Non-destructive and three-dimensional measurement of local strain development during tensile deformation in an aluminium alloy

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Abstract. Anisotropy of mechanical responses depending on crystallographic orientation causes inhomogeneous deformation on the mesoscopic scale (grain size scale). Investigation of the local plastic strain development is important for discussing recrystallization mechanisms, because the sites with higher local plastic strain may act as potential nucleation sites for recrystallization. Recently, high-resolution X-ray tomography, which is non-destructive inspection method, has been utilized for observation of the materials structure. In synchrotron radiation X-ray tomography, more than 10,000 microstructural features, like precipitates, dispersions, compounds and hydrogen pores, can be observed in aluminium alloys. We have proposed employing these microstructural features as marker gauges to measure local strains, and then have developed a method to calculate the three-dimensional strain distribution by tracking the microstructural features. In this study, we report the development of local plastic strain as a function of the grain microstructure in an aluminium alloy by means of this three-dimensional strain measurement technique. Strongly heterogeneous strain development was observed during tensile loading to 30%. In other words, some parts of the sample deform little whereas another deforms a lot. However, strain in the whole specimen was keeping harmony. Comparing the microstructure with the strain concentration that is obtained by this method has a potential to reveal potential nucleation sites of recrystallization.

1. Introduction
Metallic materials consist of a large number of individual grains which are separated by grain boundaries. The grains have crystallographic orientations. Depending on the crystallographic orientation, deformation behaviors can vary between grains. This deformation anisotropy causes concentration of deformation, i.e. local deformation variations on a mesoscopic scale (the grain size scale). The development of local plastic strain during a deformation process is one of the key issues to describe mechanical properties, such as formability and plastic anisotropy [1]. The local concentration of strain may also govern fracture. Furthermore, with regard to thermo-mechanical processing, investigations of the local plastic strain development are important for discussing recrystallization mechanisms, because the sites with higher local plastic strain seem to stimulate nucleation of recrystallization [2].
Recently, high-resolution X-ray tomography, which is a non-destructive inspection method, has been used for observations of material structures. The spatial resolution of current tomography techniques has been reduced to 1 \( \mu \text{m} \), which is close to the limit of the resolution in a visible-light-conversion type detectors. By X-ray tomography using a monochromatic X-ray beam from synchrotron radiation facilities, more than 10,000 microstructural features, like precipitates, dispersions, compounds and hydrogen pores, can e.g. be observed in aluminium alloys. We have proposed employing these microstructural features as markers to measure local strains, and have developed a method to calculate the three-dimensional strain by tracking the microstructural features \[3\]. In this study, we report research on the development of local plastic strains affected by the grain microstructure in an aluminium alloy by this method.

2. Experimental method
An in-situ tensile experiment during tomography scans were performed at BL20XU at the Japanese synchrotron radiation facility, SPring-8. A small special test rig was installed at BL20XU. First, the specimen was scanned without loading. The tensile load was given to the specimen step by step. The tomography scans were done at each step. 6 time scans were achieved up to a maximum load in this study. Table 1 explains the loading steps and macroscopic strain. The macroscopic tensile strain was approximately 30\% at the final step (Step 6). Note that the tensile displacement was controlled and a strain rate of 0.001 mm/s was used. After finishing the tensile test, the deformed specimen was removed from the test rig, and then the grain boundaries were visualized by means of a gallium application. Details of the sample and of the experimental method are given in the following.

| Loading step | Load (N) | Macroscopic strain (%) |
|--------------|----------|------------------------|
| 0 (undeformed) | 0        | 0.0                    |
| 1            | 67.9     | 3.2                    |
| 2            | 82.3     | 7.1                    |
| 3            | 91.5     | 11.4                   |
| 4            | 96.2     | 16.9                   |
| 5            | 97.9     | 23.3                   |
| 6            | 97.2     | 29.6                   |

2.1 Sample
The sample used in this study is an Al-4mass\%Cu alloy that contains Al\(_2\)Cu precipitate particles employed as tracking markers to measure local plastic strains. To obtain both a relatively large grain size and a lot of Al\(_2\)Cu particles, a thermo-mechanical treatment was applied as shown in Fig. 1. A cast ingot of Al-4mass\%Cu was manufactured by melting and casting base-materials of 3N pure aluminium and Al-33mass\%Cu alloy. The ingot was cut into small billets and homogenized at 823 K for 36 ks with water cooling. The billets were processed with 80\% cold rolling and heat-treated at 503 K for 7.2 ks as an intermediate annealing. Further 10\% additional rolling was given to the intermediate annealed sample. The samples were then recrystallized at 803 K for 45 s in a salt-bath. After recrystallization, the samples were solution-heat-treated at 768 K for 18 ks and were then cooled for 900 s in a muffle furnace to obtain a dense population of precipitate particles on the grain boundaries. With this thermo-mechanical treatment, the average grain size and particle size were approximately 100 \( \mu \text{m} \) and 2.9 \( \mu \text{m} \), respectively. Small specimens for tensile tests with cross section of 0.6 mm by 0.6 mm in the gauge (see Fig. 2) were cut from the sample by electro-discharge machining.

2.2 Synchrotron X-ray tomography
X-ray tomography was carried out at the experimental hutch at BL20XU. The set-up used in this study was the projection type microtomography as shown in Fig. 3 that possess a spatial resolution of
approximately 1 μm. Monochromatic X-ray with 20 keV energy was obtained by a (111)-(111) Si double crystal monochromater. The test rig was mounted on a high-precision rotation stage. A visible-light conversion type detector was used consisting of a scintillator (Lu₂SiO₅:Ce⁺), ×10 optics lens and with a complementary metal oxide semiconductor (CMOS) camera (Hamamatsu photonics ORCA-Flash 2.8 1920×1440 pixels). A voxel size of (0.5 μm)³ was achieved by this set-up. The sample to detector distance was adjusted to approximately 20 mm to make the object edge sharp by X-ray phase contrast enhancement. One thousand eight hundred radiographs with height and width of approximately 720 μm and 960 μm were captured during a 180 degree rotation (i.e. 0.1 degree step). The convolution back projection method was utilized for reconstruction. The linear absorption coefficient (LAC) was stored in the reconstructed slice image as a 16-bit grey depth. To reduce the image data size, a LAC value from 0 cm⁻¹ to 31 cm⁻¹ was converted into 8-bit grey depth (i.e. from 0 to 255). Note that LAC of aluminium, Al₂Cu particles were 8.56 cm⁻¹ and 84.86 cm⁻¹ at 20 keV, respectively, i.e. widely different.

![Diagram of thermo-mechanical processing for the sample preparation in this study](image1)

**Figure 1.** Diagram of thermo-mechanical processing for the sample preparation in this study

![Dimensions of the tensile specimen. (mm)](image2)

**Figure 2.** Dimensions of the tensile specimen.

![X-ray microtomography set-up at BL20XU](image3)

**Figure 3.** X-ray microtomography set-up at BL20XU

2.3 Visualization of grain boundaries

After finishing the X-ray tomography scans, the deformed specimen was removed from the test rig. The grain boundary network was visualized by applying gallium onto the specimen. It is well known that gallium with the low melting point, can penetrate aluminium grain boundaries easily and cause intergranular fracture because of liquid metal embrittlement. The LAC of gallium (239.27 cm⁻¹) is significantly higher than that of aluminium (8.56 cm⁻¹) at 20 keV. Therefore, gallium penetrated grain boundaries are seen as bright contrast within the aluminium matrix. Gallium was melted at 323 K on a
hot-plate. The specimen was dipped into the liquid gallium and then the sample surface was scratched lightly by the tip of a pair of tweezers until gallium penetrates into the sample. The specimen was mounted in a specimen holder on a rotation stage of the X-ray instrument and was scanned again. The distance between the sample and detector was set to 20 mm to allow appropriate phase contrast. The LAC values of the images obtained by the gallium application were converted into 8-bit grey depth in the range from 0 cm$^{-1}$ to 63 cm$^{-1}$. Further, images were binarized applying a threshold of 80 to determine the grain boundary positions correctly. Binarized objects with a volume under 3$^3$ voxel were deleted in binarized image, and then the distance transformation [4] and H-minima transformation [5] was applied to the binarized and modified images. $H = 8$ was used for the H-minima transformation in this study. A Watershed transformation [5] which is one of the segmentation methods was finally used. It was found that some grains were hyper fractionated by the Watershed transformation. Therefore, all results of the grain segmentation were checked, and were corrected by merging these grains into one. The three-dimensional volume rendered image after the Watershed transformation is shown in Fig. 4. 503 grains exist in the field of view. The average grain diameter of these grains is 64.5 $\mu$m. The numbers of grain boundaries, edges that form triple junction lines and vertices that form quadrupole points were 2871, 4416 and 1784, respectively. Details of this processing have been reported in ref [6].

![Figure 4. Three-dimensional image showing the grains in the sample](image)

2.4. Strain calculation
A series of X-ray tomography images measured during tensile loading were binarized with a threshold value of 147. The positions (gravity centres), volumes and surface areas of all particles were measured in the binarized images by a homemade software. To remove uncertain markers, only particles with a volume larger than 3$^3$ voxel were accepted as tracking markers for the local plastic strain measurement. Since the specimen moved slightly during tensile loading, the marker positions in all volume images of different loading steps were converted into the load step 0 (before deformation) using an Affine transformation to make tracking easy. After conversion, markers were tracked by using a matching parameter method [3]. The position, surface area and volume were considered as matching parameters, $M_p$. The weighting of these were 0.65, 0.3 and 0.05, respectively. When The matching parameter of a marker is larger than the first threshold value, $M_p^{th1}$ and the difference between the largest $M_p$ and 2nd largest $M_p$ is larger than the second threshold value, $M_p^{th2}$, those markers were matched. Approximately twenty thousand markers were tracked through all the loading steps. A Delaunay tessellation [7] was performed to make tetrahedra within the 3D space of the sample by utilizing the positions of the tracked markers. Here, we assume a linear displacement field inside the tetrahedra and displacements $u_i, v_i, w_i, u_j, v_j, w_j, u_k, v_k, w_k, u_l, v_l, w_l$ at tetrahedra vertices $i, j, k, l$ in a rectangular coordinate system, then we can calculate normal strains, $\varepsilon_x, \varepsilon_y, \varepsilon_z$ and shear strains, $\gamma_{xy}, \gamma_{xz}, \gamma_{yz}$, by the following equations;
where \( \mathbf{B} \) is given as
\[
\mathbf{B} = \frac{1}{6V} \begin{bmatrix}
    o_i & 0 & 0 \\
    0 & p_i & 0 \\
    0 & 0 & q_i \\
    p_i & o_i & 0 \\
    0 & q_i & p_i \\
    q_i & 0 & o_i \\
\end{bmatrix},
\]
\( o_i = y_k z_j + y_j z_k - y_j z_l - y_l z_k + y_l z_j \\
 p_i = x_j z_j + x_j z_k + x_k z_l - x_l z_k - x_k z_j \\
 q_i = x_l y_k + x_l y_l + x_k y_j - x_j y_k - x_k y_j - x_l y_k \\
\]
where \( x_j, y_j, z_j, x_k, y_k, z_k, x_l, y_l, z_l \) indicate the vertex positions of \( j, k, l \). Shifting indices such as \( i \rightarrow j, j \rightarrow k, k \rightarrow l, \) and \( l \rightarrow i \) brings \( \mathbf{B}_j, \mathbf{B}_k, \mathbf{B}_l \). The variable \( V \) means the tetrahedron volume. The calculated strains were arranged at the gravity center positions of the tetrahedra. Three dimensional discrete strain data were converted into volume image data with a 5 \( \mu \)m voxel size by linear interpolation to make comparison with the grain boundary images easy.

Table 2. Marker tracking result.

| Loading step | Number of tracked markers | Average of local strain (%) |
|--------------|---------------------------|-----------------------------|
| 0-1          | 41278                     | 3.2                         |
| 0-2          | 34877                     | 7.5                         |
| 0-3          | 32246                     | 12.0                        |
| 0-4          | 24762                     | 17.9                        |
| 0-5          | 23271                     | 25.9                        |
| 0-6          | 19512                     | 33.2                        |

3. Results and discussions

Results of strain mapping at a macroscopic tensile strains of 3.2% (loading step 1) and 29.6% (loading step 6) are shown in Fig. 5. The strain along the tensile direction, \( \varepsilon_z \), the other normal strain, \( \varepsilon_y \) and the shear strain, \( \gamma_{yz} \) are indicated in Fig. 5(a), (b) and (c), respectively. Some limited strain concentrations are seen at 3.2% strain. A very heterogeneous strain distribution is observed in the entire strain map at 29.6%. Regarding with the evolution of \( \varepsilon_z \), the positions at which the strain concentration occurred at an early stage of deformation are found to develop a large deformation in the later loading step. Spots in which a large strain occurs thus seem to be defined in the early stage of deformation. The undeformed domains at the early strain step did not deform preferentially at later steps. Deformation spots did not propagate widely in the present case when the final macroscopic strain was approximately 30%. The grains that show large deformation may have a lower work hardening rate compared with other grains. In the map of the \( \varepsilon_y \) strain, compression strains can be confirmed as shown in Fig. 5 (b'). The compression strain, \( \varepsilon_k \) in Fig. 5(b') have a tendency to be large at the positions where large tensile strains are observed in Fig. 5 (a'). The strain distribution of \( \varepsilon_y \) seems to roughly correspond to that of \( \varepsilon_z \).

The shear strain, \( \gamma_{yz} \) in Fig. 5 (c') are seen not to be of the same magnitude as the \( \varepsilon_z \) strain. The direction of shear deformation is different in individual grains. Although we have not measured the
crystallographic orientation of each grain, the dependence of deformation behaviour, like the deformation magnitude and shear direction is likely to depend in the grain orientation which may be determined by the non-destructive orientation measurement method, three dimensional X-ray diffraction [8]. The area having large shear strains are seen to propagate into low shear strain areas with increasing macroscopic tensile strain. This development of shear strains is different from that of the normal strain.

The distributions of local strains are shown in Fig. 6. Each strain distribution figure in Fig. 6 corresponds to the strain maps in Fig. 5. In the initial stage of tensile deformation, the distribution of local strains is narrow. Note that the distributions contain a measurement error due to the limitation of tomography resolution: because the average marker distance was 19.4 μm; if the spatial resolution of synchrotron radiation tomography is 1 μm, then the minimum measurable strain will be 5% (=1/19.4×100).

With increasing macroscopic tensile deformation, the distribution of local strains along the tensile direction shift towards positive (tensile) values and becomes wider gradually. In the final loading step 6 of 29.6%, the right tail of the distribution becomes long as shown in Fig. 6 (a'). In contrast, the bell-shaped distribution is kept in the εx strain (Fig. 6 (b')), a long left tail is observed in εy strain distribution. For the shear strain, the three components, γyz, γzx and γxy show a similar tendency. The maximum in the local shear strain did not change with tensile deformation. However, the widths increase with increasing macroscopic tensile deformation. The width of distribution in shear strains became wider than that of εx at the last step.

![Local strain maps obtained by marker tracking in X-ray tomography.](image)

**Figure 5.** Local strain maps obtained by marker tracking in X-ray tomography. (a) and (a') show the εz strain which is along with tensile direction at 3.2% and 29.6% tensile deformation, respectively. (b) and (b') are the εy strain distribution at 3.2% and 29.6% tensile deformation, respectively. (c) and (c') show the shear strains γyz at 3.2% and 29.6% tensile deformation, respectively.
Figure 6. Distributions of local strains. Each figure correspond to the strain maps in Fig. 5.

Figure 7. Changes in the average of local strain and standard deviations in the measured strain distributions. The six strain components (three normal strains and three shear strains) are plotted.

The averages of the local strains and standard deviations are plotted in Fig. 7. The dashed line shows when the local strain equals the macroscopic strain in the plot of $\varepsilon_x$ (Fig. 7 (c)). A dashed line is also inserted in the plot of $\varepsilon_x$ and $\varepsilon_y$, here it is assumed that $\varepsilon_x=\varepsilon_y=0.5\varepsilon$, this is a valid assumption if the deformation causes no volume change and is isotropic in $x$ and $y$. As seen in Fig. 7(c), the average of local strain, $\varepsilon_z$ corresponds well with the macroscopic strain, $\varepsilon$. This supports that the result of local strain measurement is reasonable. However, there are clear differences between $\varepsilon_x$ (Fig. 7(a)) and $\varepsilon_y$ (Fig.
(b)) and the macroscopic expectations. This could be due to the crystallographic texture of the sample. Although, some deviations from the dashed line are confirmed in all normal strain components, $\varepsilon_x$, $\varepsilon_y$ and $\varepsilon_z$, the volume should not change. The volume change, $\Delta V=(1+\varepsilon_x)(1+\varepsilon_y)(1+\varepsilon_z)-1$ was checked for each loading step. The measured volume changes were less than 0.15% at all loading steps. Thus, the volume change was quite small. The standard deviation is largest for the $\varepsilon_z$ strain. The standard deviation in $\varepsilon_x$ and $\varepsilon_y$ are similar and approximately half of that of $\varepsilon_z$ at all strains. The averages of the local shear strains are close to zero for all shear strain components, $\gamma_{yz}$, $\gamma_{zx}$ and $\gamma_{xy}$ at all macroscopic strains. Therefore, although the local spatial distribution of the shear strains are very heterogeneous (see Fig. 5(c)), the average remains close to zero.

Local strains in the sample is very heterogeneously distributed spatially, the stored energy will therefore also vary significantly. It is suggested that the variations of local strains are considered when the aim is to pinpoint nucleation sites and understand nucleation mechanisms. Using the present method (without the gallium step) followed by annealing to study recrystallization is planned for the future.

4. Conclusions
The development of local strain distributions has been investigated in an aluminium alloy during tensile deformation by means of a three-dimensional strain measurement technique based on X-ray tomography. Strongly heterogeneous strain development was observed after 30% tensile loading. The normal strain components continued to develop at localized sites in the sample in which large strains occur already in the early stage of deformation. The development of shear strains was different to that of the normal strains. The area having a large shear strain propagated into low shear strain areas with increasing macroscopic tensile strain. Comparing deformation microstructures and local strain concentrations, obtained by this method, may be a powerful method to reveal nucleation positions of recrystallization.

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