RESIDUAL STRESS ESTIMATION OF TI CASTING ALLOY
BY X-RAY SINGLE CRYSTAL MEASUREMENT METHOD

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ABSTRACT

Recently, titanium casting technology has attracted attention in the industrial fields. Since the casting metals involve various kinds of residual stresses due to heat shrinkage, inclusion particles and so on, the accurate estimation of residual stresses is desired in the engineering field. The aim of the present study is to evaluate the nondestructive stress of titanium casting material by X-ray stress measurement. The \( \sin^2 \psi \) method for the usual X-ray stress measurement was used to evaluate the residual stresses. However, it was unsuitable for the measurement of titanium casting materials because of their coarse grains. Therefore, the single crystal X-ray diffraction method was employed for the present system. From the present investigation, the method for single crystal systems could be applied to the residual stress measurement.

INTRODUCTION

Titanium has several advantageous properties, such as high strength, thermal and corrosion resistance, and light weight. Due to these properties, the demand for titanium is expected to rise in the future. Casting technology is an effective method to produce engineering parts from titanium. However, casting generates residual stresses within a body due to non-uniform cooling rate. Since the residual stresses influence the mechanical properties of casting materials, residual stress measurement becomes important.

This study aimed to provide the nondestructive stress evaluation of titanium casting materials using a X-ray stress measurement technique. There is currently little in the literature about X-ray stress measurement of titanium. It has been observed that a large X-ray absorption and a hexagonal closed packed (HCP) crystal system of titanium results in weak diffraction intensities. A large grain size in the casting body is another difficulty for X-ray stress measurement. Experimental approaches for X-ray stress measurements of a single crystal specimen or an individual grain of a polycrystalline specimen had already been developed by several researchers\textsuperscript{1-3}. In the present study, we measured the stresses of individual grains appeared on the surface of a titanium cast sample. The X-ray stress measurement method for single crystal system was used instead of the \( \sin^2 \psi \) method. A three-axis sample table was prepared for the flexible measurement of X-ray diffraction. After determining the crystal orientation, lattice strains at independent poles of \( hkl \) diffraction were measured and then residual stresses were calculated from the theory of elasticity.
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SAMPLE PREPARATION

The measuring sample is Ti-6Al-4V alloy manufactured by vacuum casting. After buff polishing and chemical etching, coarse crystal grains were observed on the surface of sample as shown in Fig. 1. Table 1 shows the composition of the etching solution.

Table 1 Composition of etching solution.

<table>
<thead>
<tr>
<th>Composition</th>
<th>Amount</th>
</tr>
</thead>
<tbody>
<tr>
<td>Distilled water</td>
<td>20 ml</td>
</tr>
<tr>
<td>Hydrogen peroxide (30 %)</td>
<td>5 ml</td>
</tr>
<tr>
<td>Potassium hydrate (40 %)</td>
<td>10 ml</td>
</tr>
</tbody>
</table>

Fig. 1 Surface of titanium casting sample.

PRINCIPLE OF SINGLE CRYSTAL STRESS MEASUREMENT BY X-RAY DIFFRACTION

Fig. 2 defines three types of the Cartesian coordinate system, sample coordinate $P_i$, laboratory coordinate $L_i$, and crystal coordinate $C_i$. $P_3$ is coincident with the surface normal to the sample and $L_3$ defines the normal to the diffraction plane. $\varepsilon_{33}^L$ is the normal strain measured along the $L_3$ direction by X-ray diffraction.

Fig. 3 shows the relationships between the three types of the coordinates system, where $\pi$, $\gamma$ and $\omega$ are the directional cosines between coordinate axes.

The normal strain $\varepsilon_{33}^L$ in $L_3$ direction is written as $\varepsilon_{ij}^C$ in crystal coordinate $C_i$ by the following equation:
\[ \varepsilon_{33}^{L} = \gamma_{3i}^{} \gamma_{3j}^{} \varepsilon_{ij}^{C} \]

\[ = \gamma_{3i}^2 \varepsilon_{11}^{C} + \gamma_{3j}^2 \varepsilon_{22}^{C} + \gamma_{3i}^2 \varepsilon_{33}^{C} + 2(\gamma_{3i}^{} \gamma_{3j}^{} \varepsilon_{23}^{C} + \gamma_{3j}^{} \gamma_{3i}^{} \varepsilon_{13}^{C} + \gamma_{3i}^{} \gamma_{3j}^{} \varepsilon_{12}^{C}). \]

In the HCP system, the stress-strain relation is described as follows \(^5\) using the elastic compliances \(S_{ij}\).

\[ \varepsilon_{11}^{C} = S_{11} \sigma_{11}^{C} + S_{12} \sigma_{22}^{C} + S_{13} \sigma_{33}^{C} \]
\[ \varepsilon_{22}^{C} = S_{12} \sigma_{11}^{C} + S_{13} \sigma_{22}^{C} + S_{13} \sigma_{33}^{C} \]
\[ \varepsilon_{33}^{C} = S_{13} \sigma_{11}^{C} + S_{13} \sigma_{22}^{C} + S_{13} \sigma_{33}^{C} \]
\[ 2\varepsilon_{23}^{C} = S_{44} \sigma_{23}^{C} \]
\[ 2\varepsilon_{31}^{C} = S_{44} \sigma_{31}^{C} \]
\[ 2\varepsilon_{12}^{C} = 2(S_{11} - S_{12}) \sigma_{12}^{C} \]

As the main purpose is the description of the normal strain \(\varepsilon_{33}^{L}\) in terms of the stresses \(\sigma_{ij}^{P}\) in the sample coordinate system, we use the following relationship between \(\sigma_{ij}^{C}\) and \(\sigma_{ij}^{P}\) with the help of transformation matrix \(\pi\) in Fig.3.

\[ \sigma_{ij}^{C} = \pi_{ik} \pi_{jl} \sigma_{kl}^{P}. \]

Substituting eqs. (2) and (3) for eq.(1), \(\varepsilon_{33}^{L}\) is written as follows under the condition of plane stress state.

\[ \varepsilon_{33}^{L} = (A_1 \pi_{11}^2 + A_2 \pi_{22}^2 + A_3 \pi_{12}^2 \pi_{13} + A_4 \pi_{13}^2 \pi_{11} + A_5 \pi_{12}^2 \pi_{12}) \sigma_{11}^{P} \]
\[ + (A_6 \pi_{21}^2 + A_7 \pi_{22}^2 + A_8 \pi_{23}^2 \pi_{23} + A_9 \pi_{23}^2 \pi_{21} + A_{10} \pi_{22}^2 \pi_{22}) \sigma_{22}^{P} \]
\[ + 2A_1 \pi_{13} \pi_{21} + 2A_2 \pi_{12} \pi_{22} + 2A_3 \pi_{12} \pi_{23} + A_4 (\pi_{12} \pi_{23} + \pi_{22} \pi_{21}) \]
\[ + A_5 (\pi_{13} \pi_{21} + \pi_{23} \pi_{11}) + A_6 (\pi_{12} \pi_{22} + \pi_{23} \pi_{12}) \sigma_{12}^{P}, \]

where

\[ A_1 = \gamma_{31}^2 S_{11}^2 + \gamma_{32}^2 S_{12}^2 + \gamma_{33}^2 S_{13}^2 \]
\[ A_2 = \gamma_{31}^2 S_{12}^2 + \gamma_{32}^2 S_{11}^2 + \gamma_{33}^2 S_{13}^2 \]
\[ A_3 = \gamma_{31}^2 S_{13}^2 + \gamma_{32}^2 S_{11}^2 + \gamma_{33}^2 S_{13}^2 \]
\[ A_4 = \gamma_{32}^2 S_{44} \]
\[ A_5 = \gamma_{33}^2 S_{44} \]
\[ A_6 = 2(\gamma_{31}^{} \gamma_{32}^{} (S_{11} - S_{12})) \]

Having three \(\varepsilon_{33}^{L}\) values in the independent direction, the stress components \(\sigma_{11}^{P}, \sigma_{22}^{P}\) and \(\sigma_{12}^{P}\) can be calculated from eq. (4).
As indicated in Fig.4, the HCP crystal coordinate system \((a_1, a_2, c)\) is different from the Cartesian coordinate system \((x_1, x_2, x_3)\). Therefore, the HCP coordinate system should be changed to the Cartesian coordinate system. Eq. (5) shows the transformation from Miller indices \((hkl)\) in the HCP system to the Cartesian coordinate system \((HKL)\). By the same meaning, eq. (6) is the direction indices, where \(\lambda = c/a\) is the lattice parameter ratio of the crystal system.

\[
\begin{align*}
(H & \ K & \ L) = \begin{pmatrix} h & \frac{h+2k}{\sqrt{3}} & l \end{pmatrix} : \text{plane indices} & (5) \\
[U & \ V & \ W] = \begin{pmatrix} u & \frac{u+2v}{\sqrt{3}} & \lambda w \end{pmatrix} : \text{direction indices} & (6)
\end{align*}
\]

The direction cosines of the coordinate transformation between \(L_3\) and \(C_1\) are defined by \(\gamma_{3k}\). When diffraction plane is shown by \(HKL\), \(\gamma_{3k}\) is described in the following equations:

\[
\begin{align*}
\gamma_{31} &= \frac{H}{\sqrt{H^2 + K^2 + L^2}}, & \gamma_{32} &= \frac{K}{\sqrt{H^2 + K^2 + L^2}}, & \gamma_{33} &= \frac{L}{\sqrt{H^2 + K^2 + L^2}}. & (7)
\end{align*}
\]

Furthermore, the direction cosines of the coordinate transformation between \(P_1\) and \(C_1\) are defined by \(\pi_{ij}\). We then define four angles, \(\psi, \alpha, \varphi\) and \(\beta\), as shown in Fig.5. \(\psi\) and \(\alpha\) determine the angle between the surface normal and \(C_3\) axis and \(\varphi\) is the rotating angle around \(C_3\) axis. \(\beta\) is defined as the following equation:

\[
\beta = \psi - \alpha. & (8)
\]

Finally, the directional cosine matrix \(\pi_{ij}\) is described as follows:

\[
\pi_{ij} = \begin{pmatrix}
\cos \beta & \sin \beta & 0 \\
-\sin \beta & \cos \beta & 0 \\
0 & 0 & 1
\end{pmatrix}
\begin{pmatrix}
\cos \alpha \cos \psi & \sin \alpha \cos \psi & -\sin \psi \\
\cos \alpha & 0 & 0 \\
\cos \alpha \sin \psi & \sin \alpha \sin \psi & \cos \psi
\end{pmatrix}
\] & (9)

Fig. 4 Conversion of the coordinate system.

Fig. 5 Definition of \(\alpha, \psi\) and \(\varphi\) (Stereographic projection).
EXPERIMENTAL PROCEDURE

In the present study, the Schulz reflection method was used to produce a stereographic diagram. This method requires a three-axis sample table which allows a rotation of the sample about surface normal axis and horizontal axis. Three axes are the \( \psi \), \( \chi \) and \( \theta-2\theta \) as shown in Fig. 6.

The three axes \( \psi \), \( \chi \) and \( \theta-2\theta \) intersect each other at a point on the sample surface. This point also coincides with the irradiation position of X-ray beams.

At first, a stereographic diagram was measured by rotating the sample table. Fig. 7 shows the stereographic diagram measured in the near-edge region on the surface of sample. The described poles are consistent with two crystals existing in the irradiation area.

Fig. 8 shows an enlarged view of the stereographic diagram and diffraction profiles of \( \theta-2\theta \) scan at each pole. The plane indices are specified at each pole, and peak positions are determined from the profiles. The X-ray stress measurement is based on the Bragg’s law (eq. (10)):

\[
\lambda = 2d \sin \theta, \tag{10}
\]

where \( \lambda \) is the X-ray wavelength, \( d \) the interplanar spacing and \( \theta \) the diffraction angle.

We evaluate the interplanar spacings by measuring diffraction angles. Lattice strain is then calculated from the following equation:

\[
\varepsilon = \frac{d - d_0}{d_0}, \tag{11}
\]

where \( d_0 \) is the stress free lattice spacing, \( d \) the lattice spacing measured by X-ray diffraction.

Finally, the stress components are calculated by eq. (4). If we have three strains corresponding to independent three poles, the stress components \( \sigma_{11}, \sigma_{22} \) and \( \sigma_{12} \) are calculated by the simultaneous equation. If more than three poles exist, the multiple linear regression analysis is used for the determination of the three components.

Conditions of X-ray stress measurement and the elastic compliances of titanium are shown in Table 2 and Table 3\(^7\), respectively.
Fig. 8 Enlarged stereographic diagram and $\theta$-2$\theta$ profiles at each pole.

Table 2 Conditions of X-ray stress measurement.

<table>
<thead>
<tr>
<th>Characteristic X-rays</th>
<th>CuK$\alpha$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Incident slit</td>
<td>Collimator</td>
</tr>
<tr>
<td>Receiving slit</td>
<td>Parallel beam slit</td>
</tr>
<tr>
<td>Tube voltage</td>
<td>40 kV</td>
</tr>
<tr>
<td>Tube current</td>
<td>20 mA</td>
</tr>
<tr>
<td>hkl plane</td>
<td>00·4 $\theta = 82.5^\circ$</td>
</tr>
<tr>
<td>Diffraction angle</td>
<td>21·3 $\theta = 141.5^\circ$</td>
</tr>
<tr>
<td>$\chi$-oscillation range</td>
<td>$\pm 2.0$ deg</td>
</tr>
<tr>
<td>$\psi$-oscillation range</td>
<td>$\pm 2.0$ deg</td>
</tr>
<tr>
<td>Filter</td>
<td>Nickel</td>
</tr>
<tr>
<td>Irradiated area</td>
<td>$\varnothing$ 1 mm</td>
</tr>
</tbody>
</table>

Table 3 Elastic compliances of titanium. ($1/10^2$ GPa)

<table>
<thead>
<tr>
<th>$S_{11}$</th>
<th>$S_{12}$</th>
<th>$S_{13}$</th>
<th>$S_{33}$</th>
<th>$S_{44}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.958</td>
<td>-0.462</td>
<td>-0.189</td>
<td>0.698</td>
<td>2.141</td>
</tr>
</tbody>
</table>

EXPERIMENTAL RESULTS AND DISCUSSIONS

Fig. 9 and 10 show the results of residual stresses $\sigma_{11}$ and $\sigma_{22}$ in each crystal which exists on the sample surface, respectively. One data point corresponds to the stress in one crystal. Different stresses shown by one to four data points at the individual distance from the center indicate that the stress is different from one crystal to another even in one irradiated area.
Fig. 9 Distribution of residual stress $\sigma_{11}$.

Fig. 10 Distribution of residual stress $\sigma_{22}$.

Fig. 11 Model of residual stress distribution in a homogeneous casting metal with fine grains.

Fig. 11 shows a model of residual stress distribution in a homogeneous casting metal with fine grains. In the process of solidification, the outer part first begins to solidify and then the inner part solidifies. Due to the different cooling rate between the outer part and the inner part, the hoop residual stress, $\sigma_{22}$, of the outer part becomes compressive and that of the inner part becomes tensile as shown in Fig. 11(b). The radial residual stress, $\sigma_{11}$, at the edge of the specimen must be zero and the stress at the center must be the same as the hoop residual stress. Therefore, tensile stresses distribute from the center to the edge as shown in Fig. 11(a). Usually, no shear stresses should exist in the surface. However, the present results on a coarse
grained sample shows that the stress in each grain does not obey the normal distribution like the model. Furthermore, Fig. 12 shows the non-zero shear stress distribution. The shear stresses in each crystal are different from the stresses in other crystals. The results obtained in this study suggest that the individual grains of large size exhibit a different stress state both in the magnitude and the direction of the principle axis as shown in Fig. 12. The state of different stresses in different grains of a polycrystalline material is expressed as Heyn stress\(^8\) or the intergranular stress. The present experimental results are a typical case of the intergranular stress\(^9\). Similar results were obtained in case of pure iron that had been plastically elongated by 3 %.\(^1\)

CONCLUSIONS

Residual stresses in a titanium casting material composed of coarse grains were measured by the X-ray technique for single crystal materials. The conclusions are as follows:
1. The X-ray stress measurement method for a single crystal system is formalized to measure residual stresses in an HCP crystal.
2. Even in the irradiated area, the residual stresses are different in each crystal. Furthermore, the direction of principal axis is different in each crystal.

REFERENCES