Evaluation of ductile damage progress of aluminum single crystal with prior activity of single slip system under tensile loading by using synchrotron white X-ray

J. Shibano[1,a*], K. Kajiwara[2,b], T. Tsukamoto[3], H. Kawai[3], S. Miura[1,c], S. Zhang[4,d], T. Shobu[4,e] and M. Kobayashi[1,f]

[1] Dept. of Mech. Eng., Kitami Inst. of Tech., Kouen-cho, Kitami, 090-8507 Japan.

[2] Japan Synchrotron Radiation Research Institute, Japan.

[3] Graduate students, Kitami Inst. of Tech., Kouen-cho, Kitami, 090-8507 Japan.

[4] Synchrotron Radiation Research Center, Japan Atomic Energy Agency, Japan.

[a] jshibano@mail.kitami-it.ac.jp, [b] kajiwara@spring8.or.jp, [c] miurast@mail.kitami-it.ac.jp

[d] zhang.shuoyuan@jaea.go.jp, [e] shobu.takahisa@jaea.go.jp, [f] kobayasi@mail.kitami-it.ac.jp

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Abstract. A ductile damage progress of an aluminum single crystal with the prior activity of the single slip system under tensile loading was verified by a profile analysis using white X-ray obtained in BL28B2 beam line of SPring-8. In this study, the aluminum single crystal of the purity 6N was used as a specimen prepared in I-type geometry for tensile test. A notch was introduced into one side of the center of a parallel part of the specimen by the wire electric discharge machining. White X-ray beam, which has 50 μm in both height and width, was incident into the specimen on the Bragg angle θ of 3 degrees using energy dispersive X-ray diffraction technique. The specimen was deformed by elongation in the direction of 45° to [111] and $[1\bar{1}0]$ crystal orientations, respectively, and a diffraction profile of the white X-ray from Al220 plane was analyzed. In profile analysis, an instrumental function was defined in consideration both of a divergence by a slit and a response function peculiar to the energy dispersive method. The Gauss component of integral breadth related to non-uniform strain and the Cauchy component of integral breadth related to crystallite size were determined by eliminating the broadening by the instrumental function from the diffraction profile of white X-ray. As a result, the characteristics of ductile damage progress near the notch of the aluminum single crystal were inspected from the distribution of both non-uniform strain and dislocation density.

Introduction

In order to verify experimentally the relation between the progress of ductile damage and crystal orientation, the X-ray diffraction measurements of a single crystal is valid. It is known that the shape of the diffraction X-ray profile of a metallic material obtained by X-ray diffraction method is connected to the ductile damage by plastic deformation, and many researches have been done for many years[1]~[7]. Authors are also investigating about the ductile damage evaluation of the crystal level using synchrotron radiation white X-ray. By our previous study[8], ductile damage progress behavior near the notch of the aluminum single crystal to which crystal orientation [001] turned to the direction of a tension load was clarified. At that time, the instrumental function in consideration of a response function peculiar to an energy dispersion method and the breadth by an angle of divergence was derived, and the method to determine for the Gauss component originating in a non-uniform strain or the Cauchy component originating in crystallite size from the integral breadth of a diffraction X-ray profile was proposed.

In this study, verification of the ductile damage progress behavior of an aluminum single crystal using white X-ray was performed in the BL28B2 beam line of synchrotron radiation facilities SPring-8. One-side notched specimen was produced from the aluminum single crystal of 6N purity.

Each crystal orientation of [111] and $[1\bar{1}0]$ of the specimen turns to 45° direction to a loading direction. The area near the notch of the specimen which carried out plastic deformation by the tensile load was measured with white X-ray diffraction technique. A non-uniform strain and dislocation density were calculated from the integral breadth Gauss component obtained from X-ray diffraction profile, and ductile damage progress behavior was examined based on those values.

Experimental Procedure

Specimen and tension equipment. The specimen was a single aluminum crystal of 6N purity (99.9999% pure). The specimen, with [111] and $[1\bar{1}0]$ crystal orientations which have 45° slant to a tensile load direction, was cut from the crystal disk by a low-speed diamond cutter. The specimen of I-type geometry used in tensile tests (Fig. 1) was produced by milling. The notch (width and depth of 0.34 mm and 0.20 mm, respectively) was introduced into one side of the center of a parallel section by wire electric discharge machining. The compact tension equipment (Fig. 2) was used to apply a tension load to a specimen.

Measurement facility and conditions. Measurements were performed in a BL28B2 beam line installed in the synchrotron radiation facility SPring-8. Semiconductor detector (SSD) was oriented at a horizontal diffraction angle of 6°. The slits installed at the irradiation sides were 50 μm high and 50 μm wide, and those installed at the detector sides were 100 μm high and 100 μm wide. The lattice strain was measured by the transmission of white X-ray. The gauge volume of this measurement is shown in Fig. 3. By using radioactive isotopes of Co-57 and Am-241, the energy calibration formula was calculated as follows:

$$En = 0.059066 \times CH + 0.37067 \quad [\mathrm{KeV}], \tag{1}$$

where *CH* is a channel number of MCA. An example of the transmission diffracted X-ray profile of a specimen is shown in Fig. 4. The peak of diffraction profile of Al220 was measured relative to the $[1\bar{1}0]$ crystal orientation.

First, five points near the center of specimen were measured for 90s per point in the no-load condition. The average value of the measured peak energy of each lattice plane was made into non-strain data. The stress–strain diagram of the specimen, under the strain rate of ~3.1×10^{-6}/s, is shown in Fig. 5. As illustrated in that figure, measurements near the notch were taken at applied strains of 1.0% (measurement point 1) and 5.0% (measurement point 2). A measuremet positions are shown in Fig. 6. Measurements were taken at a rate of 60s per position. During the measurements, the load was stopped and the distance between grips was held.

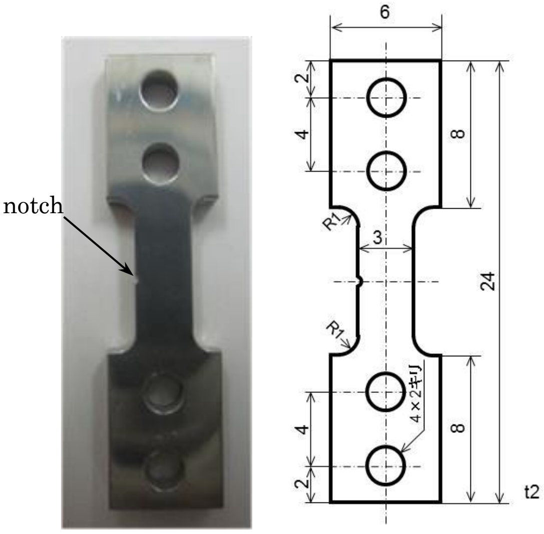


Fig.1 Specimen configuration for tensile test.

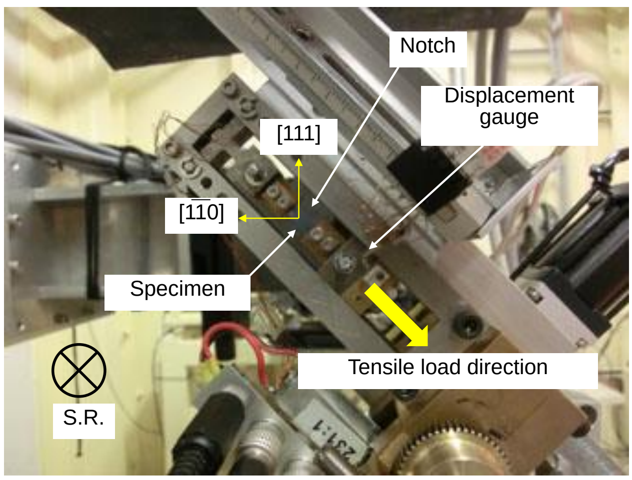


Fig.2 Compact tension equipment for strain measurement using synchrotron white X-ray.

Profile fitting. In order to consider the progress of ductility damage in detail from a diffraction X-ray profile, profile fitting was performed with the Voigt function, expressed by Eq. (2):

$$I = I_0 + A\frac{2\ln 2}{\pi^{3/2}}\frac{wC}{wG^2}\int_{-\infty}^{\infty}\frac{e^{-t^2}}{\left(\sqrt{\ln 2}\frac{wC}{wG}\right)^2+\left(\sqrt{4\ln 2}\frac{E-E_C}{wG}-t\right)^2}dt\ , \tag{2}$$

where I_0 is a background, A is the integrated intensity of a diffraction profile, wG is a gauss component of the integral breadth of a diffraction X-ray profile, wC is the Cauchy component, E is X-ray energy, and E_c is peak energy.

Calculation of non-uniform strain and dislocation density. Non-uniform strain $\Delta\varepsilon$ was calculated by Eq. (3) using true Gauss component β_G of the integral breadth of a diffraction X-ray profile and X-ray energy E_B diffracted by Bragg angle θ_B. Here, the instrumental function which is needed when determining for true Gauss component β_G is calculated using a response function and the angle of divergence of synchrotron radiation.

$$\Delta\varepsilon = \frac{\beta_G}{E_B}. \tag{3}$$

Dislocation density is calculated from non-uniform strain $\Delta\varepsilon$ as follows:

$$\rho = \frac{k\Delta\varepsilon^2}{F\mathbf{b}^2}\ , \tag{4}$$

where, **b** is a Burgers vector, F is a factor about the interaction during a dislocation, and k is a constant depending on strain distribution. F is usually set to 1, and k is set to 16.1, when setting the slip direction to [110] in a face centered cubic lattice.

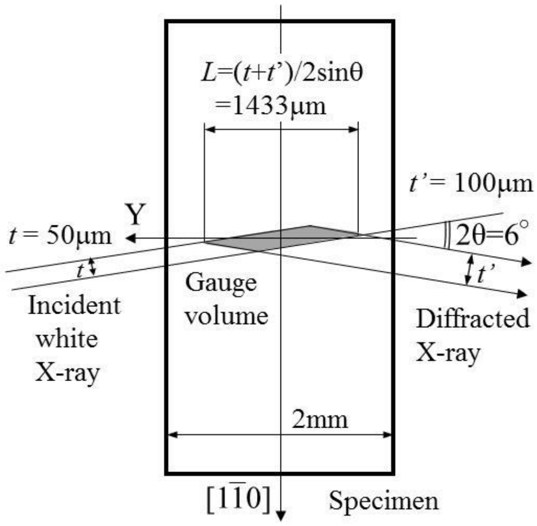


Fig.3 Schematic diagram of gauge volume using transmission diffracted X-ray.

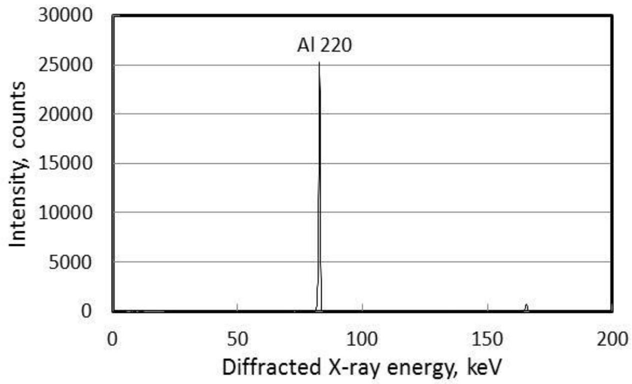


Fig.4 Diffracted X-ray profile of Al single crystal.

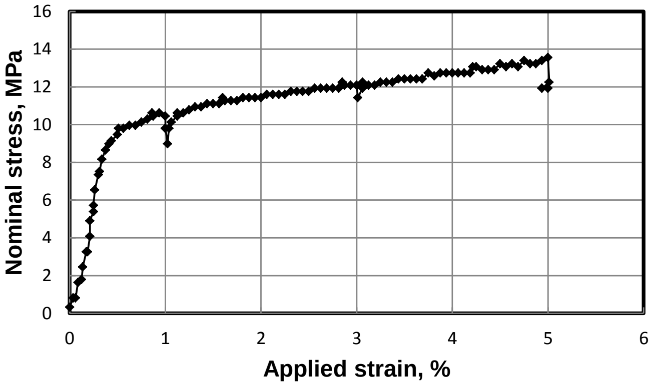


Fig.5 Stress-strain diagram and measurement points of one-side notched Al single crystal specimen.

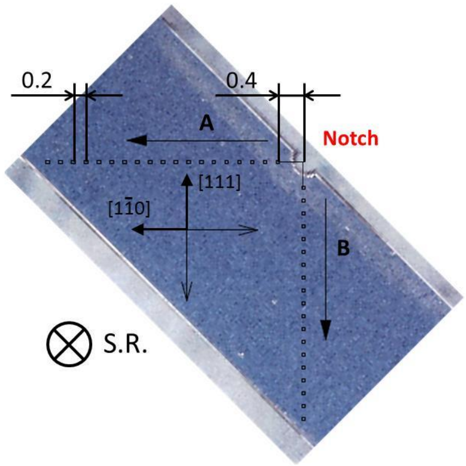


Fig.6 Measurement positions in the specimen.

Results and Discussion

Generation and rotation of subgrain. Diffraction X-ray from the aluminium single-crystal specimen decreased sharply as plastic deformation progresses. Since it was estimated both of a generation and a rotation of subgrain from this phenomenon, the rotation angle was investigated by a flat panel sensor as the two-dimensional detector. We installed the flat panel sensor in the detector side so that the diffraction X-ray which penetrated the specimen may be detected. The example of the measured transmission type Laue pattern is shown in Fig. 7. The angle of the specimen was adjusted for every position of measurement so that the spot of Al220 diffraction of Fig. 7 might be symmetrically arranged to a center on the flat panel sensor. Furthermore, the specimen angle was finely tuned by SSD so that diffraction X-ray intensity might serve as the maximum. The relative angle variations of the normal direction of Al220 diffraction plane in horizontal and vertical planes are shown in Figs. 8a and 8b, respectively. Those values in the horizontal plane shown in Fig. 8a show that the crystal orientation is changing gradually as it gets away from a notch. And, although the angle variation in the measurement direction A indicated the almost same tendency at 1% and 5% of applied strains, it indicated the reverse tendency in the measurement direction B. The angle variations in the vertical plane indicated in Fig. 8b show that those changes are very large compared with them in a horizontal plane. In particular, the angle of rotation near the notch is large at applied strain 5%. It is estimated that the slip activity of Al111 plane by tension load is larger near the notch.

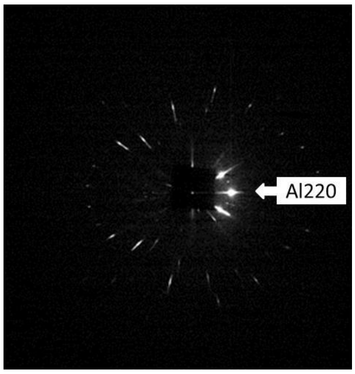
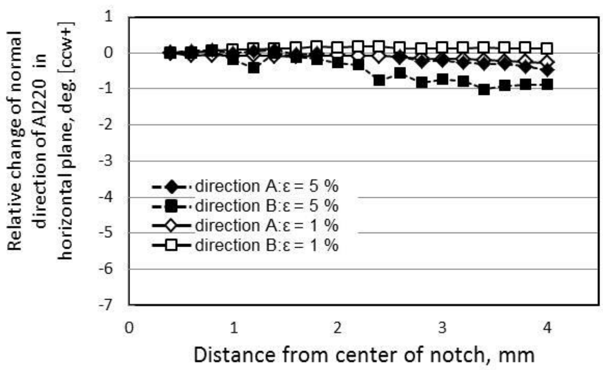
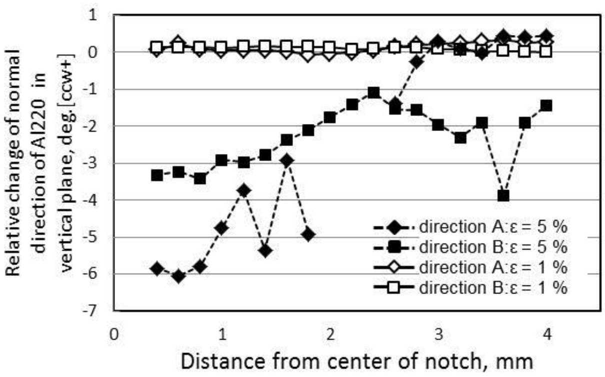


Fig.7 Transmission SR Laue diffraction pattern of Al single crystal by flat panel sensor.

Integrated-intensity distribution of a diffraction X-ray profile is shown in Fig. 9. At 1.0% of



(a) in horizontal plane



(b) in vertical plane

Fig.8 Distribution of relative rotation angle of normal direction of diffraction plane of Al220.

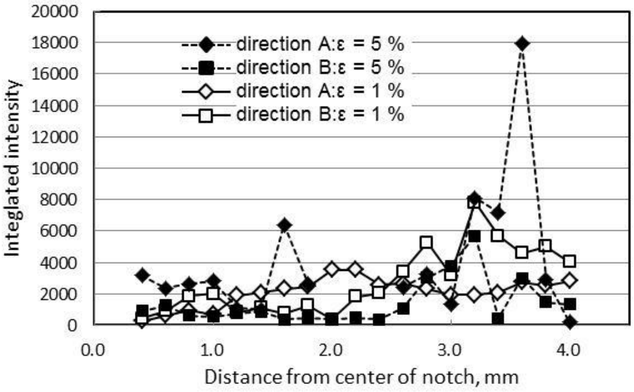


Fig.9 Integrated intensity of Al 220 diffraction profile.

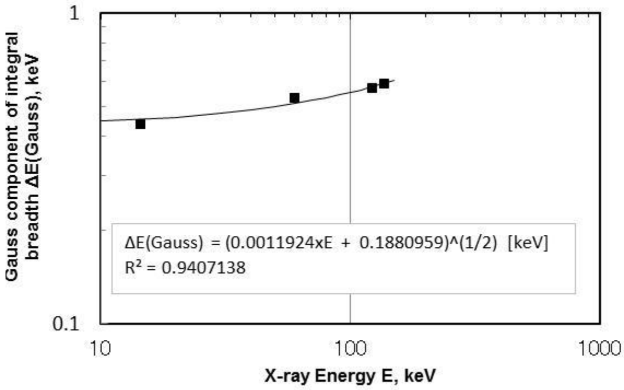


Fig.10 Gauss component of integral breadth of instrumental function in the measurement system.

applied strain, integrated intensity is low near the notch also in which measurement directions of A and B. Integrated intensity becomes so high that there are many crystal grains which satisfies X-ray diffraction conditions. Subgrains generate generally in a single crystal with the progress of ductile damage. Particularly near the notch, there is also much generation of the subgrain accompanying a development of a slip band due to ductile damage progress. Some of subgrains do not satisfy X-ray diffraction conditions by those rotations; thus the integrated intensity becomes also low. On the other hand, in the area away from the notch, it is estimated that there are few developments of subgrain, and that those rotations are also small. Accordingly, the integrated intensity may become relatively high.

Distribution of non-uniform strain and dislocation density. The measurement spectrum of Co-57 and Am-241 radioisotopes were fitted by using the Gaussian curve. The Gauss component was obtained from those integral breadth. The relation between diffraction X-ray energy *E* and Gauss component, that is, integral breadth Gauss component wG_{SSD} for the detector system, is given by

$$\mathrm{w}G_{SSD}(E) = (0.1880959 + 0.0011924 \times E)^{1/2} \quad . \tag{5}$$

The relationship between the energy *E* of diffraction X-ray and the Gauss component wG_{SSD} is shown in Fig. 10. The instrumental function is obtained by the convolutions of the breadth introduced by the detector and the divergence angle of the optical system [8]. Therefore, the Gauss component β_G arising from non-uniform strain of crystal is calculated from the integral width Gauss component *wG* of the diffracted X-ray profile. Non-uniform strain distributions at applied strains of 1% and 5% are calculated by Eq. (3), and they are shown in Fig. 11. And, the dislocation density distributions calculated by the equation (4) are shown in Fig. 12. Since both of the non-uniform strain and the dislocation density at applied strain of 5% are large in the wide measuring range compared with these values at 1% applied strain, it shows that they increase because ductile damage progresses. And, those values near the notch are slightly small relatively. It expects that the release of the strain energy by the activity of a slip plane with the progress of ductile damage has occurred more often near the notch. However, in the state where subgrains are arising and rotating, it is not in a pure single-crystal state. Therefore, the assumption for the proposed equation which calculates the instrument function may not be realized. Accordingly, the instrument function will estimate small and it expects that dislocation density is evaluated more largely. The calculation procedure of the instrument function when subgrains generate has to be reexamined.

Although specimen differs, the optical microscope photographs of the surface near the notch at applied strain of 5% in the same load condition is shown in Fig. 13. Many slip bands are generated especially along the direction of Al[111] in the line B. Since the development of slip band leads to a release of local strain energy, the non-uniform strain and the dislocation density therefore might decrease locally.

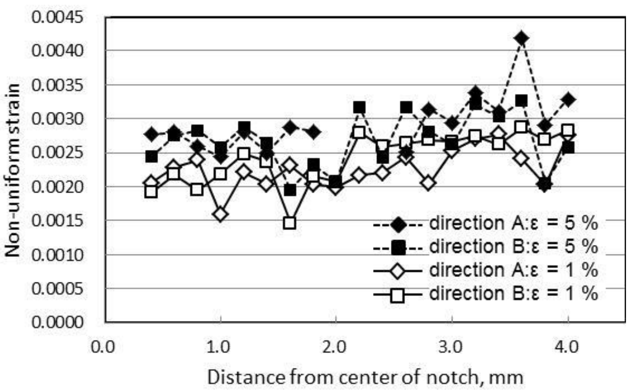


Fig.11 Distribution of non-uniform strain calculated by using Al220 diffraction profile.

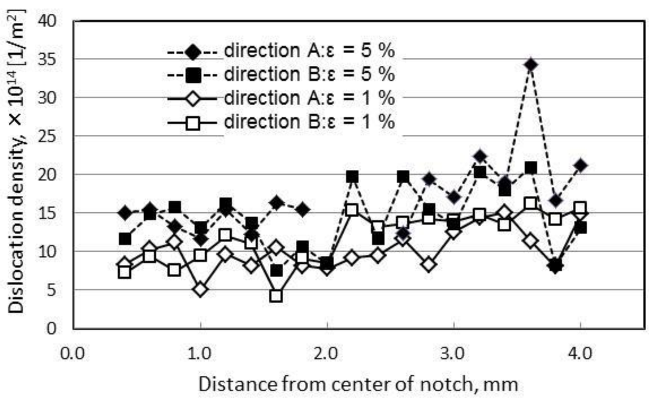


Fig.12 Distribution of dislocation density calculated by using Al 220 diffraction profile.

Conclusion

Verification of the ductile damage progress behavior of an aluminum single crystal with the prior activity of the single slip system under tensile loading was performed by using white X-ray in BL28B2 beam-line installed in synchrotron radiation facilities SPring-8. A non-uniform strain and dislocation density were calculated from the integral breadth Gauss component obtained from X-ray diffraction profile of Al220 plane, and ductile damage progress behavior was examined based on those values. The main conclusions are as follows.

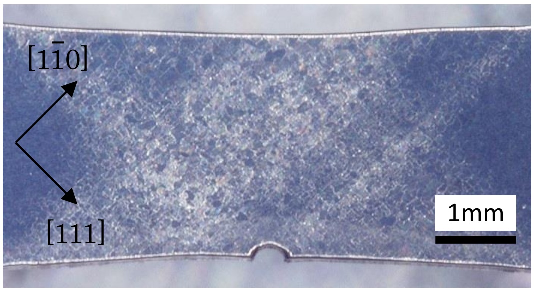


Fig.13 Distribution of slip band near notch of aluminum single crystal at applied strains of ε= 5%.

(1) Subgrains were generated and rotated near the notch with ductile damage progress. The angle variations in the vertical plane are very large compared with them in a horizontal plane.
(2) Dislocation density increased as ductile damage progressed, but it was relatively low near the notch. The influence of release of the strain energy by ductile damage progress was expected.

Acknowledgement

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