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Monitoring microstructural evolution in-situ during cyclic loading with high-resolution reciprocal space mapping

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Abstract. High-resolution reciprocal space mapping using high-energy hard x-rays has been developed to investigate the microstructure of grains located in the bulk of a metallic sample in a non-destructive way in-situ during different loading conditions. The technique allows to identify and follow individual grains and subgrains during ongoing deformation such as loading in tension and compression, during repeated cyclic deformation or even individual load cycles while simultaneously monitoring macroscopic stress and strain. Insight in the structural reorganization within single grains is gained by in-situ monitoring of the characteristic intensity distribution of Bragg reflections from individual grains during cyclic deformation of commercially pure polycrystalline aluminium. By reciprocal space mapping with high angular resolution and combined analysis of the radial and azimuthal information, individual subgrains are tracked during single load cycles with different strain amplitudes. Additionally, changes in mean peak position, peak width and asymmetry of integrated radial profiles from individual grains are analyzed as well as their orientation spread. In this manner, the microstructural evolution in grains embedded in the bulk of polycrystalline specimens is traced and linked to the changing mechanical loads during cyclic deformation.

1. Introduction
Material failure due to fatigue as a consequence of repeatedly changing load conditions is the most dominant failure reason of structural components. Details about the microstructural changes occurring in metallic materials during periodically varying loads remained largely unidentified due to limitations of the available characterization techniques as well as challenges in performing in-situ experiments.

High-resolution reciprocal space mapping (HRRSM) using hard x-rays has been developed to investigate the microstructure of grains located in the bulk of a metallic sample in a non-destructive manner in-situ during mechanical loading [1-6]. The synchrotron technique enables investigating the characteristic intensity distribution of diffraction peaks in a crystalline material in large detail and to identify and follow individual grains and subgrains during ongoing deformation [1-6]. The technique is based on the acquisition of three-dimensional reciprocal space maps of Bragg reflections and reveals the orientation spread as well as the occurring elastic strains within an individual grain. As the time for acquisition of a single map is of the order of 20 min, changes in the microstructure of individual grains can be followed during mechanical loading – even during continued tensile deformation [7]. The acquired reciprocal space maps are analyzed and presented complementarily as radial profiles or
This allows tracking the microstructural evolution through the development of subgrains and the internal stresses associated with the substructure formation in individual grains during deformation. Simultaneous monitoring of macroscopic stress and strain enables to establish a link between the resolved evolution of the substructure and the mechanical loading.

In the past, HRRSM was applied successfully to unidirectional loading [1-6], stress relaxation [8], strain path changes [9-14], unloading and strain reversal [15] as well as cyclic deformation [16,17]. Previous investigations on cyclic deformation focused on application of HRRSM either on repeated tension-tension cycling for many cycles or during an individual cycle [16,17]. Here, HRRSM is employed on cyclic deformation in (almost symmetric) tension and compression with focus on the effect of a change in strain amplitude.

2. Experimental details

High-resolution reciprocal space mapping was carried out at beam line 1-ID-E at the Advanced Photon Source (APS) at Argonne National Laboratory with a monochromatic beam of 52 keV while loading an undeformed sample in position control. Figure 1 shows a sketch of the experimental set-up.

2.1. Material

Uniaxial test specimens were manufactured from a commercially pure aluminium sheet (AA1050) homogeneously cold-rolled by 90% thickness reduction to a final thickness of 1 mm. Dog bone-shaped specimens with a gauge section of 15 mm in length and 5 mm in width were designed to fit to a custom-made load frame. After spark-cutting, tensile specimens were annealed at 600 °C for 2 h to ensure complete and homogeneous recrystallization. After annealing, the microstructure was investigated metallographically with light optical and scanning electron microscopy. The microstructure was found to be homogeneous throughout the entire cross section of the gauge showing a pronounced cube texture; grain sizes were estimated to be between 30 μm and 100 μm. Tensile testing of recrystallized specimens with a nominal strain rate of 10^{-3} s^{-1} revealed a yield strength of 16 MPa.

2.2. Experimental set-up at APS

For the synchrotron investigation, the sample was equipped with two pre-wired strain gauges (Omega KFG-3 350 Ω), one on each side at the center of the gauge section and aligned with the loading axis to monitor the axial strain in-situ. The sample was mounted in a custom-made screw-driven load frame equipped with a 1 kN load cell as presented in the insert of figure 1. Using flat grips, this load frame enables mechanical loading in tension and compression while monitoring the local microstructure within the test specimen in-situ using synchrotron radiation. The load frame was placed with the load axis horizontally on a xy-translation stage allowing to translate the sample and with this the chosen grain of interest – which gets displaced with respect to the load frame during mechanical loading – to the center of the beam after each load step. The xy-translation stage is mounted on top of a rotation stage enabling rotation of the entire load frame around the vertical z-axis to obtain the desired reciprocal space maps by rocking in small intervals of the angle ω around this axis. An additional z-translation stage provides adjustment for possible changes in the height of the selected grain of interest due to mechanical loading (cf. figure 1).

2.3. Experimental procedure for high-resolution reciprocal space mapping

Suitable grains are identified with the help of a large area detector (detector 1), an amorphous silicon flat panel from General Electrics, placed 86 cm behind the sample on a horizontal translation to cover the first 6 diffraction rings of aluminium. Individual grains are selected by finding isolated 400 diffraction peaks (i.e. peaks without any overlap with peaks from other grains) from a set of diffraction patterns (acquired at different ω angles but at same sample position to determine grain size and neighbourhood). Four bulk grains are chosen which have their 400 diffraction vector close to the loading axis. (Grains with their crystallographic [100] direction along the loading axis develop cell structures with subgrains separated by dislocation walls in tension [18] and cyclic deformation [19].)
After this, detector 1 is moved out of the beam and the diffraction peaks are investigated with significantly higher angular resolution by a Mar165 CCD (detector 2). This second detector is placed 4.65 m behind the sample at the location of 400 diffraction peaks with their diffraction vectors close to the loading axis (i.e. in the horizontal diffraction plane at a diffraction angle 2θ_{400} of 13.53° for aluminium). An entire sequence of two-dimensional detector images for each selected diffraction peak is acquired with the far detector 2 while rocking the sample around the vertical z-axis perpendicular to the scattering plane in small intervals Δω of 0.015° of the rocking angle ω. In this way three-dimensional distributions of the diffracted intensity (consisting of two dimensions, η and 2θ, on the far detector and additionally the rocking angle ω as third dimension) are obtained by stacking the images recorded for several adjacent ω intervals.

Two different projections of the acquired three-dimensional reciprocal space maps are conveniently used for analysis: azimuthal projections and radial profiles. Azimuthal projections characterize the intensity distribution of the diffraction peak in dependence on the two azimuthal angles (η,ω). They represent the pole density in dependence on the orientation as given by the angles (η,ω) and are essentially pole figures of quite high resolution. Radial profiles, on the other hand, are azimuthally integrated intensity distributions and represent the integrated intensity in dependence on either the diffraction angle 2θ (related to wave length λ and spacing dhkl between the diffracting (hkl) lattice planes by Bragg’s law 2dhkl sinθ = λ) or the length of the diffraction vector q = 2π/dhkl = 4π sinθ / λ. The latter is the preferred means of representation as (relative) differences in the diffraction vector Δq relate directly to the elastic strain

\[ ε_{el} = \frac{d - d_0}{d_0} = \frac{q - q_0}{q} = -\frac{Δq}{q}. \]

From such three-dimensional reciprocal space maps (and their two complementary projections), microstructural features such as dislocation walls and subgrains within individual grains can be identified from intensity variations in the map [1-6]: These intensity variations are caused by the slightly different and unique orientations of individual subgrains (e.g. [1-3,6,20]) in the deformation structure as the crystalline lattice becomes locally distorted by the emerging dislocation structures. Sharp peaks of high intensity in the high-resolution reciprocal space map correspond to individual subgrains, whereas a smooth cloud of lower intensity originates from the dislocations walls. From the characteristic intensity distribution of ordered dislocation structures, both the variation of elastic strains within single grains as well as the elastic strains of the embedded individual subgrains can be resolved.
3. Experimental procedure and results

3.1. Sample deformation

An undeformed and annealed dog bone-shaped tensile sample equipped with two strain gauges as described in section 2.2 was mounted in the load frame. After pre-deformation to 1% tensile strain by a macroscopic stress of 42 MPa, the specimen was cyclically deformed into saturation by 10000 tension-compression cycles with a strain amplitude $\varepsilon_{a1} = 0.4 \cdot 10^{-3}$. After this initial cycling, four grains were selected for further investigations and the sample was cyclically deformed further – for in total 30010 cycles. The last ten cycles where monitored in detail to choose the displacement for the desired stress-strain conditions along the stress-strain hysteresis curve for which data acquisition was intended. Based on this, a first tension-compression load cycle with the small strain amplitude $\varepsilon_{a1} = 0.4 \cdot 10^{-3}$ was performed for which reciprocal space mappings were undertaken at specific stress-strain conditions in such a manner that both the elastic region, as well as the yield point (or at least close to it), the plastic region and the state of maximum compression and tension were covered.

Thereafter, the strain amplitude was increased over 10 cycles to the intended strain amplitude of $0.82 \cdot 10^{-3}$. The sample was cycled in displacement control for another 10010 cycles, during which the strain amplitude decreased to a final value of $\varepsilon_{a2} = 0.68 \cdot 10^{-3}$. Again the last ten load cycles with the new strain amplitude of $\varepsilon_{a2} = 0.68 \cdot 10^{-3}$ were recorded in detail to select the displacements for the desired stress-strain conditions, before a second load cycle at the increased strain amplitude $\varepsilon_{a2}$ was performed and reciprocal space maps were acquired. (Finally, the sample was cycled for another 8500 cycles with the large strain amplitude $\varepsilon_{a2}$ before a further mapping of the grains was undertaken. The sample was then unloaded to zero stress and a final high-resolution reciprocal space map acquired.)

During acquisition of each high-resolution reciprocal space map, applied load and resulting strains were recorded by the load cell and the strain gauge, respectively. The average macroscopic stress and strain of all acquisitions are displayed in figure 2. Note, that during pausing of the motors for HRRSM after each load step, the position of the cross heads is fixed, while stresses and strains do not remain constant and change slightly. From the two hysteresis curves in figure 2, a clear increase in the width of the obtained hysteresis loop is obvious for load cycle 2 in comparison to load cycle 1. It also becomes evident that the average macroscopic strain decreased during cycling deformation.

![Figure 2](image)

**Figure 2.** Macroscopic stress and strain measured during all acquisitions of high-resolution reciprocal space maps (highlighted with circles) showing the typical hysteresis curve during both load cycles. Before the two load cycles were investigated in detail, the sample was once unloaded and re-loaded without any intermediate step (represented by the lines in the middle of the hysteresis loops) to verify the strain amplitude.

3.2. Azimuthal maps

Grains were selected after pre-deformation to 1% tensile strain and 10000 tension-compression cycles. Four grains were chosen for further investigations with up to 29 HRRSMs per grain. Figure 3 shows azimuthal projections of the four grains measured always at maximum tension: (i) after the initial 10000 cycles, when the grains were initially selected, (ii) after cycling for 40010 cycles with $\varepsilon_{a1}$ and (iii) after an increase of the strain amplitude to $\varepsilon_{a2}$ followed by another 10020 cycles. Already when identifying the grains, the intensity distribution of their diffraction peaks showed a wide spread due to the 1% tensile pre-deformation. A clear increase of their extent in reciprocal space after the increase in strain amplitude is visible for grains 1 and 3, but less pronounced for grains 2 and 4.
Figure 3. Azimuthal maps ($\eta$ horizontally, $\omega$ vertically) of the 400 diffraction peak after grain selection (left), before (middle, L5a see below) and after (right, L1b) increase of strain amplitude from $\varepsilon_{a1}$ to $\varepsilon_{a2}$ for four different grains: (a) grain 1, (b) grain 2, (c) grain 3, and (d) grain 4.

The two grains 1 and 2 were mapped in detail during the two load cycles with different strain amplitudes with 9 (load cycle 1 designated with a, figure 4a) and 11 (load cycle 2 designated with b, figure 4b) acquisitions along the hysteresis curve. The acquisitions were selected to be at the maximum tensile load before (L1a, L1b) and after the load cycle (L5a, L6b), in the elastic region during unloading (U2a; U2b, U3b) and loading (L2a; L2b, L3b), around the yield point (U3a, L3a; U4b, L4b), at the maximum compressive load (U5a, U6b) and at several interesting conditions in the plastic region along the cycle (U4a, L4a; U5b, L5b). All measurements during unloading from maximum tension and loading to maximum compression are designated with U and all measurements during unloading from compression and loading to maximum tension are designated with L.

Figure 4. Hysteresis loops (left) and azimuthal maps of grain 1 (right) during the two load cycles with different strain amplitudes $\varepsilon_{a1}$ (a) and $\varepsilon_{a2}$ (b). Red lines indicate zero load and average macroscopic strain of the total strain variation during the cycle. The designation of the acquisitions starts with L1a (L1b) at maximum tensile load, following the arrows during unloading into compression to U5a (U6b) and loading in tension with a final map at maximum tension L5a (L6b).
Both load cycles interrupted for HRRSM were nearly symmetrical in their stress amplitude around zero load (indicated by the crossing red lines). In both cases, the absolute maximal stresses in compression were only slightly smaller than the absolute maximal stresses in tension. The stress ratio $R = \sigma_{\text{max}}/\sigma_{\text{min}}$ for the hysteresis loop changed from the value $R = -0.84$ at small strain amplitude $\varepsilon_{a1} = 0.4 \cdot 10^{-3}$ to an even higher value of $R = -0.97$ for the larger strain amplitude $\varepsilon_{a2} = 0.68 \cdot 10^{-3}$.

The azimuthal maps for grain 1 shown in figure 4a and b for all acquisitions during the two load cycles demonstrate exemplarily that no major changes are detectable during a hysteresis loop, but that significant changes are caused by the increase in strain amplitude and the performed 10020 cycles.

3.3. Radial profiles

Radial profiles are analysed for both load cycles. In figure 5, the radial profiles for the first load cycle are presented for grain 1. For a better overview, the profiles are split up into two sequences of unloading into compression (figure 5a) and loading into tension (figure 5b). The radial profiles shift to higher values of the diffraction vector $q = 2\pi/d_{hkl} = 4\pi \sin \theta / \lambda$ during compressive loading and to lower values during tensile loading in accordance with the applied load.

![Figure 5](image)

**Figure 5.** Radial profiles of grain 1 for load cycle 1: (a) unloading from tension and into compression (L1a, U1a-U5a), (b) unloading from compression and loading into tension (U5a, L2a-L5a).

Figure 6 shows the mean profile position of each of the four grains during the first load cycle compared to the strain-free diffraction vector $q_0 = 2\pi/d_{400} = 6.2064 \text{ Å}^{-1}$ (based on the lattice constant $a = 4.0495 \text{ Å}$ of pure aluminium and marked by a horizontal line). Grains 1 and 3 have peak positions generally above the strain-free diffraction vector $q_0$, whereas the average diffraction vectors of grains 2 and 4 remain mainly below $q_0$ – indicating quite different neighbourhoods between the four grains.

![Figure 6](image)

**Figure 6.** Mean profile position of the four grains for load cycle 1 in dependence on (a) macroscopic strain and (b) macroscopic stress. Apparent Young’s moduli are noted next to the data of each grain.
The radial profiles are further analysed with regard to their integral profile width $\beta$ and asymmetry $\kappa$ as shown in figure 7 where the changes of both parameters during the two load cycles is illustrated for grain 1. During the first load cycle, the peak width (see figure 7a) is initially decreasing until U4a and then increasing towards the maximum compression (U5a) and further during tensile loading towards L5a. The second load cycle reveals more details due to the two additional measurements. Here it becomes obvious that the profile width is in general decreasing towards its lowest value at U5b and again strongly increasing towards the maximum compression U6b. After U6b it decreases first towards L5b, before increasing again towards L6b. The behaviour – in particular for the second load cycle – resembles a so-called butterfly pattern observed earlier [21,22] and discussed in detail in [23].

The absolute asymmetry $\kappa$ (i.e. the difference between the widths at half of the maximum intensity on both sides of the position of maximum intensity) follows a distinct pattern as well (cf. figure 7b). During the first load cycle, the asymmetry is first increasing from L1a until U4a and then decreasing towards U5a. The asymmetry is in general decreasing further until L4a and then increasing again towards maximum tensile load L5a. For the second load cycle the asymmetry is first increasing until U4b and then decreasing towards U6b. It behaves similar (but with opposite sign) during tensile loading with first decreasing until L4b and then increasing until the tensile point L6b is reached. The asymmetry is changing sign from positive to negative during loading into compression as expected from the composite model (see section 4.2), with a similar absolute value ±1.6 Å⁻¹ at maximum tension and compression during load cycle 2. The other three grains behave quite similar (cf. [23]).

### 3.4. Subgrains

In high-resolution reciprocal space maps, the existence of high-intensity peaks on a cloud of enhanced intensity reflects the developing ordered dislocation structure with perfect, slightly disoriented subgrains separated by dense dislocation walls. Each almost perfect subgrain gives rise to one individual peak of high intensity, whereas the dislocation walls contribute collectively a cloud of enhanced intensity to the diffraction peak [1-6]. For analysing subgrains, the two different contributions from subgrains and walls are separated by a mathematical approach [7,10]. Figure 8 shows the separated azimuthal map originating solely from all subgrains in grain 1 at the specific loading condition L1b just before the second load cycle. Circles mark the ten most intense peaks corresponding to the ten largest subgrains. For grain 1, up to 92 subgrains have been identified in this manner in the separated subgrain contributions of the different acquired reciprocal space maps. No clear relation between the number of identified subgrains and the stress-strain condition was found, though a tendency for fewer identified peaks around the maximum compressive load was observed for load cycle 2. With such a large number of identified subgrains, a statistical analysis can be performed (as illustrated for unidirectional tension [2], strain path changes [13,14], unloading and strain reversal [15] or cyclic deformation in tension-tension [17] – revealing a Gaussian distribution of the elastic
strains of subgrains in all cases). Alternatively, individual subgrains can be identified by their azimuthal position and followed during the loading sequence. The latter is illustrated here for the second load cycle by the largest subgrain in the azimuthal map in figure 8 marked with a red circle which could be followed through all acquisitions of grain 1.

**Figure 8.** Azimuthal map of grain 1 at maximum tensile stress after 10020 cycles with larger strain amplitude $\varepsilon_{a2}$ (load step L1b). Only the separated part formed by the high-intensity peaks of all subgrains is illustrated. The ten most intense peaks corresponding to the ten largest subgrains are marked with circles.

The radial profiles of this subgrain are shown together with the radial profiles of grain 1 in figure 9a at the maximum tensile and maximum compressive load (L1b in black and U6b in red, respectively), where the mean profile positions are marked with straight lines additionally. It is apparent, that the grain profile at maximum compression U6b looks in general mirrored to the grain profile measured under maximum tensile load L1b. The subgrain profile shifts similar to the one of grain 1 to higher $q$-values during loading into compression and to lower $q$-values during loading into tension. Small differences can be identified between the mean profile positions of grain and subgrain. The mean position of the grain is at slightly higher $q$-values than the one for the subgrain at maximum tension and at slightly lower $q$-values at the maximum compression, meaning that the subgrain experiences slightly less tensile strain at maximum tensile load and slightly less compressive strain at maximum compressive load than the grain, i.e. the subgrain experiences back strains in both cases.

**Figure 9a.** Radial profiles of grain 1 at maximum tension (L1b) and maximum compression (U6b) during load cycle 2. The additional profiles of the particular subgrain (which has been identified in all load steps and marked in the azimuthal map in figure 8 by a red circle) are scaled to a maximum of 0.5. The mean profile positions (of the fitted profiles) are marked with vertical dashed lines for the grain profiles and full lines for the subgrain profiles. (b) Average profile positions $q_{\text{mean}}$ for grain 1 (black) and the particular subgrain (red) in dependence on macroscopic strain.

The mean positions of grain and subgrain throughout load cycle 2 are displayed in detail in figure 9b, where the shift of the subgrain profile clearly reproduces the hysteresis loop as observed for the grain. Obviously, the subgrain shows slightly higher $q_{\text{mean}}$ than the entire grain at maximum tension, unloading from there and loading into compression (meaning the subgrain experiences slightly less tensile stress) and slightly lower $q_{\text{mean}}$ than the grain at maximum compression, unloading from there and loading into tension (meaning the subgrain experiences slightly less compressive stresses than the grain). This is in accordance with previous findings for tension-tension cycling [17] that there is a size effect governing the stresses of individual subgrains with the largest subgrains always experiencing the largest back stresses.
3.5. Increase in strain amplitude

The strain amplitude was increased in between the detailed acquisition of the two load cycles from $\varepsilon_{a1} = 0.4 \cdot 10^{-3}$ to $\varepsilon_{a2} = 0.68 \cdot 10^{-3}$. Before the second load cycle was measured, 10020 cycles with the increased strain amplitude $\varepsilon_{a2}$ were performed. Figure 10 visualizes the changes in the Bragg reflection along the azimuthal $\omega$ direction and with this the appearance of the grain in orientation space before and after the increase of the strain amplitude. The rocking curves of all four grains change slightly between before (blue) and after (red) the increase in $\varepsilon_{a}$. The intensity distributions become broader in width, most clearly seen for grains 1 and 3 which has earlier been indicated by the azimuthal maps in figure 3. The rocking curve of grain 4 seems to become slightly broader as well. The observed movements in $\omega$ are due to a rotation of the grains when the sample and the grains adjust to the higher strain amplitude and the different level of macroscopic strain of the second load cycle.

**Figure 10.** Rocking curves for four grains before (measurement 1, L5a) and after (measurement 2, L1b) increase of strain amplitude from $\varepsilon_{a1}$ to $\varepsilon_{a2}$. Each $\omega$ interval corresponds to $\Delta\omega = 0.015^\circ$.

Figure 11 presents an overview how radial profile position, width and asymmetry were affected by the change in strain amplitude. The radial profiles were analysed at maximum tensile load at the end of the first load cycle (L5a, measurement 1) and at maximum tensile load at the beginning of the second load cycle (L1b, measurement 2), i.e. after additional 10020 cycles with $\varepsilon_{a2}$. The profile positions decrease only slightly due to the lower macroscopic stress at L5a than at L1b (cf. figure 2). The radial profile width is increasing in general for all grains, though less pronounced for grain 1. The absolute asymmetry is increasing for all grains in a similar manner. After increasing the strain amplitude, three grains (grain 1, 3 and 4) show a positive asymmetry at maximum tension and a negative asymmetry at maximum compression as expected by the composite model (cf. section 4.2); in contrast to all other grains, grain 2 has negative asymmetry throughout. Nevertheless, profile width and asymmetry increase both with an increase in strain amplitude as expected due to increasing internal stresses.

**Figure 11.** Comparison of (a) average profile position, (b) integral width, and (c) absolute asymmetry at maximum tensile load before the increase of strain amplitude after 40010 cycles with $\varepsilon_{a1}$ (L5a, measurement 1) and after additional 10020 cycles with $\varepsilon_{a2}$. (L1b, measurement 2).

4. Discussion

4.1. Peak positions

The different average peak positions of the four grains seen in figure 6 reveal substantial internal stresses within the specimen. Grains 1 and 3 show in general compressive strain with respect to the strain-free lattice spacing, whereas grains 2 and 4 show tensile strains. These internal elastic strains
must be caused by corresponding internal stresses arising during the tensile pre-deformation as a result of load transfer between the grains under investigation and their immediate surrounding of neighbouring grains. As grains 1 and 3 experience compressive stresses, i.e. back stresses compared to the initial tensile loading, they are expected to be located in rather strong (or stiff) neighbourhoods, so they will yield first (or elastically deform more) when under the same load. In order to deform compatibly with the stronger (or stiffer) neighbourhood, they develop back stresses with respect to the applied tensile load. Vice versa, grains 2 and 4 are expected to reside in a weaker (or more compliant) surrounding which yields earlier (or elastically deforms more) than the two grains under consideration. Grains 2 and 4 will hence develop additional forward stresses, i.e. residual tensile stresses, to keep a compatible deformation.

A linear dependence of the peak position $q_{\text{mean}}$ on the macroscopic applied stress becomes evident from figure 6b. Considering that the average elastic strain $\varepsilon_{\text{el}} = (q_0/q_{\text{mean}})-1$ of each grain is related to the mean peak position, this is expected from elastic behaviour. An apparent elastic modulus $E^* = |\sigma|/|\varepsilon_{\text{el}}|$ can be determined for each of the grains. The resulting values specified in figure 6b are all above the elastic modulus $E_{[100]} = 63.5$ GPa of aluminium along its crystallographic [100] direction [24]. The increased values are partially caused by the misalignment between the tensile axis and the [100] direction along which the elastic strain is determined. Due to elastic cross contraction, any such misalignment leads to a reduction of the measured elastic strain along the 400 diffraction vector and hence to an overestimation of the elastic modulus. The variations between the apparent moduli of the four grains are, however, too large to be caused by their slightly different orientation (which can be assessed from the azimuthal position of the diffraction peaks and differ by less than $5^\circ$). The different values for the apparent moduli must hence result from the neighbouring grains affecting the stress state. The effect of the neighbourhood will not only cause load transfer between grains of different stiffness and strength, but also to possible deviations from a uniaxial stress state. According to figure 6b, a correlation exists between the apparent moduli and the residual stresses in a grain: Grains 1 and 3 which developed compressive residual stresses during tensile pre-deformation have a lower apparent modulus than grains 2 and 4 which developed tensile residual stresses. This correlation can be rationalized by the same reasoning about the neighbourhood of the different grains as outlined above. The strong and stiff neighbourhood of grains 1 and 3 experiences higher local stresses $|\sigma_{\text{loc,high}}|$ and higher elastic strains $|\varepsilon_{\text{el,high}}|$ than the weak or compliant surrounding of grains 2 and 4 ($|\sigma_{\text{loc,low}}| < |\sigma_{\text{loc,high}}|$ and $|\varepsilon_{\text{el,low}}| = |\varepsilon_{\text{el,high}}|$). Hence, the apparent Young’s moduli $E^* = |\sigma|/|\varepsilon_{\text{el,high}}|$ of grains 1 and 3 become smaller than the apparent moduli $E^* = |\sigma|/|\varepsilon_{\text{el,low}}|$ of grains 2 and 4.

4.2. Shape of radial profiles

Radial profiles of plastically deformed metals are in general asymmetric [25-27] as rationalized by the composite model of deformation structures [28,29]. The refined version of the composite model [5] takes into account individual, dislocation-free subgrains and dislocation-rich dislocation walls. During loading, subgrains yield before the walls due to their lower dislocation density. In order to maintain compatible deformation, load is transferred from the weaker subgrains to the stronger walls leading to back stresses (with respect to the applied stress) in the subgrains and forward stresses in the walls. This difference in stresses causes differences in the elastic strains between subgrains and walls which are revealed by diffraction as both components will diffract at different diffraction vectors $q = q_0/(1+\varepsilon_{\text{el}})$. Furthermore, the subprofile arising from dislocation walls is significantly broadened due to their high dislocation density. As rationalized in the refined composite model [5], each dislocation-free subgrain will contribute with its own sharp profile at a slightly different diffraction vector. The summed subprofile from all subgrains will be broadened slightly due to the Gaussian distribution of the elastic strains of individual subgrains [2,13-15,17]. The combination of the shift of the two subprofiles with their different width and volume fraction leads to asymmetric profiles. For instance, in tension, subgrains come under compressive back stresses leading to compressive strains along the loading direction and a higher diffraction vector than that of the walls which are under forward stresses and hence experience tensile elastic strains. Due to the rather sharp subprofile contributed by
all subgrains and their slightly different stresses, the maximum intensity of the diffraction peak at \( q_{\text{max}} \) is close to the average position of the subgrains, whereas the contribution of the walls occurs at lower diffraction vectors. Consequently, the average position \( q_{\text{mean}} \) of the entire peak has a lower value than \( q_{\text{max}} \) and the radial diffraction profile becomes asymmetric with larger width for lower \( q \), hence, leading to a positive (absolute) asymmetry \( \kappa \).

This asymmetry of diffraction peaks has been discovered first in tensile deformed copper [23,24] and rationalized by the original composite model [25-27]. Occurrence of the corresponding asymmetry in aluminium after tensile deformation has been confirmed much later [28]. During cyclic deformation in tension and compression, the situation becomes reversed between tension and compression and the asymmetry is expected to change sign from being positive under tension to becoming negative in compression. This has been observed on copper after unloading from tension or compression [22].

The present investigation confirms the general trend of reversing the sign of asymmetry between tension and compression as seen in figure 7, but even more importantly it reveals that the asymmetry is much more pronounced in the unloaded state than under maximum tensile or compressive load: The absolute asymmetry shows the proper sign at maximum tension and compression, but with quite small absolute values only, whereas during unloading a significant increase of the absolute amount of the asymmetry is observed. The reason for this behaviour roots in the different elastic strains of the individual subgrains and their succession of yielding during strain reversal. This idea has to be investigated further and may potentially rationalize the butte-fly behaviour of the peak width as well.

4.3. Increase in strain amplitude

An increase in strain amplitude is connected with an increase in the amplitude of the applied stresses due to work-hardening of the material mainly by dislocation accumulation in the walls. The increased dislocation density in the walls leads – from statistical reasons – even during cyclic deformation to an increase of the small orientation differences between the subgrains [31]. The increasing mutual disorientations cause an increasing orientation spread in the grains which explains both, the observed larger spread of the intensity distribution in the azimuthal maps (figure 3) and the larger width of the rocking curves (figure 10) after an increase in strain amplitude. The increased dislocation density in the walls causes a larger difference between the elastic strains of subgrains and walls and consequently a larger peak width and a larger amount of the absolute asymmetry after the increase in strain rate.

5. Conclusion

Cyclic deformation in tension and compression of macroscopic, commercially pure, polycrystalline aluminium samples was performed with a custom-made load frame. High-resolution reciprocal space mapping was used to investigate the effect of changing the strain amplitude during cycling. Independent of the strain amplitude, the acquired azimuthal maps show only minor changes along a load cycle. A peak shift of the radial profiles following the macroscopically applied stress was observed as well as indications for a characteristic behaviour of the profile width and asymmetry along a tension-compression load cycle. On the other hand, a clear difference in both shape and intensity distribution in the azimuthal maps is observed for different strain amplitudes. An increase in strain amplitude results in broadening, which can be quantified from both radial profiles and azimuthal maps and linked to structural reorganization occurring in the monitored grains. Further insight into the deformation-induced processes is of fundamental importance to understand and predict materials behaviour during cyclic deformation in order to facilitate future materials design.

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References

[1] Jakobsen B, Poulsen H F, Lienert U, Almer J, Shastri S D, Sørensen H O, Gundlach C and Pantleon W 2006 Science 312 889-92
[2] Jakobsen B, Poulsen H F, Lienert U and Pantleon W 2007 Acta mater. 55 3421-30
[3] Jakobsen B, Poulsen H F, Lienert U, Huang X and Pantleon W 2007 Scripta mater. 56 769-72
[4] Jakobsen B, Poulsen H F, Lienert U and Pantleon W 2008 Mater. Sci. Engng. A 483-484 641-3
[5] Pantleon W, Wejdemann C, Jakobsen B, Lienert U and Poulsen H F 2010 Advances in characterization of deformation structures by high resolution reciprocal space mapping Proc. 31st Risø Intern. Symp. Mater. Sci. eds N Hansen et al. (Roskilde: Risø DTU) pp 79-100
[6] Pantleon W, Wejdemann C, Jakobsen B, Poulsen H F and Lienert U 2014 High-resolution reciprocal space mapping for characterizing deformation structures Strain and dislocation gradients from diffraction eds R Barabash and G Ice (London: Imperial College Press) chapter 9 pp 322-57
[7] Pantleon W, Wejdemann C, Jakobsen B, Lienert U and Poulsen H F 2009 Mater. Sci. Engng. A 524 55-63
[8] Jakobsen B, Poulsen H F, Lienert U, Bernier J, Gundlach C and Pantleon W 2009, phys. stat. sol. (a) 206 21-30
[9] Wejdemann C, Poulsen H F, Lienert U and Pantleon W 2009 IOP Conf. Series: Mater. Sci. Engng. 3 012003:1-6
[10] Wejdemann C, Lienert U, Nielsen H B and Pantleon W 2010 Identifying individual subgrains in evolving deformation structures by high angular resolution x-ray diffraction Proc. 31st Risø Intern. Symp. Mater. Sci. eds N Hansen et al. (Roskilde: Risø DTU) pp 477-87
[11] Wejdemann C, Lienert U and Pantleon W 2010 Scripta mater. 62 794-7
[12] Wejdemann C, Lienert U and Pantleon W 2010 J. Phys.: Conf. Series 240 012160:1-4
[13] Wejdemann C, Poulsen H F, Lienert U and Pantleon W 2013 JOM 65 35-43
[14] Lienert U, Ribárik G, Ungar T, Wejdemann C, Pantleon W 2017 Synchr. Rad. News 30 35-40
[15] Diederichs A M, Thiel F, Fischer T, Lienert U and Pantleon W 2017 IOP Conf. Series: J. Phys.: Conf. Ser. 843 012031-1:9
[16] Diederichs A M, Thiel F, Lienert U and Pantleon W 2017 Procedia Struct. Integr. 7 268-74
[17] Diederichs A M, Thiel F, Lienert U and Pantleon W 2018 Int. J. Fatigue 117 206-16
[18] Huang X and Hansen N 1997 Scripta mater. 37 1-7
[19] Vidum M and Ryum N 1996 Mater. Sci. Engng. A 219 1-10
[20] Gundlach C, Pantleon W, Lauridsen E M, Margulies L, Doherty R D and Poulsen H F 2004 Scripta mater. 50 477-81
[21] Holste C and Burmeister H J 1979 phys. stat. sol. (a) 57 269-80
[22] Biermann H, Ungar T, Pfannenmüller T, Hoffmann G, Borbely A and Mughrabi H 1993 Acta metall. mater. 41 2743-53
[23] Diederichs A M 2019 Structural reorganization during cyclic deformation (Kongens Lyngby: DTU) Ph.D. thesis 252 pp
[24] Tallon J L and Wulfenden A 1979 J. Phys. Chem. Solids 40 831-7
[25] Ungar T, Mughrabi H, Rönnpagel D and Wilkens M 1984 Acta metall. 32 333-42
[26] Mughrabi H, Ungar T, Kienle W and Wilkens M 1986 Philos. Mag. A 53 793-813
[27] Mughrabi H and Ungar T 2002 Long-range internal stresses in deformed single-phase materials: the composite model and its consequences (Dislocations in solids vol 11) (Amsterdam: Elsevier) chapter 60 pp 343-411
[28] Mughrabi H 1978 Mater. Sci. Engng. 33 207-23
[29] Mughrabi H 1983 Acta metall. 31 1367-79
[30] Pantleon W, Poulsen H F, Almer J and Lienert U 2004 Mater. Sci. Engng. A 387-389 339-42
[31] Pantleon W 1999 Dislocation boundaries: formation, orientation and implications Proc. 20th Risø Intern. Symp. Mater. Sci. eds J B Bilde Sørensen et al. (Roskilde: Risø National Laboratory) pp 123-46