Study on Ductile Damage Progress of Aluminum Single Crystal using Synchrotron White X-ray

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Abstract. A ductile damage progress of FCC single crystal was verified by a profile analysis using white X-ray obtained in BL28B2 beam line of SPring-8. In this study, an 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, which has 100 microns in height and 200 microns in 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 along crystal orientation [001], and a diffraction profile of the white X-ray which penetrated it 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 direction of progress and the characteristics of ductile damage near the notch of the aluminum single crystal were clarified from the Gauss component and the Cauchy component of integral width of the single diffraction profile.

Introduction

It was shown clearly by the numerical analysis of our previous research [1] that the ductile damage progress in FCC crystal is dependent on crystal orientation. In order to verify experimentally the relation between a progress of ductile damage and a crystal orientation, the X-ray diffraction measurement with a single crystal is valid. The relation between the material damage by plastic deformation and the form of a diffracted X-ray profile is well known, and many researches have been carried out [2-8]. In order to obtain the information about non-uniform strain and crystallite size from the single diffracted X-ray profile of the material which produced a plastic deformation, an instrumental function peculiar to a measurement system must be eliminated from the diffracted X-ray profile. The instrumental function is generally determined by measuring a powder sample whose crystal grain diameter is about 10-20 microns. Although it seems that this method is effective in measurement of a polycrystalline material, it cannot use for the determination of an instrumental function in measurement of a single-crystal material. Since the diffraction direction is random in a powder sample, the influence of the divergence angle by the optical system of the detector side appears in an instrumental function. An instrumental function can be defined if there is a single crystal in which non-uniform strain and a dislocation does not exist at all. However, it is difficult to obtain such a perfect sample.

Authors are developing the procedure of measuring an internal strain of material by the energy dispersive method using a high energy white X-ray obtained in synchrotron radiation facility

SPring-8 [9]. In measurement using white X-ray, a semiconductor detector (SSD) and a pulse height analyzer (MCA) are used. SSD detects the energy of X-rays. SSD is calibrated by measuring the energy of a radioisotope or fluorescent X-ray. Although an energy spectrum of radioisotope is mono-energetic, the energy spectrum measured indicates the Gaussian distribution. Such broadening occurs by a response function with regards to the energy resolution by SSD, MCA and electronic circuits. On the contrary, by measuring mono-energetic radiations with various energy values, the response function peculiar to a measurement system can be defined as a function of energy. Furthermore, in measurement of the diffraction X-ray of a single crystal, the following three factors also influence broadening of a diffracted X-ray profile. They are a divergence of the synchrotron radiation itself, a divergence by the slits of incidence and detected side, and an arrangement error of a sample. By folding these in a previous response function, the instrumental function peculiar to the measurement system can be defined. Therefore, in the diffracted X-ray profile analysis of a single crystal, it is not necessary to measure the standard sample for defining a*n* instrumental function. It is thought with this point that the energy dispersive method by white X-ray is advantageous for the profile analysis of single crystal.

In this study, the instrumental function in consideration of broadening by both the response function peculiar to an energy dispersive method and the divergence angle was defined. Based on the function, the method to obtain the Gauss component related to non-uniform strain and the Cauchy component related to crystallite size from an integral width of diffracted X-ray profile was proposed. In the BL28B2 beam line of synchrotron radiation facility SPring-8, the aluminum single crystal was deformed to the plastic region by tensile loading. The validity of this method was examined by evaluating the distributions of the Gauss component and the Cauchy component from the integral width of diffraction profile of white X-ray which penetrate the inside of the single crystal.

Theory

Broadening of the diffraction profile by detecting device system. A semiconductor detector SSD is used for detection of the diffracted white X-ray. The X-rays which enter into SSD generate an electron and positive-hole pair proportional to the energy, and are changed into an electronic pulse with the height proportional to the number. The number of generation of the electron and positive-hole is subjected to the influence of statistical fluctuation as characteristics of SSD. The statistical fluctuation is expressed by the Gaussian type response function [10] given by

$$R\left(E_n, E_n^p\right) = \frac{1}{\sqrt{2\pi}\hat{s}\left(E_n^p\right)} \exp\left[\frac{\left(E_n - E_n^p\right)}{2\hat{s}\left(E_n^p\right)^2}\right]. \tag{1}$$

This equation shows the probability that the X-rays of the energy E_n^P will be measured as what has the energy of E_n. $\hat{s}$ of Eq. (1) is a standard deviation of Gaussian distribution, and is expressed by Eq. (2):

$$\hat{s}\left(E_n^p\right) = \frac{\Delta E_{FWHM}(E_n^P)}{2\sqrt{2ln2}}, \tag{2}$$

where ΔE_{FWHM} is a full width at half maximum (FWHM) of the measurement profile of monoenergetic X-ray of E_n^P, and it is expressed by Eq. (3) [11]:

$$\Delta E_{FWHM}(E_n^P) = \left[\Delta E_{amp}^2 + \left\{2.355\left(F_f \beta E_n^P\right)^{1/2}\right\}^2\right]^{1/2}, \tag{3}$$

where ΔE_{amp} is a decrease amount of the resolution in an electronic circuit including a

preamplifier. F_f is a Fano factor which is about 0.1. β is the generation energy of electron and positive-hole pair of the semiconductor used for SSD, and, in the case of germanium Ge, it is 2.8 [eV]. ΔE_{FWHM} is convertible for the integral width by the following relationship:

$$wG_{SSD}(E_n^P) = \frac{1}{2}\sqrt{\frac{\pi}{ln2}}\Delta E_{FWHM}(E_n^P)\,. \tag{4}$$

Breadth of the diffraction profile by angle of divergence. When incident X-ray has a divergence angle, a diffracted X-ray from a single crystal will also have the same angle of divergence by the Bragg law. Therefore, in a single crystal, it is expected by the energy dispersive method that broadening of diffraction angle becomes almost equivalent to the divergence angle of incidence X-ray. Here, if the slit of width *b* mm is installed in the location of *L* mm from the luminous source of width *a* mm, the horizontal angle of divergence of the synchrotron radiation after slit passage is given by

$$\Delta\theta = 2\,tan^{-1}\left(\frac{a+b}{2L}\right)\,[deg]\,. \tag{5}$$

By substituting Eq. (5) for the equation (6) related to the Bragg law in the energy dispersive method, Eq. (7) can be defined as an integral width Gauss component of the instrumental function by the divergence.

$$\frac{\Delta E_D}{E_n^P} = cot\,\theta_B \cdot \Delta\theta\,. \tag{6}$$

$$wG_D(E_n^P) = \Delta E_D = cot\,\theta_B \cdot \Delta\theta \cdot E_n^P\,. \tag{7}$$

Instrumental function. An instrumental function is considered to be a convolution of the breadth by the detector and the divergence angle of optical system, and the Gauss component of integral width is given by

$$wG_0 = \left(wG_{SSD}{}^2 + wG_D{}^2\right)^{1/2}\,. \tag{8}$$

Therefore, the Gauss component β_G only influenced by a non-uniform strain is obtained by Eq. (9) from the integral width Gauss component *wG* of a diffracted X-ray profile.

$$\beta_G = (wG^2 - wG_0^2)^{1/2}\,. \tag{9}$$

Since the Cauchy component β_C of integral width of a diffracted X-ray profile is affected with a crystallite size, it is considered that the component of instrumental function is zero. Then, the Cauchy component *wC* obtained by a measurement of single crystal specimen is directly set to β_C.

$$\beta_C = wC. \tag{10}$$

In order to separate the Gauss component *wG* and the Cauchy component *wC* of integral width from a diffracted X-ray profile, profile fitting was performed with the Voigt function.

Experiment Procedure

Specimen. The aluminum single crystal of the purity 6N (about 99.9999% of purity) was used as a specimen material. The specimen which was coincided crystal orientation [001] in the direction of

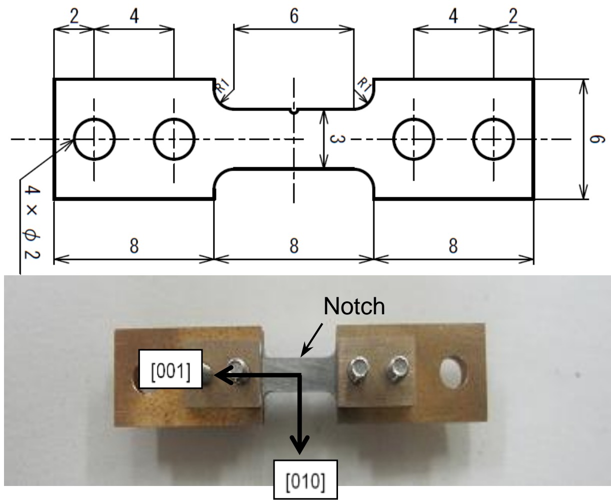

Fig.1 Specimen configuration for tensile test.

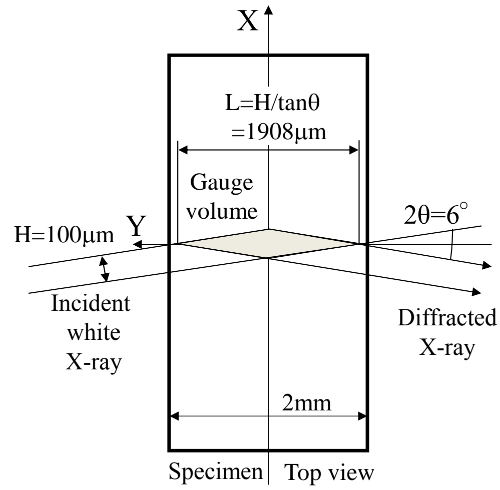

Fig.2 Schematic diagram of gauge volume using transmission diffracted X-ray.

tension was cut down with the low speed diamond cutter from the disk of the aluminum single crystal. I-shape flat specimen for tensile test as shown in Fig. 1 was produced by milling. Furthermore, the notch (width is 0.32 mm and the depth is 0.15 mm) was introduced into one side of the center of a parallel part by wire electric discharge machining.

Measurement facility and conditions. The BL28B2 beam line installed in synchrotron radiation facility SPring-8 was used for measurement. SSD was installed in the position of 6 degrees of diffraction angles in the horizontal plane. Both sizes of the slit installed in the irradiation and detector side were 200 micrometers in height, and 100 micrometers in width. The lattice strain was measured by the transmission type white X-ray method. The gauge volume of this measurement is shown in Fig. 2. The strain of each lattice plane was measured applying ±0.25-degree oscillation about the Y-axis in Fig. 2. The slit of incident side in the 2nd hatch of BL28B2 beam line is in the location of 43.5 m from a source of light with a size of 300 micrometers. Then, L=43500 mm, a=0.3 mm and b=0.1 mm are substituted for Eq. (5) based on a measurement condition and this experimental device arrangement, the horizontal angle of divergence of the synchrotron radiation is equal to

$$\Delta\theta = 5.269 \times 10^{-4}\ [\text{deg.}] = 9.195 \times 10^{-6}\ [\text{rad}]. \tag{11}$$

Results and Discussion

Tensile test and measuring point. An example of the transmission diffracted X-ray profile of a specimen is shown in Fig. 3. In relation to aluminum [001] crystal orientation, the peaks of each

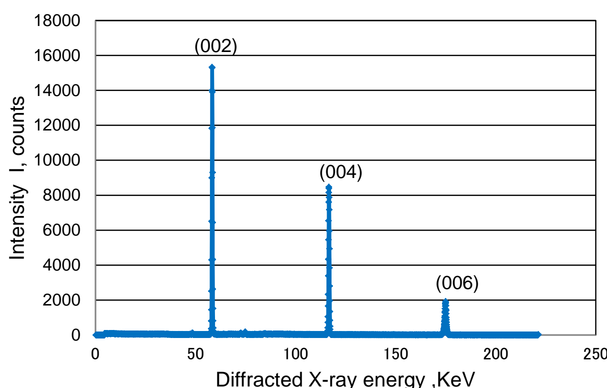

Fig.3 Diffracted X-ray profile of Al single crystal by using white X-ray.

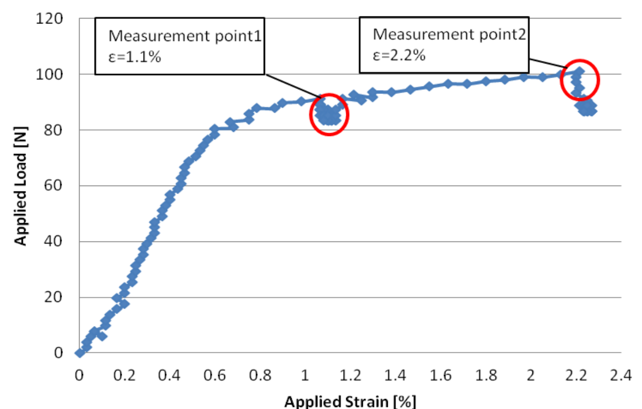

Fig.4 Measurement points of one-side notched specimen.

diffraction plane of (002), (004), and (006) were measured. The load-strain diagram of the specimen is shown in Fig. 4 when the strain rate was set to about 3.1x10^{-6}/s. As shown in Fig. 4, measurement near the notch was performed when the applied strains were 1.1% (measurement point 1) and 2.2% (measurement point 2). In the measurement point 1, the 3 mm wide and 1.2 mm long area near notch was divided at intervals of 0.1 mm wide and 0.2 mm long, and measurement of a total of 217 points was performed. In the point of measurement 2, the 1.5 mm wide and 1.2 mm long area near notch was divided at intervals of 0.1 mm wide and 0.2 mm long, and measurement of a total of 112 points was performed. During the measurement, the load was stopped and the distance between grips was held. Each point of measurement was measured in 100 seconds per point.

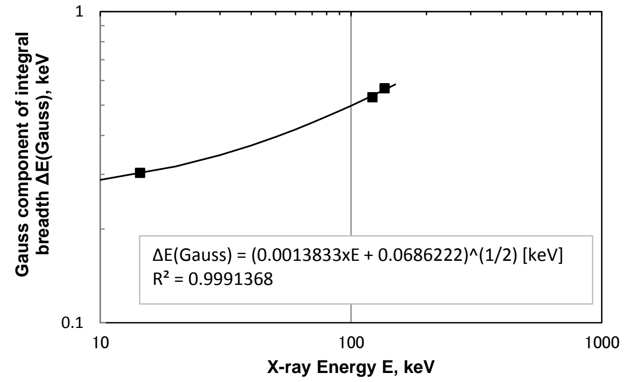

Fig.5 Gauss component of integral breadth of response function in the measurement system.

Distribution of lattice strain and integral width each component near the notch. Fitting of the measurement profile of radioisotope Co-57 was carried out by the Gaussian curve, and the Gauss component of integral width was determined. The relation between the energy of diffraction X-ray and a Gauss component was approximated by the least-square method using Eq. (4), and the Gauss component wG_{SSD} of integral width of the instrumental function is then calculated by

$$wG_{SSD}(E_n^P) = (0.0686222 + 0.0013833 \times E)^{1/2}\,. \tag{12}$$

The relation between the energy E of diffraction X-ray and the Gauss component wG_{SSD} using Eq. (12) is shown in Fig. 5. Distribution of the lattice strain of aluminum (002) in the measurement point-1 (ε = 1.1%) is shown in Fig. 6. Strain is increasing near the notch in the direction of about 45 degrees of slant. Distributions of the Gauss component β_G and the Cauchy component β_C of the integral width of a diffraction profile are shown in Fig. 7 and Fig. 8, respectively. In Fig. 7, the Gauss component as well as lattice strain is increasing in the direction of about 45 degrees of slant near the notch. Since a Gauss component is subjected to the influence of accumulation of non-uniform strain or dislocation, it is considered that ductile damage is progressing in this direction. In Fig. 8, distribution of the Cauchy component does not indicate a characteristic change. Since the Cauchy component will generally be detected if crystallite size becomes below sub-micron order, it is expected that a specimen is still a single crystal or comparatively large sub-grain generated in it.

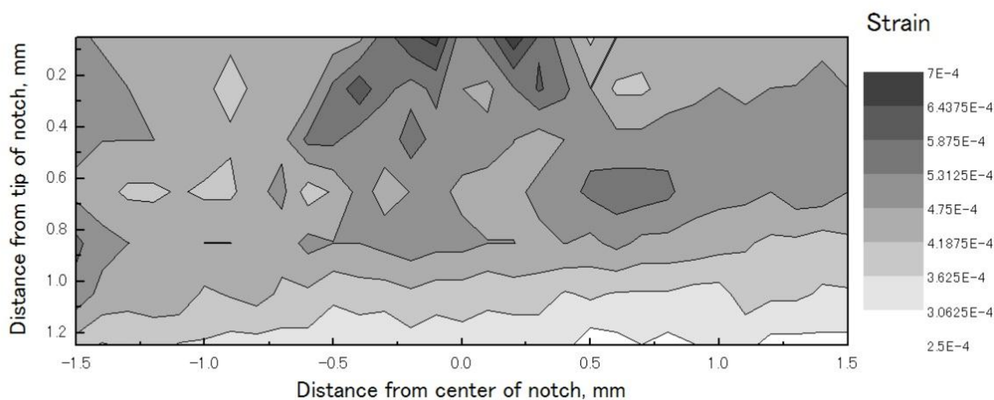

Fig.6 Distribution of lattice strain of Al(002) near notch at measurement point1 (ε=1.1%).

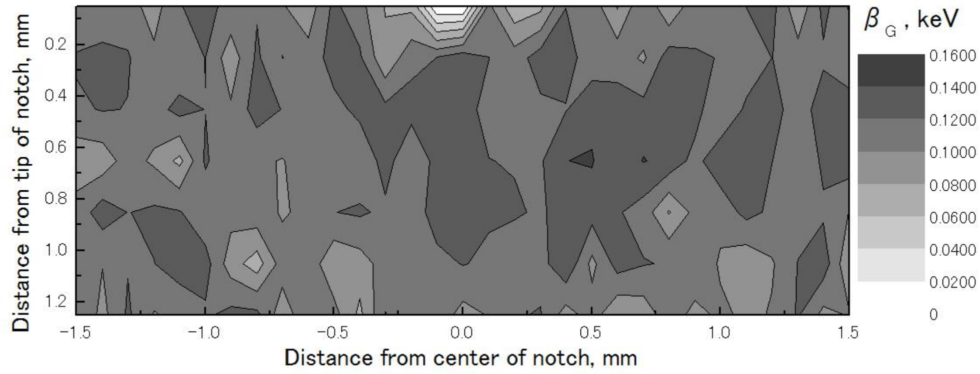

Fig.7 Gauss component β_G of integral breadth of Al(002) diffraction profile near notch at measurement point1 (ε=1.1%).

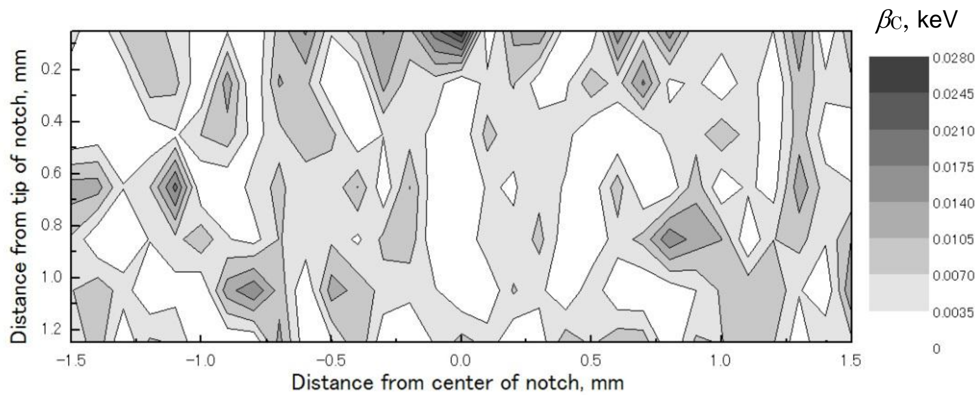

Fig. 8 Cauchy component β_C of integral breadth of Al(002) diffraction profile near notch at measurement point1 (ε=1.1%).

Distribution of the lattice strain of aluminum (002) in the measurement point-2 (ε = 2.2%) is shown in Fig. 9. The area where a lattice strain is small appeared in the direction of about 45 degrees of slant near the notch. In Fig. 10, the Gauss component β_G is also reducing along the reduction area of the strain of the direction of about 45 degrees of slant near the notch. On the other hand, the Cauchy component β_L shown in Fig. 11 is increasing along the reduction area of the strain. This result suggested generating of sub-grain in the progress direction of ductility damage. When the applied strain was 1.1%, slip bands ware already generated in the direction of about 45 degrees of slant near the notch. When the applied strain was 2.2%, much more slip bands ware generated. A development of slip band leads to release of strain energy locally. Therefore, it is considered that decreases of both lattice strain and the Gauss component related to non-uniform strain and dislocation density caused by the release of strain energy.

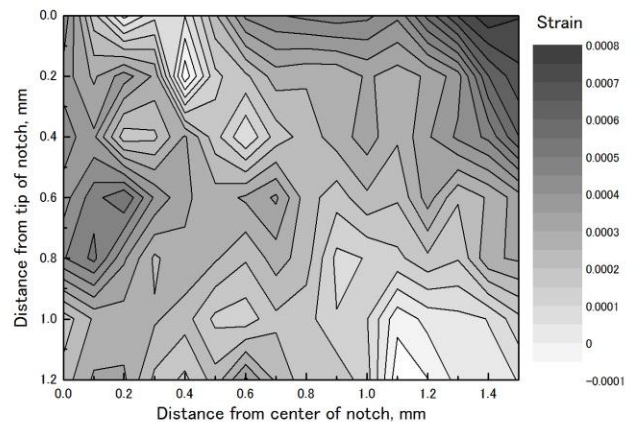

Fig.9 Distribution of lattice strain of Al(002) near notch at measurement point 2 (ε=2.2%).

Conclusion

By the profile analysis using white X-ray, the ductile damage progress of the FCC single crystal was verified. In profile analysis, the instrumental function in consideration of the breadth by an angle of divergence related to

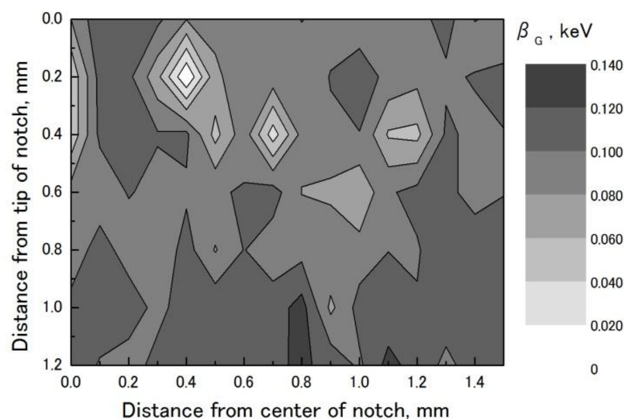

Fig.10 Distribution of Gauss component β_G of integral breadth of Al(002) diffraction profile near notch at measurement point2 (ε=2.2%).

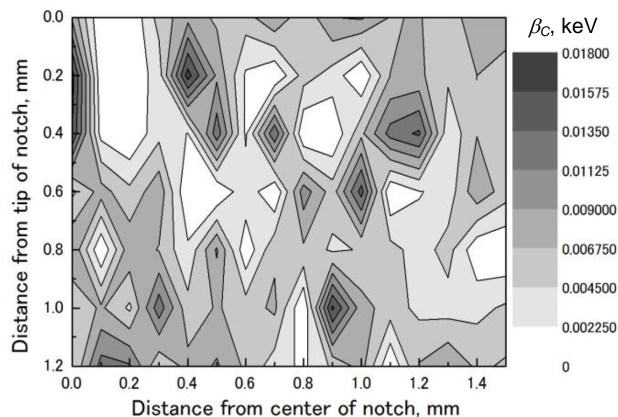

Fig.11 Distribution of Cauchy component β_C of integral breadth of Al(002) diffraction profile near notch at measurement point2 (ε=2.2%).

optical system and a response function peculiar to an energy dispersive method was defined, and the procedure of determining the Gauss component and the Cauchy component of integral width from a diffracted X-ray profile was proposed. In the BL28B2 beam line installed in synchrotron radiation facilities SPring-8, the ductile damage progress behavior of the aluminum single crystal which has crystal orientation [001] along the tensile direction was investigated using synchrotron radiation white X-ray. As a result, in 1.1% of applied strain, an increase of the lattice strain was observed in the direction of about 45 degrees of slant from the notch bottom. The Gauss component of the integral breadth also showed the same tendency, and the Cauchy component of the integral breadth hardly changed. In 2.2% of applied strain, the lattice strain reduced in the direction of about 45 degrees of slant from the notch bottom. The Gauss component of the integral breadth decreased similarly. It is considered that decreases of both lattice strain and the Gauss component related to a non-uniform strain and a dislocation density caused by the release of strain energy due to ductile damage progress. On the other hand, the Cauchy component β_L is increasing along the reduction area of the strain. This result suggested generating of sub-grain in the progress direction of ductility damage. The characteristics of the ductile damage progress near the notch of the aluminum single crystal which has crystal orientation [001] along the tensile direction was confirmed by the proposed method.

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