X-RAY STRESS DETERMINATION OF COLD-ROLLED STEEL SHEET USING ORIENTATION DISTRIBUTION FUNCTION

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ABSTRACT

Difficulties often attended determining the residual stress of cold-rolled steel sheets with crystallographic textures from the conventional X-ray stress measurement, i.e., the sin²ψ technique, because the technique is constructed on assumption that specimens are isotropic elastic polycrystalline materials. Therefore, the aim of this investigation is to develop an effective X-ray stress measurement for textured materials. At first, the relation between the stress and lattice strain measured by the X-ray diffraction is formulated by an average method. In the method, the strain is averaged as the expected value with a weight function from the crystallite orientation distribution function (ODF). Then, in order to lower the effect on the unstressed lattice spacing, an X-ray stress determination is introduced by using the derivative of the X-ray stress-strain relation. Moreover, the X-ray stress measurement is actually applied to cold-rolled steel sheets, and the stresses are determined.

INTRODUCTION

The conventional X-ray stress measurement, so-called sin²ψ technique requires the specimen to be macroscopically an isotropic polycrystalline material, which is composed of anisotropic single crystallites. However, actual polycrystalline materials are macroscopically anisotropic materials having the bias of the crystallite orientation distribution in greater or lesser degree. For instance, there are coating films by physical vapor deposition (PVD) and chemical vapor deposition (CVD) which have preferred orientations along the normal to the film surfaces, casting parts which have the anisotropy due to one-way solidification and so on.

The applicable X-ray stress measurement should be necessary for such anisotropic materials. In the present study, therefore, an effective X-ray stress measurement is formulated for textured materials on assumption that the crystalline anisotropy is represented by the crystallite orientation distribution function (ODF). At first, the relation between the stress in the specimen and the lattice strain measured by the X-ray diffraction method, which is the fundamental equation of the X-ray stress measurement, is derived from Hook’s law in the single crystal using ODF. Then, in order to lower the effect on the unstressed lattice spacing, the differential form of the X-ray stress-strain relation is introduced as an X-ray stress determination. Moreover, the X-ray stress measurement is actually applied to cold-rolled steel sheets, and discussed.
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X-RAY STRESS MEASUREMENT

A. X-ray stress analysis for cubic polycrystalline materials

Three orthogonal coordinate systems are introduced in the beginning. As shown in Figure 1, the sample coordinate system (S system) depends on the form or the rolled direction of the specimen. In order that the new $S_3$ axis is parallel to the normal direction of the diffraction plane $(hkl)$, the transformed system by the rotation with the Eulerian angles $(\phi, \psi, 0)$ is the laboratory coordinate system (L system). Three axes of the crystal coordinate system (C system) correspond to the crystallographic axes of a cubic crystal. The relation among these systems is shown in Figure 2 with three matrices of orthogonal transformations. These matrices are represented by the rotation matrix $R_\gamma(a, b, c)$ with the Eulerian angles $(a, b, c)$, i.e.

$$
\begin{align*}
\omega_y &= R_y(\phi, \psi, 0) \\
\pi^{-1}_y &= R_y(\psi, \Theta, \Phi) \\
\gamma_y &= R_y(\alpha, \beta, \gamma)
\end{align*}
$$

where $\gamma_{3i}$ is also represented by Miller indices $(hkl)$ of diffraction plane as follows:

$$
\gamma_{3i} = \frac{1}{\sqrt{h^2 + k^2 + l^2}}(h \ k \ l).
$$

Fig. 1 Relation between the sample and laboratory coordinate systems.

Fig. 2 Definitions of orthogonal transformation tensors among three coordinate systems.
In the next place, the relation between the stress \( \sigma'_{ij} \) in the S system and the strain \( \varepsilon'_{33} \) along the \( L_3 \) axis is formulated for polycrystalline materials. The generalized Hooke’s law with relation to the stress \( \sigma'_{ij} \) and strain \( \varepsilon'_{ij} \) in the C system is assumed.

\[
e_C' = S_C'_{ijkl} \varepsilon_C'_{kl}
\]

where \( S_C'_{ijkl} \) denotes the elastic compliance of single crystal. Under the transformations:

\[
\varepsilon'_{33} = \gamma_{3i} \gamma_{3j} \varepsilon_C'_{ij}, \quad \sigma'_{ij} = \pi^{-1}_{km} \pi^{-1}_{ln} \sigma_C'_{mn} ,
\]

the X-ray stress-strain relation for single crystals is obtained from Eq. 1.

\[
\varepsilon_{33} = S_{33ij} \sigma_{ij}' ,
\]

\[
S_{ijkl}' \equiv \gamma_{ia} \gamma_{jb} S_{abcdef} \pi^{-1}_{al} \pi^{-1}_{dl} .
\]

Not all crystallites contribute the diffraction phenomenon on the Bragg’s law, only crystallites that have crystal planes with normal to the \( L_3 \) axis, namely the measured lattice strain has the rotation symmetry about the axis. Thus the measured lattice strain is obtain from the average with a weight function:

\[
\overline{\varepsilon_{33}'} = \frac{\int_{0}^{2\pi} f(\gamma)\varepsilon_{33} d\gamma}{\int_{0}^{2\pi} f(\gamma) d\gamma}
\]

where the weight function \( f(\gamma) \) is given by ODF \( F(\Psi, \Theta, \Phi) \).

\[
f(\gamma) = F(\Psi(\gamma),\Theta(\gamma),\Phi(\gamma))
\]

The Eulerian angles (\( \Psi, \Theta, \Phi \)) have a parameter \( \gamma \) by the transformation rule.

\[
\pi^{-1}_{ij} = \gamma_{ik} \omega_{kj}
\]

Introducing Eq.7 into Eq.5, therefore, the relation between the X-ray stress and strain for polycrystalline materials is obtained by

\[
\overline{\varepsilon_{33}'} = \overline{S_{33ij}'} \overline{\sigma_{ij}'}
\]

for the Reuss model which assumes constant stress.

**B. X-ray stress determination**

Measuring the variation \( \theta - \theta_0 \) from the unstressed diffraction angle \( \theta_0 \), the lattice strain \( \varepsilon_{33}' \) is determined by the difference of the Bragg diffraction condition

\[
\varepsilon_{33}' = \frac{\bar{d} - d_0}{d_0} = - (\theta - \theta_0) \cot \theta_0
\]

where \( d \) and \( d_0 \) are lattice spacings in stressed and unstressed state, respectively. On the other hand, the X-ray elastic compliance \( S_{33ij}' \) is determined by Eq.6. Therefore, the X-ray stress is derived from Eq.10.

However, in order to lower the measurement error on the unstressed lattice spacing, the differential of Eq.11 for \( \phi \) or \( \sin^2 \phi \):

\[
\frac{\partial \varepsilon_{33}'}{\partial \psi} = \frac{1}{d_0} \frac{\partial \bar{d}}{\partial \psi} = - \frac{\partial \theta}{\partial \psi} \cot \theta_0
\]

is introduced. Thus the X-ray stress is determined by the differential of Eq.10.

\[
\frac{\partial \varepsilon_{33}'}{\partial \psi} = \frac{\partial S_{33ij}'}{\partial \psi} \sigma_{ij}'
\]
Especially, the X-ray elastic compliances for diffraction planes \{111\} and \{100\} is derived the independent equation on the rotation angle \(\gamma\) from Eq.6 as follows:

\[
S_y^{33y} = \left( \frac{1}{3} S_x^0 + S_x^{12} \right) \delta_y + \frac{1}{2} S_x^{44} \omega_{3y} \omega_{3y} \quad \text{for} \ \{111\},
\]
\[
S_x^{33y} = S_x^{12} \delta_y + (S_x^{11} - S_x^{12}) \omega_{3y} \omega_{3y} \quad \text{for} \ \{100\},
\]
\[
S_x^0 = S_x^{11} - S_x^{12} - \frac{1}{2} S_x^{44}
\]

where \(\delta_y\) and \((S_x^{11}, S_x^{12}, S_x^{33})\) denote the Kronecker's delta and the elastic constants of cubic crystal, respectively. Then, the \(e^{t_{33}} - \sin^2 \phi\) diagram shows linearity without the influence of the elastic anisotropy. The result is equivalent to the equation by Honda\(^8\) and Dölle et al.\(^9\) Furthermore, since the averaged X-ray elastic compliance for isotropic polycrystalline materials with \(f(\gamma) = 1\) is given by

\[
S_x^{33y} = (S_x^{12} + S_x^0 \Gamma) \delta_y + (S_x^{11} - S_x^{12} - 3S_x^0 \Gamma) \omega_{3y} \omega_{3y},
\]
\[
\Gamma = \gamma_{31} \gamma_{31}^2 + \gamma_{32} \gamma_{32}^2 + \gamma_{33} \gamma_{33}^2
\]

Eq.13 with Eq.17 in plane stress state is equal to the X-ray stress determination of the \(\sin^2 \phi\) technique in the Reuss model:

\[
\sigma^y = \frac{-\cot \theta_o}{S_x^{11} - S_x^{12} - 3S_x^0 \Gamma} \frac{\Delta \theta}{\Delta \sin^2 \psi},
\]

---

Fig. 3 Crystallite orientation distribution function of the ferrite phase for cold-rolled steel sheet
\[ \sigma^x = \sigma_{11}^x \cos^2 \phi + \sigma_{12}^x \sin 2\phi + \sigma_{22}^x \sin^2 \phi. \]  

(20)

**EXPERIMENTAL**

A very low carbon steel sheet with \(10 \times 50 \times 0.90\) mm was employed as the specimen after a cold-rolled treatment up to 80%. The ODF results of the ferrite phase are shown in Figure 3. The diffraction angles \(2\theta\) of \(\alpha\) Fe\(211\) were measured in \(\phi=0\) degrees by an X-ray diffractometer with the iso-inclination method. Then, the \(S_1\) axis was fixed along the rolled direction. Table 1 listed the X-ray stress measurement condition. \((S_{11}^c, S_{12}^c, S_{44}^c)=(7.67, -2.83, 8.57)\) TPa was used as the elastic constants of \(\alpha\) Fe single crystal.

To verify the present X-ray stress measurement, the X-ray stress values were compared with the applied stress values by loading experiments with a small four-point jig. The applied stress \(\sigma_{11}^A\) was determined by Hooke’s law:

\[ \sigma_{11}^A = E^A \varepsilon_{11}^A \]  

(21)

where the applied strain \(\varepsilon_{11}^A\) was measured by a strain gage on the specimen surface and the mechanical elastic constant \(E^A=192.9\) GPa was calculated by the Reuss model for macroscopic elastic constants.

<table>
<thead>
<tr>
<th>X-ray tube / Filter</th>
<th>Cr-K (\alpha) / Vanadium</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diffraction plane</td>
<td>(\alpha) Fe 211</td>
</tr>
<tr>
<td>Diffraction angle</td>
<td>156deg</td>
</tr>
<tr>
<td>Tube voltage &amp; current</td>
<td>30kV &amp; 10mA</td>
</tr>
<tr>
<td>Step width</td>
<td>Peak 0.3, B.G. 0.5 deg</td>
</tr>
<tr>
<td>Peak determination</td>
<td>Gaussian fitting</td>
</tr>
<tr>
<td>Fixed time</td>
<td>3s</td>
</tr>
<tr>
<td>Scanning method</td>
<td>Fixed (\phi) method</td>
</tr>
</tbody>
</table>

Fig.4 Non linearity of diffraction angles for each applied strain.
RESULTS AND DISCUSSIONS

A. Non-linearity in the $\sin^2 \phi$ diagram

By means of the $2\theta - \sin^2 \phi$ diagrams for applied stress values, the measurement results of $\alpha$ Fe211 diffraction is shown in Figure 4. These $2\theta$ values for each $\sin^2 \phi$ values are calculated by the Gaussian fitting for maximum diffraction intensities in the $2\theta$-intensity scanning. The diagrams do not show linearity for strong texture. Consequently, it is impossible to apply the conventional $\sin^2 \psi$ technique, which demands the linearity in the diagram, to the textured material as the X-ray stress determination.

![Fig.5 Gradient of lattice strain on $\phi$ for $\alpha$ Fe211.](image)

Fig.5 Gradient of lattice strain on $\phi$ for $\alpha$ Fe211.

![Fig.6 Relative intensity distribution about the normal direction of the diffraction plane $\alpha$ Fe211 by the ODF analysis in ($\phi$, $\phi$) = (0,10) degrees.](image)

Fig.6 Relative intensity distribution about the normal direction of the diffraction plane $\alpha$ Fe211 by the ODF analysis in ($\phi$, $\phi$) = (0,10) degrees.
Table 2 X-ray stress values for applied strains.

<table>
<thead>
<tr>
<th>Applied strain, $\times 10^{-6}$</th>
<th>X-ray stress by present method $\sigma_{11}^{s}$,MPa</th>
<th>X-ray stress by the $\sin^2 \phi$ technique $\sigma_{11}^{s}$,MPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>-53.7</td>
<td>-2.82</td>
</tr>
<tr>
<td>250</td>
<td>13.3</td>
<td>25.2</td>
</tr>
<tr>
<td>500</td>
<td>62.5</td>
<td>50.1</td>
</tr>
<tr>
<td>750</td>
<td>123.9</td>
<td>82.1</td>
</tr>
</tbody>
</table>

Fig. 7 Variations of X-ray stress values for applied stresses by ODF analysis and linear Reuss model.

B. Compression with applied stress

The X-ray stress values were determined from Eq.13 by the least-squares method. In the calculation, the differential values of the measured lattice strain were calculated from Eq.12 by the polynomial approximation, as shown in Figure 5. In the same way, the differential values of the X-ray elastic compliance were calculated after averaging Eq.6. In illustration of the weight function, Figure 6 shows the weight function from ODF of Fe21 1 in (4, (1, )=(0, 10 deg).

The X-ray stress values by the present method and the $\sin^2 \phi$ technique in the Reuss model are shown in Table 2. Then, the difference between the applied and X-ray stress is shown in Figure 7. The relations between the applied and X-ray stress have linearity in each method, and these gradients are 1.21 and 0.58 for the present method and the $\sin^2 \phi$ technique, respectively. In result, it is evident that the calculated values by the present method is nearer to the measured values than by the $\sin^2 \phi$ technique.

CONCLUSIONS

The X-ray stress measurement for cubic polycrystalline materials, which consists of the averaged lattice strain with ODF and the differential form of the stress-strain relation, was devol-
opened in the Reuss model. Furthermore, the X-ray stress measurement was applied to a cold-rolled steel sheet, and the X-ray stress was determined.

1. The relation between the X-ray stress and the measured lattice strain was derived from ODF. In general, the relation had non-linearity in the \( \sin^2 \phi \) diagram, but the relations for \{111\} and \{100\} had linearity. Then, the relation for isotropic materials was coincident with the conventional \( \sin^2 \phi \) technique.

2. In order to control the measurement error of the Bragg angle in unstressed state, the differential form of the relation between the X-ray stress and lattice strain was introduced. In result, the X-ray stress was possible to determine for textured materials with the nonlinear \( \sin^2 \phi \) diagrams even if the \( \sin^2 \phi \) technique was impossible to apply.

3. Actually, it was hard to apply the \( \sin^2 \phi \) technique to the cold-rolled steel sheet employed because of the nonlinearity in the \( \sin^2 \phi \) diagram.

4. As a result of loading tests, the X-ray stress by the present method was nearer the applied stress than the \( \sin^2 \phi \) technique.

REFERENCES