Transmission Imaging and Strain Mapping in the Vicinity of Internal Crack Tip Using Synchrotron White X-Ray

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TRANSMISSION IMAGING AND STRAIN MAPPING IN THE VICINITY OF INTERNAL CRACK TIP USING SYNCHROTRON WHITE X-RAY

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Abstract. A transmission imaging and a strain mapping in the vicinity of a crack tip in steel were investigated using a high energy white X-ray obtained from BL28B2 beam line at SPring-8 in Japan. Low-alloy and high-tensile steel was used as a specimen prepared in the G-type geometry with a rectangular sectional part of 5mm thickness for a four-point bending. A fatigue crack was introduced into the notch root on the tension side of the specimen by a pulsating bending load. The imaging of the crack in the specimen under the bending load was carried out by using the CCD camera that can detect indirectly the X-ray transmitted through the specimen. To measure the internal strain in the vicinity of the crack tip, the synchrotron white X-ray beam, which had a height of 80 \( \mu \)m and a width of 300 \( \mu \)m, was incident on the specimen with the Bragg angle \( \theta \) of 5 degrees using the energy dispersive X-ray diffraction technique. As the results, the transmitted image of the crack showed that the crack in the specimen was propagated deeper than that on the surface. The map of the internal strain near the crack tip could be obtained using the white X-ray with energy ranging from 50keV to 150keV. It became clear by the numerical simulation that the FWHM of diffracted X-ray profile measured near the crack tip was increased due to the steep change in the strain distribution. It was confirmed that the synchrotron white X-ray was useful for the imaging of the internal crack and the strain mapping near it.

Introduction

To clarify the local internal strain conditions in the vicinity of a fatigue crack tip is necessary to understand a growth mechanism of the fatigue crack. Therefore, the measurements of the internal stress/strain near the crack tip of a material have been performed using a synchrotron radiation [1-2]. In our previous work, a fundamental study to measure the internal strain of a low-alloy and high-tensile steel using a high energy white X-ray obtained from synchrotron radiation was carried out at SPring-8. As a result, the internal strain of the steel of 5, 10 and 15 mm thicknesses could be evaluated using the white X-ray which range of energy from 50keV to 150keV [3]. In order to measure the strain near the internal crack tip, it is necessary to detect correctly its position in the material. The aims of the present study are therefore to clarify the crack tip inside material by using a transmission imaging and then to measure the strain distribution near the estimated crack tip in the
steel specimen of 5 mm thickness using the high energy white X-ray obtained from the public beam line BL28B2 at SPring-8.

**Experimental procedure**

**Specimen.** A low-alloy and high-tensile steel (JIS G3128 SHY685) was used as a specimen prepared in the G-type geometry with a rectangular sectional part of 5mm thickness for a four-point bending as shown in Fig. 1. The notch of 0.33mm in width and 0.3mm in depth on the center of parallel part of the specimen was worked by the wire electric discharge machining. Residual stress of the specimen due to a series of working was removed by the annealing which is done at 540°C for one hour followed by slow furnace cooling under the argon gas atmosphere. Young's modulus, Poisson's ratio and the yield stress of the specimen obtained from a tensile test were 200GPa, 0.29 and 779MPa, respectively. A fatigue crack was introduced into the notch root of the specimen by the pulsating bending load of 25.2Nm with the sine wave having a frequency of 5Hz. The fatigue crack of length about 1mm from the notch root on the surface of the specimen was produced after the number of cycles of 123461, as shown in Fig. 2.

**Imaging of internal crack.** A transmission imaging of the internal crack of the specimen carried out at the public beam line BL28B2 in Super Photon Ring-8 (SPring-8). The air-cooling CCD camera with 1000 by 1024 picture elements in the photoreceptor (Hamamatsu Photonics C4880-10-14A) and the beam monitor (Hamamatsu Photonics AA40P) were used for the imaging. A resolution of photographic image obtained with the camera is 5.83 micrometers. The CCD camera was mounted in a rear direction about 530mm of the specimen, and detected indirectly the X-ray transmitted through the specimen of 5mm thickness. The transmitted image near the crack of the specimen applied with the bending load of about 25.2Nm was exposed.

**Strain measurement near the crack tip.** The experimental setup of strain measurement using high energy white X-ray at BL28B2 is shown in Fig. 3. For the strain measurement, X-axis on the notch root center of the specimen was coincided with the optical axis of synchrotron radiation. The Ge-type Solid State Detector (SSD) was fixed at the diffraction angle 2θ = 10 degrees in the X-Y plane. A multi-channel analyzer (MCA) with 4096 channels was used for the discrimination of the diffracted X-ray energy detected by an energy dispersive technique. To determine the energy calibration equation in this experiment, fluorescence X-rays of Pb-Kα1 (74.9694keV) and Pb-Kα2 (72.8042keV) from a lead material and a radioisotope of Co-57 (122keV) were measured, then it was obtained as Eq.1,

$$E_n = 0.0539xCH+0.7243 \quad [\text{keV}] ,$$  \hspace{1cm} (1)

where, CH is a channel number of MCA. The energy range per channel was therefore 53.9eV, and the discrimination of energy to about 221 keV was possible at 4096 channels.
Figure 4 shows the schematic diagram of cross section of the gauge volume by using the transmission diffracted X-ray. The strain in only orthogonal direction to the crack face was measured because of the limitation of our machine time. The strain measurement was performed under the bending load of about 25.2Nm. Applied load was verified by the strain gauge cemented on the compressive stress side of the specimen, and it was adjusted so that it might be set to -1550x10^{-6} as strain value. The stress intensity factor $K_I$ in the mode I of the specimen was about 22.67MPa m^{1/2} under the load condition. The measurement areas near the crack therefore were set up as shown in Fig. 5. 112 points in 1500 x 1500 micrometers area in the Y-Z section (at $X = 0$) were measured. The irradiated time of the synchrotron white X-ray was set to 600 seconds per measured point. The width of both divergence and receiving slits was 300\(\mu\)m, and the height of divergence slit was 100\(\mu\)m. The height of receiving slit was set to 80\(\mu\)m. The measurement strain therefore becomes an average value in a very long and slender parallelepiped volume with length of 3.44mm, width of 0.3mm and height of 0.08mm. The number of crystal grains in this gauge volume was about 6300.

The strain $\varepsilon$ is given by Eq. 2 using the energy dispersive method.

$$\varepsilon = \frac{\Delta E}{E} = \frac{E_{n0} - E_n}{E_n},$$

where $E_{n0}$ and $E_n$ are the diffracted X-ray energies detected from a non-strained and a strained specimen, respectively. In this experiment, $E_{n0}$ was obtained by measuring the same material treated with the stress relief annealing.

Results and discussion

Image of internal crack. Transmission image of the internal fatigue crack in X-Y section (at $Z = 0$) of the specimen is shown in Fig. 5. The crack length estimated by the transmission imaging was about 1.03mm, which was longer than it observed at the surface. It was confirmed that the fatigue crack propagated deeply in the steel specimen.

Strain and FWHM distributions near the internal crack. The diffracted X-ray profile of the specimen used in this experiment is shown in Fig. 6. Although a background noise was a little high, the intensity of each diffraction peak was sufficiently strong to measure. The peak energy of the diffracted X-ray was calculated by approximating the diffraction profile to the Gaussian curve.

Figure 7 shows the strain $\varepsilon_y$ mapping near the internal crack tip calculated by using the $\alpha$-Fe321 plane data, and Fig. 8 shows the strain $\varepsilon_y$ distribution along the crack growth direction (at $Y=0$). It was found that the large tensile strain occurred at the position of about 1.3mm from the notch root. The distribution indicates well the feature of the strain distribution in the vicinity of the crack tip in
the specimen under loading. However, the compressive strain was distributed near the crack tip position estimated by the transmission imaging. The compressive strain is produced usually by the compressive residual stress developed after the crack propagation. Therefore, the accurate length of internal crack tip is probably longer than 1.03mm obtained from the transmission image as shown in Fig. 5.

Figure 9 shows the full width at half maximum (FWHM) distributions of $\alpha$-Fe321 plane near the internal crack tip, and Fig. 10 shows the FWHM distribution along the crack growth direction (at Y=0). The peak position of FWHM was about 1.2mm from notch root, and this position was different from it of strain $\varepsilon_y$ distribution. Furthermore, its value was relatively larger than that with increasing the plastic strain. It can be considered that the steep change of strain near the crack tip has an effect on FWHM. Hence, the change of diffraction profile with strain distribution was carried out as shown below. The diffracted beam spectrum of white X-ray is generally detected by a solid-state detector (SSD). When the X-rays irradiate the SSD, electron-hole pairs are generated in proportion to the absorbed energy. These are converted into a voltage pulse. The voltage-pulse distribution has a statistical fluctuation and when the SSD is used, the statistical fluctuation is given as the response function [4], which is equal to the Gaussian distribution of Eq. 3,
The response function of Eq. 3 gives the probability of energy $E_p^i$ of an X-ray with regard to energy $E_n$. $\delta(E_p^i)$ is the standard deviation of the Gaussian distribution, which is obtained from the FWHM of the profile by measuring a single energy spectrum, as Eq. 4

$$\delta(E_p^i) = \frac{\Delta E_{FWHM}(E_p^i)}{2\sqrt{2\ln 2}}.$$ (4)

$\Delta E_{FWHM}(E_p^i)$ is the energy resolution [5], and the value is expressed by

$$\Delta E_{FWHM}(E_p^i) = \left[ \Delta E_{amp}^2 + \left( \frac{2.355(F\beta E_p^i)^{1/2}}{\Delta E} \right)^2 \right]^{1/2},$$ (5)

where $\Delta E_{amp}$ is due to the dark current through the SSD and to the noise in a field-effect transistor and a preamplifier, $F$ is the Fano factor (approximately 0.1), $\beta$ is the energy required for creating an electron/positive- hole pair (2.8eV for Ge semiconductor), and $E_p^i$ is the incident X-ray energy. Therefore, the diffracted beam distribution of white X-ray passing through the SSD [6] is given by

$$M(E_n) = \int_{E_n - \Delta E/2}^{E_n + \Delta E/2} R(E_n, E_p^i) I(E_p^i) dE_p^i.$$ (6)

Voltage pulses generated by the SSD reach the MCA through amplifiers. The amplitude of a voltage pulse is converted into a digital quantity by an analog-to-digital converter, which is counted to the channel corresponding to the pulse-amplitude range is represented by $\Delta E$, the final counts recorded on a channel $i$ is given as

$$M_i = \int_{E_i - \Delta E/2}^{E_i + \Delta E/2} M(E_n) dE_n.$$ (7)

When $I(E_p^i)$ is the single energy spectrum with the diffraction intensity of $I$, Eq. (7) can be approximated to Eq. (8).

$$M_i = I \times R(E_n, E_p^i).$$ (8)

The numerical simulation of the diffraction profile of $\alpha$-Fe321 plane under non-strained condition is shown in Fig. 11, where the diffracted X-ray energy $E_n^p$ set to 92.840keV for the diffraction angle 20 of 10 degrees, the diffraction intensity of $I$ and the $\Delta E_{amp}$ are assumed to be 1.0 and 0.0, respectively. Furthermore, when the measurement area contains both the strained and the non-strained region near the crack tip, the diffraction profile is simulated as shown in Fig. 12, where the X-ray energy diffracted at $\alpha$-Fe321 plane in the strained region set to 92.478keV corresponding.
to a strained crystal under the yield condition. The FWHM in Fig. 12 clearly shows an increase as compared with it in Fig. 11. For that reason, it is difficult to evaluate accurately the correlation between the FWHM and the plastic strain introduced in the vicinity of the crack tip.

Summary
A transmission imaging and a strain mapping in the vicinity of a crack tip in steel were investigated using a high energy white X-ray obtained from BL28B2 beam line at SPRing-8. As a result, the crack in the specimen of 5mm thickness under the bending load could be observed by the transmission imaging using the high energy white X-ray. The large tensile strain could be estimated near the internal crack tip. It became clear by the numerical simulation that the FWHM of diffracted X-ray profile measured near the crack tip is increased due to the steep change in the strain distribution. Therefore, it was found that the accurate evaluation of the correlation between the FWHM and the plastic strain introduced in the vicinity of the crack tip is difficult.

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