Vol 14 No 9, September 2005
1009-1963/2005/14(09)/1866-07

Chinese Physics

© 2005 Chin. Phys. Soc.
and IOP Publishing Ltd

Evaluation of multiaxial stress in textured cubic films by x-ray diffraction*

Zhang Jian-Min(张建民)[a)†] and Xu Ke-Wei(徐可为)[b)]

[a)] *College of Physics and Information Technology, Shaanxi Normal University, Xi'an 710062, China*

[b)] *State Key Laboratory for Mechanical Behavior of Materials, Xi'an Jiaotong University, Xi'an 710049, China*

(Received 22 October 2004; revised manuscript received 27 February 2005)

X-ray diffraction is used extensively to determine the residual stress in bulk or thin film materials on the assumptions that the material is composed of fine crystals with random orientation and the stress state is biaxial and homogeneous through the x-ray penetrating region. The stress is calculated from the gradient of $\varepsilon \sim \sin^2\psi$ linear relation. But the method cannot be used in textured films due to nonlinear relation. In this paper, a novel method is proposed for measuring the multiaxial stresses in cubic films with any [*hkl*] fibre texture. As an example, a detailed analysis is given for measuring three-dimensional stresses in FCC films with [111] fibre texture.

Keywords: residual stress, x-ray diffraction, thin film, fibre texture
PACC: 6180C, 7360D, 8170J

1. Introduction

Metal films have been used extensively in microelectronics and data storage devices because of their electronic, magnetic and optical properties. Residual stress in metal film is an yardstick for the reliability of the film/substrate systems. Large residual stress can cause a number of significant reliability problems such as voiding,[1−3] cracking,[4] hillocking[5,6] in the film, and buckling or delamination of the film from the substrate.[7] If the temperature changes in these systems, thermal stress develops in the film because of a mismatch in the thermal expansion coefficients between film and substrate.[8] A large temperature change produces large stress which may induce plastic deformation as well as creep deformation in the film.[9] The evaluation of the magnitude and state of residual stresses in thin film is essential to guarantee the reliability of microelectronics and data storage devices.

In general, residual stress is determined with x-ray diffraction from the measurement of the peak positions of high-angle reflections as function of the angle ψ between the normal of the specimen surface and the normal of one selected diffracting plane. The experimental results are then presented in a plot of the plane distance d (or lattice strain $\varepsilon = \dfrac{d - d_0}{d_0}$) versus $\sin^2\psi$ (the so-called $\sin^2\psi$ method). If the material is composed of fine crystals with random orientation and the stress state is biaxial and homogeneous through the x-ray penetrating region, an $\varepsilon - \sin^2\psi$ linear relation will be obtained. The stress is calculated from the gradient of $\varepsilon - \sin^2\psi$ relation with the help of x-ray elastic constant (x-REC) which can be determined experimentally or derived from single-crystal elastic constants. However, nonlinear $\varepsilon - \sin^2\psi$ relations have been reported extensively, such as a splitting for positive and negative ψ values due to the presence of shear stresses,[10,11] a curvature resulting from the presence of a stress gradient normal to the surface[12] and oscillations on the textured materials.[13] The conventional $\sin^2\psi$ method cannot be adopted in such cases.

Physical or chemical vapour deposited (PVD or CVD) films are well known to have fibre texture, that is a specific lattice plane is taken to be parallel to the film surface. Ohmi *et al* found that Cu films with (001)- and (111)- preferred orientation were grown on Si substrate by a low kinetic energy particle process.[14] Tracy and Knorr discussed the observed texture response of Cu films deposited by a variety

*Project supported by the State Key Development Program for Basic Research of China (Grant No 2004CB619302), and the National Natural Science Foundation of China (Grant No 50271038).

†E-mail: jianm_zhang@yahoo.com

of techniques, and in general, (001)-, (011)-, (111)- and (511)-oriented films dominated the response.[15] In previous papers, we made calculations of the strain energy[16] and surface energy[17] for various grain orientations relative to the film surface in a polycrystalline film composed of the face-centred cubic (FCC) metals, Cu, Al, Ag, Au, Ni, Pb and Th. The results showed that the (111) texture is induced by surface energy minimization, but the (001) and (011) texture is driven by strain energy minimization.

In this paper, a novel method is proposed for measuring the multiaxial stresses in cubic films having any $[hkl]$ fibre texture with the fibre axis perpendicular to the film surface. As an example, a detailed analysis is given for measuring three-dimension stresses in FCC films with [111] fibre texture.

2. Theory

Three orthogonal coordinate systems used in the following discussion are shown in Fig.1, The specimen coordinate system (S_i) is defined such that S_1 and S_2 axes are in the film surface and S_3 axis is perpendicular to the film surface. The laboratory coordinate system (L_i), in which diffraction is sampled, is defined such that L_3 is in the direction of the normal to the family of planes $(h'k'l')$ whose lattice plane distance d is being measured by x-ray diffraction, L_2 is in the film surface and makes an angle ϕ with S_2. The texture coordinate system (T_i), which is transformed from cubic crystal axes (A_i), is selected such that the fibre axis $[hkl]$ is taken as the T_3 direction, T_1 is in the film surface and makes an angle β with S_1.

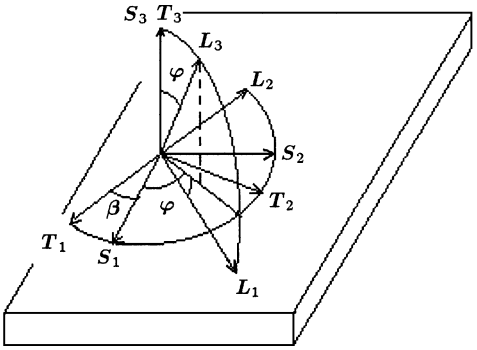


Fig.1. Three orthogonal coordinates of specimen system (S_i), laboratory system (L_i) and texture system (T_i).

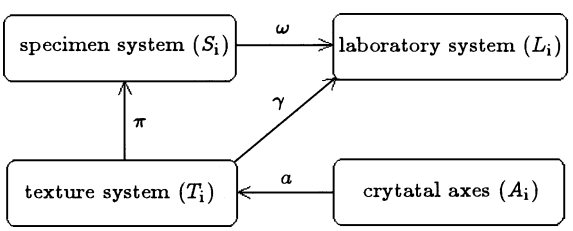


Fig.2. Various coordinate systems and transformation matrices.

The transformation matrices from (A_i) to (T_i), (T_i) to (S_i), (S_i) to (L_i) and (T_i) to (L_i), which are indicted by the arrows in Fig.2, is given by

$$(a_{ij}) = \begin{bmatrix} \dfrac{hl}{\sqrt{h^2+k^2}\sqrt{h^2+k^2+l^2}} & \dfrac{kl}{\sqrt{h^2+k^2}\sqrt{h^2+k^2+l^2}} & -\dfrac{\sqrt{h^2+k^2}}{\sqrt{h^2+k^2+l^2}} \\ -\dfrac{k}{\sqrt{h^2+k^2}} & \dfrac{h}{\sqrt{h^2+k^2}} & 0 \\ \dfrac{h}{\sqrt{h^2+k^2+l^2}} & \dfrac{k}{\sqrt{h^2+k^2+l^2}} & \dfrac{l}{\sqrt{h^2+k^2+l^2}} \end{bmatrix}, \tag{1}$$

$$(\pi_{ij}) = \begin{bmatrix} \cos\beta & \sin\beta & 0 \\ -\sin\beta & \cos\beta & 0 \\ 0 & 0 & 1 \end{bmatrix}, \tag{2}$$

$$(\omega_{ij}) = \begin{bmatrix} \cos\phi\cos\psi & \sin\phi\cos\psi & -\sin\psi \\ -\sin\phi & \cos\phi & 0 \\ \cos\phi\sin\psi & \sin\phi\sin\psi & \cos\psi \end{bmatrix}, \tag{3}$$

$$(\gamma_{ij}) = (\omega_{im})(\pi_{mj}) = \begin{bmatrix} \cos(\phi+\beta)\cos\psi & \sin(\phi+\beta)\cos\psi & -\sin\psi \\ -\sin(\phi+\beta) & \cos(\phi+\beta) & 0 \\ \cos(\phi+\beta)\sin\psi & \sin(\phi+\beta)\sin\psi & \cos\psi \end{bmatrix}, \tag{4}$$

where ψ is the angle between S_3 and L_3. For the $[hkl]$ fibre texture with the fibre axis perpendicular to the film surface, S_3 axis coincides with T_3 axis as shown in Fig.1. Because L_3 is selected along the normal to diffracting plane $(h'k'l')$, the tilt angle ψ can be calculated by

$$\cos\psi = \frac{hh' + kk' + ll'}{\sqrt{(h^2+k^2+l^2)(h'^2+k'^2+l'^2)}}, \tag{5}$$

from Eq.(1) and (4), the L_3 direction can be presented in crystal axes $(A_{\rm i})$ by

$$\begin{aligned} L_3 =& \gamma_{3k}a_{kj}A_j \\ =& \left[\frac{hl}{\sqrt{h^2+k^2}\sqrt{h^2+k^2+l^2}}\cos(\phi+\beta)\sin\psi - \frac{k}{\sqrt{h^2+k^2}}\sin(\phi+\beta)\sin\psi + \frac{h}{\sqrt{h^2+k^2+l^2}}\cos\psi\right]A_1 \\ &+ \left[\frac{kl}{\sqrt{h^2+k^2}\sqrt{h^2+k^2+l^2}}\cos(\phi+\beta)\sin\psi + \frac{h}{\sqrt{h^2+k^2}}\sin(\phi+\beta)\sin\psi + \frac{k}{\sqrt{h^2+k^2+l^2}}\cos\psi\right]A_2 \\ &- \left[\frac{\sqrt{h^2+k^2}}{\sqrt{h^2+k^2+l^2}}\cos(\phi+\beta)\sin\psi - \frac{l}{\sqrt{h^2+k^2+l^2}}\cos\psi\right]A_3. \end{aligned} \tag{6}$$

In the x-ray method, the macro-stress σ_{pq} is determined from measurement of normal strain $\varepsilon_{33}^{\rm L}$ along L_3 direction. From the relations between stress and/or strain

$$\varepsilon_{33}^{\rm L} = \gamma_{3i}\gamma_{3j}\varepsilon_{ij}^{\rm T}, \tag{7}$$

$$\varepsilon_{ij}^{\rm T} = s_{ijkl}^{\rm T}\sigma_{kl}^{\rm T}, \tag{8}$$

$$\sigma_{kl}^{\rm T} = \pi_{pk}\pi_{ql}\sigma_{pq}, \tag{9}$$

we can get

$$\varepsilon_{33}^{\rm L} = \gamma_{3i}\gamma_{3j}s_{ijkl}^{\rm T}\pi_{pk}\pi_{ql}\sigma_{pq}. \tag{10}$$

The stress and strain tensors are denoted by $(\sigma_{ij},\ \varepsilon_{ij})$, $(\sigma_{ij}^{\rm L},\ \varepsilon_{ij}^{\rm L})$ and $(\sigma_{ij}^{\rm T},\ \varepsilon_{ij}^{\rm T})$ for $(S_{\rm i})$, $(L_{\rm i})$ and $(T_{\rm i})$ coordinate systems, respectively. The compliance $s_{ijkl}^{\rm T}$ for the $(T_{\rm i})$ coordinates is related to the elastic compliance s_{mnop} in the $(A_{\rm i})$ coordinates by[18]

$$s_{ijkl}^{\rm T} = a_{im}a_{jn}a_{ko}a_{lp}s_{mnop}, \tag{11}$$

where the summation is taken from 1 to 3 for repeated subscript indices, the 81 compliances of s_{mnop} (or $s_{ijkl}^{\rm T}$) form a fourth-rank tensor and the first two subscript can be abbreviated into a single one running from 1 to 6, and the last two can be abbreviated in the same way, according to the following scheme:

Tensor notation :	11	22	33	23, 32	31, 13	12, 21
Matrix notation :	1	2	3	4	5	6

At the same time factors 2 and 4 are introduced as follows:

$s_{mnop} = s_{ij}$ when i and j are 1, 2 or 3,
$2s_{mnop} = s_{ij}$ when either i or j are 4, 5 or 6,
$4s_{mnop} = s_{ij}$ when both i and j are 4, 5 or 6.

The arrays of s_{ij} form 6×6 squares.

For the materials with cubic structures, only 3 (s_{11}, s_{12} and s_{44}) of the 36 components s_{ij} are independent,

$$s_{ij}^{\rm cub} = \begin{bmatrix} s_{11} & s_{12} & s_{12} & 0 & 0 & 0 \\ s_{12} & s_{11} & s_{12} & 0 & 0 & 0 \\ s_{12} & s_{12} & s_{11} & 0 & 0 & 0 \\ 0 & 0 & 0 & s_{44} & 0 & 0 \\ 0 & 0 & 0 & 0 & s_{44} & 0 \\ 0 & 0 & 0 & 0 & 0 & s_{44} \end{bmatrix}. \tag{12}$$

In this case, Eq.(11) can be rewritten in the form

$$\begin{aligned} s_{ijkl}^{\rm T} =& \delta_{ij}\delta_{kl}s_{12} + \frac{1}{4}(\delta_{ik}\delta_{jl} + \delta_{il}\delta_{jk})s_{44} \\ &+ a_{is}a_{js}a_{ks}a_{ls}s_0, \end{aligned} \tag{13}$$

where δ_{ij} is the Kronecker delta function defined by

$$\delta_{ij} = \begin{cases} 1 & (i = j), \\ 0 & (i \neq j), \end{cases} \tag{14}$$

$$s_0 \equiv s_{11} - s_{12} - \frac{1}{2}s_{44}. \tag{15}$$

If a general three-dimensional stress tensor is considered,

$$(\sigma_{ij}) = \begin{bmatrix} \sigma_{11} & \sigma_{12} & \sigma_{13} \\ \sigma_{12} & \sigma_{22} & \sigma_{23} \\ \sigma_{13} & \sigma_{23} & \sigma_{33} \end{bmatrix}, \tag{16}$$

we can obtain the following equation by substituting Eq.(2) into Eq.(10):

$$\begin{aligned} \varepsilon_{33}^{\mathrm{L}} =& (\gamma_{31}^2 s_{11}^{\mathrm{T}} + \gamma_{31}\gamma_{32}s_{16}^{\mathrm{T}} + \gamma_{31}\gamma_{33}s_{15}^{\mathrm{T}} + \gamma_{32}^2 s_{12}^{\mathrm{T}} + \gamma_{32}\gamma_{33}s_{14}^{\mathrm{T}} + \gamma_{33}^2 s_{13}^{\mathrm{T}})(\sigma_{11}\cos^2\beta - \sigma_{12}\sin 2\beta + \sigma_{22}\sin^2\beta) \\ &+ (\gamma_{31}^2 s_{16}^{\mathrm{T}} + \gamma_{31}\gamma_{32}s_{66}^{\mathrm{T}} + \gamma_{31}\gamma_{33}s_{56}^{\mathrm{T}} + \gamma_{32}^2 s_{26}^{\mathrm{T}} + \gamma_{32}\gamma_{33}s_{46}^{\mathrm{T}} + \gamma_{33}^2 s_{36}^{\mathrm{T}})\left(\sigma_{11}\frac{\sin 2\beta}{2} + \sigma_{12}\cos 2\beta + \sigma_{22}\frac{\sin 2\beta}{2}\right) \\ &+ (\gamma_{31}^2 s_{12}^{\mathrm{T}} + \gamma_{31}\gamma_{32}s_{26}^{\mathrm{T}} + \gamma_{31}\gamma_{33}s_{25}^{\mathrm{T}} + \gamma_{32}^2 s_{22}^{\mathrm{T}} + \gamma_{32}\gamma_{33}s_{24}^{\mathrm{T}} + \gamma_{33}^2 s_{23}^{\mathrm{T}})(\sigma_{11}\sin^2\beta + \sigma_{12}\sin 2\beta + \sigma_{22}\cos^2\beta) \\ &+ \left[(\gamma_{31}^2 s_{15}^{\mathrm{T}} + \gamma_{31}\gamma_{32}s_{56}^{\mathrm{T}} + \gamma_{31}\gamma_{33}s_{55}^{\mathrm{T}} + \gamma_{32}^2 s_{25}^{\mathrm{T}} + \gamma_{32}\gamma_{33}s_{45}^{\mathrm{T}} + \gamma_{33}^2 s_{35}^{\mathrm{T}})\,(\sigma_{13}\cos\beta - \sigma_{23}\sin\beta)\right. \\ &+ \left.(\gamma_{31}^2 s_{14}^{\mathrm{T}} + \gamma_{31}\gamma_{32}s_{46}^{\mathrm{T}} + \gamma_{31}\gamma_{33}s_{45}^{\mathrm{T}} + \gamma_{32}^2 s_{24}^{\mathrm{T}} + \gamma_{32}\gamma_{33}s_{44}^{\mathrm{T}} + \gamma_{33}^2 s_{34}^{\mathrm{T}})(\sigma_{13}\sin\beta + \sigma_{23}\cos\beta\right] \\ &+ (\gamma_{31}^2 s_{13}^{\mathrm{T}} + \gamma_{31}\gamma_{32}s_{36}^{\mathrm{T}} + \gamma_{31}\gamma_{33}s_{35}^{\mathrm{T}} + \gamma_{32}^2 s_{23}^{\mathrm{T}} + \gamma_{32}\gamma_{33}s_{34}^{\mathrm{T}} + \gamma_{33}^2 s_{33}^{\mathrm{T}})\sigma_{33}. \end{aligned} \tag{17}$$

3. Example

As an example, consider a cubic film with [111] fibre texture. The transformation matrix $a_{ij}^{(111)}$ from (A_{i}) to (T_{i}) can be obtained from Eq.(1),

$$a_{ij}^{(111)} = \begin{bmatrix} \frac{1}{\sqrt{6}} & \frac{1}{\sqrt{6}} & -\frac{\sqrt{2}}{\sqrt{3}} \\ -\frac{1}{\sqrt{2}} & \frac{1}{\sqrt{2}} & 0 \\ \frac{1}{\sqrt{3}} & \frac{1}{\sqrt{3}} & \frac{1}{\sqrt{3}} \end{bmatrix}. \tag{18}$$

From Eqs.(11)–(15), the matrix representation of s_{ijkl}^{T} is

$$s_{ij}^{\mathrm{T}} = s_{ij}^{(111)} = \begin{bmatrix} s_{11}^{\mathrm{T}} & s_{12}^{\mathrm{T}} & s_{13}^{\mathrm{T}} & 0 & s_{15}^{\mathrm{T}} & 0 \\ s_{12}^{\mathrm{T}} & s_{11}^{\mathrm{T}} & s_{13}^{\mathrm{T}} & 0 & -s_{15}^{\mathrm{T}} & 0 \\ s_{13}^{\mathrm{T}} & s_{13}^{\mathrm{T}} & s_{33}^{\mathrm{T}} & 0 & 0 & 0 \\ 0 & 0 & 0 & s_{44}^{\mathrm{T}} & 0 & -2s_{15}^{\mathrm{T}} \\ s_{15}^{\mathrm{T}} & -s_{15}^{\mathrm{T}} & 0 & 0 & s_{44}^{\mathrm{T}} & 0 \\ 0 & 0 & 0 & -2s_{15}^{\mathrm{T}} & 0 & 2(s_{11}^{\mathrm{T}} - s_{12}^{\mathrm{T}}) \end{bmatrix}, \tag{19}$$

where s_{ij}^{T} is related to the compliance of cubic crystals s_{mn} by

$$\left.\begin{aligned} s_{11}^{\mathrm{T}} &= \frac{1}{2}s_{11} + \frac{1}{2}s_{12} + \frac{1}{4}s_{44}, \\ s_{12}^{\mathrm{T}} &= \frac{1}{6}s_{11} + \frac{5}{6}s_{12} - \frac{1}{12}s_{44}, \\ s_{13}^{\mathrm{T}} &= \frac{1}{3}s_{11} + \frac{2}{3}s_{12} - \frac{1}{6}s_{44}, \\ s_{15}^{\mathrm{T}} &= -\frac{\sqrt{2}}{3}s_{11} + \frac{\sqrt{2}}{3}s_{12} + \frac{\sqrt{2}}{6}s_{44}, \\ s_{33}^{\mathrm{T}} &= \frac{1}{3}s_{11} + \frac{2}{3}s_{12} + \frac{1}{3}s_{44}, \\ s_{44}^{\mathrm{T}} &= \frac{4}{3}s_{11} - \frac{4}{3}s_{12} + \frac{1}{3}s_{44}. \end{aligned}\right\} \tag{20}$$

Substituting Eqs.(4) and (19) into Eq.(17), we have the following equation:

$$\begin{aligned}\varepsilon_{33}^{\mathrm{L}} =& s_{13}^{\mathrm{T}}(\sigma_{11}+\sigma_{22})+s_{33}^{\mathrm{T}}\sigma_{33}\\ &+\left[(s_{11}^{\mathrm{T}}+s_{12}^{\mathrm{T}}-s_{13}^{\mathrm{T}})\sigma_{11}-s_{13}^{\mathrm{T}}\sigma_{22}+(s_{13}^{\mathrm{T}}-s_{33}^{\mathrm{T}})\sigma_{33}\right]\sin^2\psi\\ &+\left\{(\sigma_{22}-\sigma_{11})(s_{11}^{\mathrm{T}}\sin^2\phi+s_{12}^{\mathrm{T}}\cos^2\phi)+\sigma_{12}(s_{11}^{\mathrm{T}}-s_{12}^{\mathrm{T}})\sin 2\phi\right.\\ &\left.+s_{15}^{\mathrm{T}}\left[\sigma_{13}\cos(2\phi+3\beta)-\sigma_{23}\sin(2\phi+3\beta)\right]\right\}\sin^2\psi\\ &+\left\{\frac{s_{15}^{\mathrm{T}}}{2}\left[(\sigma_{11}-\sigma_{22})\cos(\phi+3\beta)-2\sigma_{12}\sin(\phi+3\beta)\right]+\frac{s_{44}^{\mathrm{T}}}{2}(\sigma_{13}\cos\phi+\sigma_{23}\sin\phi)\right\}\sin 2\psi. \end{aligned}\tag{21}$$

In measuring the residual stress with x-ray diffraction, the average of the strains of diffracting planes has to be taken to obtain the x-ray strain. For thin film with FCC structure, such as Al, Cu, Ag, and Ni, the strains are usually measured from (222) or (311) plane by Cu-, Co- or Cr- Kα radiations. The values of ψ and $\phi+\beta$ have been calculated from Eqs.(5) and (6) for FCC films with (111) fibre texture, the results are summarized in Table 1 together with the average values of $\cos(2\phi+3\beta)$, $\sin(2\phi+3\beta)$, $\cos(\phi+3\beta)$ and $\sin(\phi+3\beta)$ for $\phi=0^\circ$ and $\phi=90^\circ$.

Table 1. Values of ψ and $\phi+\beta$ for (222) and (311) diffracting planes of FCC films with (111) fibre texture and corresponding average values of $\cos(2\phi+3\beta)$, $\sin(2\phi+3\beta)$, $\cos(\phi+3\beta)$ and $\sin(\phi+3\beta)$, for $\phi=0^\circ$ and $\phi=90^\circ$.

Diffracting plane	ψ	$\phi+\beta$	Average value							
			$\phi=0^\circ$				$\phi=90^\circ$			
			$\cos(2\phi+3\beta)$	$\sin(2\phi+3\beta)$	$\cos(\phi+3\beta)$	$\sin(\phi+3\beta)$	$\cos(2\phi+3\beta)$	$\sin(2\phi+3\beta)$	$\cos(\phi+3\beta)$	$\sin(\phi+3\beta)$
(222)	70.53°	0° 120° 120°	1	0	1	0	0	–1	–1	0
(311)	58.52°	30° –30° 90° –90° 150° –150°	0	0	0	0	0	0	0	0
	29.50°	60° –60° 180°	–1	0	–1	0	0	1	1	0

By substituting corresponding average values of Table 1 into equation (21), we can get the average of x-ray strain as a function of the macro-stress for each combination of ψ and ϕ.

For the case of $\phi=0^\circ$, the relations between $\bar{\varepsilon}_{33}^{\mathrm{L}}$ and the macro-stress are

$$\begin{aligned}\bar{\varepsilon}_{33}^{\mathrm{L}} =& s_{13}^{\mathrm{T}}(\sigma_{11}+\sigma_{22})+s_{33}^{\mathrm{T}}\sigma_{33}\\ &+\left[(s_{11}^{\mathrm{T}}-s_{13}^{\mathrm{T}})\sigma_{11}+(s_{12}^{\mathrm{T}}-s_{13}^{\mathrm{T}})\sigma_{22}-s_{15}^{\mathrm{T}}\sigma_{13}+(s_{13}^{\mathrm{T}}-s_{33}^{\mathrm{T}})\sigma_{33}\right]\sin^2\psi\\ &+\left[\frac{s_{15}^{\mathrm{T}}}{2}(\sigma_{22}-\sigma_{11})+\frac{s_{44}^{\mathrm{T}}}{2}\sigma_{13}\right]\sin 2\psi, \qquad (\psi=29.50^\circ)\end{aligned}\tag{22}$$

$$\begin{aligned}\bar{\varepsilon}_{33}^{\mathrm{L}} =& s_{13}^{\mathrm{T}}(\sigma_{11}+\sigma_{22})+s_{33}^{\mathrm{T}}\sigma_{33}\\ &+\left[(s_{11}^{\mathrm{T}}-s_{13}^{\mathrm{T}})\sigma_{11}+(s_{12}^{\mathrm{T}}-s_{13}^{\mathrm{T}})\sigma_{22}+(s_{13}^{\mathrm{T}}-s_{33}^{\mathrm{T}})\sigma_{33}\right]\sin^2\psi\\ &+\frac{s_{44}^{\mathrm{T}}}{2}\sigma_{13}\sin 2\psi, \qquad (\psi=58.52^\circ)\end{aligned}\tag{23}$$

$$\begin{aligned}\overline{\varepsilon}_{33}^{\mathrm{L}} =& s_{13}^{\mathrm{T}}(\sigma_{11}+\sigma_{22})+s_{33}^{\mathrm{T}}\sigma_{33}\\ &+\left[(s_{11}^{\mathrm{T}}-s_{13}^{\mathrm{T}})\sigma_{11}+(s_{12}^{\mathrm{T}}-s_{13}^{\mathrm{T}})\sigma_{22}+s_{15}^{\mathrm{T}}\sigma_{13}+(s_{13}^{\mathrm{T}}-s_{33}^{\mathrm{T}})\sigma_{33}\right]\sin^2\psi\\ &+\left[\frac{s_{15}^{\mathrm{T}}}{2}(\sigma_{11}-\sigma_{22})+\frac{s_{44}^{\mathrm{T}}}{2}\sigma_{13}\right]\sin 2\psi. \qquad (\psi=70.53^\circ)\end{aligned} \tag{24}$$

For the case of $\phi = 90^\circ$, we have the following equations

$$\begin{aligned}\overline{\varepsilon}_{33}^{\mathrm{L}} =& s_{13}^{\mathrm{T}}(\sigma_{11}+\sigma_{22})+s_{33}^{\mathrm{T}}\sigma_{33}\\ &+\left[(s_{12}^{\mathrm{T}}-s_{13}^{\mathrm{T}})\sigma_{11}+(s_{11}^{\mathrm{T}}-s_{13}^{\mathrm{T}})\sigma_{22}-s_{15}^{\mathrm{T}}\sigma_{23}+(s_{13}^{\mathrm{T}}-s_{33}^{\mathrm{T}})\sigma_{33}\right]\sin^2\psi\\ &+\left[\frac{s_{15}^{\mathrm{T}}}{2}(\sigma_{11}-\sigma_{22})+\frac{s_{44}^{\mathrm{T}}}{2}\sigma_{23}\right]\sin 2\psi, \qquad (\psi=29.50^\circ)\end{aligned} \tag{25}$$

$$\begin{aligned}\overline{\varepsilon}_{33}^{\mathrm{L}} =& s_{13}^{\mathrm{T}}(\sigma_{11}+\sigma_{22})+s_{33}^{\mathrm{T}}\sigma_{33}\\ &+\left[(s_{12}^{\mathrm{T}}-s_{13}^{\mathrm{T}})\sigma_{11}+(s_{11}^{\mathrm{T}}-s_{13}^{\mathrm{T}})\sigma_{22}+(s_{13}^{\mathrm{T}}-s_{33}^{\mathrm{T}})\sigma_{33}\right]\sin^2\psi\\ &+\frac{s_{44}^{\mathrm{T}}}{2}\sigma_{23}\sin 2\psi, \qquad (\psi=58.52^\circ),\end{aligned} \tag{26}$$

$$\begin{aligned}\overline{\varepsilon}_{33}^{\mathrm{L}} =& s_{13}^{\mathrm{T}}(\sigma_{11}+\sigma_{22})+s_{33}^{\mathrm{T}}\sigma_{33}\\ &+\left[(s_{12}^{\mathrm{T}}-s_{13}^{\mathrm{T}})\sigma_{11}+(s_{11}^{\mathrm{T}}-s_{13}^{\mathrm{T}})\sigma_{22}+s_{15}^{\mathrm{T}}\sigma_{23}+(s_{13}^{\mathrm{T}}-s_{33}^{\mathrm{T}})\sigma_{33}\right]\sin^2\psi\\ &-\left[\frac{s_{15}^{\mathrm{T}}}{2}(\sigma_{11}-\sigma_{22})-\frac{s_{44}^{\mathrm{T}}}{2}\sigma_{23}\right]\sin 2\psi. \qquad (\psi=70.53^\circ)\end{aligned} \tag{27}$$

For any $(h'k'l')$ diffracting plane, the average normal strain $\overline{\varepsilon}_{33}^{\mathrm{L}}$ along (ϕ,ψ) orientation can be rewritten as

$$\overline{\varepsilon}_{33}^{\mathrm{L}} = \varepsilon_{\phi\psi} = \frac{d_{\phi\psi}-d_0}{d_0}, \tag{28}$$

where d_0 is the unstressed spacing of the $(h'k'l')$ diffracting plane and can be calculated from unstressed lattice constant a_0,[19]

$$d_0 = \frac{a_0}{\sqrt{h'^2+k'^2+l'^2}}, \tag{29}$$

where $d_{\phi\psi}$ is the stressed plane spacing along $(\phi,\ \psi)$ orientation and can be determined from the diffraction peak position θ of the $(h'k'l')$ plane and the radiation wavelength λ through Bragg's law:

$$2d_{\phi\psi}\sin\theta = \lambda. \tag{30}$$

By substituting the six determined values of x-ray strain into Eqs.(22)–(27) and corresponding single-crystal compliances into Eq.(19) and then into Eqs.(22)–(27), three-dimensional (residual) stresses in thin FCC films or bulk materials with [111] fibre texture can be determined by solving Eqs.(22)–(27).

It is noted that the accuracy of the three-dimensional (residual) stresses are determined by the accuracy of the average normal strain $\overline{\varepsilon}_{33}^{\mathrm{L}} = \varepsilon_{\phi\psi}$ which is determined further by the unstressed and stressed plane spacing d_0 and $d_{\phi\psi}$ of the $(h'k'l')$ diffracting plane. In order to avoid large errors in $\overline{\varepsilon}_{33}^{\mathrm{L}} = \varepsilon_{\phi\psi}$, the unstressed plane spacing d_0 or unstressed lattice constant a_0 must be known very accurately. Since $d_{\phi\psi}$ is also required to have an accurate value, Kα doublet should be separated, the intensity should be corrected and the environment temperature had better be kept constant. Furthermore, from differential formula of Bragg's equation (30)

$$\frac{\Delta d_{\phi\psi}}{d_{\phi\psi}} = -\mathrm{ctg}\theta\cdot\Delta\theta + \frac{\Delta\lambda}{\lambda}, \tag{31}$$

we know that the error in $d_{\phi\psi}$ comes from both errors of the diffraction peak position $\Delta\theta$ and errors of the radiation wavelength $\Delta\lambda$. Since the accuracy of the x-ray wavelength has already come up to 10^{-7}nm and the errors of the radiation wavelength $\Delta\lambda$ are kept constant for all measurements, the $\Delta\lambda$ can be neglected and Eq.(31) becomes

$$\frac{\Delta d_{\phi\psi}}{d_{\phi\psi}} = -\mathrm{ctg}\theta\cdot\Delta\theta. \tag{32}$$

Obviously, a large θ of a selected $(h'k'l')$ diffracting plane will result in a small error in $d_{\phi\psi}$ for a certain $\Delta\theta$.

4. Conclusions

A novel x-ray method is proposed for the evaluation of three-dimensional (residual) stress in cubic films having any $[hkl]$ fibre texture with the fibre axis perpendicular to the film surface. The method can also be extended to other crystal systems.

References

[1] Bϕrgesen P, Lee J K, Gleixner R and Li C Y 1992 *Appl. Phys. Lett.* **60** 1076

[2] Vinci R P, Marieb T N and Bravman J C 1993 *Mat. Res. Soc. Symp. Proc.* **308** 297

[3] Gungor M R and Maroudas D 1998 *Surf. Sci.* **415** L1055

[4] Zhang J M and Xu K W 2004 *Chin. Phys.* **13** 205

[5] Chaudhari P 1974 *J. Appl. Phys.* **45** 4339

[6] Gerth D, Katzer D and Schwarxer R 1992 *Mater. Sci. Forum* **94–96** 557

[7] Shute C J and Cohen J B 1991 *J. Mater. Res.* **6** 950

[8] Zhang J M, Xu K W and Ji V 2001 *Appl. Surf. Sci.* **180** 1

[9] Zhang J M and Xu K W 2004 *Acta Phys. Sin.* **53** 2439 (in Chinese)

[10] Hauk V M 1983 *Adv. In x-ray Anal.* **27** 101

[11] Noyan I C and Cohen J B 1983 *Adv. In x-ray Anal.* **27** 129

[12] Dölle H 1979 *J. Appl. Cryst.* **12** 489

[13] Brakman C M 1983 *J. Appl. Cryst.* **16** 325

[14] Ohmi T, Saito T, Otsuki M and Shibata T 1991 *J. Electrochem. Soc.* **138** 1089

[15] Tracy D P and Knorr D B 1993 *J. Elec. Mater.* **22** 611

[16] Zhang J M, Xu K W and Ji V 2002 *Appl. Surf. Sci.* **185** 177

[17] Zhang J M, Ma F and Xu K W 2004 *Appl. Surf. Sci.* **229** 34

[18] Nye J F 1972 *Physical Properties of Crystals* (Oxford: Clarendon) p131

[19] Pearson W B 1958 *A Handbook of Lattice Spacings and Structures of Metals and Alloy* (New York: Pergamon)