General paper

X-Ray Stress Measurement of Silicon Single Crystal

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Abstract: In X-ray stress measurement of a single crystal, the control of crystal oscillation is required in order to obtain a perfect diffraction profile. Accurate diffraction profiles can be measured by using the $\chi\psi$ -oscillation method proposed in this study. The elasticity applied stresses in a silicon single crystal used as a material of semiconductor devices was measured using this oscillation method. Lattice strain was obtained from the peak shift of the diffraction profiles. The stress was calculated using the lattice strain of three different diffraction planes. As a result, the measured stress agreed well with the applied stress evaluated using the strain gage. Therefore, the possibility of X-ray stress measurement of a single crystal using the $\chi\psi$ -oscillation method was confirmed. Also, the relationship between the displacement error of the specimens and the stress error was theoretically examined. It was confirmed that the effect of misalignment on X-ray stress measurement of single crystal materials is much larger than in the case of polycrystalline materials. In this study, a microscope was used in order to set the specimen.

Key words: X-ray stress measurement, Single crystal, Diffraction profile, Semiconductor device, Lattice strain

1. INTRODUCTION

Semiconductor devices are manufactured by bonding different materials. Residual stresses or thermal stresses are generated during device production or operation because of the difference in the thermal expansion coefficients of the different materials. These stresses cause dynamic problems in the material, e.g., peeling of the bonded interface, disconnection of the wiring, and generation of dislocations, resulting in deteriorated electrical characteristics. Therefore, stress evaluation is important to ensure the reliability of semiconductor devices. The dimensions of each component in the semiconductor devices are on a submicron order. Even if the wiring is polycrystalline, there is only one crystal in the cross-sectional direction of the wiring, thus the stress measurement of a single crystal is required.

For the stress measurement of crystalline materials, the X-ray stress-measurement method is applicable. This method can be used to measure the stresses in bulk materials, thus it is the optimum method for application to actual machines, such as semiconductor devices and micromachines.

During the 1940s to 1970s, several articles were devoted to the study of X-ray stress measurement of single crystals and the theoretical background was already established [1]. However, experimental analysis was difficult compared with that for the stress measurement of polycrystalline materials. As a result, there have been few reports on this subject [2-4]. For example, in the stress measurement of polycrystalline materials, Debye-sherrer (DS) diffraction ring can be measured when X-rays are emitted from any direction. However, in single-crystal materials, a diffraction spot on an imaginary DS circle can be measured when X-rays are emitted only from a specific direction. Therefore, the control of crystal oscillation is required in order to obtain an ideal diffraction profile.

It is necessary to determine the optimum experimental conditions by examining the stress-measurement accuracy in order to establish an effective stress measurement method of a single crystal. In this study, a stress test of the X-ray stress-measurement apparatus was carried out using a silicon single crystal and the factors which influenced the stress measurement accuracy were examined.

If a method for the stress measurement of a single crystal can be established, various developments can be expected, including the assurance of the reliability of semiconductor devices as well as further examination of the crack growth mechanism in a single crystal.

2. PRINCIPLE

The relationship between stress and strain of a single crystal is expressed as follows [5]. Figure 1 shows the crystal system X_i and the laboratory system L_i with respect to the specimen system P_i and the definition of ψ - and ϕ -angle. The transformation matrix between the

Fig. 1. Coordinate system.

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specimen system P_i and the laboratory system L_i can be expressed as

$$\sigma'_{ij} = \omega_{ik} \omega_{jl} \sigma_{kl} \quad , \quad (1)$$

where

$$\omega = \begin{pmatrix} \cos \phi & \sin \phi & 0 \\ -\cos \psi \sin \phi & \cos \psi \cos \phi & \sin \psi \\ \sin \psi \sin \phi & -\sin \psi \cos \phi & \cos \psi \end{pmatrix} \quad . \quad (2)$$

The normal strain ϵ'_{33} along the direction L_3 is expressed by the strain tensor of the crystal system as

$$\epsilon'_{33} = \gamma_{3i} \gamma_{3j} \epsilon^*_{ij} = \gamma_{31}^2 \epsilon^*_{11} + \gamma_{32}^2 \epsilon^*_{22} + \gamma_{33}^2 \epsilon^*_{33} + 2(\gamma_{32} \gamma_{33} \epsilon^*_{23} + \gamma_{33} \gamma_{31} \epsilon^*_{31} + \gamma_{31} \gamma_{32} \epsilon^*_{12}) \quad , \quad (3)$$

where γ_{3k} is the direction cosine of the direction L_3 for the crystal system X_i , and is expressed as the following equations when the diffraction plane is expressed as hkl .

$$\gamma_{31} = \frac{h}{\sqrt{h^2 + k^2 + l^2}}, \quad \gamma_{32} = \frac{k}{\sqrt{h^2 + k^2 + l^2}}, \quad \gamma_{33} = \frac{l}{\sqrt{h^2 + k^2 + l^2}} \quad . \quad (4)$$

The strain tensor ϵ^*_{ij} is expressed by the stress tensor σ^*_{ij} along the crystal system as

$$\begin{aligned} \epsilon^*_{11} &= (S_{11} - S_{12}) \sigma^*_{11} + S_{12} (\sigma^*_{11} + \sigma^*_{22} + \sigma^*_{33}), \\ \epsilon^*_{22} &= (S_{11} - S_{12}) \sigma^*_{22} + S_{12} (\sigma^*_{11} + \sigma^*_{22} + \sigma^*_{33}), \\ \epsilon^*_{33} &= (S_{11} - S_{12}) \sigma^*_{33} + S_{12} (\sigma^*_{11} + \sigma^*_{22} + \sigma^*_{33}), \\ 2\epsilon^*_{23} &= S_{44} \sigma^*_{23}, \quad 2\epsilon^*_{31} = S_{44} \sigma^*_{31}, \quad 2\epsilon^*_{12} = S_{44} \sigma^*_{12} \quad . \end{aligned} \quad (5)$$

Next, the stress σ^*_{ij} along the crystal system is expressed by the stress σ_{mn} along the specimen system as

$$\sigma^*_{ij} = \pi_{mi} \pi_{nj} \sigma_{mn} \quad . \quad (6)$$

If the direction P_1 is expressed as $[w_1, w_2, w_3]$ of the tensile direction and P_3 is expressed as $[n_1, n_2, n_3]$ of the normal direction of the specimen surface, then the transformation matrix π_{ij} is expressed as

$$\pi_{ij} = \begin{pmatrix} \pi_{11} & \pi_{12} & \pi_{13} \\ \pi_{21} & \pi_{22} & \pi_{23} \\ \pi_{31} & \pi_{32} & \pi_{33} \end{pmatrix} \quad , \quad (7)$$

where

$$\begin{aligned} \pi_{11} &= \frac{w_1}{\sqrt{w_1^2 + w_2^2 + w_3^2}}, \quad \pi_{12} = \frac{w_2}{\sqrt{w_1^2 + w_2^2 + w_3^2}}, \\ \pi_{13} &= \frac{w_3}{\sqrt{w_1^2 + w_2^2 + w_3^2}}, \quad \pi_{31} = \frac{n_1}{\sqrt{n_1^2 + n_2^2 + n_3^2}}, \\ \pi_{32} &= \frac{n_2}{\sqrt{n_1^2 + n_2^2 + n_3^2}}, \quad \pi_{33} = \frac{n_3}{\sqrt{n_1^2 + n_2^2 + n_3^2}}, \\ \pi_{21} &= \pi_{13} \pi_{32} - \pi_{12} \pi_{33}, \quad \pi_{22} = \pi_{11} \pi_{33} - \pi_{13} \pi_{31}, \\ \pi_{23} &= \pi_{12} \pi_{31} - \pi_{11} \pi_{32} \quad . \end{aligned}$$

From Eqs. (3) to (7), ϵ'_{33} is calculated as

$$\begin{aligned} \epsilon'_{33} &= S_0 \sum_i \sum_j \sigma_{ij} \left(\sum_k \gamma_{3k}^2 \pi_{ik} \pi_{jk} \right) + S_{12} (\sigma_{11} + \sigma_{22} + \sigma_{33}) \\ &+ \frac{1}{2} S_{44} \sum_i \sum_j \sigma_{ij} \left(\sum_k \gamma_{3k} \pi_{ik} \right) \left(\sum_k \gamma_{3k} \pi_{jk} \right) \quad , \end{aligned} \quad (8)$$

where

$$S_0 = S_{11} - S_{12} - \frac{1}{2} S_{44} \quad .$$

On the other hand, the next transformation can be made.

$$\begin{aligned} &\frac{1}{2} S_{44} \sum_i \sum_j \sigma_{ij} \left(\sum_k \gamma_{3k} \pi_{ik} \right) \left(\sum_k \gamma_{3k} \pi_{jk} \right) \\ &= \frac{1}{2} S_{44} \sum_i \sum_j \sigma_{ij} \omega_{3i} \omega_{3j} \quad . \end{aligned} \quad (9)$$

Therefore,

$$\begin{aligned} \epsilon'_{33} &= S_0 \sum_i \sum_j \sigma_{ij} \left(\sum_k \gamma_{3k}^2 \pi_{ik} \pi_{jk} \right) + S_{12} (\sigma_{11} + \sigma_{22} + \sigma_{33}) \\ &+ \frac{1}{2} S_{44} \sigma_{33} \\ &+ \frac{1}{2} S_{44} (\sigma_{11} \sin^2 \phi - \sigma_{12} \sin 2\phi + \sigma_{22} \cos^2 \phi - \sigma_{33}) \sin^2 \psi \\ &+ \frac{1}{2} S_{44} (\sigma_{31} \sin \phi - \sigma_{23} \cos \phi) \sin 2\psi \quad . \end{aligned} \quad (10)$$

If the plane stress condition is assumed, then

$$\sigma_{33} = \sigma_{23} = \sigma_{31} = 0 \quad . \quad (11)$$

Equation (10) is therefore expressed as

$$\begin{aligned} \varepsilon'_{33} &= S_0 \sum_i \sum_j \sigma_{ij} \left(\sum_k \gamma_{3k}^2 \pi_{ik} \pi_{jk} \right) + S_{12} (\sigma_{11} + \sigma_{22}) \\ &+ \frac{1}{2} S_{44} (\sigma_{11} \sin^2 \phi - \sigma_{12} \sin 2\phi + \sigma_{22} \cos^2 \phi) \sin^2 \psi \\ &= S_0 \left[(\gamma_{31}^2 \pi_{11}^2 + \gamma_{32}^2 \pi_{12}^2 + \gamma_{33}^2 \pi_{13}^2) \sigma_{11} \right. \\ &+ 2(\gamma_{31}^2 \pi_{11} \pi_{21} + \gamma_{32}^2 \pi_{12} \pi_{22} + \gamma_{33}^2 \pi_{13} \pi_{23}) \sigma_{12} \\ &+ (\gamma_{31}^2 \pi_{21}^2 + \gamma_{32}^2 \pi_{22}^2 + \gamma_{33}^2 \pi_{23}^2) \sigma_{22} \left. \right] + S_{12} (\sigma_{11} + \sigma_{22}) \\ &+ \frac{1}{2} S_{44} (\sigma_{11} \sin^2 \phi - \sigma_{12} \sin 2\phi + \sigma_{22} \cos^2 \phi) \sin^2 \psi. \end{aligned} \quad (12)$$

For the stress measurement of a single crystal specimen, it is necessary to measure the strain of the diffraction plane distance expressed by at least three independent combinations of ϕ and ψ . Three stresses σ_{11} , σ_{22} , σ_{12} are obtained by solving the simultaneous equations given by Eq. (12).

ϕ and ψ are calculated as follows.

$$\tan \phi = -\frac{q_1}{q_2}, \quad \sin^2 \psi = q_1^2 + q_2^2, \quad (13)$$

$$q_1 = P_1 L_3, \quad q_2 = P_2 L_3, \quad (14)$$

$$\begin{aligned} P_1 &= [\pi_{11} \pi_{12} \pi_{13}], \quad P_2 = [\pi_{21} \pi_{22} \pi_{23}], \\ L_1 &= [\gamma_{31} \gamma_{32} \gamma_{33}]. \end{aligned} \quad (15)$$

The strain ε'_{33} of the diffraction plane distance is calculated by measuring the diffraction angle 2θ as follows

$$\varepsilon'_{33} = -\frac{(\theta - \theta_0)}{\tan \theta_0}. \quad (16)$$

If the diffraction angle of the unstressed condition is considered as $2\theta_0$, the measured stress expresses the absolute value, and if the diffraction angle in the initial state is measured and this angle is assumed as $2\theta_0$, the measured stress is the relative value for the initial state.

3. EXPERIMENTAL PROCEDURE

3.1. Specimens and Loading Method

The sample used in this study was a silicon wafer for which the plane orientation was (001) and the orientation in the flat direction was [110]. Specimens of loading directions [110] and [100], with 40 mm length and 5 mm width, were cut from the wafer 0.45 mm thick and were called the (001)[110]-specimen and the (001)[100]-specimen, respectively. A four-point bending device which can be installed on the crystal oscillating device was used to apply strain. The inside and outside spans of the load pin were 10 mm and 30 mm, respectively. The prestress of

Table 1. Stiffness matrices of silicon [6].

C_{11} , GPa	167.4
C_{12} , GPa	65.2
C_{44} , GPa	79.6

Table 2. Oscillation condition and X-ray measurement condition.

Characteristic X-ray	Co-K α_1
χ -oscillation range, deg	± 10 (Continuous)
ψ -oscillation range, deg	± 2 (0.1 deg step)
Tube voltage, kV	30
Tube current, mA	8
Pinhole, mm	0.8

Table 3. Experimental conditions of diffraction angle measurement.

(a) (001)[110]-specimen.

Diffraction plane	135	$\bar{1}\bar{3}5$	404
Diffraction angle, deg	153.99	153.99	137.41
ϕ -angle, deg	116.57	-26.57	45
ψ -angle, deg	32.31	32.31	45
Applied stress, MPa	27, 54, 80		

(b) (001)[100]-specimen.

Diffraction plane	315	$3\bar{1}5$	$\bar{1}\bar{3}5$
Diffraction angle, deg	153.99	153.99	153.99
ϕ -angle, deg	108.43	71.57	-18.43
ψ -angle, deg	32.31	32.31	32.31
Applied stress, MPa	14, 27, 41		

the (001)[110]-specimen and the (001)[100]-specimen was set at about 36 MPa and 41 MPa, respectively, in order to hold the specimen, and this condition was considered as the initial state. From this initial state, the stresses in three steps, as referred in Table 3, were taken as the relative stresses. In each step, the diffraction angles of three different planes were measured using X-rays, and the stresses were calculated from the peak shift. The mechanical stress was calculated using the output from the strain gage fixed onto the back of the specimen; the stiffness matrices of silicon are listed in Table 1 [6].

3.2. X-ray Stress-measurement Procedure

First, the combinations of the diffraction planes and the characteristic X-rays are determined. Next, the incident angles of the X-rays, ψ and ϕ , are calculated from the plane index and the loading direction of the specimen using Eq. (13). The specimen is set on the basis of ψ and ϕ , and the diffraction profile is measured while the specimen is oscillated. For the generation of X-rays and the detection of the diffraction X-rays, PSPC-MSF [Rigaku] was utilized. The position-sensitive proportional counter (PSPC) with a detection angle of ± 14 degrees and 2048 channels was utilized. The X-ray measurement

conditions, as well as the combinations of the diffraction planes, the diffraction angles, the X-ray Incident angles and the load condition for this X-ray stress measurement, are listed in Tables 2 and 3, respectively.

3.3. Oscillation Method

In the case of detecting the diffracted X-rays using the PSPC, even if X-rays are emitted to the specimens from the calculated incident angles, ψ and ϕ , the diffracted X-rays may not be adequately detected. As some of the causes for this, the following are considered: the lattice strain is unknown, the slippage of the specimen orientation, the turbulence of the atomic arrangement, and the difference between the actual lattice constant and that obtained from the reference. Therefore, in order to obtain an ideal diffraction profile, crystal oscillation around the angle at which the specimen is set is required. As Fig. 2 indicates, the biaxial oscillation method, called the " $\chi\psi$ -oscillation method" has been used in this study. The axis of χ -oscillation coincides with the direction of incident X-rays, and the axis of ψ -oscillation coincides with the vertical direction of the χ -rotating axis.

Figure 3 shows the crystal-oscillation apparatus and the X-ray stress-measurement apparatus used in this study. The automatic goniometer stage (ψ -stage), which can be changed by angle ψ within ± 35 deg, was mounted on an automatic rotating stage (χ -stage) which can rotate around the incident X-rays. The Z-stage for the height adjuster and the ϕ -rotating stage (ϕ -stage) for changing the orientation of the specimen are mounted on the ψ - and χ -stages. In addition, a four-point bending device is included. This crystal-oscillation apparatus is set on the XY-stage such that the oscillation center coincides with the incident X-rays. The oscillation condition was ± 2 deg in 0.1 deg steps around the ψ -axis, and continuous oscillation of ± 10 deg around the χ -axis for ψ -oscillation of 0.1 deg. As Fig. 4 indicates, ψ -oscillation is performed in order to satisfy the optimum diffraction condition, and χ -oscillation is performed such that the diffraction lines pass through the PSPC. The formation process of the diffraction profile is described below. Immediately after the oscillation starts, no diffraction line is detected. However, the diffraction lines are gradually detected as the ψ -angle increases (process ① indicated in Fig. 4), and after reaching the highest intensity (process ②), their intensity gradually decreases (process ③). Lastly, the diffraction lines become undetectable. Similarly, immediately after the oscillation in the χ -direction starts, no diffraction lines are detected. However, when the diffraction lines pass through the PSPC, the diffraction lines of maximum intensity are detected. Then, they become undetectable again. Therefore, it is necessary to ensure that the diffraction lines are not detected at either the beginning or the end of the measurement in order to confirm that the oscillation range is sufficient. In this study, it is possible to confirm whether the oscillation range is sufficient during measurement because of using the PSPC. By following the above procedure, the diffraction lines can be accurately detected, and high reproducibility of the measured diffraction profiles can be

Fig. 2. Oscillation system.

Fig. 3. Overview of the stress analyzer and the crystal-oscillating device developed in this study.

Fig. 4. Schematic illustration of the measurement process.

realized.

Each rotating stage was combined so that the tip of the thin pin set on the ϕ rotating stage would not move during the oscillation. The oscillating center was made to coincide with the measurement point using a microscope in the horizontal direction, whose crossmark coincides with the oscillation center. By following the above method, the setting accuracy of the specimen was within approximately 10 μm .

4. EFFECT OF SETTING ERROR OF SPECIMEN ON X-RAY STRESS MEASUREMENT

4.1. Peak Shift of Diffraction Profile

In X-ray stress measurement, the displacement error affects the stress-measurement value. Thus, the relationship between the displacement error and the stress error was theoretically investigated. On the size of the stress error for the displacement error of the specimen, the stress measurement of a single crystal was compared with the stress measurement of polycrystalline materials using the iso-inclination method (the Ω -diffractometer method).

The relationship between the displacement error of a specimen and the stress error in the stress measurement employing iso-inclination with the PSPC is described. The influence of incorrectly setting the specimen in X-ray stress measurement by the side-inclination method (the ψ -diffractometer method) was clarified by Goto [7], and the expression in the case of the iso-inclination method is described below. As Fig. 5(a) indicates, when a displacement error occurs between a stress measurement region and the oscillation center, the diffraction angle detected in the PSPC can be given by

$$2\theta_1 = 2\theta_0 + \frac{L \sin 2\theta_0}{\Delta\alpha \sin(\theta_0 + \psi)} + \left\{ R_0 + \frac{L \cos 2\theta_0}{\sin(\theta_0 + \psi)} \right\} \frac{\tan\left(\frac{\sigma}{K} \sin^2 \psi\right)}{\Delta\alpha} \quad (17)$$

(L : displacement error; R_0 : distance from oscillation center to PSPC; K : X-ray stress constant; $2\theta_0$: diffraction angle under unstressed condition; σ : actual stress; and $2\theta_1$: detected diffraction angle). $\Delta\alpha$ indicates the length per deg of the PSPC and is given as

$$\Delta\alpha = \frac{R_0 \tan \alpha}{\alpha}, \quad (18)$$

where 2α is the range of the detection angle of the PSPC.

Therefore, the stress error can be evaluated by comparing the stress calculated from the $2\theta_1$ - $\sin^2 \psi$ chart with the stress σ .

The relationship between the displacement error of a

specimen and the stress error, in the case of the X-ray stress measurement of a single crystal using a PSPC, was investigated. As Fig. 5(b) indicates, when displacement error occurs between a stress-measurement region and the oscillation center, the diffraction angle detected in the PSPC can be expressed as

$$2\theta_1 = 2\theta_0 + \frac{L' \sin(180 - 2\theta_0)}{\Delta\alpha} - \frac{(R_0 - L' \cos 2\eta) \tan(2\theta_0 - 2\theta')}{\Delta\alpha}, \quad (19)$$

where $2\theta'$ is the diffraction angle when the stress is σ , and $2\eta = 180 - 2\theta_0$. L' is given as

$$L' = \frac{L}{\cos(\psi - \eta)}, \quad (20)$$

where ψ is the angle between P_3 and L_3 in Fig. 1, and $\Delta\alpha$ is similar to that given by Eq. (18).

(a) The case of polycrystalline materials.

(b) The case of single-crystalline materials.

Fig. 5. X-ray path to the PSPC in the case of the displacement error of the specimen.

Therefore, the stress calculated from Eqs. (12) and (16) using the relationship between $2\theta_0$ and $2\theta_1$ can be compared with the actual applied stress σ .

4.2. Relation between Displacement Error and Stress Error

The (001)[110]-specimen was assumed to be composed of single-crystalline material and the combinations of the measured diffraction planes were of three types: (1) 135, $\bar{1}\bar{3}\bar{5}$, 404, (2) 135, 315, $3\bar{1}\bar{5}$, and (3) 135, 224, $\bar{2}\bar{2}\bar{4}$. For polycrystalline materials, the diffraction angle and the stress constant were assumed to be 154 deg and $-200 \sim -500$ MPa/deg, respectively. The effect of the displacement error on the measured stress values was theoretically investigated. The parameters for the stress error calculation are listed in Table 4.

The relationship between the stress error at the displacement error is shown in Fig. 6. In the case of the stress measurement of polycrystalline materials, the amount of change in the stress error was approximately 0.03 %/ μm . Even when a displacement error of 100 μm occurred, the stress error was about 3 %. In the stress measurement of single-crystalline materials, the amount of change in the stress error is approximately 0.3~2.7 %/ μm . Therefore, it was confirmed that the stress error of single-crystalline materials is 10 to 90 times that of polycrystalline materials. The difference in the stress error resulting from the different combinations of the diffraction planes is dependent on the diffraction angle. In combination (2), the stress error is smaller than that in the other combinations, because the diffraction angle of all diffraction planes is a large angle of approximately 154 deg in which the highly precise measurement of strain is possible. In combination (3), which includes 224 and $\bar{2}\bar{2}\bar{4}$, in which the diffraction angle is 121.44 deg, the stress error is the largest. Combination (1) is similar to the combination considered in this study and the amount of change of the stress error is 0.94 %/ μm ; the setting error must be below approximately 10 μm in order for the stress error to be within 10 %. Therefore, it is necessary to improve the specimen setting accuracy for the center of the optical system and to select the diffraction planes which have a high diffraction angle.

5. RESULTS AND DISCUSSION

5.1. Diffraction Profile

The diffraction profiles of the 135 and 404 diffraction of the (001)[110]-specimen under the applied stress of 37 MPa are shown in Figs. 7(a) and (b), respectively. The diffraction profile of the 135 diffraction was separated into two peaks with approximately 1 deg difference. This was equal to the difference between the diffraction angles of $\text{CoK}\alpha_1$ and $\text{CoK}\alpha_2$. The intensity ratio of both diffraction profiles was 2:1, and this was equal to the intensity ratio of $\text{K}\alpha_1$ to $\text{K}\alpha_2$. Therefore, it is confirmed that an ideal diffraction profile can be obtained using the $\chi\psi$ -oscillation method.

In the diffraction profile of the 404 diffraction, the angular resolution decreased because this diffraction

Table 4. Parameters for the calculation of the stress error.

R_0 , mm		200
α , deg		14
σ , MPa		50
$2\theta_0$, deg	531 family	154
	422 family	121.66
	404	137.41

Fig. 6. Relationship between the stress error and setting error.

Fig. 7. Diffraction profiles of the (001)[110]-specimen.

Fig. 8. Comparison of measured and theoretical diffraction angles of three different planes for each applied stress of (001)[110]-specimen.

angle is low, thus the profile of $K\alpha_1$ overlapped with that of $K\alpha_2$.

As the method for determining the diffraction angle, the half-value breadth method of $K\alpha_1$ was utilized for the 531 family and the two-thirds value breadth method of $K\alpha_1$ was utilized for the 404 diffraction.

5.2. Results of Stress Measurement

In each diffraction plane, the diffraction angle measurement was carried out three times for each load. The stress calculation was carried out by substituting the mean value of the three measurements in Eqs. (16) and (12).

The result of comparing the measured diffraction angle with the theoretical diffraction angle in each diffraction plane is shown in Fig. 8. The plotted point indicates the mean value of three measurements and the error band indicates the dispersion of the three measurements. The peak shift for each of the applied stresses in each diffraction plane agreed closely with the theoretical value. The range of the dispersion of the 531 family and the 404 diffraction plane was 0.001~0.005 deg and 0.005~0.008 deg, respectively.

Young's moduli of the [110]-direction and the [100]-

(a) (001)[110]-specimen.

(b) (001)[100]-specimen.

Fig. 9. Results of X-ray stress measurement.

direction, calculated using the stiffness matrices listed in Table 1, were 170 GPa and 130 GPa, respectively. σ_{11} and σ_{22} agree with principal stresses, and σ_{12} is the shear stress which acts in (110) and (100). Theoretically, σ_{11} agrees with the applied stress, and σ_{12} and σ_{22} are equal to 0 MPa. The relationship between the measured X-ray stresses and the applied stresses in the (001)[110]- and (001)[100]-specimens are shown in Figs. 9(a) and (b), respectively. The X- and Y-axes in these figures indicate the relative amount of change from the initial state.

In the (001)[110]-specimen, as Fig. 9(a) indicates, σ_{11} increased in close proportion to the applied stress. The measured stress σ_{11} was lower than the applied stress by approximately 9 to 12 MPa. Therefore, the largest measured stress error was 12 MPa. However, when a linear approximation is performed on the measured stress value from the first step to the third step, the amount of change was about 51 MPa, therefore, the measured stress agreed with the applied stress of approximately 53 MPa with approximately 2 MPa error. σ_{12} was 0 MPa, thus this

agreed with the theoretical value. However, σ_{22} did not agree with the theoretical value, and increased with increasing applied stress. As some of the causes of this, the following are considered: the effect of cylindric bending, and the fact that the bending deformation was restricted by the pin of the four-point bending device.

In the (001)[100]-specimen, as Fig. 9(b) indicates, σ_{11} was increased in close proportion to the applied stress. The measured stress σ_{11} was lower than the applied stress by approximately 8 to 11 MPa. Therefore, the largest measured stress error was 11 MPa. However, when a linear approximation was performed on the measured stress value from the first step to the third step, the amount of change was about 25 MPa, thus the measured stress agreed with the applied stress of about 27 MPa with about 2 MPa error. σ_{22} and σ_{12} were included in the narrow band which ranged from -6 to 0 MPa. σ_{22} agreed closely with the theoretical value and did not increase, unlike that of the (001)[110]-specimen.

As the results of stress measurement indicated, the measured stresses were generally lower than the applied stresses. As the reason for this, it was considered that the amount of change in the measured stress was small from the initial state to the first step. This behavior was similar to that of the diffraction angle of the 135 diffraction shown in Fig. 8, therefore, this diffraction plane may have a large effect on the measured stress. It is necessary to investigate the stress in a small strain region, namely, the behavior of the lattice strain, in the future.

6. CONCLUSIONS

In this study, the X-ray stress measurement apparatus for performing the $\chi\psi$ -oscillation method was experimentally produced, and a stress test of this apparatus was carried out. On the basis of the results, the following conclusions were obtained.

(1) The measured stress agreed closely with the theoretical stress. Therefore, X-ray stress measurement of a single crystal using the $\chi\psi$ -oscillation method is possible.

(2) In the case of the stress measurement of single crystals, the setting of the sample for the optimum X-ray system must be precisely calibrated, unlike that in the stress measurement of polycrystalline materials. For example, under the measurement conditions of the (001)[110]-specimen used in this study, if the stress error is assumed to be less than 10 %, the precision of the setting of the sample must be less than approximately 10 μm .

(3) In the case of the stress measurement of single-crystalline materials, if the plane stress condition is assumed, the diffraction angles of three diffraction planes must be measured. Then, if the diffraction angle of all combined diffraction planes is at the higher angle side, the precision of the stress measurement is improved.

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