Development of a Strain Evaluation Technique Using a Micro Focus X-ray Beam

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A strain evaluation technique using a micro focus X-ray beam was developed. An electron gun with two electron lenses was used in order to steadily make a fine focus of an electron beam on a copper target. A target current was 24 μ A, and an X-ray brightness 162×10^9 W/m² was obtained. X-ray beam was condensed to a converging angle 0.09 deg with a convergent unit, and the minimum focus diameter was 60 μ m. A Cu-K α_2 's peak can be easily split from a K α_1 's peak, because the converging angle of the X-ray beam is low and an X-ray brightness is high. A charge coupled device camera can be set in order to observe the irradiated area on a sample. A cubic ZrO₂-10 mol%Y₂O₃ single crystal cut along a (001) plane was selected as a sample to ensure a measurement technique. The difference in a Miller index from an integer was less than 0.4%. A lattice parameter of the specimen was 0.51442 ± 0.00002 nm, and the reference value was 0.5144 nm. Strain was loaded to the sample with a four-point bending jig. A strain tensor was analyzed from the difference in the lattice parameters which were calculated using from 15 to 50 planes. Although the some measured values had a more than 100×10^{-6} difference from the estimated ones, the other measured values were almost same as the estimated ones within experimental error.

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1. Introduction

High performance composite materials are developed for various regions, such as electrics, optics, structural and heat resistant material in order to achieve the synergistic properties. Stress is concentrated between different elastic coefficient interfaces or different thermal expansion interfaces, and the evaluation technique is required. However, the smaller and more complex the devices are, the more difficult to evaluate the stress concentration.

The $\sin^2 \psi$ method using X-ray, which is the most well-known evaluation of the stress, is inadequate for the orientated structure, because the random crystal direction is assumed in this method. Hanabusa *et al.*¹⁾ and Tanaka *et al.*,^{2,3)} applied an improved $\sin^2 \psi$ diagram for the fibrous texture that is completely orientated in a thin film, but there are few studies on an incomplete orientated structure. A specimen which has large strain gradient in an X-ray irradiated region is also inadequate, because the averaged strain of a plenty of grains is detected with the $\sin^2 \psi$ method. From these restrictions, the irradiated diameter is required more than 300 μ m.

A new strain-evaluation technique which overcomes these problems should be developed for the micro-structures. Suyama *et al.*⁴⁾ irradiated a micro focus X-ray beam through $\phi 1$ mm pinhole on one grain (mean grain size = approximately 20 nm) of a coarsened Fe+3%Si specimen, and detected the reflected X-ray with a Position Sensitive Proportional Counter. The structure in the irradiated area is a single crystal, and the principal strain distribution is calculated by the changes in the lattice spacing. However the precise strain-evaluation technology for finer crystals should be developed.

Although a high intensity and micro focus X-ray beam should be used in order to evaluate stress in a micro region, it can not be obtained except in synchrotron radiation facilities.^{5,6)} In this study, the strain evaluation technology using a small-scale micro focus X-ray diffraction system is developed.

Fig. 1 Schematic diagram of the developed apparatus.

2. Specifications of the Developed Apparatus

Following specifications should be satisfied in order to detect reflection X-rays from one single crystal and precisely evaluate the strain.

- (1) An X-ray source producing a high brightness X-ray in steady.
- (2) A convergent unit producing a low converging angle and micro focus X-ray beam.
- (3) A goniometer detecting diffracted X-rays in precise.

An apparatus that satisfies all these specifications were developed with RIGAKU Co. A schematic diagram of the developed apparatus is shown in Fig. 1, and the specifications are mentioned below.

2.1 X-ray source

A rotating anode is a well-known apparatus as a high power X-ray source. For example, in the case of ultraX18 (RIGAKU Co.), the tube voltage and the target current is 40 kV and 30 mA, respectively. However, the X-ray brightness, which is calculated by dividing the maximum allowable load by the focus size on a target, is low, 12×10^9 W/m².

An electron gun with two electron lenses was used in order to steadily make a fine focus of the electron beam on a copper target. A single LaB₆ crystal, of which the emission area is

$\phi 200 \mu\text{m}$, was used as an emission source of the electron beam, because its work function is lower and a lifetime is longer compared with tungsten. An X-ray is generated from $2.2 \times 3.36 \mu\text{m}^2$ on the target. The target current was $24 \mu\text{A}$, and the X-ray brightness $162 \times 10^9 \text{ W/m}^2$ was obtained, when the tube voltage was 50 kV, and the tube current was 0.7 mA.

2.2 Convergent unit

The X-ray generated on the target is condensed to the converging angle 0.2 deg with an artificial multilayer mirror unit. This mirror unit consists of two mirrors made of tungsten and silicon, and condenses the X-ray by reflecting once on each mirror.

The converging angle of the X-ray beam was decreased to 0.09 deg with an aperture ($\phi 0.2 \text{ mm}$), and the minimum focus diameter was $60 \mu\text{m}$. The converging angle was measured by a profile shown in Fig. 2, of the direct beam around the 2θ origin. The focus diameters in a horizontal plane and a vertical plane were measured by scanning a $\phi 20 \mu\text{m}$ pinhole around the focus position, as shown in Fig. 3. Figure 4 shows the diffraction profile of a (004) plane of a cubic ZrO_2 –10 mol% Y_2O_3 single crystal. The $\text{Cu-K}\alpha_2$'s peak is completely split from the $\text{K}\alpha_1$'s peak, because the converging angle of the X-ray beam is low and the X-ray brightness is high.

2.3 Goniometer

The incident angle ω , and the diffraction angle 2θ , are detected by rotary encoder. Their angle resolution is 10^{-4} deg/step . A charge coupled device camera, which magnifies 500 times on a monitor, can be set in order to observe the irradiated area on a sample. A four-point bending jig can be set on a sample stage in order to measure the diffraction angle of the sample with a load.

3. Experimental Procedures

A cubic ZrO_2 –10 mol% Y_2O_3 single crystal cut along a (001) plane ($5 \times 5 \times 45 \text{ mm}^3$) was selected as a sample to ensure a measurement technique. Strain (300×10^{-6}) was loaded to the sample with the four-point bending jig. Measuring points with X-ray and a strain gauge are shown in Fig. 5.

Strain tensor was analyzed from the difference in the lattice parameters,⁷⁾ which were calculated using from 15 to 50 planes.

Fig. 2 Direct beam profile around 2θ origin.

Fig. 3 Dimension of direct beam at focus position.

Fig. 4 Diffraction pattern of ZrO_2 –10 mol% Y_2O_3 (004) plane.

Fig. 5 Strain measuring positions on a specimen.

Fig. 6 Relationship between the unit cell a_i and the Cartesian coordinates u_i .

Consider a crystal with the lattice parameters a_i and α_i is attached to a Cartesian-coordinate system u_i as shown in Fig. 6, that is the u_1 axis parallel to a_1 , the u_2 axis in the plane of a_1 and a_2 , and the u_3 axis perpendicular to that plane.

Real-lattice vectors in the Cartesian-coordinate system are expressed as,

$$A = \begin{pmatrix} a_1 & a_2 \cos \alpha_3 & -a_3 \cos \alpha_2 \\ 0 & a_2 \sin \alpha_3 & -a_3 \sin \alpha_2 \cos \beta_1 \\ 0 & 0 & 1/b_3 \end{pmatrix}, \quad (1)$$

where the β_i s and b_i s are the reciprocal-lattice parameters for the unit cell.

Let A_0 and A_1 be the matrix for the unstrained unit cell and the strained cell, respectively. A matrix T which transforms from A_0 to A_1 is defined as follows,

$$A_1 = TA_0. \quad (2)$$

From the definition of the strain tensor ϵ_{ij} , the strain tensor in the Cartesian-coordinate system is given by

$$\epsilon_{ij} = (T_{ij} + T_{ji})/2 - I_{ij}, \quad (3)$$

where I is a unit matrix.

Fig. 7 Principal strain distribution.

The strain tensor contains both a hydrostatic strain $\Delta/3$ and a distortion strain term, therefore eq. (3) can be written in terms of the two components

$$\boldsymbol{\varepsilon} = \boldsymbol{\varepsilon}_d + \frac{\Delta}{3} \mathbf{I}, \quad (4)$$

where $\Delta = \varepsilon_{11} + \varepsilon_{22} + \varepsilon_{33}$. $\boldsymbol{\varepsilon}_d$ is the distortional term,

$$\boldsymbol{\varepsilon}_d = \begin{pmatrix} \varepsilon_{11} - \Delta/3 & \varepsilon_{12} & \varepsilon_{13} \\ \varepsilon_{12} & \varepsilon_{22} - \Delta/3 & \varepsilon_{23} \\ \varepsilon_{13} & \varepsilon_{23} & \varepsilon_{33} - \Delta/3 \end{pmatrix}. \quad (5)$$

Principal strains, ε_1 , ε_2 and ε_3 , are calculated as eigenvalues of $\boldsymbol{\varepsilon}_d$.

4. Results

A part of the result of a crystal orientation analysis for the specimen without a load is shown in Table 1. All the diffraction intensity exceeds 500 s^{-1} , and it follows that an extremely high brightness X-ray is irradiated on the specimen. The differences in the Miller indices from integers were less than 0.4%. The lattice parameter of the specimen was $0.51442 \pm 0.00002 \text{ nm}$, and the reference value was 0.5144 nm .⁸⁾ From these results, it follows that a crystal orientation and lattice parameters can be extremely precisely analyzed by the developed apparatus.

Principal strains of each measuring point were calculated using from eqs. (1) to (5). A principal strain distribution with a load is shown in Fig. 7. Errors of the principal strains were evaluated from errors of the lattice parameters. A principal strain estimated from the strain gauge's value and the crystal orientation is also plotted in Fig. 7. Although the some

measured values had a more than 100×10^{-6} difference from the estimated ones, the other measured values were almost same as the estimated ones within experimental error. In conclusion, the micro focus X-ray diffraction system can be used for strain evaluation in micro region.

5. Summary

Strain evaluation technology which can be used in a small-scale system was developed.

- (1) An X-ray source which produced high brightness X-ray in steady was obtained. The target current was $24 \mu\text{A}$, and the X-ray brightness was $162 \times 10^9 \text{ W/m}^2$.
- (2) A convergent unit which produced a low converging angle (0.09 deg) and a fine focus X-ray beam ($\phi 60 \mu\text{m}$) was installed.
- (3) A goniometer which detects reflection X-rays with an angle resolution for 2θ and ω of 10^{-4} deg/step was used.
- (4) The principal strain distribution in a cubic ZrO_2 – $10 \text{ mol\%Y}_2\text{O}_3$ single crystal was measured. Most of the measured values were almost same as the estimated ones within experimental error.

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Table 1 Reflection index of ZrO_2 – $10 \text{ mol\%Y}_2\text{O}_3$.

Miller index			Intensity, I/s^{-1}
h	k	l	
0.000	0.000	4.000	3313
0.998	–1.001	5.000	1061
0.001	0.000	6.000	836
4.000	–0.001	4.000	640
2.999	–1.001	5.000	826
2.001	–0.001	5.999	1287
–0.004	–1.999	6.000	1106
4.001	1.999	3.999	515