Effect of temperature gradient on simultaneously experimental determination of thermal expansion coefficients and elastic modulus of thin film materials

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Some specific experimental methods to simultaneously determine the thermal expansion coefficients $\alpha_T$ and biaxial elastic modulus $E_T/(1-\nu_T)$ of thin film materials have been reported recently. In these methods, the deflections or the curvature change of the thin films, deposited on two different types of circular disks with known material properties, generally can be measured with a variety of optical techniques. The temperature-dependent deflection behaviors of thin films are then obtained by heating the samples in the range from room temperature to a slightly higher temperature level at which the physical properties and microstructures of thin film materials still remain unchanged. By using the relations between stress, deflection, and temperature, the physical properties of thin films can be finally calculated by using the slopes of two lines in the stress versus temperature plot. These relations, however, are formulated under the condition of uniform temperature rise. If the heating processes of samples are conducted in the condition that there exists a small steady-state temperature gradient along the thickness of samples due to the effect of natural heat convection on the upper surface of thin film, the formulation mentioned above shall be modified. It is found that the deflection of sample induced by the small temperature gradient along the thickness due to natural heat convection is very significant and comparable to that induced by uniform temperature rise. Consequently, if the effect of this temperature gradient is carelessly disregarded in physical modeling, a significantly different value of elastic modulus may be misleadingly obtained. Some cases are exemplified and illustrated to show the influence of temperature gradient on the evaluation of material properties. © 2004 American Institute of Physics. [DOI: 10.1063/1.1789629]

I. INTRODUCTION

Thin film materials have wide applications in a variety of electronic devices and hard coating technology of machine parts such as antireflection coatings, optical waveguides, metal oxide semiconductor devices, insulators in electronic devices, and narrow-bandpass filters. It is well known that the physical properties of thin film material are quite different from bulk material.1–3 The discrepancy between them may be as high as one order in magnitude. Consequently, an accurate determination of mechanical properties of thin film materials, such as thermal expansion coefficient and elastic modulus, becomes extremely important and has attracted extensive attention in recent years. However, only few results have been reported.

The total residual stress on a thin film material is generally composed of intrinsic stress and thermal stress. The former is generated during film growth due to relatively complicated microscopic mechanisms, such as the different spacing of atoms in a growing film, the incorporation of excess vacancies, the presence of impurities, and bombardment by energetic particles, while the latter results from the difference in the thermal expansion coefficients between adjacent layers. Residual stresses induced in the thin film material significantly influence not only the mechanical performance of coatings such as spallation resistance, thermal cycling life, and fatigue properties but also the optical, electrical, and magnetic behaviors of layer devices due to the cracking, interfacial delamination, and the change of physical properties due to their stress and deformation dependence in nature. A reliable evaluation of intrinsic and thermal stresses is dependent on the accurate values of thermal expansion coefficient and Young’s modulus of thin film materials.

Generally, elastic modulus and thermal expansion coefficient of thin film materials can be individually and independently determined by different experimental methods. For instance, a variety of methods, such as the tensile test method,4 the bending tests,5 the mechanical deflection method,6 the suspended flexural vibration method,7 the resonance frequency method,8 and the photoacoustic measuring technique,9 have been proposed to determine the elastic modulus of the coating materials. On the other hand, the optical levered laser technique,10 the bending beam technique,11 and the capacitance cell method12 have been reported to determine the thermal expansion coefficient of thin film materials. Recently, the method to use a sample curvature technique to measure thermal stresses of thin films of the same material deposited on two different substrates with known properties has been reported.13–17 The temperature-dependent deflection behaviors of thin films are obtained by

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heating the samples in the range from room temperature to a slightly higher temperature level at which the physical properties and microstructures of thin film materials still remain unchanged. By analyzing the thermal stress data as a function of temperature, the thermal expansion coefficients $\alpha_F$ and biaxial elastic modulus $E_F/(1 - \nu_F)$ of thin film materials can be related to the deflection of the circular substrate at a distance $R$ from the center of the substrate by using the modified Stoney’s formula

$$\sigma = \frac{E_s d_e^2 \delta}{3(1 - \nu_s) R^2 d_F},$$

where $\sigma$ is the stress in the thin film which includes both the intrinsic stress $\sigma_i$, produced during film deposition, and the thermal stress $\sigma_{th}$ due to thermal expansion mismatch between the film and the substrate; $\delta$ is the average deflection of the substrate measured at the radius $R$ on the substrate due to the action of stress $\sigma$; $E_s$ and $\nu_s$ are the Young’s modulus and Poisson’s ratio of the substrate, respectively; $d_s$ and $d_F$ are the thicknesses of the substrate and the thin film layer, respectively.

**II. MECHANICAL MODEL**

For a structure with a very small film to substrate thickness ratio, generally, it can be satisfactorily assumed that an isotropic homogeneous stress distribution is induced in the film layer. The internal stress in thin film layer, $\sigma$, can then be related to the deflection of the circular substrate at a distance $R$ from the center of the substrate by using the modified Stoney’s formula

$$\sigma = \frac{E_s d_e^2 \delta}{3(1 - \nu_s) R^2 d_F}.$$  

where $\sigma$ is the stress in the thin film which includes both the intrinsic stress $\sigma_i$, produced during film deposition, and the thermal stress $\sigma_{th}$ due to thermal expansion mismatch between the film and the substrate; $\delta$ is the average deflection of the substrate measured at the radius $R$ on the substrate due to the action of stress $\sigma$; $E_s$ and $\nu_s$ are the Young’s modulus and Poisson’s ratio of the substrate, respectively; $d_s$ and $d_F$ are the thicknesses of the substrate and the thin film layer, respectively.

**A. Samples with uniform temperature distributions along thickness**

If the temperature is uniformly distributed in both the substrate and the thin film, the stress in the thin film can be written as

$$\sigma = \sigma_i + \sigma_{th} = \sigma_i + (\alpha_s - \alpha_F) \frac{E_F}{1 - \nu_F} (T_2 - T_1),$$

where $\alpha$ is the thermal expansion coefficients; subscripts $S$ and $F$ denote the physical properties of the substrate and the film layer, respectively; and $T_2$ and $T_1$ are the temperatures of stress measurement and film deposition, respectively. Therefore, if $\delta$ represents the thermal deflection of substrate only due to uniform temperature rise from $T_1$ to $T_2$, by using Eqs. (1) and (2), its relation with material properties and temperature can be written as

$$\delta = \left[ \frac{3d_F E_F/(1 - \nu_F)}{d_s E_s/(1 - \nu_s)} \right] [(\alpha_s - \alpha_F)(T_2 - T_1)].$$

If all the physical properties, as shown in Eq. (2), are temperature independent, the slope of the measured stress-temperature curve is equal to

$$\frac{d\sigma}{dT} = \frac{d\sigma_{th}}{dT} = (\alpha_s - \alpha_F) \frac{E_F}{1 - \nu_F}.$$
are misused directly, a very significant error in estimating physical properties of thin film material will be probably taken place.

1. Theoretical temperature distributions along the thickness of the sample

a. Prediction of overall heat transfer coefficient $h_C$. Prediction of $h_C$ in natural convection on a vertical surface of substrate can be accurately performed by using the well-known Squire-Eckert formulation and the equations proposed by Churchill and Chu in the field of natural heat convection. The average overall heat transfer coefficient $h_C$, proposed by Churchill-Chu correlation equation is given by

$$
\overline{h}_C = N_{Ut} \frac{k}{L},
$$

(5a)

where

$$
N_{Ut} = 0.68 + 0.67 \text{Ra}^{1/4}_L \left[ 1 + \left( \frac{0.492}{\text{Pr}} \right)^{9/16} \right]^{-4/9},
$$

(5b)

$$
\text{Ra}_L = \frac{g \beta \Delta T L^3}{\nu \alpha},
$$

(5c)

and

$$
\text{Pr} = \frac{\nu}{\alpha},
$$

(5d)

in which $N_{Ut}$ is the average Nusselt number; $L$ is the vertical length of the plate; $\text{Ra}_L$ is the Reynolds number; $\text{Pr}$ is the Prandtl number; $\nu$ is the kinematic viscosity; $\alpha$ is the thermal diffusivity; $\beta$ is the reciprocal of absolute ambient temperature; $g$ is the gravitational acceleration; and $k$ is the thermal conductivity.

b. Evaluation of steady-state temperature distributions along the thickness of sample. If the sample is originally at uniform temperature $T_I$ and then heated from the bottom surface of the substrate, the temperature distributions along the thickness of the film and the substrate can be accurately evaluated by solving the following steady-state heat conduction equation and associated thermal boundary conditions in which the effect of interfacial thermal contact resistance between the thin film and the substrate is neglected. The one-dimensional steady-state heat conduction equation of the two-layer sample, as shown in Fig. 1(b), along thickness direction, $\xi$, can be expressed as

$$
d^2 T_\xi / d\xi^2 = 0, \quad 0 \leq \xi \leq d_S; \quad d^2 T_F / d\xi^2 = 0, \quad d_S \leq \xi \leq d_S + d_F;
$$

(6)

where $T_\xi(\xi)$ and $T_F(\xi)$ are the temperature distributions along the substrate and the thin film, respectively; the axis $\xi$ is along the thickness of the layered plate and it originated at the bottom surface of the substrate, as shown in Fig. 1(b).

The associated thermal boundary conditions are as follows:

$$
-k_S \frac{dT_S}{d\xi} = q_0 \quad \text{at} \quad \xi = 0, \quad \text{at} \quad \xi = d_S.
$$

(7a)

$$
k_F \frac{dT_F}{d\xi} = h_C (T_F - T_A) \quad \text{at} \quad \xi = d_S + d_F.
$$

(7b)

$$
-k_F \frac{dT_F}{d\xi} = h_C (T_F - T_A) \quad \text{at} \quad \xi = d_S + d_F,
$$

(7c)

$$
T_F = (T_2)_U \quad \text{at} \quad \xi = d_S + d_F,
$$

(7d)

$$
T_S = (T_2)_B \quad \text{at} \quad \xi = 0,
$$

(7e)

where $q_0$ is the heat flux prescribed on the bottom surface of substrate by the hot storage; $R_{th}$ denotes the interfacial thermal contact resistance between the thin film and the substrate; $T_A, (T_2)_U,$ and $(T_2)_B$ are the ambient temperature, the upper surface temperature of thin film layer, and the bottom surface temperature of substrate, respectively.

The solutions of Eq. (6) that satisfy the boundary conditions (7a)–(7e) can be easily obtained as

$$
T_S(\xi) = a_1 + a_2 \xi, \quad 0 \leq \xi \leq d_S;
$$

(8a)

$$
T_F(\xi) = a_3 + a_4 \xi, \quad d_S < \xi \leq d_S + d_F;
$$

(8b)

where

$$
a_1 = (T_2)_U + h_C [(T_2)_U - T_A] (d_F/k_F + d_S/k_S)
$$

$$
+ R_{th} h_C [(T_2)_U - T_A] = (T_2)_B,
$$

(8c)

$$
a_2 = - h_C [(T_2)_U - T_A] / k_S,
$$

(8d)

$$
a_3 = T_2 + h_C [(T_2)_U - T_A] (d_F + d_S) / k_F,
$$

(8e)

$$
a_4 = - h_C [(T_2)_U - T_A] / k_F.
$$

(8f)

If the thickness of film layer is much thinner than that of the substrate, i.e., $d_F \ll d_S$, and the value of interfacial thermal contact resistance between the film and the substrate, $R_{th}$, is much smaller than $d_S/k_S$ in Eq. (8b), then the temperature gradient in the substrate, $a_2$, as shown in Eq. (8c), can be briefly written as

$$
a_2 = dT_S/d\xi \equiv [(T_2)_U - (T_2)_B] / d_S.
$$

(8g)

In convenience of deflection and stress calculations, another coordinate $z$, which is parallel to $\xi$ axis with relation $z = \xi - e$ (where $e$ is distance between the bottom surface and the neutral surface of the structure), is adopted in analysis. Therefore, the temperature distributions in terms of $z$ can be expressed as

$$
T_S(z) = b_1 + b_2 z, \quad -e \leq z \leq d_S - e;
$$

(9a)

$$
T_F(z) = b_3 + b_4 z, \quad d_S - e \leq z \leq d_S + d_F - e;
$$

(9b)

where

$$
b_1 = a_1 + a_2 e, \quad b_2 = a_2, \quad b_3 = a_3 + a_4 e, \quad b_4 = a_4.
$$

(9c)
2. Deflection of two-layer circular sample due to nonuniform temperature rise

The mechanical equilibrium equation of a circular sample with radius $R$ subjected to a lateral loading $p(r)$ can be formulated and expressed as follows:\(^{22}\)

$$\frac{d^2w}{dr^2} - \frac{1}{r} \frac{dw}{dr} + \left( \frac{d^2w}{dr^2} + \frac{1}{r} \frac{dw}{dr} \right) = \frac{p(r)}{D}.$$  (10a)

The associated mechanical boundary conditions are\(^{22}\)

$$w(0) = 0, \quad \frac{d^2w}{dr^2} + \frac{v_s}{r} \frac{dw}{dr} = -\frac{M_T}{D} \text{ at } r = R,$$  (10b)

where $w(r)$ is the flexure of the circular plate; $p(r)$ is the lateral loading density along $z$ axis and equal to zero in our problem; $M_T$ is the thermal moment induced in the specimen as the temperature at the upper surface of thin film is increased from $T_1$ to $T_2$ [i.e., $(T_2)_u$ and $(T_2)_b$ at the upper surface of this film and the bottom surface of substrate, respectively]. $D$ is the flexure rigidity of the plate having the form

$$D = \int_{d_s}^{d_s - e} \frac{E_s}{1 - \nu_s} z^2 dz + \int_{d_s - e}^{d_s + d_s - e} \frac{E_F}{1 - \nu_F} z^2 dz$$

$$= \left[ \frac{E_F d_F}{1 - \nu_F} \right] \left( \frac{d_F^2}{3} + d_f(d_s - e) + (d_s - e)^2 \right)$$

$$+ \left[ \frac{E_s d_s}{1 - \nu_s} \right] \left( \frac{d_s^2}{3} + d_s e + e^2 \right),$$  (11a)

where

$$e = \frac{d_F^2 E_F}{2[(d_F E_F)/(1 - \nu_F) + (d_s E_s)/(1 - \nu_s)]}. $$  (11b)

If the thickness of film layer is much thinner than that of the substrate, i.e., $d_s \ll d_F$, then Eqs. (11a) and (11b) can be, respectively, reduced to

$$D \equiv \frac{1}{12} \left( \frac{E_s d_s^3}{1 - \nu_s} + \frac{1}{4} \frac{E_F d_F d_s^3}{1 - \nu_F} \right), \quad e \equiv \frac{d_s}{2} \left[ 1 - \frac{d_F}{d_s} \frac{E_F}{E_s} \right].$$  (12)

Consequently, by using the boundary conditions in Eq. (10b), the solution of Eq. (10a) can be obtained and written as

$$w(r) = -\frac{M_T r^2}{2D(1 + \nu_s)}. $$  (13)

The thermal bending moment $M_T$ induced in the specimen as the temperature at the upper surface of thin film is increased from $T_1$ to $T_2$ can be written as

$$M_T = \int_{d_s - e}^{d_s - e} z \left( \frac{E_s}{1 - \nu_s} \right) [\alpha_s(T_f(z) - T_1)] dz$$

$$+ \int_{d_s - e}^{d_s + d_s - e} z \left( \frac{E_F}{1 - \nu_F} \right) [\alpha_F(T_f(z) - T_1)] dz$$

$$= (M_T)_{\text{non}} + (M_T)_{\text{uni}},$$  (14a)

where

$$(M_T)_{\text{non}} \equiv \left( \frac{2d_s^2}{12} \frac{E_s a s}{1 - \nu_s} \right) \left[ (T_2)_u - (T_2)_b \right] d_s \left( \frac{E_s a_s}{1 - \nu_s} \right),$$  (14b)

$$(M_T)_{\text{uni}} \equiv \left( \frac{E_F}{1 - \nu_F} \right) \left( \frac{d_F d_F}{2} \right) [(\alpha_s - \alpha_F) [(T_2)_u - T_1]],$$  (14c)

in which $(M_T)_{\text{non}}$ and $(M_T)_{\text{uni}}$ represent the thermal bending moment contributed due to temperature gradient (nonuniform temperature distribution) along the thickness of the substrate and uniform temperature rise in the sample, respectively. Moreover, it can be seen that these two components have the same order in magnitude and shall be considered simultaneously in analysis.

Therefore, the deflection of specimen at $r=R$ due to thermal bending moment can be obtained by substituting Eqs. (15) into Eqs. (14) and expressed as

$$\delta = w(R) = \delta_{\text{non}} + \delta_{\text{uni}},$$  (15a)

where

$$\delta_{\text{non}} \equiv \frac{b_2 \alpha_s R^2}{2} \left( \frac{(T_2)_u - (T_2)_b)\alpha_s R^2}{2d_s} \right),$$  (15b)

<table>
<thead>
<tr>
<th>Substrate</th>
<th>Young’s modulus $E_s$ (GPa)</th>
<th>Poisson’s ratio $v_s$</th>
<th>Thermal expansion coefficient $a_s$ ($°C^{-1}$)</th>
<th>Thermal conductivity $k_s$ (W/m K)</th>
</tr>
</thead>
<tbody>
<tr>
<td>BK-7</td>
<td>81.0</td>
<td>0.208</td>
<td>7.40×10^{-6}</td>
<td>1.5</td>
</tr>
<tr>
<td>Pyrex</td>
<td>62.7</td>
<td>0.200</td>
<td>3.25×10^{-6}</td>
<td>1.3</td>
</tr>
</tbody>
</table>

See Ref. 14.

TABLE II. Temperature distributions and temperature gradient of substrate.

<table>
<thead>
<tr>
<th>Substrate</th>
<th>$(T_2)_u$ (°C)</th>
<th>$(T_2)_b$ (°C)</th>
<th>$dT_2/dz$ (°C/mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>BK-7</td>
<td>31</td>
<td>31.032</td>
<td>0.021</td>
</tr>
<tr>
<td>Pyrex</td>
<td>40</td>
<td>40.112</td>
<td>0.075</td>
</tr>
<tr>
<td>substrate</td>
<td>52</td>
<td>52.239</td>
<td>0.159</td>
</tr>
<tr>
<td></td>
<td>70</td>
<td>70.458</td>
<td>0.305</td>
</tr>
<tr>
<td></td>
<td>31</td>
<td>31.038</td>
<td>0.025</td>
</tr>
<tr>
<td>Pyrex</td>
<td>40</td>
<td>40.129</td>
<td>0.086</td>
</tr>
<tr>
<td>substrate</td>
<td>52</td>
<td>52.276</td>
<td>0.184</td>
</tr>
<tr>
<td></td>
<td>70</td>
<td>70.528</td>
<td>0.352</td>
</tr>
</tbody>
</table>

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uniform terms can be evaluated by subtracting individually linearly related to temperature rise and are easily adopted to shall be modified by using and is calculated by using the following relation: 

\[ \delta_{\text{uni}} = \frac{3dF}{dF} \frac{E_F}{(1 - \nu_F)} \left[ (\alpha_S - \alpha_T)(T_2 - T_1) \right]^2. \] (15c)

It is satisfactorily assumed that the stress in the thin film layer is uniformly distributed due to its very thin thickness and is calculated by using the following relation:

\[ \sigma_{th} = \frac{F_T}{dF} = \frac{2M_TdS}{dF} = \frac{2(M_T)_{\text{non}} + 2(M_T)_{\text{uni}}}{dSdF} \]

\[ = (\sigma_{th})_{\text{non}} + (\sigma_{th})_{\text{uni}}, \] (16a)

where

\[ (\sigma_{th})_{\text{non}} = \frac{b_3dF}{6dF} E_S \frac{\alpha_S}{1 - \nu_S} \frac{[(T_2)_{U} - (T_2)_{D}]dS}{6dF} \left( \frac{E_S \alpha_S}{1 - \nu_S} \right), \] (16b)

\[ (\sigma_{th})_{\text{uni}} = \frac{\left[ (\alpha_S - \alpha_3) [(T_2)_{U} - T_1] \right]}{3(1 - \nu_S)R^2dF}, \] (16c)

where \( F_T \) is the resultant thermal force exerted on the thin film layer. In other words, the relations between displacement, thermal stress, and temperature under the condition of nonuniform temperature distributions along the thickness shall be modified from Eqs. (1) and (3) to (15) and (16), respectively.

It shall be emphasized that since the parameter \( b_2 \) in \((\sigma_{th})_{\text{non}}\) is nonlinearly dependent on temperature rise due to the effect of natural heat convection, only the uniform parts of thermal deflection, \( \delta_{\text{uni}} \), and thermal stress, \((\sigma_{th})_{\text{uni}}\), are linearly related to temperature rise and are easily adopted to determine the material properties of thin film layer. These uniform terms can be evaluated by subtracting individually the nonuniform terms from the total thermal deflection and total thermal stress, respectively. In other words, the Eq. (4) shall be modified by using \((\sigma_{th})_{\text{uni}}\) instead of \(\sigma_{th}\) as follows:

\[ \frac{d(\sigma_{th})_{\text{uni}}}{dT} = \frac{(\alpha_S - \alpha_T)}{1 - \nu_F} \frac{E_F}{1 - \nu_F}. \] (17)

Consequently, two kinds of physical properties of thin film layer, i.e., \(E_F/(1 - \nu_F)\) and \(\alpha_T\), can be easily evaluated by using the samples with the same thin film material deposited on two different substrate materials through Eq. (17). For the specific case of uniform temperature distribution without temperature gradient along the thickness of the substrate, i.e., \(b_2 = 0\) [i.e., \((T_2)_U = (T_2)_D = T_2\)], then \(\delta_{\text{non}} = (\sigma_{th})_{\text{non}} = 0\), the corresponding maximum deflection of plate at \(r = R\) and stress in the thin film layer then completely become identical to Eqs. (1) and (3), respectively.

### III. EXAMPLES UNDER INVESTIGATION

To evaluate the effects of temperature gradient, the literature, which simultaneously determines the thermal expansion coefficient and the elastic modulus of Ta \(_2\)O \(_5\) thin film using phase shifting interferometry, is exemplified and studied again. In this reference, the samples, which were deposited with the same thin film material upon two different substrates, were heated from the bottom of the substrate by a hot stage linked with an electric temperature controller; while a thermocouple was attached to the upper surface of thin film layer to sense the temperature variation over there. Moreover, a phase shifting Twyman-Green interferometer apparatus was installed in the middle of the open space surface of thin film layer to optically detect the deformation fringes of samples. Since all the experiments were conducted in an open room space, the upper surface of the thin film was mainly cooled by the effect of natural heat convection. Consequently, a temperature gradient along the thickness of the samples will be inherently introduced even in the condition of steady-state heat conduction. The physical properties of two different kinds of substrate are listed in Table I. Experimental results show that the shape of the bare BK-7 substrate is upwardly concave, while the bare Pyrex substrate is convex. After film deposition, the shape of the BK-7 coated substrate is less concave and thus presents compressive film stress. On the other hand, the shape of the Pyrex coated

<table>
<thead>
<tr>
<th>( T_F ) (°C)</th>
<th>( h_C ) (W/m(^2) K)</th>
<th>( \delta ) (μm)</th>
<th>( \delta_{\text{non}} ) (μm)</th>
<th>( \delta_{\text{uni}} ) (μm)</th>
<th>( \sigma_{th} ) (GPa)</th>
<th>( \sigma_{th} ) (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>26</td>
<td>...</td>
<td>−0.155</td>
<td>0</td>
<td>−0.155</td>
<td>−0.398</td>
<td>−0.398</td>
</tr>
<tr>
<td>31</td>
<td>6.4</td>
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<td>−0.385</td>
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<tr>
<td>40</td>
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<td>0.028</td>
<td>−0.143</td>
<td>−0.295</td>
<td>−0.367</td>
</tr>
<tr>
<td>52</td>
<td>9.2</td>
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<td>−0.206</td>
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<tr>
<td>70</td>
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<td>0.110</td>
<td>−0.134</td>
<td>−0.054</td>
<td>−0.344</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>( T_F ) (°C)</th>
<th>( h_C ) (W/m(^2) K)</th>
<th>( \delta ) (μm)</th>
<th>( \delta_{\text{non}} ) (μm)</th>
<th>( \delta_{\text{uni}} ) (μm)</th>
<th>( \sigma_{th} ) (GPa)</th>
<th>( \sigma_{th} ) (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>26</td>
<td>...</td>
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<td>0</td>
<td>−0.199</td>
<td>−0.391</td>
<td>−0.391</td>
</tr>
<tr>
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<td>0.004</td>
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</tr>
<tr>
<td>40</td>
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</tr>
<tr>
<td>52</td>
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<td>0.030</td>
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<td>−0.419</td>
</tr>
<tr>
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<td>10.4</td>
<td>−0.173</td>
<td>0.056</td>
<td>−0.229</td>
<td>−0.338</td>
<td>−0.450</td>
</tr>
</tbody>
</table>

Reference 14.
substrate is more convex and thus also shows the compressive film stress. The thicknesses of substrate and thin film layer are 1.5 mm and 0.299 µm, respectively. There is only few information on the interfacial thermal contact resistance \( R_{th} \) between the film and the substrate; its value is, in general, within the range of \( 1 \times 10^{-6} - 1 \times 10^{-9} \) m² K/W.\(^{23,24} \) In the present study a maximum value of \( 1 \times 10^{-6} \) m² K/W, which is still much smaller than \( d_3/k_3 (\approx 1 \times 10^{-3} \) m² K/W), is adopted in the temperature calculation. It can be seen that the influence of this maximum interfacial thermal contact resistance on temperature distributions is still less than 0.2% and can be satisfactorily disregarded in the calculation in this case.

If the value of thermal conductivity of the thin film is approximately the same order as the bulk, the temperature distributions, temperature gradients, thermal deflection, and stress due to uniform temperature distribution, i.e., \( (T_2)_{UL}, (T_2)_B, dT_3/dz, \delta_{uni}, \) and \( (\sigma_{th})_{uni} \) are evaluated based on Eqs. (8), (15), and (16) and shown as in Tables II and III, respectively.

The thermal deflections and thermal stresses versus temperature for two different substrate materials are shown in Figs. 2 and 3, respectively. Only the parts of thermal deflection and thermal stress induced by uniform temperature rise can be adopted to determine the physical properties of thin film layer. A simple linearization scheme of least square method is adopted to evaluate the slopes \( d(\sigma_{th})_{uni}/dT \) of two different substrate materials. The physical properties of thin film material \( \text{Ta}_2\text{O}_5 \) evaluated by using Eqs. (17) instead of Eq. (4) in the present study are shown as in Table IV.

However, if we did not take account of the effect of...
temperature gradient along the thickness of the sample and directly adopt Eqs. (1), (3), and (4) under the assumption of uniform temperature rise, the values of biaxial elastic modulus and coefficient of thermal expansion become 1549.4 GPa and $(2.42 \times 10^{-6}) \, ^\circ\text{C}^{-1}$, respectively. The value of the elastic biaxial modulus becomes too high and is almost 15 times more than the bulk or three times more than that obtained in the present study.

It was reported that the physical properties of thin film materials are closely related to the interface conditions between thin film layer and substrate, microscopic structures, porosity ratio, defects, and thickness of thin film layer. The value of interface conditions between thin film layer and substrate, microscopic structures, with which Mother Nature ensures a minimum argument on this issue somewhat still exists. The error of the dual-substrate method may come from several sources. One of the most negative characteristics of this method is that the slope of the measured stress-temperature curve, as shown in Eq. (17), is extremely small. For instance, if the values of $(\alpha_s - \alpha_f)$ and $E_f/(1 - \nu_f)$ are of the order of $10^{-6} \, ^\circ\text{C}^{-1}$ and $10^5 \, \text{GPa}$, respectively, the slope of stress-temperature curve is only about $10^{-4} \, \text{GPa/}^\circ\text{C}$. In other words, a $50 ^\circ\text{C}$ temperature rise in sample only approximately results in $5 \times 10^{-3} \, \text{GPa}$ increase in film stress or $10^{-2} \, \mu\text{m}$ change in thermal deflection. Consequently, the deduced thin film properties are very sensitive to the errors of experimental measurement and substrate property. For instance, if the Young’s modulus and thermal expansion coefficient are 15% higher for BK-7 glass and 15% lower for Pyrex glass than those listed in Table I, the values of biaxial elastic modulus and coefficient of thermal expansion will become 156.6 GPa and $(7.68 \times 10^{-6}) \, ^\circ\text{C}^{-1}$, respectively. Consequently, it is highly desirable to use single crystal structures, with which Mother Nature ensures a minimum variation of substrate structures. The structure and property variations of BK-7 and Pyrex glasses, however, are not easy to predict during their forming processes. Even with single crystal substrates, the measured substrate property data errors can propagate into the thin film properties. The errors of the substrate properties are probably major contributors to the unreasonable results; the deduced biaxial modulus listed in this paper, therefore, may not be reliable.

IV. CONCLUDING REMARKS

In this paper, modified deflection, thermal stress, and temperature relations due to the existence of temperature gradient along the thickness of samples are proposed and formulated in analytical modeling of some experimental methods to simultaneously determine the thermal expansion coefficients $\alpha_f$ and biaxial elastic modulus $E_f/(1 - \nu_f)$ of thin film materials. It is seen that the small temperature gradient induced along the thickness of samples has a very significant influence on the accurate determination of elastic biaxial modulus and thermal expansion coefficient of thin film materials and shall be taken account of in analysis. If the effect of this temperature gradient is carelessly disregarded in physical modeling, a significantly different value of elastic modulus may be misleadingly obtained.

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