Evaluation of Fatigue Damage by Diffraction Contrast Tomography Using Synchrotron Radiation

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Abstract
The three dimensional grain mapping technique for polycrystalline material, that is termed X-ray diffraction contrast tomography (DCT) has been proposed. In the present study, the measurement of DCT was conducted in SPring-8 and the condition of measurement and data procedure were discussed. Developed technique was applied to aluminium alloy and stainless steel. The shape and location of grain could be determined by the developed three-dimensional mapping technique using the apparatus in SPring-8, BL19B2 bending beam line. Next, for evaluation of plastic deformation, the internal grain orientation spread of the individual grain was counted. The grain orientation spread is caused by the mosaicity, which related to the change of microstructure. The grain orientation spread increased with increased tension stress in the plastic deformation region. It is possible to evaluate fatigue damage in microstructure, such as crack initiation, by the DCT technique and using the grain orientation spread as one of the fatigue damage parameters.

Keywords: Synchrotron radiation, Imaging, Diffraction contrast tomography, Plastic deformation, Fatigue damage

1. Introduction
The three-dimensional grain mapping techniques for polycrystalline materials have attracted a growing interest. The new real space mapping techniques, such as ‘three-dimensional X-ray diffraction microscopy’ (3DXRD) [1] and differential aperture X-ray microscopy’ (DAXM) [2], based on the diffraction of synchrotron beam, have been proposed. These non-destructive bulk mapping techniques were used to analyze microstructural changes related to deformation and annealing processes in metallic samples. The other different data acquisition strategy, aiming at simultaneous reconstruction of the absorption and grain microstructure of a material, has been proposed [3]-[5]. The procedure is termed X-ray diffraction contrast tomography (DCT), which is similar to conventional X-ray absorption contrast tomography. DCT can share a common experimental setup with conventional X-ray CT. Projected images of grain are obtained using the occasionally occurring diffraction contribution to the X-ray attenuation coefficient each time a grain fulfils the diffraction condition. The three-dimensional grain shapes are reconstructed from these projections. The DCT can provide simultaneous access to the sample’s three dimensional grain arrangement (shapes and crystallographic orientations) and microstructural features visible in X-ray absorption contrast (cracks, porosity etc.).

The development of the DCT technique has been centered mainly on the European Synchrotron Radiation Facility (ESRF). In the present study, the measurement conditions and image processing for three-dimensional grain mapping in SPring-8 were discussed. Two types of DCT data acquisition strategy, termed the direct beam case[4] and combined case[5], are applied. In the direct beam case, the projection image of direct beam area is used for data analysis. In the data set, the amount of spot overlap on the direct beam area leads to the breakdown of reconstruction of grain shape. Combined case was modified from direct beam case, and the diffraction spot on the outside direct beam area is also used. Next, the information about the grain structure could be obtained from the diffraction condition during the measurement of DCT. Therefore, the change of grain structure due to static load and cyclic loading can be observed by DCT. The developed DCT technique was applied to the
evaluation of plastic deformation of individual grains in a specimen in tensile test and in fatigue test.

2. Experimental procedure of DCT

2.1 Principle of DCT

The apparatus used for diffraction contrast tomography is identical to a conventional microtomographic imaging setup, with only two moving elements – the rotation of the sample (ω) and the translation of the sample out of the beam to record reference images of the incoming beam profile, as shown in Fig. 1. During acquisition of an optimized tomographic scan (ω), undeformed grains embedded in the bulk of a polycrystalline sample give rise to distinct diffraction contrasts which can be observed in the transmitted beam each time a grain fulfills the Bragg diffraction condition. By extracting and sorting these contrasts into groups belonging to individual grains, it is possible to reconstruct the three-dimensional grain shapes by parallel beam geometry algebraic reconstruction techniques. As in direct-beam only case, the grains are imaged the extinction contrast that is occasionally observed in the transmitted beam when the grains are aligned in the diffraction condition. In the combined case, variant extinction spot is replaced to the corresponding diffraction spot on the same detector, which is set closely behind the sample. Once the diffraction / extinction spot pairs have been assigned to a grain, its crystallographic orientation can be calculated.

Fig. 1 An overview of the acquisition geometry of DCT, showing the synchrotron beam, the sample, and both direct and diffraction images on the detector.

2.2 Materials and specimen

To investigate the measurement conditions and develop the image processing for DCT technique, the sample of aluminum alloy Al 1050 (99%Al), which is a wire rod of 1mm in the diameter, is prepared. In order to produce a coarse-grained microstructure, the aluminium alloy sample receives a recrystallization annealing treatment for 1h at 723K. Next, for evaluating the plastic deformation by the developed DCT technique, austenitic stainless steel (JIS type SUS304) and industrial pure iron are employed. SUS304 stainless steel and pure iron receive a recrystallization annealing treatment for 30 minutes at 1300K and 2 hours at 1373K, respectively. The average grain size was 71µm for SUS304 steel, and 120µm for pure iron. After the heat treatment, 0.2% proof stress was 255MPa for SUS304 steel and 184 MPa for pure iron. The geometry of sample is shown in Fig. 2. The sample of SUS304 steel has rectangular bar with 0.3mm in width and 0.3mm in thickness. That of pure iron is a wire rod of 0.3mm in the diameter, and tab plates are attached at the end of wire for applying static loading and cyclic loading.
2.3 Experimental setup

The synchrotron experiment is performed at BL19B2 beam line of SPring-8, the brightest synchrotron radiation facility in Japan. This beam line delivers an X-ray beam with high brightness and high spatial coherence, allowing to perform micro-tomography with high spatial resolution in the micro-meter range [7] [8]. The polychromatic synchrotron beam is monochromated to 28keV for aluminium alloy sample and 37keV for SUS304 steel and pure iron sample using a Si(111) double-crystal monochromator, and two-dimensional projection images are recorded on a high-resolution detector system based on a transparent luminescent screen, light optics and a CCD camera. The sample to detector distance is 10mm for the aluminium alloy and 20mm for SUS304 steel and pure iron, respectively, and effective pixel size of 2.7µm is chosen for this experiment. The beam at the sample position is limited to 1.0mm × 1.0mm by means of X-ray slits. Projection image is obtained every 0.032° over 180° for aluminium alloy, and every 0.04° over 180° for SUS304 steel and pure iron, respectively.

To evaluate the plastic deformation and fatigue damage inside the grain, the static tensional loading and cyclic loading is applied to a specimen. The automatic tensile testing machine, which consists of load cell and linear electric actuator, was developed as shown in Fig. 3. It can be placed on the sample rotation stage for tomography scan. In static tensional test, the sample is subjected to tension stress, which increases from elastic to plastic deformation, such as 0MPa, 100MPa, 270MPa and 380MPa for SUS304 steel, and 70MPa, 160MPa and 220MPa for pure iron, respectively. Tensional stress is applied to the sample during the data acquisition. In fatigue tests, cyclic partially pulsating loading is applied to the specimen by the tensile testing machine. Fatigue test is carried out under displacement control condition, and total displacement range in this experiment is set to be 65mm (total strain range ∆ε is
1.05%) for pure iron. The fatigue test is interrupted and DCT measurement is conducted at certain number of cycles.

### 2.4 Data procedure

#### 2.4.1 Removal of the absorption

One example of projection image is shown in Fig. 4. One extinction spot appeared in the direct beam area, and the corresponding diffraction spot having same shape to extinction spot was observed outside direct beam. In the first step several background removal are applied to the raw images. The processing for the direct beam area in the center of an image consists of the standard flat field correction, two-dimensional median filtering for noise reduction, and removal of the absorption background. The absorption background is calculated by application of a one-dimensional median filter to a stack of projection images, which are built from equally spaced images centered around the current projection and covering an adequate angular range. By calculating pixel by pixel the one-dimensional median value along the stack, diffraction events can be rejected and obtain a good estimate of the absorption background. Fig. 5(a) shows one of the direct beam areas in projection image for which one of the large grain happens to fulfil the Bragg condition. The absorption background (Fig. 5(b)) is obtained by the one-dimensional median filter applied to a stack of 50 projection images. The extinction spot, corresponding to a projection of the grain volume, can be obtained, as shown in Fig. 5(c). For the area outside direct beam, the standard flat field correction is applied and diffraction spots are detected by threshold processing.

![Figure 4](image1.png)

**Fig. 4** Projection image with an extinction spot (black) and paired with their corresponding diffraction spot.

![Figure 5](image2.png)

**Fig. 5** Removal of the absorption background, (a) Projection image, (b) corresponding absorption background, (c) extinction spot obtained by logarithmic subtraction of the images to the left.

#### 2.4.2 Spot summation and segmentation of diffraction and extinction spot

Diffraction and extinction spots can spread over a range of successive images and only part of the diffracting grain may be visible in each of the individual projection images. Therefore the
summation of contributions belonging to the same spot is performed using the three-dimen-

dimensional connected area.

The diffraction and extinction spots are approximately similar in shape and intensity, although
of opposite sign. The extinction spot corresponding to each diffraction spot can be identified
by template matching. The crystallographic orientation of each grain can be calculated from
the location of pairs of diffraction and extinction spot. The information of diffraction spot is
convenient to help the segmentation process of the extinction spots, which often have
considerable overlap. In direct case, extinction spots are used for the reconstruction of grain.
In combine case, diffraction spot is also used for variant extinction spot, and it is replaced to a
corresponding diffraction spot which is detected by template matching.

2.4.3 Sorting of diffraction and extinction spot

The segmented extinction spots are sorted into sets belonging to the same grain. The spot
sorting can be accomplished by means of the following two filtering steps. The extinction
spot is a projection of the grain and hence is invariant during rotation around the vertical axis.
The top and bottom vertical limits of the extinction spots belonging to the same grain are
employed as the criterion. At second step, center lines of the thresholded spots are
backprojected into the sample plane, taking into account the respective rotation angles. Fig.
6(a) shows the two-dimensional back-projection of the center line of the extinction spots. The
intersection point of lines by back-projecting of the extinction spot projections defines an
approximate possible grain position.

The set of extinction/diffraction spot projections are normalized with respect to integrated
intensity. The tomographic reconstruct of grain was solved for each grain, using a standard,
parallel beam geometry algebraic reconstruction algorithm (ART) [6]. Fig. 6(b) show the
reconstructed two dimensional grain volume from the grain set extinction spot projections in
Fig. 6(a). By stacking the reconstructed two dimensional slices, the corresponding three
dimensional grain volumes can be assembled, as shown in Fig. 6(c).

3. Results and discussions

3.1 Grain mapping

The results of reconstruction of grain for sample of aluminium alloy and stainless steel are
shown in Fig. 7 and 8, respectively. The individual grain sub-volumes are segmented, labeled
(color coded). Grains for aluminium ally were calculated by direct case, and that for stainless
steel were obtained by combine case. The shape and location of grain for both materials can
be determined by three-dimensional mapping technique using the apparatus in BL19B2
bending beam line. For stainless steel, some variant extinction spots were observed, and the number of extinction spots used for the reconstruction in combine case is larger than that in direct case. Combine case is useful for sample where many grains align in diameter direction. Average grain size measured from DCT shown in Fig. 8(c) is 72.2 µm. this value coincides with that measured from metallographic observation (71 µm).

![Image](image1.png)

Fig. 7 Three-dimensional reconstruction of grains of aluminum alloy sample. (a) top view, (b) side view

![Image](image2.png)

Fig. 8 Three-dimensional reconstruction of grains of austenitic stainless steel sample.

### 3.2 Evaluation of plastic deformation

#### 3.2.1 Static tensional tests

For evaluation of plastic deformation, the grain orientation spread of the individual grain was counted. As mentioned previously, an individual diffraction spot may extend angularly from extinction spot location. Individual diffraction spots that are visible give some measure of the orientation spread within grains. The grain orientation spread could be caused by the mosaicity (i.e. sub-grain misorientation) or the curvature of grain caused by misorientation, which is related to the damage of the microstructure in materials.

The set of images of diffraction spot, which is observed at same rotation angle ω and could belong to the same grain, is shown in Fig. 9. The contrast of diffraction spot for σ=380MPa is smaller than that for σ=0MPa, on the other hand, the shape of the summation of diffraction spots for σ=0MPa is globally in good agreement with that for σ=380MPa, and then the grain
orientation spread $\Delta \omega$ increases from $0.40^\circ$ to $0.95^\circ$. The diffraction spots for several tensional stresses are counted. The histogram of grain orientation spread for stainless steel is shown in Fig. 10. 0.2% proof stress was 255MPa for SUS304 steel. It is found from Fig. 10 that the histogram for $\sigma=100$MPa is almost identical to that for the 0MPa. In the plastic deformation region, the mode value of the grain orientation spread increases with tension stress. Histogram of grain orientation spread for pure iron is shown in Fig. 11. 0.2% proof stress of pure iron is 184MPa. It is found that the tendency of histogram for pure iron is good agreement with that for stainless steel. In elastic deformation region, histogram for 70MPa is almost identical to that for 160MPa. In plastic deformation region, the mode value of $\Delta \omega$ for $\sigma=220$MPa become to be large.

(a) 0 MPa
(b) 0 MPa

Fig. 10 Histogram of grain orientation spread for stainless steel subjected to static tension

Fig. 11 Histogram of grain orientation spread for stainless steel subjected to static tension

3.2.2 **Cyclic loading fatigue tests**

Histogram of grain spread orientation for pure iron subjected to cyclic loading is shown in Fig 12. It is found from Fig. 12 that mode value of grain spread orientation stayed unchanged against the number of cycles. Frequency of diffraction spot at mode value ($\Delta \omega = 0.3$)
becomes smaller after cyclic loading, and diffraction spot, having large grain orientation spread, increases compared with initial value. It is considered that the successive plastic deformation in cyclic loading develops in a particular grain or diffraction plane in the sample.

The diffraction spots which belong to certain grain are sort by diffraction plane and its grain orientation spread is measured. The change of grain orientation spread of certain grain for each diffraction plane is shown in Fig. 13. Plane shown in Fig. 13 indicates crystallographically equivalent plane. There is no change in plane \{211\}, and grain orientation spread of plane \{200\} increases slightly by 0.16° in a grain. On the other hand, grain orientation spread of plane \{110\} increase significantly by 0.2~0.6° during cyclic loading. Change of grain orientation spread is observed mainly on plane \{110\}. Plane \{110\} is one of slip plane for the material having body-centered cubic lattice (BCC), such as pure iron.

![Fig. 12](image12.png)

**Fig. 12** Histogram of grain orientation spread for pure iron subjected to cyclic loading

![Fig. 13](image13.png)

**Fig. 13** Change of grain orientation spread for pure iron subjected to cyclic loading.

### 3.2.3 Discussion
Grain orientation spread is observed inside grain during sample rotation when diffraction condition is fulfilled, so that grain orientation spread could be equivalent to full width at half maximum (FWHM) of rocking curve. FWHM is related to the amount of plastic strain[9],[10]. In static tensional test, grain orientation spread increases with increasing of applied stress in plastic region. In fatigue tests, it is found that change of grain orientation spread occurs in a particular grain and diffraction plane. Diffraction plane, where the most change of grain orientation spread is observed, coincides with the slip plane of pure iron. It could be considered that grain orientation spread reflected change of grain structure, especially the slip behaviour.
The number of grain orientation spread in the static tensional tests is more than that in fatigue test. High stress in the static tensional test induces the movement of many slip planes on grains; on the other hand, low stress in the fatigue test induces the movement of primary slip system on particular grains. Therefore, it is possible to identify the grain of crack initiation by evaluating the change of grain orientation spread. The visualization of change of grain orientation spread is shown in Fig. 14. Grains are color-coded according to amount of grain orientation spread change. Red indicates the grain having amount of grain orientation spread change above 0.56°. It is found that the grains shown by red are located near the surface. The growth of slip band and crack initiation occurs on the surface of material. It could be considered that this visualization of change of grain orientation spread shows accumulation of fatigue damage, and DCT technique provide the evaluation method for fatigue damage.

4. Conclusion

The measurements of DCT were conducted in SPring-8 and the condition of measurement and data procedure were discussed. Developed technique was applied to aluminium alloy, stainless steel and industrial pure iron. It was found that the shape and location of grain can be determined by three-dimensional mapping technique (direct beam method and combine method) using the apparatus in SPring-8, BL19B2 bending beam line. For evaluation of plastic deformation, the internal grain orientation spread of the individual grain was counted in the static tensional test and fatigue test. The grain orientation spread increased with an increase in the tension stress in the plastic deformation region in static tensional test. In fatigue test, grain orientation spread increased on the particular grain and diffraction plane which coincides with slip plane. It is found that fatigue damage in microstructure, such as crack initiation, can be evaluated by the DCT technique and the grain orientation spread is one of the effective fatigue damage parameters.

References

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