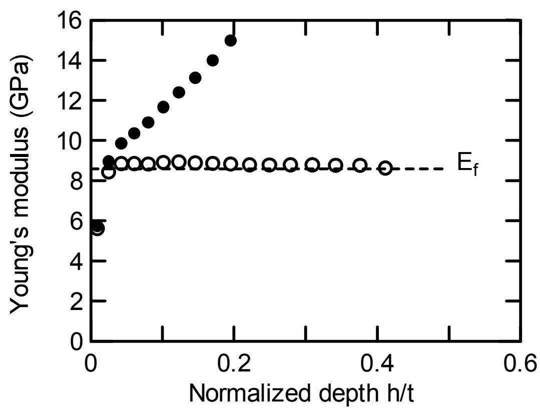


Fig. 10. Plot of 'intrinsic' film modulus predicted by the present model (5) for the porous silica low-k of thickness of 300nm (○). Only the Young's modulus of silicon substrate 170 GPa was assumed in the calculation. The experimental values of composite modulus used in the calculation (●) are also plotted for comparison. The horizontal dotted line shows the average of the experimental values in the indentation depth from 5 to 8 nm regarded as the experimental intrinsic film modulus.

agreement with the 'intrinsic' elastic modulus of the film E_f estimated from the average of the experimental values in the indentation depth from 5 to 8 nm. The agreement holds up to the indentation depth of 30 % of the film thickness. It is suggested that our model enables to evaluate the Young's modulus of low-k film on silicon substrate by a small number of measurements with a larger indentation depth by using even a nanoindentation testing machine that is not equipped with CSM.

6. CONCLUSIONS

We established a method to extract the intrinsic Young's modulus of low-k films from nanoindentation data of composite film/substrate modulus having a strong dependence on indentation depth due to stiffness of silicon substrate. Our model successfully extracted the intrinsic Young's modulus of the porous silica low-k film for the nanoindentation data at the indentation depths up to 30 % of the film thickness.

The present model is an improvement of the model proposed by Saha-Nix [5], introducing an additional scaling parameter for indentation depth. The scaling parameter introduced in present work is a linear function of the elastic modulus of film material relative to that of substrate. Thus, our equation includes parameters to be empirically determined as the model of Doerner and Nix [3]. Consequently, application of the present model is also limited to the specific film materials investigated in this study. However, from a view point of practical use, we think that our method is quite effective in evaluation of the 'film-only' Young's modulus of porous silica low-k films being of great interest in advanced semiconductor material industries.

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