INTRODUCTION

Ni₃Al has been investigated by many researchers as a heat resisting material for the future generation. The investigation of the grain boundary brittleness of polycrystalline Ni₃Al is one of these studies. To prevent intergranular fracture, a unidirectional solidification process or a floating zone method[1] is applied. However, an application of X-ray stress measurement for such materials is very difficult because they consist of coarse grains and have a preferred orientation. Therefore, if we can obtain good information about stress by using X-ray diffraction, it would be very useful for evaluating the mechanical behavior of such Ni₃Al materials.

In this investigation, we try to obtain stress in Ni₃Al consisting of coarse grains and preferred orientation, by using two X-ray diffraction techniques. An X-Y plane oscillation method which is a mechanical oscillation applied for coarse grains, and a method which utilizes diffraction lines belonging to one zone axis which is applied for the preferred orientation. Moreover, a 2θ as high as 167° was chosen for the diffraction angle. In a usual X-ray measurement which uses a goniometer, such a high angle 2θ cannot be used, however, an Imaging Plate (IP)[2,3] makes this possible. The X-ray stress measurement at high angle 2θ is highly precise. At first we obtained an understanding of the state of preferred orientation of the specimen, which consisted of coarse grains, by measuring the pole figure. Secondly, we obtained X-ray elastic constants and the stress constant with the present method, and examined the validity of the X-ray elastic constants by comparing with elastic constants which were calculated from the elastic compliances of a Ni₃Al single crystal.

EXPERIMENTAL PROCEDURE

Material and Test Specimen

Ni₃Al bar ingots were prepared by an argon arc-melting method from Ni and Al lumps with a purity of 99.99 mass% respectively. The specimen for X-ray stress measurements was cut from
Bottan side of solidification

Figure 1 Dimensions of test specimens, sampling position of test specimen for X-ray stress measurement.

the ingot using a wire electrodischage machine. The sampling position of the specimen in the ingot and the dimensions of the specimen are shown in Fig. 1. The surface of the specimen was finished by buffing after emery-polishing.

**X-Ray Observation**

A Debye-Scherrer ring was recorded on the IP using a Rigaku MSF X-ray stress analyzer, fitted with a Debye-Scherrer camera. The detailed conditions of X-ray observation are given in Table I. The coordinate system and the symbols used in the X-ray stress measurement are defined in Fig.2.

The strain $\varepsilon_{\text{app}}$ was also applied by a four point bending jig, and the strain was read by a strain gage pasted on the specimen. The $\varepsilon_{\text{app}}$ was established at three levels: 0, 200 and 400×10^{-6}.

An X-Y plane oscillation was done by using an X-Y stage. We put a four point bending jig on the stage. The X-Y stage has stepping-motors on the X axis and Y axis respectively, and the amount of the movement of the X axis and Y axis can be controlled by a personal computer. The X-ray irradiation point moves as shown by the arrow drawn in Fig.2 for the specimen. In the measurement, the specimen was covered with vinyl tape except for the inner span. The X-ray irradiation area is 8×22mm.

**Measurement of the Mechanical Elastic Constant**

The mechanical Young's modulus of Ni$_3$Al was measured by a tension test. The tension specimen was JIS No.5 B (gage dimensions: 16×4×2mm). The specimen was cut from

<table>
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<tr>
<th>Table I X-ray diffraction condition.</th>
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<tr>
<td>Equipment</td>
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<tr>
<td>Tube voltage</td>
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<tr>
<td>Tube current</td>
</tr>
<tr>
<td>Exposure time</td>
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<tr>
<td>Camera length</td>
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<tr>
<td>Collimator</td>
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<tr>
<td>Diffraction plane</td>
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<td>Characteristic X-ray</td>
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<tr>
<td>Filter</td>
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<tr>
<td>Diffraction angle</td>
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<tr>
<td>$\eta_{(180-20)}/2$</td>
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<tr>
<td>$\Psi_{0(\psi)}$ low</td>
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<tr>
<td>$\Psi_{0(\psi)}$ high</td>
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the same part that the specimen for X-ray stress measurement was cut from with the tensile axis coinciding with that of the specimen for X-ray stress measurement. The speed of tensile testing was 0.5 mm/min, and the strain was detected by strain gages.

As a result, the Young's modulus $E_M$ was found to be 197 GPa. Moreover, Poisson's ratio $v_M$ which was measured by a bending test using the X-ray specimens was found to be 0.353.

RESULTS AND DISCUSSION

Structural Features and State of Preferred Orientation in Specimen

Figure 3 shows the macrophotographs of the ingot. The Ni$_3$Al ingot consists of columnar crystals which have a diameter of 300-500 μm. The columnar crystals grew in the direction of solidification which is almost perpendicular to the measurement plane of the specimen.

Figure 4 shows the X-ray diffraction intensity distribution of Ni$_3$Al 220 in the direction of $\psi$ obtained from a specimen using a goniometer. We found that this Ni$_3$Al has very strong preferred orientation and $\{110\}$ planes are parallel to the measurement plane of the specimen because a very strong diffraction line appears near $\psi=0^\circ$. We also notice that two strong diffraction lines appear at intervals of 60 degrees. We consider this as follows. Figure 5 is a schematic illustration.

Figure 2 Coordinate system and symbols used in X-ray stress measurement.

(a) Longitudinal direction.  (b) Transverse direction.

Figure 3 Macrophotographs of Ni$_3$Al ingot made by an arc-melting method.
which shows positions of \(<110>\) poles of a Ni$_3$Al single crystal. The \((110)\) plane has freedom of rotation around the \([110]\) axis. If we consider an ideal intensity distribution of 220 diffraction in the direction of \(\psi\), we can understand that these diffraction lines which appear at intervals of 60 degrees belong to one zone axis.

It is very difficult to analyze stresses correctly for such a material in which diffraction intensity distribution depend on \(\psi\), by an ordinary \(\sin^2 \psi\) method[4]. However, according to Yoshioka et al [5], if we use the method utilizing the diffraction lines belonging to one zone axis, we can calculate the stress correctly from the slope of the \(2\theta - \sin^2 \psi\) diagram.

**X-ray Elastic Constant and Stress Constant**

Figure 6 shows a pole figure of Ni$_3$Al 220 diffraction obtained from the specimen by the Schultz reflection method. We found poles (dots with center (a)) and 60 degrees away (b) from the center. The pole figure did not have an ideal distribution such as a pole figure obtained from a thin film with fiber texture, because of the coarse grain[6]. Most of the specimens actually did not have two strong diffraction lines which exist at intervals of 60 degrees in the direction of \(\psi\).

![Intensity distribution of Ni$_3$Al 220 diffraction in the direction of \(\psi\).](image)

**Figure 4** Intensity distribution of Ni$_3$Al 220 diffraction in the direction of \(\psi\).

![Schematic illustration of positions of \(<110>\) pole and ideal intensity distribution of Ni$_3$Al 220 diffraction in the direction of \(\psi\).](image)

**Figure 5** Schematic illustration of positions of \(<110>\) pole and ideal intensity distribution of Ni$_3$Al 220 diffraction in the direction of \(\psi\).
Figure 6 Pole figure of 220 diffraction plane of the specimen.

Figure 7 shows Debye-Scherrer rings for $\psi_0=0^\circ$ obtained by IP. In figure, (b) X-Y plane oscillation was given and in (a) it was not given. Figure 8 shows also the diffraction profiles in the direction of $+\eta(\alpha=180^\circ)$ of the Debye-Scherrer rings in Figure 7.

When the X-Y plane oscillation was not applied, the Debye-Scherrer ring was spotty in spite of the concentration of the 220 reflection in the direction of $\psi_0$. The shape of the peak profile is not clear either. However, when the X-Y plane oscillation was applied, the diffraction became evident around the ring. And the shape of the diffraction profile has sharpened, too.

Figure 9 also shows the change of the Debye-Scherrer ring with $\psi_0$. We found that it changes according to the distribution of $\{110\}$ poles in the direction of $\psi_0$. When we looked at the intensity distribution of the diffraction profile in the direction of $+\eta$ for these Debye-Scherrer rings, we found that $\psi_0=-6^\circ$ and $\psi_0=54^\circ$ had strong intensity. Therefore, it was expected that there were diffraction lines from diffraction planes which belong to one zone axis in the vicinity.
(a) No X Y plane oscillation.  
(b) X Y plane oscillation.  

**Figure 8** Diffraction profile in the direction of $+ \eta$ of Debye-Scherrer ring in Fig.7.

<table>
<thead>
<tr>
<th>$\psi_0 = -6^\circ$</th>
<th>$\psi_0 = 10^\circ$</th>
<th>$\psi_0 = 20^\circ$</th>
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<tbody>
<tr>
<td>$\psi_0 = 30^\circ$</td>
<td>$\psi_0 = 40^\circ$</td>
<td>$\psi_0 = 54^\circ$</td>
</tr>
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</table>

**Figure 9** Changes in Debye-Scherrer ring with $\psi_0$.

Figure 10 shows the relation between intensity of the peak profile in the direction of $+ \eta$ of the Debye-Scherrer ring and $\psi$, and the relation between $2\theta$ and $\psi$. The $\psi$ of the horizontal axis is obtained by adding $\eta$ to $\psi_0$. The relation intensity and $\psi$ become straight line upper right for the reasons of an optics system[5].

Figure 11 shows $2\theta - \sin^2 \psi$ diagrams for applied strain $\varepsilon_{\text{app}} = 0$, 200 and $400 \times 10^{-6}$. The $2\theta$ corresponds to $\psi$ where the strongest intensity was obtained in Fig.10. Figure 12 also shows the relation between slope M of the $2\theta - \sin^2 \psi$ diagrams and applied stress $\sigma_{\text{app}}$, and the relation between the intercept $2\theta_0$ and $\varepsilon_{\text{app}}$. The $\sigma_{\text{app}}$ was obtained by multiplying $\varepsilon_{\text{app}}$ and $E_M$. 

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Figure 10 Changes in diffraction intensity distribution and of $2\theta$ with $\psi$.

Figure 11 $2\theta - \sin^2 \psi$ diagram.

Figure 12 Changes in slope and intercept of $2\theta - \sin^2 \psi$ diagrams with applied stress.
Table II  X-ray elastic constants and stress constant of Ni₃Al.

<table>
<thead>
<tr>
<th>X-ray elastic constant</th>
<th>Stress constant</th>
<th>Young's modulus</th>
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<tbody>
<tr>
<td>Ex/1+νX(Gpa)</td>
<td>Ex (GPa)</td>
<td>K (MPa/deg)</td>
</tr>
<tr>
<td>151</td>
<td>209</td>
<td>-157</td>
</tr>
<tr>
<td>197</td>
<td></td>
<td>197</td>
</tr>
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</table>

The X-ray elastic constant Ex and stress constant K were calculated from the inclination of two straight lines of Fig.12[4], and are shown in Table II.

We consider a model of polycrystalline Ni₃Al in which {110} planes turn as in Fig.13. The coordinates system in this figure corresponds to the coordinate system shown in Fig.2. An elastic constant E in the direction of the X-axis shown in the figure can be calculated from the elastic compliance of a Ni₃Al single crystal by using equations (1) and (2)[7]. The Reuss model is included in the calculation process for equation (1). When the elastic compliances S₁₁= 8.04, S₄₄= 7.58 and S₁₂= -3.07(TPa)¹ of Ni₃Al single crystal [8] were substituted in these equations, E was calculated to be 227GPa. The X-ray elastic constant Ex was about 10% smaller than this value. In this model, all grains in the specimen are considered in the calculation. However, the stress measured by X-rays is the stress in grains which contribute to the diffraction. We think that the measurement accuracy for the above-mentioned reason is included in an error of about 10%. Though we must examine this in the future, we think that we obtained an almost satisfactory value.

\[
\frac{1}{E} = \frac{9}{16} S₀ + S₁₂ + \frac{1}{2} S₄₄
\]  

\[
\sigma = \frac{1}{S₀} E (\varepsilon - \varepsilon₀)
\]  

Figure 13 Model of polycrystalline Ni₃Al in which {110} planes turn.

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CONCLUSIONS

An X-ray stress measurement method using X-Y plane oscillation and a method to utilize diffraction lines belonging to one zone axis was applied to Ni$_3$Al consisting of coarse grains with preferred orientation.

The results are as follows.

(1) X-ray elastic constant $E_x$ was found to be 209 GPa and nearly equal to the elastic constant $E=227$ GPa calculated from the elastic compliances of a Ni$_3$Al single crystal.

(2) From (1), we can obtain almost accurate stress values for Ni$_3$Al consisting of coarse grains with preferred orientation by the present X-ray stress measurement method.

REFERENCES