Elastic Constants and X-Ray Stress Measurement of Cubic Thin Films with Fiber Texture*

Keisuke TANAKA**, Yoshiaki AKINIWAD**, Toshimasa ITO** and Kaoru INOUE***

A new X-ray method is proposed to measure the residual stress in cubic polycrystalline films having the fiber texture with the axis of $\langle 111 \rangle$, $\langle 100 \rangle$ and $\langle 100 \rangle$ perpendicular to the film surface. The elastic constants of textured thin films were calculated on the bases of Reuss and Voigt models. According to the analysis based on Reuss model, the relation between the strain measured by X-ray, $\varepsilon_{ab}$, and $\sin^2 \phi$ for the equi-biaxial stress is linear for the cases of $\langle 111 \rangle$ and $\langle 100 \rangle$ fiber textures. For the other cases, the relation is non-linear. A method to determine the stress from non-linear relations is proposed. The analysis based on Voigt model gives the linear relation between $\varepsilon_{ab}$ and $\sin^2 \phi$ for any case of fiber texture. In thin films made of materials with low anisotropy, both models give nearly identical relation.

**Key Words:** Experimental Stress Analysis, X-ray Stress Measurement, Residual Stress, Thin Films, Fiber Texture, Reuss Model, Voigt Model, Micromechanics

1. Introduction

The thin film technology has been used in a wide variety of production processes of machine parts as well as electronic devices. The physical and mechanical properties of thin films coated on substrate are very much dependent on the residual stress in thin films. The X-ray diffraction method is expected to be one of the most useful techniques to measure the residual stress in polycrystalline thin films. The classical $\sin^2 \phi$ method of X-ray stress measurement, however, is not necessarily applicable, especially when films have strong texture.

Polycrystalline thin films of cubic crystals often possess the fiber texture whose axis is $\langle 111 \rangle$, $\langle 100 \rangle$ and $\langle 110 \rangle$ perpendicular to the film surface. Table 1 summarizes common fiber textures observed in cubic thin films$^{(1)-(9)}$. Under the assumption of the equi-biaxial residual stress, several modifications of the $\sin^2 \phi$ method have been proposed for the cases of $\langle 111 \rangle$, $\langle 100 \rangle$ and $\langle 110 \rangle$ texture$^{(11)-(13)}$. However, the residual stress state in the thin film is not always equi-biaxial.

Tanaka et al. proposed a new method of X-ray stress measurement of non-equilibiaxial residual stresses in cubic films with the $\langle 111 \rangle$ fiber axis perpendicular to the film surface$^{(10)}$, and successfully applied the new method to determine the residual stress in patterned aluminum thin films sputtered on silicon wafers$^{(12)}$.

In the present paper, the elastic constants of cubic thin film with $\langle 111 \rangle$, $\langle 100 \rangle$ and $\langle 110 \rangle$ fiber textures were determined from the elastic constants of single crystals on the bases of Reuss and Voigt models$^{(13)-(14)}$. The X-ray method is proposed to determine the non-equilibiaxial state of residual stresses in thin films with three kinds of fiber textures.

2. Relation between Stress and Strain

2.1 Transformation of coordinates

First, crystal coordinates are transformed from the original $X$ to $X'$ as shown Fig. 1, where $X'$ is the axis of fiber texture. The transformation matrix $a_\alpha$ ($X'_i=a_\alpha X_i$) is

<table>
<thead>
<tr>
<th>Fiber axis</th>
<th>Materials</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\langle 111 \rangle$</td>
<td>Al$^{(1)}$, Cu$^{(1)}$, Mo$^{(6)}$, TiC$^{(2)}$, TiN$^{(5)}$</td>
</tr>
<tr>
<td>$\langle 100 \rangle$</td>
<td>Cu$^{(2)}$, TiC$^{(2)}$, TiN$^{(5)}$</td>
</tr>
<tr>
<td>$\langle 110 \rangle$</td>
<td>Mo$^{(6)}$, Ni$^{(5)}$, TiC$^{(2)}$, TiN$^{(5)}$</td>
</tr>
</tbody>
</table>

Table 1 Examples of fiber texture of cubic thin films

---

* Received 28th September, 1998
** Department of Mechanical Engineering, Nagoya University, Furo-cho, Chikusa-ku, Nagoya 464-8063, Japan. E-mail: K_tanaka@mech.nagoya-u.ac.jp
*** LCD Module Design Engineering Dept., Toshiba Co. Ltd., 50 Kamiyobe, Yobe-ku, Himeji 671-1295, Japan

JSME International Journal
Fig. 1 Transformation of cubic crystal axes

Fig. 2 Coordinates of specimen, $P_1$, laboratory system, $L_i$, and crystal system, $X_i$

\[
(a_i) = \begin{bmatrix}
\cos \chi_1 \cos \chi_2 & \cos \chi_1 \sin \chi_2 & -\sin \chi_1 \\
-\sin \chi_2 & \cos \chi_2 & 0 \\
\sin \chi_1 \cos \chi_2 & \sin \chi_1 \sin \chi_2 & \cos \chi_1 
\end{bmatrix}
\]  

where $\chi_1$ and $\chi_2$ are the angles shown in Fig. 1.

Figure 2 shows specimen coordinates ($P_1$) and laboratory coordinates ($L_i$) taken on the specimen, where $P_3$ is perpendicular to the film surface. The crystal axis $X_i$ is coincident with the $P_i$ axis, and the $X_i$ axis is rotated from the $P_i$ axis by orientation angle $\beta$ as shown in Fig. 2. The $L_3$ axis is the direction along which the normal strain is measured by X-ray diffraction. The transformation matrices among four coordinate systems are shown in Fig. 3. The transformation matrix $\beta$ is given by

\[
(\beta) = \begin{bmatrix}
\cos \beta & \sin \beta & 0 \\
-\sin \beta & \cos \beta & 0 \\
0 & 0 & 1
\end{bmatrix}
\]

The matrix $R$ is given as the product of $\beta$ and $a$ as

\[
R = R(\beta, a)
\]  

where repeated indices mean the summation convention from 1 to 3. From Fig. 2, the matrix $\omega$ is given as

\[
(\omega) = \begin{bmatrix}
\cos \phi & \cos \phi & \cos \phi \\
-\sin \phi & \cos \phi & 0 \\
\sin \phi & \cos \phi & \cos \phi 
\end{bmatrix}
\]

The relation between strain $\varepsilon_{ij}$ and stress $\sigma_{ij}$ of a crystal with orientation $\beta$ expressed in specimen coordinates is given by

\[
\varepsilon_{ij} = s_{ijkl}^P \sigma_{kl}
\]

where $s_{ijkl}^P$ is the elastic compliance of a crystal in specimen coordinates. The compliance $s_{ijkl}^P$ is obtained by tensor transformation from the crystal compliance $s_{ijkl}$ expressed in the original crystal system $X_i$ as follows:

\[
s_{ijkl}^P = \eta_{ijklpqrs} s_{pqrs}^X
\]

2.2 Reuss model

2.2.1 Mechanical elastic compliance In Reuss model, for elastic deformation of polycrystals, each crystal is assumed to be subjected to the same stress equivalent to the macrostress. The elastic strain in a crystal is given by Eq. (5). The compliance $s_{ijkl}^P$ is a function of the orientation angle $\beta$. The mechanical macrostrain is the mean value of $\varepsilon_{ij}$ averaged from $\beta = 0$ to $2\pi$ as follows:

\[
\bar{\varepsilon}_{ij} = \frac{1}{2\pi} \int_{0}^{2\pi} \varepsilon_{ij} d\beta
\]

because $\sigma_{ij} = \sigma_{ii}$ in Reuss model. Since thin films are transversely isotropic in the $P_1-P_3$ plane, the matrix representation of $\bar{\varepsilon}_{ij}$ has the following form:

\[
(\bar{\varepsilon}_{ij}) = \begin{bmatrix}
\bar{\varepsilon}_{11} & \bar{\varepsilon}_{12} & \bar{\varepsilon}_{13} \\
\bar{\varepsilon}_{12} & \bar{\varepsilon}_{22} & \bar{\varepsilon}_{23} \\
\bar{\varepsilon}_{13} & \bar{\varepsilon}_{23} & \bar{\varepsilon}_{33}
\end{bmatrix}
\]

2.2.2 X-ray strain The strain measured by X-ray diffraction is the average of the normal strain $\varepsilon_{33}$ in the $L_3$ direction. It is related to the strain and stress in a crystal as

\[
\varepsilon_{33} = \omega_{33} + \varepsilon_{33}^P
\]

By taking the average of the strain of crystals which satisfy the diffraction condition, we have

\[
\bar{\varepsilon}_{33} = \omega_{33} + \bar{\varepsilon}_{33}^P
\]

The stress, $\sigma_{ij} = \sigma_{ii}$, is determined from the measure-
ment of $\varepsilon_{k1}$ by the X-ray method.

2.3 Voigt model

2.3.1 Mechanical elastic compliance Voigt model assumes that the strain in each crystal is uniform and equal to the macrostrain\(^{[14]}\). The stress in a crystal is given by

$$\sigma_{\beta} = C_{\beta\kappa} \varepsilon_{k\kappa} \ (11)$$

where $C_{\beta\kappa}$ is transformed from the crystal elastic stiffness $C_{\kappa\kappa}$ as

$$C_{\beta\kappa} = \sum_{\kappa=1}^{3} C_{\kappa\kappa} \cos \theta_{\beta\kappa} \sin \theta_{\beta\kappa}$$

Since $\varepsilon_{k\kappa}$ is equal to macrostrain $\varepsilon_{k1}$, the stress averaged, from $\beta = \phi$ to $2\pi$ is

$$\delta_{\beta} = \frac{1}{2\pi} \int_{0}^{2\pi} \varepsilon_{k1} \sin \theta_{\beta\kappa} \ d\theta_{\beta\kappa}$$

Because of transverse isotropy, the matrix representation of the average stiffness is expressed by

$$\left[ \begin{array}{cccccc} \tilde{c}_{11}^* & \tilde{c}_{12}^* & \tilde{c}_{13}^* & 0 & 0 & 0 \\
\tilde{c}_{12}^* & \tilde{c}_{22}^* & \tilde{c}_{13}^* & 0 & 0 & 0 \\
\tilde{c}_{13}^* & \tilde{c}_{13}^* & \tilde{c}_{33}^* & 0 & 0 & 0 \\
0 & 0 & 0 & \tilde{c}_{44}^* & 0 & 0 \\
0 & 0 & 0 & 0 & \tilde{c}_{44}^* & 0 \\
0 & 0 & 0 & 0 & 0 & \tilde{c}_{44}^* \end{array} \right]$$

(13)

where the independent stiffnesses are $c_{11}$, $c_{12}$, $c_{13}$, $c_{33}$, and $c_{44}$\(^{[15]}\). The inverse matrix of $\tilde{c}_{\beta\kappa}$ denoted by $(\tilde{c}_{\beta\kappa})^{-1}$ is the mechanical elastic compliance by Voigt model.

2.3.2 X-ray strain Since the strain is uniform in Voigt model, the strain measured by X-rays is equal to the macrostrain, and is given by

$$\varepsilon_{k1} = \varepsilon_{k1} - \varepsilon_{k1} \sin \theta_{\beta\kappa} \ (15)$$

The macrostress $\delta_{\beta}$ is determined from the measurement of $\varepsilon_{k1}$.

3. 〈111〉 Fiber Texture

3.1 Reuss model

3.1.1 Mechanical elastic constants The $X_1$, $X_2$, and $X_3$ axes of the transformed crystal system are [111], [110] and [111], respectively. The transformation matrix $x_{1\kappa}$ is obtained by substituting $x_1 = \cos^{-1}(1/\sqrt{3})$ and $x_0 = \pi/4$ into Eq. (1). The mechanical elastic compliance obtained from Eq.(13) is as follows:

$$\tilde{c}_{11} = s_{11} - s_0/2 \ \ \ \ \ \ \ \ \ \ \ \ (16)$$

$$\tilde{c}_{12} = s_{12} + s_0/6$$

$$\tilde{c}_{13} = s_{11} + s_0/3$$

$$\tilde{c}_{22} = s_{12} + s_0/3$$

$$\tilde{c}_{33} = s_{11} - s_0/2$$

$$\tilde{c}_{44} = s_{14} + 4s_0/3$$

where $s_0$ is the single crystal elastic compliance and $s_0$ is anisotropy index defined by

$$s_0 = s_{11} - s_{12} - s_{14}/2 \ \ \ \ (17)$$

3.1.2 Determination of stress from X-ray strain The strain $\varepsilon_{k1}$ in a crystal with orientation angle $\beta$ is related to the macrostress $\delta_{\beta}$, which is equal to the stress acting on the crystal $\sigma_{\beta}$, as

$$\dot{\varepsilon}_{k1} = \frac{1}{4} s_{14} (\sigma_{11} + \sigma_{22} - 2\sigma_{33})$$

$$+ \frac{1}{12} (3s_{14} + 2s_0) (\sigma_{11} - \sigma_{22}) \cos 2\beta + 2\sigma_{12} \sin 2\beta$$

$$- \frac{\sqrt{2}}{3} s_0 (\sigma_{11} \cos (2\phi + 2\beta)$$

$$- \sigma_{12} \sin (2\phi + 2\beta)) \sin^2 \phi$$

$$+ \left[ - \frac{\sqrt{2}}{6} s_0 (\sigma_{11} - \sigma_{22}) \cos (\phi + 3\beta)$$

$$- 2\sigma_{12} \sin (\phi + 3\beta) \right] \sin 2\phi$$

$$+ \left( \frac{3+3}{3} s_{14} + 4s_0 \right) (\sigma_{11} \cos \phi + \sigma_{22} \sin \phi) \sin 2\phi$$

$$+ \frac{1}{3} (3s_{14} + s_0) (\sigma_{11} + \sigma_{22}) + \frac{1}{3} (3s_{14} - 2s_0) \sigma_{33}$$

The residual stresses in thin film is in most cases under the plane stress state, i.e. $\sigma_{13} = \sigma_{23} = \sigma_{33} = 0$. For this case, the above equation becomes

$$\dot{\varepsilon}_{k1} = \frac{1}{4} s_{14} (\sigma_{11} + \sigma_{22})$$

$$+ \frac{1}{12} (3s_{14} + 2s_0) (\sigma_{11} - \sigma_{22}) \cos 2\beta$$

$$+ 2\sigma_{12} \sin 2\beta) \sin^2 \phi$$

$$- \frac{\sqrt{2}}{3} s_0 (\sigma_{11} - \sigma_{22}) \cos (\phi + 3\beta)$$

$$- 2\sigma_{12} \sin (\phi + 3\beta) \sin 2\phi$$

$$+ \frac{1}{3} (3s_{14} + s_0) (\sigma_{11} + \sigma_{22})$$

For the case of the equi-biaxial state of stress, i.e. $\sigma_{11} = \sigma_{22} = \sigma_{33} = \sigma$ we have

$$\dot{\varepsilon}_{k1} = \frac{1}{2} s_{14} \sigma \sin^2 \phi + \frac{2}{3} (3s_{14} + s_0) \sigma$$

The stress $\sigma$ can be determined from the slope of the linear relation between $\varepsilon_{k1}$ and $\sin^2 \phi$. This equation was derived by Hauk el al.\(^{[10]}\) and Korhonen et al.\(^{[2]}\).

For the case of non-equiaxial stresses, the stress components, $\sigma_{11}$, $\sigma_{22}$ and $\sigma_{33}$, can be determined by the method proposed by Tanaka et al.\(^{[10a,11]}\) The orientation angle $\beta$ can be determined for each diffraction on the basis of a stereographic projection of poles for cubic crystal with [111] pole at the center. Table 2 summarizes $\beta$ values and the means of $\sin 3\beta$ and $\cos 3\beta$ for various diffractions which can be used for stress analysis. Table 2 also indicates the tilt angle $\phi$ for each diffraction. Since the mean $\sin 3\beta = 0$, the X-ray strain for $\phi = 0^\circ$ is given by

$$\dot{\varepsilon}_{k1}(\phi = 0) = \frac{1}{6} (3s_{14} + s_0) \sigma_{11} - \sigma_{22} \sigma_{33} \sin^2 \phi$$

$$+ \frac{\sqrt{2}}{6} s_0 (\sigma_{11} - \sigma_{22}) \cos 3\beta \sin 2\phi$$

$$+ \frac{1}{3} (3s_{14} + s_0) (\sigma_{11} + \sigma_{22})$$

(21)

Similarly, for the case of $\phi = 90^\circ$, by substituting $90^\circ - \beta$ for $\beta$ in Eq.(19) and taking the average, we have

$$\dot{\varepsilon}_{k1}(\phi = 90) = \frac{1}{6} (3s_{14} + s_0) \sigma_{11} - \sigma_{22} \sigma_{33} \sin^2 \phi$$

$$+ \frac{\sqrt{2}}{6} s_0 (\sigma_{11} - \sigma_{22}) \cos 3\beta \cos 2\phi$$

$$+ \frac{1}{3} (3s_{14} + s_0) (\sigma_{11} + \sigma_{22})$$

(22)
Table 2 Orientation angle $\beta$ for (111) fiber texture

<table>
<thead>
<tr>
<th>Diffraction plane</th>
<th>(222)</th>
<th>(422)</th>
<th>(331)</th>
<th>(311)</th>
<th>(220)</th>
<th>(420)</th>
<th>(331)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tilt angle $\psi$ (deg)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0</td>
<td>19.47</td>
<td>22.00</td>
<td>29.50</td>
<td>35.26</td>
<td>39.23</td>
<td>48.53</td>
<td></td>
</tr>
<tr>
<td>$\sin^2 \psi$</td>
<td>0</td>
<td>0.111</td>
<td>0.140</td>
<td>0.242</td>
<td>0.333</td>
<td>0.400</td>
<td>0.561</td>
</tr>
<tr>
<td>$\beta$ for $\phi = 0^\circ$ (deg)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0 to $-2\pi$</td>
<td>$60^\circ$ (422)</td>
<td>$60^\circ$ (224)</td>
<td>$0^\circ$ (311)</td>
<td>$0^\circ$ (110)</td>
<td>$0^\circ$ (311)</td>
<td>$0^\circ$ (110)</td>
<td>$0^\circ$ (331)</td>
</tr>
<tr>
<td>$\cos^3 \beta$</td>
<td>0</td>
<td>-1</td>
<td>1</td>
<td>-1</td>
<td>1</td>
<td>0</td>
<td>1</td>
</tr>
<tr>
<td>$\sin \beta$</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Diffraction plane</th>
<th>(400)</th>
<th>(311)</th>
<th>(422)</th>
<th>(222)</th>
<th>(420)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tilt angle $\psi$ (deg)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>54.74</td>
<td>58.52</td>
<td>61.87</td>
<td>70.53</td>
<td>75.04</td>
<td></td>
</tr>
<tr>
<td>$\sin^2 \psi$</td>
<td>0.667</td>
<td>0.727</td>
<td>0.778</td>
<td>0.889</td>
<td>0.933</td>
</tr>
<tr>
<td>$\beta$ for $\phi = 0^\circ$ (deg)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$60^\circ$ (400)</td>
<td>$30^\circ$ (311)</td>
<td>$19.11^\circ$ (422)</td>
<td>$-19.11^\circ$ (224)</td>
<td>$100.89^\circ$ (422)</td>
<td>$40.89^\circ$ (422)</td>
</tr>
<tr>
<td>$-60^\circ$ (040)</td>
<td>$-90^\circ$ (311)</td>
<td>$-100.89^\circ$ (422)</td>
<td>$120^\circ$ (224)</td>
<td>$-79.11^\circ$ (422)</td>
<td>$160.89^\circ$ (042)</td>
</tr>
<tr>
<td>$180^\circ$ (004)</td>
<td>$150^\circ$ (113)</td>
<td>$139.11^\circ$ (224)</td>
<td>$-120^\circ$ (222)</td>
<td>$-160.89^\circ$ (204)</td>
<td></td>
</tr>
<tr>
<td>$\cos^3 \beta$</td>
<td>-1</td>
<td>0</td>
<td>0.540</td>
<td>1</td>
<td>-0.540</td>
</tr>
<tr>
<td>$\sin \beta$</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
</tbody>
</table>

\[
\varepsilon_{ij}^b(\phi=90^\circ) = \frac{1}{6}(-s_0s_1 + (3s_{44} + s_0)s_{12}) \sin^2 \psi \\
+ \frac{\sqrt{3}}{6} s_0 (s_1 - s_2) \cos 3\beta \sin 2\psi \\
+ \frac{1}{3} (3s_{12} + s_0)(s_1 + s_2) \\
(22)
\]

From Eqs. (21) and (22), we can determine $s_{11}$ and $s_{22}$. By taking the sum and deduction of Eqs. (21) and (22), we have

\[
F_i = [\varepsilon_{ij}^b(\phi=0^\circ) - \varepsilon_{ij}^b(\phi=90^\circ)]/2 \\
(23)
\]

\[
F_{ii} = [\varepsilon_{ij}^b(\phi=0^\circ) - \varepsilon_{ij}^b(\phi=90^\circ)]/2 \\
=(s_{11} - s_{22})Y \\
(24)
\]

where

\[
Y = \frac{1}{12} (3s_{44} + 2s_0) \sin^3 \psi - \frac{\sqrt{3}}{6} s_0 \cos 3\beta \sin 2\psi \\
(25)
\]

The sum of $s_{11} + s_{22}$ is determined from the linear relation between $F_i$ and $\sin^2 \psi$, and the deduction of $s_{11} - s_{22}$ from the linear relation between $F_{ii}$ and $Y$. The value of $\sin 3\beta$ is listed in Table 2 for each diffraction plane.

The stress $s_{12}$ can be determined from the X-ray strain for $\phi=45^\circ$. By substituting $\phi=45^\circ$ and $\beta=45^\circ$ for $\beta$ in Eq. (19) and taking the average of the strain, we have

\[
\varepsilon_{ij}^b(\phi=45^\circ) = \frac{1}{4} s_0 (s_{11} + s_{22}) \sin^2 \psi \\
+ s_{12} \left[ \left( \frac{1}{2} s_4 + \frac{1}{3} s_0 \right) \sin^2 \psi - \frac{\sqrt{3}}{2} s_0 \cos 3\beta \sin 2\psi \right] \\
+ \left( s_{12} + \frac{1}{3} s_0 \right)(s_{11} + s_{22}) \\
(26)
\]

The difference between $\varepsilon_{ij}^b(\phi=45^\circ)$ and $F_i$ is expressed as

\[
F_{ii} = \varepsilon_{ij}^b(\phi=45^\circ) - F_i \\
= 2s_{12} Y \\
(27)
\]

Because $F_1$ is known from Eqs. (23) and (24), $F_{ii}$ can be calculated. From the relation between $F_{ii}$ and $Y$, $s_{12}$ is determined.

### 3.2 Voigt model

#### 3.2.1 Mechanical elastic constants

The mechanical elastic stiffness $\varepsilon_{ij}^b$ is given by

\[
\varepsilon_{ii}^b = c_{11} - c_0 a/2 \\
\varepsilon_{ii}^b = c_{12} + c_0 a/2 \\
\varepsilon_{ii}^b = c_{13} + c_0 a/2 \\
\varepsilon_{ii}^b = c_{21} - 2c_0 a/3 \\
\varepsilon_{ii}^b = c_{22} + c_0 a/3 \\
\varepsilon_{ii}^b = c_{23} + c_0 a/3 \\
(28)
\]

where $c_0$ is the anisotropy index defined by

\[
c_0 = c_{11} - c_{12} - 2c_4 \\
(29)
\]

The inverse of the stiffness matrix is the mecha-
cal compliance of the film which is given by
\[ \varepsilon_{\text{film}} = \begin{pmatrix} \frac{1}{3}s_{11} + \frac{2}{3}s_{12} + \frac{11}{24}s_{44} - \frac{3s_{12}^2}{8(4s_{11} + 3s_{12})} \\ \frac{1}{3}s_{11} + \frac{2}{3}s_{12} - \frac{7}{24}s_{44} + \frac{3s_{12}^2}{8(4s_{11} + 3s_{12})} \\ s_{12} = \frac{1}{2}s_{11} + \frac{1}{2}s_{12} \\ s_{33} = \frac{1}{2}s_{11} - \frac{1}{2}s_{12} \\ s_{66} = 6s_{12}(s_{11} - s_{12}) \\ s_{36} = \frac{1}{2}s_{11} + \frac{1}{2}s_{12} + \frac{3s_{12}^2}{2s_{11} + 3s_{12}} \end{pmatrix} \]
(30)

3.2.2 Determination of stress from X-ray strain

In Voigt model, the strain \( \varepsilon_{\text{film}} \) is related to the macro-stress by Eq.(15), and is independent of the orientation angle \( \beta \). For simplicity of expression, the macrostress \( \sigma_\beta \) in Eq.(15) is denoted by \( \sigma_0 \) in the following. By using the compliance \( s_\beta \), Eq.(15) can be written as
\[ \varepsilon_{\text{film}} = \frac{1}{s_{11}}[(s_{11} - s_{12})\cos\phi + 2s_{12}\sin\phi]\sin^2\phi \\
+ \frac{1}{s_{12}}[(s_{11} - s_{12})\cos\phi + 2s_{12}\sin\phi]\sin^2\phi \\
+ \frac{1}{s_{33}}(s_{11} + s_{12})\sin\phi \sin 2\phi \]
(31)

For the case of the plane stress, \( \sigma_1 = \sigma_2 = \sigma_3 = 0 \), the strain for \( \phi = 0^\circ, 90^\circ \), and \( 45^\circ \) are given by
\[ \varepsilon_{\text{film}}(\phi = 0^\circ) = \frac{1}{2}s_{11}(s_{11} - s_{12})\sin^2\phi \\
\varepsilon_{\text{film}}(\phi = 90^\circ) = \frac{1}{2}s_{12}(s_{11} + s_{12})\sin^2\phi \]
(32, 33)

\[ \varepsilon_{\text{film}}(\phi = 45^\circ) = \frac{1}{2}[s_{11}(s_{11} - s_{12})\sin^2\phi + s_{12}(s_{11} + s_{12})] \]
(34)

From the above three equations, we can obtain the following relations:
\[ F_1 = \frac{1}{4}(2s_{11} + 3s_{12})\sigma_1 + \frac{s_{12}}{4}\sigma_2 \\
+ \frac{s_{11} - s_{12}}{4}\sigma_3 \\
F_2 = \frac{1}{4}(2s_{11} + 3s_{12})\sigma_1 + \frac{s_{12}}{4}\sigma_2 \\
+ \frac{s_{11} - s_{12}}{4}\sigma_3 \\
F_{11} = \frac{1}{4}(2s_{11} + 3s_{12})\sigma_1 + \frac{s_{12}}{4}\sigma_2 \\
+ \frac{s_{11} - s_{12}}{4}\sigma_3 \]
(35, 36, 37)

From the slope of the linear relation of \( F_1, F_2, \) and \( F_{11} \) against \( \sin^2\phi \), the three stress components, \( \sigma_1, \sigma_2, \) and \( \sigma_3 \) are determined.

For the case of the equi-biaxial stress, \( \sigma_1 = \sigma_2 = \sigma \), the strain and stress are reduced to the relation between Eq.(20) which is derived on the basis of Reuss model.

4. \( \langle 100 \rangle \) Fiber Texture

4.1 Reuss model

4.1.1 Mechanical elastic constants

The transformed system \( (X^\prime) \) is identical to the original crystal system \( X \); \( a_0 \) is the unit matrix. The mechanical elastic compliance obtained from Eq.(7) is as follows:
\[ \varepsilon_{\text{film}} = \begin{pmatrix} s_{11} - s_{12}/4 \\ s_{12} + s_{11}/4 \\ s_{11} - s_{12}/4 \\ s_{12} + s_{11}/4 \\ s_{11} - s_{12}/4 \\ s_{12} + s_{11}/4 \end{pmatrix} \]
(38)

4.1.2 Determination of stress from X-ray strain

The relation between \( \varepsilon_{\text{film}} \) and \( \sigma_\beta \) is
\[ \varepsilon_{\text{film}} = \begin{pmatrix} s_{11} + 2s_{12}/4(\sigma_1 + \sigma_2 - 2\sigma_3) \\ + \frac{s_{11} - s_{12}}{4}\cos 2\phi + s_{12}\cos 2\phi + 2\beta \sin 2\phi \sin 2\phi \\ + \frac{s_{11} + s_{12}}{2}\sin 2\phi - s_{12}\sin 2\phi + 2\beta \sin 2\phi \sin 2\phi \\ + s_{11}(\sigma_1 + \sigma_2) + s_{12}\sigma_3 \end{pmatrix} \]
(39)

Under the assumption of the equi-biaxial stress, \( \sigma_1 = \sigma_2 = \sigma, \) and \( \sigma_3 = \sigma_3 = 0 \), we have
\[ \varepsilon_{\text{film}} = \begin{pmatrix} s_{11} + 2s_{12}/4(\sigma_1 + \sigma_2) \\ + \frac{s_{11} - s_{12}}{4}\cos 2\phi + s_{12}\cos 2\phi + 2\beta \sin 2\phi \sin 2\phi \\ + \frac{s_{11} + s_{12}}{2}\sin 2\phi - s_{12}\sin 2\phi + 2\beta \sin 2\phi \sin 2\phi \\ + s_{11}(\sigma_1 + \sigma_2) + s_{12}\sigma_3 \end{pmatrix} \]
(40)

This equation was derived by Hauk et al\(^{33} \). From the linear relation between \( \varepsilon_{\text{film}} \) and \( \sin^2\phi \), \( \sigma \) can be determined.

For non-equibiarcial plane stress case, the stress \( \sigma_1, \sigma_2, \) and \( \sigma_3 \) can be determined as before. The orientation angle \( \beta \) is determined as before for each diffraction. Table 3 summarizes the \( \beta \) value and the means of \( \sin 4\beta \) and \( \cos 4\beta \). The value of \( \sin 4\beta = 0 \).

The X-ray strain for \( \phi = 0^\circ \) and \( 90^\circ \) can be obtained as before. They are
\[ \varepsilon_{\text{film}}(\phi = 0^\circ) = \begin{pmatrix} \frac{1}{4}(2s_{44} + 3s_{44})\sigma_1 + \frac{s_{44}}{4}\sigma_2 \\ + \frac{s_{44}}{4}\sigma_3 \cos 4\beta \sin^2\phi + s_{44}\sigma_1 + s_{44}\sigma_2 \end{pmatrix} \]
(41)

\[ \varepsilon_{\text{film}}(\phi = 90^\circ) = \begin{pmatrix} \frac{1}{4}s_{44}(\sigma_1 + \sigma_2) + \frac{1}{4}(2s_{44} + 3s_{44})\sigma_2 \\ - \frac{s_{44}}{4}\sigma_3 \cos 4\beta \sin^2\phi + s_{44}\sigma_1 + s_{44}\sigma_2 \end{pmatrix} \]
(42)

By taking the sum and deduction of two strains, we have
\[ F_1 = \frac{1}{4}(2s_{44} + 3s_{44})\sigma_1 + \frac{s_{44}}{4}\sigma_2 \\
+ \frac{s_{44}}{4}\sigma_3 \cos 4\beta \sin^2\phi + s_{44}\sigma_1 + s_{44}\sigma_2 \]
(43)

\[ F_{11} = \frac{1}{4}(2s_{44} + 3s_{44})\sigma_1 + \frac{s_{44}}{4}\sigma_2 \\
+ \frac{s_{44}}{4}\sigma_3 \cos 4\beta \sin^2\phi + s_{44}\sigma_1 + s_{44}\sigma_2 \]
(44)

where
\[ V = [(s_{44} + s_{44})\cos 4\beta]/4 \]
(45)

The stress, \( \sigma_1, \sigma_2, \) can be determined from the linear relations between \( F_1 \) and \( \sin^2\phi \) and between \( F_{11} \) and \( V \).

The X-ray strain for \( \phi = 45^\circ \) is obtained as
\[ \varepsilon_{\text{film}}(\phi = 45^\circ) = \begin{pmatrix} \frac{1}{4}(s_{44} + s_{44})\sigma_1 + \frac{s_{44}}{4}\sigma_2 \end{pmatrix} \]
(46)

JSME International Journal
Table 3: Orientation angle β for 〈100〉 fiber texture

<table>
<thead>
<tr>
<th>Diffraction plane</th>
<th>(400)</th>
<th>(511)</th>
<th>(311)</th>
<th>(420)</th>
<th>(422)</th>
<th>(220)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tilt angle ψ (deg)</td>
<td>0</td>
<td>15.79</td>
<td>25.24</td>
<td>26.57</td>
<td>35.26</td>
<td>45</td>
</tr>
<tr>
<td>sin²ψ</td>
<td>0</td>
<td>0.074</td>
<td>0.182</td>
<td>0.200</td>
<td>0.333</td>
<td>0.5</td>
</tr>
<tr>
<td>β for φ = 0 deg</td>
<td>0</td>
<td>45°</td>
<td>45°</td>
<td>0°</td>
<td>45°</td>
<td>0°</td>
</tr>
<tr>
<td></td>
<td>0≤2π</td>
<td>(115)</td>
<td>(113)</td>
<td>(204)</td>
<td>(224)</td>
<td>(202)</td>
</tr>
<tr>
<td></td>
<td>15°</td>
<td>135°</td>
<td>135°</td>
<td>90°</td>
<td>135°</td>
<td>90°</td>
</tr>
<tr>
<td></td>
<td>-135°</td>
<td>-135°</td>
<td>-135°</td>
<td>180°</td>
<td>-135°</td>
<td>180°</td>
</tr>
<tr>
<td>cos⁴β</td>
<td>0</td>
<td>-1</td>
<td>-1</td>
<td>1</td>
<td>-1</td>
<td>1</td>
</tr>
<tr>
<td>sin⁴β</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
</tbody>
</table>

\[ + \frac{2}{3} (s_{44} + s_{90} + s_{40}\cos\beta) \sin^2\psi + s_{12}(s_{11} + s_{22}) \]

(46)

The difference between \(\varepsilon_{33}(\phi=45°)\) and \(F_i\) is expressed as

\[ F_{33} = \varepsilon_{33}(\phi=45°) - F_i \]

(47)

From the linear relation between \(F_{33}\) and \(V\), \(\sigma_{12}\) can be determined.

4.2 Voigt model

4.2.1 Mechanical elastic constants

The mechanical elastic stiffness of thin film is obtained as follows:

\[ \begin{align*}
    c_{11}^t &= c_{11} - c_0/4 \\
    c_{12}^t &= c_{12} + c_0/4 \\
    c_{13}^t &= c_{13} \\
    c_{23}^t &= c_{23} \\
    c_{44}^t &= c_{44}
\end{align*} \]

(48)

By taking the inverse matrix of \(c_{ij}^t\), the elastic compliance is obtained as follows:

\[ \begin{align*}
    s_{11} &= \frac{1}{2}s_{11} + \frac{1}{2}s_{12} + \frac{s_{11} - s_{12}}{2(s_{44} + s_{90})}s_{44} \\
    s_{12} &= \frac{1}{2}s_{11} + \frac{1}{2}s_{12} - \frac{s_{11} - s_{12}}{2(s_{44} + s_{90})}s_{44} \\
    s_{13} &= s_{12} \\
    s_{23} &= s_{11} \\
    s_{44} &= s_{44}
\end{align*} \]

(49)

4.2.2 Determination of stress from X-ray strain

The stress can be determined by the some procedure as described in section 3.2.2, where \(s_{ij}\) are given by Eq. (49). For the case of the equi-biaxial stress, the relation between the X-ray strain and the stress is equivalent to Eq.(40) derived on the basis of Reuss model.

5. 〈110〉 Fiber Texture

5.1 Reuss model

The \(X_i, X_j, X_k\) axes of the transformed crystal coordinates are [001] [110] and [110], respectively. The \((a_i)\) matrix is determined by substituting \(x_i=\pi/2\) and \(x_3=\pi/4\) into Eq.(1). The mechanical elastic compliance is calculated as follows:

\[ \begin{align*}
    &\varepsilon_{i3} = s_{11} - 7s_{80}/16 \\
    &\varepsilon_{i3} = s_{11} + 3s_{80}/16 \\
    &\varepsilon_{i3} = s_{11} + s_{80}/4 \\
    &\varepsilon_{i3} = s_{11} - s_{80}/2 \\
    &\varepsilon_{i3} = s_{44} + s_{80}
\end{align*} \]

(50)

5.1.1 Determination of stress from X-ray strain

The relation between \(\varepsilon_{33}\) and stress \(\sigma_{ij}\) is obtained as

\[ \varepsilon_{33} = \frac{1}{4}(s_{44} + s_{90} + s_{12})(\sigma_{11} + \sigma_{22}) - \frac{1}{2}(s_{44} + s_{90})\sigma_{33} + \frac{s_{80}}{8}(\sigma_{11} + \sigma_{22} - 2\sigma_{33})\cos(2(\phi + \beta)) \]

Table 4 Orientation angle $\beta$ for $\langle 110 \rangle$ fiber texture

<table>
<thead>
<tr>
<th>Diffraction plane</th>
<th>(220)</th>
<th>(331)</th>
<th>(420)</th>
<th>(422)</th>
<th>(311)</th>
<th>(222)</th>
<th>(333)</th>
<th>(511)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tilt angle $\psi$ (\text{deg})</td>
<td>0</td>
<td>13.26</td>
<td>18.43</td>
<td>30</td>
<td>31.48</td>
<td>35.26</td>
<td>35.26</td>
<td>35.26</td>
</tr>
<tr>
<td>$\sin^2 \psi$</td>
<td>0</td>
<td>0.0526</td>
<td>0.0999</td>
<td>0.25</td>
<td>0.2727</td>
<td>0.3333</td>
<td>0.3333</td>
<td>0.3333</td>
</tr>
<tr>
<td>$\beta$ for $\phi = 0^\circ$ (\text{deg})</td>
<td>0 - 360(^\circ)</td>
<td>0° (331)</td>
<td>180° (331)</td>
<td>90° (240)</td>
<td>35.26° (422)</td>
<td>-35.26° (422)</td>
<td>144.74° (242)</td>
<td>-144.74° (422)</td>
</tr>
<tr>
<td>$\cos^2 \beta$</td>
<td>0</td>
<td>1</td>
<td>-1</td>
<td>0.3333</td>
<td>-0.3333</td>
<td>1</td>
<td>1</td>
<td>-0.7778</td>
</tr>
<tr>
<td>$\cos \beta$</td>
<td>0</td>
<td>1</td>
<td>1</td>
<td>-0.7778</td>
<td>-0.7778</td>
<td>1</td>
<td>1</td>
<td>0.2099</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Diffraction plane</th>
<th>(400)</th>
<th>(331)</th>
<th>(420)</th>
<th>(422)</th>
<th>(511)</th>
<th>(220)</th>
<th>(331)</th>
<th>(420)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tilt angle $\psi$ (\text{deg})</td>
<td>45</td>
<td>49.54</td>
<td>50.77</td>
<td>54.74</td>
<td>57.02</td>
<td>60</td>
<td>64.76</td>
<td>71.6</td>
</tr>
<tr>
<td>$\sin^2 \psi$</td>
<td>0.5</td>
<td>0.5789</td>
<td>0.6</td>
<td>0.6667</td>
<td>0.7037</td>
<td>0.75</td>
<td>0.8182</td>
<td>0.9</td>
</tr>
<tr>
<td>$\beta$ for $\phi = 0^\circ$ (\text{deg})</td>
<td>90° (040)</td>
<td>-90° (040)</td>
<td>25.24° (331)</td>
<td>54.74° (042)</td>
<td>25.24° (331)</td>
<td>-54.74° (042)</td>
<td>0° (224)</td>
<td>76.74° (511)</td>
</tr>
<tr>
<td>$\cos^2 \beta$</td>
<td>-1</td>
<td>0.6364</td>
<td>-0.3333</td>
<td>1</td>
<td>-0.8947</td>
<td>0.3333</td>
<td>1</td>
<td>-1</td>
</tr>
<tr>
<td>$\cos \beta$</td>
<td>1</td>
<td>-0.9016</td>
<td>-0.7778</td>
<td>1</td>
<td>0.6011</td>
<td>-0.7778</td>
<td>1</td>
<td>1</td>
</tr>
</tbody>
</table>

\[
\begin{align*}
+ \frac{4s_4 + 3s_8}{16} (s_1 - s_2) \cos 2\phi + 2s_2 \sin 2\phi &
+ \frac{3s_8}{16} (s_1 - s_2) \cos 2(\phi + 2\beta) + 2 \cos 2\beta \\
- \frac{3}{8} s_8 (s_2 \sin 2(\phi + 2\beta) + 2 \sin 2\beta) &
+ \left[ \frac{1}{2} s_8 \sin (\phi + 2\beta) + 2 \sin 2\beta \right] \\
+ \frac{1}{2} (s_4 + s_8) (s_3 \cos \phi + s_2 \sin \phi) &
- \frac{1}{4} s_8 (s_1 - s_2) \cos 2\beta - 2s_2 \sin 2\beta \\
+ \frac{1}{4} (4s_4 + s_8) (s_1 - s_2) &
+ \frac{1}{2} (s_2 \sin 2\beta) (s_1 - s_2)
\end{align*}
\]

Here, we assume the plane stress, i.e. \(s_1 = s_2 = s_3 = 0\). Table 4 summarizes the values for various diffractions. The mean values of \(\sin 2\beta\) and \(\sin 4\beta\) are zero. The X-ray strain for \(\phi = 0^\circ\) is

\[
\varepsilon_{\phi=0} = \frac{1}{8} \left[ (s_2 + s_8 + s_0 \cos 2\beta) (s_1 + s_2) \right]
+ \frac{1}{16} (4s_4 + 3s_8 + 3s_8 \cos 4\beta)
+ \frac{1}{4} s_8 (s_1 - s_2) \cos 2\beta + \frac{1}{4} (4s_4 + s_8) (s_1 + s_2)
\]

The sum and deduction of two strains are described as

\[
F_1 = \left[ \varepsilon_{\phi=0} + \varepsilon_{\phi=90^\circ} \right] / 2
=(s_1 + s_2) W_1 + \frac{1}{4} (4s_4 + s_8) (s_1 + s_2)
\]

where

\[
W_1 = \frac{1}{8} (2s_4 + s_8 + s_8 \cos 2\beta) \sin^2 \phi
\]

and

\[
F_{11} = \left[ \varepsilon_{\phi=0} - \varepsilon_{\phi=90^\circ} \right] / 2
=(s_1 - s_2) W_1
\]

where

\[
W_{11} = \frac{1}{16} (4s_4 + 3s_8 + 3s_8 \cos 4\beta)
+ \frac{1}{4} s_8 (s_1 - s_2) \sin^2 \phi + \frac{1}{4} (s_4 + 3s_8) \cos 2\beta
\]

From the relations between \(F_1\) and \(W_1\), and between \(F_{11}\) and \(W_{11}\), \(s_1\) and \(s_2\) can be determined.

The strain \(\varepsilon_{\phi=45^\circ}\) is

\[
\varepsilon_{\phi=45^\circ} = \frac{1}{8} (s_2 + s_8 + s_0 \cos 2\beta) (s_1 + s_2)
+ \frac{1}{4} (4s_4 + s_8 + s_8 \cos 4\beta)
+ \frac{1}{4} s_8 (s_1 - s_2) \sin^2 \phi
+ \frac{1}{4} (s_4 + s_8) \cos 2\beta + \frac{1}{4} (4s_4 + s_8) (s_1 + s_2)
\]
From the above equation, we can derive the following equation to determine $\sigma_2$:

$$F_{i} = \varepsilon_2(\phi = 45^\circ) - F_1 = 2\sigma_2 W_{ii}$$  

(59)

where

$$W_{ii} = \frac{1}{16} \left[ (4s_4 + 3s_3 - 3s_0 \cos 4\beta) + 6s_0 \cos 2\beta \right] \sin^2 \phi + \frac{1}{2} (s_4 + s_3 - s_0 \cos 2\beta)$$  

(60)

For the case of the equi-biaxial stress $\sigma_1 = \sigma_2 = \sigma$, the X-ray strain is given by the following equation irrespective of $\phi$ value:

$$\varepsilon_2 = \frac{1}{4} (2s_4 + s_3 + s_0 \cos 2\beta) \sigma \sin^2 \phi + \frac{1}{2} (s_4 + s_3 + s_0) \sigma$$  

(61)

This equation was derived by Hauk et al. and Ejiri et al. Only for the case of $<110> fiber$ texture, the X-ray strain is not a linear function of $\sin^2 \phi$ even under the plane stress state. The above equation can be written as

$$\varepsilon_2 = \sigma W_0 + \frac{1}{2} (s_4 + s_3 + s_0) \sigma$$  

(62)

where

$$W_0 = \frac{1}{4} (2s_4 + s_3 + s_0 \cos 2\beta) \sin^2 \phi$$  

(63)

By plotting $\varepsilon_2$ as a function of $W_0$, the stress $\sigma$ can be obtained from the slope of the linear relation.

5.2 Voigt model

5.2.1 Mechanical elastic constants The mechanical elastic stiffness is obtained as follows:

$$\begin{align*}
\varepsilon_1 &= c_{11} - 2c_{0}/16 \\
\varepsilon_2 &= c_{12} + 3c_{0}/16 \\
\varepsilon_3 &= 2c_{11} - c_{0}/2 \\
\varepsilon_4 &= c_{11} + c_{0}/2 \\
\varepsilon_5 &= c_{11} + c_{0}/4
\end{align*}$$

(64)

The corresponding compliance ($s_0$) can be obtained by calculating the inverse matrix of $\varepsilon_0$.

5.2.2 Determination of stress from X-ray strain The stress can be determined by the same procedure as used in section 3.2.2, where $s_0$ can be determined from Eqs. (64).

6. Applications to Various Thin Films

6.1 Mechanical elastic constants

The anisotropy of elastic properties of polycrystals is a function of the anisotropy index $s_0$ defined by Eq. (17). When $s_0$ is zero, the crystal is isotropic. Here we chose two materials with high and low $s_0$ values for each case of fiber texture shown in Table 1. The single crystal compliances of five materials are listed in Table 5(10-18). Aluminum and TiN are materials with low anisotropy, and the others have high anisotropy.

<table>
<thead>
<tr>
<th>Table 5</th>
<th>Elastic compliances of single crystal</th>
</tr>
</thead>
<tbody>
<tr>
<td>Material</td>
<td>Lattice constant [Å]</td>
</tr>
<tr>
<td>---------</td>
<td>------------------------</td>
</tr>
<tr>
<td>Al</td>
<td>4.0948</td>
</tr>
<tr>
<td>Au</td>
<td>4.1786</td>
</tr>
<tr>
<td>Ni</td>
<td>3.5226</td>
</tr>
<tr>
<td>TiN</td>
<td>4.240</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Kamem et al.</td>
</tr>
<tr>
<td>AIP Handbook(7)</td>
</tr>
<tr>
<td>AIP Handbook(7)</td>
</tr>
<tr>
<td>AIP Handbook(7)</td>
</tr>
<tr>
<td>AIP Handbook(7)</td>
</tr>
<tr>
<td>Perry(10)</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Table 6</th>
<th>Mechanical elastic compliances of cubic thin films with fiber texture</th>
</tr>
</thead>
<tbody>
<tr>
<td>Material</td>
<td>$s_{11}$</td>
</tr>
<tr>
<td>---------</td>
<td>---------</td>
</tr>
<tr>
<td>Texture</td>
<td></td>
</tr>
<tr>
<td>Al</td>
<td>&lt;110&gt;</td>
</tr>
<tr>
<td></td>
<td>13.940</td>
</tr>
<tr>
<td></td>
<td>&lt;110&gt;</td>
</tr>
<tr>
<td></td>
<td>-5.114</td>
</tr>
<tr>
<td></td>
<td>-0.248</td>
</tr>
<tr>
<td></td>
<td>-0.460</td>
</tr>
<tr>
<td></td>
<td>2.383</td>
</tr>
<tr>
<td></td>
<td>5.525</td>
</tr>
<tr>
<td>Au</td>
<td></td>
</tr>
<tr>
<td></td>
<td>13.855</td>
</tr>
<tr>
<td></td>
<td>&lt;110&gt;</td>
</tr>
<tr>
<td></td>
<td>-5.107</td>
</tr>
<tr>
<td></td>
<td>-0.246</td>
</tr>
<tr>
<td></td>
<td>-0.454</td>
</tr>
<tr>
<td></td>
<td>2.346</td>
</tr>
<tr>
<td></td>
<td>5.525</td>
</tr>
<tr>
<td>Cu</td>
<td></td>
</tr>
<tr>
<td></td>
<td>13.263</td>
</tr>
<tr>
<td></td>
<td>&lt;110&gt;</td>
</tr>
<tr>
<td></td>
<td>-5.102</td>
</tr>
<tr>
<td></td>
<td>-0.246</td>
</tr>
<tr>
<td></td>
<td>-0.454</td>
</tr>
<tr>
<td></td>
<td>2.346</td>
</tr>
<tr>
<td></td>
<td>5.525</td>
</tr>
<tr>
<td>Ni</td>
<td></td>
</tr>
<tr>
<td></td>
<td>13.263</td>
</tr>
<tr>
<td></td>
<td>&lt;110&gt;</td>
</tr>
<tr>
<td></td>
<td>-5.102</td>
</tr>
<tr>
<td></td>
<td>-0.246</td>
</tr>
<tr>
<td></td>
<td>-0.454</td>
</tr>
<tr>
<td></td>
<td>2.346</td>
</tr>
<tr>
<td></td>
<td>5.525</td>
</tr>
</tbody>
</table>

The mechanical compliances, $s_{11}$ (Reuss model) and $s_{12}$ (Voigt model) calculated from the single crystal compliance $s_0$ are summarized in Table 6. For the case of $<100>$ fiber texture, the following relation are held: $s_{11} = s_{12} = s_{13}$, $s_{11} = s_{12} = s_{44}$, $s_{44} = s_{55}$. For the case of $<111>$ fiber texture, $s_{11} = s_{12}$, $s_{13} = s_{55}$. There is a slight difference in the compliances between TiN with $<100>$ and $<110>$ fiber textures. The compliances based on Reuss model is larger than that based on Voigt model. The difference is larger for the material with high anisotropy.

Polycrystals with random orientation in three dimensions are isotropic, and have two independent compliances, $s_{11}$ and $s_{12}$. They are calculated from single crystal compliances $s_0$ as

$$s_{11} = s_{55} = s_{13} = s_{44}$$

(65)

for Reuss model(13), and for Voigt model(14)

$$\begin{align*}
\frac{s_{11}}{s_{12}} &= \frac{5s_{66} + 2s_{22} + 2s_{33}}{5s_{66} + 2s_{22} + 2s_{33}} \\
\frac{s_{12}}{s_{13}} &= \frac{5s_{66} + 2s_{22} + 2s_{33}}{5s_{66} + 2s_{22} + 2s_{33}}
\end{align*}$$

(66)

The $s_{11}$ and $s_{12}$ are given by

$$s_{11} = 2(s_{44} - s_{55})$$

(67)

$$s_{12} = 2(s_{44} - s_{55})$$

(68)

Table 7 summarizes the values calculated by using Eqs. (65) to (68). Those are the compliances of polycrystals without any texture. The difference in compliances between materials with and without texture.
becomes larger as the anisotropy index gets larger.

### 6.2 X-ray stress measurement

In the X-ray method, the strain $\varepsilon_{50}$ is determined from the change in the diffraction angle 2$\theta$ from that of the stress-free state 2$\theta_0$ as

$$\varepsilon_{50} = -\cot \theta (\theta - \theta_0) \quad (69)$$

For the case of the equi-biaxial state of thin films with $\langle 111 \rangle$ and $\langle 100 \rangle$ fiber textures, the linear relation between $\varepsilon_{50}$ and $\sin^2 \phi$ is satisfied. When we use the same diffraction plane, the precise value of $\theta_0$ is not necessary as in the classical $\sin^2 \phi$ method.

For the case of $\langle 110 \rangle$ fiber texture, the relation between $\varepsilon_{50}$ and $\sin^2 \phi$, Eq.(61), is not linear. To determine the stress, we plot $\varepsilon_{50}$ against $W_0$ as given by Eq.(62). Even for this case, if we can use the same diffraction plane as different $\phi$ values, the precise values of $\theta_0$ is not required\(^{131}\). In our previous paper of Al film with $\langle 111 \rangle$ fiber texture, we are 222 and 331 diffractions, by which we can measure the strain at two tilt angles (see Table 2). When it is difficult to measure strains at two tilt angles, we have to measure 2$\theta_0$ by using powder.

---

**Table 7** Mechanical elastic compliances of cubic poly crystals with random orientation

<table>
<thead>
<tr>
<th>Material</th>
<th>Al</th>
<th>Au</th>
<th>TiN</th>
<th>Cu</th>
<th>Ni</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reuss model</td>
<td>$\varepsilon_{50}^{Al}$ (1/TPa)</td>
<td>$\varepsilon_{50}^{Al}$ (1/TPa)</td>
<td>$\varepsilon_{50}^{Al}$ (1/TPa)</td>
<td>$\varepsilon_{50}^{Al}$ (1/TPa)</td>
<td>$\varepsilon_{50}^{Al}$ (1/TPa)</td>
</tr>
<tr>
<td>Voigt model</td>
<td>$\varepsilon_{50}^{Al}$ (1/TPa)</td>
<td>$\varepsilon_{50}^{Al}$ (1/TPa)</td>
<td>$\varepsilon_{50}^{Al}$ (1/TPa)</td>
<td>$\varepsilon_{50}^{Al}$ (1/TPa)</td>
<td>$\varepsilon_{50}^{Al}$ (1/TPa)</td>
</tr>
</tbody>
</table>

**Table 8** Diffraction planes of TiN

<table>
<thead>
<tr>
<th>Characteristic X-Ray</th>
<th>Diffraction plane</th>
<th>Diffraction angle (deg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu-K $\alpha$</td>
<td>(333)(511)</td>
<td>141.74</td>
</tr>
<tr>
<td></td>
<td>(422)</td>
<td>125.93</td>
</tr>
<tr>
<td></td>
<td>(420)</td>
<td>108.81</td>
</tr>
<tr>
<td></td>
<td>(331)</td>
<td>104.85</td>
</tr>
<tr>
<td>Co-K $\alpha$</td>
<td>(420)</td>
<td>141.52</td>
</tr>
<tr>
<td></td>
<td>(331)</td>
<td>133.92</td>
</tr>
<tr>
<td></td>
<td>(400)</td>
<td>115.23</td>
</tr>
<tr>
<td>Fe-K $\alpha$</td>
<td>(331)</td>
<td>169.55</td>
</tr>
<tr>
<td></td>
<td>(400)</td>
<td>132.09</td>
</tr>
<tr>
<td></td>
<td>(222)</td>
<td>104.64</td>
</tr>
<tr>
<td>Mn-K $\alpha$</td>
<td>(400)</td>
<td>165.54</td>
</tr>
<tr>
<td></td>
<td>(222)</td>
<td>118.44</td>
</tr>
<tr>
<td></td>
<td>(311)</td>
<td>110.68</td>
</tr>
<tr>
<td>Cr-K $\alpha$</td>
<td>(222)</td>
<td>138.74</td>
</tr>
<tr>
<td></td>
<td>(311)</td>
<td>127.28</td>
</tr>
<tr>
<td></td>
<td>(220)</td>
<td>99.66</td>
</tr>
<tr>
<td>V-K $\alpha$</td>
<td>(311)</td>
<td>156.85</td>
</tr>
<tr>
<td></td>
<td>(220)</td>
<td>113.33</td>
</tr>
</tbody>
</table>

**Table 9** Diffraction planes of Ni

<table>
<thead>
<tr>
<th>Characteristic X-Ray</th>
<th>Diffraction plane</th>
<th>Diffraction angle (deg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu-K $\alpha$</td>
<td>(331)</td>
<td>156.10</td>
</tr>
<tr>
<td></td>
<td>(400)</td>
<td>144.97</td>
</tr>
<tr>
<td>Co-K $\alpha$</td>
<td>(311)</td>
<td>122.10</td>
</tr>
<tr>
<td>Fe-K $\alpha$</td>
<td>(331)</td>
<td>123.28</td>
</tr>
<tr>
<td></td>
<td>(220)</td>
<td>114.82</td>
</tr>
<tr>
<td>Mn-K $\alpha$</td>
<td>(311)</td>
<td>163.58</td>
</tr>
<tr>
<td></td>
<td>(220)</td>
<td>115.12</td>
</tr>
<tr>
<td>Cr-K $\alpha$</td>
<td>(220)</td>
<td>133.66</td>
</tr>
</tbody>
</table>

The non-linearity between $\varepsilon_{50}$ and $\sin^2 \phi$ is dependent on the values of anisotropy index $s_0$. If $s_0$ is very low, we can use the classical $\sin^2 \phi$ plot to obtain the stress value as described below. In the following the influence of anisotropy index on the relation between $\varepsilon_{50}$ and $\sin^2 \phi$ will be examined for TiN and Ni films with $\langle 110 \rangle$ fiber texture.

Tables 8 and 9 show the diffraction angles of various diffractions which can be used for stress measurements of TiN and Ni thin films. The values of $\beta$ are listed in Table 4. The relation between $\varepsilon_{50}$ and $\sin^2 \phi$ for $\phi=0^\circ$ is examined for three cases: (a) the equi-biaxial case $\sigma_{11}=\sigma_{22}=\sigma$, (b) the uni-axial case $\sigma_{11}=\sigma$, $\sigma_{22}=0$, and (c) the shear case $\sigma_{11}=\sigma$, $\sigma_{22}=-\sigma$.

The relation between $\varepsilon_{50}/\sigma$ and $\sin^2 \phi$ for three stress states for TiN and Ni films are shown in Figs. 4 and 5, respectively. The open marks indicate the strains based on Reuss model which can be measured at more than two tilt angles. The solid marks mean the strain measured at one tilt angles. The dotted line indicates the relation based on Voigt model, and the solid line is the linear regression line for all the data plotted. For the cases of the equi-biaxial and uni-axial stresses of TiN, the relation is almost linear, and Reuss and Voigt models give nearly identical relation. The slope is nearly equal to $s_{50}/2$, because $s_0$ is small. The $s_{40}$ value is also close to the mechanical compliance $\varepsilon_{50}^*/\sigma$ of random polycrystals shown in Table 7. The mechanical compliance measured for bulk polycrystals with random orientation can be used for X-ray stress measurement as utilized by Miki et al\(^{19}\). For the case of the shear stress, there is some scatter even for TiN films. The relation between $\varepsilon_{50}/\sigma$ and $\sin^2 \phi$ of Ni films is not linear even in the case of the equi-biaxial stress. The method described in the present paper will be useful for X-ray stress analysis. The stress values calculated by using Reuss and Voigt models are different. It is necessary to decide which
Fig. 4 Relation between strain and $\sin^2 \phi$ for TiN with <110> fiber texture

Fig. 5 Relation between strain and $\sin^2 \phi$ for Ni with <110> fiber texture
model is appropriate for the material measured.

7. Conclusions

The X-ray method to measure the residual stress in polycrystalline thin films having fiber texture with the axis of \(\langle 111\rangle\), \(\langle 100\rangle\) and \(\langle 110\rangle\) perpendicular to the film surface is proposed on the basis of Reuss and Voigt models. The results are summarized at follows:

1. According to the analysis based on Reuss model, the relation between the X-ray strain \(\varepsilon_{11}\) and \(\sin^2 \phi\) for the equi-biaxial stress is linear for the cases of \(\langle 111\rangle\) and \(\langle 100\rangle\) fiber textures. For the other cases, the relation is not linear, because of the anistropic elastic properties of crystal.

2. The analysis based on Voigt model gives the linear relation between \(\varepsilon_{11}\) and \(\sin^2 \phi\) for all three fiber texture.

3. When the anisotropy index is low, such as TiN, the relation between \(\varepsilon_{11}\) and \(\sin^2 \phi\) based on Reuss model is almost linear and nearly identical to the relation derived from Voigt model. The bulk elastic constant can be used for X-ray elastic constants as an approximation.

4. When thin films are made of anisotropic crystals and the relation between \(\varepsilon_{11}\) and \(\sin^2 \phi\) is nonlinear, the method proposed in the present paper will be useful for the determination of residual stresses.

References