Mechanical properties characterization of PECVD nitride films with rapid thermal annealing using finite element simulation

Chih-Yang Chiang[1,*], Hong-Yi Yan[2], Zhi-Kai Huang[2] and Kuo-Shen Chen[1,2]

[1] Institute of Nanotechnology and Microsystems Engineering
[2] Department of Mechanical Engineering, National Cheng Kung University
Tainan, Taiwan
[*] Corresponding Author, Email: q26981024@mail.ncku.edu.tw

ABSTRACT

This paper presents the development of a new model for extracting elastic modulus from nanoindentation testing data and its subsequent application for deterimining the Young's modulus of plasma-enhanced chemical vapour deposited (PECVD) silicon nitride films after rapid thermal annealing (RTA), processed between 400 and 800 ^{o}C. Primary test data indicate that the modulus varied significantly with different RTA temperatures. However, the traditional data reduction scheme cannot exclude the substrate effect and it results in depth-dependent modulus. In this work, the substrate effect can be significantly reduced by the proposed model and the accurate data reduction implies that it is possible to provide more understanding to accurate correlate the fabrication process parameters and the resulted mechanical behavior of thin films in the future.

Keywords: nanoindentation, substrate effect, rapid thermal annealing, silicon nitride, finite element analysis

1 INTRODUCTION

Plasma-enhanced chemical vapour deposited (PECVD) silicon nitrides films have been widely used in microelectromechanical systems (MEMS) and integrated circuit (IC) devices as a mask or a structural material. The mechanical properties of PECVD nitride subjected to thermal processing are traditionally important information for designing and evaluating the structural integrity of the above devices. In addition, since the PECVD process is usually performed at a lower temperature, it usually results in a porous microstructure in the deposited films. Such a microstructure is usually required to be further densified by a high temperature annealing process. This heat treatment also change the associated material properties, such as elastic modulus, hardness, toughness, and residual stresses of the material. In order to avoid excess amount of thermal budget, the annealing is usually performed by rapid thermal annealing (RTA) scheme. Hence, it is important to obtain the relation between RTA parameters and mechanical properties of silicon nitrides .

Recently, instrumentation micro- and nano-indentations, due to their testing scale and relative ease of specimen preparation, have been widely used for characterising mechanical properties of micro- and nano-scale materials. Traditionally, the applied load (i.e. *P*) and penetration depth (i.e. *d*), obtained from experimental data can be used to deduce the mechanical properties using the relation proposed by Oliver and Pharr [1]. However, it must emphasized here that the model mentioned above [1] is primarily used for bulk materials. For a thin film attached to a substrate, this bulk model could result in considerable errors. In particular, previous investigation indicated that the indentation behavior for hard film/soft substrate and soft film/hard substrate combinations could be quite different [2]. Based on Oliver and Pharr's model [1], Yan *et al.*[3] found that the annealing temperature could influence the elastic modulus of nitride film and the reduced Young's modulus could be smaller or larger than that of the silicon substrate. However, since the formula proposed by Oliver and Pharr has been founded to be overestimated with increasing penetration depth on the case of soft film/hard substrate and is opposite on the other combination [2], the results obtained by using that model could potentially result in considerable errors. In order to reduce the substarte effect for better data reduction, a modified model should be developed.

King [4] developed a model of punch that attempted to account for the influence of the substrate compliance by including a term to represent the contribution of the substrate. The resulted reduced elastic modulus of the film become a mixture quantities of both the film and the substrate varying between the limits of "film-only" properties and "substrate-only" properties. Saha extened King's analysis to nanoindentation with Berkovich indenter. Although it showed considerable improvement over the original Oliver and Pharr model, this modified King model did not work well for larger penetration depth [5]. Strictly speaking, King's work is for a flat punch, where the projected area is unchanged during the entire indentation. This is not true for nanoindentation using conic, Berkovich, and even sopherical indenters. As a result, based on Saha-King [4,5] model and by considering the presence of a substrate and the fact of depth-dependent projected area, a modified model for extracting Young's modulus of films from the associated nanoindentation test data is proposed and developed. Subsequent finite element (FE) simulations indicate that this model can effectively eliminate the

artificial depth-dependent Young's modulus and retrieve accurate results.

2 SPECIMEN FABRICATION AND NANOINDENTATION

The overall experimental flow is shown in Fig. 1. PECVD nitrides (500 nm) were deposited on 4-in. silicon wafers using a Nano-Architect Research/ BR-2000LL PECVD system located at the semiconductor research center of National Chiao-Tung University (NCTU) at temperatures between 250 and 400 ^{o}C with a pressure of 5 Torrs based on the following reaction formula:

$$SiH_4(g) + NH_3 \xrightarrow[\Delta]{RF+N_2(g)} SiN_x : H(s) + 3H_2(g) \quad (1)$$

After deposition, the wafers were die-sawed into square chips (10 mm × 10 mm). Specimens were allowed to undergo RTA processes using an Annealsys/AS-One 100 RTA system (Annealsys Corporation, Montpellier, France) at temperatures of 400, 600 and 800 ^{o}C with an annealing period of 60 seconds. The elastic moduli of these nitride specimens were subsequently characterized by a MTS Nano Indenter XP nanoindentor with a Berkovich indenter (MTS Corp., Eden Prairie, MN, USA) using a load-controlled manner. By contact mechanics, it is possible to correlate the initial unloading slope S with the reduced elastic modulus E_r as [1]:

$$S = \gamma \frac{2}{\sqrt{\pi}} E_r \sqrt{A(h_c)} \quad (2)$$

Where γ equals 1.034 for Berkovich indenter [4] and the reduced modulus E_r is defined as:

$$\frac{1}{E_r} = \frac{1-\nu_s^2}{E_s} + \frac{1-\nu_i^2}{E_i} \quad (3)$$

where the subscripts 'i' and 's' represent the indenter and the substrate, respectively.

By setting the peak load at 30.8 mN, the experimental *P–d* curves were obtained. As shown in Fig. 2, it can be found that the curvature of the *P–d* curves rises with RTA temperature, which implies the hardness also increases.

On the other hand, by directly using Oliver, the Young's modulus can also be found. However, as mentioned before that the results would be questionable due to substrate effect. Instead, the proposed model will be used to perforrm the data reduction here.

Fig. 3 shows the fundamental flow of the proposed model. A dimensional analysis is performed to reduce the governing variables. Based on Saha-King model, a reasonable from to rerpresent the reduced modulus is proposed with coefficients to be determined. Finite element models are then constructed and extensive software experiemnts are followed to extract the coefficients. Finally, standard benchmark problems are used to test if the model can eliminate the substrate effect. Once it is validated, we then use the model to perform the data reduction of the experimental data addressed in this work.

3 CONSTRUCTION OF FINITE ELEMENT MODELS

The finite element model, shown in Fig. 4 using commercial available FEA package ABAQUS STANDARD V. 6.10, represents a nanoindentation processes using a cone-shape rigid indenter. By matching the contact area with a half-angle θ of 70.3°, this model can also represent the indentation process using a Berkovich indenter [6].

Since the region of interest, which in the vicinity of the indenter tip, is very small in comparison with the overall specimen size, it is convinient to model the structure far away from the indentation zone by using infinite elements. Furthermore, it is also reasonable to use axisymmetric model for reducing the computational cost. In this model, regular axisymmetric quadratic finite elements (CAX8) is used to the model the region of interest, and the infinite elements (CINAX5R) are adopted to model the far region (total element number: 35627). The interface of thin film and substrate is defined as perfectly bonded, which means there are no delamination or slippage occured. The interaction between the indenter and the top surface of the thin film is defined as a sliding surface, which is applicable for friction or non-friction contact. Based on previous investigation [7], for friction coefficient less than 0.2, the effect from friction contact in the nanoindentation can be negliglble [7]. The mesh is shown in Fig. 4.

Two loading steps: indenter loading and unloading, are used to simulate nanoindentation. During loading, the rigid indenter moves along the y-axis and penetrates the film up to a preset maxium depth; during unloading, the indenter returns to its initial position. The relationship between the simulation and the experiment of standard specimen (fused silica) is shown in Fig. 5. and both results match each other very well. This implies that the current mesh density and model are appropriate.

4 EXPERIMENTS AND DATA REDUCTIONS

As mentioned above, the model proposed a reasonable expression of the reduce moduls shown in Eq.(4). I.e.,

$$\frac{1}{E_r} = \frac{1-\nu_i^2}{E_i} + \frac{1-\nu_f^2}{E_f}(1-e^{-\beta}) + \frac{1-\nu_s^2}{E_s}e^{-\beta} \quad (4)$$

where E_i, E_f and E_s are the elastic modulus of the indenter, film, and substrate, respectively. Eq.(4) is similar to the original form proposed by King and Saha [4,5]. However, in King and Saha [4,5], in those work the exponent term is assumed to be explicitly related to the ratio between the effective indenter width (a) and the film thickness (t), which is only valid for punch. As a result, we use a scaling function β to repressent the relation. By extensive finite element analyses, it is possible to obtain β numerically, which is a power function of normalized projected contact area, as shown in Fig. 6.

It is found that the indentation test is not very sensitive to differences to the strain hardening rate, but is quite sensitive to the variations in either Young's modulus (E) or yield strength (Y), with the loading portion sensitive to both Y and E, and the unloading primarily sensitive to E [8]. As a result, for the purpose of simplicity, it is reasonable to assume the film being a elastic-perfect plastic material by neglecting possible strain-hardening effect. By limiting the parameters to be varied to Y and E and fitting the P–d curve, we are able to obtain the material properties.

One way to evaluate the accuracy of the proposed model is to perfrom finite element nanoindentation simulations with different peak penetration depths with assumed constant material properties. Table I provides neceaary material data for the FE simulation. The simulated nanoindentation responses are then reduced by both the Oliver and Pharr and the proposed models and the results are shown in Fig. 7. Under perfect situations, the extracted Young's modulus should be independent from the indentation depth. However, as one can see, the Oliver and Pharr's results exhibit strong depth-dependent artifacial phenomena. On the contrary, the modulus obtained by the proposed model are nearly constant for different penetration depth. This validates the effectiveness of the proposed model.

Finally, the extracted Young's modulus of PECVD silicon nitride with different RTAs are shown in Fig. 8. It can be seen that for RTA temperatures below 400 ^{o}C, the modulus are relatively insensitive to RTA temperatures. However, once the RTA temperatures exceed 400 ^{o}C, elastic modulus increases significantly with temperature. By the proposed model, it is expected that the accurate Young's modulus can be obtained, which serves critical fundamental information for subsequent structural design and reliability evaluation of microsystems.

5 DISCUSSION AND CONCLUSION

The modulus shown in Fig. 7 depend on the penetration depth. However, it should be emphasised that the depth-dependent property is not a physically phenomenon but merely the consequence of using an improper data reduction model. It can be found that although the proposed model behaves much better than the Oliver and Pharr model, the substrate effect is still not fully eliminated. More sophisticated modeling works are suggested as future investigations to further improve the data reduction process.

This paper presents the characterization of elastic modulus of PECVD silicon nitride films subjected to RTA processing between 400 and 800 ^{o}C by menas of experiments and modeling. For eliminating artifacts presented in the experimental data due to strong substrate effects. The proposed model demonstated its ability to retrieve accurate material modulus by excluding the substrate effect.

Silicon nitride is an important mechanical materials for microsystems. Accurate information on the correlation between thermal processing parameter and the elastic modulus of nitide is vital for smicrosystem structure design and reliability analysis. As a result, we believe that the contribution of this work is to provide a better approach to accurate extract material data by removing key artifacts. And this provides fundations for subsequent micro mechanics analysiss and optimization for micro fabrication process design.

ACKNOWLEDGEMENT

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Table 1: Input mechanical parameters for nanoindentation finite element analysis.

	without RTA	400 ^{o}C	600 ^{o}C	800 ^{o}C
E_f (GPa)	90	102	170	200
Y_f (GPa)	4.5	4.5	7	9
υ_f	0.27	0.27	0.27	0.27
E_s (GPa)	180	180	180	180
Y_s (GPa)	5	5	6	6
υ_s	0.25	0.25	0.25	0.25

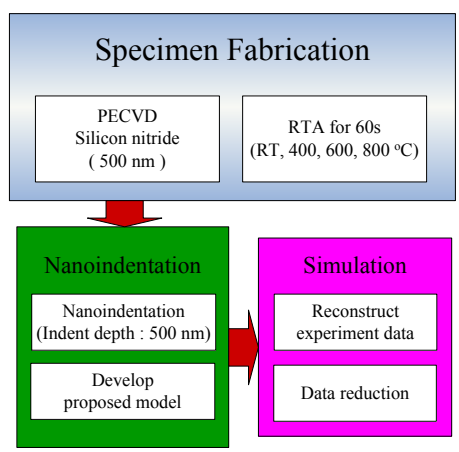

Figure 1: The overall research structure.

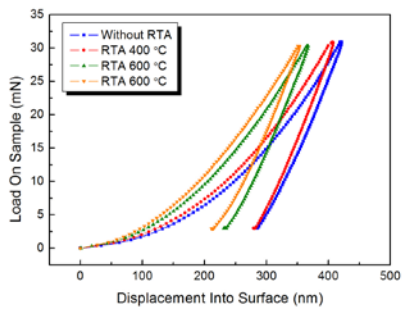

Figure 2: Experimental data of nanoindentation on silicon nitride films at various temperatures.

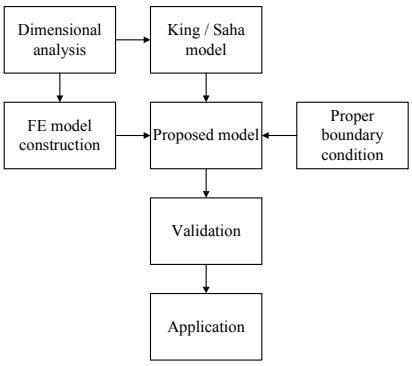

Figure 3: Flow chart for developing the proposed model.

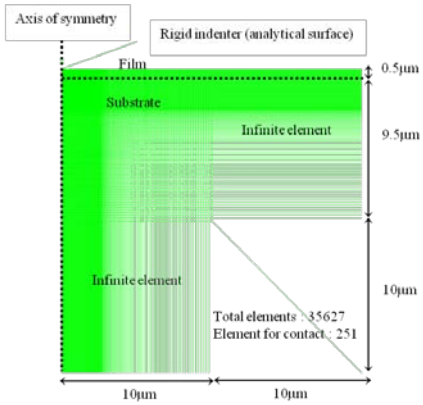

Figure 4: The finite element mesh and indicated dimensions

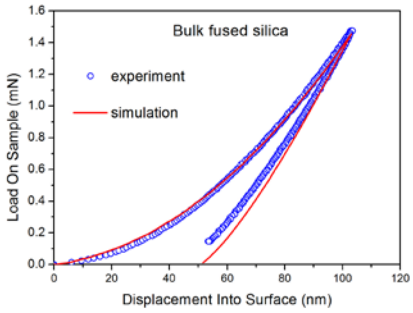

Figure 5: Comparison between the experiment and simulation of indentation for a standard fused silica specimen.

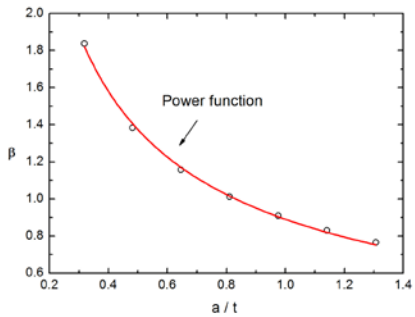

Figure 6: Function β as a function of normalize projected contact area.

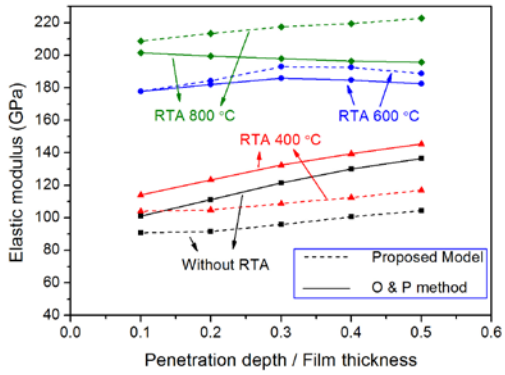

Figure 7: Elastic modulus extraction *w.r.t.* penetration depth by using the proposed and Oliver/Pharr models.

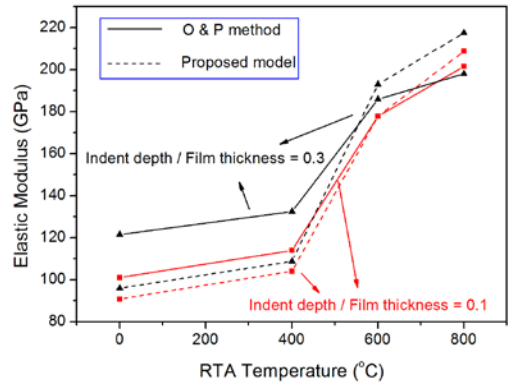

Figure 8: Elastic modulus of nitride *w.r.t.* RTA temperature at different indent depths using the proposed and Oliver/Pharr models

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