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Full length article

Relationships between 3D grain structure and local inhomogeneous deformation: A laboratory-based multimodal X-ray tomography investigation

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ABSTRACT

Relationships between the three-dimensional (3D) grain structure and the local strain distribution within individual grains are investigated. Multimodal X-ray tomography, i.e. attenuation tomography (ACT) and diffraction contrast tomography (LabDCT), was applied, for the first time using a laboratory instrument, to non-destructively characterize the grain structure and the local plastic deformation behavior of a fully recrystallized Al-4mass%Cu alloy. The evolution of the internal strain distribution during tensile deformation was measured in-situ by means of a microstructural feature tracking (MFT) method based on ACT. By a correlation analysis of the microstructural parameters of 855 grains, it was investigated if and how the initial crystallographic orientations and sizes of the grains affect the local deformations. In addition, effects of grain boundaries were analyzed. Only weak correlations are found. It is suggested that specific interactions between neighboring grains, which depend on parameters such as grain shapes and orientation differences, are of critical importance for the development of the local strains.

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1. Introduction

It is by now well recognized that significant local variations, and thus severe heterogeneities, exist in both the deformation microstructures and distributions of local strain in deformed polycrystalline metallic materials [1–5]. Although the evolution of deformation microstructures follows a common framework [6], local microstructural variations exist on several scales: Sample scale, grain scale and within the individual grains [7]; and on the latter two scales the crystallographic orientations of the grains are of importance [8]. Similarly, various types of strain measurements have documented local strain variations on all these scales [9–14]. Heterogeneities in the local microstructure and strain distributions in deformed metals are of critical importance for subsequent mechanical processing, for recrystallization and for crack nucleation and growth. It is thus essential to understand the formation of such local heterogeneities and to be able to control their development.

To advance the field, it is considered necessary to combine knowledge about the microstructures and the local strain distributions; not just consider one or the other separately. With that aim already much work has been done combining microstructural characterization with digital image correlation (DIC) [15,16] measurements of the local strains [17,18]. However, such measurements are of course limited to surface investigations, which may not represent the full 3D situation in the bulk of the material.

Motivated by this limitation, much attention has recently been on non-destructive 3D inspection methods. Especially, methods that can image 3D microstructures including different phases, cracks, grain morphologies, crystallographic orientations and local strains, like three-dimensional X-ray Laue microdiffraction [19], three-dimensional X-ray diffraction (3DXRD) [20–22], high energy diffraction microscopy (HEDM) [23,24] and diffraction contrast tomography (DCT) [25,26], have been used for research on
deformation and fracture of polycrystalline metallic materials. For example, 3DXRD has been used to follow grain rotations during straining [27–29] and to examine the relationships between stresses and grain structures in both the elastic and plastic regimes of tensile deformation within austenitic stainless steel [30,31]. As another example, DCT has been used to analyze propagation of short fatigue cracks and to investigate relations between crystallographic slip and fracture surfaces in a metastable beta titanium alloy [32]. So far these studies have focused mainly on the development of local grain misorientations during plastic deformation, based on which the actual heterogeneities of the local plastic strains cannot readily be derived [33].

Several methods exist for determining the local 3D strain distribution by synchrotron X-rays, including digital volume correlation (DVC) [34–36], which is an extension of the DIC methodology, and microstructural feature tracking (MFT) [37–43], which allows faster strain quantifications in larger volumes than DVC. These synchrotron methods are very powerful, and the experiments done so far have already documented interesting and unexpected correlations between microstructure and strain. The work has indeed underpinned the need for many more measurements on a wide range of metals, alloys, deformation modes and strains etc., beyond what would be possible at synchrotron sources.

The aim of this work is to use a laboratory X-ray method for non-destructive 3D characterization of both the initial grain microstructure and the development of local strains by MFT during in-situ tensile deformation of an Al-4mass%Cu alloy. It should be noted that the laboratory method does not allow tracking of the lattice rotations, and hence intragranular misorientation gradients, during the course of the tensile deformation as has previously been done by synchrotron techniques [44–47]. However, laboratory diffraction contrast tomography (LabDCT) [48–50] is used for imaging the grains and their crystallographic orientations in the undeformed state of the sample, after which absorption contrast tomography (ACT) is used to map second phase particles as the basis for MFT local strain measurements during tensile deformation. As hundreds of grains are investigated by this multimodal approach, a statistical as well as a local analysis of the spatial distribution of strains and their developments are done, effects of initial grain orientations as well as grain sizes are quantified and possible strain localizations near grain boundaries are analyzed. The local plastic strain results obtained in this study provide important additional information to the current lattice rotation results for understanding the heterogeneous local plastic deformation behavior.

## 2. Experiments and analysis methods

### 2.1. Material

A small ingot of the Al-4mass%Cu alloy was prepared by gravity casting. To obtain a suitable microstructure for this research, a tailored thermomechanical treatment was applied before machining a tensile specimen (see Fig. 1(a)). A piece cut from the ingot with a size of $30 \times 40 \times 20$ mm$^3$ was homogenized at 823 K for $3.6 \times 10^4$ s (10 h), quenched in water, and cold-rolled to 90% reduction. The cold-rolled sample was heated at 803 K for 45 s in a salt-bath to obtain a fully recrystallized coarse-grained microstructure. Finally, the recrystallized sample was solution treated at 768 K for 1.8 $\times$ 10$^4$ s (5 h) in an electrical furnace and cooled to 727 K in 9.0 $\times$ 10$^3$ s (15 min) in the furnace to obtain precipitated $\alpha_2$Cu particles in the microstructure. After the furnace cooling, the sample was quenched in water. The thermomechanical process brought a weak crystallographic texture, and dispersed particles not only at grain boundaries but also within grain interiors, where the particles had a slight tendency to align along the rolling direction.

A tensile specimen with gauge dimensions of $1.25 \times 100 \times 0.50$ mm$^3$ (Fig. 1(b)) was cut from the sample with the tensile axis along the transverse direction (TD) of the cold rolling.

### 2.2. In-situ tensile experiment

To visualize in 3D the initial grain structure and the positions of the intermetallic particles during in-situ tensile testing, a ZEISS Xradia 520 Versa X-ray micro-CT system equipped with a LabDCT module was used. The tensile specimen was mounted in a tensile rig designed especially for this instrument. First, LabDCT measurements were performed for the undeformed specimen. In total 181 X-ray diffraction images were collected by a 2D detector with an exposure time of 100 s during a 360° specimen rotation for a single LabDCT volume mapping with a voxel size of 4 $\mu$m. Four LabDCT volumes were mapped to cover the whole uniform tensile gauge volume. The detailed conditions of the LabDCT measurement are listed in Table 1.

<table>
<thead>
<tr>
<th>LabDCT</th>
<th>ACT</th>
</tr>
</thead>
<tbody>
<tr>
<td>X-ray tube power</td>
<td>10 W</td>
</tr>
<tr>
<td>Accelerating voltage</td>
<td>110 kV</td>
</tr>
<tr>
<td>Sample-to-source distances</td>
<td>14.5 mm</td>
</tr>
<tr>
<td>Sample-to-detector distances</td>
<td>16 mm</td>
</tr>
<tr>
<td>Aperture size</td>
<td>250 $\times$ 750 $\mu$m$^2$</td>
</tr>
<tr>
<td>Beam-stop size</td>
<td>2.5 $\times$ 2.5 mm$^2$</td>
</tr>
</tbody>
</table>

After the LabDCT scanning, ACT was performed to obtain the spatial distribution of the $\alpha_2$Cu particles, which is utilized for local strain measurement. In total 2401 absorption contrast images (radiographs) were recorded by the 2D detector with an exposure time of 10 s during a 360° sample rotation. The detailed conditions of the ACT measurement are listed in Table 1. The radiographs were reconstructed into a 3D volumetric image, which is a stack of slice images with 16-bit grey levels. The resulting spatial resolution was 1.8 $\mu$m in the reconstructed 3D image.

Load was then employed to the specimen using the tensile rig to different strains at a strain rate of $1.8 \times 10^{-3}$ s$^{-1}$. ACT scans were conducted repeatedly using the same experimental parameters after each strain step, while keeping the tensile deformation at each target strain. The details on the experimental set-up are further explained in reference [51].

The load-displacement curve recorded during the in-situ tensile test is shown in Fig. 2, with inserts showing the specimen appearance at each loading step. The ACT scans were conducted from the initial undeformed state (0th loading step) to a state just before fracture (7th loading step). It should be noted that for the initial undeformed state, two ACT scans with the relative specimen mounting position shifted vertically by 50 $\mu$m were performed to determine the strain uncertainty assuming all strain variations between the two volumes to be measurement uncertainties. Some stress relaxations are observed during the holding periods when the ACT scans were performed, because the tensile deformation was temporarily stopped (see Fig. 2). The material yielded already during the 1st loading step (0.56% tensile deformed) and gradually worked hardened from the 1st to the 5th loading step (23.9% tensile deformed). Necking occurred around the 5th loading step and the sample fractured soon after the 7th loading step (38.4% tensile deformed). Additionally, the fracture surface was also scanned in the 8th step. In this study, focus is however on analyzing the region of homogeneous deformation before necking occurs (up to the 4th loading step).
Fig. 1. (a) Thermomechanical treatment of the Al-4mass%Cu alloy used in this study and (b) Shape of the small specimen designed for in-situ tensile testing.

Fig. 2. Load-displacement curve recorded in the in-situ tensile test. Specimen appearance obtained by ACT in each loading step is shown. Macroscopic tensile strains indicated at each ACT image were measured from the specimen elongation obtained from the ACT images. The grains located in the specimen center, which are used in the analysis, are also presented at 0% deformation as a colored grain map.
2.3. Grain structure of gauge volume

The 3D microstructure before deformation is shown in Fig. 3. The figure reveals that enough Al<sub>2</sub>Cu intermetallic particles are nearly uniformly distributed ensuring a proper strain evaluation, and that the sample consists of almost equiaxed grains. The total number of grains in the characterized gauge volume is 4804, see Fig. 3 (b). The map indicates that grains of similar orientation (similar color) do not cluster spatially. Furthermore, it could be confirmed that the texture of the sample is very weak. The average grain size, defined as the average diameter of the spheres of equivalent volumes, is 50.7 μm. With regard to the Al<sub>2</sub>Cu particles recorded by ACT, their average size and average distance to neighbors are 7.3 μm and 19.7 μm, respectively. This size and the homogeneous spatial distribution (see Fig. 3(a)) made them suitable to utilize as gauge markers to measure local plastic strains.

Only grains in the center of the specimen gauge volume are examined, as a finite element analysis confirmed small stress concentration effects from grips and at the sample surface for the present specimen shape (see section 5.1 in the supplementary material). There are 1281 such interior grains with an average grain size of 47.8 μm in the analysis volume. However, only the grains with at least one strain measurement point (see definition in next section) were analyzed. 855 grains with an average size of 61.0 μm fulfill this criterium. The grains examined in this study are presented in Fig. 2 as a colored grain map next to the 0% (undeformed) ACT volume.

2.4. Local strain mapping

The local strain within the deformed specimen was measured by means of the MFT method [39]. The series of reconstructed ACT images obtained during tensile deformation was analyzed as follows: First, the grey values of the images were rescaled on an 8-bit level, by assigning the average of the air and Al peak values to 0 and 64, respectively. The Al<sub>2</sub>Cu particles in the 8-bit rescaled images were segmented by binarization applying a suitable threshold value of 99. The gravity center positions, volumes and surface areas of the segmented particles were extracted and particles with volumes > 9 voxels were utilized for the local strain measurement in this study. From 14,117 to 24,158 particles were detected at each loading step as listed in Table 2. This difference is due to different image qualities [51].

The particle positions were tracked for each strain increment. At first, the same several tens of relatively large particles were found by manual inspection in two successive strain steps. Then, the spatial distribution of these marker particles in the first of the two successive deformation steps was estimated by virtually deforming the sample by means of a linear interpolation of the displacement vector field in order to simulate the expected particle positions after deformation. This process makes marker matching easy. By considering distance, volume and surface area as matching parameters, all particles (not only the selected ones) in the first and second successive deformation steps were matched, and unreliable particles were eliminated. Here, the automatic matching algorithm reported in reference [39] was applied. The number of particles that were successfully and reliably tracked from the 0th step (undeformed) up to the 4th step (15.6% tensile strain) was 4998 (after manually checking and removing large strain uncertainties due to connected, or almost connected, particles). The average distance between the tracked marker particles was 27.5 μm in the undeformed state.

The local strains were determined by assembling tetrahedrons with the tracked marker particles as vertices. The Delaunay tessellation algorithm [52] was utilized for creating tetrahedrons. Strains of each tetrahedron were solved based on the finite element framework assuming linear displacement fields within tetrahedrons, as follows:

\[
\begin{bmatrix}
\varepsilon_x \\
\varepsilon_y \\
\varepsilon_z \\
\gamma_{xy} \\
\gamma_{xz} \\
\gamma_{yz}
\end{bmatrix}
= \mathbf{B} \begin{bmatrix}
u_i \\
v_i \\
w_i \\
u_j \\
w_j \\
u_k \\
w_k \\
u_l \\
w_l
\end{bmatrix},
\]

\(i = 1, 2, ..., 15\)

Fig. 3. Microstructure of Al-4mass%Cu sample before deformation. (a) Al<sub>2</sub>Cu particles observed by ACT and (b) 3D grain maps obtained by LabDCT. The grain structure is colored according to the inverse pole figure along the tensile direction.
where $e_{x}$, $e_{y}$, $e_{z}$ and $\gamma_{xy}$, $\gamma_{yz}$, $\gamma_{zx}$ are normal and shear strain components, $B$ is the strain-displacement matrix, $u$, $v$ and $w$ are displacements at tetrahedron vertices and the subscripts $i$, $j$, $k$, $l$ of displacements correspond to the four tetrahedron vertices. The $B$ matrix used in this study is $B = [ B_i B_j B_k B_l ]$, where:

$$B_i = \frac{1}{6 V_{\text{tetra}}} \begin{bmatrix} o_i & 0 & 0 \\ 0 & p_i & 0 \\ 0 & 0 & q_i \\ p_i & q_i & o_i \\ q_i & 0 & p_i \end{bmatrix} \tag{2}$$

$$o_i = y_i z_j + y_k z_i - y_j z_k - y_k z_i - y_j z_j \tag{3}$$

$$p_i = x_j z_k + x_k z_j - x_i z_k - x_k z_i - x_i z_j \tag{4}$$

$$q_i = x_k y_j + x_k y_j + x_j y_k - x_j y_k - x_j y_j \tag{5}$$

Here $V_{\text{tetra}}$ is the tetrahedron volume, $x$, $y$ and $z$ with subscripts are the positions of the four tetrahedron vertices and $B_i$, $B_j$, $B_k$ and $B_l$ can be obtained by shifting indices such that $i \rightarrow j$, $j \rightarrow k$, $k \rightarrow l$ and $l \rightarrow i$. The gravity center of the tetrahedron was also calculated and used for the correlation analysis with the grain structure.

A total of 27974 strain measurement points were obtained from the 4998 tracked marker particles. The average number of strain measurement points in the analyzed 835 grains was 7.95. In order to visualize the strain distributions and easily compare these with the 3D grain map (Fig. 3(b)), 3D images with a voxel size of 4 µm, corresponding to the voxel size of the LabDCT grain reconstruction, were also prepared by assigning the strain of a tetrahedron to the voxels belonging to that tetrahedron. These strain data assigned to the voxels were used for the correlation analysis with the grain structure. The details are described in the next section.

The uncertainty of the strain measured by the MFT method was evaluated based on the two vertically shifted undeformed ACT volumes. The average standard deviation of the measured six strain components in the undeformed state was found to be approximately 0.045; for details see reference [51]. It should be noted that the uncertainty is affected by both the image quality (spatial resolution) and the marker distance (tetrahedron size). Considering this uncertainty, we only analyzed the data for applied strains of 8.5% and 15.6%, though the specimen was also scanned at 0.56% and 2.8% deformation during the experiment.

2.5. Local strain data for correlation analysis

To investigate relationships between grain structure and inhomogeneous deformation, the individual grains, which typically contain several strain measurement tetrahedrons, are used as the unit, and all the strains assigned to individual LabDCT voxels within a grain are averaged to determine the grain averaged strain values. This is done for each of the six strain components. Similarly, equivalent strain is also calculated from the six strain components by the following equation:

$$\varepsilon_{\text{equiv}} = \sqrt{\frac{2}{3} \left( e_x^2 + e_y^2 + e_z^2 \right) + \frac{1}{3} \left( \gamma_{xy}^2 + \gamma_{yz}^2 + \gamma_{zx}^2 \right) } \tag{6}$$

and averaged over all the voxels within a grain. Not only the averages, but also the standard deviations are computed, and the average of the standard deviations of the six strain components, $\sigma_{\text{six}}$, can be used as a representative parameter for the magnitude of inhomogeneous deformation within a grain. $\sigma_{\text{six}}$ is calculated as follows;

$$\sigma_{\text{six}} = \frac{1}{6} \left( \sigma_{e_x} + \sigma_{e_y} + \sigma_{e_z} + \sigma_{\gamma_{xy}} + \sigma_{\gamma_{yz}} + \sigma_{\gamma_{zx}} \right) \tag{7}$$

where $\sigma_{e_x}$, $\sigma_{e_y}$, $\sigma_{e_z}$, $\sigma_{\gamma_{xy}}$, $\sigma_{\gamma_{yz}}$, and $\sigma_{\gamma_{zx}}$ are the standard deviations of each of the six strain components in one grain. The analysis based on strains obtained by this averaging method are called "strain based analysis", as relationships between the grain structure and strains are evaluated per grain.

Strains utilizing every strain measurement point (i.e. utilizing each individual tetrahedron) were also analyzed. This is referred to as the "strain point based analysis". The strain point based analysis was used for investigations of a possible preferential deformation near grain boundaries. This is feasible because the spatial distribution of strain points is almost homogeneous over the entire investigated volume, also near grain boundaries, as confirmed in a preliminary study of the present specimen [51].

2.6. Correlative analysis of grain parameters

In order to clarify possible correlations between local strain and grain parameters (e.g. grain size, Taylor factor and distance to grain boundaries), the following analysis was adapted: For each selected grain parameter, the grains were sorted in ascending order of that parameter. Box-and-whisker plots were made with suitable binning intervals. The average, median and interquartile range (IQR, i.e. middle 50%) were measured in each bin. IQR was used for the box drawing. The minimum and maximum values to draw whiskers were determined within the range of the IQR ± 1.5 × IQR width. The values that are outside this range are referred to as outliers. This correlation analysis was conducted for 8.5% and 15.6% tensile deformation. Only the plots for 15.6% tensile deformation are shown here, but similar tendencies were observed at 8.5% tensile deformation.

3. Results

3.1. Local strain development

The morphologies and orientations of the grains within the central part of the specimen, which are analyzed in the following, are shown in Fig. 4 (a) (see also Fig. 3 for an overview). The development of the internal strain (equivalent strain) is shown in Fig. 4 (b)-(d). With increasing macroscopic tensile strain, the strains within the individual grains increase, and local strain concentrations develop gradually. The small strain concentrations observed in the undeformed state (b) are due to the uncertainty in determining the particle displacement as discussed in reference [51]. It should be noted that as a consequence of this strain uncertainty, the hydrostatic (volumetric) strain component for each measured strain point can deviate from the as-expected value of zero for volume conserved plastic deformation. This noise-induced non-zero hydrostatic strain component is not physical. It is, however, kept and treated on equal terms with the uncertainty in the shear strain components for calculation of equivalent strain. Even though this leads to an increase in the variance of the equivalent strain, this variance increase (as shown in Fig. 4 (b)) is significantly smaller than the equivalent strain values of the plastically deformed sample analyzed here (see Fig. 4 (c) and (d)). It is therefore expected to have only a minor impact on the following quantitative analysis.

Fig. 5 shows the development of equivalent strains by the strain point based analysis and grain based analysis. For both analysis methods the strain distributions shift toward larger values and become wider with increasing macroscopic strain. These changes correspond to the development of strain concentrations as seen in Fig. 4 (c) and (d). The broadening means that inhomogeneous deformation develops locally. The distributions are observed to be narrower in the grain based analysis as compared to the strain point based analysis. This is caused by averaging over each of the
Fig. 4. Comparison of (a) internal grain structure (colored by IPF (ref. to Fig. 3 (b)) with (b)-(d) development of equivalent strains. Strain distribution at 0% was obtained from two undeformed ACT volumes.

Fig. 5. Comparison of equivalent strain development between (a) Strain point based and (b) Grain based analysis.

strain components within the individual grains in the grain based analysis.

3.2. Effect of grain size on local strain development

The observed relations between the local strain and initial grain size are shown in Fig. 6. Here, the grain based equivalent strain and the average of standard deviation $\sigma_{six}$ are shown. Fig. 6(a) reveals that the equivalent strains averaged over the individual grains only correlate very weakly to the grain size, and that there are slightly larger strain variations for small grains (i.e. some small grains take more deformation than others of similar size). In Fig. 6(b) $\sigma_{six}$, which represents the magnitude of inhomogeneous deformation within a grain, correlate positively with grain size, in the sense that the average of the standard deviation increases with increasing grain size. This suggests a higher tendency of inhomogeneous deformation within larger grains.

3.3. Effect of initial grain orientation on the local strain development

Fig. 7 shows how the equivalent strain and $\sigma_{six}$ depend on the Taylor factor of the initial grain orientations. The Taylor factor was calculated following Taylor’s original approach [53] by using the strain tensor for uniaxial tensile deformation on the $\{111\}<110>$ slip system applying a Poisson’s ratio of 0.5 (plastic deformation), i.e. $( 0 \ -0.156/2 \ 0 )$. Neither elasticity nor elastic anisotropy was considered. Fig. 7(a) reveals that the strain is nearly independent of Taylor factor. Similarly, no clear correlations between Taylor factor and $\sigma_{six}$ can be observed in the correlation plot shown in Fig. 7(b). This suggests that deformability of individual grains does not systematically affect the inhomogeneous deformation of the grains. An alternative visualization of the correlation between grain orientation and deformation is shown in Fig. 8. Here the equivalent strains after 15.6% tension and the initial orientations of the individual grains are plotted in inverse pole figures.
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**Fig. 6.** Local plastic strain development within the sample at 15.6% tension versus grain size (grain based analysis). (a) Equivalent strain and (b) Average of the standard deviation in the six strain components. Box-and-whisker plots with 10 μm intervals. The box corresponds to the IQR, the line and rhombus mark the median and average, respectively, the whiskers indicate min and max within 1.5 IQR and outliers are shown as circles.

**Fig. 7.** Local plastic strain development within the sample at 15.6% tension versus Taylor factor. (a) Equivalent strain and (b) Average of the standard deviation in the six strain components. Box-and-whisker plots with 0.15 interval.

**Fig. 8.** Inverse pole figure plot of equivalent strains at 15.6% tensile deformation. Grains with tensile strains (a) smaller or (b) larger than the average tensile strain of εz = 19% are plotted. Note that εz = 19% is higher than the macroscopic tensile deformation of the gauge volume due to strain concentration at the central 1/3 volume.

3.4. Effect of grain boundaries on local strain development

Grain boundaries (GBs) may be sites where deformability is discontinuous, and they may thus become sources of stress concentrations in polycrystalline microstructures. Possible effects of the distance from a GB on the deformation were therefore examined by using the strain point based analysis. The distances from the (IPFs) together with the values of the Taylor factor for uniaxial tensile deformation. The IPF plots in Fig. 8(a) and (b) are of grains with tensile strain, εz smaller or larger than the peak of the strain distribution, respectively. High and low equivalent strains are seen for all orientations. This tendency is somehow in contrast to the common knowledge that grains with low Taylor factor are generally easy to deform and can accumulate high local strains.
strain measurement points to the nearest GB was determined and used to plot equivalent strain as a function of distance to a GB. The result is shown in Fig. 9, where strain measurement points in grains smaller and larger than the mean grain size of 83 μm are plotted separately. No correlations are observed meaning that on average we do not observe strain localization at grain boundaries. Focusing, however, on the outliers, locations with small distances to GBs tend to occasionally experience very high strain values, but very low equivalent strain values are also observed here. This matches well with the visual impression seen in Fig. 4 (d).

3.5. Effect of grain shape on local strain development

To examine possible effects of grain shape on the local strain development, the shape of each grain was characterized by means of the principal axes of the grain. The principal axes, P1, P2 and P3 (P1 ≥ P2 ≥ P3) were measured by using the moments of the 3D digital images [54]. Three types of shape features were considered, that is “flat”, “spherical” and “elongated” shapes by the ratio of P3/P2, P1/P3 and P1/P2, respectively. The angle between the longest principal axis of a grain, P1, and the tensile direction was also measured to investigate potential effects of grain-shape orientation on the equivalent strain of grains.

The correlations between grain shape and equivalent strain are shown in Fig. 10. The details of the grain shape are shown in section 5.4 in the supplemental material. No statistically significant effects are observed for any of the cases. The result of the grain-shape orientation analysis is shown in Fig. 11. Here, only elongated grains, with the P1/P2 axes ratio above 1.5, are plotted (323 grains). Some fluctuation can be found in the correlation plot of strain versus angle. Whether the fluctuations are statistically significant is uncertain, and the data will not be discussed further in the following.

4. Discussion

The results have shown that laboratory-based multimodal X-ray tomography is a powerful technique for studying local plastic deformation of polycrystalline materials containing second phase particles. The spatial resolution of LabDCT is currently about 5 μm for grains with size >15–20 μm, in contrast to submicron for synchrotron DCT. On the other hand, the spatial resolution of laboratory ACT can nowadays be submicron, which is to a certain extent comparable to that of synchrotron ACT. This is mainly because the ACT resolution is limited only by the detector pixel size, while it is the low X-ray flux from laboratory X-ray tubes that is the limiting factor for LabDCT. Mapping a reasonably large volume of a typical sample with a grain size of 40-50 μm takes about 5–15 h for both ACT and LabDCT [55–58]. Though this is much longer than with synchrotron techniques. The unlimited access to a laboratory instrument remedies this situation.

With laboratory-based multimodal X-ray tomography, the local distribution of plastic strains within individual grains can be quantified with reasonable uncertainty based on the relative displacement of particles at different hardening stages of a stress-strain curve and correlated to the initial grain structure. In the following, the present results will be further analyzed and compared to those obtained previously, with focus on previous 3D results obtained using in-situ synchrotron techniques.

4.1. Relations between the local strain distributions and microstructural parameters

Heterogeneities developing during plastic deformation in either local strains or local crystallographic grain subdivision is by now well accepted to be the common pattern and is generally considered to be caused by anisotropic environments and thus grain-grain interactions [30,31,59,60]. The present results reveal very significant local strain heterogeneities both within individual grains and between grains. Large local strain concentrations within grains, which cannot be ascribed to strain measurement uncertainty (as discussed in [51]), are also obvious. Focusing first on grain averaged strain values, the present investigation reveals that grains with high average strains are not clustered macroscopically in the sample. The same is the case for grains with low average strains (see Fig. 12).

A tempting expectation for strain variations from grain to grain, would be that grains which deform much initially when the sample is exposed to the external load, will cease deforming at later stages as other less deformed grains take the load. This is, however, not what we observe. On the contrary, we observe that the grains deforming first and most, continue to do so all the way up to 15.6% elongation. This is seen when inspecting the whole volume (Fig. 4) and quantified in Fig. 13 where the evolution in equivalent strain distributions obtained from 100 strain measurement points are shown for grains with low and high equivalent strains after 8.5% deformation. Fig. 13(a) reveals that the low strained points are narrowly distributed and when the tensile deformation is increased to 15.6% the ‘add on’ strains are still rather low. For the highly strained points after 8.5% deformation (Fig. 13(b)) the strain distribution is wider and upon further tensile deformation the ‘add on’ strains cover the whole range from 0 to 9.9 in equivalent strain, meaning that some of the initially heavily strained grains do continue to strain more than average even when approaching fracture.
Another grain averaged result observed in this work is the equivalent strain independency of both grain size and Taylor factor. It is generally accepted that larger grains may be straining more and thus developing larger interior crystallographic misorientations than smaller grains [33,61-63]. We do observe a weak tendency that the average standard deviation in the six strain components increase with increasing grain size (Fig. 6(b)). We find that the equivalent strain is largely independent of the Taylor factor (Fig. 7). Although not matching the theoretical prediction [53,64], the present results are in general agreement with recent 3D synchrotron X-ray characterization results: Toda et al. have utilized the diffraction-amalgamated grain boundary tracking method to investigate the tensile deformation behavior of a similar Al-Cu alloy [59]. Only a limited number of grains were followed in that work, but very similar local plastic strain patterns and grain size/orientation dependencies were obtained. Using 3DXRD and HEDM, Pokharel et al. have quantified the local plastic deformation of 4930 Cu grains during tensile testing [44]. The results showed a clear relation between grain size and grain-averaged misorientation. If it is assumed that large local misorientation implies large local strain variation, their results are in good agreement with the present findings (see Fig. 6(b)).
Focusing next on the local strain variations, we note that by the present method using marker particles to map the local strain distributions, it is of course not possible to map strains on a scale finer than that of the particle distribution. In addition, the results are averaged within the individual volumes defined by tetrahedrons with the tracked marker particles as vertices. This means that some very local ‘hot spots’ may be smeared out, and possible local effects of grain boundaries/triple junctions may contribute to the results in the nearby tetrahedrons. To analyze the possible effects of the latter, the tetrahedrons are further grouped into different types according to the vertex distributions with respect to the grain boundaries and triple junctions. The results (see section 5.5 in the supplemental material) show that the vertex distributions of tetrahedrons does have an impact on the calculated strains. However, it does not change the overall conclusions.

Furthermore, in Ref. [51], it was analyzed if more particles were located near GBs in the present sample, which would have affected the results. It was, however, concluded that this is not the case. The results shown in Fig. 9, revealing that on average the equivalent strains are similar near GBs and in the grain interiors, are thus considered to be unbiased. It is thus interesting to note that Fig. 9 documents a significantly larger scatter in strain values near the GBs than away from them. At some boundaries, or locally along some GB segments, very large strains build up, whereas nothing special is observed at other GB segments. Similar conclusions have also drawn by Chen et al [65], based on their 2D high-resolution DIC plus EBSD analysis. This matches also perfectly with electron microscopy investigations of the dislocation structures near grain boundaries [66,67], where dislocation pile-up/accumulation is observed at some boundaries or boundary segments, while at others the dislocation structure is identical to that in the bulk of the grain. In this respect, it also must be considered that some grain boundary zones with high dislocation densities and large misorientations (note: these are not equivalent to strains) are quite narrow [68–71] in the order of a few µm, which is below the spatial resolution of the present method. An asset of 3D as compared to 2D measurements, is that 3D measurements allow determination of the true particle distance to the nearest grain boundary. 3D measurements with better spatial resolution by utilizing high resolution ACT, like synchrotron CT, for samples with finer particle spacing, would be a way forward to further clarify this issue.
As discussed in many papers [e.g. 60,72,73], grain-grain interactions may be essential for the strain evolution in the individual grains. It has for example been observed that high strain regions can be located between grains with large differences in Taylor factor, while low strain regions have been found between grains with similar orientations (i.e. similar Taylor factors) [59]. We have thus analyzed the difference in Taylor factor between each individual grain and its neighbors as well as the difference in Taylor factor between all the neighboring grains. The results are shown in Fig. 14. No correlations can be stated based on the present data, which comprises 855 individual grains.

Whereas the present results to a certain extent deviate from theoretical predictions, they agree in general with those from synchrotron measurements, despite differences in applied methodology and material. This suggests that the observed complexity of the plastic deformation process is quite representative of FCC polycrystalline materials with medium to high stacking fault energies (like Al and Cu). The small elastic anisotropy of these materials may be a reason for the observed lack of dependences. It could also be because, in addition to the parameters studied above, parameters such as number of neighboring grains, as well as spatial arrangement (with respect to the active slip systems) and the orientation relationship between neighboring grains also affect the grain-grain interaction, and hence the local strain development.

As a result of grain-grain interaction, the local strain variation within a grain can be as large as that between grains (see e.g. the grain marked by the circle A in Fig. 15), which can to a certain extent smear out the strain difference between grains. In some cases, a significant amount of shear strains develops (see e.g. the grains marked by the circle B in Fig. 15), which leads to large equivalent strains and makes Taylor’s original assumption of uniform strain for all grains in the gauge volume invalid. One consequence of this is that the grain orientation rotation path upon deformation can deviate from that predicted based on Taylor model [46,74], thereby affecting the correlation results. Moreover, macroscale band structure can be seen in some 2D sections of the 3D strain map (see e.g. those marked by the arrows in Fig. 15). Similar observations have been reported based on DIC measurements on sample surfaces (see e.g. [65]). These transgranular bands do not align with the 45° shear directions, and are coarse compared to the grain scale. The present results further reveal that this banding is only visible on 2D sections but not in the 3D grain averaged strain map (see Fig. 12), suggesting that the banding structure only penetrates part of the grain. This means that a part of a grain may be part of
a high strain band while another part resides in a low strain band. It is clear that when the local strains are averaged over a grain, such macroscopic transgranular banding will weaken any possible strain dependence on grain size and orientation. However, effects of the transgranular strain banding and the additional parameters discussed above on the local plastic deformation are difficult to quantify. To advance the understanding of the mechanisms behind the present experimental observations, complementary experimental investigations of strain (by MFT) and grain structure (by e.g. synchrotron methods [44,74] or 3D EBSD [75,76]) of both the starting and the deformed volume(s) for different material systems as well as crystal plasticity simulations [77–80], using the measured 3D initial microstructure as input, may be essential.

5. Conclusions

Laboratory X-ray methods were applied for non-destructive 3D characterization of both the grain microstructure (by LabDCT) and the development of local strains (by ACT and MFT) in an in-situ investigation of tensile deformation of Al-4mass%Cu. Effects of grain size, shape and orientation as well as grain boundaries on local strains were analyzed in 3D. In total, 855 bulk grains were included in the analysis. It was found that:

• Large local strain variations were present both within individual grains and between different grains.
• No clear correlations could be established between the grain averaged plastic strains and either the grain size or the grain orientation.
• It was observed that the grains deforming first and most, continue to do so all the way up to 15.6% elongation.
• Although some high plastic strains were seen at some grain boundary regions, on average there was no difference in strain at the grain boundary and grain interior regions.

As these results to a large degree agree with those from recent synchrotron experiments for other FCC materials, it is suggested that the observed complexity of the plastic deformation process is quite representative of FCC polycrystalline materials with medium to high stacking fault energies. The results further indicate that grain neighbor relations significantly affect the evolution of local strains, even though no clear correlation could be established between Taylor factors of neighboring grains for the present data. To further advance the understanding, crystal plasticity finite element simulations based on the present experimental data may be the way forward, thereby enabling a detailed analysis of grain–grain interactions.

Although the spatial resolution of the present strain distribution is limited by both the particle distribution and the ACT resolution of the laboratory X-ray system, we have clearly demonstrated the usefulness and potentials of non-destructive 3D characterization of plastic deformation by a combination of laboratory X-ray ACT and MFT. This method allows easy access to investigations of different materials, strains, strain rates etc., which we consider necessary for furthering the understanding of the evolution of strain heterogeneities during plastic deformation, and to provide 4D data (x, y, z, strain level) to validate and advance theoretical modelling.

Declaration of Competing Interest

The authors declare that they have no conflict of interest.

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Supplementary materials

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