X-RAY STRESS MEASUREMENT OF NICKEL-BASE SINGLE CRYSTAL
SUPERALOY USING TWO-DIMENSIONAL DETECTOR

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ABSTRACT

The stress in a single crystal of nickel-base superalloy with 72% volume fraction of γ'-phase was measured by the X-ray method. The specimen whose surface normal was parallel to [001] direction was oscillated around φ-axis during recording of the X-ray diffraction pattern with a two-dimensional position sensitive proportional counter (PSPC). The stress was determined from the measured strain using the multiple regression method and the two-tilt method. The uniaxial stress was applied along [100] direction and three stress components were measured with the X-ray methods. The stress along [100] direction, \( \sigma_{11} \), measured with the X-ray method increased proportionally to the applied uniaxial stress, and the measured stress was about 5% smaller than the applied stress. The other stress components, \( \sigma_{22} \) and \( \sigma_{12} \), did not change with the applied stress. Those two methods were applied to measure the machining residual stress of the specimen made for fatigue tests. The residual stress was a compression of about 700 MPa on the surface and quickly decreased to zero at about 15 \( \mu \)m beneath the surface. The increase in the full-width at half maximum was observed within the depth of about 15 \( \mu \)m from the machined surface, corresponding to the depth of the compression zone.

INTRODUCTION

Nickel-base superalloys are widely used as high temperature materials in turbine blade of gas turbine engines. The superalloys are monocrytsalline or have coarse-grained structure to achieve a high creep resistance. The fatigue properties are also important to guarantee the performance as turbine blades. The residual stress may have an influence on creep properties as well as fatigue strength. To understand the influence of the residual stress on high temperature properties of superalloys, the X-ray measurement of stress in single grains is necessary. The multiple regression method is a versatile method to measure the stress in single crystals [1], and more simple two-tilt method can be used for the cases of cubic single crystals with special orientation [2]. These two methods were applied to measure the stress in single crystals of silicon in our previous papers [2,3].

In the present study, the multiple regression method and two-tilt method are used for stress measurement of single crystals of nickel-base superalloys whose crystalline structure is cubic. The accuracy of the measured stress was first examined to measure the loading stress of the specimen subjected to four-point bending. The method was applied to measure the residual stress below the ground surface. The full width at half maximum (FWHM) of the diffraction profiles is also measured to observe the distribution of plastic strain below the surface.
METHOD OF X-RAY STRESS MEASUREMENT OF SINGLE CRYSTALS

Figure 1 shows the relation among three coordinate systems: the specimen coordinates, \( P_i \), the crystal coordinates, \( X_i \), and the laboratory coordinates, \( L_i \). The transformation matrices are shown in Fig. 2. Figure 3 shows the specimen coordinates, \( P_1, P_2, P_3 \), where the [001] direction is normal to the surface, and the normal stress in \( P_1 \) direction [100] is \( \sigma_{11} \) and that in \( P_2 \) direction [010] is \( \sigma_{22} \). The normal strain along \( L_3 \) direction, \( \varepsilon_{33} \), is measured by X-ray diffraction, where the inclination angles, \( \varphi \) and \( \psi \), are shown. By assuming plane stress, the measured strain is related to the stress in terms of the specimen coordinate as follows [1,3]. The relation between \( \varepsilon_{33} \) and the stress in the specimen coordinates \( \sigma_{ij} \) is given as

\[
\varepsilon_{33} = s_{0} \left( \gamma_{31} \sigma_{11} + \gamma_{32} \sigma_{12} + \gamma_{33} \sigma_{22} \right) + \frac{1}{2} s_{44} \left( \sigma_{11} + \sigma_{22} \right) + s_{ij} \left( \sigma_{ij} \right)
\]

(2)

where \( s_{ij} \) is the single crystal compliances and \( s_{0} = s_{11} - s_{12} - s_{44} / 2 \). The values of \( \gamma_{3i} \) is the direction cosines of \( L_3 \) to the crystal coordinate \( X_i \) and is given for \( hkl \) diffraction by

\[
\gamma_{31} = \frac{h}{\sqrt{h^2 + k^2 + l^2}}, \quad \gamma_{32} = \frac{k}{\sqrt{h^2 + k^2 + l^2}}, \quad \gamma_{33} = \frac{l}{\sqrt{h^2 + k^2 + l^2}}
\]

(3)

The direction of \( P_1 \) is expressed in terms of the crystalline coordinates as \( [\pi_{11}, \pi_{12}, \pi_{13}] \) and the surface normal \( P_3 \) is \( [\pi_{31}, \pi_{32}, \pi_{33}] \). The inclination angles, \( \psi \) and \( \varphi \), are given by

\[
\tan \varphi = \omega_{32} / \omega_{31}, \quad \sin^2 \psi = \omega_{31}^2 + \omega_{32}^2
\]

(4)

\[
\omega_{31} = \gamma_{31} \pi_{11} + \gamma_{32} \pi_{12} + \gamma_{33} \pi_{13}, \quad \omega_{32} = \gamma_{31} \pi_{21} + \gamma_{32} \pi_{22} + \gamma_{33} \pi_{23}
\]

(5)

Fig. 1. Relation among three coordinate systems: specimen \( P_i \), crystal \( X_i \), and laboratory \( L_i \).

Fig. 2. Transformation matrices of coordinates.
In the stress measurement, the strains in several directions, $\varepsilon_{33}^{L(n)} (n=1,2,3\ldots)$, are measured and they are related to stresses as

$$\varepsilon_{33}^{L(n)} = A_n \sigma_{11} + B_n \sigma_{12} + C_n \sigma_{22}$$  \hspace{1cm} (6)

$$A_n = s_0 \left( \gamma_{31}^2 \pi_{11}^2 + \gamma_{32}^2 \pi_{12}^2 + \gamma_{33}^2 \pi_{13}^2 \right) + s_{12} + \frac{1}{2} s_{44} \cos^2 \varphi \sin^2 \psi$$

$$B_n = 2 s_0 \left( \gamma_{33}^2 \pi_{11} \pi_{21} + \gamma_{33}^2 \pi_{12} \pi_{22} + \gamma_{33}^2 \pi_{13} \pi_{23} \right) + \frac{1}{2} s_{44} \sin^2 \varphi \sin^2 \psi$$

$$C_n = s_0 \left( \gamma_{32}^2 \pi_{21}^2 + \gamma_{32}^2 \pi_{22}^2 + \gamma_{32}^2 \pi_{23}^2 \right) + s_{12} + \frac{1}{2} s_{44} \sin^2 \varphi \sin^2 \psi$$  \hspace{1cm} (7)

The strain is determined from the change of diffraction angles.

$$\varepsilon_{33}^{L(n)} = -\frac{1}{2} \cot \theta_0 (2 \theta_n - 2 \theta_0)$$  \hspace{1cm} (8)

where $2 \theta_n$ is the diffraction angle and $2 \theta_0$ is the angle for stress-free materials. From eqns. (6) and (8), the following equation is derived

$$2 \theta_n = -\frac{2 \sigma_{11}}{\cot \theta_0} A_n - \frac{2 \sigma_{12}}{\cot \theta_0} B_n - \frac{2 \sigma_{22}}{\cot \theta_0} C_n + 2 \theta_0 = \alpha_{11} A_n + \alpha_{12} B_n + \alpha_{22} C_n + 2 \theta_0$$  \hspace{1cm} (9)

The stresses $\sigma_{11}, \sigma_{12}, \sigma_{22}$ are related to $\alpha_{11}, \alpha_{12}, \alpha_{22}$ as

$$\sigma_{11} = -\frac{1}{2} \alpha_{11} \cot \theta_0, \quad \sigma_{12} = -\frac{1}{2} \alpha_{12} \cot \theta_0, \quad \sigma_{22} = -\frac{1}{2} \alpha_{22} \cot \theta_0$$  \hspace{1cm} (10)

The stresses can be determined from more than five measured diffraction angles by multiple regression. The stereographic projection is shown in Fig. 4. In the multiple regression method, the strains for five diffractions 133, 133, 313, 313, 331 are measured. Table 1 summarizes the inclination angles for five diffractions.

When the single crystal has [001] axis perpendicular to the specimen surface, a simple two-tilt
method can be used. Figure 4 shows the standard stereographic projection. The 420 diffraction can be obtained at two inclination angles \( \psi \) for the cases of \( \varphi = 0 \) and \( 90^\circ \). The X-ray measured strain is related to the stress as follows [2]:

\[
\varepsilon_\psi = \frac{s_{44} + 2s_{0}}{4} \left[ (\sigma_{11} + \sigma_{22}) + (\sigma_{11} - \sigma_{22}) \cos 2\varphi \right] \sin^2 \psi + \frac{s_{44}}{2} \sigma_{12} \sin 2\varphi \sin^2 \psi + s_{12} (\sigma_{11} + \sigma_{22})
\]

(11)

The slope of the \( 2\theta \cdot \sin^2 \psi \) diagrams for \( \varphi = 0^\circ \) and \( \varphi = 90^\circ \) are

\[
M_{\varphi=0} = \frac{\partial 2\theta}{\partial \sin^2 \psi} = -\left( s_{44} + 2s_{0} \right) \sigma_{11} \tan \theta_0 \left( \frac{180}{\pi} \right)
\]

(12)

\[
M_{\varphi=90} = \frac{\partial 2\theta}{\partial \sin^2 \psi} = -\left( s_{44} + 2s_{0} \right) \sigma_{22} \tan \theta_0 \left( \frac{180}{\pi} \right)
\]

(13)

From the slope, the stresses, \( \sigma_{11} \) and \( \sigma_{22} \), are determined. This method is simpler and does not require the precise \( d_0 \) value for stress determination. The inclination angles for the two-tilt method are summarized in Table 2.

**EXPERIMENTAL PROCEDURE**

The experimental material is a nickel-base single crystal superalloy. The microstructure is mainly \( \gamma' \)-phase with a volume fraction of 28 % of \( \gamma \)-phase. To measure the loading stress under four-point bending, a plate specimen with 10 mm in length, 1mm in thickness and 50 mm in length was machined to have the orientation shown in Fig. 1. The residual stress of the ground surface of the bar specimen with a diameter of 6.35 mm for fatigue tests was measured after successive removal of the surface layer by electropolishing. The axial direction of the bar specimen was parallel to the \( [001] \) direction of the crystal. The axial residual stress on the surface with the normal direction of \( [100], [010], [100], [010] \) was measured.

The X-ray equipment used had a two-dimensional detector (Bruker AXS D8 DISCOVER with GADDS). The Cr-\( K\alpha \) radiation was used for stress measurement. The X-ray was collimated by double pinhole of 0.5 mm in diameter. In the experiments, the angle \( \varphi \) was oscillated between \( \pm 3.0^\circ \), after setting the calculated inclination angles of \( \varphi \) and \( \psi \).

### Table 1 \( \varphi \)-angle and \( \psi \)-angle of 3 3 1 diffraction.

<table>
<thead>
<tr>
<th>Diffraction plane</th>
<th>3 1 3</th>
<th>1 3 3</th>
<th>1 3 3</th>
<th>3 1 3</th>
<th>3 3 1</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \varphi )-angle, deg</td>
<td>18.44</td>
<td>71.57</td>
<td>108.44</td>
<td>161.57</td>
<td>44.75</td>
</tr>
<tr>
<td>( \psi )-angle, deg</td>
<td>46.51</td>
<td></td>
<td></td>
<td></td>
<td>76.74</td>
</tr>
</tbody>
</table>

### Table 2 \( \varphi \)-angle and \( \psi \)-angle of 4 2 0 diffraction.

<table>
<thead>
<tr>
<th>Diffraction plane</th>
<th>2 0 4</th>
<th>0 2 4</th>
<th>4 0 2</th>
<th>0 4 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \varphi )-angle, deg</td>
<td>0</td>
<td>90</td>
<td>0</td>
<td>90</td>
</tr>
<tr>
<td>( \psi )-angle, deg</td>
<td>26.57</td>
<td></td>
<td>63.44</td>
<td></td>
</tr>
</tbody>
</table>
EXPERIMENTAL RESULTS AND DISCUSSION

RELATION BETWEEN X-RAY MEASURED STRESS AND APPLIED STRESS

Figure 5(a) shows the diffraction image recorded on two-dimensional detector. It is possible to see the movement of the diffraction pattern by the oscillation of the inclination angle $\varphi$, and the diffraction profile is obtained as the intensity distribution as shown in Fig. 5(b), where the square indicate the region of integration of the intensity. Two peaks for $K\alpha 1$ and $K\alpha 2$ radiation can be seen, while diffractions from $\gamma'$- and $\gamma$-phases are not separable.

The elastic compliances of the single crystals required for stress calculation is derived as follows. Table 4 summarizes the published values of stiffnesses, $c_{ij}$, for $\gamma'$- and $\gamma$-phases [4], and the anisotropy constant $A$ and Poisson's ratio $\nu$ are calculated by

$$A = \frac{2c_{44}}{c_{11} - c_{12}} = \frac{2(s_{11} - s_{12})}{s_{44}}$$

(14)

$$\nu = \frac{c_{12}}{c_{11} + c_{12}} = -\frac{s_{12}}{s_{11}}$$

(15)

The stiffnesses of composite of $\gamma'$- and $\gamma$-phases calculated by Voigt and Reuss models [5] are presented in the table. Two models give nearly identical values, and $A = 2.840$ and $\nu = 0.2827$. The Young's modulus $E$ is given as

$$E = \frac{1}{s_{11}} = \frac{(c_{11} - c_{12})(c_{11} + 2c_{12})}{c_{11} + c_{12}}$$

(16)

![Diffraction pattern and profile of 313 diffraction.](image)
Young's modulus is 126 GPa for $\gamma$-phase, 118 GPa for $\gamma'$-phase, and 120 GPa for the average. The measured value of the present material is 128 GPa, which is larger than the former three. Because $A$ and $\nu$ are rather insensitive to the material microstructure [6], the stiffnesses of the materials are derived using $A = 2.840$, $\nu = 0.2827$, and the measured Young's modulus. Table 4 summarizes the values of stiffnesses and compliances used for stress calculation.

<table>
<thead>
<tr>
<th>Table 3</th>
<th>Elastic constants of single crystal of $\gamma$, $\gamma'$ phase</th>
</tr>
</thead>
<tbody>
<tr>
<td>$c_{11}$ GPa</td>
<td>$c_{12}$ GPa</td>
</tr>
<tr>
<td>$\gamma$-phase</td>
<td>243</td>
</tr>
<tr>
<td>$\gamma'$-phase</td>
<td>225</td>
</tr>
<tr>
<td>Voigt model</td>
<td>230.0</td>
</tr>
<tr>
<td>Reuss model</td>
<td>229.8</td>
</tr>
<tr>
<td>Average</td>
<td>229.9</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Table 4</th>
<th>Elastic constants of single crystal of $V_{\gamma'}=0.72$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$c_{11}$ GPa</td>
<td>$c_{12}$ GPa</td>
</tr>
<tr>
<td>245.5</td>
<td>152.2</td>
</tr>
</tbody>
</table>

Fig. 6 Change of X-ray stress with applied stress for 331 diffraction. (multiple regression method)

Fig. 7 Change of X-ray stress with applied stress for 420 diffraction. (two-tilt method)
The changes of the X-ray measured stresses as a function of the applied stress is shown in Fig. 6 for the case of the multiple regression method. The diffraction angle for stress-free state determined by the regression method was $2\theta_0 = 139.054\pm 0.002^\circ$ independent of the applied stress. The relation between the stress $\sigma_{11}$ (MPa) and the applied stress $\sigma_A$ (MPa) is given by

$$\sigma_{11} = -32.1 + 0.950\sigma_A \quad (17)$$

The stresses, $\sigma_{12}$ and $\sigma_{22}$, do not change with loading. Figure 7 show the changes of the stresses with the applied stress, and the following relation is obtained.

$$\sigma_{11} = -20.9 + 0.953\sigma_A \quad (18)$$

The X-ray value of $\sigma_{11}$ is nearly equal to the applied stress for both methods and the difference is about 5%.

**DIFFRACTION PROFILES AND FWHM**

The diffraction profiles is broadened on the surface and becomes sharper at the depth of 15$\mu$m. The diffractions from $\gamma'$- and $\gamma$-phases are not separated, so they are treated as a single peak. The diffraction profile is assumed to be a pseudo-Voigt function. The peak position and the full width at half maximum (FWHM) were determined from the K$\alpha$1 diffractions separated from measured doublet. Similar results were obtained for the other diffractions. The penetration depth of 331 diffraction is 3.9 $\mu$m for $\psi = 46.51^\circ$, 1.3 $\mu$m for $\psi = 76.74^\circ$. The depth is larger for 420 diffraction and 5.3 $\mu$m for $\psi = 26.57^\circ$ and 2.6 $\mu$m for $\psi = 63.44^\circ$. The mean depth is 2.7$\mu$m for 331 diffraction and 4.0 $\mu$m for 420 diffraction. Figure 8 shows the distribution of FWHM, where each measured value is plotted at the positions of each penetration depth. The FWHM is increased to the depth of about 15 $\mu$m, and takes a small constant value below that depth. It can be concluded that the plastic zone is spread about 15 $\mu$m below the ground surface.
DISTRIBUTION OF RESIDUAL STRESS

The residual stress value measured on the ground surface by the multiple regression method was usually larger compression than that by the two-tilt method. This is because the residual compressive stress has a steep gradient below the surface and the 313 diffraction used for the multiple regression method has a shallower penetration depth. The multiple regression method gives the stress averaged over a shallower region below the surface than the two-tilt method. Figure 9 shows the distribution of the residual stress, where the each data is plotted at each penetration depth. The compressive stress close to the surface is -700 MPa and diminishes quickly to zero at the depth of about 15μm.

CONCLUSIONS

The stress in a single crystal of nickel-base superalloy with 72% volume fraction of γ'-phase was measured by the X-ray method. The specimen whose surface normal was parallel to [001] direction was oscillated around ϕ-axis during recording of the X-ray diffraction pattern with a two-dimensional position sensitive proportional counter (PSPC). The stress was determined from the measured strain using the multiple regression method and the two-tilt method. The obtained results are summarized as follows:
(1) A two-dimensional detector was successfully applied to measure the strain in single crystals only by using the oscillation of one axis during recording.
(2) The stress along [100] direction, σ_{11}, measured with the X-ray method increased proportionally to the applied uniaxial stress, and the measured stress was about 5% smaller than the applied stress. The other stress components, σ_{22} and σ_{12}, did not change with the applied stress.
(3) The plastically deformed depth below the ground surface superalloy was accompanied by the increase of the full width at half maximum. It was about 15 μm in depth.
(4) The residual stress on the ground surface was a large compression of about 700 MPa and diminished quickly to zero at the depth of about 15 μm.

REFERENCES