Measurement of X-Ray Elastic Constants of Ni$_3$Al using an Imaging Plate

Tokimasa Goto
Graduate student, Kanazawa University, Kakuma-machi
Kanazawa, 920-1192 Japan

and

Toshihiko Sasaki and Yukio Hirose
Department of Materials Science and Engineering, Kanazawa University
Kakuma-machi, Kanazawa, 920-1192 Japan

Abstract

X-ray stress measurement was used to examine an arc-melted Ni$_3$Al intermetallic compound, which consists of coarse grains and exhibits strongly preferred orientation. An imaging plate (IP) was used as an X-ray detector, and continuous Debye-Scherrer diffraction rings were obtained from such coarse grained material by applying an X-Y plane oscillation method. Mechanical bending stress was applied to the specimen to measure X-ray elastic constants and a stress constant. An ideal orientation method was adopted to obtain these parameters. It uses the diffraction lines which belong to the same zone axis. The experimental elastic constant was compared with the theoretical values calculated from the elastic compliance of Ni$_3$Al single crystal.

The results obtained are summarized as follows;

(1) The X-ray elastic constants measured from Ni$_3$Al 220 diffraction peaks which appeared at $\psi=0.537^\circ$ and $60.537^\circ$ were $E_X/(1+\nu_X)=176$GPa, $E_X=220$GPa and $\nu_X=0.253$.

(2) The theoretical elastic constant $E_0$ at $\psi=0^\circ$ was calculated to be 207GPa from the elastic compliances of Ni$_3$Al single crystal. This value corresponds to the elastic constant in the direction of the tensile axis for the grains which contribute to diffraction. The elastic constant $E_{60}$ at $\psi=60^\circ$ was similarly obtained as 228GPa. The experimental value of the X-ray elastic constant $E_X$ (220GPa) was intermediate between these theoretical values.

Introduction

The intermetallic compound Ni$_3$Al is of interest as a new high temperature structural material. However, it lacks sufficient ductility and toughness for practical use. Room temperature brittleness is an important problem, and techniques such as adding metallic elements and unidirectional solidification process have been tried to improve performance. Unidirectional solidification is a method to improve room temperature brittleness by control of structure, and it does not influence other characteristics as the method adding metallic elements does. However, the conventional $\sin^2\psi$ method is not applicable, because Ni$_3$Al made by unidirectional solidification consists of coarse grains and exhibits strongly preferred orientation. In the present study, the X-ray stress measurement was used to examine an arc-melted Ni$_3$Al intermetallic compound, which consists of coarse grains and exhibits strongly preferred orientation. X-ray elastic constants and a stress constant were obtained by applying the X-Y plane oscillation method and the ideal orientation method. The experimental elastic constant was compared with the theoretical values calculated from the elastic compliance of Ni$_3$Al single crystal.
This document was presented at the Denver X-ray Conference (DXC) on Applications of X-ray Analysis.

Sponsored by the International Centre for Diffraction Data (ICDD).

This document is provided by ICDD in cooperation with the authors and presenters of the DXC for the express purpose of educating the scientific community.

All copyrights for the document are retained by ICDD.

Usage is restricted for the purposes of education and scientific research.

DXC Website – www.dxcicdd.com  ICDD Website - www.icdd.com
crystal. From the good agreement of the experimental value and the theoretical values, we conclude that this X-ray stress measurement method is effectively applicable to Ni₃Al which consists of coarse grains and exhibits strongly preferred orientation.

Experimental procedure

Material and test specimen

Ni₃Al bar ingots were prepared by an argon arc-melting method from Ni and Al lumps with purity of 99.99mass%. The specimen used for the X-ray stress measurement was cut from the ingot by a wire electrodischage machine. The specimen shape was 8mmx4mmx60mm. The long direction of the specimen corresponds to the long direction of the ingot. The measured plane (XY plane in Fig.1) of the specimen was parallel to the solidification bottom of the ingot. The surface of the specimen was finished up by electrolytic polishing.

X-ray observation

The detailed conditions of the X-ray stress measurement are given in Table I. Debye-Scherrer(D-S) diffraction rings were recorded on an imaging plate (IP) [1~4] by using an X-ray device fitted up with a D-S camera. The coordinate system and symbols used in the X-ray stress measurement are illustrated in Fig.1. In the X-ray stress measurement, mechanical bending strain of five steps (ε_{app}= 0, 100, 200, 300 and 400×10^{-6} ) was applied to the specimen by using a four point bending jig. The applied strain was measured by a strain gage pasted on the specimen.

Fig. 1. Schematic illustration of the X-Y plane oscillation, and the coordinate system and symbols used in the X-ray stress measurement.
The X-Y plane oscillation method

The X-Y plane oscillation method increases the number of grains which contribute to diffraction in the X-ray stress measurement for coarse grained materials. Arrows on the XY plane of the specimen in Fig.1 show a track of the X-ray irradiation position during the X-Y plane oscillation. It was carried out using an XY table under control of a personal-computer. The oscillation range is 8mm×22mm, and about 800 grains came to contribute to diffraction.

The ideal orientation method

The conventional sin²ψ method [5] is not applicable to materials which exhibit strongly preferred orientation, because it is based on the assumption of an elastically isotropic body. The ideal orientation method [6] which uses diffraction peaks which belong to the same zone axis was applied to the stress analysis. These diffraction peaks are not under the influence of elastic anisotropy, and stress can be calculated from the linear relation in the 2θ - sin²ψ diagram as well as the conventional sin²ψ method. In the present study, two diffraction peaks which appeared at about ψ₁=0° and ψ₂=60° were used. The X-ray elastic constants \( E_x / (1 + \nu_x) \) and \( \nu_x \) can be obtained by solving the following simultaneous equations (1) and (2);

\[
\frac{1 + \nu_x}{E_x} = \frac{1}{2} \left( \frac{\sigma_M}{\sigma_{app}} \right) \cdot \frac{\pi}{180} \cot \theta_0 \quad (1)
\]

\[
\frac{\nu_x}{E_x} = \frac{1}{2} \left( \frac{\partial (2\theta_{\psi=0})}{\partial \sigma_{app}} \right) \cdot \frac{\pi}{180} \cot \theta_0 \quad (2)
\]

where, \( \sigma_{app} \) is applied stress given by the product of a mechanically determined Young’s modulus \( E_M \) and applied strain \( \varepsilon_{app} \), and M is the slope of the linear relation obtained from only two points (ψ₁=0° and ψ₂=60°) in the 2θ - sin²ψ, and given by

### Table I. Conditions of X-ray stress measurement.

<table>
<thead>
<tr>
<th>Conditions</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tube voltage</td>
<td>30 (kV)</td>
</tr>
<tr>
<td>Tube current</td>
<td>10 (mA)</td>
</tr>
<tr>
<td>Exposure time</td>
<td>600 (sec)</td>
</tr>
<tr>
<td>Camera length</td>
<td>125 (mm)</td>
</tr>
<tr>
<td>Collimator</td>
<td>8 (mm)</td>
</tr>
<tr>
<td>Diffraction plane</td>
<td>Ni₃Al 220</td>
</tr>
<tr>
<td>Characteristic X-ray</td>
<td>VKα</td>
</tr>
<tr>
<td>Filter</td>
<td>Ti</td>
</tr>
<tr>
<td>Diffraction angle</td>
<td>166.927 (deg)</td>
</tr>
<tr>
<td>( \eta - (180-20)/2 )</td>
<td>6.537 (deg)</td>
</tr>
<tr>
<td>( \psi_0 ) low</td>
<td>-7, -6, -5 (deg)</td>
</tr>
<tr>
<td>( \psi_0 ) high</td>
<td>53, 54, 55 (deg)</td>
</tr>
</tbody>
</table>
\[ M = \frac{2\theta_2 - 2\theta_1}{\sin^2 \psi_2 - \sin^2 \psi_1} \]  
\[ (3) \]

The stress constant \( K \) is also given by

\[ K = -\frac{E_x}{2(1 + \nu_x)} \frac{\pi}{180} \cot \theta_0 \]  
\[ (4) \]

**Results and discussion**

**State of preferred orientation in the Ni₃Al ingot**

Structure photographs of the Ni₃Al ingot are shown in Fig. 2. These photographs show that columnar crystals which have a diameter of 300~500\( \mu \)m grew up to the top of the ingot from its solidification bottom. The long direction of these columnar crystals almost exactly corresponds to the \( Z \)-axis of the sample coordinate system as shown in Fig. 1.

Figure 3 shows a \{110\} pole figure measured on the XY-plane of the specimen by the Schulz reflection method. \{110\} crystal planes of Ni₃Al (cubic system) have the azimuth relation of 60° or 90° to each other. From this pole figure, it is thought that the normal direction of \{110\} crystal planes correspond to the \( Z \)-axis ((a) in Fig.3) and cone plane of vertical angle 120°centering on \( Z \)-axis ((b) in Fig.3). However, the accumulation positions of \{110\} poles in the pole figure were discrete and not in ideal symmetry due to the influence of the coarse grains.

![Structure photographs of Ni₃Al ingot](image)

(a) Long direction.  
(b) Transverse direction.

Fig.2. Structure photographs of Ni₃Al ingot.

**Effect of the X-Y plane oscillation method**

Figure 4(a) shows a D-S diffraction ring obtained from a fixed specimen. The incident X-ray beam was vertical to the specimen. On the other hand, Fig.4(b) is a D-S diffraction ring obtained by applying the X-Y plane oscillation to the measurement. From these results, it is found that the X-Y plane oscillation makes the D-S diffraction rings more continuous.

In the ideal orientation method, diffraction profiles on the +\( \eta \) side (\( \alpha = 180^\circ \)) of the D-S diffraction rings are used for the stress analysis. Therefore, the measured D-S diffraction rings should be continuous. The diffraction profiles on the +\( \eta \) side of the D-S diffraction ring in Fig.4(a) and Fig.(4b) are shown in Fig.5. Excellent diffraction profiles could be obtained by
applying the X-Y plane oscillation method to the measurement.

![Pole figure](image)

**Fig.3.** \{110\} pole figure.

**Change of the D-S diffraction ring for \( \psi_0 \)**

D-S diffraction rings measured on each \( \psi_0 \) by applying the X-Y plane oscillation are shown in Fig.6. These D-S diffraction rings changed with the density distribution of \{110\} poles in the direction of \( \psi \) (\( \psi = \psi_0 + \eta \) when \( \alpha = 180^\circ \)). Figure 7 shows the peak intensity distribution of the diffraction profile on the +\( \eta \) side of the D-S diffraction ring for \( \psi \). Very strong peaks appear at \( \psi = 0.537^\circ \) and \( 60.537^\circ \). Because these peaks had the azimuth relation of \( 60^\circ \) to each other, they were regarded as diffraction peaks reflected by \{110\} crystal planes on the same zone axis.

![Diffraction rings](image)

(a) D-S diffraction ring measured on fixed specimen.  
(b) D-S diffraction ring measured on oscillated specimen.

**Fig.4.** D-S diffraction rings measured by IP.
(a) Diffraction profile measured on fixed specimen.  
(b) Diffraction profile measured on oscillated specimen.

Fig.5. Diffraction profiles measured on the \( +\eta \) side of the D-S diffraction ring.

Fig.6. D-S diffraction rings measured on each \( \psi_0 \).
X-ray elastic constants and stress constant

In Fig. 7, the relation between the diffraction angle 2θ and ψ could be approximated to a straight line. The reason has been reported by Yoshioka et al [6]. The diffraction angles 2θ at the peak positions (ψ=0.537° and 60.537°) were found in Fig. 7. Figure 8 shows a 2θ-sin²ψ diagram drawn by using 2θ obtained at two ψ in Fig. 7. Results in cases of applied strain ε_{app} = 0, 100, 200, 300, 400×10⁻⁶ are also shown in Fig. 8.

In Fig. 9, open circles (○) show the relation between the slope M of the straight line in the 2θ-sin²ψ diagram and applied stress σ_{app}, and closed circles (●) show the relation between the intercept 2θ_{ψ=0} of the straight line in the 2θ-sin²ψ diagram and applied stress σ_{app}. Applied stress σ_{app} was obtained by multiplying applied strain ε_{app} and mechanical young's modulus E_M (210GPa). From the linear relation in Fig. 9, an X-ray elastic constant E_X and a stress constant K were obtained by using equations (1) to (4). They are shown in Table II.

Fig. 7. Changes in diffraction angle 2θ and diffraction intensity of notable 220 diffraction lines which belong to the same zone axis observed at ψ=0.537° and 60.537°.

Fig. 8. 2θ-sin²ψ diagram.
Fig. 9. Slope in the $2\theta$-sin$^2\psi$ diagram (M) and intercept ($2\theta_{\psi=0}$) as a function of applied stress.

Table II. X-ray elastic constants, stress constant and mechanical Young's modulus obtained in the present study.

<table>
<thead>
<tr>
<th>X-ray compliance</th>
<th>X-ray elastic constant</th>
<th>Stress constant K</th>
<th>Mechanical Young's modulus E_M GPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>$(1+\nu_X)/E_X$</td>
<td>$\nu_X/E_X$</td>
<td>$E_X/(1+\nu_X)$</td>
<td>$E_X$</td>
</tr>
<tr>
<td>$10^{-3}$/GPa</td>
<td>$10^{-4}$/GPa</td>
<td>GPa</td>
<td>GPa</td>
</tr>
</tbody>
</table>

Theoretical elastic constant of Ni$_3$Al [7]

The X-ray elastic constant $E_X$ which had been obtained by the present method was compared with theoretical elastic constants calculated from the elastic compliance of Ni$_3$Al single crystal [7]. The effectiveness of this X-ray stress measurement method for Ni$_3$Al was confirmed. The theory assumes that a [110] crystal axis of all grains in the specimen completely correspond to P$_3$ axis as shown in Fig. 10.

Fig. 10. Sample model in theoretical calculation, and relation between P system and I system.
Table III. Elastic compliances of Ni$_3$Al single crystal[8].

<table>
<thead>
<tr>
<th>Compliance</th>
<th>$S_{11}$</th>
<th>$S_{44}$</th>
<th>$S_{12}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>(TPa)$^{-1}$</td>
<td>8.04</td>
<td>7.58</td>
<td>-3.07</td>
</tr>
</tbody>
</table>

However, these grains are variable in rotation around the [110] axis. Moreover, the Reuss model and cubic system are assumed. It is through that the peak which appears at $\psi=60^\circ$ as shown in Fig.7 is an accumulation of diffraction lines from specific grains, in which one of [101], [101], [011] or [011] crystal axis corresponds to $\psi=60^\circ$. An elastic constant $E_0$ (in the direction of $P_1$) in these grains given by

$$\frac{1}{E_{60}} = S_0 \left( \cos^4 \gamma + \frac{1}{2} \sin^4 \gamma \right) + S_{12} + \frac{1}{2} S_{44}$$

where, $S_{11}$, $S_{12}$, $S_{44}$ and $S_0 = S_{11} - S_{12} - S_{44}/2$ are the elastic compliances of a cubic single crystal, and $\gamma$ is a rotation angle which converts the I system into the S system in the conversion process such as crystal coordinate system (C) $\rightarrow$ intermediate coordinate system (I) $\rightarrow$ sample coordinate system (S). In this case ((110) preferred orientations), $\gamma$ is given by the following equation using [h k l].

$$\gamma = \arctan \left( \frac{k - h}{\sqrt{2} \cdot l} \right)$$

On the other hand, the strong peak which appears at $\psi=0^\circ$ is an accumulation of diffraction lines from all grains on the specimen surface. In this case, elastic constant $E_0$ in the direction of the $P_1$ axis in these grains is given by

$$\frac{1}{E_0} = \frac{9}{16} S_0 + S_{12} + \frac{1}{2} S_{44}$$

These values obtained by using the elastic compliances of Ni$_3$Al single crystal shown in Table III were $E_0=207$GPa and $E_{60}=228$GPa. Because $E_0$ is an average of all grains while $E_{60}$ is limited to the grains which are in a specific direction, these values are different from each other. It is thought that the elastic constant measured by X-rays at each $\psi$ when applied stress is given to the specimen corresponds to these values. The X-ray elastic constant $E_X$ which had been obtained by using two $\psi$ points was 220GPa. Because the X-ray elastic constant $E_X$ was intermediate between the theoretical values, it can be judged to be an almost appropriate value. Therefore, it has become clear that this X-ray stress measurement method can be effectively applied to Ni$_3$Al which consists of coarse grains and exhibits strongly preferred orientation.
Conclusion

X-ray stress measurement which adopts the X-Y plane oscillation method and the ideal orientation method was applied to Ni$_3$Al which consists of coarse grains and exhibits strongly preferred orientation. Moreover, the reliability of the X-ray elastic constant obtained by this X-ray stress measurement method was confirmed by comparing with the theoretical values obtained from the elastic compliance of Ni$_3$Al single crystal.

The results obtained are summarized as follows;
(1) The X-ray elastic constants measured from Ni$_3$Al 220 diffraction peaks which appeared at $\psi =0.537^\circ$ and $60.537^\circ$ were $E_x/(1+\nu_x)=176$GPa, $E_x=220$GPa and $\nu_x=0.253$.
(2) The theoretical elastic constant $E_0$ at $\psi =0^\circ$ was calculated as 207GPa from the elastic compliances of Ni$_3$Al single crystal. This value refers to the elastic constant in the direction of the tensile axis for the grains which contribute to diffraction. The elastic constant $E_{60}$ at $\psi =60^\circ$ was similarly obtained as 228GPa. The experimental value of X-ray elastic constant $E_x$ (220GPa) is an intermediate value between these theoretical values.

References