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Young's Modulus Measurement of Thin Films by Resonant Frequency Method Using Magnetostrictive Resonator

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Abstract: At present, there are many methods about Young's modulus measurement of thin films, but so far there is no recognized simple, non-destructive and cheaper standard measurement method. Considering thin films with various thicknesses were sputter deposited on the magnetostrictive resonator and monitoring the resonator's first-order longitudinal resonant frequency shift both before and after deposition induced by external magnetic field, an Young's modulus assessing method based on classical laminated plate theory is presented in this paper. Using the measured natural frequencies of Au, Cu, Cr, Al and SiC materials with various thicknesses in the literature, the Young's modulus of the five materials with various thicknesses are calculated by the method in this paper. In comparison with the Young's modulus calculated by the other methods, it is found that the calculated Young's modulus for various thicknesses are in good agreement with the Young's modulus values in the literature. Considering the simple and non-destructive characteristics of this method, which can effectively describe the effect of the thickness on the Young's modulus, it has the potential to become a standard assessing method of thin film Young's modulus.

Keywords: Magnetostrictvie resonator, Resonant frequency method, Young's modulus of thin film

1 Introduction

With the development of modern science and technology, the researches about mechanical behavior and characteristics of microelectromechanical systems (MEMS) received increasing attention [De Wolf (2003), Spearing (2000), Van Spengen (2003)].

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In order to better improve the functionality and reliability of MEMS devices, first of all it needs to be understood that the inherent mechanical properties are related to the thin film as interconnect and packaging materials in MEMS, and that these mechanical properties, especially Young's modulus, are the key data in the numerical simulation and design. In many cases, the Young's modulus of thin films is not the same with corresponding bulk material, so the accurate measurement method has become an important problem and received many concern from engineering field [Arzt (1998), Liang, Li and Jiang (2002)].

So far, there are many methods, including destructive and non-destructive methods, to evaluate the Young's modulus of thin film. Destructive methods [Allen et al (1987), Espinosa, Prorok and Fischer (2003), Espinosa, Prorok and Peng (2004), Greek et al (1999), Haque and Saif (2002), Kiesewetter et al (1992), Prorok and Espinosa (2002), Petersen and Guarnieri (1979), Sharpe, Yuan and Edwards (1997), Stolken and Evans (1998), Tsuchiya et al (1998), Vlassak and Nix (1992), Ye et al (1996), Yu, Hsu and Fang (2005)] usually finishing the test structure cause the destruction of structure. Obviously, they require relatively long and complex fabrication processes, as well as expensive equipment to measure [Chinmulgund, Inturi and Barnard (1995), Huang and Spaepen (2000), Schneider and Tucker (1996), Zhou, Zhou and Yang et al (2006)], and the thin film structure and material properties may be affected by production process. In contrast, non-destructive method [Bellan and Dhers (2004), Cros, Gat and Saurel (1997), Dirras et al (2004), Peraud et al (1997), Schneider and Tucker (1996)] uses direct measurement, and does not change the thin film properties in any way. In fact, although there are many methods used to evaluate Young's modulus of thin film, there is no recognized standard method, which is simple, inexpensive, and non-destructive [Liang and Prorok (2007)]. With the rapid development of materials and electric science, all kinds of smart materials, such as piezoelectric materials [Arockiarajan and Menzel (2007); Chen, Kao and Chen (2009); Deepak and Ranjan (2009); Wu, Chiu and Wang (2008); Wu and Liu (2007)], shape memory alloys [Erdogan and Murat (2009)], giant magnetostrictive materials [Ghosh and Gopalakrishnan (2004); Zhou and Zhou (2007), Zhou, Zheng and Zhou (2006), Zhou, Zhou and Zheng (2007, 2009)], have been widely studied and used. Giant magnetostrictive material, especially, is highly attractive for sensor, actuator and resonator etc, not only due to its small size and low prices, but also because of its passive and wireless nature. So a new measurement technique that employs a magnetostrictive resonator is presented by Schmidt and Grimes (2001), shown to be suitable for evaluating elastic properties of thin film and of the potential to become the standard technique in terms of assessing elastic properties. Liang and Prorok (2007) adopt this technique to measure the elastic properties of various thin films and offer a discussion of the errors involved. However, the Young's modulus calculated by Schmidt and Grimes (2001) and Liang and Prorok (2007) using GMM resonator technique is the same for various thicknesses of thin film. In fact, the Young's modulus may not be the same for different film thicknesses.

For the characteristics that Young's modulus changes with the thickness of thin film, and considering the thin film and the magnetostrictive resonator as a laminated structure, a Young's modulus evaluating method using magnetostrictive resonator driven by external magnetic field is proposed in this paper. Substituted the Young's modulus obtained by Liang and Prorok (2007) into this method, the calculated result about the relative frequency shift versus film thickness for various materials has significant deviation with experiment data, which obviously due to the fact that the Young's modulus in Liang's paper does not change with the thin film thicknesses. Then using the frequency data measured by experiment, the Young's modulus for various materials and thicknesses is calculated by this method. It is found that the calculated Young's modulus for various thicknesses in this paper is in good agreement with the results determined by the other methods in literature. And this method has the potential to become a standard evaluating method of thin film Young's modulus considering its characteristics such as being simple, non-destructive, and able to effectively describe the effect of the thickness on the Young's modulus,.

2 Basic Equations

The laminated structure including the magnetostrictive resonator is shown in Fig. 1, and thin film sputter deposition on the magnetostrictive resonator. A harmonically alternating or transient magnetic field excitation generates mechanical oscillations in the magnetostrictive resonator. The oscillations are mainly along the length direction of the resonator. The Young's modulus of thin film can evaluate by measuring the change of the first-order longitudinal natural frequency before and after coating thin film. The length, width and thickness of laminated structure are l, b and t respectively, where the thicknesses of the magnetostrictive resonator and thin film are t_1 and t_2 respectively.

Considering the magnetostrictive resonator shown in Fig. 1 as a laminated structure, according to classical laminated plate theory, the strain of structure determined by middle surface strain ε^0 and curvature κ is as follows

$$\begin{Bmatrix} \varepsilon_x \\ \varepsilon_y \\ \gamma_{xy} \end{Bmatrix} = \begin{Bmatrix} \varepsilon_x^0 \\ \varepsilon_y^0 \\ \gamma_{xy}^0 \end{Bmatrix} + z \begin{Bmatrix} \kappa_x \\ \kappa_y \\ \kappa_{xy} \end{Bmatrix} = \{\varepsilon^0\} + z\{\kappa\} \tag{1}$$

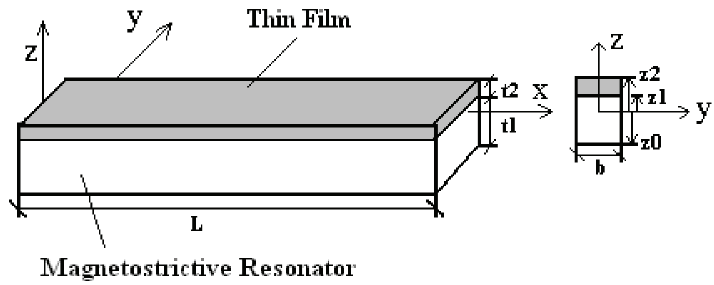

Figure 1: The Sketch of laminated structure including giant magnetostrictive resonator.

Where, the middle surface strain ε^0 and curvature κ are as follows,

$$\begin{Bmatrix} \varepsilon_x^0 \\ \varepsilon_y^0 \\ \gamma_{xy}^0 \end{Bmatrix} = \begin{Bmatrix} \frac{\partial u}{\partial x} \\ \frac{\partial v}{\partial y} \\ \frac{\partial u}{\partial y} + \frac{\partial v}{\partial x} \end{Bmatrix}, \quad \begin{Bmatrix} \kappa_x \\ \kappa_y \\ \kappa_{xy} \end{Bmatrix} = \begin{Bmatrix} -\frac{\partial^2 w}{\partial x^2} \\ -\frac{\partial^2 w}{\partial y^2} \\ -2\frac{\partial^2 w}{\partial x \partial y} \end{Bmatrix} \tag{2}$$

In which, u and v are the displacement of middle surface in x and y direction respectively, and w is the deflection.

For magnetostrictive materials, the relation for magnetic field, magnetization and strain are nonlinear and coupling (Zheng and Liu (2005), Liu and Zheng (2005)). Considering the magnetostrictive resonator working for given pre-stress and bias magnetic field, the simplified linear constitutive relationships can be used as follows.

$$\{\boldsymbol{\sigma}\} = [\mathbf{Q}]\{\boldsymbol{\varepsilon}\} - [d]\{H\} \tag{3}$$

Where, $[\mathbf{Q}]$ is stiffness matrix, $[d]$ is magnetostrictive coupling coefficients and $\{H\}$ is external magnetic field.

Therefore, the constitutive relationships of all layers can be uniformly written as the form,

$$\{\boldsymbol{\sigma}\} = [\mathbf{Q}](\{\boldsymbol{\varepsilon}\} - \{\boldsymbol{\Lambda}\}) = [\mathbf{Q}]\{\boldsymbol{\varepsilon}\} - [\mathbf{Q}]\{\boldsymbol{\Lambda}\} \tag{4}$$

Where, the stress vector $\{\boldsymbol{\sigma}\}$ and the magnetostrictive strain vector $\{\boldsymbol{\Lambda}\}$ are respectively shown as follows,

$$\{\boldsymbol{\sigma}\} = \begin{bmatrix} \sigma_x & \sigma_y & \tau_{xy} \end{bmatrix}^T, \quad \{\boldsymbol{\Lambda}\} = \begin{bmatrix} \Lambda_x & \Lambda_y & \Lambda_{xy} \end{bmatrix}^T \tag{5}$$

In Eq.4, the term $[\mathbf{Q}]\{\mathbf{\Lambda}\}$ is equivalent stress generated by the magnetostrictive resonator driven by external magnetic field, and this term only exists for magnetostrictive layer.

In comparison of Eq. 4 and Eq. 5, for magnetostrictvie layer, we have

$$[\mathbf{Q}]\{\mathbf{\Lambda}\} = [d]\{H\} \tag{6}$$

Substituting Eq. 1 into Eq. 4 and integrating the resulting equation along the z direction, we get the matrix form of the constitutive relations as follows,

$$\begin{Bmatrix} N \\ M \end{Bmatrix} = \begin{bmatrix} A & C \\ C & D \end{bmatrix} \begin{Bmatrix} \varepsilon^0 \\ \kappa \end{Bmatrix} - \begin{Bmatrix} N_\Lambda \\ M_\Lambda \end{Bmatrix} \tag{7}$$

In which,

$$(A_{ij}, C_{ij}, D_{ij}) = \int_{-t/2}^{t/2} \left(1, z, z^2\right) Q_{ij} dz \tag{8}$$

$$N = \int_t \{\sigma\} dz, M = \int_t \{\sigma\} z dz \tag{9}$$

$$N_\Lambda = \int_{t_a} [Q]\{\Lambda\} dz = \int_{t_a} [d]\{H\} dz, \quad M_\Lambda = \int_{t_a} z[Q]\{\Lambda\} dz = \int_{t_a} z[d]\{H\} dz \tag{10}$$

The matrixes A, C and D are tensile stiffness, coupling stiffness and bending stiffness of laminated plate respectively. From the view of physical meaning, we know that N and M are internal force and moment respectively, while N_Λ and M_Λ are parts contributed by the magnetostrictive layer only.

When an external magnetic field is applied, the total strain energy of laminated structure can be expressed by the form

$$U = \frac{1}{2} \iint\limits_A \left\{ \varepsilon^{0^T} \kappa^T \right\} \begin{bmatrix} A & C \\ C & D \end{bmatrix} \begin{Bmatrix} \varepsilon^0 \\ \kappa \end{Bmatrix} dA - \iint \left\{ \varepsilon^{0^T} \quad \kappa^T \right\} \begin{Bmatrix} N_\Lambda \\ M_\Lambda \end{Bmatrix} dA \tag{11}$$

Denote $\{r\} = \begin{bmatrix} u & v & w \end{bmatrix}^T$, we have

$$\begin{Bmatrix} \varepsilon^0 \\ \kappa \end{Bmatrix} = [L]\{r\} \tag{12}$$

Where

$$L = \begin{bmatrix} \frac{\partial}{\partial x} & 0 & \frac{\partial}{\partial y} & 0 & 0 & 0 \\ 0 & \frac{\partial}{\partial y} & \frac{\partial}{\partial x} & 0 & 0 & 0 \\ 0 & 0 & 0 & -\frac{\partial^2}{\partial x^2} & -\frac{\partial^2}{\partial y^2} & -\frac{\partial^2}{\partial x \partial y} \end{bmatrix}^T \tag{13}$$

Then the displacement vector at any point of laminated plate can be written as

$$\begin{Bmatrix} X \\ Y \\ Z \end{Bmatrix} = \begin{bmatrix} 1 & 0 & -z\frac{\partial}{\partial x} \\ 0 & 1 & -z\frac{\partial}{\partial y} \\ 0 & 0 & 1 \end{bmatrix} \begin{Bmatrix} u \\ v \\ w \end{Bmatrix} = [\Psi]\{r\} \tag{14}$$

So, the total kinetic energy of laminated structure can be formulated as the matrix forms

$$T = \iiint\limits_V \frac{1}{2}\rho \left\{\dot{X} \quad \dot{Y} \quad \dot{Z}\right\} \begin{Bmatrix} \dot{X} \\ \dot{Y} \\ \dot{Z} \end{Bmatrix} dV = \iiint\limits_V \frac{1}{2}\rho \dot{r}^T [\Psi]^T [\Psi] \dot{r} dV \tag{15}$$

Divide the plate into $n \times m$ elements of four nodes. To each node, five degrees of freedom are employed, i.e. (u, v, w, α, β), where $\alpha = \partial w/\partial y$ and $\beta = -\partial w/\partial x$. Denote $\delta_i = \begin{bmatrix} u_i & v_i & w_i & \alpha_i & \beta_i \end{bmatrix}$ (i=1, 2, 3, 4), the node displacement column in the element can be written as $\{\delta^e\} = \begin{bmatrix} \delta_1 & \delta_2 & \delta_3 & \delta_4 \end{bmatrix}^T$. The middle surface displacement in the element can be formulated by the interpolation polynomial of the form $u = \sum_{i=1}^{4} N_{ui} u_i$, $v = \sum_{i=1}^{4} N_{vi} v_i$ and $w = \sum_{i=1}^{4} \left(N_{wi} w_i + N_{\alpha i} \alpha_i + N_{\beta i} \beta_i\right)$. Where, N_{ui}, N_{vi}, N_{wi},$N_{\alpha i}$ and $N_{\beta i}$ represent shape function. Then the matrix form of middle surface displacement can be expressed as

$$\{r\} = \begin{bmatrix} N_1 & N_2 & N_3 & N_4 \end{bmatrix} \begin{Bmatrix} \delta_1^T \\ \delta_2^T \\ \delta_3^T \\ \delta_4^T \end{Bmatrix} = [N]\{\delta^e\} \tag{16}$$

Where,

$$N_i = \begin{bmatrix} N_{ui} & & & & \\ & N_{vi} & & & \\ & & N_{wi} & N_{\alpha i} & N_{\beta i} \end{bmatrix} \tag{17}$$

Substituting Eq. 16 into Eq. 12, we have

$$\begin{Bmatrix} \varepsilon^0 \\ \kappa \end{Bmatrix} = [L]\{r\} = [L][N]\{\delta^e\} = [B]\{\delta^e\} \tag{18}$$

In which $[B] = [L][N]$. Denote the Lagrange function by $\Pi = (T - U)$ when ignoring the external force work. Then substituting Eqs. 16 and 18 into Eqs. 11 and 15, and the resulting equations into the definition formula, we get

$$\begin{aligned}\Pi =&(T-U)\\ =&\frac{1}{2}\iint\limits_{A}(\dot{\delta}^{eT}(\int_t(\Psi N)^T\rho\Psi N dz)\dot{\delta}^e-\\ &\delta^{eT}B^T\begin{bmatrix}A & C\\ C & D\end{bmatrix}B\delta^e+2\delta^{eT}B^T\begin{bmatrix}N_\Lambda\\ M_\Lambda\end{bmatrix})dA\end{aligned} \tag{19}$$

Appling the Hamiltonian principle of the form $\delta\int_{t_1}^{t_2}\Pi dt=0$ to the above equation, we get a system of ordinary differential equations of the dynamic system in the following matrix form

$$[M]\{\ddot{a}\}+[K]\{a\}=\{F\} \tag{20}$$

Where, $\{a\}$ and $\{\ddot{a}\}$ are the columns consisting of node displacement and relevant accelerations; $[M]$ and $[K]$ are the global mass and stiffness matrices respectively, and $\{F\}$ is the global load column generated by magnetic field. Their element matrices or columns can be explicitly formulated by

$$[M]^k=\iint\limits_{S_e}(\int_t(\Psi N)^T\rho\Psi N dz)dS \tag{21}$$

$$[K]^k=\iint\limits_{S_e}B^T\begin{bmatrix}A & C\\ C & D\end{bmatrix}BdS \tag{22}$$

$$\{F\}=\iint\limits_{S_e}B^T\begin{bmatrix}N_\Lambda\\ M_\Lambda\end{bmatrix}dS \tag{23}$$

If all of material constants are given, the natural frequencies of laminated structure can be obtained by modal analysis according to Eq. 20. If the natural frequencies measured by experiment and the Young's modulus of thin film as the only unknown quantity, it can be calculated by Eq. 20 too.

3 Results and Discussions

Here, we give a case study of a laminated structure with a size of $8.0\times 1.6mm^3$, and the thickness of the magnetostrictive layer is $28\mu m$. The commercially magnetostrictive material, Metglas^{TM} 2826MB, is selected as the magnetostrictive resonator and its material parameters are taken as: the Young's modulus E_m = $105GPa$ and the density ρ_m = $7.9\times 10^3 kg\,/\,m^3$. The density of five thin films, i.e. Au, Cu, Cr, Al and SiC, are selected as $\rho_{\text{Au}} = 19.34 \times 10^3 kg\,/\,m^3$, $\rho_{\text{Cu}} = 8.96 \times 10^3 kg\,/\,m^3$, $\rho_{\text{Cr}} = 7.19 \times 10^3 kg\,/\,m^3$, $\rho_{\text{Al}} = 2.70 \times 10^3 kg\,/\,m^3$, $\rho_{\text{SiC}} = 3.20 \times 10^3 kg\,/\,m^3$. The results of the thickness of the five thin films and the corresponding measured frequencies are selected from Liang's paper (Liang and Prorok (2007)).

For the uncoated magnetostrictive resonator, the experiment measured frequency of the first-order longitudinal vibration is 276.8kHz. According to the calculated formula $f_m = \frac{1}{2l}\sqrt{\frac{E_m}{\rho_m(1-\nu^2)}}$, the calculated frequency of the first-order longitudinal vibration is 238.86kHz. But the calculated frequency of the first-order longitudinal vibration using the method in this paper is 277.2kHz. Clearly, the calculated result by the method in this paper is more accurate than the formula.

According to the change of frequency before and after deposition, an approximate calculation formula for Young's modulus of thin film is presented in Liang's paper. And the calculated Young's modulus for five materials are $E_{\text{Au}} = 75.9$GPa, $E_{\text{Cr}} = 130.8$GPa, $E_{\text{Cu}} = 139.2$GPa, $E_{\text{Al}} = 55.4$GPa and $E_{\text{SiC}} = 160.4$GPa respectively. Obviously, the Young's modulus determined in Liang's paper are the same for various film thicknesses, and the calculated Young's modulus is effective only when the thickness of thin film is much smaller than the thickness of magnetostrictive layer. In fact, the Young's modulus should be changed along with the film thickness increasing in the deposition process. Substituting these values of Young's moduli into the method in this paper, the calculated frequencies of the first-order longitudinal vibration for various thicknesses and the experimental results in Liang's paper are compared and shown in Fig. 2. The solid point represents the calculated results and the hollow point represents the experiment measurement results in this figure. From the Fig. 2, we can see obviously that the calculated results and experimental measurement results have a great deviation, which shows that the Young's modulus should not be constant under various thicknesses and the approximation Young's modulus given by Liang and Prorok (2007) is not reliable when the thickness of thin film has larger changes.

Substituting the different film thicknesses and corresponding frequencies of experimental measurement into the method in this paper, the calculated Young's modulus versus thickness of thin films is shown in Fig. 3. From this figure we can see that the Young's modulus for various thicknesses is not constant and has nonlinear relationships with the thicknesses. The compared results with the Young's modulus calculated by the other methods and the calculated Young's modulus for various thicknesses determined by the method in this paper are compared and shown in Fig 4., where the solid lines are on behalf of the range of Young's modulus in the literature (i.e Au: 53$\sim$130GPa; Cr: 107$\sim$275GPa; Cu: 90$\sim$129GPa; Al: 47$\sim$90GPa; SiC: 100$\sim$452GPa), the square on behalf of the calculated Young's modulus by Liang and Prorok (2007), and the circle on behalf of the calculated Young's mod-

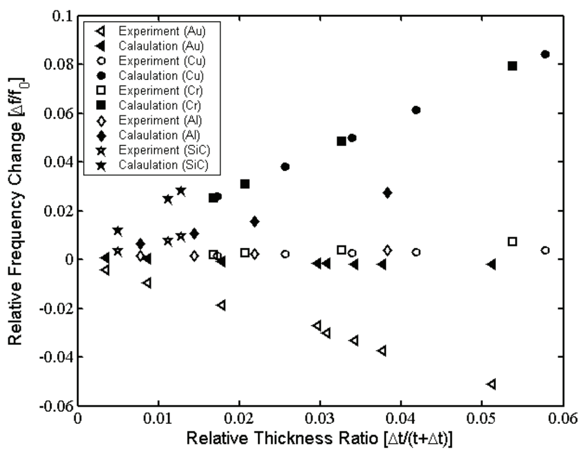

Figure 2: The relative frequency shift versus film thickness percentage of the laminated structure.

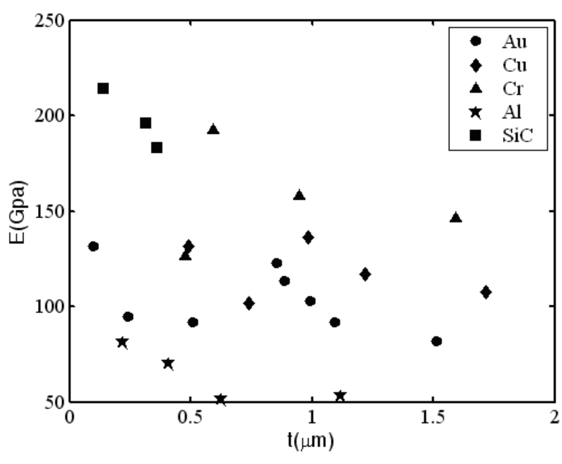

Figure 3: The Young's modulus changes with the thickness of thin films for various materials.

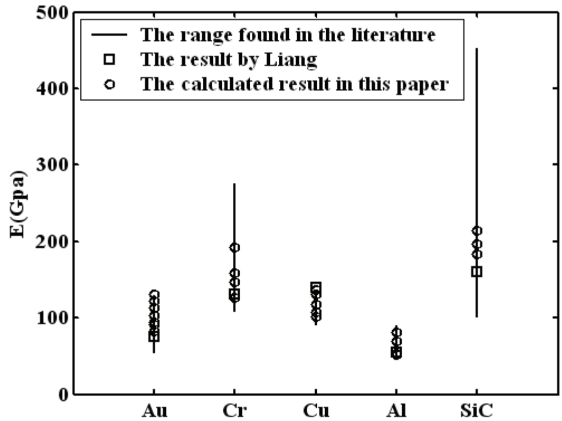

Figure 4: The evaluated Young's modulus for each material falls in the range found in the literature.

ulus for various thicknesses determined by the method in this paper. As shown by Fig. 4, the calculated results in this paper are coincident with the results in the literature.

4 Conclusions

A Young's modulus evaluating method for various thin film thicknesses by monitoring the magnetostrictive resonator's first-order longitudinal resonant frequency shift both before and after deposition induced by external magnetic field, is presented in this paper based on classical laminated plate theory. Substituting the experimental data of frequencies into this method, the Young's modulus for various thicknesses is calculated. From the results figures, it is found that the calculated Young's modulus for various thicknesses is in good agreement with the Young's modulus calculated by the other methods in the literature. This technique that employs a magnetostrictive resonator has shown to be adept at evaluating thin film Young's modulus and with the potential to become the standard measurement technique considering the characteristics of this method such as being simple, inexpensive to perform, requiring no post-deposition fabrication, non-destructive, and able to effectively describe the effect of the thickness on the Young's modulus.

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