Plate model to evaluate interfacial adhesion of anisotropy thin film in CSN test

XUEJUN ZHENG[*,‡]
Institute of Fundamental Mechanics and Materials Engineering, Xiangtan University, Xiangtan, Hunan 411105, People's Republic of China; Faculty of Material Photoelectronic Physics, Xiangtan University, Xiangtan, Hunan 411105, People's Republic of China
E-mail: xj_zheng2002@hotmail.com

S. F. DENG
Faculty of Material Photoelectronic Physics, Xiangtan University, Xiangtan, Hunan 411105, People's Republic of China

YICHUN ZHOU
Institute of Fundamental Mechanics and Materials Engineering, Xiangtan University, Xiangtan, Hunan 411105, People's Republic of China; Faculty of Material Photoelectronic Physics, Xiangtan University, Xiangtan, Hunan 411105, People's Republic of China

XIN TAN
Faculty of Material Photoelectronic Physics, Xiangtan University, Xiangtan, Hunan 411105, People's Republic of China

PENG JIANG
Engineer School, National University of Defence Technology, Changsha, Hunan 410073, People's Republic of China

Cross-sectional nanoindentation (CSN) is a new *test* especially designed for measuring interfacial adhesion of thin film with nanoindenter and scanning electron microscopy (SEM) [1, 2]. Sanchez *et al*. [1] proposed the indentation analytical models, in which interfacial crack area of ceramic thin film was considered as isotropic or simplified as an assembly of a tapered beam and circular plate, to measure interfacial adhesion of Si_xN_y/SiO_2 ceramic–ceramic thin film system. However, many materials in engineering technology are anisotropic, especially ferroelectric/piezoelectric thin films used in microelectromechanical systems (MEMS) [3]. In this paper, based on the anisotropic cylindrical plate theory in which the effect of transverse shear is considered, an analytical expression for the critical energy release rates per unit new interfacial crack area is derived. Interfacial adhesion of SiN and PZT thin film evaluated by the anisotropic plate model is compared with the results evaluated by the previous models. The investigation provides the foundation to estimate the interfacial adhesion of anisotropic ceramic thin film systems in CSN test.

In CSN test of multiplayer thin film structures [1], one side of the pyramidal indenter was parallel to the interface between thin film and oxide-layer/substrate and the point of the indentation load was at a distance d from the interface (shown in Fig. 1a). The three-sided indenter was driven perpendicularly into the cross section and then was taken out. The indentation position and indentation depth Δ (or indentation load) can be adjusted, and a schematic diagram of the interfacial fracture procedure is shown in Fig. 1. The interfacial cracks, characteristic of brittle materials loaded with pyramidal indenters [4], propagated on loading through the silicon substrate and the strong silicon–silicon oxide interface. In Fig. 1b, a wedge produced by the indenter is composed of the multilayer thin film/oxide-layer/substrate structure, and the original wedge length without the interfacial crack is defined as $2b$. When the crack reaches the weak thin film–silicon oxide interface, it tilts out of the original planes following the thin film–silicon oxide interface. Finally, when the indentation depth Δ (or the indentation load W) increases, the maximum length of the interfacial crack at thin film–silicon oxide interface is $2a$. In Fig. 1c, Δ and w_0 are defined as the indentation depth and the maximum vertical displacement, respectively. The delamination of ceramic thin film can be simplified to a bending plate given in Fig. 2. The coordinate origin and plane or θ coincide respectively with the orthotropic pole and plate mid-plane, and z-axis perpendicular to the mid-plane is the elastic principal axis.

According to the basic assumptions of anisotropic plate theory [5], the displacement and stress fields can

[*] *Present address*: Faculty of Material Photoelectronic Physics, Xiangtan University, Xiangtan, Hunan 411105, People's Republic of China.
[‡] Author to whom all correspondence should be addressed.

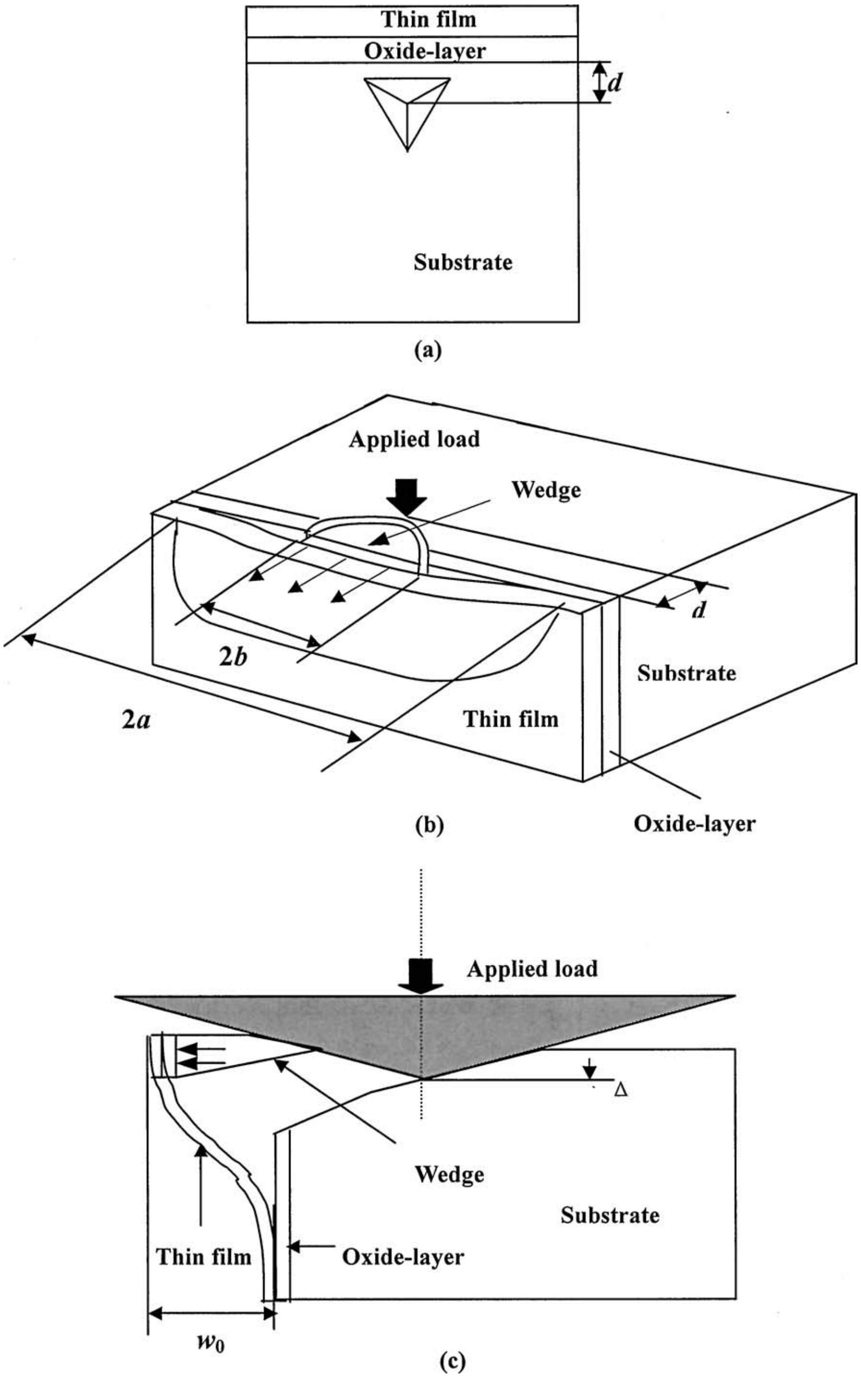

Figure 1 (a) Orientation and placement of the indentation, (b) schematic of CSN test configuration, and (c) side elevation of CSN test configuration.

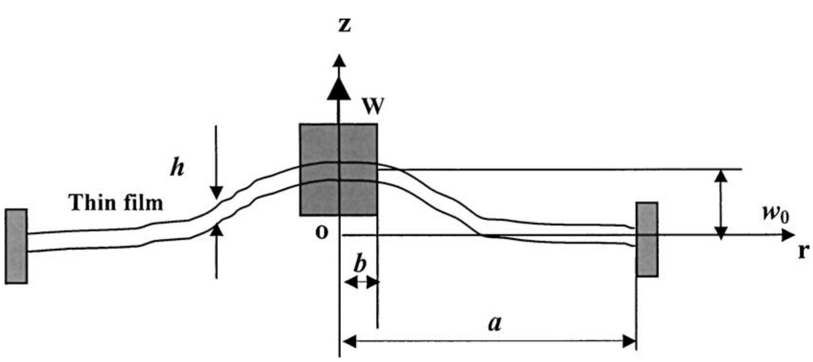

Figure 2 Schematic of the elastic bending circular plate model.

be expressed as

$$\left.\begin{aligned}
u(r,\theta,z) &= -z\frac{\partial w(r,\theta)}{\partial r} + \frac{z}{2}\left(\frac{h^2}{4} - \frac{z^2}{3}\right)\frac{1}{G_{rz}}\varphi(r,\theta),\\
v(r,\theta,z) &= -\frac{z}{r}\frac{\partial w(r,\theta)}{\partial \theta} + \frac{z}{2}\left(\frac{h^2}{4} - \frac{z^2}{3}\right)\frac{1}{G_{\theta z}}\psi(r,\theta),\\
w(r,\theta,z) &= w(r,\theta),\\
\tau_{rz} &= \frac{1}{2}\left(\frac{h_2}{4} - z^2\right)\varphi,\\
\tau_{\theta z} &= \frac{1}{2}\left(\frac{h_2}{4} - z^2\right)\psi,
\end{aligned}\right\} \tag{1}$$

where $\varphi(r,\theta)$, $\psi(r,\theta)$ are arbitrary unknown functions, $G_{rz}, G_{\theta z}$ shear elastic modules, and h is the thickness of thin film. There is only axial load $Z^{\pm}(r,\theta)$, and $X^{\pm} = Y^{\pm} = 0$ on the upper and lower surfaces of the plate. Substituting the geometric and constitutive equations into equilibrium equations, w, φ, ψ can be given as follows,

$$\left.\begin{aligned}
&\frac{1}{r}\frac{\partial(r\varphi)}{\partial r} = -\frac{12}{h^3}(Z^+ + Z^-),\\
&D_1\frac{\partial}{\partial r}\left(r\frac{\partial^2 w}{\partial r^2}\right) - D_2\frac{1}{r}\frac{\partial w}{\partial r} + \frac{h^2}{10G_{rz}}\\
&\quad\times\left[D_2\frac{\varphi}{r} - D_1\frac{\partial}{\partial r}\left(r\frac{\partial \varphi}{\partial r}\right)\right] + \frac{h^3}{12}(r\varphi)\\
&\quad = A_2\frac{h^2}{10}(Z^+ + Z^-) - A_1\frac{h^2}{10}\frac{\partial}{\partial r}r(Z^+ + Z^-),\\
&\frac{\partial^2\psi}{\partial r^2} + \frac{1}{r}\frac{\partial \psi}{\partial r} - \left(\frac{1}{r^2} + \frac{5hG_{\theta z}}{6D_k}\right)\psi = 0.
\end{aligned}\right\} \tag{2}$$

where A_1, A_2, D_1, D_2, D_k are parameters determined by the elastic coefficients of thin film. For transversely isotropic materials, the internal forces and moments can be simplified as

$$M_{r\theta} = \frac{h^2}{10}\left(\frac{d\psi}{dr} - \frac{\psi}{r}\right), \quad Q_\theta = \frac{h^3}{12}\psi(r,\theta). \tag{3}$$

Because of the force boundary conditions $Z^+ = Z^- = 0$ and axis-symmetry $M_{r\theta} = Q_\theta = 0$, one can determine the following functions by Equations 2 and 3

$$\varphi(r) = \frac{c_1}{r} \quad \text{and} \quad \psi(r,\theta) = 0. \tag{4}$$

where c_1 is a integral constant. Substituting them into Equation 3, the differential equation is

$$\frac{d^3 w}{dr^3} + \frac{1}{r}\frac{d^2 w}{dr^2} - \frac{1}{r^2}\frac{dw}{dr} = -\frac{h^3}{12D}\frac{c_1}{r}. \tag{5}$$

where, $D = \frac{h^3}{12}Q_{11}$, Q_{11} is the elastic coefficient of thin film. The displacement boundary conditions can be expressed as

$$\begin{aligned}
&(w)_{r=b} = w_0, \quad \left(\frac{dw}{dr}\right)_{r=b} = 0,\\
&(w)_{r=a} = 0, \quad \left(\frac{dw}{dr}\right)_{r=a} = 0.
\end{aligned} \tag{6}$$

The strain ε_{ij} and stress σ_{ij} fields could be obtained by the geometric equations and constitutive equations, respectively. Considering transverse *shear*, the elastic strain energy of semicircular bending plate will be given as

$$U = \frac{1}{2}\int \frac{1}{2}\sigma_{ij}\varepsilon_{ij}\,dV = \frac{\pi w_0^2}{4}F(\lambda, Q_{ij}). \tag{7}$$

$$\begin{aligned}
&F(\lambda, Q_{ij})\\
&= \frac{h^3(1-\lambda^2)^3 - 4h^3\lambda^2(1-\lambda^2)\ln^2\lambda}{3a^2\left[\frac{1}{2}(1-\lambda^2)^2 - 2\lambda^2\ln^2\lambda\right]^2}\\
&\quad\times Q_{11} - \frac{4h^5(1-\lambda^2)^2\ln\lambda}{15a^4\left[\frac{1}{2}(1-\lambda^2)^2 - 2\lambda^2\ln^2\lambda\right]^2}\frac{Q_{11}^2}{Q_{44}}\\
&\quad+ \frac{17h^7(1-\lambda^2)^3}{630a^6\lambda^2\left[\frac{1}{2}(1-\lambda^2)^2 - 2\lambda^2\ln^2\lambda\right]^2}\\
&\quad\times\frac{(Q_{11} - Q_{12})Q_{11}^2}{Q_{44}^2}
\end{aligned} \tag{8}$$

where $\lambda = b/a$, Q_{11}, Q_{12}, Q_{44}, and V are the elastic coefficients of thin film materials and circular plate volume. Then, the critical energy release rate can be written

$$\begin{aligned}
G(\Delta a) &= -\left(\frac{U(A+\Delta A) - U(A)}{\Delta A}\right)_{w_0}\\
&= -\frac{1}{\pi a}\left(\frac{\partial U}{\partial a}\right)_{w_0}
\end{aligned} \tag{9}$$

where A is the delamination area corresponding to one-half of circular plate. If the original wedge radius b, the maximum radius of interfacial delamination area a, and the maximum vertical displacement w_0 are obtained, the critical energy release rate per unit crack can be calculated by Equation 9.

Generally, the critical energy release rate per unit crack is used to characterize interfacial adhesion of thin film/substrate system [6, 7]. According to the experimental results in Ref. [1], the measured parameters such as b, a (A can be calculated), and w_0 could be recorded by SEM, and then the interfacial adhesion of Si_xN_y/SiO_2 thin film can be calculated by Equation 9 and the results are listed in Table I. It is obvious that interfacial adhesions evaluated by the anisotropic plate model are greater than evaluated by the assembly tapered beams and isotropic plate models [1]. The main reason is that the transversal shear effect is considered, and the interfacial adhesion evaluated by the anisotropic plate model increases due to the contribution of shear strain energy.

To understand the anisotropic plate model, transversely isotropic PZT thin film materials are discussed

TABLE I Interfacial adhesion of SiN thin film evaluated by the different models

Test	1	2	3	4	5	6	7	8	9	10	11	12	13	14
A (μm^2) [1]	386	348	499	772	736	663	784	945	1305	1787	2350	2157	2273	2760
G-assembly beam (J/m^2) [1]	3.0	1.8	2.2	1.6	2.9	2.1	2.6	2.6	1.3	1.4	1.4	1.4	2.2	1.3
G-isotropic plate (J/m^2) [1]	3.1	1.9	2.3	1.7	3.0	2.2	2.6	2.6	1.4	1.4	1.4	1.4	2.1	1.4
G-anisotropic plate (J/m^2)	3.9	2.7	3.1	2.1	3.4	2.6	3.1	3.1	1.6	1.6	1.6	1.7	2.6	1.6

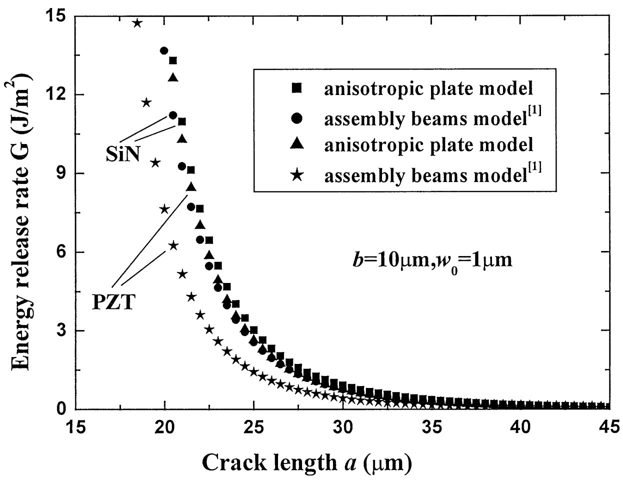

Figure 3 The variation of interface adhesion G with the crack length a, when the size of extrapolated composite b and maximum vertical displacements w_0 are 10 and 1000 μm, respectively.

as an example. The parameters corresponding to the critical state are assumed as $b = 10$ μm and $w_0 = 1000$ μm, and the elastic constants of PZT thin film are determined as $Q_{11} = 13.9 \times 10^{10}$ N/m^2, $Q_{12} = 7.78 \times 10^{10}$ N/m^2, and $Q_{44} = 2.56 \times 10^{10}$ N/m^2 [8]. The variation of interfacial adhesion with the maximum radius of interfacial delamination area a are described as Fig. 3. One can conclude that interfacial adhesions evaluated by the anisotropic plate and assembly tapered beams models differ little for isotropic SiN thin film, however there *is* a large difference for transversely isotropic PZT thin film.

In summary, a plate model based on anisotropic plate theory is proposed to evaluate interfacial adhesion of transversely isotropic thin films. For isotropic thin films, the interfacial adhesions evaluated by the anisotropic plate model are larger than that evaluated with the previous models, because the *shear* strain energy is considered in the former model. In CSN test, the anisotropic plate model should be used to evaluate the interfacial adhesion of anisotropic ceramic thin films.

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