Structural Reorganization During Cyclic Deformation

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Abstract

A major failure reason for structural materials is fatigue-related damage due to repeatedly changing mechanical loads. During cyclic loading, dislocations self-organize into characteristic ordered structures, which play a decisive role for the materials lifetime. The synchrotron technique High Resolution Reciprocal Space Mapping (HRRSM) using high energy X-rays was successfully applied to characterize these heterogeneous deformation structures evolving during cyclic deformation of commercially pure, polycrystalline aluminium AA1050. Insight into the structural reorganization within single grains embedded in the bulk material is gained by in-situ monitoring of the microstructural evolution during individual tension-compression load cycles and after selected numbers of cycles along tension-tension or tension-compression cycling sequences. By High Resolution Reciprocal Space Mapping individual subgrains can be resolved in the bulk of polycrystalline specimens and their fate, their individual orientation and elastic strains, tracked during different loading regimes. With this approach, the evolution of the intragranular structure in selected grains was followed.

Four or five grains were monitored during each of in total four weeks of beam time at Argonne Photon Source and PETRA and a detailed analysis of their subgrain structure is presented for selected grains. Initially, the microstructural changes during tension-tension cycling were investigated, where the azimuthal maps and radial profiles of in total four grains were analyzed during a cycling sequence of 7350 cycles. It was possible to follow the same subgrain over the entire cycling sequence. It is concluded that the microstructure is stable during the saturation stage of cyclic deformation, since only minor microstructural changes where observed in azimuthal maps and radial profiles during cycling sequences. It was however shown that major changes are occurring during the first cycles after tensile loading possibly due to structural reorganization for adaptation of the cyclic deformation condition.

During in total three weeks of beam time tension-compression cycling was investigated, where finally up to 60 acquisitions were done for individual grains monitoring the microstructural changes during tension-compression cycling and along five subsequent tension-compression load cycles. A characteristic behavior of the peak profile width and peak profile asymmetry was revealed. It was observed for the first time that the maximum asymmetry does not occur at the maximum tension and compression, but around the yield points. This is attributed to a size effect of the subgrains. The elastic back strains of subgrains are Gaussian distributed with larger subgrains showing larger back strains implying a size effect. Four subgrains were followed during three subsequent load cycles with fourteen HRRSM acquisitions along each hysteresis. The subgrains showed different behaviors of the local elastic strains. All investigated grains showed different values for the profile position, width and asymmetry as well. It is therefore concluded that the local environment is of high importance for the behavior of grains and subgrains under applied stress.

The detailed characterization of the microstructure during cyclic loading by in-situ monitoring of the internal structure within individual grains facilitates the understanding of the material behaviour during cyclic deformation by providing experimental findings for the development of models predicting the materials performance.
Dansk resumé


Det var muligt at følge det samme underkorn igennem hele den cykliske belastningssækvens. Det konkluderes at mikrostrukturer er stabil under mætningsstadiet af den cykliske deformation, eftersom kun mindre ændringer i mikrostruktur blev observeret i de azimutale projektioner og radiale profiler under den cykliske belastningssækvens. Det blev derimod vist, at der sker store ændringer under de første cyklusser efter deformation i træk, muligvis pga. strukturel reorganisering som tilpasning til forholdet ved den cykliske deformation.


Den detaljerede karakterisering af mikrostrukturerne under cyklisk belastning ved hjælp af in-situ monitorering af den interne struktur af individuelle korn muliggør forståelsen af materialeopførsel under cyklisk deformation ved at frembringe eksperimentel data, der kan bruges til udvikling af modeller, som kan forudsige materialeopførsel.
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1 Introduction

Failure due to fatigue is still one of the major problems for structural materials, where the material often fails unexpected and abrupt after fluctuating stresses due to repeated periodically varying load over a long time. Still 90% of all mechanical service failures are found to be related to fatigue [ASM08], which gives enormous issues for the lifetime design and sustainability of the components. While steels and titanium alloys can be designed with a large buffer to an expected fatigue limit, no clear fatigue limit can be estimated for non-ferrous alloys such as aluminium alloys. The key for this lies in the metal microstructure and the microstructural development during cyclic deformation, which is changing drastically on the intragranular level. Many authors have tried early on to quantify the effects [BAS69, FEL67, MUG78], but in-situ experiments especially within the field of electron microscopy were difficult and hence the knowledge about the structural reorganization during cyclic deformation is still limited. It is however known, that dislocations are introduced in the metal during plastic deformation. During cyclic deformation these dislocations organize themselves in dislocation networks with dislocation-rich and dislocation-poor regions, which can be of influence for potential fatigue failure.

Earlier work using laboratory X-ray sources revealed the asymmetric character of radial X-ray peak profiles resulting from a heterogeneous dislocation structure and prevailing long-range internal stresses. [MUG83, UNG83] Because of various advantages such as the higher beam energy to penetrate larger samples and higher angular resolution, synchrotron techniques were applied for the present investigations. In particular High Resolution Reciprocal Space Mapping (HRRSM) was used in this work for investigations of embedded grains and subgrains in a macroscopic tensile sample with a high angular resolution to enable straightforward application of theories dealing with the microstructures from cyclic deformation. Due to local distortions of the crystalline lattice within an individual grain caused by the introduced dislocation structures, the intensity distribution of each individual diffraction peak is not completely smooth. These dislocation structures consist of dislocation-free subgrains separated by dislocation walls, where the subgrains cause sharp high-intensity peaks on top of a cloud of smooth intensity. To reveal this, hard X-rays were used to penetrate through a 1 mm thick tensile sample
mounted in a custom-made screw-driven load frame, while macroscopic stress and strain were monitored.

The following thesis aims to give further insight into the cyclic deformation behaviour of the fcc metal aluminium, which has barely been investigated until now.

The focus will be on in-situ investigations applying the synchrotron technique High Resolution Reciprocal Space Mapping (HRRSM) with the use of a custom-made load frame. Experiments were performed during one week of beam time at PETRA P07 (Hamburg, Germany) and three beam times each of one week duration at APS 1-ID-E (Argonne National Laboratory, IL, USA). During the project efforts were done to improve and further characterize the mechanical testing with the load frame to approve it for tension-compression testing after the first two beam times. The experiments were further improved from beam time to beam time and the thesis should be read with this in mind. The data analysis is based on X-ray line profile analysis principles established by Ungar et al. [UNG83, UNG01] and the composite model by Mughrabi et al. [MUG83, MUG02] with algorithms developed during an earlier Ph.D. project by Christian Wejdemann [WEJ11]. Large amounts of data were acquired during the experiments and only selected but representative results will be presented.

In the present thesis grains and subgrains embedded in a macroscopic tensile sample were investigated for the first time in detail during various conditions along a stress-strain hysteresis and during cycling. Besides the extensive analysis of results from synchrotron experiments, complementary studies on the cyclic deformation behaviour of the investigated aluminium samples including mechanical testing and metallographic analysis with DTU in-house equipment will be presented. HRRSM as diffraction technique can only give to limited information about the spatial arrangement. An outlook will present some first trials with the newly developed synchrotron technique Dark Field Microscopy, which is performed at ESRF (Grenoble, France) as a promising technique to also characterize the microstructural changes in 3D.
2 Background

2.1 Mechanical behaviour of fcc metals during cyclic deformation

2.1.1 Mechanical Deformation

During mechanical loading of metals, plastic deformation occurs on the microscale by motion of dislocations causing a fraction of dislocations to be stored in the material. When fcc metals are exposed to cyclic loading, a hardening (or softening) behavior can be observed, which is closely related to their initial microstructure. However, the structural behavior during cyclic deformation is still not documented in detail and not fully understood on the micro level.

2.1.1.1 Uniaxial Deformation

If a metallic component is deformed in uniaxial tension it responds in a characteristic way. The material will first deform elastically (reversible deformation) and when the stress is large enough, above the yield stress, plastic deformation will occur. The relation between the stress \( \sigma \) and the strain \( \varepsilon \) is described for the elastic case by Hooke’s law with the material specific Young’s modulus \( E \):

\[
\sigma = E \varepsilon \tag{2.1}
\]

Young’s modulus can be derived from the slope of the linear part of the curve for low stresses and strains, which is corresponding to the linear elastic regime. A higher modulus implies that more stress is needed for the same amount of strain. The elastic strain \( \varepsilon_{per} \), which is perpendicular to the loading axis can be connected to the axial strain by the Poisson ratio \( v \):

\[
\varepsilon_{per} = -\varepsilon v \tag{2.2}
\]
If an effective Young’s modulus is calculated with a strain different to the axial strain $\varepsilon$ it will differ from the effective Young’s modulus for loading conditions.

Presented values are engineering stress and strains, not taking the change of area during deformation into consideration.

2.1.1.2 Cyclic stress-strain hysteresis

Cyclic deformation is characterized by periodically repeated loads. Fatigue failures are defined as failures due to the application of varying stresses, which are much lower than the stress required for failure from just a single loading, over a large number of cycles [ASM08]. Failures due to fatigue show a characteristic fracture surface showing oscillations due to slow crack propagation.

An example of the stress variation for tension-tension and tension-compression cycling is shown in Figure 2.1. Tension-tension cycling is applied, when the load during cycling is always positive and of tensile character. Ideal tension-compression cycling is applied, when the load is reversed symmetrical around zero load and is both of tensile and compressive character in an equal way. Tension-compression cycling is the most common procedure for fatigue testing.

![Stress Strain Hysteresis Diagram](image)

**Figure 2.1:** Example for the stress fluctuation for (a) tension-tension and (b) tension-compression cycling. Reproduced from [ASM08].

During cyclic tension-compression deformation the material responds with a characteristic stress-strain hysteresis loop as shown in Figure 2.2. The sample experiences a characteristic maximum and minimum stress $\sigma_{\text{max}}$ under maximum tensile and compressive load. The total stress $\Delta\sigma$ (vertical height of the loop) and strain $\Delta\varepsilon$ (width of the loop) are the differences between the maximum and minimum stress.
and strain. The total strain $\Delta \varepsilon$ is the sum of $\Delta \varepsilon_{pl}$ for the plastic regime and the strain $\Delta \varepsilon_e$. The strain $\Delta \varepsilon_{pl}$ is defined as the difference of the strain measured at the maximum load and the strain measured for the extrapolation of the elastic region of the corresponding branch (as shown in the figure), which is the difference between the opposite maximum load and the extrapolated strain limiting the elastic region (following Hooke’s law). The Young’s modulus $E$ can be determined from the slope of the elastic regime in the hysteresis.

![Figure 2.2](image)

Figure 2.2: Example for a cyclic stress-strain hysteresis curve. The parameters for characterization of a cyclic hysteresis are marked. Reproduced from [ASM08].

The cyclic stress amplitude $\sigma_a$ is defined as half of the cyclic stress range $\Delta \sigma$, which is the difference between the maximum and minimum stress. The strain amplitude $\varepsilon$ is $\Delta \varepsilon/2$ in accordance to the definition of the stress amplitude $\sigma_a$. The strain amplitude is kept constant during the cycling, when the test is strain-controlled.

$$\sigma_a = \frac{\sigma_{\text{max}} - \sigma_{\text{min}}}{2} \quad (2.3)$$

$$\varepsilon_a = \frac{\varepsilon_{\text{max}} - \varepsilon_{\text{min}}}{2} \quad (2.4)$$
The origin of the shape of the hysteresis loop is described in more details in connection with the composite model in section 2.3.1. It is influenced by internal stresses causing the so-called Bauschinger effect, which states that the yield stress is lower after reversed load in comparison to the yield stress during the initial loading.

The stress ratio $R$ is the ratio between the minimum and maximum cylic stress $\sigma_{\text{min}}$ and $\sigma_{\text{max}}$ according to equation 2.5. For calculation of the stress ratio the maximum and minimum stresses of the hysteresis were used.

$$R = \frac{\sigma_{\text{min}}}{\sigma_{\text{max}}}$$

Changes of the stress amplitude during strain-controlled cycling are indications for microstructural processes such as hardening or softening. Is the stress-amplitude constant the material is in saturation. The hysteresis loops change in case of cyclic hardening or softening with successive cycles in a characteristic way as shown in Figure 2.3. The total stress $\Delta \sigma$ increases during hardening and decreases during softening. Low strength metals such as pure fcc metals show in general a cyclic hardening behaviour. However, softening can be observed as well. The behaviour can be observed in strain-controlled tests.

Figure 2.3: Typical stress response (left) and stress-strain response hysteresis (left) for (a) cyclic hardening showing an increase in the stress $\Delta \sigma$ (or strain amplitude $\sigma_a$) and (b) cyclic softening showing a decrease in the stress $\Delta \sigma$ (or strain amplitude $\sigma_a$). The number of cycles is written next to the hysteresis curves. Reproduced from [ASM08].
2.1.2 Cyclic response curves for fcc metals

The cyclic response of the discussed fcc metals copper, nickel and aluminium was found to show, in principle, similar stages of initial hardening, saturation, softening or secondary hardening for all materials but in different extends depending on the strain amplitude. The appearance of a saturation stage is however debated for aluminium. The following should give a short overview about the different cyclic responses.

The hardening or softening behavior is in general related to the dislocation microstructure, which will be introduced in detail in section 2.3. If the initial dislocation density is low, such as at the beginning of the cyclic deformation, the dislocation density increases with cycling and increases the stress required to deform the material. If the dislocation density is high, as in later stages along the cycling, the dislocations rearrange, it reduces the stress required and softening behavior can occur. The dislocation distribution is expected to be stable within the saturation stage.

Feltner et al. [FEL67] investigated the cyclic deformation behavior for annealed polycrystalline fcc metals. They stated that the hysteresis loop for materials with a medium to high stacking fault energy such as copper and aluminium are symmetrical with respect to both, stress and strain. The hardening rate has been found to depend on the cycling strain amplitude and decreases with a decreased strain amplitude [FEL67], which has also been shown by various authors for the different fcc metals. [POL84, BUQ01, LI10]

2.1.2.1 Copper

As shown in Figure 2.4 Polak et al. [POL84] presented cyclic response curves for polycrystalline copper with an average grain size of 30 µm cyclically deformed with different strain amplitudes. All specimens reveal an initial hardening stage. The hardening stage is occurring during a higher number of cycles for lower strain amplitudes. A saturation stage (with constant stress) follows the initial hardening. For strain amplitudes up to $2 \cdot 10^{-3}$ softening occurs after the saturation stage. For higher strain amplitudes a second hardening can be observed after the saturation stage. At very high amplitudes only initial hardening and saturation is observed.
Mughrabi et al. [MUG78] observed three regimes for the cyclic saturation stress (cf. Figure 2.6a) in dependence of the cycling amplitude in a range of $1.55 \times 10^{-5}$ to $2 \times 10^{-2}$. These show an increase of saturation stress due to initial work hardening behaviour for low strain amplitudes and again for high amplitudes and a clear intermediate plateau for medium strain amplitudes between $6 \times 10^{-5}$ to $7.5 \times 10^{-3}$, where PSBs were formed.

### 2.1.2.2 Nickel

Three stages were identified in the cyclic deformation curve of polycrystalline nickel as well. They are shown in Figure 2.5, where the relation of the saturation stress to the plastic strain amplitude is shown in a cyclic stress strain curve [BUQ01]. Nickel experiences an increase of the saturation stress (hardening) for low strain amplitudes, then a saturation steady-stage at medium deformation amplitudes and again a second hardening stage with further stress increase with increasing strain amplitudes.
Figure 2.5: Cyclic deformation behaviour of nickel visualized by the dependence of the saturation stress $\sigma_{sat}$ in dependence of the plastic strain amplitude $\varepsilon_{pl}$. Reproduced from Buque et al. [BUQ01].

2.1.2.3 Aluminium

The cyclic work hardening and softening behavior of aluminium has been studied in detail by Snowden et al., Vorren et al. [SNO63, VID96a, VOR87, VOR88] and recently by Li et al. [LI10] and Nellesen et al [NEL16] for single crystals and Videm et al. [VID96b] and Madhoun et al. [MAD03] for polycrystals. Figure 2.6a shows cyclic stress-strain curves, which can be observed for pure aluminium single crystals during cycling. In general the cyclic loading response of Aluminium single crystals was stated to show a hardening-softening-secondary hardening behavior, where the degree of softening and secondary hardening has been shown to be slightly dependent on the strain amplitude and the crystallographic orientation with respect to the loading axis. The cyclic hardening curve is shown in Figure 2.6b and shifted to higher stresses with increasing plastic strain amplitude. The initial cycling hardening stage is also slightly shorter. [VID96a, LI10] A detailed analysis of the cyclic hardening behaviour for varying strain amplitudes was made by Videm et al. [VID96b].
Figure 2.6: (a) Comparison of the cyclic stress strain curves for different oriented aluminium single crystals and copper single crystals. The classic CSS curve for Cu established by Mughrabi et al. [MUG78] shows a distinct and stable plateau, which is not clearly visible for Al. (b) Cyclic hardening curves of [576] single crystals at different strain amplitudes. The CSS curve is shifted to higher stresses with increasing strain amplitude. Reproduced from [LI10]

Videm et al. [VID96b] presented results for cyclically deformed polycrystalline aluminium, though with rather large grain sizes of in average 110 µm, cf. Figure 2.7. For polycrystalline aluminium a rapid initial hardening was followed by a softening. For high strain amplitudes (from $1.1 \cdot 10^{-4}$) a second hardening before failure was observed. Videm et al. states as well that a saturation stage does not exist for polycrystalline aluminium, instead continuous softening followed by secondary hardening was observed.
Figure 2.7: Cyclic hardening curve for polycrystalline aluminium for different strain amplitudes showing initial hardening, softening and secondary hardening. Reproduced from [VID96a].

Madhoun et al. [MAD03] has investigated more recently the cyclic hardening behaviour for polycrystalline aluminium with an average grain size of 40 µm for different strain amplitudes and reveals a saturation stage. All curves (cf. Figure 2.8) show an initial rapid hardening during the first few cycles. The hardening stage is extended over a larger number of cycles for lower strain amplitudes. After the initial hardening the hardening rate decreases and the stress becomes nearly constant. Though the highest strain amplitude reveals possibly a softening after the initial hardening. Madhoun's results differ clearly in the extend of the initial hardening stage and in the absence of secondary hardening from the curves presented by [LI10] for single crystals, which is due to the small number of investigated cycles.

An increased cyclic strain hardening was stated to be the result of the operation of multiple glide systems at higher strain amplitudes. The activation of a secondary slip system will cause the formation of stabilizing dislocation structures and with this an increase in the stress amplitude. These observations are similar to the ones presented for copper.
A similar cyclic hardening behavior was presented for aluminium single crystals oriented for double slip by Nellesen et al. [NEL16] for the first 100 cycles with a displacement amplitude of 20 µm, cf. Figure 2.9. It is shown that depending on the crystal orientation and stress amplitudes the initial hardening takes less than 25 cycles and is followed by softening before the saturation stage is reached from approximately 50 cycles on. This also shows that the initial microstructural changes can appear very early on for aluminium, which is one of the major differences to the other fcc materials.
2.2 Dislocation arrangements in fcc metals after cyclic deformation

The research on cyclic deformation and fatigue has been of interest for more than 150 years. Feltner et al. [FEL67] distinguished the response of materials on cyclic deformation and introduced so-called wavy slip and planar slip materials. Wavy slip materials are materials with easily occurring cross slip often (but not exclusively) due to a high stacking fault energy. A well-known diagram shown in Figure 2.10 from Mughrabi et al. [MUG10] shows that materials, which have a high tendency for cross-slip (so-called wavy slip materials such as copper and aluminium) can develop dislocation cell structures during cyclic deformation with a relatively low strain amplitude before so-called persistent slip bands develop. Persistent slip bands as described in the next section are observed for higher strain amplitudes. Mixed structures occurring for high planar slip for the transition regions between cell structures, persistent slip bands and planar arrangements.

![Figure 2.10](image)

**Figure 2.10:** Dependence of the characteristic microstructure developing during cycling deformation in dependence of the tendency for planar slip and cycling strain amplitude. Reproduced from [MUG10].

Persistent slip bands (PSB) and cellular structures consist, in general, of regions free of dislocations, which are the cell interiors, and dislocation-rich areas separating the cells, which are the so-called dislocation walls. The walls are parallel for PSB structures. The mechanical implications of this heterogeneous dislocation distribution will be discussed in detail in relation to the composite model in section 2.3.1, which was developed based on the observation of PSBs. A schematic example for an
idealized cell structure is given in Figure 2.11. When axial load is applied the dislocation-free cell interior perpendicular to the loading axis and the dislocation-rich walls parallel to the loading axis experience different stresses. In general regions with a lower dislocation density like the cells yield easier than areas with large dislocation densities like the walls, which have a higher resistance against plastic deformation. When the critical shear stress of the slip systems in the cells is reached, mobile dislocations will glide. Dislocations gliding in the cells are stopped at the walls and stored at the interface as seen in Figure 2.11a. They will then induce elastic stresses in the wall component and the cell interiors, while reducing the stress in the cells, and ensure in this way compatible deformation. The stresses are forward in the walls and backward in the cells.

![Figure 2.11: Idealized sketch of a [001]-oriented single crystal with a heterogeneous dislocation structure consisting of dislocation rich walls and dislocation-poor cell interiors under axial deformation, (a) \( b_{\text{res}} \) is the resulting Burger vector of the individual Burgers vector for the two slip systems indicated with straight lines. (b) Cells and walls experience different internal stresses indicated by arrows (compressive stresses in cells and tensile stresses in walls). Reproduced from [MUG02].](image)

These structure were found to develop mainly under the conditions of multiple slip for uniaxial loading. Similar structures are obtained in cyclic deformation conditions under single slip and multiple slip. Typical examples are fcc metals like copper, nickel and aluminium.

Heterogeneous dislocation structures can develop in the later stages of fatigue as so-called persistent slip bands or ladder structures, which cause surface roughness and with this possible crack initiation
and nucleation points. The term persistent slip bands is used confusingly both for dislocation structures and the resulting surface structures. They are called persistent, because the surface structure reappears after polishing the initial structure away during new cyclic deformation. In this way the fatigue life of investigated samples containing PSB was prolonged by removing the surface layer [THO56]. It is hence of importance to gain more knowledge about the formation of dislocation structures characteristic for cyclic deformation inside grains.

2.2.1 Cyclic Deformation in Copper

One of the classical materials developing these characteristic structures is copper, which has been investigated intensively [e.g. by MUG78, WIN81, UNG83, MUG83, POL84, LIU94, HUA01, WEJ13]. The dislocation structures will be described, since the composite model has been developed based on investigations of the dislocation structures for copper.

The term persistent slip band is in connection with dislocation structures used to describe a ladder structure consisting of channels separated with a typical wall spacing of approximately 1.5 \( \mu \text{m} \), which are believed to accommodate plastic deformation during cyclic hardening. In the following the term ladder-structure will be used for the characteristic dislocation structures consisting of a dislocation-free cells separated by parallel narrow walls of high dislocation density. The surface appearance will be designated as PSB.

Winter et al. [WIN81] tested copper polycrystals with grain sizes of 100 – 300 \( \mu \text{m} \) at low and high strain amplitudes into saturation. The most prominent type of dislocation structure observed were ladder structures. It was found that few ladder structures were observed in samples tested with low strain amplitudes \((1.1 \cdot 10^{-4}\text{ and } 3.1 \cdot 10^{-4})\) at a small fraction of the fatigue life (cf. Figure 2.12a) and the amount was increasing with increasing strain amplitude and number of cycles (cf. Figure 2.12b). The ladder-structures were found to usually be parallel to the \( \{111\} \) plane but not always aligned with the primary slip plane (marked as P in the figures by [WIN81]). The matrix around the ladder-structures contains irregular dislocation free regions (or channels, bright) separated by dislocation-rich arrays (dark). PSBs
were observed to cross low-angle boundaries without major modifications but terminate at high-angle grain boundaries. At higher strain amplitudes (>5.1·10^{-4}) the ladders were found following more than one slip plane indicating the presence of multiple slip (cf. Figure 2.12).

![Image](image1)

**Figure 2.12:** Persistent slip band (PSB) observed in TEM foils prepared from cyclically deformed copper polycrystals (a) following primary slip plane in a sample fatigued at low strain amplitudes (1.1·10^{-4}). (b) A large amount of PSB following the (111) planes in a sample fatigued at high strain amplitude (6·10^{-4}). (c) Several PSB following both the primary and the secondary slip plane in a sample fatigued at high strain amplitude (6·10^{-4})

The primary slip plane is marked with P, the secondary slip plane with S, the tensile axis with T. Reproduced from [WIN81].

Next to the ladder-structures and the surrounding matrix structure, other structures were found to a large extend in the samples fatigued at high strain amplitudes. Cellular structures with misoriented cells
(cf. Figure 2.13a) and labyrinth structures (cf. Figure 2.13b) were observed. Both structures are assumed to have formed under multiple slip conditions. The misorientations of the cells are parallel to one axis and the cell size is around 1 µm. The direction of the walls is more random and they do not necessarily follow the \{111\} planes like the PSB. Labyrinth structures have not been observed for copper single crystals. However this inclination of the walls of cells and labyrinths follows roughly the appearance of PSBs.

The appearance of these structures is usually related to the crystallographic direction along the tensile axis in the standard stereographic triangle (cf. Figure 2.13). The structures in copper are not clearly be related to the direction of the tensile axis, which indicates that the neighbourhood plays a strong role (eventually stronger than the orientation of the loading axis) on the internal stresses and with this on the developing dislocation structures inside a grain. [WIN81]
Figure 2.13: (a) Cell structures with large misorientations (recognizable by different shades of grey due to orientation contrast) between the cells and (b) labyrinth dislocation cell structure observed in TEM foils of copper after cyclic deformation at high strain amplitudes. (c) Standard stereographic triangle showing the relation between grain orientation and characteristic dislocation structures. No clear systematic was found in copper. Reproduced from [WIN81]

A dislocation free zone of circa 1 µm size was observed next to high-angle boundaries. It was suggested that this zone accommodates plastic strains from the neighbouring grains, since its width is similar to the channel width of PSB. [WIN81]

2.2.2 Cyclic Deformation in Nickel

It has been found that the appearance of dislocation structures can be dependent on the crystallographic orientation of the individual grains. This was investigated in detail by Buque et al. for Nickel [BUQ01]. The saturated microstructure developed during cyclic deformation of pure nickel at room temperature was investigated applying EBSD and ECCI imaging in the SEM to correlate the grain orientation with the spatial appearance of the grain and the surrounding neighbourhood. The material was recrystallized to grain sizes of 35 µm and texture-free with random distribution of the grain orientation.

Grains were investigated after cyclic loading with two different strain amplitudes $\varepsilon_a = 5 \cdot 10^{-4}$ and $\varepsilon_a = 1 \cdot 10^{-3}$ and a relation of the appearance of the dislocation structure to their crystallographic direction
along the loading axis was observed. The authors differ in general between a fragmented wall structure (with loading axis near [111]), a labyrinth structure (for grains with loading axis near [100]) and a patch structure for (with loading axis near [011]). A bundle structure has been observed for all orientations in between. These structures were observed both for surface and bulk grains and can be related to regions within the standard stereographic triangle (SST) as shown in Figure 2.14. Rather sharp boundary lines distinguish the regions within the triangle, where the different types of structures appear. It has been observed that an increase in the strain amplitude causes a condensation (meaning a reduction of the wall thickness, cf. Figure 2.14b) of the structures and a development of a more clear cell structure for the bundle and patch structure. The bundle configuration can be compared to ladder structures observed in crystals oriented for single slip. First, the configuration has a vein-like shape with dislocation-free channels, which transforms into a ladder structure (with 1 µm wide cells) parallel to the primary slip system in a amplitude range of \( \varepsilon_a = 1 \cdot 10^{-4} \) to \( \varepsilon_a = 1 \cdot 10^{-3} \). For amplitudes between \( \varepsilon_a = 1 \cdot 10^{-3} \) to \( \varepsilon_a = 2 \cdot 10^{-3} \) it turns into a cell structure, whereas only (equiaxed) cells are observed after cycling with \( \varepsilon_a = 5 \cdot 10^{-3} \).

![Figure 2.14](image.png)

Figure 2.14: Typical dislocations structure found for differently oriented grains in nickel polycrystals after cycling with (a) \( \varepsilon_a = 5 \cdot 10^{-4} \) and (b) \( \varepsilon_a = 5 \cdot 10^{-3} \) visualized in the standard stereographic triangle. After [BUQ01].

The effect of the strain amplitude on the amount of dislocation-rich regions (referred to as DR in figure) and dislocation poor areas such as channels and cell interiors (referred to as PR in figure) has been
investigated in detail and is displayed in Figure 2.15. This shows a clear decrease of the width of the dislocation-rich regions with increasing strain amplitude since the dislocation poor regions on the other hand remain nearly constant. The general tendency is the same for all observed dislocation structures.

Figure 2.15: Size of (a) dislocation-rich (DR) and (b) dislocation-poor (PR) regions in relation to the plastic strain cycling amplitude. The general tendencies (grey) are independent of the specific structure type, which is indicated by different symbols. Reproduced from [BUQ01].

The authors suggest that the intrinsic properties of the grains are having a dominant effect on the intragranular changes during cyclic deformation. This has been indicated by the close relation between crystallographic direction along the loading axis and the developing dislocation structure, strong differences of the structure within one grain in dependence of the experienced strain amplitude and a similar behaviour of bulk and surface grains (and with this a missing effect of the free surface). Contradictory were dislocation patterns associated with multiple slip (ladder and cell structures) observed in a regions, where single slip is expected for a single crystal. This indicates that the dislocation patterns developing in a grain in a polycrystal are different than in a single crystal, if both have a similar orientation towards the loading axis. [BUQ01]

2.2.3 Cyclic Deformation of Aluminium

While extensive and detailed research has been performed on fatigued copper, only limited and inconsistent characterization work has been done on pure aluminium exposed to cyclic deformation.
Dislocation structures in aluminium have been described and designated inconsistently in literature. These inconsistencies are often due to the limited characterization techniques available in the early time, where much of the research on fatigued fcc metals was performed.

Due to its high stacking fault energy (SFE), aluminium has a special role within the group of fcc metals such as Cu, Ni and Ag and therefore shows different dislocation patterns and a different cyclic hardening behavior, which has been pointed out by several authors [GRO63, CHA87, VID96b, KON02, LI10].

In addition to the observed differences in the cyclic response between copper and aluminium differences in the surface slip morphology as well as in the appearances of dislocation patterns are visible in aluminium when compared to Cu. This has been in focus in most of the publications on cyclically deformed aluminium [KIN67, MIT68, ARN69, MIT69, MIT70, VOR87, VOR88, ZHA95, VID96b, ZHA96b, ZHA96c, LI10].

The typical slip morphology on the surface of a cyclically deformed aluminium crystal is characterized by intrusions and extrusions at low strain amplitudes and is thus similar to what is observed for other fcc metals (cf. Figure 2.16a). The deformation can reach a saturation at higher strain amplitudes, which is visible in an increasing distortion and deformation of the slip bands. The increased surface roughness is seen as one of the major factors for fatigue fracture. Depending on strain amplitude, the surface patterns have also been described as cord-like (low strain amplitudes) and tweed-like structures (high strain amplitudes) for fatigued [001] Al single crystals [VID96b].

![Figure 2.16: (a) Typical surface morphology of cyclically deformed Al at low strain amplitudes showing a wavy slip structure with intrusions and extrusions, similar to other fcc metals such as Ni and Cu. Reproduced from [LI10].](image-url)
Figure 2.17: Crystallographic direction along the loading axis of grains showing different surface features, reproduced from [ARN69].

Figure 2.17 shows an overview of the relation between surface morphology and grain orientations in fatigued polycrystalline aluminium published by Arnell et al. [ARN69]. Different symbols are related to different appearances of striations on the surface. When the loading axis moves from the center of the stereographic triangle towards the corners, the surface features change from conventional fatigue striations to other structures, which do not show signs for concentrated slip on any particular plane, as observed by King et al [KIN67]. The planes on which the features develop are consistent with systems of high resolved shear stress. The right side triangle in figure 3 shows, that the surface orientation of the grains have only a small effect on the type of surface feature. [ARN69]

It has been shown by several authors [MIT69, MIT70, VOR87, VOR88, CHA89, ZHA96a], that the appearance of intense slip banding at the surface corresponds to the formation of dislocation cell structures. Since Al has a high SFE, cross slip can continuously occur during cyclic deformation and supports formation of cell-like structures. Cell structures are the major dislocation arrangement for cyclically deformed aluminium single crystals independent of their orientation, since it has been observed for Al single crystals with various orientations such as [001]Al, [-579]Al and [-123]Al. [LI10]

Mitchell et al. have investigated dislocation structures below the surface showing striations in TEM foils of polycrystalline aluminium after cyclic deformation and found a correlation between the surface markings and internal dislocation structures [MIT69, MIT70]. Foils cut parallel to the surface have been investigated to obtain a correlation between the markings at the surface and the underlying dislocation structures. When surface markings are only visible along the primary slip plane, diffuse but aligned
dislocation clusters can be observed below the surface. Further cycling produces irregular deformation bands and a distinct dislocation cell structure in the material. Cell structures in polycrystalline Al have been observed in the earliest publications at high stress amplitudes (with fatigue life less than $10^5$ cycles such as the strain amplitudes applied in the HRRSM experiments) and were described as fatigue-induced substructures [SEG59, GRO63a, GRO63b] or early stages of the ladder structures known from copper [MUG01]. The dislocation structures become gradually more pronounced during the first hundred cycles. Once they are established they remain stable. [GRO63, SNO63, MIT69, MIT70] This in accordance with the appearance of a small initial hardening stage in the cyclic hardening curves presented above.

In general random dislocation distributions (cf. Figure 2.18a+b) are the dominant observation at low number of cycles low strain amplitudes. The development of dislocation tangles and patches to cell structures (cf. Figure 2.18c) and the increase of dislocation density with the development of cyclic hardening has been observed by several authors using TEM imaging of thin foils [SNO63, GRO63a, GRO63b, MIT68, MIT69, MIT70, CHA87, VOR87, VOR88, ZHA96a, VID96, LI09, LI10], but the terms used for the observed intermediate dislocation structures are often inconsistent. Grosskreutz et al. discuss that, until a certain cycling level is reached, the observation of dislocation structures and the type of substructure is dependent on the grain orientation with respect to the tensile axis in polycrystalline Al. Hence more favorably oriented grains start earlier to form heterogenous dislocation arrangements such as tangles or dislocation clusters (after 500 cycles) or cell-structure (after 2000-3000 cycles). [GRO63b] Grains oriented for multiple-slip will most likely start to form cell-structures first [SEG59, GRO63a, GRO63b, SNO63].
Figure 2.18: TEM images of (a) dislocation tangle and dislocation loops in the active glide plane in a very early stage of cyclic deformation, (b) dislocation tangles and loops after progressed cycling start to form loop patches [GRO63b], (c) dislocation tangles spread out in diffuse bands parallel to the glide plane after further cycling. Reproduced from [SNO63].

Videm et al. correlated the appearance of dislocation structures with the stages observed in the cyclic hardening curve. During the primary hardening stage the previously described heterogeneous dislocation distribution consisting of irregular dislocation tangles can be observed. The dislocation density increases with increasing stress. The dislocation distribution becomes more regular during the softening stage. With continued cycling the dislocations begin to cluster into bundles and form a well-defined wall structure with walls parallel to \(\{100\}\). Labyrinth structures have been observed in this stage as well. During the secondary hardening, walls begin to break down and cell structures develop. Segall et al. and Grosskreutz et al. describe the formation of cell structures as the formation of subgrains during fatigue at high stress [SEG59, GRO63a, GRO63b]. Cell structures develop at the end of the softening stage or the beginning of the secondary hardening stage [GRO63a, VOR88, LI10].

As described before, cell structures appear in later stages of fatigue and are often associated with the formation of a surface pattern. Grosskreutz et al. have shown that cellular structures can develop after 3000 cycles at at strain amplitude of \(2 \cdot 10^{-3}\). The probability of cell structure or subgrain formation increases with higher strain amplitudes and higher number of cycles. [GRO63b] A combination of cell structures and dislocation patches can be found at low strain amplitudes, depending on the grain orientation (cf. Figure 2.18). [GRO63a] Figure 2.19 shows example of clear cell structures observed in TEM foils. Differences in orientation contrast indicate misorientations between cells in the dislocation
cell structures. It has been observed that the misorientations increase with the number of cycles due to further plastic deformation, where dislocations accumulate in cell boundaries [GRO63b, MIT70] in accordance to observations for copper cell structures by Winter et al. [WIN81]. Cell structures after $8 \times 10^5$ cycles show a significant increase in misorientations in comparison to cell structures observed after $2 \times 10^5$ cycles [MIT70]. Apart from these findings, no big attention has been put on characterizing the misoriented cell structures in aluminium or the measurements of misorientations.

Li et al. observed cellular structures in a tested Al [-123][-455] bicrystal after $10^4$ cycles at room temperature. After further cycling ($2 \times 10^6$ cycles), these structures developed into clear ladder-like structures (cf. Figure 2.20a). This type of behavior has also been found by Zhai et al. (cf. Figure 2.20b) and Vorren et al. (cf. Figure 2.20c) [VOR88, ZHA96a]. The dislocation cells in the ladder structures observed by Li et al. also show significant differences in orientation. The spacing in the elongated structures was approximately 5 µm, which is two times the spacing observed in wall structures of polycrystalline Al fatigued at 77K (2.8 µm) and much higher than the wall spacing observed for copper at room temperature, which is circa 1 µm [CHA87]. Zhai et al. observed spacings of 4 - 10 µm for cells in aluminium [ZHA96a]. The striking long walls have been described as persistent slip lines (PSL), since

Figure 2.19: TEM images of foils prepared from aluminium after cyclic deformation showing (a) an elongated cell-structure in polycrystalline Al after $2 \times 10^6$ cycles at $\varepsilon=3 \times 10^{-4}$. Loop patches are visible at A. Reproduced from [GRO63a] (b) a clear cell structures showing differences in orientation contrast associated with misorientations after fatigue at room temperature for $2 \times 10^5$ - $8 \times 10^5$ cycles, reproduced from [MIT70].
they appear on the surface of the crystal (cf. Figure 2.21a). Figure 2.21 shows diagrams of the surface pattern in Al and Cu due to the position of ladder-like structures below the surface. Li et al. describe a sharper slip line pattern at the surface in Al compared to Cu created by the striking dark lines (PSL) [LI09]. The observation of dislocation walls associated with the slip lines occurring on the sample surface has been described by Videm et al. as well [VID96a].

![Figure 2.20: Ladder-like structures in an (a) Al bicrystal fatigued at 2·10⁶ cycles, reproduced from [Li09]. (b) Al single crystal fatigued at 5·10⁶ cycles, reproduced from [ZHA96a], (c) Al single crystal after cycling at ε = ±7.1·10⁻⁴ until after the secondary hardening had started, reproduced from [VOR88].](image)
Figure 2.21: Schematic diagram of surface profiles and the dislocation structures in (a) Al, (b) Cu, reproduced from [Li09].

In general it was found by [CHA87] that the appearance of the dislocation structure can vary dependent on the temperature. Figure 2.22 gives an overview about experimentally observed dislocation structures as a function of temperature showing that different structures can be formed for a given set of conditions. The observed dislocation structures are basically categorized by the same types in Al and Cu. At temperatures below room temperature vein and labyrinth structures were observed in aluminium after cyclic deformation. The dominant dislocation structure in aluminium was found to be a three-dimensional cell structure. Dislocations of the secondary slip system are more moveable at higher strain amplitudes and contribute to the observed structures, which makes a clear distinction of the structures in aluminium at high strain amplitudes easier than at lower strain amplitudes.
Koneva et al. categorized metals in regard to their typical dislocation structures appearing during cyclic deformation. [KON02] They place the observed “chaotic” (in comparison to copper) dislocation substructures in aluminium together in a group with observations for aluminium alloys (AlMg) and certain types of austenitic steels. Koneva et al. separates them clearly from those dislocation structures observed in Cu and Ni. Aluminium dislocation structures are described as a chaotic distribution of dislocations with dislocation loops of a small diameter, which is in accordance to descriptions of other authors for early stages of fatigue, and the development of cellular substructures with further loading as it has been described above. The cell walls in single crystals often follow certain directions thus the cell structures are not isotropic throughout the whole crystal. Misorientations appear at cell walls with an increased number of loading cycles. Figure 2.23 shows a sketch of the dislocation substructures typically observed for single and polycrystalline aluminium and typical structures seen in Cu after Koneva et al. [KON02]. It has to be noted that various authors have described clear observations of patchy substructures and well-defined wall structures or labyrinth structures for aluminium as well. The chaotic distribution of dislocations has often been described as tangles or clusters. Hence the grouping into characteristic dislocation structures described by Koneva et al. is only in limited accordance to the descriptions above, but emphasizes the difference in cyclic deformation behavior in comparison to Cu.
Figure 2.23: Schematic sketch of dislocation distributions appearing during cyclic deformation typical for (1) Al and (2) Cu, reproduced from [KON02]. (1) a: chaotic, b: cellular, c: clear subgrain substructures; (2) a: tangles, b: veins, c: walls, d: labyrinth, e: patchy substructures.

In summary, the slip mode of Al and the observed dislocation structures differ from those observed in other fcc metals, which is mainly related to a higher SFE increasing the probability of secondary slip and cross slip. Ladder-like PSB structures as typically observed for copper have only been observed in limited ranges of temperature and for high strain amplitudes [CHAR89, LI09]. Dislocation structures have been designated inconsistently in literature and cell structures have been wrongly described as ladder-structures [CHA88]. It has been discussed that aluminium shows a number of similarities, but also some differences in comparison with copper. However, no clear grouping of dislocation pattern in relation to grain orientation and strain amplitude such as it has been done for nickel [BUQ01] and copper [WIN81] has been published yet.
2.3 Heterogenous dislocation structures - the Composite Model

2.3.1 Model description

As described above, fcc metals including the investigated aluminium, are developing characteristic dislocation structures during cyclic deformation. The most common type are three-dimensional cell structures, which consist of dislocation-rich walls and dislocation-poor cell interiors called subgrains. The composite model has been derived from investigations of the dislocation cell structures (in form of persistent slip bands) developed in cyclically deformed copper.

To describe plastic deformation and the internal elastic strains arising from these heterogeneous dislocation structures, the concept of the composite model has been proposed. The aim of the model is to describe the local mechanical properties inside a grain following the compatibility requirements during plastic deformation. It was found that the curvature of dislocations varies spatially in a characteristic way in channels of persistent slip bands (PSB) in copper. For this to be possible, the local stress has to vary inside the channel. Based on these observations the composite model was formulated for (single slip) heterogeneous dislocation structures similar to PSBs based on the idea that a crystal containing a heterogeneous dislocation structure responds like a two-component composite material to external stresses. In grains with a heterogeneous internal structure the macroscopic applied stress $\tau$ is redistributed on a local scale. The dislocation-rich regions are seen as hard regions and the dislocation-poor regions (which are later called subgrains) as soft regions, where the flow stress is higher than the applied stress for hard and lower for soft regions. The macroscopic flow stress $\tau$ can be calculated by the weighted local flow stresses. Following the rule of mixture and with this the essence of the composite model, the macroscopic flow stress is the sum of the local flow stresses of the walls $\tau_w$ and cells $\tau_c$ connected by their volume fraction $f_c$ and $f_w$ (cf. eq. 2.6). The sum of the volume fractions of the cells and walls has to be one, cf. eq. 2.7.

$$\tau = \tau_c f_c + \tau_w f_w$$  \hspace{1cm} (2.6)
\[ f_c + f_w = 1 \] (2.7)

Mughrabi’s composite model [MUG83] has first been formulated for single slip after the observation of deformation induced peak profile broadening and is valid in a similar way for structures caused by multiple slip, where the shear stresses \( \tau_c, \tau_w \) can be replaced in the equations by the normal stresses \( \sigma, \sigma_c, \sigma_w \). The composite model for multiple slip was developed by to the observation of asymmetric peak profiles as discussed later in section 2.3.2.

The Taylor-type flow stress relation is formulated for the local flow stresses \( \tau_w \) and \( \tau_c \). They are related to the locally varying dislocation densities of the cells \( \rho_c \) and walls \( \rho_w \) (eq. 2.8 and 2.9) with the geometrical constants \( \alpha_c \) and \( \alpha_w \) equal to \( \alpha = 0.4 \) [PUS82].

\[ \tau_w = \alpha_w Gb \sqrt{\rho_w} \] (2.8)

\[ \tau_c = \alpha_c Gb \sqrt{\rho_c} \] (2.9)

Figure 2.24 visualizes the yielding of the walls and cell interiors (referred to as channels by Mughrabi) as expected by the composite model when applying macroscopic stress both for the initially described case of single-slip (cf. Figure 2.24a) and multiple slip for a stress-strain hysteresis (cf. Figure 2.24b). Figure 2.24s shows the behaviour of the wall and subgrain component during tensile loading and unloading. First both the cell interiors and the walls deform elastically. Then the soft cell interiors begin to yield plastically, while the hard walls are still deformed elastically (microyielding). During further loading into tension, the local stress in the cells stays at the constant level \( \tau_c \) due to the assumed ideal plastic behaviour. On the other hand the stress in the walls increases above the applied stress while equation 2.6 is valid.
This is achieved by local internal stresses given by equation 2.10. The average of the internal stresses vanishes as given by equation 2.11.

\[
\Delta \tau_c = \tau_c - \tau \quad \text{and} \quad \Delta \tau_w = \tau_c - \tau \quad (2.10)
\]

\[
f_c \Delta \tau_c + f_w \Delta \tau_w = f_c \tau_c - f_c \tau + f_w \tau_c - f_w \tau = \tau - (f_c + f_w) \tau = 0 \quad (2.11)
\]

If the stress is high enough the walls begin to yield plastically as well and both components are deforming plastically (macroyielding). When the sample is unloaded to zero load assuming the stresses are preserved, the internal stresses are revealed clearly. Then the stresses of the components remain differently being positive for the walls, which experience the so-called forward stress \( \Delta \tau_w \), and negative for the cells, experiencing the so-called back stress \( \Delta \tau_c \). The magnitude of the stresses is defined by equation 2.12. As a consequence the channels and walls will begin to yield at the same stress level plastically during second loading and at exactly the same stress where unloading started.

\[
0 = \Delta \tau_c f_c + \Delta \tau_w f_w \quad (2.12)
\]

The composite model can describe cyclic deformation as well. The yielding behaviour of walls and cell interiors during loading into compression is shown in Figure 2.24b. If the load is reversed the same behaviour as initially discussed for the first loading into tension is observed. During loading into compression both components deform elastically, before the cell interiors begin to yield first. At the macroscopic yield point, plastic yielding of the walls and with this common plastic yielding of cells and walls is observed. The presence of internal stresses is a possible explanation for the Bauschinger effect, where the yield stress is lowered after reversing the load direction due to internal compressive stresses (after initial tensile deformation) or internal tensile stresses (after initial compressive deformation) for the cells. The Bauschinger effect is giving the characteristic shape of the stress-strain hysteresis for cyclic deformation as shown in Figure 2.24b. During the loading into compression internal stresses are reduced to zero (after the cells begin to yield and before yielding of the walls, corresponding to the point of intersection in Figure 2.24b). The internal stresses will then during loading into tension built up again, but with an opposite sign. The hysteresis will repeat itself in the following cycles.
Figure 2.24: (a) Schematic mechanical response of the dislocation-rich walls and the dislocation-poor cells (here: channels) (top) on macroscopic stress (bottom), reproduced from [MUG83]. Walls and cells yield at different stresses $\tau_c$ and $\tau_w$ and the residual stresses are different ($\Delta\tau_w$ and $\Delta\tau_c$), when unloading the sample. (b) The example applied for cyclic deformation showing the yielding behaviour of cells and walls during loading into compression. Here the axial stresses $\sigma_w$ and $\sigma_c$ and the axial strain $\varepsilon$ are used. Reproduced from [MUG02].

2.3.2 Experimental evidence of long-range internal stresses

It has been discussed that the origin of long-range internal stresses, as the residual forward and back stresses are usually referred to in literature, is due to interfacial dislocations accommodating the elastic-plastic strain mismatch of the soft cells and hard walls. As explained in the beginning of the chapter, dislocations resulting from the initial gliding within the subgrain are stored at the interface due to geometrical constraints. It is assumed that the strain mismatch between the cells and walls is accommodated by these so-called misfit dislocations, which are dislocations located at the cell-wall boundary. These interface dislocations are expected to accumulate, when the cells are yielding
plastically before the walls begin to yield plastically as well. The most common tool to characterize stresses arising in the crystal lattice has been X-ray diffraction, where local internal elastic strains can be observed by a variation in the diffraction angle due to local changes of the lattice parameter.

2.3.2.1 Basics of X-Ray Diffraction

X-rays are electromagnetic waves, which a wavelength of 0.01 – 10 nm. For classical X-ray diffraction, X-rays of low energy (< 10 keV) produced by X-ray tubes are used. The produced X-ray spectrum consists of a continuous spectrum so-called Bremsstrahlung (braking radiation) and characteristic X-rays, corresponding in energy to electron jumps from an outer to an inner shell (in Bohr’s atom model with discrete energy levels for electrons).

Hard X-rays are usually characterized by their low wavelength and high energy (larger than 10 keV). They are typically produced by synchrotron sources, where electrons are accelerated to high speed and forced to change their direction periodically by various types of magnets (e.g. insertion magnets, bending magnets and focussing magnets) inside a storage ring. Insertion devices or so-called undulators are a set of magnets, which force the electrons to follow an oscillatory trajectory with small amplitudes. The emission of X-rays occurs in phase and the radiation from each bend interferes with those from the following bends producing in this way a more intense and nearly monochromatic beam with low bandwidth.

X-Ray diffraction is a well-established analytical technique to study the crystal structure of a crystalline material (e.g. for identifying different phases, texture) and for lattice strain analysis. It is based on the interaction of X-rays, which are scattered elastically on the crystal lattice coherently in an angle 2θ as shown in Figure 2.25.
Figure 2.25: Sketch describing an incoming X-ray wave with wave vector $k_{in}$, which is scattered elastically on an object with a scattering angle $2\theta$. This is resulting in an outgoing beam with wave vector $k_{out}$ and a scattering vector $q$. The length of the incoming and outgoing wave vector is $|k| = 2\pi/\lambda$.

The diffraction vector $q$ after equation 2.13 is the difference of the incoming and outgoing wave vector. It can be related to the diffraction angle according to equation 2.14 and corresponds to the vector of the reciprocal lattice $g^*$.

$$q = k_{in} - k_{out} \quad \quad (2.13)$$

$$q = \frac{4\pi}{\lambda} \sin \theta \quad \quad (2.14)$$

This effect has been described by Laue in 1912, who discovered that crystalline materials such as metals can act as three-dimensional diffraction objects for X-rays, which have a wavelength similar to their lattice spacing. Bragg’s law describes the interaction of X-rays with a defined wavelength $\lambda$ with a sample with a crystalline lattice with lattice spacing $d$, where a diffracted beam is observed in a diffraction angle $2\theta$, when the equation 2.15 (with $n$ being a positive integer) is fulfilled and constructive interference of the incoming X-rays occurs.

$$n \lambda = 2d \sin \theta \quad \quad (2.15)$$

Bragg’s equation (cf. eq. 2.15) is illustrated in Figure 2.26, where $n$ is a positive integer, $\lambda$ is the wavelength of the incident X-rays, $\theta$ the angle between the incident X-rays and the lattice planes and $d$ the lattice spacing for this family of lattice planes. If the so-called Bragg condition is fulfilled, diffraction...
will occur for the corresponding set of crystallographic (hkl) lattice planes. The angle of reflection is the same as the incident angle $\theta$.

![Bragg Equation Diagram](image)

Figure 2.26: Illustration of the Bragg equation. The X-ray beam of a wavelength $\lambda$ penetrates the crystalline sample with the lattice spacing $d$ in an incident angle $\theta$ between different lattice planes and the incoming beam.

In classical X-Ray diffraction the sample will be scanned for different $2\theta$ angles and the X-Ray intensity is detected. Intensity peaks are expected at certain $2\theta$ (so-called Bragg peaks) corresponding to the characteristic lattice spacings for the material. These individual X-ray peak profiles can be analysed further regarding their position, width and their intensity.

Both diffraction angles as well as the intensities of the diffracted beam are a function of the crystal structure [BRA08]. The outer electrons of the material act as scattering objects and influence the magnitude of diffraction. This means that lattice defects such as dislocations and internal stresses disturb the periodicity and structure of the lattice. The intensity of the diffracted beam is directly related to the amplitude of a so-called structure factor, which is a mathematical function describing the amplitude and phase of a wave diffracted from crystal lattice planes (hkl). The reader is referred to Brand et al. [BRA08] for more details in determination of the structure factor for an fcc material. X-ray profiles of plastically deformed crystals can reveal a peak shift or a peak broadening. [BRA08, WAR90]
In general it is differentiated between stresses of first order, which are macrostresses (e.g. residual stresses) being homogeneous over several grains, stresses of the second order, microstresses being homogeneous within one grain, and stresses of third order being microstresses inhomogeneous within one grain e.g. because of the presence of heterogeneous intragranular structures such as dislocation networks. Macrostresses as well as microstresses, which are homogeneous within the grain, can be detected by a peak shift of the X-ray profile. Microstresses of third order, which are related to locally different strains within the grain, can only be identified from the analysis of the peak width and peak shape. Stresses are always measured indirectly by determining the local elastic strain. A statement about the stress can then be made (using Hooke’s law), when the stress-state is known (e.g. uniaxial). Especially the local elastic strains causing microstresses of third order will be of interest of the thesis.

For the following it is important that we deal with a metallic and crystalline material, which is characterized by a three-dimensional crystalline atomic structure with a periodic lattice. The concept of the reciprocal lattice is introduced shortly, which is the Fourier transformation of the real crystalline lattice.

In general any plane can be described by a vector (indicated by bold type) perpendicular to it. The reciprocal lattice is constructed by lattice vectors \( \mathbf{a}_1, \mathbf{a}_2 \) and \( \mathbf{a}_3 \) in real space and the basis reciprocal vector \( \mathbf{g}_1^*, \mathbf{g}_2^*, \mathbf{g}_3^* \) are defined as described in the following:

\[
\mathbf{g}_1^* = 2\pi \frac{\mathbf{a}_2 \times \mathbf{a}_3}{\mathbf{a}_1 \cdot (\mathbf{a}_2 \times \mathbf{a}_3)} \quad (2.16)
\]

\[
\mathbf{g}_2^* = 2\pi \frac{\mathbf{a}_3 \times \mathbf{a}_1}{\mathbf{a}_1 \cdot (\mathbf{a}_2 \times \mathbf{a}_3)} \quad (2.17)
\]

\[
\mathbf{g}_3^* = 2\pi \frac{\mathbf{a}_1 \times \mathbf{a}_2}{\mathbf{a}_1 \cdot (\mathbf{a}_2 \times \mathbf{a}_3)} \quad (2.18)
\]

This means that e.g. the reciprocal space vector \( \mathbf{g}_1^* \) is perpendicular to the lattice plane in real space defined by \( \mathbf{a}_2 \) and \( \mathbf{a}_3 \). Any reciprocal space vector \( \mathbf{g}_{hkl}^* \) is perpendicular to a set of lattice planes (hkl) and can be calculated according to equation 2.19.
\[ \mathbf{g}_{hkl}^* = h\mathbf{g}_1^* + k\mathbf{g}_2^* + l\mathbf{g}_3^* \]  

(2.19)

\[ |\mathbf{g}_{hkl}^*| = \frac{2\pi}{d_{hkl}} \]  

(2.20)

The length of the reciprocal space vector is related to the distance between the lattice planes \(1/d_{hkl}\) as given in equation 2.20, thus the unit generally used for observations derived from reciprocal space (and as we later see with this from the diffraction vector \(\mathbf{q}\)) is Å\(^{-1}\).

Figure 2.27: Ewalds sphere with the reciprocal lattice. Lattice points located on the reflecting sphere will cause diffraction. Lattice points within the limiting sphere can cause diffraction after rotation of the sample. Reproduced from [BRA08].

The Ewalds sphere (cf. Figure 2.27) with a radius of \(1/\lambda\) is used to visualize the objects that contribute to diffraction when satisfying Bragg’s law. The reciprocal lattice points are equidistant (corresponding to the lattice spacing \(2\pi/d_{hkl}\)) along a straight line with successive order of the corresponding Bragg reflection (eg. 111, 222, 333, 444 ...). The size of the points (or diffraction objects) is in principle related to the perfection of the crystal (eg. if the crystal lattice is distorted or not) determining the precise Bragg angle or angular range for deformed crystals, where diffraction will occur. The diffraction vector with origin at 000 is normal to the lattice planes hkl. Points that are located on the reflection sphere are contributing to diffraction and other points lying within the limiting sphere can possibly contribute to diffraction, when the specimen is rotated.
When identifying microscopic elastic strains within a grain as described later by HRRSM, different regions of the grain correspond to slightly different positions of reciprocal lattice points, while the reciprocal lattice points shift (and with this the diffraction angle changes) when macroscopic strains are applied. [BRA08, WAR90]

2.3.2.2 Characterization of long-range internal stresses

Long-range internal stresses have been characterized mainly using classical X-ray diffraction and peak profile analysis by Ungar et al. [UNG83, MUG83, MUG02]. The major observation was, that X-ray peak profiles are asymmetric, when originating from a heterogeneous dislocation distribution present in plastically deformed fcc metals. This is seen as an experimental evidence for long-range internal stresses as proposed by the composite model. Ungar et al. [UNG83, MUG86] investigated intensively copper. Classical analysis of X-ray peak profiles obtained from cyclically deformed aluminium has not been published by the same extend. The origin of the asymmetric peak profiles can be explained by the composite model. Both the cell and the wall component will give a symmetric peak profile, but at slightly different diffraction conditions due to their different internal stresses. The resulting asymmetric peak profile is the superposition of those two relatively shifted subprofiles. In general, two different cases are distinguished under tensile load depending on the diffraction geometry: The axial case with the diffraction vector $q$ being parallel to the deformation axis, and the side case with the diffraction vector $q$ perpendicular to the loading axis. These are demonstrated in Figure 2.28. The asymmetry will appear differently for the two cases. For the axial case, which is the case investigated in the presented experiments, the asymmetry is positive with a tail appearing at lower $q$ or $2\theta$ values. The wall profile is shifted to lower $q$ or $2\theta$, because of tensile forward stresses, and the subgrain profile to higher due to compressive back strains. The local strains are of opposite sign for the side case and the resulting peak profile asymmetry is negative with a tail at higher $q$ or $2\theta$. 
Figure 2.28: Example for the asymmetric radial peak profile obtained for tensile deformed [001]-oriented copper single crystal. The shift of the wall and peak profile in comparison to the mean profile position $\theta_0$ are indicated and in opposite directions for the two cases. (a) axial case (020 reflection investigated) with positive asymmetry and (b) side case (002 reflection investigated) with negative asymmetry. Reproduced from [UNG83] with added markings for the subgrain and wall profile. The angular scale in the images was corrected to $\Delta \theta = 2'$ instead of $1'$ in [UNG91].

Initially X-Ray diffraction was done on copper single crystals after crystallization but before deformation and on the same copper single crystals deformed in uniaxial tension. The (002) reflections were investigated for the described axial case, where an increase in the profile width and an increasing asymmetry was observed with larger tensile deformation as shown in Figure 2.29. [UNG83] Cyclically deformed samples show a smaller extent of asymmetry than samples deformed to large plastic strains. [BIE92]
The composite model was revised by Pantleon et al. [PAN10] stating that the subprofile of the subgrain is a superposition of many profiles resulting from the individual subgrains. The reason for the refinement being that the dislocation density in the subgrain regions has been found to be too low to cause the observed broadening of the subgrain peak profile and that individual subgrains had different back strains. The traditional composite model considers only one broadened subgrain component including all subgrains present, but does not differ between the individual subgrains. It also assumes that all subgrains experience the same stress and that the signal is therefore broadened due to the dislocation density. The broadening can though be explained when considering that the individual subgrains experience different local elastic strains and with this occur at different diffraction vectors $q$ or Bragg angles $2\theta$. The profiles of the individual subgrains are expected to be rather sharp, since they originate from nearly dislocation-free regions. The approach will be discussed in detail, when describing the applied technique High Resolution Reciprocal Space Mapping in section 3.4.

Recrystallized polycrystalline copper samples were investigated after fatigue at a strain amplitude of $5 \cdot 10^{-3}$ for 2000 cycles into saturation by Biermann et al. [BIE92]. The tested samples were stopped at six selected points as shown in Figure 2.30a along the stress-strain hysteresis and unloaded to zero.
stress before further investigations with X-Ray diffraction. Measurements were done after unloading from the maximum tensile and compressive load, at half the plastic strain after strain reversal and unloading and at zero plastic strain, though not within the elastic region nor close to the yield point. It was found that, the asymmetry changes sign when the radial peak profile is measured at reversed load. Figure 2.30b shows a radial peak profile obtained at the maximum tensile and maximum compressive load (after unloading) for cyclically deformed copper. The profile at the maximum tension shows a positive asymmetry and the profile at maximum compression a negative asymmetry. These observations are relevant for the later presented HRRSM results.

Figure 2.30: (a) Selected conditions along the cyclic stress-strain hysteresis for analysis with X-Ray diffraction (b) Radial peak profile for the (200) Bragg reflection of cyclically deformed copper obtained for a sample unloaded from the maximum tension $T$ and maximum compression $C$ (corresponding to 1 and 4 in (a)). The sample surface investigated post mortem was perpendicular to the loading axis, hence the axial case was measured with positive asymmetry for the profile measured after unloading from maximum tensile load. Reproduced from [BIE92].

The radial profiles obtained for the positions along the hysteresis show only a small asymmetry, which is largest at maximum tension and maximum compression. It is nearly zero for the samples investigated after unloading from half of the reversed strain amplitude, because in this condition the internal stresses are at their minimum as proposed by the composite model (cf. Figure 2.24b). The extend of
the asymmetry is increasing during loading to the maximum stress. The sign of the asymmetry for samples measured after unloading from half the strain amplitude was found to be sometimes of the sign of asymmetry measured for the profile for the maximum stress and sometimes of the reversed sign.

The local elastic strain of the grains was derived from the difference of the local strains $\Delta \varepsilon_w$ and $\Delta \varepsilon_c$ determined from the subprofile positions for the wall and the subgrain (designated with c for cell) and is shown in Figure 2.31. The local elastic strain of the grain follows in principle a hysteresis loop being minimal (nearly zero) for the measurements after unloading from half the strain amplitude for the same reasons as the profile appears nearly symmetric. Local elastic strains are of an order between $-6 \cdot 10^{-4}$ to $6 \cdot 10^{-4}$, while the macroscopic strain was more than one magnitude larger of the order of approximately $-2 \cdot 10^{-3}$ to $2 \cdot 10^{-3}$. The local strain $\Delta \varepsilon_c$ corresponds to what is described as “naive” asymmetry in the following.

\[ \Delta \varepsilon_{w} - \Delta \varepsilon_{c} \]

\[ (\Delta \varepsilon_{w} - \Delta \varepsilon_{c}) \cdot 10^{-4} \]

\[ (\Delta \varepsilon_{w} - \Delta \varepsilon_{c}) / \text{MPa} \]

\[ (\Delta \varepsilon_{w} - \Delta \varepsilon_{c}) / \text{rad} \]

\[ \epsilon_{pl} \cdot 10^{3} \]

\[ \text{FWHM} / 10^{-4} \text{ rad} \]

\[ \epsilon_{pl} \cdot 10^{3} \]

**Figure 2.31:** (a) Local elastic strain of the grain deduced from the difference of the local elastic strains for cells and walls in dependence of the macroscopically applied plastic strain and (b) the radial profile full width at half maximum measured for radial peak profiles for samples unloaded from six selected conditions along a cyclic stress-strain hysteresis. [BIE92]

A characteristic butterfly pattern was observed for the width of the profiles measured after unloading from the six discussed conditions in dependence of the macroscopic strain. The full width of half maximum (FWHM) is largest at the maximum compression and tension, just as the asymmetry. The
width is decreased for samples unloaded from half the strain amplitude in comparison to the measured width for the samples unloaded from maximum tension and compression. The width is nearly similar for the samples unloaded from zero strain, with a value in between. The FWHM is of the order $3 \cdot 10^{-4}$ rad to $6 \cdot 10^{-4}$ rad.
3 Experimental Methods

3.1 Material

Tensile test specimens were manufactured from a commercially pure aluminium alloy AA1050 sheet homogeneously cold-rolled to 90% thickness reduction to a final thickness of 1 mm. The composition of the material is displayed in Table 3.1. AA1050 is mostly used as a sheet material where moderate strength is required and known for its excellent corrosion resistance.

<table>
<thead>
<tr>
<th></th>
<th>Si</th>
<th>Fe</th>
<th>Cu</th>
<th>Mg</th>
<th>Zn</th>
<th>Ti</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>Min.:</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>99.5</td>
</tr>
<tr>
<td>Max.:</td>
<td>0.25</td>
<td>0.4</td>
<td>0.05</td>
<td>0.05</td>
<td>0.07</td>
<td>0.05</td>
<td>-</td>
</tr>
</tbody>
</table>

Table 3.1: Composition of the commercially pure aluminium alloy AA1050 (as provided by the producer) used for all presented investigations.

Dog bone-shaped specimens as shown in Figure 3.1 with a gauge section of 15 mm in length and 5 mm in width were designed to fit into a clamp holder in a custom-made screw-driven load frame, which will be described in detail in section 3.2.2. Sample cutting was done by spark erosion cutting to minimize the mechanical impact on the microstructure.

![Figure 3.1: Dimensions of the tensile test sample manufactured from a 1 mm thick AA1050 sheet](image)

Tensile specimens were annealed at 600 °C for two hours to ensure complete and homogeneous recrystallization. The microstructure after annealing was investigated metallographically. The hardness is 45 HV<sub>0.05</sub> before heat treatment and around 22 HV<sub>0.05</sub> (cf. Figure 7.16) after heat treatment.
Using both, light optical microscopy and scanning electron microscopy, grain sizes were estimated to be between 30 µm and 100 µm, and the microstructure was found to be homogeneous throughout the entire cross section of the gauge showing a pronounced cube texture. An exemplary area investigated by Electron Backscatter Diffraction is presented in Figure 3.2.
Figure 3.2: EBSD analysis of recrystallized AA1050 before any mechanical deformation. A large area of 1.5x1.5 mm was investigated. (a) Left: EBSD orientation map (where the crystal orientation along the tensile direction is coloured according to the triangle shown in the corner) of the grain structure showing various sizes appearing with uniform orientations. Right: texture analysis of the presented data showing a cube texture. Acquired with Bruker EBSD System at 20 kV at a working distance of 12 mm and 1 µm step size (b) Distribution analysis of the grain sizes for the presented data indicating the presence of some large and some small grains but mostly a distribution around 50-60 µm. The average grain size was though determined to 90 µm, because of the presence of some larger grains.
3.2 Mechanical Testing

3.2.1 Mechanical Pre-deformation – MTS Acumen

Some of the investigated tensile specimens were pre-deformed in tension and pre-fatigued using an MTS Acumen 3 kN Electrodynamic Test System with pneumatic grips and equipped with Station Manager MTS FlexTest 40 as shown in Figure 3.3.

![Figure 3.3: MTS Acumen 3 kN Electrodynamic Test System with pneumatic grips and equipped with Station Manager MTS FlexTest 40. An AA1050 tensile sample is mounted in the grips as shown in the enlarged square.](image)

However various issues occurred using the in-house set up, because of small sample sizes that made the alignment in the grips difficult and a load cell designed for much larger loads. Bending during mounting and testing with higher displacement amplitudes (larger than 20 µm) was one of the major issues.

The desired displacement amplitude was approached in an adaptive way during the first 10 cycles from a slightly lower displacement amplitude to ensure the correct values. The strain was calculated from the displacement of the grips.
3.2.1.1 Tensile Deformation

Figure 3.4 shows a typical stress-strain curve for the annealed AA1050 sample deformed to 1% tensile strain (as done for the samples investigated later) with a nominal strain rate of $10^{-3}$ s$^{-1}$ using the MTS Acumen machine. Pneumatic grips were used to mount the sample and the axial center of the gauge was aligned with the center of the grips. The upper grip was then moved to the same displacement to keep a constant grip distance for all samples. The gripping pressure was adjusted to 2 bar, which was found to be the reasonable pressure for a soft material such as annealed aluminium. A steep increase during the short elastic regime is visible before yielding starts at approximately 80 N corresponding to a yield strength of 16 MPa.

![Figure 3.4: Engineering stress-strain curve of bone-shaped AA1050 sample deformed by 150 µm to 1% strain with MTS Acumen. The load was zeroed after mounting.](image)

3.2.1.2 Tension-Compression Cycling

Figure 3.5 shows exemplary load curves acquired during the tension-compression cycling after 1% tensile deformation of bone-shaped AA1050 samples by the 3 kN MTS Acumen machine with a two different displacement amplitudes corresponding to strain amplitudes $\varepsilon_a$ of $6.7 \cdot 10^{-4}$ and $1.3 \cdot 10^{-3}$. No macroscopic bending and wobbling of the sample was visible for those displacement amplitudes (but for higher values) during cycling. Cycling was done with the chosen displacement amplitude above and below the maximum displacement after pre-deformation (set point 0.150 mm) using a tapered
ramp shape with 10 Hz. A Peak-Valley compensator was used to achieve a uniform peak-valley ramp signal.

Figure 3.5: The maximum and minimum load over number of cycles for tension-compression cycling at (a) a displacement of 10 \( \mu \text{m} \) (corresponding to a strain amplitude of \( 6.7 \cdot 10^{-4} \)) for the first 100 000 cycles without visible fracture. It was still no fracture observed after 500 000 cycles, but only the first 100 000 cycles were demonstrated (due to calculation time). (b) A displacement amplitude of 20 \( \mu \text{m} \) (corresponding to a strain amplitude of \( 1.3 \cdot 10^{-3} \)) for 95000 cycles and fracture starting after 89150 cycles.

It is visible for both curves that the load amplitude (difference between maximum and minimum load) is first increasing before reaching a plateau, which is more striking for the higher strain amplitude. For the lower strain amplitude the load amplitude seems then to decrease continuously during further testing as described in section tension-compression testing as well. For the higher strain amplitude the load amplitude is then increasing again before remaining nearly constant until fracture. The oscillations of the data points causing a wide curve is due to the peak-and-valley read out. The tests were done under displacement-controlled conditions and the strain is calculated based on the displacement values.
3.2.2 Equipment for in-situ deformation – custom made load frame

The load frame used during the synchrotron experiments was initially designed for in-situ high energy X-ray studies at synchrotron installations investigating monotonic uni-axial loading. It was then redesigned and improved for tension-compression cycling as shown in Figure 3.6a by a PETRA III/CHESS(inSitu) collaboration. The load frame is designed to be attached to a rotation stage and to not obstruct the incoming and outcoming X-ray beams over rotation ranges of about ±70 degree. It allows measurements with both a near-field and far-field detector as described in section 3.4, while simultaneously applying mechanical load.

A bone-shaped sample is equipped with strain gauges and screwed into a clamp holder (cf. Figure 3.6b) in the center of the frame. The deformation takes place by a motor-driven lead screw and the load is read out by a 1 kN load cell, which was calibrated with lead bricks. The deformation rate is controlled by the rotating speed of the screw.

To ensure a tight grip the clamps were first loosely screwed onto the sample and the sample was pulled until small changes in load were noticed, which is then the zero displacement, because the shoulder (cf. Figure 2.1b left, marked with an arrow) are at this point touching the sample causing real deformation. Flat plates (cf. Figure 2.1b right) are then screwed tightly on the ends of the tensile loaded sample to avoid buckling. It is important to ensure that all load frame components attached to the sample holder are screwed tightly to avoid slipping or jam up during deformation causing “empty” deformation/pulling. After the beam time at PETRA a test sample was always deformed before the real experiment to ensure, that the load frame is assembled correctly and the deformation occurs as expected.
Figure 3.6: Load frame used for tensile and compressive deformation in-situ during High Resolution Reciprocal Space Mapping. A dog bone shaped sample (with two strain gauges) is mounted in horizontal orientation in the load frame and loaded by a motor-driven lead screw. (b) An example of the mounting of the bone-shaped flat specimens in the clamp-holder of the load frame. Left: Grips for the bone-shaped specimen, where the shoulders (marked with blue arrows) are touching the sample. Right: A plate is screwed on the specimen to fix the sample into the clamp enabling compression loading.

Because small bending was noticed during this procedure in tests of the load frame after the first beamtimes, the sample mounting was then monitored in detail by following the strain and load over time (so-called timescan) during the mounting. It is presented in the following for the sample used during investigations at the second APS beamtime. The sample was mounted, where it experienced a strain around $10^{-3}$ and a compression force of ca. -60 N after tight clamping as shown in Figure 3.7. The sample deformation was identified as bending by the use of two strain gauges, attached to the sample front and back sides, behaving in the opposite way (cf. Figure 3.7a). This effect is consistent with a slightly curvature of the unmounted samples possibly due to the soft material or manufacturing. However, it is not expected that the experienced load and strain will cause the development of an irreversible compression structure.
Figure 3.7: Measures for strain and force over time during loose and tight clamping of the tensile sample into the clamp-holder. (a) Timescan of the strain measured by the two strain gauges (red and blue) showing that the strain develops in opposite directions indicating a bending of the sample. The time, where the tight screwing of the sample started is marked with a dashed blue line. Irregularities are due to an overflow from short circuit of the strain gauge cables when touching each other during screwing. (b) Timescan of the load measured by the load cell showing that the sample experienced at the end -60 N during mounting.

3.2.2.1 Strain Measurement

To recognize and avoid a bending of the sample both during mounting and later during the experiment two strain gauges were used, one attached each side of the specimen aligned with the tensile axis and centered with respect to the gauge section (cf. Figure 3.8). In the first APS experiment, only one pre-wired strain gauge of type Omega KFG-3 350 Ω was used, because the problem of sample bending was still unknown. Later two strain gauges of the type HBM LY13 350 Ω were used and connected by light wires. Strain gauges are used to measure the axial strain by a strain sensitive copper grid, which is elongating or compressing during deformation. The change of the electrical resistance due to changes in the pattern geometry can be measured and the read out voltage converted (using the Wheatstone bridge and a gauge factor) into strain. The presented strain is the mean strain calculated from both strain gauges. The zero strain was calibrated from the unmounted or loosely mounted sample, which is the strain at zero load of the clamped sample.
The tensile cycling of the first APS experiment was done displacement controlled with a predefined displacement, while the tension-compression cycling in later experiments was done displacement controlled where the displacement was defined through the desired strain values. In both cases the cycling was done by moving the motor with a constant displacement, but the way of deciding on the displacement was different. This was done due to a backlash of the load frame, when reversing load.

3.2.2.2 Tension-Compression Deformation

The load frame was initially designed for tensile deformation and then redesigned to allow tension-compression deformation. It was therefore of interest to verify the instrument response and to exclude affects from the load frame (e.g. load frame stiffness). For this a tensile-compression test deformation was performed as shown in Figure 3.9, before synchrotron experiments. Here the two strain gauges are displayed (in red and blue) and Young’s modulus and Buckling stress were determined from the displayed load-strain hysteresis. It is visible that the strain gauges begin to behave opposite of each other during the last part of compressive deformation at around -180 N corresponding to -36 MPa. This is the critical buckling stress meaning that no bending will occur above -36 MPa. A cycling of 0.1% strain around the zero load will be below the buckling and hence possible in the following experiments.

The beginning differences of 0.15% in initial strain and the elastic regime are related to a bending of the sample during mounting due to forcing the sample into the holder when tight screwing the clamps or straightening the initial sample curvature e.g. from transport or minor misalignment. The sample adapters need to be carefully aligned before sample mounting to minimize the bending during
mounting and to avoid buckling of the sample at higher deformations. This findings lead to a monitoring of the load and strain during mounting in further experiments to limit the bending experienced by the sample.

The Young’s modulus was determined to 70 GPa as expected from literature indicating no load frame hardening in the beginning of tensile deformation.

![Strain Gauge Back and Front](image)

**Figure 3.9:** Load frame test to identify buckling regime. A load-strain curve was measured with both strain gauges (blue and red) and buckling was detected at -180 N. The strain gauges start at different values due to sample bending during mounting.

A further challenge during tension-compression cycling was the backlash, which means that the load frame screw had to move a long distance after changing loading direction before the sample was affected by deformation. An example for the displacement needed to achieve a full stress-strain hysteresis is shown in Figure 3.10, where the stress remains constant during a nominal displacement of approximately 100 µm. Therefore, the displacement expected to achieve a desired strain is different from the nominal displacement, which needs to be driven in reality for tension-compression cycling. For this reason the displacement limits for cycling were determined by the strain read from the strain gauge signals and detailed load cycles were measured to determine the displacements for the desired stress-strain conditions before mapping along a stress-strain hysteresis. The backlash was first characterized in the second and third APS experiment.
Figure 3.10: Example for (a) two stress-strain hysteresis measured by the load cell and two strain gauges (cyan and red) and the hysteresis with the calculated average strain (purple) and (a) the corresponding changes of stress with the driven displacement (in µm) along in total ten load cycles. It is visible that a large nominal displacement of ca. 100 µm is driven with constant stress caused by the backlash of the load frame. To obtain a tension-compression stress-strain hysteresis demonstrated in (a) a significant larger nominal displacement has to be driven than a common displacement amplitude.

A possible replacement of the leadscrew with a satellite screw was discussed to enhance the speed and remove the backlash when the load is reversed.

3.2.2.3 Cyclic response curve

The change of maximum and minimum load and strain for 9000 cycles tension-compression cycling with the load frame is presented in the following. High number of cycles were very time-consuming caused by the slow speed and backlash of the motor screw. Due to the backlash the displacement limits for cycling were first found manually by driving the motor to the desired strain amplitudes (contrary to cycling with the MTS Acumen, where the displacement is converted into a strain amplitude). Further cycling was then done by driving it automatically to the identified displacement positions. The presented cycling in Figure 3.12 was done for a sample equipped with two strain gauges (red and blue) after tensile deformation to 1% average strain. The first ten cycles are presented in Figure 3.11
showing that the hysteresis curves measured by the two strain gauges differ and also vary from cycle to cycle. The starting value for cycling in tension is almost \(0.5 \cdot 10^{-4}\) different in strain, though only \(0.2 \cdot 10^{-4}\) different in strain at the maximum compression for the two strain gauges indicating bending. While the strain at maximum compression and the branch for loading into tension is almost similar for all ten cycles, the strain at the maximum tension and the branch of unloading into compression of the hysteresis and is clearly different for each cycle possibly due to a softening of the load frame upon stress reversal. The first hysteresis was very wide and then they converge more and more to the same values and a narrower hysteresis while cyclic deformation continues.

The presented cycling is not symmetrically around zero load, because it was chosen to cycle \(2 \cdot 10^{-3}\) strain below the maximum tension after 1% pre-deformation detected by one of the strain gauges ending at approximately 200 N (which is higher than the load after 1% pre-deformation in displacement with the MTS Acumen). The average cycling strain amplitude was approximately \(10^{-3}\) though slightly smaller for the blue curves and slightly larger for the red curves. The strain amplitude \(10^{-3}\) (meaning the hysteresis covers in total \(2 \cdot 10^{-3}\) strain difference) was the maximum strain amplitude used in all presented experiments, because higher strain amplitudes will cause buckling and early failure as discussed before. The sample was then cycled further to 9000 cycles and the changes of load and strain are shown in Figure 3.12. It resembles the general observation for aluminium that both load and strain are dropping over time during cycling. Hence it seems to be a material specific behaviour and not related to the mechanical testing equipment used. Detailed mechanical data based on the introduced observations will be presented for the individual synchrotron experiments in the following.
Figure 3.11: The first ten cycles after 1% pre-deformation in tension detected by two strain gauges (blue and red). Differences in the hysteresis measured by each of the two strain gauges and measured for each cycle are visible. The direction of loading and unloading are marked with arrows. The hystereses are approaching more similar values over the cycles resulting in a decrease of the strain amplitude. The maximum strain amplitude is marked with arrows and dashed lines.
Figure 3.12: Load and strain data of a AA1050 sample cycled for 9000 cycles after 1% tensile predeformation using the load frame for synchrotron experiments. (a) Maximum and minimum load of the sample. (b) Maximum and minimum strain detected by the two strain gauges attached to the back (DMS1) and front (DMS2). (c) Change of the load amplitude over number of cycles showing a steep decrease until ca. 5000 cycles but further decrease until 9000 cycles. (d) Change of strain amplitude for the two different strain gauges DMS1 and DMS2 showing a continuous decrease of the strain amplitude during cycling.
3.3 Scanning Electron Microscopy

Scanning Electron Microscopy (SEM) was used to gain a first insight into the microstructure of mechanically deformed samples. In this way suitable testing parameters (e.g. strain amplitude and number of cycles) resulting in subgrain structures were determined. SEM is a broadly used tool to analyse topography and morphology, crystallography and orientation of grains in macroscopic samples allowing the investigation of large areas using different signals and detectors. The conductive sample is scanned by a focused electron beam and backscattered electrons (BSE) or secondary electrons (SE) can be detected. Detectable secondary electrons are generated close to the surface and are widely used to image a topography contrast such as fracture surfaces, while BSE are used to image the material using mean atomic number (Z) or orientation contrast. Electron microscopy investigations are done after testing and the microstructure can be influenced by metallographic preparation before the investigations. Moreover the analysis is restricted to the surface with a limited angular resolution.

In Figure 3.13 the field-emission gun scanning electron microscope FEI Nova Nano 600 used for the presented investigations is shown.

![High resolution scanning electron microscope FEI Nova Nano SEM 600 equipped with SE and height-adjustable EBSD detector. The BSE detector is mounted on the pole piece within the chamber and hence not visible.](image)

Figure 3.13: High resolution scanning electron microscope FEI Nova Nano SEM 600 equipped with SE and height-adjustable EBSD detector. The BSE detector is mounted on the pole piece within the chamber and hence not visible.
3.3.1 Sample Preparation

For analyzing the grain structure by orientation imaging and EBSD an even sample surface is required. The metallographic preparation of aluminium was very challenging because of the low material hardness (especially in an undeformed state) and much time was spent on finding an acceptable way of preparing tensile test samples for SEM analysis. The final approach with applying electropolishing as the final step is described below.

First, the center of the gauge of the dog bone was cut using Strues Acutom precision cutting machine. It was not possible to use embedded samples due to several challenges: The required electropolishing for the final step, the large sample size, the limitation in chamber space and drifting problems, when tilting the sample.

Hence, the cut piece was ground and polished manually. First, the sample surface was grinded on 4000 sandpaper for 9 minutes to remove a surface layer of 100 µm. It was then polished for 3 minutes on a MOL cloth using 3 µm diamonds and STRUERS red lubricant (suspension of oil and water used for soft materials). The sample was cleaned in ultrasound bath after each preparation step to remove grinding material to minimize scratching and indenting of the surface in further preparation steps. It was then flushed with ethanol and dried in warm air. Next, it was polished for three minutes on a NAP cloth using 1 µm diamonds using red lubricant. Further polishing with smaller diamond sizes resulted in to many scratches and was therefore omitted. Finally, the center of the sample was electropolished using a square mask of 0.5 cm² area and A2 solution for 15 seconds with 20 V. Longer electropolishing times or chemical polishing using OPS solution as the final step resulted in a too strong topography making orientation imaging difficult. Defects visible at the presented BSE images are scratches from preparation, pressed in or fallen out grinding particles resulting in holes and etching marks from locally too aggressive electropolishing.
3.3.2 Electron Channeling Contrast Imaging

Electron Channeling Contrast Imaging (ECCI) was presented by Zaefferer et al [ZAE14] as a powerful technique to image grain orientations, intragranular misorientations and crystal defects such as dislocations. This requires SEM microscopes, which allow small beam diameter, small beam convergence and high current density. In this way a high angular resolution of up to 0.1° allowing the detailed imaging of subgrain structures in large areas can be achieved in SEM investigations without application of transmission electron microscopy.

The origin of orientation contrast for BSE signal is described schematically in Figure 3.14. If the lattice planes of the grain are parallel to the incident beam, the electron beam can channel through and the responded signal is low hence resulting in a dark appearance of the grain in the image. Depending on the inclination of the lattice planes with respect to the incoming electron beam the intensity will be stronger and the grain will appear in different shades of grey up to very bright for grains oriented in non-channeling directions.

Figure 3.14: Schematic path of backscattered electrons for orientation imaging. The intensity of BSE (black arrows) depends on the orientation of the grain.
Figure 3.15: Tilt series for 0°, 2° and 4° degrees for the same region of a grain deformed 15% in tensile deformation showing the sensitivity of ECCI for the grain orientation. Dark spots are dirt from preparation.

The presented BSE images (e.g. Figure 3.15) were acquired with 15 keV at 1.7 nA in analytical mode for high beam current density and a low working distance of 4-5 mm using the spot after first crossover allowing a parallel beam with low convergence, which were found to be the best imaging conditions. The orientation of selected imaged grains was analysed using Electron Backscatter Diffraction (EBSD) by using hardness indents as markers for the location. A combination of EBSD with image controlled diffraction conditions (cECCI) is a possibility for future investigations to gain more detailed information about the orientation relationship.

3.3.3 Electron Backscatter Diffraction

Electron Backscatter Diffraction (EBSD) is a SEM-based technique and has been established as a widely used method for the characterization of the microstructure of crystalline materials. Conventional EBSD
can provide the visualization of the microstructure (with a spatial resolution of up to ca. 20 - 60 nm for Aluminium at 20kV) and orientations as well as local misorientations (up to 0.1° [BRIT18] for conventional EBSD). Furthermore crystallographic textures and phases can be identified.

When obtaining EBSD patterns, the electron beam scans across an area in a given step size at a sample surface tilted in an angle of 70°. From the interaction of the electron beam with the inclined lattice planes, cones of electrons are formed resulting in a diffraction pattern at the phosphor screen, which converts the pattern into an electronic signal. The angles between the Kikuchi bands and the width is characteristic for the crystal structure of the investigated material. From the diffraction pattern the characteristic crystal structure and orientation can be determined providing information about the phase and local orientation. To achieve the tilt in the present investigations a pre-tilt holder was used. The loading axis of the investigated samples was always oriented as shown in Figure 2.1.

![Figure 3.16: Schematic EBSD set-up with the inclined sample and the resulting diffraction pattern on the detector. The orientation of the tensile direction for all presented samples is marked in black. The reference coordinate system is marked in red. Modified from [SCH09].](image)

In the following two different visualizations of EBSD measurements are presented: The Image Quality (IQ) map (cf. Figure 3.17) and an orientation map colored according to the crystallographic directions along a certain macroscopic axis (TD, RD or ND) (cf. Figure 3.18). The IQ-map displays the investigated region in a grey-scale dependent on the local indexing quality of the diffraction pattern acquired at the measurement points. The quality of the diffraction pattern is influenced by many factors such as
microscope settings, location on the screen, crystal orientation and crystal distortion. To obtain a good pattern quality the microscope settings are chosen to give a high signal-to-noise ratio and a high pattern contrast. Black regions in the IQ-map indicate a poor indexing of the pattern either due to a high distortion (e.g. boundaries or highly deformed areas) or poor preparation (poor pattern signal), which does not allow the indexing of a required amount of bands in the diffraction pattern to the selected material. A typical example for an Image Quality map is shown in Figure 3.17.

![Image](image.png)

**Figure 3.17: Example for an Image Quality (IQ) map acquired by EBSD from a cyclically deformed AA1050 sample.** The image quality map gives information about, how good the pattern of the individual measurement points was indexed. The pixel size corresponds to the step size of 100 nm. Pixel at grain boundaries lead to poor image quality and appear dark.

One of the main applications of EBSD is the investigation of local orientations, textures and local misorientations. For this, the investigated microstructure can be displayed with a color-code for the corresponding orientations in an orientation map, where crystallographic directions are color-coded according to the inverse pole figure (cf. Figure 3.18 left). The color-code is dependent on the chosen reference microscope axis, which is typically the Transverse Direction (TD, [100]), Normal Direction (ND, [001]) or Reference Direction (RD, [010]) of the sample (cf. Figure 3.16b). A typical example for the three orientation maps (for the RD, TD, ND plane) is shown in Figure 3.18. Orientation maps shown in
The following are usually color-coded according to RD, which is nearly parallel to the deformation axis of the sample (cf. Figure 3.16).

The presented EBSD data were acquired using the FEI Nova Nano SEM equipped with a Bruker EBSD System at a detector distance of 18.5 mm, using a 20 kV beam with 2.1 nA in analytical mode. The step size for acquisition corresponds to the pixel size and is given in connection with the presented data. A finer step size means a longer acquisition time, but a better resolution. To improve the pattern quality for indexing, several frames of the diffraction pattern at the same location can be measured and averaged before indexing. This was done for poorly prepared or highly deformed samples (e.g. 50% tensile strain).

![Figure 3.18: Example for orientation maps acquired with EBSD from cyclically deformed AA1050 sample with a step size of 100 nm. The three different maps reveal the crystallographic direction along a chosen macroscopic direction according to the color-code in the inverse pole figure (left) and their relative direction. left: color coded according to RD, middle: according to TD and left: according to ND.](image)
3.4 High Resolution Reciprocal Space Mapping

High Resolution Reciprocal Space Mapping (HRRSM) is a diffraction technique using synchrotron radiation. It combines the use of hard high-energy focused X-rays to penetrate bulk metal samples and a significantly higher angular resolution of 0.004° than it can be provided by recent electron microscopes. The technique allows fast mapping of reciprocal space while in-situ investigating the microstructure under mechanical deformation. [JAK07]

The experiments were performed either at Beamline P07 at PETRA (DESY, Hamburg, Germany) or 1-ID-E at Argonne Photon Source (Argonne National Laboratory, IL, USA) providing as third generation synchrotron facilities a X-ray beam of high brilliance. An experimental hutch with several meters space was needed for setting up of the far-field detector.

3.4.1 Data acquisition

3.4.1.1 Set-Up

Reciprocal space maps were acquired at selected stress and strain conditions by HRRSM at two different synchrotron installations. Similar set-ups were used for the presented experiments performed at Beamline P07 at PETRA (Germany) or 1-ID-E at Argonne Photon Source (IL, USA) and will be explained in detail for APS. Differences of the set-ups are highlighted at the end of this section.
Figure 3.19: Sketch of the diffraction geometry and the position of the detectors used at APS, 1-ID-E. The tensile axis \( x \) of the sample is almost parallel to the diffraction vector.

The general set-up is exemplarily shown in Figure 3.19 for APS. It includes two detectors with a near-field detector for the selection of suitable grains and a far-field detector for acquisition of maps with high angular resolution of the monitored Bragg reflections.

The sample was equipped with at least one strain gauge at the center of the gauge section and aligned with the tensile axis to monitor the axial strain in-situ. It is mounted in a custom-made screw-driven load frame as described in section 3.2.2. Using flat grips, this load frame allows for mechanical loading in tension and compression while monitoring the local microstructure within the tensile specimen in-situ using synchrotron radiation. The load frame was placed with the load axis horizontally on a \( xy \)-translation stage allowing to move the sample and with this the selected grain of interest – which becomes displaced with respect to the load frame during mechanical loading – to the center of the beam after each loading step. The \( xy \)-translation stage is mounted on top of a rotation stage allowing rotation of the entire load frame around the vertical \( z \)-axis to obtain reciprocal space maps by rocking in small intervals around this axis. An additional \( z \)-translation stage allows adjustment for possible
changes in the height of the selected grain of interest due to mechanical loading. The set-up with the load frame mounted on the different motor stages is shown Figure 3.20a. The tensile axis of the sample (and the load frame) was aligned to be almost parallel to the diffraction vector, which is the so-called axial case [UNG83]. In this manner grains can be investigated with a specific crystallographic direction along TD.

![Figure 3.20](image)

**Figure 3.20:** (a) Image of the load frame used for HRRSM at APS, 1-ID-E. The sample equipped with a strain gauge is positioned in the center of rotation on top of several translation and rotation stages for alignment and acquisition. (b) Image of set-up at APS, 1-ID-E showing the round far-field detector approximately 5 m behind the sample in the load frame.

The 1 mm thick sample is penetrated at the selected location by the incoming beam. The beam is focused in vertical direction to a Gaussian width of 10 µm and narrowed in horizontal direction to 50 µm allowing a complete illumination of individual grains with sizes up to 50 µm.

After alignment of the beam and before any further analysis, it is ensured (by alignment with a pin, cf. Figure 3.21) that the beam is in the center of rotation and the same area is illuminated during rotations of the load frame around a vertical axis.
Figure 3.21: Load frame with a pin to align the equipment to ensure the sample is in the center of rotation around a vertical axis before the experiment. The tip of the needle is followed and the position adjusted so that during 360° rotations of the load frame it does neither move in vertical nor in horizontal direction.

For investigations two detectors, a near and a distant detector to image the diffracted beam, are used. The sample can be moved horizontally in x-direction and downstream in y-direction, the height can be adjusted in z and rotated around the z-axis with the angle $\omega$. HRRSM are acquired by the far-field detector 2, while rocking the sample around the vertical z axis perpendicular to the scattering plane and hence perpendicular to the tensile axis of the sample. The far-field detector and the rocking will be described further in section 3.4.1.3.

Suitable grains are identified with the help of a large area detector, an amorphous silicon flat panel from General Electrics (detector 1, near-field) with 2048x2048 pixels and a pixel size of 200 $\mu$m, placed 86 cm behind the sample on a horizontal translation to cover the first 6 diffraction rings of aluminium including the 400 ring. After this, the near detector 1 is moved out of the beam and the diffraction peaks are investigated with higher angular resolution by a Mar165 CCD (detector 2, far-field) with 2048x2048 pixels placed 4.65 m behind the sample on the location of the 400 diffraction peaks with diffraction vector close to the tensile axis (i.e. in the horizontal diffraction plane at a diffraction angle $2\theta_{400}$ of 13.53° for aluminium at 52 keV). The pixel size of the far-field detector corresponds to 79 $\mu$m.
and covers an angular $\eta$-range of 4.8°. All grains are (if not differently specified) detected within this range.

HRRSM was carried out at beam line 1-ID-E at the Advanced Photon Source at Argonne National Laboratory with a monochromatic beam of 52 keV. The set-up applied at beam line P07 at PETRA III was similar to the set-up used at APS. The major difference was, that the far-field detector was placed right instead of left with respect to the sample covering a different $\eta$ range of the diffraction ring. This implied exclusively consequences for the image reading in the data treatment. The experiment was performed with a monochromatic beam of 53 keV. Similar to the APS set-up the load frame with the sample was placed with the load axis horizontally on a $xy$ translation stage on top of a rotation stage allowing rotation of the entire load frame around a vertical $z$-axis. Translation along $z$ could be achieved by the heavy duty hexapod.

Grains were selected with the help of a Perkin Elmer detector placed 70 cm behind the sample on a horizontal translation. The diffraction peaks were investigated by a MarCCD placed 3.9 m behind the beam on the appropriate location for 400 diffraction peaks with diffraction vectors close to the tensile axis, i.e. in the horizontal diffraction plane at a diffraction angle $2\theta_{400}$ of 13.27° for aluminium for 53 keV.

The incoming beam intensity was measured by a diode (at PETRA) or split ion chamber (at APS) for each individual image and backgrounds were acquired for the near-field detector used during further image analysis. Furthermore, a stability test was performed each beam time to ensure the reproducibility of the results.

Before acquisition of each grain, a centering scan is done in the $x$- and $z$-direction and in $\omega$ to ensure that the grain is fully (and always equally) illuminated by the incoming beam. Centering must be done as grains move with tensile loading physically. A typical centering procedure is shortly described below. The sample is translated or rotated in small steps around the expected grain position and the intensity is detected. The sample is then moved to the position, where the center of intensity peak was detected.
A typical procedure is to start with an \( \omega \)-scan (around the expected \( \omega \)) to identify the correct angular range, where diffracted intensity can be detected from the selected grain. Then first an \( x \)-scan is done in the identified \( \omega \)-scan (the load frame is rotated through a given \( \omega \) interval for each \( x \)-position), where the sample is translated in small steps along the \( x \)-direction in the region, where the grain is expected. The sample is then moved to the position, where the center of intensity peak (which is identified by software using a Gauss-fit) was found. At this position the same procedure is repeated for the \( z \)-axis and the sample is then translated to the \( z \)-position found for the center of the intensity peak. This procedure is done before any HRRSM acquisition for a grain and very time-consuming. It is also used to identify grains on the nearfield detector to exclude neighbours with similar orientations.

The centering scan slightly vary for different acquisitions e.g. because the required fit for the intensity profile, which can cause intensity variation between the acquired HRRSM.

### 3.4.1.2 Near-field Detector

A near-field detector (cf. Figure 3.22a) is used to identify grains for further investigations from the diffraction pattern (cf. Figure 3.22b), which are fulfilling certain criteria. The grains are chosen from a limited \( \eta \)-range, which can be detected by the far-field detector (cf. Figure 3.22b, green square). It is furthermore of importance for the clarity of the analysis that the grains have no neighbours with similar orientation to avoid an overlap of the diffraction signals (e.g. only one visible maximum when doing a centering scan in the location and angle range of interest). Because the grains are illuminated and mapped at the far-field detector by a 10x50 \( \mu \)m beam, the grain size should be of 30-50 \( \mu \)m to allow a full illumination of the entire grain. By acquiring a set of diffraction patterns for various \( \omega \) angles and sample positions, different grains can be found in different areas in the sample. The grain size can be determined during a centering scan in \( x \) and \( z \) direction by scanning the diffraction spot through the beam and obtaining the integrated intensity profile and with this the profile width in dependence of the sample position. An appearance of double peaks in such an intensity profile of the centering scans can be an indication for a neighbouring grain with same orientation or location and must be avoided.

Data from the near-field detector can also be used to reconstruct the grain neighbourhood an all their orientations, which is a possibility for future investigations.
Figure 3.22: (a) Near-field detector PE used at PETRA, (b) Diffraction pattern of a pre-deformed AA1050 tensile sample acquired at the near-field detector for a certain sample position. The different rings diffracted from different fcc lattice planes (and with this different 2θ) are indicated. The green square indicates approximately the part of the ring that can be detected with the far-field detector.

3.4.1.3 Far-field Detector

The diffraction spots for grains found at determined sample positions can be observed with the far-field detector (cf. Figure 3.23) after moving the near-field detector out of the beam. Figure 3.24 gives an example for the different appearance of the diffraction spot on the far-field detector depending on the degree of deformation. The diffraction spots resulting from the annealed and undeformed sample (cf. Figure 3.24a) are distinct round spots and not broadened. Section 3.1 shows that the grains in the undeformed sample are nearly uniform in orientation with an undisturbed crystal lattice. Figure 3.24b and Figure 3.24c show typical examples for an image acquired at the far-field detector for cyclically deformed AA1050 in the experiments. It is clearly visible that a diffraction spot of the deformed samples are broadened. The broadening is due to lattice defects introduced into the microstructure during deformation. The distance from the detector to the location, where the beam
interacts with the sample is calculated from the desired $2\theta$, which is characteristic for the investigated material and the desired reflection.

Figure 3.23: Height-adjustable MarCCD Far-field detector used at PETRA.

Figure 3.24: Far-field raw image of a part of the 400 diffraction ring in (a) an undeformed and annealed AA1050 sample showing distinct round spots and (b) for another specimen without major tensile pre-deformation but after tension-compression cycling showing slightly broader spots. (c) Far-field raw image for a specimen initially deformed to 1% in tension and then cyclically deformed in tension-compression showing even broader spots.
Before acquisition of reciprocal space maps a centering scan as described in 3.4.1.1 is done to ensure that the whole grain is centered within the beam.

For HRRSM an entire sequence of two-dimensional images of the 400 diffraction peak are acquired with the far-field detector, while rocking the sample around the vertical axis perpendicular to the scattering plane in small intervals $\Delta \omega$ (typically 0.015° and 5 s exposure time) of the rocking angle $\omega$ with constant velocity. Each image is acquired at the sample positions from centering, while moving the rocking motor through an interval of 0.015° and measuring the intensity detected over the time. For this the loadframe is rotated in small angular intervals around the rotation axis, while exposing the sample for a certain time. As a constant speed during the acquisition interval $\Delta \omega$ is required, the rotation motor needs to perform a lot of different motions for accelerating and decelerating, which is very time-consuming. An $\omega$-range from $\omega_{\text{start}}$ to $\omega_{\text{end}}$, which should cover the entire reflection around $\omega_{\text{cen}}$, defined from the grain centering of the corresponding grain. The next slice is acquired for the same angular interval at a different angle. The sum of those intervals then covers the total $\omega$-range. An example for a typical image sequence is shown in Figure 3.25.

![Figure 3.25](image_url)

**Figure 3.25:** Raw far-field image data (acquired at APS) showing the (broadened) Bragg reflection of interest during different rocking intervals $\omega$ of 0.075° apart from each other. For overview reasons only 11 images of the sequence containing in total 50 images are displayed. The $\omega$ interval in the experiment was 0.015°.
Before final acquisition with fine $\omega$-intervals, an $\omega$ centering scan was done with the rotation motor to determine the $\omega$-range for the map. The final rocking curve in Figure 3.26 shows the integrated intensity in the image over the number of acquisition (or rather the continuously increasing rocking angle $\omega$). It shows that the $\omega$-range was chosen in a way to contain the maximum peak of the intensity of the selected Bragg reflection covering in this case a range from 0.515° to 1.265° with 50 images and an $\omega$ step size of 0.015° with the peak center at 0.9°.

![Rocking curve](image.png)

Figure 3.26: Rocking curve for the image sequence shown in Figure 3.25 indicating the number of acquisition and the position of the shown images are marked with blue arrows. The individual omega slices are acquired with an interval of 0.015°.

By stacking the images recorded for several adjacent $\omega$ intervals, three-dimensional distributions (two directions on the detector and the additional rocking direction) of the diffracted intensity are obtained representing three-dimensional reciprocal space maps of the reflection.

The time of the far-field detector between the image acquisitions is limited by the speed of the rotation motor. Overhead of the motor due to acceleration and deceleration is one of the major time limitations for the experiment. Future experiments can be optimized using a fast area detector such as the pixirad2, which can also provide smaller pixel sizes (60 $\mu$m) and with this a higher resolution.
3.4.2 Data analysis

The acquired data from the far-field detector was analysed extensively based on matlab scripts and algorithms developed by Wejdemann et al. [WEJ11]. Selected results for the different beam times are shown in the corresponding section.

Each raw image was corrected with regards to background, incoming beam intensity and cosmic rays before further analysis. Because of the time-consuming measurements the beam intensity can fluctuate and needs readjustment making a constant incoming beam intensity unobtainable. Hence the data needed to be corrected for the incoming beam intensity to allow a comparison of the results. Cosmic rays in form of exceptionally high intensity pixels were removed from the image. During acquisition, a region of interest (ROI) on the detector containing the entire reflection was determined to minimize the calculation time for data analysis. First a correction for the detector background was done in the region of interest by substracting the calculated mean intensity in areas on both sides of the reflection (as described in detail by [WEJ11]). The individual images (cf. Figure 3.27) were then normalized with the incoming beam intensity measured by a diode (PETRA) or split ion chamber (APS) during acquisition to correct for variations of the beam intensity.

After the corrections of each individual image, all images are stacked to produce a three dimensional reciprocal space map including the two detector directions ($x_{ROI}$ and $y_{ROI}$ corresponding to $\eta$ and $2\theta$) and one rocking direction $\omega$. 

Figure 3.27: Integrated detector image for a deformed grain after adding all far-field images for different $\omega$ angles together. The region of interest is defined for the $x$ direction ($x_{\text{ROI}}$) and $y$ direction ($y_{\text{ROI}}$).

The acquired three-dimensional intensity distributions of the selected Bragg reflection can be more easily assessed in two complementary projections: azimuthal projections and classical radial X-ray line profiles, which represent the distributions of lattice plane inclinations and normal strains, respectively.

3.4.2.1 Azimuthal Maps

Azimuthal maps are the projection of the three-dimensional intensity distribution onto the so-called azimuthal plane along the scattering vector showing local orientation differences (representing the different inclinations of (400) planes) indicated by locally different intensities (cf. Figure 3.28). The vertical axis in the map corresponds to the data acquired for different rocking angles $\omega$ and the horizontal axis to the detector direction along the ring, i.e. the azimuthal angle $\eta$ ($x_{\text{ROI}}$, 1 pixel corresponds to 0.0052°).
Figure 3.28: Typical example for an azimuthal map for a deformed grain (from first APS beam time) covering a rocking angle range $\omega$ from 0° to 0.75° (50 images with steps of 0.015°) and an $\eta$ range of 1.25°.

The individual azimuthal maps are scaled by their maximum intensity level, where the highest intensity fraction has been cut off. All measured intensities are sorted by the visualization software and for reasons of visual appearance a cut-off of the 250 highest intensity values was done. The maps are then scaled using the maximum value after the cut-off as a maximum value in the color-code. The azimuthal maps are in general put on the same scale (after taking all intensity values into consideration and then scaling by a common maximum value) for reasons of comparison, when they are presented as a sequence (e.g. for a load cycle). Meaning that the same intensity values correspond to the same colors i.e. red for high intensity and blue for low intensity.

To highlight desired features the azimuthal maps can be scaled linearly between their individual minimum and the maximum value including all pixels intensities, which will be mentioned in the figure subtext. This was done, for example, for azimuthal maps obtained from grains, which were not pre-deformed in tension (third APS beam time), because their intensity distribution is narrow due to the small plastic strain.

Figure 3.29 shows an example of azimuthal maps scaled linearly between zero and different maximum values. The maximum value is lower, when the 250 pixels with the highest intensity are shown saturated (corresponding to circa 2% of all pixels for the given example with 12050 pixels in total) in comparison
to when all pixels and their intensity are included to refine the scale. This is done to remove noise in the image (cf. normal probability plot) or to highlight the main peaks in the intensity distribution.

Figure 3.29: Example of azimuthal maps, where the intensity is scaled linearly between zero and different maximum intensities for the color scale. The maximum value for scaling is determined by the pixels with highest intensity. If the 250 pixels with highest intensity are not considered (top) the maximum intensity is lower and the scaled azimuthal map will appear different in case when all pixels and their intensities are included to define the color code, i.e. the maximum intense pixel defines the maximum of the color code (bottom). The normal probability plot (right) of the two intensity distributions shows that a deduction of the 250 pixels with highest intensity gives a less curved intensity distribution.

The heterogeneous intensity distribution in the azimuthal maps is composed of a component with high-intensity peaks and a spread out cloud of lower intensity. For further analysis, each azimuthal map is partitioned as illustrated in Figure 3.30 into a smooth cloud component and the peak component containing all high-intensity peaks using mathematical approaches [JAK06, WEJ11]. The cloud of lower intensity represents the dislocation-rich wall component, whereas the peak component constitutes the subgrain component and represents the sum of the diffracted intensities from all subgrains present in the grain.

Each high-intensity peak corresponding to a local maximum in the peak component, e.g. marked in Figure 3.30, represents a nearly dislocation-free subgrain and can be analysed individually. In this way,
individual subgrains can be identified (cf. example Figure 3.31) by their corresponding high-intensity peaks and traced from acquisition to acquisition. The peaks are sorted after their intensity starting with the highest number for the most intense peak and thus largest subgrain. For statistical analysis the peaks are usually grouped in groups from peaks 1-10, peaks 11-30 and peaks 31-80. In some cases, it cannot be excluded that the diffracted intensity may origin from more than one subgrain, in particular, for peaks of lower intensity, they will therefore be neglected in the further analysis [WEJ11].

Figure 3.30: Example for partitioning of the intensity distribution: (a) azimuthal map and the two corresponding extracted components, (b) the peak component comprising the high-intensity peaks from the subgrains (three subgrains are exemplarily marked by a white circle) and (c) the smooth cloud component originating from the dislocation walls.

Figure 3.31: Example for the automatic peak identification using matlab for the separated high intensity fraction of one azimuthal map. The peaks are numbered after their intensity (1 for the highest intensity) and can be traced manually from acquisition to acquisition. The three maps show the same azimuthal map of peak component in Figure 3.30 but highlight the different subgrains 1 to 10, 11 to 30 and 31 to 80.

For the analysis of the subgrain, projection is done for a restricted region $\eta$ and $\omega$. The azimuthal map contains in the first place no information about strains. The strains can be accessed by analyzing the radial profiles.
3.4.2.2 Radial Profiles

Radial profiles are the projection of the intensity distribution onto the radial direction (integration in the direction perpendicular to the diffraction vector) as described in detail by Wejdemann et al. [WEJ11]. They can be calculated for each reciprocal map taking the detector position into consideration. The value \( q \) is calculated at the corners of each pixel and the \( q \)-width of one pixel being \( 10^{-4} \text{Å}^{-1} \) for the presented set-up.

Radial profiles are either calculated for the entire map (grain) or for a restricted range of \( \omega \) and \( \eta \) (individual subgrain). Radial profiles are widely used in classical X-ray diffraction to determine interplanar spacings and lattice strains.

To calculate radial profiles the exact beam position has to be determined by the use of a reference measurements with CeO\(_2\) powder producing rings with known diffraction angles \( 2\theta \) on the far-field detector. From this the radius and the exact position (horizontal and vertical shift and exact distance) of the detector with regard to specimen and beam can be calculated.

Radial profiles can be described by several parameters. The diffraction vector \( q \) is related through the wave length lambda \( \lambda \) (here e.g. \( \lambda_{52keV} \) equal to \( 0.2384 \text{Å}^{-1} \) for the used X-rays at APS) to the Bragg angle \( (2\theta_{400,Al} \text{ equal to } 13.53^\circ).\)

From the earlier introduced Bragg equation 2.16 with the Bragg angle \( 2\theta \) and the X-ray wave length it follows that the diffraction vector is inversely proportional to the lattice spacing (cf. eq. 3.3). A larger lattice spacing results in a smaller \( q \). Tensile stresses will result in a larger lattice spacing \( d_{400} \) and thus in a lower values for the diffraction vector \( q \) and \( 2\theta \) (cf. eq. 3.4) resulting in a peak profile shift. Consequently, the local lattice parameter changes can be deduced from the shift of the radial profiles and the local elastic strain within the grain can be calculated from equation 3.5.

\[
q = \frac{2\pi}{d_{400}} \quad \text{using} \quad d_{400} = \frac{\lambda}{2\sin\theta} 
\]

\[
q = \frac{4\pi}{\lambda} \sin\theta 
\]

\[
\varepsilon = \frac{d - d_0}{d_0} = \frac{q_{\text{mean},L0}}{q_{\text{mean}} - 1}
\]
The profile of a heterogeneous microstructure of the grain with subgrains present corresponds according to the composite model [MUG02, PAN10] to a superposition of profiles (wall and all present subgrains) from the different structural elements existing within the grain as demonstrated in Figure 3.32. An ordered structure causes a superposition of at two subprofiles (wall and subgrain), so the profile appears asymmetric because of different internal stresses present in the heterogeneous microstructure causing variations in the diffraction vector $q_{\text{mean}}$ of the subprofiles. The asymmetry is therefore depending on the sign and local elastic strains between the subgrains and walls. Note that the separation of the wall and subgrain part is always done in the azimuthal maps not by curve fitting in the radial profile.

![Graph](https://via.placeholder.com/150)

**Figure 3.32:** Example for the separation of the grain profile (black) into a profile from the wall part (blue) and profile from all subgrains (red). Additionally the profiles of the ten most intense subgrains (red, thin lines) normalized to the corresponding maximum intensity. The grain profile is the sum of the (blue) wall and the (red) subgrain profile. The red subgrain profile is the sum of all existing subgrain profiles (here only ten selected for clarity) according to the revised composite model [PAN10].

Radial profiles can be calculated for each high intensity peak (equal to a subgrain) and characterized in the same manner with regards to position, width and asymmetry as the radial profile for the grain in the way described above. However, only the position is of interest here giving information about the local elastic strain experienced by the individual subgrain.
In order to reveal internal stresses and strains, the average peak positions of the subgrain profiles are compared to the average position of the entire grain they belong to. Based on the measured peak shift $\Delta q$ between the subgrain profile and the grain profile the internal elastic strain $\varepsilon$ of an individual subgrain with respect to the entire grain can be calculated directly.

$$\Delta q = q_{\text{subgrain}} - q_{\text{mean}}$$ (3.6)

A positive peak shift $\Delta q$ of a specific subgrain profile is associated with an elastic compressive strains of the corresponding subgrain. The local elastic strain of each subgrain embedded within the measured grain is calculated according to equation 3.7.

$$\varepsilon = \frac{d_{\text{subgrain}} - d_{\text{mean}}}{d_{\text{mean}}} = \frac{q_{\text{grain}} - q_{\text{subgrain}}}{q_{\text{subgrain}}} = \frac{\Delta q}{q_{\text{subgrain}}}$$ (3.7)

In this way information about the local stress-state (e.g., compressive, tensile stresses) of individual subgrains can be gained. In general only the peak shift $\Delta q$ was calculated and presented, but a clear relation to the difference between the subgrain profile position and the peak profile position can be made and from this a direct relation to $\Delta q_w$ using the fraction for the walls $f_w$ and $f_c$ for the fraction of the cells.

$$\Delta q_w = \frac{f_w}{f_c} \Delta q$$ (3.8)

Figure 3.33 visualizes the characteristic parameters used for the description of the appearance of the radial profile on one example.

The position of the radial profiles is characterized by either the mean position $q_{\text{mean}}$ or the position at maximum intensity $q_{\text{max}}$. The $q_{\text{mean}}$ of the fit profile is the same as $q_{\text{mean}}$ of the original profile (originating from the programming of the software).
Figure 3.33: Example for a radial profile with the characteristic parameters used for description. The profile position can be described by the \( q \)-value at maximum intensity \( q_{\text{max}} \) (red line) or the averaged \( q_{\text{mean}} \) (black line). The profile width is described by the integral width \( \beta \) (green arrow) or the Full Width at Half Maximum (FWHM, red arrows), which is split up in FWHM\(_{\text{low}}\) and FWHM\(_{\text{high}}\). The square indicates the \( q \)-width used for calculation of the integral width.

Furthermore the width of the profile can be characterized and three different values are used for this. The main value for characterizing the profile width is the integral width \( \beta \). The integral width is the integrated intensity \( I_{\text{int}} \) of the peak, in a range of defined \( q \)-values (here with at least 1/50 of the maximum intensity) divided by the maximum intensity \( I_{\text{max}} \) as defined in equation 3.9.

\[
\beta = \frac{I_{\text{int}}}{I_{\text{max}}} \tag{3.9}
\]

It is equivalent to the area of the rectangle as visualized in Figure 3.33, which has the same maximum as the maximum intensity of the profile and the same area as the area under the peak. The integral width is determined from the original profile. The integral width of the fit profile has in the software the same value as the integral width of the original profile.
Secondly the full width at half maximum (FWHM) is used to characterize the profile width. The total FWHM is calculated for the original profile (without fit) and the same value is overwritten to the fit profile.

Profiles can be fitted using a split pseudo-Voigt profile. It is a combination of two half pseudo-Voigt profiles, which are a linear combination of a Gauss profile and a Lorentz profile. The two half pseudo-Voigt profiles are on each side of the maximum intensity to allow fitting of an asymmetric profile. A split pseudo-Voigt profile is given by equation 3.10, where \( I_{\text{max}} \) corresponds to the maximum intensity, \( f_L \) to the Lorentz fraction, and \( q_{\text{max}} \) for the position of the maximum intensity. The two variables \( WHM_{\text{low}} \) and \( WHM_{\text{high}} \) are the half width at maximum intensity at the side of lower \( q \)-values than the maximum intensity and at the side of higher \( q \)-values than the maximum intensity (cf. Figure 3.33). [WEJ11].

\[
I(q) = I_{\text{max}} \times \begin{cases} 
  f_L \left[ 1 + \left( \frac{q - q_{\text{max}}}{WHM_{\text{low}}} \right)^2 \right]^{-1} + (1 - f_L) \left[ \exp \left( -\frac{(q - q_{\text{max}})^2}{WHM_{\text{low}}^2 / \ln 2} \right) \right] & \text{for } q \leq q_{\text{max}} \\
  f_L \left[ 1 + \left( \frac{q - q_{\text{max}}}{WHM_{\text{high}}} \right)^2 \right]^{-1} + (1 - f_L) \left[ \exp \left( -\frac{(q - q_{\text{max}})^2}{WHM_{\text{high}}^2 / \ln 2} \right) \right] & \text{for } q > q_{\text{max}} 
\end{cases}
\]  

\[
FWHM = WHM_{\text{high}} + WHM_{\text{low}} = \frac{FWHM_{\text{high}}}{2} + \frac{FWHM_{\text{low}}}{2}
\]  

The WHM measured for lower \( q \)-values than \( q_{\text{max}} \) (\( WHM_{\text{low}} \)) and the FWHM measured for higher \( q \)-values than \( q_{\text{max}} \) (\( WHM_{\text{high}} \)) are used to calculated the profile asymmetry. They are in sum the total FWHM as visible in Figure 3.33.

The last width is the FWHM of the fitted profile \( FWHM_{\text{fit}} \). This is calculated as half the sum of \( FWHM_{\text{low}} \) and \( FWHM_{\text{high}} \).
The profile width is besides changes in the dislocation density strongly influenced by the present internal stresses. The presence of ordered structures and the extent of internal stresses can be estimated from the profile asymmetry.

The asymmetry is calculated for the fitted profile only and can be displayed in three different ways:

1. “Naive” asymmetry
The so-called “naive” asymmetry gives a first estimation about the profile shape from the magnitude of the difference between the position of the maximum \( q_{\text{max}} \) from the fitted profiles and to the average \( q_{\text{mean}} \) according to equation (3.12).

\[
\kappa_{\text{naive}} = q_{\text{max, fit}} - q_{\text{mean, meas}} \quad (3.12)
\]

2. Absolute asymmetry
In the following the absolute asymmetry value as given in equation (3.13) is usually used to quantify the asymmetry behaviour. This is the difference between the FWHM measured at lower \( q \)-values and the FWHM measured at higher \( q \)-values than \( q_{\text{max}} \).

\[
\kappa_{\text{abs}} = \text{FWHM}_{\text{low}} - \text{FWHM}_{\text{high}} \quad (3.13)
\]

3. Relative asymmetry
Another way to display the asymmetry is the relative asymmetry calculated from the FWHM as given in equation (3.14).

\[
\kappa_{\text{rel}} = \frac{\text{FWHM}_{\text{low}} - \text{FWHM}_{\text{high}}}{\text{FWHM}_{\text{low}} + \text{FWHM}_{\text{high}}} \quad (3.14)
\]

In all investigated cases is the diffraction vector almost parallel to the tensile axis (this condition corresponds to the axial case in literature). The asymmetry measures are constructed in such a manner that all have positive values for profiles with a longer tail at the low \( q \) range.
4 Experimental Results

This section summarizes selected results from initial electron microscopy investigations (section 4.1) and four beam times applying High Resolution Reciprocal Space Mapping (section 4.2-4.5). Both, grains and load steps, are named with regards to each individual beam time, hence in some occasions the same name are used in different beam times. This means grain 2 of e.g. the first APS beam time (section 4.2) is a different grain than grain 2 for the third APS beam time (section 4.5) and the results of each section are independent of each other. A cycling sequence is defined as the cyclic deformation of a sample with a selected number of cycles and strain amplitudes. The cycling was interrupted after selected numbers of cycles and HRRSM measurements were obtained. The cycling was then continued after measurements. An individual load cycle is defined as one full cyclic stress-strain hysteresis. When investigating the material during an individual load cycle, the deformation was interrupted at selected stress-strain conditions and continued after measurement. A cycling sequence consists in principle of many load cycles.

4.1 Microstructural Evolution as investigated by Electron Microscopy

To gain further insight into the microstructure of aluminium after cyclic deformation and to determine the testing parameters used in HRRSM experiments, tensile samples of the same material and the same heat treatment were mechanically tested using the MTS Acumen at DTU MEK. To reduce manufacturing costs of the bone-shaped samples, some of the experiments were done with stripes of the dimensions 32mm x 9mm x 1 mm (similar dimensions of the bone-shaped samples though without the notches) annealed for two hours at 600 °C. Cross sections along the plane perpendicular to the tensile axis were prepared metallographically for further investigations using electron microscopy as described in the experimental methods.
4.1.1 Uniaxial Deformation

Some of the samples investigated during the beam times were pre-deformed in tension. To investigate the microstructure after tensile deformation further and to compare it with the microstructure developed after cyclic deformation, electron microscopy analysis was done on samples after uniaxial tensile deformation. The tensile deformation was done on annealed AA1050 samples using the MTS Acumen with a grip speed of 0.015 mm/s in a similar way to the pre-deformation of the investigated samples as shown in Figure 3.4. The samples samples differ however in their gauge width and are not bone-shaped. Hence the presented tensile curve in Figure 4.1 differs from the curve in Figure 3.4.

![Tensile test curve](image)

Figure 4.1: Tensile test curve of an annealed AA1050 sample (in stripe shape) with indicated strains based on a 15 mm gauge length. The arrows mark the strains, at which tests were interrupted for the different samples investigated.

Tensile deformation has been terminated at different strains along the stress-strain curve to perform microstructural investigations. Figure 4.2 shows first an EBSD map of the grain structure after annealing without deformation and after 5% tensile deformation. Not many changes within the grains are visible in the undeformed condition despite the fine step size. There is however an extreme texture difference between the two presented areas, they are hence not fully representative. Figure 4.3 shows the
exemplary grain structure for a tensile sample deformed to 10%, 30%, 45% and 50% tensile deformation. A clear elongation of the grains in tensile direction and a developing substructure is visible. The orientation indexing quality of the recorded diffraction pattern is decreasing with increasing deformation due to an increased amount of dislocations and lattice distortions. The quality of the preparation on the other hand increases slightly with stronger deformation due to work-hardening of the material. Several maps were acquired with different step sizes to determine a suitable step size for revealing the features of interest. Step sizes of 500 nm was found to be fine enough to uncover the relevant features. A finer step size was used for less deformed samples in Figure 4.3 (note that a higher magnification was used as well for time reasons), to allow revealing possibly less clearly pronounced features in comparison to samples after higher deformation.

![Figure 4.2](image)

**Figure 4.2:** Orientation maps (colored according to the crystallographic direction along the tensile direction) for samples after (a) annealing and 0% (b) after 5% uniaxial tensile deformation. Black areas are due to indexing problems because of a poor sample preparation. The step size was 250 nm with 20 ms exposure time. The tensile axis is oriented vertically.
Figure 4.3: Orientation maps acquired by EBSD (colored according to crystallographic direction along the tensile direction) for samples deformed to a different degrees of in uniaxial tension acquired with the same magnification for (a) 10% (b) 30% (c) 45% (d) after fracture at ca. 50%. The step size was 500 nm with 10 ms exposure time. Because of increasing indexing difficulties due to deformation the indexed pattern for (d) was averaged over 3 frames. The tensile axis is oriented vertically.

From these investigations it can be seen that a clear tensile deformation structure is first formed in the samples investigated after 10% tensile deformation and higher. Only some indications for intragranular changes are visible in the red grains in Figure 4.2b after 5% deformation. The fact that red grains are less substructured, is most likely an artefact of the color code, which might be an effect of the visibility of the different shades of red.
Selected grains were imaged in detail by orientation contrast imaging using BSE. A visualization of the same grains as mapped by EBSD using BSE imaging is challenging, since the imaging geometry is different. The sample is not tilted for BSE imaging but tilted 70 ° for EBSD. Figure 4.4 shows how the grains were re-identified using a hardness indent as marker for orientation. The fine EBSD maps were usually acquired in a reasonable distance far from the hardness indent, to ensure no influence of the indent. Some grains were selected for detailed images shown in Figure 4.5 and Figure 4.6.

![Figure 4.4: Identification of grains mapped by EBSD using BSE. Left: BSE image including the hardness indent on the top middle (marked with a circle) imaging the grain structure at a low magnification. Right top: coarse EBSD orientation map including the hardness indent on the top middle and right bottom: fine EBSD map as shown in Figure 4.3b. By scanning the sample with a coarse step size in the surrounding of the hardness indent, the same region as mapped in a fine step size by EBSD can be identified. Grains can then be refound by their shape and internal structure (exemplary marked by arrows). Some grains were selected for detailed imaging and are marked with a name K1-K4. The tensile axis is oriented vertically.](image)

The highly magnified image in Figure 4.5 of the internal grain structure of K1 shows a clear ladder structure as observed for fcc metals like copper after tensile deformation [HUA98, HUA01]. The subgrain structure shown in Figure 4.5 seems to follow clear directions of approximately 45 ° towards
the tensile axis and the cell sizes vary in size from about 0.5 µm – 5 µm. A more wavy appearance of the subgrain structure was observed in other grains (cf. Figure 4.6c).

Figure 4.5: SEM BSE image of a region K1 within a grain after 30% tensile deformation (marked with a blue square). A ladder structure indicating multiple slip with varying cell sizes is clearly visible. The tensile axis is oriented vertically.
Figure 4.6: SEM BSE images of region (a) K2 (b) K3 (c) K4 after 30% tensile deformation showing a similar structure. The ladder structure can appear more wavy in highly substructure grains as visible for K4. The tensile axis is oriented vertically.

4.1.2 Cyclic Deformation

The following section intends to give an insight in the microstructural evolution during cyclic deformation in AA1050 for different cycling conditions and confirms the presence of subgrains. Various samples have been investigated by metallography and electron microscopy after cyclic deformation testing done using the MTS Acumen. The morphology of the subgrain structures and their appearance after cycling were studied. Challenges were encountered with the sample preparation for ECCI and EBSD of the commercially pure aluminium AA1050 material in the soft, annealed condition, but it was still possible to follow the evolution of the microstructure despite some artefacts, appearing as black features on EBSD maps as well as on microscopy images.

Different cycling sequences were investigated for displacement amplitudes between 2.5 µm and 40 µm corresponding to a range of strain amplitudes from $1.6 \times 10^{-4}$ to $2.7 \times 10^{-3}$. Cycling was done during the beam time experiments with a strain amplitude of $6 \times 10^{-4}$ to $7 \times 10^{-4}$, which corresponds to a displacement amplitude of 10 µm, or lower. To investigate whether the material would develop the subgrain structures, which were of interest for the investigations applying HRRSM, microscopy was initially done on samples cyclically deformed with higher strain amplitudes than the samples investigated later with
HRRSM. Investigations with ECCI of selected samples cycled with a strain amplitude of 6.7·10⁻⁴ commensurate with the strain amplitude applied during beam times showed however, that similar structures are existent in the samples cycled with lower strain amplitudes.

Two cyclic deformation sequences for two different strain amplitudes were investigated in detail by SEM applying ECCI and EBSD. All investigated samples were pre-deformed to 1% tensile strain before further cycling. In the presented image the tensile direction of the sample is always oriented vertically.

- The first series was done for a strain amplitude of 1.3·10⁻³ (corresponding to a displacement amplitude of 20 μm) with a strain rate of 10⁻³ s⁻¹ and a cycling frequency of 10 Hz. Samples were investigated with EBSD after selected number of cycles. Failure occurred at a varying number of cycles between 60000 – 90000 cycles. Three samples were investigated after failure at 62500 cycles, 70050 cycles and 89150 cycles.

- Another series was performed with a strain amplitude of 2.7·10⁻³ (corresponding to a displacement amplitude of 40 μm) with a strain rate of 10⁻³ s⁻¹ and a frequency of 10 Hz. Fracture occurred, much earlier than for the lower strain amplitude, i.e. after 12500 cycles. Samples were investigated with EBSD after selected number of cycles.

Some samples cycled with a strain amplitude of 0.67·10⁻³ were investigated as well, but were only imaged with backscattered electron contrast in the SEM.

For comparison with the microstructure before cyclic deformation, the reader is referred to chapter 3.1, where the microstructure of the annealed condition without deformation was presented.

4.1.2.1 Investigations of the surface structure

After cyclic deformation an increased surface roughness on the center of the gauge could be identified by visual inspection. To document the change in surface roughness, the surface of the gauge was investigated before metallographic preparation. The surface of the gauge was first imaged with a stereomicroscope Leica MZ125 as exemplarily shown for the test series cycled with a strain amplitude of εₐ = 2.6·10⁻³ shown in Figure 4.7. An increase in surface roughness has already occurred after 100
cycles. Both the width of the region with increased surface roughness and the magnitude of the roughness seem to increase with increasing number of cycles. The surface roughness (Ra and Rz) was measured with a handheld roughness measurer Mahr MarSurf PS1 using a total measurement length of 5.6 mm. The roughness was measured in the center of the gauge, perpendicular to the tensile axis, though the location slightly varied between samples depending on the exact location, where the roughness was apparent. The results should therefore only be used for a qualitative interpretation, since the roughness depends on the exact chosen location. The results presented in Figure 4.8 for Ra and Rz are the average of three measurements along three different lines. The graphs showing the average roughness values show a clear increase in surface roughness with increasing number of cycles. For both samples, the roughness increases only slowly during the first 1000 cycles before a steep increase in the surface roughness can be detected. The surface roughness increases earlier for the samples with a higher strain amplitude. The sample, which was investigated after a number of cycles close to the number of cycles, where failure was observed, shows higher average roughness values for the higher strain amplitude e.g. the series for εa = 2.6·10⁻³ shows a higher final value of Ra and Rz being nearly 3.5 µm and the series for εa = 1.3·10⁻³ shows a lower final value of only 2.5 µm.
Figure 4.7: Stereomicroscope images of the center of the gauge after different number of cycles with a strain amplitude of $\varepsilon_a = 2.6 \cdot 10^{-3}$ to show exemplarily the increase of surface roughness both in width and distinctness with increasing number of cycles. The markings unfortunately denoted the number of cycles performed before further investigation. The tensile axis is oriented horizontally.

Figure 4.8: Average $R_a$ and $R_z$ values for qualitative roughness measurements obtained for two cycling series with a strain amplitude of (a) $\varepsilon_a = 1.3 \cdot 10^{-3}$ and (b) $\varepsilon_a = 2.6 \cdot 10^{-3}$. 

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Secondary electron SEM images were obtained from selected samples to image the surface roughness at high magnification (cf. Figure 4.9). A characteristic symmetric pattern was observed indicating that the surface roughness can be related to the intrusions and extrusions described by [LI10]. These features are a consequence of persistent slip lines or dislocation walls appearing as a sharp pattern on the sample surface. [LI09, VID96a] The surface structure reappeared after removal by grinding and continued cycling for 1000 additional cycles. This confirms them as persistent slip lines.

Using the BSE detector for a top-view on the sample a clear relation of the apparent structure and the different grains becomes visible as shown in Figure 4.10. The slip lines seem to depend in their extend and orientation on the individual grain. They are terminated at the grain boundaries, which confirms that they are crystallographic and not macroscopic. This was also stated by [ARN96], who described an orientation dependence of the visible surface features for cyclically deformed aluminium.
Figure 4.9: SEM-SE images of the surface roughness observed after 1000 cycles with a strain amplitude of $\varepsilon_a = 2.6 \cdot 10^{-3}$ with increasing magnification from (a) to (d). A wavy macroscopic topography is visible in (a), which consists of areas with parallel intrusions and extrusions as magnified in (d).

Figure 4.10: SEM-BSE images showing a clear relation of the appearance of the extrusions and the individual grains. (a) For several grains and (b) at higher magnification for a triple point between grains in the center of the image. The slip lines terminate at the grain boundaries and are of different orientation and extend in the different grains.

As described in section 2.2.3 it was discussed, that the appearance of intense slip banding is an indication for the presence of ordered structures within the grains during cyclic deformation [CHAR89, MIT69, MIT70, VOR87, VOR88, ZHA96a]. Subgrain structures were however also present in samples
cyclically deformed at lower strain amplitudes, which did not show a macroscopic surface roughness. So ordered structures are required for surface roughness, but from the absence of surface roughness the absence of ordered structures cannot be concluded.

4.1.2.2 SEM investigations of the microstructure

Figure 4.11 shows orientation maps acquired with EBSD after 2000 cycles, 5000 cycles, 20 000 cycles and after failure at 62500 cycles to show the subgrain formation during the cyclic deformation with $\varepsilon_a = 1.3 \cdot 10^{-3}$. No subgrains or internal misorientations are clearly discernible in the grain structure after annealing (cf. Figure 3.2 and Figure 4.2a) as well as after the first 2000 cycles (cf. Figure 4.11a). The orientation map obtained after 2000 cycles also shows that the pre-deformation to 1% tensile strain and the experience of compressive load during mounting did not visibly influence in the microstructure as resolved by EBSD. After 5000 cycles however, the intragranular microstructure has changed and orientation differences (in form of color variations) are visible within grains. The substructuring of the grains seems to increase with the number of cycles until failure. The investigated regions were far from the fracture surface, hence no clear indications of cracking are visible in the images. It is difficult to see the increased amount of subgrain structure in the orientation maps, which are colored to highlight the individual grains. With this color-code it can be difficult to get a proper impression of the extend of the present substructure. Therefore, the corresponding Image Quality maps are presented in Figure 4.12. They show more clearly the progressive and localized changes due to the formation of a substructure with increased number of cycles.
Figure 4.11: Orientation maps acquired by EBSD at the same magnification for samples after different number of cycles for $\varepsilon_s = 1.3 \cdot 10^{-3}$ (colored according to the crystallographic direction along the tensile direction and the color legend IPF in (a)). (a) After 2000 cycles, (b) after 5000 cycles, (c) after 20000 cycles, (d) after failure at 62500 cycles. A clear evolution of the intragranular structure is visible. The subdivision seems to increase with number of cycles. Subgrains were first discernible in the sample investigated after 5000 cycles. All maps were acquired for a similar area with a comparable magnification. The step size was 250 nm.
Figure 4.12: Image Quality acquired by EBSD at the same magnification for samples after different number of cycles for $\epsilon_a = 1.3 \cdot 10^{-3}$ (a) after 2000 cycles (b) after 5000 cycles (c) after 20 000 cycles (d) after failure at 62500 cycles. All maps are acquired for a similar area with a comparable magnification. The stepsize is 250 nm.

Similar investigations as for the first series were made with a strain amplitude of $\epsilon_a = 2.6 \cdot 10^{-3}$ twice as high as the one presented already. Figure 4.13 shows an example for orientation maps acquired with EBSD for samples after 1000 cycles, 2000 cycles, 5000 cycles and after failure at 12500 cycles. Evidence for subgrain formation is already visible after 1000 cycles and strongly present after 5000 cycles.
Figure 4.13: Orientation maps acquired by EBSD at the same magnification for samples after different number of cycles for $\varepsilon = 2.6 \cdot 10^{-3}$ (colored according to the crystallographic direction along the tensile direction and the color legend IPF in (a)). (a) After 1000 cycles, (b) after 2000 cycles, (c) after 5000 cycles, (d) after failure at 12500 cycles. All maps were acquired for a similar area with a comparable magnification. The step size was 250 nm.
Figure 4.14: Image Quality acquired by EBSD at the same magnification for samples after different number of cycles for $\varepsilon_a = 2.6 \times 10^{-3}$ (a) after 1000 cycles (b) after 2000 cycles (c) after 5000 cycles (d) after failure at 12500 cycles. All maps were acquired for a similar area with a comparable magnification. The stepsize was 250 nm.

Selected samples were investigated using backscattered electron contrast to image the local subgrain structures present within selected grains. The exact conditions of the samples (number of cycles and strain amplitude) are mentioned in the figure caption. It was found, that both elongated structures and equiaxed structures were present in the same samples with equiaxed structures being the predominant microstructure. Individual subgrains are easily recognized by their distinct orientation reflected in a different shade of grey. Subgrains were and visible in nearly all investigated grains. In some cases, the subgrains appeared to be aligned in “linear structures” (eg. cf. Figure 4.15a+b+c, parallel lines). Some grains contained domains, where only a limited part of the grain was highly substructured (cf. Figure
4.15b) into subgrains (or subgrains of different sizes (cf. Figure 4.15f), or where different domains of the grain showed different substructure morphologies such as elongated structures and equiaxed subgrains (cf. Figure 4.15c).
Figure 4.15: Exemplary SEM-BSE images showing the appearance of subgrains in AA1050. (a) 40500 cycles, $\varepsilon_s = 0.67 \cdot 10^{-3}$ (b) 5000 cycles, $\varepsilon_s = 1.3 \cdot 10^{-3}$ (c) 5000 cycles, $\varepsilon_s = 1.3 \cdot 10^{-3}$ (d) 5000 cycles, $\varepsilon_s = 2.6 \cdot 10^{-3}$ (e) 10000 cycles, $\varepsilon_s = 0.67 \cdot 10^{-3}$ (f) 10000 cycles, $\varepsilon_s = 1.3 \cdot 10^{-3}$. Examples for aligned subgrains are indicated by blue lines.

In some grains elongated structures were observed (cf. Figure 4.16). The elongated structures sometimes appeared to be composed of long parallel walls with nearly dislocation-free interior (as far as it was possible to image). This confirms statements of literature, where elongated or ladder like structures were observed in aluminium single crystals with TEM [GRO63a].
Another finding was continuation of the microstructural features across grain boundaries. Different examples are shown in Figure 4.17. Differently than what was observed for ladder structures in copper, the elongated or equiaxed structures seem sometimes to cross grain boundaries (e.g. Figure 4.17b). Only a few selected grain boundaries are showing this phenomenon, which needs further investigations. This could be an artefact of the technique, where a channeling contrast can be from another grain below, when the grain boundary has a shallow angle. In some cases a wave-like progression of the microstructural changes that possibly initiated at grain boundaries is visible. Further studies are though needed to fully document these observations, but they clearly show that the local environment is possibly strongly influencing the formation of individual subgrains.
Figure 4.17: Exemplary SEM-BSE images showing subgrain structures seemingly continuing over grain boundaries. (a) 10000 cycles, $\varepsilon_a = 2 \cdot 10^{-3}$ (b) 5000 cycles, $\varepsilon_a = 1.3 \cdot 10^{-3}$ (c) 5000 cycles, $\varepsilon_a = 1.3 \cdot 10^{-3}$ (d) 5000 cycles, $\varepsilon_a = 1.3 \cdot 10^{-3}$, wavelike cells observed next to a triple point (at the right top, not in image).

Backscattered electrons provide a high angular resolution and can be used to image dislocations in high resolution FEG SEMs, if the microscope settings are set accordingly. A first approach was done to image dislocations, which in the present case appear as bright, smeary (due to limited resolution achieved by the microscope settings) features within grains (cf. Figure 4.18d+f+g) or at subgrain boundaries (cf. Figure 4.18b+c+h). By tilting the sample in rather small angular steps, some features can be highlighted. Larger subgrains or elongated structures often showed the presence of dislocations appearing to organize themselves into walls cf. Figure 4.18a+e+g). The parallel appearance of equiaxed subgrains
and the presence of elongated structures in the grains with indication for cells indicate that the observed elongated structures may be a prior stage to the subgrain formation in deformed aluminium.
EBSD was used to give a first impression of the extend of microstructural changes. However, no clear relation between a preferred orientation for microstructural changes is visible. Selected grains in the sample taken out after in total 5000 cycles with a strain amplitude of $\varepsilon_a = 1.3 \cdot 10^{-3}$ were investigated additionally with EBSD and a finer step size of 125 nm and after using backscattered electrons to image the local subgrain structures for comparison. The obtained orientation maps for one example are shown in Figure 4.19. A grain showing local subgrain formation in one region of the grain was imaged by ECCI. A similar region (slightly larger than the SEM image) of the same grain was analysed by EBSD. The three obtained orientation maps color-coded in regard to RD, TD and ND are presented. It is visible, that the structure can be partly revealed and orientation changes of the region are indicated (in the first and third map). But with the limited resolution of EBSD it was not possible to measure the individual subgrains with the color coding used.
Figure 4.19: Example for a grain showing subgrain formation in a sample cyclically deformed for 5000 cycles with a strain amplitude $\varepsilon_s = 1.3 \cdot 10^{-3}$ (part of fig. b) analysed complementary with ECCI and EBSD. Top left and top middle: SEM-BSE image of the grain showing subgrain formation. Top right: IQ map acquired by EBSD of nearly the same region (indicated on the SEM image with an orange dashed rectangle). Bottom left to right: Orientation maps color coded with respect to the crystallographic direction along RD, TD and ND. The used color code can reveal the subgrain structures only to a limited extend. A step size of 125 nm was used.

In summary the investigations revealed the expected features in the form of equiaxed subgrain structures developing as a result of cyclic deformation. The size of subgrains varies between 1 – 4 µm. Figure 4.15a and Figure 4.15e confirmed that subgrains were observed for strain amplitudes and number of cycles relevant for the presented HRRSM investigations as well. The investigations revealed several interesting features i.e. elongated structures and possibly structures crossing grain boundaries. However further work is needed to further document and substantiate these findings. This could for example be done by statistically analyzing a certain number of grains and relating
the observed microstructure to the grain orientation and the nature of the grain boundary as it was done for copper [WIN81] and nickel [BUQ01].

The EBSD technique was demonstrated to be limited, when trying to resolve individual subgrains. In the following cyclically deformed aluminium will be analysed for various conditions of cyclic deformation in-situ and non destructive by HRRSM to gain a further insight into the behaviour of individual subgrains.
4.2 Tension-tension cycling sequence after pre-deformation (APS1)

First attempts on monitoring the microstructure during cyclic deformation using HRRSM were made. For this a pre-deformed sample cycled in tension-tension into saturation was investigated during a further tension-tension cycling sequence. It was found that no major changes occur in the later cycling stage, since it was possible to follow the same subgrain over 7350 cycles. Minor changes in the radial profile width and asymmetry were however observed after the initial 800 cycles after a tensile loading indicating structural reorganization during cycling after loading.

4.2.1 Sample Deformation

4.2.1.1 Pre-deformation

Prior to the in-situ investigation by HRRSM, cyclic pre-deformation in tension was carried out in order to introduce a microstructure conform to cyclic deformation in the specimen using MTS Acumen. The investigated sample was initially deformed by 1% in tension with a cross head speed of 0.015 mm/s and then cycled in tension-tension at a rate of 0.5 Hz under displacement control with a displacement amplitude of 10 µm corresponding to a nominal engineering strain amplitude \( \varepsilon_a = 6.7 \times 10^{-4} \). 18000 cycles were performed by repeatedly decreasing the displacement by 20 µm from the maximal displacement of 150 µm achieved after tensile deformation to 1% and increasing it again to 150 µm, so the maximum tensile load was not exceeded during cycling. During the cyclic deformation, a stress ratio \( R \) between the stresses at minimum and maximum load of 0.65 was observed.

4.2.1.2 In-Situ Deformation

For the synchrotron investigations, the sample was equipped with a pre-wired strain gauge Omega KFG-3 350 Ω glued onto the center of the gauge section and aligned with the tension axis to monitor the axial strain in-situ.
Four individual grains of similar orientation with their crystallographic [001] direction along the loading axis have been selected after mounting and tensile loading using the near-field detector in the pre-deformed specimen. High resolution reciprocal space maps for each of them have been obtained for 11 different loading steps. The details of the loading steps are summarized in Table 4.1.

<table>
<thead>
<tr>
<th>Load step</th>
<th>Acquisition after</th>
</tr>
</thead>
<tbody>
<tr>
<td>L0</td>
<td>Pre-deformation to $\varepsilon=1%$, 18000 cycles, $\varepsilon_a=6.7\cdot10^{-4}$ + Mounting and tensile loading</td>
</tr>
<tr>
<td>C1 – C3</td>
<td>$\varepsilon_a=0.8\cdot10^{-4}$, 7155 cycles,</td>
</tr>
<tr>
<td>L1</td>
<td>Loading to $\varepsilon_a=0.1%$, i.e. 1.1% total tensile strain</td>
</tr>
<tr>
<td>L2</td>
<td>Loading to $\varepsilon_a=0.3%$, i.e. 1.3% total tensile strain</td>
</tr>
<tr>
<td>C4</td>
<td>$\varepsilon_a=2.3\cdot10^{-4}$, 800 cycles</td>
</tr>
<tr>
<td>C5</td>
<td>$\varepsilon_a=2.3\cdot10^{-4}$, 2650 cycles</td>
</tr>
<tr>
<td>C6</td>
<td>$\varepsilon_a=2.3\cdot10^{-4}$, 550 cycles</td>
</tr>
<tr>
<td>C7</td>
<td>$\varepsilon_a=2.3\cdot10^{-4}$, 1150 cycles</td>
</tr>
<tr>
<td>C8</td>
<td>$\varepsilon_a=2.3\cdot10^{-4}$, 2200 cycles</td>
</tr>
</tbody>
</table>

Table 4.1: Designation of load steps for acquisition.

The sample was first loaded elastically in uni-axial tension to the highest stress experienced during pre-deformation (load step L0) so that the stress corresponded to the one measured after 1% pre-deformation in tension with the MTS Acumen. Tension-tension cycling was then performed with a low strain amplitude such that the displacement during cycling were lowered and never exceeded the displacement after the initial uni-axial loading step L0. The sample was cycled for 7155 cycles in total (load steps C1-C3) in position control of the movable cross head. A constant displacement amplitude (similar to the one used at pre-deformation) for the cross head was chosen causing an effective
macroscopic strain amplitude $\varepsilon_a$ of $0.8 \cdot 10^{-4}$ as measured by the strain gauge (rather than the nominal strain amplitude determined from the cross head displacement). In later experiments the cycling displacement amplitude was determined by the measured strain, in this way larger strain amplitudes were achieved. With the strain amplitude of $\varepsilon_a = 0.8 \cdot 10^{-4}$ a lower stress ratio $R$ of 0.29 than during the pre-deformation was experienced.

Earlier experiments applying HRRSM were done at strongly deformed samples resulting in extensive peak broadening. As neither the pre-deformation, nor these first cycles caused a significant broadening of the diffraction peaks to the desired extent (to apply the technique in the established way), the sample was further loaded in uni-directional tension by an additional macroscopic strain $\Delta\varepsilon_a$ of 0.1% (L1) and 0.3% (L2), i.e. to a total tensile strain (macroscopic strain plus nominal pre-strain of 1%) of 1.1% and 1.3% respectively, the latter being the highest strain the sample experienced during the entire experiment. The displacement achieved at the end of L2, i.e. after 1.3% strain, served again as maximal displacement of the cross head during the following cycling sequence. Further tension-tension cycling sequences (load steps C4-C8) were then performed under position control of the cross head, by decreasing the displacement by a larger amount corresponding to a macroscopic strain amplitude $\varepsilon_a$ of $2.3 \cdot 10^{-4}$.

This second cycling with in total 7350 cycles (C8) after loading to a total tensile strain of 1.3% (L2) is discussed in detail.

After each loading step (L0, C1-C3, L1, L2, C4-C8, cf. Table 4.1), high resolution reciprocal space maps for each of the four grains were collected. Each acquisition takes about 15 minutes, depending on the number of necessary $\Delta\omega$ intervals to acquire the entire orientation spread developed in the grain. Each small rocking interval requires in total 17 s including time for motor movements and an exposure time of 5 s. Usually, between 40 and 60 different rocking intervals were acquired for each reciprocal space map. The time for centering of each grain and acquisition of HRRSM for all four grains after each load step was about two hours.

During acquisition of each high resolution reciprocal space map, the applied load and the resulting average strains were recorded by the load cell and the strain gauge, respectively and saved together with the image data. The average macroscopic stress and strain during each acquisition are displayed
in Figure 4.20. During pausing of the motors for acquisition of the HRRSM after each loading step, the position of the cross heads is fixed, while stresses and strains do not remain constant. Therefore the macroscopic stresses and strains differ between the acquisitions for the four grains. This is in particular prominent for the acquisition after tensile deformation to a total strain of 1.3% (L2), where the stress drops from 39.6 MPa to 38.6 MPa between the two repeated measurements of grain 1. Simultaneously, the macroscopic strain measured by the strain gauges decreases, indicating a relaxation in the loaded parts of the load frame. After performing different numbers of tension-tension cycles a similar behaviour is observed: During the acquisition period for the four grains, a decrease of both the macroscopic axial stress and strain was detected after each load step. These differences of stresses and strains between the first and the last grain measured at each load step decrease with the increased number of cycles. Additionally, an overall decrease in macroscopic stress and strain is observed from after the first tensile loading (L2) to the last cycling (C8) (from 39.6 MPa to 37.2 MPa and from a strain of about $2.94 \cdot 10^{-3}$ to $2.90 \cdot 10^{-3}$). Notably, the behaviour of the material after the first cycling (C4) following immediately after the loading step seems to be different from that of the following cycling steps (C5-C8). While the stress decreases continuously, the strain increases during the first cycling (C4) in comparison to the strains after loading (L2), e.g. for the time of acquisition of grain 2 from $2.93 \cdot 10^{-3}$ to $2.94 \cdot 10^{-3}$. 
Figure 4.20: Average macroscopic axial stress and strain (a) for all HRRSM acquisitions for all grains 1 to 4. The red circle indicates the states for the second cycling, which will be discussed in detail. (b) for the second cycling after loading L2. An additional acquisition step has been performed for grain 1, immediately after loading L2 and again after acquiring data sets for grains 2 to 4. The entire acquisition sequence is indicated by arrows.

During the tension-tension cycling, load cell and strain gauge were monitored additionally with a frequency of 1 Hz. The resulting macroscopic stress strain curves for the last load cycle of each of the load steps C4-C8 are presented in Figure 4.21. They clearly reveal a mechanical hysteresis and only slight changes with increasing number of cycles as seen by the decrease of the average macroscopic strain.
Figure 4.21: Macroscopic hysteresis curves obtained during in-situ cyclic loading. Full circles connected by solid lines indicate the stresses and strains recorded during the last cycle of each of the performed cycling loadings (C4 to C8). Due to the read-out frequency of 1 Hz, absolute maxima and minima within the cycles are not always captured; therefore, these are traced from the entire data set and marked with crosses in the corresponding color.

4.2.2 HRRSM

4.2.2.1 Azimuthal maps

For four different grains in total up to 11 acquisitions were done, where Figure 4.22 shows exemplary azimuthal projections of the 400 diffraction peak for load step C4. The selected grains differ significantly in the appearance of their orientation distributions visualized by their azimuthal projections.
Figure 4.22: Azimuthal projections for grain (a) 1 (b) 2 (c) 3 (d) 4 after load step C4. It’s important to note that the size of the maps is different for all four grains e.g. grain 3 has a very small map (ROI$_y$ = 81) in comparison to grain 4 (ROI$_y$ = 151).

To visualize the effect of tensile loading on the appearance of the Bragg reflection in the HRRSM, Figure 4.23 shows the azimuthal maps before (C3) and after loading to 1.1% (L1) and again after loading to 1.3% strain (L2). A clear increase in size and broadening is visible for the second loading. In addition there are areas with locally high intensity are visible for both reflections. Grain 4 seems to consist of two parts possibly corresponding to two domains within the grain, which are spreading out and moving from each other with tensile loading. For Grain 1, only one central high-intensity area is split up in several smaller areas of high intensity. However a certain degree of substructure is already visible after C3.
Figure 4.23: Azimuthal maps of the selected Bragg reflection for grain 1 (top) and grain 4 (bottom). A clear broadening of the reflection is visible from C3 to L1 and to L2. The maps are scaled linearly between their individual minimum and maximum intensity to visualize the substructure because of the compact intensity distribution for C3.

All azimuthal maps of grain 1 and 4 after loading to 1.3% tensile strain and after each cycling step are shown in Figure 4.23. A clear increase in the width of the diffraction peak becomes visible after loading from 1.1% to 1.3% total tensile strain, i.e. from L1 to L2, as shown above, but the appearance of the azimuthal map does not change significantly during the subsequent cycling as shown in Figure 4.24 (C4-C8). However, small changes in the intensity can be recognized indicating possibly changes in the substructure, while the grain shape in orientation space remains rather constant. Unfortunately, the intensity of the incoming beam dropped significantly after loading step C8 hence causing low intensity for the maps designated with C8. While analysis of the mechanical data and integrated profiles is reliable, only limited information on the subgrains is available from this acquisition.
Figure 4.24: Azimuthal maps (a) of grain 1 for the load steps L2 to C8. (b) of grain 4. The projections do not differ significantly from L2 to C7 though local changes of the peak intensities (exemplary marked in C4 and C5 with a white circle) can be observed; after C8 the beam intensity dropped significantly causing a lower intensity and a different signal to noise ratio. For comparison purposes, measurements were normalized with the intensity of the incoming beam and scaled in the same manner (L2-C7).

Figure 4.25: Microstructural development during cycling from L2 to C7: (a) the total integrated (not normalized) intensity of grain 1 in comparison with the extracted intensity of the subgrain component and (b) the subgrain volume fraction for all four grains.

Figure 4.25 shows the development of the overall measured intensity of grain 1 during the second cycling in comparison to the intensity of the separated subgrain component after partitioning. The total integrated intensity shows only small variations due to fluctuations in the incoming beam intensity. The intensity of the subgrain component (integrated intensity of the peak component) is about 40% of the
The volume fraction of subgrains is calculated by dividing the integrated intensity of the peak component by the total integrated intensity. As obvious from Figure 4.25b, all four grains show a rather constant subgrain volume fraction for all acquisitions taken along the second cycling. The volume fraction of grain 3 is with 70% significantly higher than the volume fraction of 40% measured for all other grains, which is related to the more narrow appearance of the grain in the azimuthal map with a lower cloud component allowing an easier separation of the subgrain component and does not indicate a major difference in the deformed structure. Acquisition after load step C8 is not included here due to the significant drop in the beam intensity.

Figure 4.26 shows the evolution of the azimuthal width. As expected from Figure 4.23 is the azimuthal width clearly increasing during tensile loading to L1 and L2, while it is more constant during the cycling sequence C1-C3 and C4-C8.

4.2.2.2 Radial profiles

Figure 4.27 presents all radial profiles obtained for grain 1. Only selected profiles will be shown in the further analysis, because the plots get easily confusing when containing too many profiles. Nevertheless, this gives a good overview, how the radial profiles of the grain behave during the presented measurement (cf. Figure 4.20). The acquisition sequence starts with the black profile
obtained after mounting and tensile loading. Only minor changes are visible in the profile appearance during the first cycling C1-C3 with low amplitude (actually only the blue profile is visible, because the profiles are so identical that they overlap). Loading to 1.1% strain causes a shift of the profile to the following black profile at lower \( q \)-values. The second tensile loading shifts the profile further to lower \( q \)-values, where the profiles of the second cycling L2-C8 overlap. The shifts are all according to the expectations from the applied load. Finally a measurement was done after unloading of the sample to nearly zero load, which causes a significant shift of the profile to higher \( q \)-values (higher than the profile for L0 that was already measured under tensile loading). The second cycling C4-C8 will be discussed in detail in the following.

Figure 4.27: All radial profiles obtained with HRRSM for grain 1, normalized with the maximum intensity. The different load regimes are marked for overview reasons.

Figure 4.28a shows the radial profiles for the second cycling C4-C8 after loading to L2. As discussed in section 3.4.2.2 a larger lattice spacing results in a smaller \( q_{\text{mean}} \). While the profiles shift to lower \( q_{\text{mean}} \) during the tensile loading of the sample (cf. Figure 4.28b), the profiles shift to slightly higher \( q_{\text{mean}} \) during the cycling sequence (from black over green to red in Figure 4.28b) in full accordance with the applied stresses (which is decreasing along the cycling). The shift in peak position during cycling (\( q_{\text{mean}} = 1.9 \times 10^{-4} \) Å\(^{-1}\)) is quite small in comparison to the shift during tensile loading (\( q_{\text{mean}} = 1.2 \times 10^{-3} \) Å\(^{-1}\)). The shift of the peak position during the second cycling is exemplarily shown in Figure 4.28c for grain 1; \( \Delta q \) shifts to
smaller values after the first cycling after loading and increases during cycling following closely the macroscopic stress shown in Figure 4.20. Note that there is a difference in the mean peak positions between the four grains seen in Figure 4.28c, for all load steps despite their similar orientation. This is probably caused by the different neighbouring grains in the polycrystalline specimen or due to stresses e.g. from sample bending.

![Graphs showing radial profiles and peak positions](image)

**Figure 4.28:** (a) Radial profiles for the second cycling sequence after loading to 1.3% total tensile strain (black profile) for grain 1 (normalized to a maximum intensity of 1). The radial profiles shift during cycling to slightly higher values of the diffraction vector (profiles from green to red). (b) Peak position in dependence of the macroscopic strain for all four grains and all load steps. (c) Peak position for grain 1 for the second cycling L2-C8 after loading to 1.3% total tensile strain.
Figure 4.29 presents the integral width $\beta_g$ of the radial profiles for each of the four grains in dependence of the macroscopic strain. A clear increase in the integral width is observed after each tensile loading step (L1, L2), while the detailed display of the second cycling in Figure 4.29b shows a slight decrease in width after the first cycling C4 after tensile loading L2. During further cycling (C4-C8), the integral width does not change significantly any longer, but stays almost constant. Notably, the integral width differs significantly between the four grains.

![Figure 4.29: Integral width $\beta_g$ of the radial profiles for grain 1 to 4 as function of the macroscopic strain: (a) for all load steps, and (b) for the second cycling L2-C8 after loading to 1.3% total tensile strain.]

As discussed the asymmetry in the radial profiles indicates the presence of an ordered structure as rationalized by the composite model. The asymmetry values obtained by fitting split pseudo-Voigt functions to the experimental data are shown in Figure 4.30 for all four investigated grains. For grains 2 to 4, the absolute asymmetry $\kappa$ increases significantly during the first tensile loading from $2\cdot10^{-4}$ Å$^{-1}$ to $4.8\cdot10^{-4}$ Å$^{-1}$ and for grains 3 and 4 also during the second tensile loading to a maximum of $6\cdot10^{-4}$ Å$^{-1}$, while it remains almost constant for grains 1 and 2 during the cycling after the second tensile loading. Grain 1 starts with a much higher asymmetry of $5.1\cdot10^{-4}$ Å$^{-1}$ after pre-deformation and mounting and shows a decrease during the first tensile loading. However, all grains develop a similar absolute asymmetry after L2. Small variations are measured during the cycling itself as shown in detail for load steps L2-C8 in Figure 4.30b.
Figure 4.30: Absolute asymmetry $\kappa$ of the radial profiles for grain 1 to 4 as function of the macroscopic strain: (a) for all load steps, and (b) for the second cycling L2-C8 after loading to 1.3% total tensile strain.

The absolute asymmetry is positive for all measurements indicating that the subgrains have larger diffraction vectors and smaller lattice spacings than the dislocations walls. This means that compared to the average of each grain, the dislocation walls experience tensile strains and the subgrains experience compressive strains — in full accordance with the predictions of the composite model for observations along the tensile axis, (i.e. in an axial case under tension). In the following, the subgrain component will be analysed in more detail.

### 4.2.2.3 Subgrains

For further analysis, each azimuthal map is partitioned into a smooth cloud component and the peak component containing all high-intensity peaks as described in section 3.4.2.1. The 100 most intense high-intensity peaks are identified for each reciprocal space map; each of these peaks is presumed to originate from a single subgrain. In the less deformed specimen (until L1), less than these 100 subgrains were found for all grains. After a tensile loading to 1.3% total tensile strain (L2 and the subsequent cyclic loadings C4-C8) at least 100 subgrains were identified, where the 80 most intense and thus largest subgrains will be analysed in the following.
Individual subgrains can be identified for each grain by their corresponding high-intensity peaks and traced from load step to load step, as for example, the four high-intensity peaks marked for grain 1 in Figure 4.31.

**Figure 4.31:** Azimuthal map of the peak component for load steps L2-C8 of the second cycling for grain 1. Four peaks from four individual subgrains 1-4 found in all load steps (L2-C8) are marked as examples.

Both the $\omega$ and the $\eta$ positions of the subgrains can be visualized in a normal probability plot to gain statistical information about the location of the subgrains in orientation space. With this indications about the general intragranular structure can be gained, which can for example consist of domains in that the subgrains are mainly identified. The distributions of the subgrain locations in $\omega$ and $\eta$ for the different grains are shown in **Figure 4.32**. All grains show in general an even distribution for the subgrains both in $\eta$ and in $\omega$. Rather uniform distributions are visible for grain 2 and 3 confirming their narrow appearance in orientation space. For grain 3 the distribution appears almost as a Gaussian distribution, with some deviations for higher $\eta$ and $\omega$, which can possible indicate the presence of a domain at higher angles. A split up in two domains is very clear for grain 4, where one domain includes subgrains at lower $\eta$ and the other domain subgrains at higher $\eta$ angles. Grain 1 seems to have a small domain at lower $\eta$ angles, which is the smeared-out intensity as visible to the left in the azimuthal map.
Figure 4.32: Normal probability plot for $\eta$ and $\omega$ position of the 80 subgrains with the highest intensity identified by the software for (a) grain 1 (b) grain 2 (c) grain 3 (d) grain 4. An azimuthal map for L2 with the ten peaks of highest intensity marked is included for visualization.

The radial peak positions of the 80 largest subgrains of grain 1 are shown exemplarily in Figure 4.33 for L2 and C7 (after 5150 cycles). The mean positions of the individual subgrain profiles are not spread around the mean position of the grain profile and only a few subgrains have diffraction vectors lower than that of the grain average. In general, the radial positions of the subgrains are found at larger diffraction vectors than the average of the grain indicating elastic back strains. Instead of being distributed around the average peak position $q_{\text{mean}}$ of the entire grain, they are gathering around the position of the peak maximum $q_{\text{max}}$ of the profile of the entire grain as indicated by the dashed lines representing the peak shift between the position of the maximum intensity and the average position in Figure 4.33. In the normal probability plot shown in Figure 4.33, the data points of each load step follow almost a straight line revealing that the peak shifts $\Delta q$ of the individual subgrains follow a Gauss distribution (similar to [WEJ13] for a tensile deformed sample). Comparing the two load steps L2 and C7, a shift to slightly higher peak shifts is visible after cycling accompanied by an increase in the slope reflecting a decrease in the spread of the subgrain profile positions. The mean value of the distribution increases from $3.6\cdot10^{-4}$ Å$^{-1}$ for L2 to $4.1\cdot10^{-4}$ Å$^{-1}$ for C7, while the standard deviation of the distribution decreases slightly from $3.6\cdot10^{-4}$ Å$^{-1}$ to $3.2\cdot10^{-4}$ Å$^{-1}$. For the majority of the subgrains, an increase in the peak shift $\Delta q$ is expected from Figure 4.33.
Figure 4.33: Normal probability plot of the peak shift $\Delta q$ between the position of subgrain profiles and the mean position of the entire grain for the 80 most intense high-intensity peaks of grain 1 for L2 and C7. Best linear fits to the data are shown as lines indicating that the peak positions follow a Gaussian distribution around the position $q_{\text{mean}}$ of the peak maximum with a narrower spread after cycling. The peak shift $\Delta q_{\text{max(grain)}} = q_{\text{max(grain)}} - q_{\text{mean(grain)}}$ of the maximum intensity of the corresponding radial grain profile is marked as a dashed line.

Four high-intensity peaks (as marked Figure 4.31) were identified in all azimuthal projections of grain 1 and followed individually for all load steps from L2 to C8. These four high-intensity peaks were chosen among the ten highest intensities and are, hence, four of the largest subgrains identified in grain 1. In Figure 4.34, the radial profiles of the four selected subgrains are shown together with the corresponding radial profiles of grain 1 (thin lines) for load steps L2-C8. According to Figure 4.28a the grain profile shifts to higher diffraction vectors during cycling. For all four subgrains, the profiles shift to higher $q$-values with cycling as well and follow the grain behaviour (from black to red).

The integrated intensity calculated from the radial subgrain profiles (of the normalized data) for each load step is shown for the four selected subgrains in Figure 4.35a. The intensity varies for most of the acquisitions between $2.5 \times 10^4$ counts and $5.5 \times 10^4$ counts and is in average the largest for subgrain 1 and the smallest for subgrain 4 indicating that subgrain 4 is smaller (i.e. has a smaller volume) than
The subgrain 1. From their detected intensity, all four subgrains are expected to be of comparable size and their size remains nearly unchanged during all load steps (confirming that it is indeed the same subgrain). The almost constant values underline that it was possible for these four subgrains to trace their individual position and intensity after each load step.

Figure 4.35b shows the development of the peak shift Δq between the mean position of the subgrain and the entire grain profile. After tensile loading (L2), subgrains 1 and 2 differ significantly in their peak shift (Δq = 3.4·10⁻⁴ Å⁻¹ and Δq = 6.9·10⁻⁴ Å⁻¹) in 3.5·10⁻⁴ Å⁻¹, while the profiles of subgrain 3 and 4 have comparable peak shifts (Δq = 4.73·10⁻⁴ Å⁻¹ and Δq = 4.76·10⁻⁴ Å⁻¹) in between these values. The subgrain profiles for subgrain 1, 3 and 4 develop larger shifts Δq leading in general to higher peak shifts after 5150 cycles (C7) than after L2 with largest gains for subgrains 1 (2.1·10⁻⁴ Å⁻¹) and 4 (1.8·10⁻⁴ Å⁻¹). Subgrain 2 which had the largest peak shift after L2 nearly kept this value and decreased the peak shift only slightly.

In summary, the peak shifts of all four subgrains converge to more similar values after C7 in comparison to their starting values after L2, which is also indicated by the narrower spread of the peak shift distribution of all 80 subgrains of C7 in comparison to L2 discussed in Figure 4.29. The dashed black line represents the mean peak shift of all 80 subgrains in the analysed grain 1, which is in general smaller than the individually presented peak shifts for the four selected and large subgrains. Figure 14c shows similar trends for the internal elastic strain calculated after equation 3.7. The experienced elastic strain is always negative, i.e. a elastic back strain. Initially (after L2) the values are quite different from subgrain to subgrain (Δε = 6·10⁻⁵), but more similar values (Δε = 2·10⁻⁵) are adapted after C7 indicating a levelling of the elastic strains in the cyclic deformation structure.

The elastic back strains of the four selected subgrains, which were among the largest, are in general higher than the mean elastic strain of all 80 subgrains. This gives an indication that the larger subgrains experience a higher elastic back strain than the majority of (smaller) subgrains in the grain. To provide more evidence for this size effect the variation of the elastic strains experienced by the individual subgrains as determined from the peak shift of their corresponding high-intensity peak is shown in Figure 4.36a for the 80 largest subgrains of all load steps L2 to C7. The 80 peaks with highest intensity
from each load step were grouped in three groups of intense (corresponding to the 10 largest subgrains, green), medium intense (corresponding to the 20 medium sized subgrains having smaller sizes than the 10 largest ones, red) and low intense (the 50 smallest of the 80 considered subgrains, blue) peaks. Only few high-intensity peaks reach intensities up to $9 \cdot 10^5$ counts and can be related to exceptionally big subgrains. The majority of the peaks has intensities between $1 \cdot 10^5$ counts and $3 \cdot 10^5$ counts meaning that the majority of subgrains are of comparable size. Most of the subgrains experience negative elastic strains related to their positive peak shift $\Delta q$ as already seen from Figure 4.33. The average elastic strain experienced by the subgrains of each of these three groups was calculated and marked with a coloured dashed line. These lines reveal the presence of a clear size effect, the largest subgrains (green) experience in average an elastic back strain, which is two times higher than that of the smaller subgrains (blue). For a more detailed assessment of this size effect, the data from the high-intensity peaks for the identified subgrains in grain 1 after L2 and C7 are ordered with respect to their intensity in Figure 4.36b and Figure 4.36c. Figure 4.36b shows the decreasing intensity of the peaks proportional to the volume of subgrains. In addition, the peak shifts $\Delta q$ of the subgrains from L2 and C7 are shown in the same order in Figure 4.36c, revealing a tendency for the less intense peaks originating from smaller subgrains to have lower peak shift $\Delta q$ and hence a lower elastic back strain than the high intense peaks from larger subgrains.
Figure 4.34: Radial profiles of grain 1 and the four selected subgrains 1-4 marked in Fig. 7 for load steps L2 to C8. Both grain and subgrain profiles shift to higher diffraction vectors during cycling. The maximum intensity is normalized to 1 for the grain profile and to 0.5 for each subgrain profile.
Figure 4.35: (a) Integrated intensity of the four selected subgrains 1-4 for the load steps L2 to C7. Subgrain 1 corresponds to the most intense peak and thus the largest subgrain. (b) Peak shift $\Delta q$ for the four selected subgrains and (c) development of the internal elastic strain during acquisition for load steps L2 and C7. The mean peak shift and elastic strain experienced by all 80 subgrains of grain 1 are marked with a dotted black line.
Figure 4.36: Analysis of the 80 largest subgrains in grain 1 for load steps L2 to C7: (a) Peak intensities and corresponding elastic strains for the 80 largest subgrains of each load step divided in three groups according to their order with respect to their peak intensity (1:10 in green, 11:30 in red and 31:80 in blue). The average elastic strain has been marked for each group with a dashed line of same colour, indicating that larger subgrains experience in average a higher elastic back strain than smaller subgrains. (b) Peak intensities of the subgrains in order of decreasing intensity for load steps L2 and C7. (c) Peak shift \( \Delta q \) of the subgrains in order of decreasing intensity for L2 and C7 revealing a decrease in \( \Delta q \) for decreasing peak intensity of the subgrain profiles.
4.3 Tension-Compression Half-Cycle after pre-deformation (PETRA)

During the first beam time at APS the effects of tensile loading and continuous tension-tension cycling were studied. The aim of this experiment at PETRA was to follow changes in the deformation structure of selected grains during a single tension-compression load cycle in a pre-fatigued specimen, which was unloaded from the maximum compression. It was the first approach to map the microstructural changes during parts of a tension-compression load cycle.

4.3.1 Sample Deformation

The specimen was pre-fatigued using MTS Acumen. Cyclic pre-deformation in tension-compression was carried out in order to introduce a microstructure conform to cyclic deformation in the specimen prior to the in-situ investigations by HRRSM. The investigated sample was initially deformed by tension to a strain of 1%. Afterwards, displacement-controlled tension-compression cycling was performed with a rate of 0.5 Hz and a displacement amplitude of 20 µm corresponding to a nominal strain amplitude of \( \varepsilon_a = 1.3 \cdot 10^{-3} \). Cyclic deformation was stopped after 44350 cycles (corresponding to 230 accumulated strain with a stress ratio of -0.93 and a maximum load of 160 N corresponding to 32 MPa) at the end of the compression half cycle and unloaded to zero load. For the synchrotron experiment the sample was mounted in the transportable screw-driven load frame. Both, strain and load force, were monitored during deformation. By utilizing two pre-wired strain gauges at the opposing sides of the strain gauge of the specimen it was detected, if the sample starts to bend during compression.

An incomplete load cycle (cf. Figure 4.37) measured at selected conditions by HRRSM was achieved by following the hysteresis curve from the nearly unloaded state to maximal tensile load and continued into compression to a relevant load. For obtaining reciprocal space maps the deformation was interrupted after achieving selected forces. Since the pre-deformation was stopped at the highest compression stress in the hysteresis curve, it was aimed for characterization of the deformation structure after mounting and after loading the sample with as little as possible load (almost 0 N, L0). The loading was thus interrupted for a first time at 70 N (L1) to investigate the behavior during elastic
loading. For the following mapping (at 100 N, L2), the deformation was stopped after a significant change in strain. The tensile loading was stopped at the maximum force of 140 N (L3) corresponding approximately to the highest tensile load the sample experienced during cyclic pre-deformation. During unloading along the hysteresis curve to 60 N (U1) only small changes in strain were observed, whereas at the a compressive load of -100 N (U2) a significant compressive strain was attained. For stopping the deformation manually when reaching the desired loads, a quite slow deformation speed of 3 µm/s was chosen (this was necessary, because the expected backlash of the load frame has not been determined before). This was very time-costly only allowing 6 acquisitions along the hysteresis during the beam time. Due to these time limitations, no data was recorded at the maximal compressive force of -140 N. The problems with bending and backlash were first observed during this beam time and resulted in a further characterization of the load-frame for tension-compression cycling as described in section 3.2.2.2.

Figure 4.37: Hysteresis curve showing the incomplete load cycle measured in-situ by HRRSM. Reciprocal space maps were obtained at the designated macroscopic stress-strain conditions (L0-3, U1-2) while pausing the deformation for the acquisition. The maximal stress of nearly 30 MPa corresponds to the maximal tensile load experienced during the cyclic pre-deformation of the sample.
4.3.2 HRRSM

4.3.2.1 Azimuthal maps

At each of the load steps where the deformation was interrupted, reciprocal space maps of in total five different grains were collected; all having a (400) direction close to the tensile axis. An example for the appearance of these grains under maximum tensile loading is shown in Figure 4.38. As observed before, the grains differ significantly in their appearance in reciprocal space despite their similar orientation.

![Azimuthal maps acquired at the maximum tension L3 for all grains](image)

(a) (b)

(c) (d)

(e)

Figure 4.38: Azimuthal maps acquired at the maximum tension L3 for all grains (a) Grain A (b) Grain B (c) Grain C (D) Grain D (e) Grain E, normalized with the incoming beam intensity.

To visualize the changes of the azimuthal projection, all projections are exemplarily shown for grain B in Figure 4.39. Only minor changes in the intensity distribution are visible during the measured half-cycle. The overall behavior of all grains is similar and only exemplary data presented.

Figure 4.40a shows the azimuthal projection of grain A for the initial configuration (load step L0). Due to the pre-deformation of the sample the azimuthal maps shows many local intensity differences and
a large azimuthal width. Hence a significant part of the total intensity can be separated as the high-intensity fraction from subgrains. Up to the maximum of 100 high-intensity peaks corresponding to 100 individual subgrains were identified for each reciprocal space map. One subgrain of grain A (which could be identified for all load steps in the azimuthal projections) is highlighted in Figure 4.40b and selected for exemplary analysis.

Figure 4.39: Exemplary azimuthal maps for the half-cycle for grain B. Only minor changes in the intensity distribution are visible during the half cycle, though differences in the azimuthal position between L3 and U2 are visible.
Figure 4.40: Azimuthal projection of the 400 diffraction peak from grain A before any significant tensile loading (L0). The intensity distribution is not smooth and one obvious high-intensity peak corresponding to a single subgrain is marked by a white circle. (b) High intensity peak fraction corresponding to all subgrains for all azimuthal maps acquired during the half-cycle for grain A. The same subgrain is found in all maps and marked by a circle. The scaling in (a) was done in respect to the maximum intensity for the individual map and in (b) in respect to a common maximum intensity for all maps.

4.3.2.2 Radial Profiles

Figure 4.41 shows radial peak profiles for grain A. The obtained radial profiles can be further analyzed regarding their position, width and asymmetry and provide strain information as described in section 3.4.2.2. The peak position changes as shown in Figure 4.41b to smaller values (red) during tensile loading, due to increasing elastic strain and increasing lattice plane spacing, and conversely to higher values (green) during unloading and compression due to decreasing or negative strain and a smaller lattice plane spacing as a consequence of the elastic deformation.
The peak position behaves similar for all measured grains and reproduces in general the hysteresis curve of the in-situ experiment (cf. Figure 4.37). Each grain differs though significantly in their $q_{\text{mean}}$ for the half-cycle from the other grains. Figure 4.42b shows the mean peak positions $q_{\text{mean}}$ for three different grains for all six different loading steps (i) in dependence of the total macroscopic strain (in colors) measured by the strain gauges and (ii) in dependence of the purely elastic strains calculated with the $q_{\text{mean}}$ of L0 according to equation 3.7 calculated from the deviation of the actual peak position from the initial value.

Figure 4.42 shows the change of the diffraction vector during the half-cycle in dependence of the macroscopic strain. The absolute values of the scattering vector $q$, however, are surprisingly different from grain to grain, they differ by 0.07 Å⁻¹ between the three presented grains in Figure 4.42b. It is also striking, that the elastic strain $\varepsilon$ (green) calculated from the peak position with respect to the presumed strain-free initial value $q_{\text{mean,L0}}$ not only differs from the total strain measured by the strain gauges but also varies from grain to grain. This indicates the presence of large internal elastic stresses within the specimen, most likely caused by the bending through clamping when mounting the sample in the tensile test machine or load frame and from pre-deformation and 44350 cycles resulting in a large accumulated strain.

![Graphs showing peak positions and strains](image)

**Figure 4.41**: (a) Radial peak profiles of grain A for the six different loading steps during tensile and subsequent compressive loading of the pre-fatigued sample. b) Mean peak position of grain A as a function of the total strain measured by the strain gauges.
Figure 4.42: (a) Peak position in dependence of the macroscopic strain measured by the strain gauges for all five grains showing that all grains are moving around different \( q_{\text{mean}} \) (with a difference of 0.01 Å⁻¹) during the half-cycle. (b) Mean peak position of three selected grains as a function of the total strain measured by the strain gauges (magenta, red, blue) and the elastic strain (green) calculated from the peak position according to equation 3.7.

The behavior of the integral peak width for grain A is demonstrated in Figure 4.43. Initially, the width is slightly increasing in general during loading to the maximum tensile load and then further increasing during unloading into compression. The peak width of all 5 grains is shown in Figure 4.43b, where it is visible that the peak width and behavior of the different grains is different. It seems like three groups can be distinguished, where grain A and B behave in the same manner with similar integral widths. Grain C and E behave slightly different to the first group but have again similar integral widths, with a peak width that is increasing until L1 (Grain E) or L2 (Grain C) and then decreasing to L3 and increasing again into compression. Grain D has a higher integral width than all other grains, which increases first during loading until L2 and decreases before the maximum tension L3 is reached opposite to all other grains. The width also decreases during unloading and loading into compression.
A similar analysis can be performed for the absolute asymmetry of the grain profiles. Figure 4.44 shows that the absolute asymmetry differs significantly from grain to grain as well as the width and peak. The grains with the highest integral width seem to also show the highest asymmetry possibly indicating a stronger heterogeneous substructure introduced by deformation than some with lower values. Three of the grains (grain A, B and E) show a negative absolute asymmetry and two of the grains (D and C) a positive asymmetry along the whole half-cycle. The asymmetry behaves against expectations in the opposite measures the integral width, i.e. in general increasing when the integral width was observed to decrease and vice versa. As shown in section 2.3.2 is the asymmetry expected to be positive for all grains at the maximum tension (for the presented axial case), which is in this case only true for two of the five grains. The investigated sample was unloaded from the maximum compression during pre-deformation with another equipment than the load frame. It was then loaded to maximum tension again during the HRRSM experiment. A possible explanation for several grains showing a negative asymmetry might be, that the experienced maximum compression before unloading was larger than the maximum tension during loading at the HRRSM set-up.
Figure 4.44: Absolute asymmetry in relation to the macroscopic strain for all five grains. The asymmetry differs for all grains and is for some always positive and for some always negative along the measured half-cycle.

In general all radial profile characteristics seem to differ significantly from grain to grain giving indications for differences in the local stress-situation and intragranular structure e.g. because of the local neighbourhood or sample bending prior to the experiment, which would cause very different stresses within the grain depending on their location and the stiffness of their local environment within the sample.

4.3.2.3 Subgrains

Radial peak profiles of subgrains can be analyzed in the same way as the profiles for the grains demonstrated above. Figure 4.45a shows the radial profiles for the single subgrain of grain A marked in Figure 4.40b. This particular subgrain (among others) could be identified for all six load steps by its specific position in the azimuthal projections for grain A. The positions of the radial profiles of the subgrain behave similar to the radial profiles observed for the entire grain A. As shown in Figure 4.45b, the peak position of the particular subgrain shifts to lower values of \( q \) during tensile loading and higher tensile strains and to higher values of \( q \) during unloading and compression, while neither peak shape, nor peak width changes significantly.
Figure 4.45: (a) Radial peak profiles for the selected subgrain from grain A marked in Figure 3 for all load steps. The profiles shift similar to the profile of the entire grain A. b) Mean peak position of the selected subgrain as a function of the macroscopic total strain for all load steps. The peak position follows the applied load and reproduces the measured hysteresis curve.

Since up to 100 high-intensity peaks are identified in each reciprocal space map, a statistical analysis of their peak positions can be attempted in addition to the analysis of the fate of individual subgrains. Figure 4.46 presents a normal probability plot of the peak positions of the 80 largest subgrains. Essentially each subgrain has a different peak position (and hence experiences a different elastic strain); these peak positions are Gaussian distributed for each load step and shift in accordance with the applied load. The peak shifts are shown in Figure 4.46b. The overlap of the distributions indicate that the peak shift is increasing during loading to higher values and then returning towards the initial values during unloading and loading into compression.
Figure 4.46: (a) Normal probability plot of the peak positions of the 80 most intense high-intensity peaks corresponding to the 80 largest subgrains of grain A for all six load steps. Best linear fits to the data are shown as red lines indicating that the peak positions of the subgrains follow a Gaussian distribution at each load step. (b) Normal probability plot of the peak shift $\Delta q$ for all load steps.
4.4 Tension-compression cycling after pre-deformation (APS2)

Continuous tension-tension cycling and tensile loading were analysed for the first APS beamtime. During the beam time at PETRA microstructural changes during an incomplete load cycle were investigated, but the load frame needed further development and characterization as described in section 3.2.2 before continuous tension-compression cycling could be done. After these improvements the aim of the second APS beam time was to monitor the behaviour of the sample along continuous tension-compression cycling and along a complete load cycle for different strain amplitudes. The presented analysis will focus on two complete load cycles at two different strain amplitudes. The overall results achieved for the cycling sequence were similar to the ones presented for the first APS beam time. It is shown that the 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 behavior of the profile width and asymmetry along a tension-compression load cycle. Changes in the azimuthal maps are visible after an increase in strain amplitude.

4.4.1 Sample Deformation

An undeformed and annealed bone-shaped tensile sample equipped with two strain gauges as described in section 3.1 was used. During further load frame testing after the first beamtimes it was observed, that the sample mounting should be monitored in detail, which was demonstrated in section 3.2.2. The sample was screwed into the holder and predeformed to 1% tension to a load of 210 N and a corresponding macroscopic strain of 42 MPa. It was then cyclically deformed for 1000 cycles into saturation with a strain amplitude $\varepsilon_{a1} = 0.4 \cdot 10^{-3}$. After this grains were selected for further investigations and the sample was cyclically deformed further for in total 30010 cycles. The last ten cycles were monitored in detail to choose and verify the displacement for the desired stress-strain conditions to acquire data along the stress-strain hysteresis (cf. Figure 4.47). Conditions for measurements were selected in a way that both the elastic region as well as the yield point (or at least close to it), the plastic region and the maximum compression and tension were mapped. It is visible in Figure 4.47a that the
hysteresis measured with strain gauge 1 and strain gauge 2 differ from each other, where the strain gauge 1 detects a larger strain amplitude than strain gauge 2 (e.g. $\varepsilon_{a1,1} = 0.5 \cdot 10^{-3}$ and $\varepsilon_{a1,2} = 0.375 \cdot 10^{-3}$). The differences in absolute positions may indicate a buckling.

Figure 4.47: (a) Macroscopic stress and strain measured with two strain gauges (red and cyan) and the calculated average strain (magenta) and (b) Macroscopic stress and displacement (here called tension because of the motor name) for the last ten load cycles before the detailed mapping of load cycle 1. The displacements for the selected stress-strain conditions along the load cycle are marked with a star. The direction of unloading from tension and loading into compression (Ua) and loading into tension (La) is marked with an arrow. (c) Increase of the strain amplitude from $\varepsilon_{a1}$ to $\varepsilon_{a2}$. It reveals however a higher strain amplitude for the average strain than it was observed for the individual load cycle in Figure 4.49.
After this, a first tension-compression hysteresis was mapped in detail. Thereafter strain amplitude was increased over several cycles (cf. Figure 4.47c) from $\varepsilon_{a1}$ to $\varepsilon_{a2} = 0.68 \cdot 10^{-3}$ and the sample was cycled for 10010 cycles with an increased strain amplitude of $\varepsilon_{a2}$ into saturation. Again ten load cycles were mapped in detail to select the displacements for the desired stress-strain conditions (cf. Figure 4.48), before the second load cycle with the increased strain amplitude was mapped in detail. Comparing Figure 4.47c and Figure 4.48 it becomes clear, that the strain amplitude was higher before the 10000 cycles, this caused the average mean strain to be lower after cycling. Conditions for measurements were selected in a way that both the elastic region as well as the yield point (or at least close to it), the plastic region and the maximum compression and tension were mapped. It is visible that the hysteresis differ when measured with strain gauge 1 ($\varepsilon_{a1,1}$ and $\varepsilon_{a1,1}$) and strain gauge 2 ($\varepsilon_{a2,1}$ and $\varepsilon_{a2,1}$), where the strain gauge 1 detects a larger strain amplitude than strain gauge 2 (eg. $\varepsilon_{a1,1} = 0.5 \cdot 10^{-3}$ and $\varepsilon_{a1,2} = 0.375 \cdot 10^{-3}$; $\varepsilon_{a2,1} = 0.88 \cdot 10^{-3}$ and $\varepsilon_{a2,2} = 0.65 \cdot 10^{-3}$). The stress ratio for the hysteresis before increase of the strain amplitude was $R = -0.78$ and for the hysteresis with increased amplitude $R = -0.9$. The stress ratio is less negative than the ideal ratio $R=-1$ for symmetric tension-compression cycling. This means that the sample experiences larger stress in tension than in compression during cycling.
Figure 4.48: (a) Macroscopic stress and strain measured with 2 strain gauges (red and cyan) and the calculated average strain (magenta) and (b) Macroscopic stress and displacement (here called tension because of the motor name) for the last ten load cycles before the detailed mapping of load cycle 2. The displacements for the selected stress-strain conditions along the load cycle are marked with a star. One additional measurement in the elastic region was made along the load cycle with increased strain amplitude, which is marked with a circle around the star. The direction of unloading from tension and loading into compression (Ub) and loading into tension (Lb) is marked with an arrow.

Finally the sample was cycled again for 8500 cycles with the same displacement as for \( \varepsilon_{\text{a2}} \) before HRRSM of the grains were acquired again. The sample was then unloaded to zero stress. To achieve the desired strain amplitudes the load frame had to realize a total displacement difference of 171 \( \mu m \) and 197 \( \mu m \) due to the backlash. A clear increase in the width of the measured hysteresis is visible for load cycle 2 in comparison to load cycle 1.

Figure 4.49 describes the stress-strain conditions (marked with circles), where data were acquired. All grains were always mapped at the maximum tension after cycling. In general the macroscopic stress and strain seems to drop during cycling with the same amplitude, as observed before during the first APS beam time and in section 3.2.2, so the starting strain after the first 10000 and grain hunting is nearly 0.007 despite a pre-deformation to an average strain of 0.01 as measured by one of the strain gauges.
Figure 4.49: Macroscopic stress and strain measured during the acquisition. The conditions for acquisition are marked with a circle. The sample was unloaded and loaded without intermediate steps (to verify the cycling amplitude) before the detailed load cycle causing the line in the middle of the hysteresis.

4.4.2 HRRSM

4.4.2.1 Azimuthal maps

Grains were selected after pre-deformation to 1% tensile strain and 10 000 cycles and four grains were chosen for further investigations with up to 29 acquisitions per grain. Figure 4.50 shows azimuthal maps of the four grains measured at the maximum tension after the initial 10 000 cycles, when the grains were initially selected, after cycling for 40020 cycles with $\varepsilon_{a1}$ and after an increase of the strain amplitude to $\varepsilon_{a2}$ and another 10020 cycles. Already when identifying the grains, they showed a broad appearance due to 1% tensile deformation. A clear increase of the extend in reciprocal space after an increase in strain amplitude is visible for grain 1 and grain 3, but less pronounced for grain 2 and grain 4.
Figure 4.50: Azimuthal maps of the four acquired grains after grain selection (left), before (middle) and after (right) increase of the strain amplitude from $\varepsilon_{a1}$ to $\varepsilon_{a2}$ for (a) Grain 1 (b) Grain 2 (c) Grain 3 (d) Grain 4.

Two grains (grain 1 and grain 2) were mapped in detail during two load cycles with different strain amplitudes with 9 (load cycle 1 marked with a, Figure 4.51) and 11 acquisitions (load cycle 2 marked with b, two additional acquisitions) along the hysteresis. Both load cycles are nearly symmetrical in their stress amplitude around zero load (indicated with a red cross). The stress ratio for the interrupted load cycle obtained with strain amplitude $\varepsilon_{a1}$ is $R = -0.84$ and for the load cycle obtained with strain amplitude $\varepsilon_{a2}$ is $R = -0.97$ and increased in comparison to the stress ratios of the 10 continuous cycles before
detailed measurements of the load cycle. The acquisitions were selected to be at the maximum tension 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 stress (U5a, U6b) and at several interesting conditions in the plastic region along the cycle (U4a, L4a; U5b, L5b). All measurements during unloading from the 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.

The azimuthal maps of grain 1 are in Figure 4.51b and Figure 4.52b for the two load cycles demonstrating exemplarily that no major changes are visible during the full hystereses, but an obvious change due to the increase in strain amplitude and the performed 10000 cycles.

**Figure 4.51**: (a) Tension-compression stress-strain hysteresis for strain amplitude $\varepsilon_{a1}$. Red lines indicate the zero load and the average macroscopic strain of the total strain variation. All acquisitions are designated starting with L1a at the maximum tensile load and then following the arrows with unloading into compression to U5a and again tensile loading acquiring another map at the maximum tension L5a. (b) Azimuthal maps exemplary for grain 1 for selected stress-strain conditions along load cycle 1.
Figure 4.52: (a) Tension-compression stress-strain hysteresis for strain amplitude $\varepsilon_{a2}$. Red lines indicate the zero load and the average macroscopic strain. All acquisitions are designated starting with L1b at the maximum tension and then following the arrows with unloading into compression to U6b and again at maximum tension acquiring another map L6b. (b) Azimuthal maps exemplary for grain 1 for selected stress-strain conditions along load cycle 2.

4.4.2.2 Radial profiles

Radial profiles are analysed for each load cycle. The radial profiles for the two load cycles for grain 1 are presented in Figure 4.53. For overview reasons they are split up into sequences for unloading into compression and for loading into tension.
Figure 4.53: Radial profiles of grain 1 for (a) load cycle 1, unloading into compression (L1a, U1a-U5a), (b) load cycle 1, loading into tension (U5a, L2a-L5a), (c) load cycle 2, unloading into compression (L1b, U1b-U6b), (d) load cycle 2, loading into tension (U6b, L2b-L6b).

It is visible that the radial profiles shift to higher $q$-values during compressive loading and to lower $q$-values during tensile loading.
Figure 4.54: (a) Mean profile position $q_{\text{mean}}$ in dependence of the progressing measurements, where the shift to higher values during unloading and to lower values during loading is visible. An additional unloading to maximum compression (marked with c) from maximum tension (marked with t) was done, where data were collected only for grain 2 (corresponds to the blue peak, before each load cycle) and even less measurements were done for grain 3 and 4 during the load cycles. The strain-free $q_0$ is indicated with a dashed line. An increase of the strain amplitude and cycling was done between the two load cycles, which is marked with an orange line. (b) Change of the diffraction vector in dependence on the macroscopic strain for the load cycle with $\varepsilon_{a1}$. (c) Change of the diffraction vector in dependence on the macroscopic strain for the load cycle with $\varepsilon_{a2}$.

Figure 4.54 shows the mean profile position in detail for all acquisitions for all four grains (grain 1 in black) for the two load cycles. The increase of the strain amplitude and 10010 cycles were done between the investigated load cycles. Additional measurements e.g. unloading and loading before the
load cycle were performed for grain 2 (blue) and less measurements during the load cycles for grain 3 and grain 4 (red and green). This plot should visualize the increase and decrease of the diffraction vector at the mean profile position during the load cycles. The mean profile positions are again clearly different for all grains. The movement of the profile during the cycle is 0.005 Å⁻¹ for grain 1 both during load cycle 1 and load cycle 2. The strain-free diffraction vector \( q_o = 6.2064 \text{ Å}^{-1} \) (calculated using eq. 3.3 with the strain-free lattice spacing \( d(Al)_{400} = 4.0495 \text{ Å} \)) was marked with a dashed line, where grain 1 and 3 are above the strain-free \( q_o \) and grain 2 and 4 mainly below.

In Figure 4.54b and Figure 4.54c it is clearly visible that the profile positions reproduce the stress-strain hysteresis for all grains in both load cycles, with a different \( q_{\text{mean}} \) from grain to grain. It becomes obvious, that grain 1 and grain 3 and grain 2 and grain 4 are lying at more similar values. Figure 4.55 shows the mean profile position in dependence on the macroscopic stress for the four grains and all acquisition conditions during the two load cycles, which are resulting in a straight line. Young’s modulus can be calculated using the local elastic strain that can be calculated from the peak position for the grains (using eq. 3.5 and \( q_{\text{mean}} \)). The calculated values are shown in the figure, assuming an uniaxial stress state with the applied stress. Grain 1 and grain 3 have similar \( q_{\text{mean}} \) values and a rather similar Young’s moduli. The same applies to grain 2 and grain 4. Grain 2 and grain 4 have though an significant higher apparent Young’s modulus above the theoretical value for aluminium along [001] than grain 1 and grain 3. To analyse if the effective Young’s modules varies because of an orientation effect, the different deviations from the tensile axis due to \( \theta \) and \( \eta \) were investigated in detail for the four presented grains. All four grains vary in their \( \eta \) positions, it was however found that grain 1 and grain 3 are more similar in their \( \eta \)-range (\( \eta_1 = -3.3346^\circ \) to \(-2.7114^\circ \), \( \eta_3 = -1.8667^\circ \) to \(-1.4587^\circ \)) and grain 2 and grain 4 are similar in their \( \eta \)-range (\( \eta_2 = -0.1368^\circ \) to \(0.4345^\circ \), \( \eta_4 = 1.2023^\circ \) to \(1.7515^\circ \)). This can be a possible explanation for the differences observed for the Young’s modulus. The measurement of a lower microscopic strain due to a larger deviation in \( \eta \) can result in a higher effective Young’s modulus being a possible explanation for the different values. But the different peak positions also indicate different environments. This could indicate that grain 1 and 3 have a similiar neighbourhood (of similiar stiffness) and grain 2 and 4. The spatial positions of the grains are possibly an explanation, since grain 1 (\( x_1 = 0, z_1 = -0.55 \)) and grain 3 (\( x_3 = 0.75, z_3 = -0.55 \)) have a similar \( z \) coordinate. Grain 2 (\( x_2 = 0.75, z_2 \)
\(x_4 = -0.155, z_4 = 0.05\) are more different from grain 1 and grain 3 and also from each other. The location within the sample is thus not necessarily a full explanation for the observed effect.

Figure 4.55: Mean profile position \(q_{\text{mean}}\) in dependence on the macroscopic stress for the four grains. The individual Young’s moduli are presented next to the data of the corresponding grain and different for all four grains. \(q_0\) represents the stress-free diffraction vector.

The radial profiles can be further analysed with regard to their profile width and asymmetry as shown in Figure 4.56 and Figure 4.57. The change of the profile width and asymmetry is shown along both load cycles. During the first load cycle the peak width is first decreasing until U4a and then increasing towards the maximum compression and further during tensile loading. The second load cycle reveals more details due to the two additional measurements. Here it is visible that the peak width is in general decreasing towards U5b and again strongly increased towards the maximum compression U6b. After U6b it decreases towards L5b and increases for L6b. The curve in particular for the second load cycle presents a pattern, which is similar to the so-called butterfly pattern observed and discussed in detail for the third APS beam time. Figure 4.56b shows the change of the peak width for all four grains. The preliminary butterfly pattern is most clear for grain 1, but only vaguely recognizable for grain 2 for load cycle 1 as well as load cycle 2. Grain 2 however seems to behave generally very different from the other three grains. In general, the width is different for all grains, where grain 4 shows the highest value for the integral peak width \(\beta\), followed by grain 1 and grain 3. Grain 2 has the overall lowest peak width.
is also striking that the peak width differ between before and after the load cycle. The peak width both after the first and the second load cycle is increased in comparison to the starting value for grain 1 and grain 3, but nearly the same or slightly decreased for grain 4 and grain 2, which could be an effect of the interruption for measurement.

![Figure 4.56: Integral peak width $\beta$ in dependence on the macroscopic strain visualising the change of the peak width along the two load cycles for (a) grain 1 and (b) all four grains. The peak width of the maximum tension before and after both load cycles is marked with an ellipse.](image)

The absolute asymmetry seems to follow a distinct pattern as well. The asymmetry is during the first cycle increasing until U4a and then decreasing towards U5a. It is in general further decreasing until L4a and then increased at the maximum tensile load. For the second load cycle the asymmetry is increasing until U4b and decreasing until U6b. It behaves similar (but opposite) during tensile loading with decreasing until L4b and increasing until the tensile point L6b is reached. The asymmetry is changing sign from positive to negative during loading into compression as expected, with a similar absolute value (for load cycle 2 is $\kappa$ equal to circa $\pm 1.6 \, \text{Å}^{-1}$). In Figure 4.57b the change of the asymmetry is shown for all four grains, where the asymmetry values are similar to the width different for all grains. Especially grain 2 appears to behave differently again with showing only negative asymmetry values for all acquisitions. Grain 3 and grain 4 are changing the sign of the asymmetry as expected of grain 1. The asymmetry of the maximum tensile load after the second load cycle is for all grains higher than at the maximum tension at the beginning of the load cycle. It is lower or similar for the first load cycle.
4.4.2.3 Subgrains

Up to 92 subgrains were identified in the separated high intensity part of the HRRSM for grain 1. No clear relation between the number of identified grains and the stress-strain condition was found, though a tendency for fewer identified peaks around the maximum compressive strain was observed for load cycle 2. For the 92 peaks identified for load step L1b, only the 80 most intensity peaks are selected for further analysis. The following Figure 4.58 shows only the ten subgrains with the highest intensity for overview reasons, which are marked with circles in the azimuthal map in Figure 4.58a. The highest intensity peak is highlighted in red in all figures. The intensity of the ten high intensity peaks are given in Figure 4.58b, where it is visible that only the first three peaks have a significantly high intensity, which is also highlighted by the linearly scaled azimuthal map (where only three peaks of higher intensity are visible). The radial profiles of these ten subgrains are shown in Figure 4.58c, where most of them seem to overlap and to be at quite similar positions. Figure 4.58d shows the distribution of the mean profile position for the ten subgrains, where it is visible that most of them have similar profile positions, which are at larger lower q (the mean profile position of the total subgrain fraction $q_{\text{mean}}(\text{subgrain total}) = 6.20639 \, \text{Å}^{-1}$, marked in blue) than the mean profile position of the grain ($q_{\text{mean}}(\text{grain}) = 6.20641 \, \text{Å}^{-1}$, marked in green). The subgrain with the highest intensity however is

Figure 4.57: Absolute asymmetry $\kappa$ in dependence on the macroscopic strain for (a) grain 1 and (b) all four grains.
showing the largest deviation, but it is also expected to be the largest subgrain and hence experiencing the largest strain resulting in the largest peak shift in comparison to the other subgrains.

Figure 4.58: (a) Azimuthal map of the separated high intensity peak fraction for grain 1 for load step L1b, scaled linearly. Ten peaks of the highest intensity are corresponding to ten large subgrains are marked with white circles. The subgrain which will be analysed further is highlighted with a red circle. (b) Integrated intensity for the ten subgrains of highest intensity marked in the azimuthal map. (c) Radial profile of the grain and of the ten subgrains (scaled differently with the maximum intensity of the subgrain set to 0.5 for visualisation) corresponding to the high intensity peaks in (a) for load step L1b. (d) Normal probability plot for the peak positions of the subgrains with the highest intensity. $q_{\text{mean}}$ of the grain is marked with a green dotted line. $q_{\text{mean}}$ of the total subgrain component is marked with a blue dotted line. The single subgrain with the highest intensity is marked in red.
One of the subgrains with highest intensity (marked in red in Figure 4.58) was identified for all load steps during the second load cycle and followed through the cycles. Eventhough it is the most intense subgrain for load step L1b, it is not identified as the most intense subgrain for all load step along the cycle.

The radial profiles of the subgrain were plot together with the radial profiles of the grain (presented in Figure 4.53) in Figure 4.59. The subgrain profiles shift similar to the grain to higher q-values during loading into compression and to lower q-values during loading into tension. The position is displayed in detail in Figure 4.59c, where the shift of the subgrain profile clearly reproduces a hysteresis as observed for the grain as well. The profile position of the subgrain is at slightly higher values during unloading from tension and loading into compression (meaning the subgrain experiences slightly less tensile stress) and at slightly lower values during compression and loading into tension meaning the subgrain experiences slightly less compressive stresses than the grain.
Figure 4.59: Radial profiles of grain 1 and one subgrain identified for all load steps. The profiles of the subgrain were scaled differently for visualization. (a) Unloading and loading into compression, the profiles shift to higher $q$-values. (b) Loading into tension, the profiles shift to lower $q$-values. (c) Grain (black) and subgrain (red) profile positions $q_{\text{mean}}$ in dependence on the macroscopic strain reproducing the hysteresis for both. The subgrain shows slightly higher $q_{\text{mean}}$ at tension and unloading from there and slightly lower $q_{\text{mean}}$ at compression and loading into tension.

Figure 4.60 gives an example of the grain and subgrain profile at the maximum tensile and maximum compressive strain (L1b in black and U6b in red), where the mean profile positions are marked with straight lines. It is apparent, that the grain profile at the maximum compression U6b looks in general mirrored to the grain profile measured under maximum tensile strain L1b. Small differences can be identified for the mean profile position between the grain and subgrain. The mean position of the grain
is at higher $q$-values than the one for the subgrain at maximum tension and at lower $q$-values at the maximum compression, meaning the subgrain experiences slightly less compressive strain in compression and slightly less tensile strain in tension than the grain itself (i.e. always back strains).

![Graph showing radial profiles at maximum tension (L1b) and maximum compression (U6b).](image)

**Figure 4.60:** Radial profiles at maximum tension (L1b) and maximum compression (U6b) in load cycle 2 of grain 1 and one subgrain identified at both load steps. The profile of the subgrain was scaled differently to a maximum of 0.5 for visualization. The mean profile position (of the fitted profile) is marked with a dashed line for the grain profile and with a full line for the subgrain profile.

### 4.4.2.4 Increase in strain amplitude

The strain amplitude was increased in between the detailed acquisition of the two load cycles from $\varepsilon_{a1}$ to $\varepsilon_{a2}$. Before the second load cycle was measured, 10010 cycles with an increased strain amplitude $\varepsilon_{a2}$ were done. Figure 4.61 visualizes the changes in the Bragg reflection along the azimuthal direction and with this the appearance of the grain in orientation space before and after the increase of the strain amplitude. The rocking curve of all four grains changes slightly between before (blue) and after (red) an increase of the strain amplitude. Besides a movement in $\omega$ the rocking curves also seem to become broader in width, most clearly for grain 1 and 3. This has already been indicated by the azimuthal maps in Figure 4.50. The curve of grain 4 seems to become slightly broader as well. Changes in $\omega$ are due to
a rotation of the grain when the sample and the grains are adjusting to the higher strain amplitude and different levels of macroscopic strain.

Figure 4.61: Rocking curve for grain 1-4 for before (measurement 1, L5a) and after (measurement 2, L1b) increase of the strain amplitude from $\varepsilon_{a1}$ to $\varepsilon_{a2}$ and 10010 cycles at high strain amplitude.

Figure 4.62 gives an overview how the radial profile position, width and asymmetry has been influenced by the change of the strain amplitude. The presented measurements were done at the maximum tension after the first load cycle (L5a, measurement 1) and the maximum tension at the beginning of the second load cycle (L1b, measurement 2), which is after 10010 cycles with $\varepsilon_{a2}$. The profile position is nearly the same, due to a similiar macroscopic stress at L5a and L1b. The radial profile width is in general increasing for all grains, though less pronounced for grain 1 as also visible in Figure 4.62b. The absolute asymmetry is increasing for all grains. After increasing the strain amplitude, three grains (grain 1, 3 and 4) have a positive asymmetry as expected. Grain 2 shows a negative asymmetry in contrast to all other grains. This shows that both, the profile width and asymmetry are increasing with an increase
in macroscopic strain amplitude as it is expected by the composite model due to an increase in local internal stresses.

Figure 4.62: Difference in (a) mean profile position, (b) integral width, (c) absolute asymmetry after the increase of strain amplitude from (1) cycling after 40011 cycles with $\varepsilon_{a1}$ and (2) cycling after additional 10020 cycles with $\varepsilon_{a2}$. The measurements were done at the maximum tensile load.
4.5 Tension-Compression cycling without pre-deformation (APS3)

The final HRRSM beam time described within this thesis is the third beam time at APS. It follows up on the findings and achievements especially of the second APS beam time. The focus of this beam time is on several subsequential tension-compression load cycles measured in detail at even more conditions than done at the second APS beam time. During the second beam time at APS indications were found, that the profile width and profile asymmetry changes in a characteristic way along a load cycle, which was shown for two load cycles with different strain amplitudes. The third beam time should confirm those findings and to reveal more details in the change of the profile width and asymmetry during a cyclic stress-strain hysteresis. Aim of the experiment was to gain information about the changes during load cycles following each other directly and to determine if the characteristic changes observed during one load cycle are representative. For this an undeformed sample was used and no significant tensile loading was done before cycling to ensure that observed deformation structure is a result of cyclic deformation. The grains were additionally mapped during low numbers of cycles, to follow the initial development of the microstructure for cyclic deformation.

Changes of the appearance of the grain in the azimuthal map were clearly visible after 5000 cycles in comparison to 500 cycles. A characteristic behavior of the profile width and asymmetry was observed during three subsequent load cycles. Four subgrains were identified within one grain for those three subsequent load cycles and the evolution of their local elastic strain was investigated. It is shown that the behavior of the local elastic strain during the three subsequent load cycles differs significantly from each other, most likely influenced by their local neighbourhood.

4.5.1 Sample Deformation

For this experiment an undeformed bone-shaped tensile sample was mounted in the custom-made load frame. The macroscopic strain experienced by the sample was monitored using two attached strain gauges and the macroscopic load was continuously measured by a load cell. The reported macroscopic strain is the average calculated by the two strains measured on each side of the sample surface. The mechanical hysteresis in Figure 4.63 shows that the sample experienced a compression force of -4 MPa
(corresponding to -20 N) during mounting (M) before it was loaded to the maximum of the anticipated hysteresis (L0, near 160 N equivalent to 32 MPa) in tension (black curve). It is desired to have as little pre-deformation as possible to minimize the influence of a microstructure developed during tensile loading, however, it is necessary to load the sample in tension to ensure a stable condition for cyclic deformation. After stabilizing the sample with as little tensile loading as reasonable (approximately 30 MPa), the sample was cyclically deformed for 5010 cycles with a fixed strain amplitude of 6.3·10^{-4} and a strain rate of 6.7·10^{-3}s^{-1} into saturation (R = -0.95). HRRSMs were acquired several times during these 5010 cycles (after 50, 200 and 500 cycles), to obtain information about the early cycling stage, and again after in total 10000 cycles. All grains were selected after mounting and measured with HRRSM before tensile loading was done for stabilization.

Five tension-compression load cycles with the same strain amplitude were investigated in detail after the initial 5010 cycles. The first three load cycles were mapped by HRRSM at in total 15 selected points L0-L14 for two (grain G4 and grain G5) of the four selected grains and are presented in Figure 4.63 as well. Fewer points were mapped for the two following load cycles (only 5 for grain G4 and G5) and for the other grains (grain G2 and G3 were only mapped at the maximum tensile and maximum compressive load during all five load cycles) because of time limitations. The naming is for this results chosen after the real grain names (given during the experiment), therefore no grain 1 was investigated. No major differences in the observations were made for load cycle 4 and 5 and G2 and G3, hence they are excluded from the following presentation of the results.
Figure 4.63: Loading of the tensile sample to about 30 MPa and three stress-strain hysteresis loops measured after 5010 cycles. The load cycle was paused at selected conditions (marked with a star) and reciprocal space maps were acquired with HRRSM. The macroscopic strain was measured by two strain gauges and the average value is displayed. The stress is measured by the load cell. The zero stress and strain levels are marked with a dashed line, showing that the hysteresis is not completely symmetric around zero strain.

Figure 4.63 highlights the conditions for HRRSM acquisition during three subsequent load cycles, which were mapped in detail. The points were chosen according to the displacements and load identified from a previous load cycle performed for this purpose. Before acquiring a HRRSM the motor moved the grips to the predefined displacements corresponding to these points. To get a good coverage of the course of the hysteresis, the measurement points were selected to cover the maximal (L0 and L14) and the minimal (L7) displacement, the elastic region (L1-L2, L8-L9), the transition from elastic to plastic (L3 and L10) and different stages of the plastic region region (L4-L6, L11-L13). The cyclic deformation was interrupted for HRRSM of the grains at each of these points. Load steps L2 and L9 correspond to an almost unloaded condition from either tension or compression. It is however visible that the hysteresis is not fully symmetric having a slightly larger part of the macroscopic strain within the tensile regime.

Two more full load cycles shown (in cyan and blue) were mapped at exactly the same displacements though with small differences in stress as visible in Figure 4.63. As mentioned above, two more load cycles were partly mapped with only one intermediate step during unloading and loading (i.e. only L0, L4, L7, L10 and L14), they are excluded from the figures for clarity. After these five narrowly mapped
cycles, the sample was cycled for additional 4985 cycles and mapped again at L0, to observe possible changes after longer cycling of in total 10 000 cycles.

4.5.2 HRRSM

Four grains were mapped with up to 60 HRRSM acquisitions in total (for grain G4 and grain G5). The appearance of the grains in reciprocal space at the maximum tension before the first load cycle was performed (i.e. after 5010 cycles) is shown in the azimuthal maps in Figure 4.64. Grain G4 and especially grain G5 show an increased amount of internal structure and were therefore the most promising candidates to characterize in detail in the further experiment. It is striking, that all grains appear much smaller and narrower in the azimuthal projection than all grains presented before due to the significantly lower tensile load the specimen has compared to other samples, which have been predeformed using either the load frame or the MTS Acumen.
Figure 4.64: Azimuthal maps at the maximum tension before the first load cycle was acquired for (a) Grain G2 (b) Grain G3 (c) Grain G4 and (d) Grain G5. The azimuthal maps are scaled linearly to reveal the internal structure more clearly.

4.5.2.1 Tension-compression cycling

All grains were mapped by HRRSM after selected number of cycles during the first 5010 cycles. HRRSM was acquired after mounting and grain selection, after tensile loading and the first 50 cycles, after 200 cycles, after 500 cycles, after 5010 cycles and after in total 10000 cycles. The corresponding stress and strain conditions are shown in Figure 4.65. It is visible that the stress measured at maximum tension increases during the first 500 cycles and then decreases between 500 and 10000 cycles. The strain on the other hand shows an initial increase until 500 cycles as well and decreases further before decreasing to 10000 cycles.

The shown azimuthal maps for grain G5 in Figure 4.66 show a clear change in appearance after tensile loading and the first 50 cycles and from 500 to 5010 cycles and almost no difference between 50 and 500 and 5010 and 10000 cycles. This indicates that only limited changes of the microstructure are expected during the first 500 cycles and again between 5000 and 10000 cycles. It is left unclear, if there are changes after tensile loading and before the first 50 cycles as well as when the changes happen between 500 and 5010 cycles as no data are available. The change of the azimuthal width is shown in Figure 4.67 behaves as expected from the azimuthal maps with an increase between 0 and 50 cycles and another clear increase between 500 and 5010 cycles. A further increase is shown between 5010 and 10000 cycles, which is not as clearly apparent form the azimuthal maps. The absolute value of the
azimuthal width after cycling is only about a quarter of the azimuthal width measured for the sample investigated during a cycling sequence in the first APS beamtime (cf. Figure 4.26).

Figure 4.65: The macroscopic (a) stress and (b) strain in dependence of the number of cycles. HRRSM were acquired for the conditions marked with a circle. The condition at 0 cycles corresponds to the condition after mounting, where grains were selected and mapped for the first time.

Figure 4.66: Azimuthal maps for grain G5 after grain selection and different number of cycles during the initial cycling up to 5010 cycles and after 10000 cycles. The first map was done after mounting and grain selection, but before initial loading to the condition corresponding to L0.
Figure 4.67: Azimuthal width for the azimuthal maps presented in Figure 4.66. The reading direction is indicated with an arrow, since the strain was negative after mounting and increased due to minor tensile loading for cycling.

A clear increase in macroscopic stress and strain is visible due to the tensile loading of the sample after mounting and the observation under tensile load (cf. Figure 4.68a). The corresponding radial profiles of the grain G5 are shown in Figure 4.68b. A clear shift of the profile is visible between the profile measured after mounting and after cycling for 50 cycles (including initial tensile loading). As observed before, only minor changes are visible for the radial profiles obtained after different numbers of cycles. The changes in macroscopic stress and profile position, width and asymmetry are shown in Figure 4.69. The strain was slightly negative after mounting, hence a large deviation is visible in comparison to the other maps acquired after a minor tensile loading and cycling.
Figure 4.68: (a) Macroscopic stress measured by the load cell and macroscopic strain (average of the strain measured by the two strain gauges) for the initial 5010 cycles (at the maximum tension, but both for maximum tension and maximum compression at 5010 cycles) and after 10 000 cycles. (b) Corresponding radial profiles for grain G5 after sample mounting, after tensile loading and 50 cycles, 200 cycles, 500 cycles, 5010 cycles (both for maximum tension and maximum compression for comparison) and 10000 cycles.

Figure 4.69 shows the peak profile width (here FWHM), position and absolute asymmetry of the demonstrated radial profiles as well as the azimuthal width gained from the azimuthal maps for comparison. The stress increases slightly during the first 500 cycles, causing a profile shift to lower $q_{\text{mean}}$ (after the initial shift due to the tensile loading). The profile shift then to higher $q_{\text{mean}}$ again, because the macroscopic stress is decreased after 5010 cycles and decreases further at 10 000 cycles (cf. Figure 4.69a), maybe indicating a cyclic softening or relaxation. The profile width is in general increasing with continuing cycling. It actually increases more during cycling than between mounting and the width measured after the first 50 cycles and tensile loading. The increase of width during cycling is $1.54 \cdot 10^{-4}$ and the difference between mounting and the first measurement $2.5 \cdot 10^{-5}$. The profile absolute asymmetry is having a large positive value after mounting (and experiencing compressive load). During tensile loading the asymmetry decreases to $1.8 \cdot 10^{-4}$. 
Figure 4.69: (a) Mean peak profile positions $q_{\text{mean}}$, (c) FWHM, (d) the absolute asymmetry $\kappa$ for the profiles presented in Figure 4.68 for grain G5 (the maximum compression is excluded). The reading direction is indicated with an arrow. The strain was negative after mounting and increased due to tensile loading before cycling.

4.5.2.2 Subsequent tension-compression load cycles

4.5.2.2.1 Entire grains
In the following the analysis of the HRRSM obtained during the first three of the subsequent load cycles will be demonstrated. Subgrains were analysed in detail for grain G5 only. The grain is about 40 $\mu$m large.

The azimuthal map can be reconstructed for each load step along the hysteresis as displayed exemplarily for load cycle 1 in Figure 4.70. No major changes are visible in the appearance of the grain in the azimuthal maps, as observed for azimuthal maps acquired during a load cycle before in the second APS beam time.
Figure 4.70: Exemplary visualization of the 15 azimuthal maps acquired for grain G5 for 15 conditions along the first load cycle (same as the red cycle in Figure 4.63) of the subsequent load cycles.

Figure 4.71 shows the radial profiles for the measurements at the maximum L0, L7 and transition conditions L3 near to the yield and L10 normalized with the maximum intensity. The profile shifts during unloading and loading into compression from the tensile state L0 (black) towards the compressive state L7 to higher diffraction angles $2\theta$ (“proportional” to the diffraction vector $q$ cf. eq. 3.4) and during tensile loading reversingly towards lower angles. The profile L14 at maximal tensile load for the next cycle is at exactly the same $2\theta$ as the profile for L0 (cf. Figure 4.71).
Figure 4.71: (a) Radial Profiles of Grain G5 for load steps L0, L3, L7, L10, L14 during load cycle 1. The profile shifts to higher $2\theta$ from the tensile point (L0, black) during unloading and loading into compression (L7, cyan) and back to the same position during loading in tension (L14, red).

Figure 4.72 shows the radial profiles for the maximum tension and maximum compression from four load cycles following each other. This should visualize again, that the profile attains always the same position during the cycles.

Figure 4.72: Radial peak profiles obtained at the maximum tension and maximum compression for four subsequent load cycles for grain G5.
For Figure 4.73 the local elastic strain of grain G5 was calculated according to equation 3.5 using \( q_{\text{mean}} \) of L9 as the reference condition at zero load and displayed in relation to the macroscopic strain, which is the average measured by the strain gauges. The peak profiles shift to lower and higher values along the load cycle and hence the local elastic strain is (just as the macroscopically measured strain) decreasing during unloading and loading into compression and increasing during unloading from compression and loading into tension. The local elastic strain follows perfectly the applied stress and is of tensile character during unloading for L0-L2 and tensile loading L10-L14 in accordance with the macroscopically applied strain. But already from L3, where the macroscopic strain still is positive, the grain experiences local compressive strain until L9. The local strain amplitude is with \( \varepsilon_a = 4.75 \times 10^{-4} \) lower than the macroscopic strain amplitude (\( \varepsilon_a = 6.3 \times 10^{-4} \)).

![Image of hysteresis curves](image)

**Figure 4.73:** The hysteresis curves from the local elastic strain (from peak position) in relation to the macroscopic strain (as measured by the strain gauges) for (a) the first load cycle (zero strain is marked with a dotted line) and (b) the first three of the subsequent load cycles.

The radial profiles can be analysed with regards to their width (integral width \( \beta \) and FWHM) and asymmetry as well. Both measures for the width of the radial profiles show a characteristic butterfly pattern for all investigated load cycles both for grain G5 (cf. Figure 4.74) and grain G4 (cf. Figure 4.75). For grain G2 and G3 measurements were only done at the maximum compressive and tensile load, but an variation between the FWHM (higher values at the maximum tension and lower values at maximum compression) was observed as well for the five measured load cycles. The profile width and asymmetry
will only be discussed in detail for grain G5 in the following, because the subgrains are analysed for grain G5 only, but the graphs are presented for grain G4 as well to support the observations. The change of the two measures for the profile width during the three subsequent load cycles is presented in Figure 4.74. During the first load cycle the FWHM of grain G5 (cf. Figure 4.74a) is having the maximum value of \(2.5 \times 10^{-3} \, \text{ Å}^{-1}\) under maximum tensile load \(L_0\). The FWHM is then dropping significantly until \(L_4\) to \(2.25 \times 10^{-3} \, \text{ Å}^{-1}\). The FWHM is increasing again until \(2.4 \times 10^{-3} \, \text{ Å}^{-1}\) at \(L_7\) after a small plateau between \(L_4\) and \(L_5\). After the maximum compressive load the FWHM drops during tensile loading until \(L_{10}\) and increases then after a plateau between \(L_{10}-L_{12}\) until \(2.46 \times 10^{-3} \, \text{ Å}^{-1}\) at the maximum tension \(L_{14}\). This behaviour is referred to as butterfly (cf. Figure 2.31). No major changes in the behaviour of the FWHM are visible during these three subsequent load cycles. In comparison to the integral width (cf. Figure 4.74a) shows the FWHM a more systematic and smooth behaviour. The integral width has stronger deviations along the curve, but mimicks the behaviour of the FWHM. Both widths are presented to proof that the behaviour is not an artefact of the used measure for the width.
Figure 4.74: (a) Full Width Half Maximum of the peak profiles (for the axial case) for Grain G5 for Load Cycle 1 and (b) FWHM for the first three of the subsequent load cycles. (c) Integral width for Load Cycle 1 and (d) the integral width for the first three of the subsequent load cycles. All measured load cycles show a butterfly-pattern with smaller variations.
Figure 4.75: (a) Full Width Half Maximum of the peak profiles (for the axial case) for Grain G4 for Load Cycle 1 and (b) FWHM for the first three of the subsequent load cycles. (c) Integral width for Load Cycle 1 and (d) the integral width for the first three of the subsequent load cycles. All measured load cycles show a butterfly-pattern with smaller variations.

Figure 4.76 shows the change in the asymmetry (calculated after section 3.4.3) monitored for the first three subsequent load cycles for grain G5. A characteristic pattern is followed and described in detail for load cycle 1. The absolute asymmetry in load cycle 1 starts with a positive value of $0.7 \times 10^{-4}$ Å$^{-1}$ at the maximum tensile load and increases steeply to the maximum value of $2.2 \times 10^{-4}$ Å$^{-1}$ at L3. The absolute asymmetry drops then slowly until to negative values at compressive load L7 (even more clear for grain G4 in Figure 4.77). The radial profile asymmetry becomes reversed at the maximum compression L7. During unloading from compression and loading to $0.2 \times 10^{-3}$ macroscopic strain at L10 the absolute
asymmetry drops to the minimum of \(-0.9 \times 10^{-4} \text{ Å}^3\) until it increases again after L12. It is nearly constant during the acquisitions L2-L4 and L10-L12. The curve is similar for all three load cycles (cf. Figure 4.76) except for load cycle 3 (blue), where the asymmetry of L4 has already dropped significantly. Both the behaviour of naive asymmetry calculated from the peak profile positions \(q_{\text{mean}}\) and \(q_{\text{max}}\) (cf. Figure 4.76 bottom) and the relative asymmetry (cf. Figure 4.76 middle) is similar to behaviour of the absolute asymmetry. This shows that the finding is not an artefact of the specific measure for the asymmetry. The same characteristic pattern was observed even more clearly for grain G4 (cf. Figure 4.77).
Figure 4.76: Evolution of the asymmetry of the radial peak profiles for Grain G5 for Load Cycle 1 (right) and for the first three subsequent load cycles (left) in dependence of the macroscopic strain. All measured curves follow the same trends for all grains. Top: absolute asymmetry, middle: relative asymmetry calculated from the FWHM, bottom: “naive” asymmetry calculated from the peak position.
4.5.2.2 Subgrain identification

From the intensity distribution measured for the whole grain at 15 conditions along the hysteresis, the fraction representing the contribution from the high intensity peaks can be separated for each load step. Because the sample was only cyclically deformed in tension-compression and experienced no pre-deformation, the azimuthal width of the grain is narrow and the intensity distribution smoother than observed for tensile deformed samples investigated in previous experiments [WEJ11, WEJ13], for which the separation algorithms were developed. The separation with the default parameters used after Wejdemann et al. [WEJ11], was not successful for the HRRSM acquired during the third beam time, because of this narrow intensity distribution. Hence tests were done to change the knot distance for finding an optimum parameter for these HRRSM. The knot distance is an important parameter in the separation procedure using a bi-cubic spline for each azimuthal layer. It has a large effect on the separated intensity cloud and was therefore alternated for the presented investigations. It divides the azimuthal layer into rectangles by a regular grid defined by a set of points, which are called knots. [WEJ11].

Figure 4.78 shows the azimuthal maps of the grain G5 and the separated high intensity peak component for different knot distances used as input parameter for the separation procedure. Figure 4.79 shows the corresponding integrated intensity of the HRRSM in comparison for the grain (in red) and the separated high intensity fraction associated with subgrains (in black). The standard value used for all
other separations was set to [30 10], which means that the distance between the knots is 30 voxel in the \( \eta \) direction and 10 to 30 voxels in the \( \omega \) direction. [WEJ11, WEJ13] It is visible, that both after splitting with [60 20] and [30 10] the separated azimuthal map looks nearly the same. Figure 4.79 confirms, that only very little intensity was separated and attributed to the walls, since the subgrain component has nearly the same intensity as the grain. The other extreme is a separation with [12 4], where only very little intensity is left in the subgrain component, but an increased amount of artefacts visible in the azimuthal map. The separation for [24 8] and [18 6] appear to be more plausible with a limited amount of artefacts and a reasonable fraction of intensity for the subgrain component.

![Images of azimuthal maps for different knot distance parameters](image)

**Figure 4.78:** Example for the azimuthal maps for the total intensity from grain G5 (top) and of the high intensity peak component obtained by separation (bottom) with different knot distance parameters (given in the corresponding HRRSM).

To limit the occurrence of artefacts, a separation with [24 8] was done. With this parameters (among others) for separation, some local intensity maxima associated with dislocation-free subgrains can be identified. However, some artefacts lying in the outskirts were automatically identified as subgrains (since they are local maxima) as shown in Figure 4.80. These artefacts were manually excluded from further statistical analysis for example by the use of a region of interest composed of two rectangles. Figure 4.81 shows the intensity for the first 20 identified subgrains for selected load steps of load cycle 1. It is clearly visible that only a few peaks of very high intensity are identified (up to about 7) and that it does not make sense to analyse at subgrains with a higher order of size than 15, because the intensity
is close to zero. It seems fewer subgrains of high intensity are identified at the maximum compression L7, which will be investigated further.

Figure 4.79: Total intensity for each acquisition of grain G5 for the total HRRSM (grain, red) and the intensity of the separated high intensity fraction (subgrain, black) for five different knot distances used in the separation of the intensity distributions into wall and subgrain component.
Figure 4.80: Azimuthal map of the separated subgrain component for a knot distance of [24 8] (a) with the automatically identified ten most intense peaks and (b) marked with the area of interest (composed of two rectangles) used for further analysis of the subgrains number. The maps are scaled linearly for a more clear visualization of the high intensity peaks.

Figure 4.81 shows the intensity for the first 20 identified subgrains for selected load steps of load cycle 1. It is clearly visible that only a few peaks of very high intensity are identified (up to about 7) and that it does not make sense to analyse subgrains with a higher order of size than 15, because the intensity is close to zero. It seems fewer subgrains of high intensity are identified at the maximum compression L7, which will be investigated further.
Figure 4.81: Integrated intensity shown for the largest 20 subgrains (with highest intensity) identified automatically at the load steps L0, L3, L7, L10 in the first of the three subsequent load cycles for grain G5.

The number of subgrains identified for each load step during the first three subsequent load cycles for a fixed area of interest as shown in Figure 4.81b (to exclude artefacts) is summarized in Figure 4.82. These numbers are only approximations, because the area of interest was fixed for all load steps and it was observed, that the azimuthal positions can vary slightly from measurement to measurement. Even though the numbers might be approximations, a general trend is visible with a decreasing number of identified subgrains during unloading and loading into compression showing a minimum around the maximum compression L7. The number of identified subgrains increases again during unloading from compression and tensile loading. This is in accordance with the observation, that HRRSM appear
smoother around the maximum compression and more compact (cf. Figure 4.70). It was also more difficult to refine the same subgrains for acquisitions made around the maximum compression.

![Graph showing number of identified subgrains](image)

**Figure 4.82:** Number of identified subgrains within a fixed area of interest (as shown in figure b) for all load steps during the first three subsequent load cycles. The maximum compression is marked with a dashed line and the maximum tension is marked with a full line separating the load cycles. The number of identified grains seems to be higher at maximum tension than at maximum compression.

As mentioned the intensity distribution appears to be smoother around the maximum compression with increasing smoothness up to L7. Figure 4.83 shows the total intensity measured for the grain and the intensity contribution of all subgrains (i.e. the entire subgrain component) for all acquisitions made during the three subsequent load cycles. There is an overall tendency for a decrease of the total intensity during unloading into compression to L7. This is though only visible for load cycle 1 and 3, for load cycle 2 is the intensity nearly constant during unloading and loading into compression. At the same time an increase in the intensity of the intensity of the peak contribution to approximately 50% is visible. The fraction of the subgrains is therewith apparently changing along the load cycle. For load cycle 1 the subgrain fraction is nearly the same at the maximum tension L0 and L14. The same is the case for load cycle 3. For load cycle 2 it however increases slightly during the load cycle, meaning that the value for the intensity of the peak contribution is not the same at L0 and L14 for load cycle 2. This effect is however minor.
Figure 4.83: Total integrated intensity of the HRRSM acquired for the grain and the separated subgrain part for the first three subsequent load cycles.

4.5.2.2.3 Subgrains - Results

Four subgrains, that were found from the high-intensity peaks in nearly all of the 43 subsequently acquired azimuthal maps for grain G5 along the three load cycles, were selected for detailed analysis. These four selected high-intensity peaks corresponding to four subgrains were assigned to subgrain numbers 1 to 4. The azimuthal map of the separated intensity distribution for all subgrains is shown in Figure 4.84, where the four selected subgrains are marked exemplarily for load steps L0, L3, L7, L10 for load cycle 1.

In particular from the azimuthal map at L0 it is visible that at least two orientation domains are present. Both domains are lying at different azimuthal positions, where subgrains 1 and 2 are part of one domain and subgrain 3 is part of the other, while grain 4 is located completely in the outskirts of the orientation spread. To investigate, if the visible orientation domains are related to spatial domains within the grain, the data were afterwards analysed (with a centering scan) for a different region of interest, corresponding to the specific azimuthal positions of the two domains. In this way the location of the detected intensity within the tensile sample can be determined, even though the technique does in general not provide spatial information. It was revealed that the diffraction spots for subgrain 1 and 2 and subgrain 3 originate from slightly different spatial positions within the grain verifying the existence of spatial domains, which are identified in orientation space. The center of gravity of the measured
intensity is for the entire grain $x = -4.41, z = 2.351$ for the domain including subgrain 3 $x = -4.425, z = 2.351$ and for the domain including the two subgrains $1$ and $2$ $x = -4.40, z = 2.352$. This means that the spatial regions have different positions along the tensile axis corresponding to the sample $x$-axis.

![Azimuthal maps of the separated subgrain contribution exemplary for load steps L0, L3, L7 and L10 of the first load cycle. Four of the identified high intensity peaks, which could be followed throughout, corresponding to subgrains are marked with white circles. The maps are scaled linearly for a more clear visualization of the high intensity peaks.](image)

The radial profile can be displayed for the identified subgrains at each load step. Figure 4.85a shows the radial profile of the grain and the radial profile of subgrains 1 to 4 for L0, where in general subgrain 1 shows the highest intensity followed by 3, 2 (at least for L0, for some other load steps it might be the other way around) and 4. Hence subgrain 1 is in general the largest and subgrain 4 the smallest subgrain. Subgrain 2 and 3 are of similar, intermediate size. Figure 4.85b highlights, that it was possible to follow those four subgrains for several load steps. All subgrain profiles are moving towards higher $2\theta$ during unloading and loading into compression and to lower $2\theta$ during tensile loading. The peak shift $\Delta q$, which is the difference between the mean position of the individual radial profile for the subgrain minus the mean peak position of the grain profile, is analysed. The local elastic strain for each subgrain is calculated according to equation 3.7 based on the peak shift.
Figure 4.85: Radial profile of the grain G5 and the four marked subgrains at load step L0 in the first load cycle. The profiles are normalized with the maximum intensity of the grain. The subgrain profiles are scaled relatively to the grain and multiplied by a factor 100 for visibility. (b) Radial profiles of grain and subgrains for the steps L0, L3, L7, L10.

Figure 4.86 shows the local elastic strain with respect to the grain for each subgrain 1-4 calculated for all load steps for the three subsequent load cycle 1 (red), load cycle 2 (cyan), load cycle 3 (blue), where the corresponding peak could be identified, in dependence of macroscopic strain. For each subgrain a characteristic behavior is observed through all three load cycles though intermediate exceptions are visible which are likely due to difficulties in the peak identification. Subgrain 1 and subgrain 2 seem to behave quite similar, while subgrain 3 and 4 show a different “hysteresis”. It is striking that all hystereses start and end with compressive strain at the maximum tension L0/L14 or very close to it (subgrain 3). Subgrains 3 and 4 experience compressive strains at nearly all conditions even though the macroscopic strain is positive at the conditions L0-L2 and L9-L14. For subgrain 1 and 2 the local elastic strain is increasing during unloading and loading into compression and then decreasing during unloading and loading into tension. Subgrain 3 behaves in an opposite manner with generally decreasing elastic strain during unloading and loading to maximum compression L7 and increasing local elastic strain during unloading from compression and loading into tension. It actually also shows an opposite sense (counter-clockwise) of the hysteresis. For subgrain 4 the local elastic strain decreases until L4, then it increases as for subgrain 1 and 2 until L7. After this it increases though further until L12.
and decreases first then alike subgrain 1 and 2 until L14. Subgrain 1 and 2 experience though tensile strain from L5, while subgrain 4 only possible experiences tensile strains around L10-L13. The local subgrain strain behaviour for subgrain 1 and 2 correlates with the behaviour expected from the grain asymmetry with a steep increase of the local elastic strain during unloading until L10 and again a steep decrease during unloading from compression until L3.

![Calculated local elastic strain relative to the grain G5 in dependence of the macroscopic strain for four subgrains for all three subsequent load cycles.](image)

**Figure 4.86:** Calculated local elastic strain relative to the grain G5 in dependence of the macroscopic strain for four subgrains for all three subsequent load cycles. The zero strain levels are marked with dashed lines. The reading direction is indicated by arrows, not the different sense (counterclockwise) of the hysteresis for subgrain 3 in comparison to subgrain 1, 2 and 4.
To find a possible explanation for the characteristic behavior of the width and asymmetry of the grain peak profile, both the width (cf. Figure 4.87) and the absolute asymmetry (cf. Figure 4.88) were analysed for the total subgrain profile (the radial profile resulting from all individual subgrain profiles) and the profile for the wall component. The analysed width is calculated as half of the difference of the two FWHM created obtained from the fit profile (with split pseudo Voigt) as described in section 3.4.3. It was of interest to reveal, if the characteristic behavior was visible for one of the components or if it is a possible combination of the components being dominant for the characteristic response of the grain at certain conditions. For the width it is clear, that the width of the total subgrain profile shows a similar response as the FWHM observed for the grain. This is an indication, that the change of profile width is mostly related to the subgrains and their internal stresses. The width measured from the wall profile is on the other hand first overall increasing steeply during unloading and loading until maximum compressive load and then decreasing during unloading from compression and loading into tension.

The same analysis was done for the absolute asymmetry obtained from the fitted profiles, where similar tendencies of the initial increase during unloading, decrease during loading into compression and an increase during loading into tension are visible for the total subgrain profile asymmetry as for the grain. It is nevertheless less clear from the two presented asymmetries, where the characteristic change of the asymmetry may have its origin.

![Graphs](a) LC 1 subgrain total width [Å⁻¹] vs. macroscopic strain ε \times 10^{-3} 
[b] LC 1 wall total width [Å⁻¹] vs. macroscopic strain ε \times 10^{-3}

Figure 4.87: Width calculated from the fitted profiles for (a) for the total subgrain component and (b) the total wall component for load cycle 1 for grain G5. The subgrain component shows the characteristic butterfly pattern as observed for the grain.
An approach to analyse the subgrains statistically was made for clarification of the influence of the subgrains on the characteristic change of the grain peak profile width and asymmetry. As discussed before there was only a small number of subgrains (in comparison to the other cycling experiments after tensile pre-deformation of the sample) identified for each HRRSM and some of the identified peaks were partly artefacts from the separation. For the presented statistical analysis only the first seven subgrains were chosen for each load step, which might even include a few artefacts (resulting in exceptional values). A distribution analysis is difficult with only 7 subgrains, hence the results are not very clear and no systematic changes were observed.
Figure 4.89: Logarithmic integrated intensity of the individual subgrains in dependence of the peak shift $\Delta q = q_{\text{mean(subgrain)}} - q_{\text{mean(grain)}}$ for (a) Load Cycle 1 (b) Load Cycle 2 and (c) Load Cycle 3 for the load steps L0, L3, L7 and L10 for grain G5.

Figure 4.89 shows the intensity of the most intense subgrains in relation to the difference of the mean peak position between the grain and the subgrain profile to possibly show, if there is a size effect as investigated in earlier beam times. It is however not possible to find a clear relation. First of all grains of higher intensity seem to show similar peak shifts as grains of lower intensity and secondly there is no systematic change in the amount of peak shift visible for the different illustrated load steps.

For the distribution analysis (cf. Figure 4.90) only the first seven subgrains were chosen for each load step. A distribution analysis is difficult with only 7 subgrains, hence the results are not very clear and no systematic changes were observed.
Figure 4.90: Normal probability plot for the peak shift $\Delta q$ for 7 identified subgrains in HRRSM of (a) Load cycle 1 (b) Load Cycle 2 (c) Load Cycle 3 for the load steps L0 (black), L3 (green), L7 (cyan), L10 (blue) of grain G5.
4.6 Summary

The results of in total four beam times investigating cyclically deformed AA1050 by applying HRRSM were presented. Before discussion, the observed phenomena and described observations are shortly summarized in this section. The first beam time at APS dealt with the investigations of a tension-tension cycling sequence. The beam time at PETRA and all other APS beam times after this were dealing with tension-compression cycling. While at PETRA only half a tension-compression load cycle was performed, it was possible to acquire measurements for tension-compression cycling sequences as well as for entire load cycles with different strain amplitudes in the second and third beam time at APS. Similar observations like the peak shift along a load cycle were often found in the different experiments and independent of the mechanical testing parameters or sample history. These observations can therefore be generalized for the presented experiments.

The major observations of all beam times are for overview reasons structured into observations on the grain and subgrain level and statements gained from the analysis of azimuthal maps and radial profiles.

4.6.1 Grain level

4.6.1.1 Azimuthal maps

Grains appear very different in azimuthal maps, both in shape and extent. In general, the azimuthal maps of a particular grain appear without major changes during cycling sequences in the later cycling stage, which are assumed to be in the saturation regime, of up to 32505 cycles (highest total number of cycles observed for the pre-fatigued sample investigated at the first APS beam time) and during measurements along tension-compression load cycles. This observation is the same for grains in all samples independent of sample history and pre-deformation. The observation seems also to be independent of the strain amplitude during cyclic deformation. However, some changes in the appearance of grains in the azimuthal map were observed:

- The appearance of the azimuthal maps changes drastically with tensile loading. The map was also found to transform during the early stages of cycling between 500 and 5010 cycles with a
strain amplitude of 6.3 $\cdot 10^{-4}$.

- The azimuthal width of grains after tensile pre-deformation is significantly larger than for grains without unidirectional pre-deformation. The azimuthal width increases after tensile loading and after an increase of the strain amplitude.

- Orientation domains were observed in several of the azimuthal maps. They can eventually be related to spatial domains by repeating the centering procedure for a different region of interest ($\omega$ and $\eta$).

- It was observed, that the appearance of the grains (without pre-deformation) in the azimuthal map slightly changes along a tension-compression load cycle. It is more difficult to re-find subgrains close to maximum compression e.g. due to a more difficult separation and identification. The intensity distribution in the azimuthal map becomes narrower close to the maximum compression being the most narrow at maximum compressive load.

### 4.6.1.2 Radial profiles

The radial profiles reveal only minor changes for measurements obtained cycling sequences both for tension-tension and tension-compression cycling. Measurements were done at the maximum tension after cycling for comparability.

- The peak position after each cycling step remains nearly the same. Changes could be related to the change of macroscopic stress e.g. due to cyclic softening or stress relaxation in the set-up. The profile width and asymmetry was observed to change during the initial cycling directly after tensile loading (e.g. first 800 cycles at the first APS beam time). The width and asymmetry remained nearly constant during later cycling.

- After an increase of the cycling amplitude the profile width and asymmetry increases, the profile position did change in accordance with the macroscopic stress.

A characteristic behavior of the profile position, width and asymmetry was observed in investigations along individual cycles of the tension-compression stress-strain hystereses:

- The radial profiles show a distinct shift in profile position between maximum tension and
maximum compression. The peak position at maximum tension is though nearly the same again after each load cycle. The same is valid for compression.

- The peak profile positions ($q_{\text{mean}}$) follow a hysteresis, increasing during unloading and loading into compression until it reaches the maximum value at maximum compression and decreasing during unloading from compression and tensile loading until it reaches the minimum value at maximum tension.

- Usually a shift in the sign of the asymmetry (from positive to negative) is observed when unloading and loading into compression. The expected profile asymmetry at the maximum compression is negative and was usually negative. The profile asymmetry observed at the maximum tension is expected to be positive character but was observed to be both positive and negative for different grains. This is possibly due to sample bending experienced during mounting and pre-deformation.

- A characteristic butterfly pattern was observed for the evolution of the peak profile width during tension-compression load cycles.

- A characteristic pattern for the evolution of the peak profile asymmetry was observed during tension-compression load cycles.

- The key finding was that the profile asymmetry was observed to increase during unloading from tension and from compression, even though the peak width is decreasing. It was also observed to reverse when loading again from compression towards tension.

It was observed for all samples that the peak profile position, width and asymmetry observed at maximum tension (as well as after unloading and loading into compression) differs from grain to grain. Sometimes groups of grains with similar values can be observed.
4.6.2 Subgrain level

4.6.2.1 Azimuthal maps

It was possible to identify subgrains for all grains from their azimuthal maps. The number of identified subgrains was found to be dependent on the sample history in particular on the amount of tensile pre-deformation. In general a maximum of 100 subgrains (from which 80 were analysed) was found after tensile pre-deformation to 1% tensile strain. Fewer high intensity peaks corresponding to subgrains were observed in samples without uniaxial pre-deformation. Especially for samples with a low amount of tensile deformation it was obvious that fewer subgrains were found in azimuthal maps obtained at conditions close to or at the maximum tension and even less close to or at the maximum compression. The separation of the subgrain component is more difficult for samples without or with only minor tensile pre-deformation due to a more narrow intensity distribution.

- The subgrains are in general evenly distributed in the azimuthal maps. However orientation domains were observed for some grains with regions of a higher subgrain density in comparison to others.
- The subgrain fraction during cycling is overall constant but varies from grain to grain. The subgrain fraction varies slightly within a load cycle.

4.6.2.2 Radial profiles

The analysis of the subgrain positions was of interest to reveal local elastic strains. For this the peak shift, the differences between the peak position of the grain and the subgrains were analysed. Besides statistical analysis of the subgrains and their individual strain in respect to the grain was it possible for the first time to follow four subgrains and their radial profiles over in total three subsequent load cycles and along cycling with up to 7350 cycles.

- Statistical analysis revealed that the radial peak positions of the subgrains are Gaussian distributed around the radial peak position at maximum intensity of the grain. The mean peak shift was observed to change during cycling sequences to slightly higher values indicating that
in total larger local elastic strains are observed for the individual subgrains after a cycling sequence.

- A size effect of the subgrains was observed concerning the peak shift. It was shown that larger subgrains (corresponding to peaks of highest intensity) experience high local elastic strains and therefore show a larger peak shift $\Delta q$ than smaller subgrains (corresponding to peaks of lower intensity).

- It was possible to analyse selected subgrains (up to four) and their radial profiles along load cycles. It was shown for the first time (in the last beam time) that the local elastic strain each subgrain experiences with respect to the grain follows different evolution. One of the possibly reasons was found to be the local environment.

- The shift in peak position of the subgrain was reproduced over several load cycles for the same subgrain. The individual profiles of the subgrains follow however the overall motion of the grain (e.g. shifting to higher values at lower stresses and vice versa).

- The total subgrain and wall component were analysed along the individual load cycles as well to obtain information about the origin of the characteristic behaviour for the profile with and asymmetry of the grain during a cyclic tension-compression stress-strain hysteresis. The radial profiles of the total subgrain fraction show a characteristic butterfly behaviour during a tension-compression load cycle, hence it was assumed, that the behavior of the profile width from the grain profile is influenced by the subgrains. The radial profiles of the total subgrain fraction does not show the characteristic evolution of the asymmetry during a tension-compression load cycle.

It was not possible to obtain statistical information from samples without pre-deformation, due to the limited amount of identified subgrains.
5 Discussion

Bone-shaped AA1050 samples were cyclically deformed after annealing and investigated in-situ by HRRSM. Mughrabi et al. [MUG02] have proposed the presence of long-range internal stresses, which are evolving in a characteristic manner during tension-compression load cycles and cycling. These long-range internal stresses were identified as the major reason for the observation of the Bauschinger effect [BAU1886, MAS25]. Long-range internal stresses are according to the composite model caused by the heterogeneous microstructure developing during cyclic deformation. The aim of the present investigations was to gain a further insight into the microstructural reorganization of subgrains during cycling and to reveal in detail the developing internal stresses of the grain and subgrains during individual tension-compression load cycles.

Investigations were thus done both, for cycling sequences (investigations after certain number of cycles during ongoing cycling) for tension-tension and tension-compression cycling and along the tension-compression cyclic stress-strain hysteresis curve. The main observations of the presented results will be discussed in the following and compared with theoretical predictions from the composite model.

5.1 Cycling sequence

The microstructural changes during cycling sequences were presented for tension-tension cycling for a pre-deformed sample with in total 32505 cycles (cf. chapter 4.2), which was again deformed in tension during the measurements and where a cycling sequence of 7350 cycles after the additional tensile loading was investigated in detail. Microstructural changes were investigated as well for tension-compression cycling for an undeformed sample, where especially the microstructural changes during the first 10000 cycles were of interest (cf. chapter 4.5). High resolution reciprocal space mapping was performed in-situ after each loading or cycling step at the same cross-head position during all acquisitions for the same sample (usually at maximum tension), while macroscopic stress and strain were monitored.

No major changes in the azimuthal maps and in the radial profiles were observed after cycling with different number of cycles in the later stages of cycling (>5010 cycles, cf. Figure 4.66).
It was also possible to follow individual subgrains over 7350 cycles indicating that a stable microstructure was formed, where subgrains were stable during the entire cycling sequence. For the sample investigated at the first beam time it was shown, that the diffracted intensity related to the high-intensity peaks and the volume fraction of the subgrain component in each grain (e.g. about 40%) stay almost constant during the second cycling (cf. Figure 4.25b), furthermore indicating no major changes in the deformation structures in the grains during the performed cycling (C4-C7). By identifying the most intense high-intensity peaks in the high resolution reciprocal space maps from all six load steps (L2 and C4-C8), it was possible to follow four large subgrains through the performed 7350 tension-tension cycles corresponding to an accumulated strain of 6.7, even if a drop in beam intensity after loading step C8 was observed. The constant subgrain volume fraction and the fact that the very same subgrains were found after 7350 cycles indicate that changes in the deformation structure established during tension to 1.3% are rather minor despite the significant number of cycles and confirm the presence of quite stationary deformation structures during the pre-deforming tension-tension cycles. This is further supported by the behaviour of the radial profiles of the entire grains. The peak position after cycling remains almost constant (cf. Figure 4.72) despite the fact that during an individual single cycle significant changes of the peak position occurs (cf. Figure 4.54). As all measurements presented were acquired after pausing the cyclic deformation at the point of maximal displacement, i.e. the upper reversion point of the hysteresis curve, similar peak positions are expected; the observed systematic shift of the mean radial position of each grain towards higher diffraction vectors is caused by stress relaxation during cycling (cf. Figure 4.28).

In general, during the later cycling stages it was expected that the samples were cycled into the saturation regime, where a stable subgrain structure was formed. It is hence not expected that major changes occur due to significant reorganization of the dislocations and the formation of subgrains along the cycling in the saturation stage. Mughrabi et al. [MUG02] describes it as a steady-state or quasi-stationary stage, where the microstructural changes during an individual cycle are self-compensating. Internal stresses are in balance and develop in the same manner during each cycle. [ESS79, GUI84,
MUG02] The microstructural change in this stage is a minor increase in the geometrically necessary dislocation density in the walls, which will result in development of sharper subgrain boundaries with higher misorientations with progressing cycling as observed by Grosskreutz et al. and Mitchell et al. [GRO06a, MIT07].

Cyclic saturation is in general characterized by a constant stress-amplitude during cycling. The existence of a saturation stage after the initial hardening is well known from other fcc metals such as copper and nickel, but has been in discussion for aluminium. Videm et. al [VID96b] showed that polycrystalline aluminium with an average grain size of 110 μm, which is slightly larger than for the investigated samples, experienced cyclic softening after the initial hardening (cf. Figure 2.7). It was however shown by Madhoun et al. [MAD03] for polycrystalline aluminium with similar grain sizes as investigated in the HRRSM experiments, that the stress amplitude is constant during the early cycling (first 100 cycles), where the very first initial hardening was found to occur during the first 20 cycles for a strain amplitude of 6.25·10⁻⁴ (cf. Figure 2.8). This strain amplitude is in a similar range to the presently investigated strain amplitudes, indicating that a very fast initial hardening might occur in the presently investigated samples as well. Cyclic hardening curves measured on aluminium single crystals show that the initial hardening is completed after the first 1000 cycles for strain amplitudes of 1.23·10⁻³ (cf. Figure 2.6).

Present studies on annealed AA1050 dog bone samples (cf. section 3.2) have shown in all cyclic tests performed with the load frame that for a constant displacement amplitude both the stress amplitude and strain amplitude decrease continuously during cycling independent of the strain amplitude (cf. Figure 3.5 and Figure 3.12). The strain amplitude seems to however influence the initial changes of the amplitude as shown in Figure 3.5 for cyclic hardening curves obtained at the MTS Acumen for AA1050 bones and shown in Figure 3.12 for tension-compression cycling with the load frame. The initial changes of the stress amplitude as described in section 3.2 are for any strain amplitude completed latest after around 1000 cycles, which was also the highest value for similar strain amplitudes found in literature.

An initial decrease in stress and strain after loading can possibly be related to relaxation processes in the set-up (e.g. observed for L2 in Figure 4.20). For the measurements shown for the sample investigated at the first APS beam time (cf. Figure 4.20), the macroscopic strain measured by the strain gauges decreases in a similar manner during the individual acquisition sequence (where the cross head
position is kept constant) as during subsequent cycling steps (where the cross head is moved in position control to a smaller displacement and back again but kept constant for acquisition). This cannot be attributed to stress relaxation by forward plastic deformation in the specimen, rather stress relaxation occurs in the entire load frame. Even so, the effect of stress relaxation becomes less and less pronounced after more and more cycles (from 1.35 MPa after the first cycling after loading to 0.15 MPa after the last cycling) as well as the associated strain decrease (from $1.95 \times 10^5$ to $1.4 \times 10^5$) indicating a lower sensitivity of the setup towards further stress relaxation. The simultaneous decrease of the stress and strain during cycling indicates a softening of the experimental set-up, which hinders the observation and confirmation of the expected saturation behavior of the material. Neither softening nor true saturation can be excluded.

Even though no major changes occur during cycling at larger numbers of cycles, it was found that changes possibly occur in the early stage of cycling at low numbers of cycles. It was observed that the grains showed more pronounced changes (in width and asymmetry) of the radial peak profiles after the first 800 cycles after a tensile loading than during the following in total 6650 cycles for the sample investigated during the first APS beam time. The integral width of the radial profiles decreases significantly during the first cycling step (C4), but stays nearly constant for all following cycling sequences (C5-C8) indicating very little structural reorganization as also evidenced by the azimuthal maps. In this respect, the integral width after the first 800 cycles after loading (C4) seems to differ for all four grains from the trends observed for the profiles during subsequent cycling up to 6650 further cycles. This may indicate that during the first cycling step after tensile loading reorganization occurs, where the deformation structure introduced by tensile loading adjusts slightly to accommodate cyclic deformation as described by Mughrabi et al. [MUG78] for pure copper. This is further substantiated by an increase in the macroscopic strain amplitude between L2 and C4. As the first HRRSM was acquired after already 800 cycles after tensile loading, this initial reordering cannot be analysed in more detail. Quantification of the changes during the first 800 cycles hinted towards only a minor adaption of the deformation structure to the new deformation condition. If any structural adaption has occurred, it may have already been completed then, as no obvious changes occur during the following cycling steps.
To investigate the microstructural changes during the early cycling, the sample investigated at the third beam time with tension-compression cycling was stopped and investigated after mounting and grain selection and after 50, 200, 500, 5010 and 10 000 cycles. Changes in the azimuthal maps were visible in the map for 50 cycles in comparison to the appearance after mounting and grain selection and in the map for 5010 cycles in comparison to the map acquired after 500 cycles as shown in Figure 4.66 and confirmed by the changes of the azimuthal width in Figure 4.67. From this it can be established that microstructural changes seem to occur within the first 50 cycles (though initial tensile loading was done as well), similar to the statements Madhoun et al. [MAD03] and [VID96b] made about an initial hardening during the very first cycles for high strain amplitudes like the performed 6.3·10⁻⁴. Microstructural changes occur again between 500 and 5010 cycles at the given strain amplitude. Unfortunately, no measurements were done between 500 and 5010 cycles, it is hence unclear when the observed changes appeared.

5.2 Size effect

Subgrains were identified for all investigated samples though to a different extent. A relation between the extent of the grain in the azimuthal map, which was shown to be dependent on the degree of tensile strain, and the number of identified subgrains was found.

The individual peak positions of the subgrains follow for all conditions a Gaussian distribution (e.g. cf. Figure 4.46a) centred around the position of the peak maximum as expected from the refined composite model [PAN10]. Additionally, a peculiar size effect was discovered based on a statistical analysis of the individual subgrain profile positions and their peak shifts. Larger subgrains experience larger internal back strains than smaller ones (cf. Figure 4.36). This effect can be explained by presuming that larger subgrains have a lower yield stress than smaller ones as dislocations require lower resolved shear stresses to pass between obstacles of larger distances by bowing [KUH82], evidenced, for instance, by deformation structures with smaller average subgrain sizes having reportedly larger yield strengths [STA72, GIL80, RAJ86]. When applying increasing tensile stresses, large subgrains yield first and experience locally the largest plastic strain. Consequently, they develop the largest interface dislocation density and hence the largest elastic back strains.
It was shown that some subgrains can be identified for nearly all load steps along the three subsequent load cycles for the sample investigated during the third beam time. The different subgrains show a different local behavior (cf. Figure 4.86). Variations in the three load cycles are possibly due to the manual analysis of the intensity peaks, though small variations might also be caused by a possible minor change of the subgrain shape due to plastic deformation experienced during the subsequent load cycles and with this their response. The grain structure of the investigated grain appears to consist of two major domains as visible in the azimuthal projections (cf. Figure 4.84), where subgrain 1 (and possibly subgrain 2) and subgrain 3 are belonging to the different domains at opposite sides of the azimuthal map. The local elastic strain in those subgrains seem to behave in an opposite way as shown in Figure 4.86. Subgrain 4 shows an entirely different behavior. This shows that the local behavior of the subgrains must be influenced by their size and most importantly their environment.

5.3 Tension-compression load cycle

The evolution during individual load cycles was investigated for several subsequent load cycles, by interrupting the load cycle at desired stress-strain conditions and acquiring HRRSM data. Up to five subsequent load cycles were recorded in detail for the third APS beam time. It was observed that all grains show different values for the profile position, width and asymmetry (cf. Figure 4.54, Figure 4.55 and Figure 4.56). The asymmetry was usually positive in tension and negative in compression, though some grains show a negative asymmetry in tension as well (and throughout the entire load cycle). This behaviour is likely attributed to the local environment. Each individual grain is surrounded by other grains with different sizes and orientations each of those showing an individual response to external stresses. The different grains are thus embedded within different environments of different stiffnesses. Each grain and its environment can in principle be considered as a composite of a higher order, following similar behavior as the subgrains, which are embedded in an environment of other subgrains, where each of the subgrains was found to show locally different elastic strains. However, some influence of sample bending prior to the experiment cannot be entirely excluded.
The radial profiles obtained from individual tension-compression load cycles were analysed regarding their profile position, width and asymmetry. This was done for up to three subsequent load cycles to verify the findings. It was found that the peak profile positions at the maximum tension are nearly the same during several subsequent load cycles. The same is observed for the peak position at maximum compression (cf. Figure 4.63). The profile positions measured at maximum tension after longer cycling were however found to be slightly shifted after cycling with a higher number of cycles caused by a drop in macroscopic stress and strain, cf. Figure 4.20. It was observed for several grains at various conditions cf. chapter 4.3 - 4.5, that the peak position shifts significantly during a cyclic stress-strain hysteresis, before the same value at the maximum tension is attained again. The peak profile position shifts to higher values during unloading and loading into compression and to lower values during unloading from compression and loading into tension. For the derived elastic strain of the grain this means, that the local elastic strain increases during tensile loading and decreases during unloading and loading into compression, as expected from Hooke’s law:

$$\epsilon_{el}^{\text{strain}} = \frac{\sigma}{E} \quad (5.1)$$

This trivial observation of the peak shift along a load cycle shows that the experimental set-up is working and results are reliable. More interestingly, both the radial profile width and the radial profile asymmetry, change in a very characteristic way, which has not been measured in such a detail before. The documented behaviour was observed for several grains and for three subsequent load cycles from the third APS beam time for tension-compression cycling without pre-deformation.

The primary findings of the work related to this thesis were the observations of a characteristic behaviour of the peak profile width and asymmetry during a tension-compression load cycle. While the profile width in principle changes according to theoretical expectations from the composite model and first experimental attempts in measuring by Biermann et.al [BIE92], the observed characteristic behaviour of the profile asymmetry was completely unexpected and not predicted by the existing theoretical models or documented in previous experiments.
Before discussing the experimental findings in detail, the theoretically expected behaviour of the profile width and asymmetry during a tension-compression load cycle will be derived in the following. First, the relative asymmetry \( \kappa_{\text{rel}} \) can be derived from the difference between the elastic strain of the grain and the total subgrain component:

\[
\kappa_{\text{rel}} = \varepsilon^{\text{grain}} - \varepsilon^{\text{subgrain}} = -\Delta\varepsilon^{\text{subgrain}} \quad (5.2)
\]

\[
\Delta\varepsilon^{\text{subgrain}} = \frac{\Delta\sigma^{\text{subgrain}}}{E} \quad (5.3)
\]

The local elastic strain of the subgrain is derived from the local elastic stress of the subgrain presented in Figure 5.1a. The behaviour of the local elastic stress for the entire subgrain component can be constructed from the composite model as described for the walls and cells in section 2.3.1. Figure 5.1a shows the expected ideal response without work-hardening. If the sample experiences work hardening the edges will appear more curved with a smoother transition. The changes of the relative asymmetry during a tension-compression load cycle constructed from the behaviour of the local elastic stress predicted by the composite model is shown in Figure 5.1b in red. The green curve in Figure 5.1b should visualize the experimentally observed behaviour, which will be described in detail when discussing the asymmetry later. For the investigated samples only limited work hardening is expected, the evolution of the asymmetry should therefore be similar to the theoretical expected shape shown in Figure 5.1 with less pronounced tails.
Figure 5.1: Expected hysteresis behaviour of the local subgrain stress in dependence of the macroscopic stress-strain curve and (b) the expected hysteresis behaviour for the asymmetry (red), since it is derived from the curve in (a). The green curve is visualizing how the curve for the asymmetry observed in the experiments approximately appears.

The theoretical profile width can be constructed from the composite model as well. According to the composite model the peak profile $I$ is a superposition of a wall $I_w$ and a cell (subgrain) $I_c$ component as shown in equation 5.4.

$$I(2\theta) = f_w I_w(2\theta) + f_c I_c(2\theta)$$  \hspace{1cm} (5.4)

Assuming a Gaussian distribution for both subprofiles (as presented for the subgrains e.g. in Figure 4.46), the integral width of the superposition is given by equation 5.5.

$$\beta^2 = f_w^2 \beta_w^2 + f_c^2 \beta_c^2 + f_w f_c \Delta q_{cw}^2$$  \hspace{1cm} (5.5)

This shows that we measure the last term with our experiments, the difference between the profile position of the total subgrain component and the wall profile $\Delta q_{cw}$, which can be related according to
equation 3.8 to the difference of the profile position between the grain and the subgrain $\Delta q$. The difference $\Delta q_{cw}$ is changing during our tension-compression load cycle in a characteristic manner due to internal stresses experienced by the subgrains and walls. This change influences the behaviour of the integral width resulting in a slightly different butterfly-pattern, when relating the peak width to the macroscopic strain observed during a tension-compression load cycle.

$$\Delta q_{cw} = q_c - q_w = (q_c - q_{\text{mean}}) - (q_w - q_{\text{mean}}) = \Delta q_c - \Delta q_w$$

(5.6)

With

$$f_w\Delta q_w + f_c\Delta q_c = 0$$

(5.7)

$$\Delta q_{cw} = \Delta q_c \left(1 + \frac{f_c}{f_w}\right) = \frac{\Delta q_c}{f_w}$$

(5.8)

The absolute asymmetry $\kappa_{\text{abs}}$ can be derived from the difference of the mean $q$-value for the profiles of the wall and individual subgrain component according to equation 5.9.

$$\kappa_{\text{abs}} = q_{\text{max}} - q_{\text{mean}} \approx q_c - q_{\text{mean}} = \Delta q_c = f_w\Delta q$$

(5.9)

From this equation the internal peak width of the radial profile and the absolute asymmetry can be related to each other as shown in Figure 5.2 and as expected from the composite model for a two-component material. The square of the integral width shows a parabolic behaviour due to the square of $\beta$. The width is however be expected to be dependent on the asymmetry as shown in equation 5.10, which would mean that the width is expected to increase and not decrease, when the asymmetry is increasing. The maximum of the asymmetry is therefore expected to be, where the maximum of peak width is observed.

$$\beta^2 = c + \frac{f_c}{f_w} \kappa_{\text{abs}}^2$$

(5.10)
\[ \beta = \sqrt{c + \frac{f}{f_w} \kappa_{abs}^2} \]  \hspace{1cm} (5.11)

**Figure 5.2:** Constructed expected behaviour of the evolution of the integral width $\beta^2$ and the asymmetry $\kappa_{abs}$ in dependence of the macroscopic stress and strain for a tension-compression stress strain hysteresis.

Both theoretical curves however show less detail than the experimentally measured behaviour, though the behaviour of the width follows in principle the predicted changes.

The peak width is related to the dislocation density and to the heterogeneity of the structure and the internal strains of the subgrains. For three subsequent individual tension-compression load cycles a characteristic manner of the change in the peak width was found both for the integral width as well as for the FHWM similar for each load cycle. The width was observed to decrease during elastic unloading and loading, to stay nearly constant for the measurements around the macroscopic yield point. During macroscopic plastic deformation the profile width increases again, possibly due a built up of internal stresses, until the maximum.
A similar behaviour of the peak profile width during a tension-compression stress-strain hysteresis was revealed by Biermann et al [BIE92] applying classical X-ray diffraction. The considerable differences to this experiment are that the measurements presented here are performed in-situ on the same sample without unloading and several more load conditions. Taking this into consideration, only two of the measurements presented (L9 after unloading from the maximum compression and L2 after unloading from the maximum tension, cf. Figure 2.30) are comparable to the earlier work of Biermann et al [BIE92]. From the described diverse measurements it can be seen, that the butterfly pattern is more complex than described by Biermann et al [BIE92], where the minima were measured in samples unloaded from the plastic regime. Moreover from the experiments presented here, it becomes clear that the values obtained for measurements of the maximum compression and maximum tension by Biermann et al. equivalent to L2 and L9 are not corresponding to the actual maximum, which seem to occur at higher values than the previously measured states. This is a result of the findings in the presented experiment, that the integral width is decreasing during unloading.

In order to investigate if this characteristic change of the peak width can be attributed to the change in width of the total subgrain or the total wall profile this behaviour was investigated individually. It was found that this change in the peak width is mainly affected by the total subgrain component, which shows the same characteristic butterfly pattern for the change in its width during a load cycle (cf. Figure 4.74 and Figure 4.75). This supports the idea, that the internal stresses of the individual subgrains (as described by the composite model) and with this the width of the total subgrain profile changes and consequently the width of the grain profile changes. The width is expected to be the highest, when the difference $\Delta q_{\text{xx}}$ between the elastic strains of the walls and cells is the highest and with this have the largest difference in peak profile position causing a broad profile. This is as expected the case at the maximum tension. It is expected at maximum compression as well. This was also shown by [BIE92].

The experimental results show however, that the width at the maximum compression is slightly lower than the width at maximum tension. This is very likely due a non-symmetric behaviour of the applied stress-strain hysteresis. The measured stress-strain hysteresis (eg. Figure 4.63 for experiments during the third APS beamtime) has a slightly larger part within the tension regime than within the compression regime causing a shift and distortion of the ideal butterfly with perfect symmetry.
A key finding was the observation of a characteristic behaviour (cf. Figure 4.76 and Figure 4.77) for the evolution of the asymmetry during a tension-compression behaviour. The asymmetry was most surprisingly increasing up to the macroscopic yield point during unloading and loading into tension, where the overall profile width was found to decrease towards maximum compression and vice versa resulting in a characteristic curved parallelogram (cf. Figure 4.77).

A comparison of the theoretical prediction derived from the subgrain stress and the experimental finding is shown in Figure 5.1b (red for the theoretical and green for the experimental behaviour, arrows indication the direction from unloading from maximum tension). Theoretically an initial decrease of the profile asymmetry (in accordance with the initial decrease of profile width) is expected. This means the change of the measured asymmetry is not just initially different and puzzling (due to a decrease in width) as shown in Figure 5.1b but also in the complete opposite direction of the theoretically predicted asymmetry (i.e. counterclockwise, green arrow). The maximum and minimum asymmetry where not observed at the maximum and minimum tension but close to the macroscopic yield point. As shown in Figure 5.1 and Figure 5.2 the experimentally measured behaviour is not according to predictions from literature and the composite model, but in fact nearly the opposite. The behaviour was found for all three types of asymmetry investigated to exclude influences of the fit procedure. The asymmetry changes sign for most of the investigated grains (with some exceptions, cf. Figure 4.56), when unloaded from compression in comparison to when unloaded from tension as described by Ungar et al. [UNG83,UNG94] and is positive at the maximum tension.

The only possible explanation for the unpredicted and new behaviour of the asymmetry is the size-effect of the subgrains described in section 5.2. Larger subgrains were found to have larger strain and are concluded to yield first. All subgrains will yield successively depending on their size until the macroscopic yield point is reached. Because of this is the asymmetry, even though a decrease in profile width (due total decrease in internal stresses) is observed, increasing up to the yield point and only from the macroscopic yield point behaving as expected. This is not predicted by the composite model, which only takes two major components, the wall and the subgrain component, into consideration. Different elastic strains of different elastic subgrains are already take care in the refined version of the
composite model [PAN10]. In order to model the evolution of width and asymmetry, the size dependent response would have to be considered additionally together with a size distribution leading to the observed Gaussian distribution of the elastic strains of individual subgrains.

Another clear explanation for the characteristic asymmetry behavior was despite extensive data analysis not found. The analysis of the total cloud and subgrain component could not reveal, why the asymmetry is increasing during unloading, when the profile width is increasing. The current separation procedure limits the identification of subgrains, if they are in their intensity close to the base intensity of the grain because of very similar orientations as it was the case for the sample investigated in the third beamtime, where the asymmetry behaviour was observed the best. An increase of the angular resolution of the acquired data is needed for a better identification. This can be done by using another detector and aiming for a smaller pixel size.
6 Conclusion

High Resolution Reciprocal Space mapping was applied in in-situ measurements to gain information about the local internal strains in cyclically deformed polycrystalline aluminium along cycling sequences and in individual tension-compression load cycles. Azimuthal projections and radial profiles were presented for grains embedded within macroscopic tensile samples investigated at in total four weeks of beam time applying different mechanical testing conditions. The successful application of HRRSM on a cyclically deformed polycrystalline metal was demonstrated and novel scientific results on the dynamics of grains and subgrains during different regimes of cyclic deformation were obtained. The novel findings gained from the analysis of individual grains and subgrains during cyclic deformation by HRRSM are shortly listed:

- It was possible to identify subgrains in the microstructure formed by cyclic deformation applying HRRSM. The individual subgrains and their local elastic strain could be followed over a large number of cycles and along several tension-compression stress-strain hystereses.
- From the analysis of azimuthal projections it was concluded that the microstructure of the grain is stable during the investigated cycling conditions and along the single load cycles. Individual subgrains are stable during unloading from maximum tension and loading into compression and during unloading from compression and loading to tension. This was confirmed by the successful identification of exactly the same subgrains both after a larger number of cycles and along several load cycles.
- The size effect associated with larger subgrains experiencing larger local elastic strains than smaller subgrains was experimentally confirmed. This implies an earlier onset of yielding for larger individual subgrains than for smaller individual subgrains.
- Detailed observation of characteristic pattern for the changes of profile width (“butterfly” pattern) and profile asymmetry during a tension-compression load cycle were presented for the first time.
- A central finding was, that the asymmetry behaves in an opposite sense to the theoretically
predicted asymmetry even showing an initial increase during unloading from maximum tension and decrease to more negative values during unloading from maximum compression i.e. always increasing in amount (absolute value) during unloading before decreasing and reversing. In the macroscopic elastic regime the asymmetry behaves opposite to the profile width (e.g. increasing when the width is decreasing). This has been interpreted to be a result of the yielding behaviour of the individual subgrains and the size effect and can not be explained by the classical composite model.

- Four subgrains were followed and their radial profiles analysed for three subsequent tension-compression load cycles. The local elastic strain with respect to the grain was found to be different for all subgrains and possibly dependent on their local environment. It was also found that all subgrains show different values for the profile position, width and asymmetry even though the overall behaviour is the same. This is discussed to be an effect of their local environment as well.
- It was observed that the profile width and profile asymmetry increases after increasing the cycling strain amplitude. This observation is in accordance to the composite model.

For future investigations it is necessary to do even more statistics both on grains and subgrains to verify general observations and to separate them from exceptions. This requires in the first place an improvement of the speed for data acquisition and the data analysis. To observe fatigue phenomena also in the regime of crack initiation and crack propagation higher number of cycles have to be performed with the custom-made load frame, requiring an improvement of the cycling speed as well. First attempts were made to acquire data “on-the-fly” by using another faster detector, where the overhead time could be reduced. In this way large amounts of data can be acquired in a short time.

Dark field X-Ray microscopy is presented as a promising technique for complementary investigations providing high spatial resolution. SEM Orientation imaging and EBSD were demonstrated as useful characterization tools for large area analysis with good angular resolution.

The findings revealed a variety of different aspects in the microstructural behavior of grains and subgrains. Exceptions were often found for a phenomenon that was observed in several cases, hence
statistical analysis is of importance in future investigations and the further development of the
presented analysis can then help to evolve more precise models for cyclic deformation. The complexity
of the results obtained from investigating microstructural evolution during cyclic deformation is
represented by the many researches dealing with cyclic deformation and fatigue, which will be in focus
of interest as a fascinating and challenging research field for many years.
7 Appendix

7.1 Outlook: Dark Field Microscopy

7.1.1 Introduction

In previous chapters High Resolution Reciprocal Space Mapping (HRRSM) was demonstrated as a powerful tool to gain insight into the local elastic stresses of grains and subgrains in a macroscopic tensile sample under deformation. However, the grains are only visualized in orientation space and only limited spatial information of the real crystalline lattice can be gained. As demonstrated in chapter 2 the intragranular microstructure of grains exposed to cyclic deformation undergoes a continuous development especially in the early stages of cyclic deformation, where dislocations organize themselves into heterogeneous networks. It is of high interest to map in real space the development of the heterogeneous dislocation distributions and the formation of subgrains in-situ during cyclic deformation and to investigate the local neighbourhood of elastically strained grains and subgrains by a multiscale characterization approach.

A promising technique, which will allow a non-destructive three-dimensional mapping of the intragranular structure, orientations and strains is the recently developed synchrotron technique Dark Field X-ray Microscopy (DFXRM). DFXRM has been successfully applied to image subgrains (and their evolution during annealing) in deformed aluminium, strain fields and domains in ferroelectric materials [SIM18] and strain fields around individual dislocations in three dimensions [JAK19].

Similar to an electron microscope DFXRM enables the investigation on different lengths scales e.g. by the implementation of a fast pre-scanning with 3DXRD or Diffraction Contrast Tomography (DCT) before a DFXRM study. In this way, a larger region within the sample can be analysed before zooming in on the feature of interest. An example of multiscale mapping is the investigation of 10% tensile deformed AA1050 which is is shown in Figure 7.1. A volume consisting of several grains was first mapped by DCT and one grain was chosen for further analysis. The inhomogeneous orientation within the grain was
mapped and related to the three-dimensional appearance of the grain. From this subgrains were selected and their shape and internal strain was analysed further. [SIM15, SIM16]

Figure 7.1: Example of multiscale characterization of tensile deformed AA1050 (a) Diffraction contrast tomography provided a 3D grain map. (b) One grain was selected for further analysis of the intragranular orientations with DFXRM. (c) Subgrains were selected from this grain and analysed as well. Both the spatial and angular resolution are increasing from right to left with increased magnification being 300nm and 0.03° for the subgrain analysis. Reproduced from [SIM15].

Another example to show the potential of DFXRM in the characterization of the intragranular structure is given in Figure 7.2. Here a grain embedded in recrystallized AA1050 sample was mapped revealing internal misorientations of up to 0.05°. An angular distribution is provided for each voxel. [SIM16]

Figure 7.2: One layer obtained for a grain embedded in a recrystallized AA1050 needle. The local misorientations and FWHM can be obtained for each voxel. Reproduced from [SIM16].
As part of this Thesis work first attempts to apply Dark field X-ray microscopy (DFXRM) on cyclically deformed AA1050 samples were done. Preliminary results are presented below. The aim of the investigations was to image the orientation domains and subgrains within an embedded grain after cyclic deformation with high spatial resolution. A direct comparison was provided between maps of the microstructure after annealing but before deformation and the microstructure after cyclic deformation with established parameters. These results can possibly be directly combined with information about the internal stresses gained for the same sample by HRRSM. At the same time the data generated were test data sets for the validation of a new topo-tomography code, intended to provide a 3D reconstruction.

As a result a 3D reconstruction of the undeformed sample was obtained. The final reconstruction for the cyclically deformed sample however failed and below only preliminary results of the 3D shape are presented.

7.1.2 Experimental methods

7.1.2.1 Material

The trials to visualize the grain structure of cyclically deformed aluminium in 3D were done investigating samples manufactured from the material used for previously demonstrated HRRSM investigations. Two grains in two samples were studied during a beam time for obtaining tomography data from selected grains. The reference Sample A remained undeformed after annealing as described in the material section. Sample B was prepared from the tensile sample investigated during the second APS beam time. Sample B was thus initially strained in tension to a strain of 1% and then cycled for about 60000 tension-compression cycles with a final strain amplitude of 0.75·10^{-3}. The detailed sample deformation for sample B is described in chapter 3.1. Subgrain structures consisting of dislocation-rich walls and dislocation-free cell interiors were found in sample B after deformation. The intragranular structure was investigated by HRRSM in section 4.4 and by electron microscopy. Figure 7.3 shows representative SEM images of the microstructure observed in SEM investigations for sample A and
sample B. Sample A shows no clear intragranular structure, while subgrain structures are clearly visible in sample B. The subgrains are typically of sizes of 2 µm to 5 µm.

![Image](c) (b)

**Figure 7.3:** (a) SEM-BSE image of an annealed and undeformed AA1050 sample showing no subgrain structures inside the grains. The visible irregularities are dirt and defects introduced by sample preparation due to the high material softness. (b) SEM-BSE image of a grain observed in Sample B showing a clear subgrain structure with different orientations.

### 7.1.2.2 Sample preparation

Small lamellae with a size of 1 mm x 3.5 mm x 0.6 mm was cut out of the center of the gauge section, which has been analysed by SEM prior to the sample preparation. Hence the cuts were made from a sample previously prepared for electron microscopy as described in section . The sample was cut using a Struers Accutom 50 high precision cutting machine with lowest possible force and speed to minimize the mechanical deformation introduced during cutting. A sketch shows the take-out location of the samples from bone-shaped tensile test specimens in Figure 7.4. The long axis of the lamellae corresponds to the loading axis during tensile and cyclic deformation. Several lamellae were cut for each sample with a thickness of around 600 µm. The lamellae were then chemically thinned in a 15% solution of sodium hydroxide and distilled water for 7 min at 55 °C to further reduce the thickness to 300 µm for DFM analysis and to remove the deformation layer from cutting. However only the most central lamellae was used in DFXRM experiments. Those were finally attached to a brass cylinder using melted resin (cf. Figure 7.5) and mounted in the goniometer with the long axis parallel to the rotation axis $\omega$.  

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Figure 7.4: Schematic overview of the sample preparation for X-Ray Diffraction Microscopy investigations at ID06, ESRF. Sample A and B are taken out of the center a bone-shaped sample.

Figure 7.5: Image of the AA1050 lamella after chemical etching and mounting on to the brass cylinder before attaching to the goniometer.

7.1.2.3 Basic principles of Dark Field X-Ray microscopy

Dark Field X-Ray Microscopy has been established at beam line ID-06 at the European Synchrotron Facility (ESRF) using hard X-rays. Inspired by the principles of dark field microscopy in a Transmission Electron microscopes, an objective is placed between the sample and detector in the diffracted beam. By the use of high energy X-rays it is possible to penetrate macroscopic samples to image grains embedded within the bulk in a non-destructive manner. A spatial resolution of ~100 nm and an angular resolution of ~0.001° can be achieved by DFXRM. [SIM15]

Figure 7.6 illustrates the basic principle of the set-up used at ID-06 at ESRF for DFXRM. A crystalline sample with dimensions of about 1 milimeter is mounted in the center of rotation of a goniometer. The sample to objective and objective to detector distance can be varied – with the total length varying between 2 – 6 m – in order to provide the desired magnification ratio. The goniometer allows a rotation
ω, with the rotation axis being parallel to the diffraction vector \( \mathbf{q} \). The goniometer can then be rotated by a base tilt \( \mu \) and the sample can be tilted in two orthogonal directions with the sample tilts \( \chi \) and \( \Phi \). The tilt motors are used to align the grain of interest with the rotation axis of \( \omega \) parallel to the diffraction vector \( \mathbf{q} \). The detector can be translated in two directions to allow the observations of inclined reflections in e.g. a pre-defined environment such as a load frame. The coordinate system is described in detail by Poulsen et al. [POU17].

The Bragg diffracted beam is magnified by the objective, which is the central optical element in the set-up, and then detected by a CCD detector. The objective consists of a compound refractive X-ray lens, a CRL [SNI96]. The numerical aperture is relatively small while the setup exhibits a high angular resolution. The objective is also used as a filter for the diffracted signal of interest, since it only allows diffracted signals if the diffraction vector is in its origin close to a desired point in reciprocal space. In this way a certain feature e.g. grain can be selected out of diffracting volume consisting of many grains.

![Diagram](image)

**Figure 7.6**: (a) Sketch of the geometry used for Dark Field X-Ray Microscopy. The mm-sized sample (blue) is mounted on a goniometer. The goniometer can be tilted in \( \mu \) (base tilt), \( \phi \) and \( \chi \) (sample tilts) and rotated in \( \omega \). The diffraction vector \( \mathbf{q} \) is parallel to the rotation axis of \( \omega \). The diffracted beam (characterized by the two angles \( 2\theta \) and \( \eta \)) is magnified by the objective CRL and detected by a CCD detector. (b) Detailed geometry of the goniometer rotations. The laboratory coordinate system has its origin in the center of rotation of the goniometer. Reproduced from [POU17].
To study how orientations are spatially distributed within a 3D volume of a grain, the sample is rotated in the rocking direction (rotation around the y-axis) and in the so-called rolling direction (rotation along η, cf. Figure 7.6b). From the intensity variations a 2D map can be generated of the mosaic spread. Likewise 2D maps can be acquired of the axial strain component. This is performed by a scan of the “2θ-arm”, that is a simultaneous vertical translation and rotation of the objective and vertical translation of the detector.

Basically two different measuring approaches can be pursued to obtain volumetric mapping in 3 dimensions:

1. A narrow line beam can be used to illuminate a “slice” of the material and the sample is then stepwise translated through the beam. This is repeated for several layers until the desired volume has been investigated. A 3D map is then obtained by stacking the different layers on top of each other. This has been the approach for most of the previous DFXRM studies. However, data acquisition using this approach is time-consuming.

2. In the so-called topo-tomography collection mode [LUD01] data is acquired in a tomographic approach by scanning the fully illuminated grain of interest in w by at least a rotation of 180 degree. This modality can also be used with microscopy - this is in principle a faster approach. However, reconstruction software for magnified topo-tomography is not yet established, which was the one chosen for the experiment.

The actual data acquisition involved a 3D grid of three rotations. In the outer loop the grain of interest is rotated around ω with equidistant steps Δω. The sample is rotated around the scattering vector, which is aligned parallel to the rotation axis of ω. At each ω-setting the tilt µ is moved in equidistant steps Δµ, where at each step Δµ a rocking of the sample in small steps Δγ (equal to the step size Δµ) in a (new) direction γ perpendicular to the base tilt µ is done. With this procedure a set of images is acquired for each ω position corresponding to different tilts in µ and γ.
The corresponding steps $\Delta \phi$ and $\Delta \chi$ in $\phi$ and $\chi$ to align the rotation axis $\omega$ with the diffraction vector can be calculated according to equation 7.1 and 7.2, assuming a position of $(\phi_0, \chi_0)$ for the diffracting lattice point.

$$\Delta \chi = \chi - \chi_0 = -\cos(\omega) \Delta \gamma$$  \hspace{1cm} (7.1)

$$\Delta \phi = \phi - \phi_0 = \frac{\sin(\omega)}{\cos(\chi_0)} \Delta \gamma$$  \hspace{1cm} (7.2)

To improve the quality of the reconstruction a square slit was used in front of the refractive lenses. Following a topo-tomography reconstruction procedure as described in detail by Cereser et al. [CER17] the aim was to reconstruct the shape of the grain as well as to provide a map of the internal orientation gradients. The reader is referred to the publications by Poulsen et al. [POU17] for more details about the resolution function and construction algorithms. A strain resolution of up to $10^{-5}$ can be achieved. [SIM16]

### 7.1.2.4 Data acquisition and data treatment

Topo-tomography scans were done for one grain in sample A and one grain in sample B. The beam energy was 17 keV with using an Si(111) Bragg-Bragg monochromater. The desired (200) reflection was observed of a $2\theta$ of 20.75° for a lattice spacing of 4.0495 Å using a horizontal scattering geometry with $\eta = 90^\circ$. The far-field detector was placed 5.53 m behind the sample. The objective was positioned 0.204 m behind the sample giving a magnification of 18.7. The beam was defined to a box beam of 150 $\mu$m x 150 $\mu$m by a slit and the camera was binned 4x4. The CRL is comprised of lenslets with a radius of 50 $\mu$m and a thickness of 2 mm. Figure 7.7 shows an image of the experimental DFXRM set-up used for topo-tomography scans.
Figure 7.7: Experimental set-up at ID06 at ESRF used for topo-tomography showing the goniometer with the mounted sample (using a horizontal scattering geometry, marked with an arrow), the objective and detector. The incoming beam is coming from the right in the image.

For sample A the grain size was determined to be 30 µm. The motor ω was rotated in a range of 0° to 180° in steps of 0.8° for Δω. The motor γ was rotated in in total seven steps of a step size of 0.032° in the range of -0.012° to 0.012°. As described above for each γ a rotation in μ is done with the same parameters. Hence μ was rotated in a step size of 0.032° in the range of -0.012° to 0.012° as well. The exposure time for each image was 2 seconds to obtain a good signal to noise ratio with intensities of around 5000.

For sample B the grain size was determined 38 µm. In this case ω was rotated in a range of 0° to 180° in steps of first 0.8° (for the first 96 projections) and then because of time limitations in steps of 1.6° for Δω. The motor γ was rotated in eleven steps with a step size of 0.0585° in the range of -0.32° to 0.32°. The motor μ was rotated at each γ in steps of 0.0585° and in the range of -0.32° to 0.32° as well. The exposure time for each image was 2 seconds.

After data acquisition the background was subtracted from the raw data and the data was corrected for a varying beam intensity by normalizing to incoming beam intensity. Pixel with extremely high intensity were removed and the intensity for negative pixels and pixels outside of the identified regions containing a diffraction signal (identified by binarizing the image ) were set to zero. Data cleaning was
done with the software package Recon3D [CER17] to minimize the noise contribution and to enhance the diffraction signal intensity, which has still been an issue for the presented experiment. Figure 7.8 gives an example for an image before and after the cleaning procedure. [CER17]

![Image](image.png)

**Figure 7.8.** Image acquired by DFM topo-tomography scan before (left) and after (right) cleaning. The appearance of the diffracted signal is clearly improved after cleaning using Recon3D. Both images are scaled to the max intensity. Reproduced from [CER17].

### 7.1.3 Preliminary Results on cyclically deformed AA1050

#### 7.1.3.1 Sample A – AA1050 after annealing

The cleaned data sets with an optimized signal-to-noise ratio was analysed using Matlab and the program Recon3D, which was developed specifically to analyse DFXRM topo-tomography data. The Recon3D algorithm comprises two steps:

- For a given w-setting the images acquired at various f and ω positions are summed. The result is an approximation of the integrated intensity. Using these “integrated intensity projections” a 3D reconstruction can be obtained by applying a traditional tomography algorithm, e.g. an iterative algebraic solution such as ART. The result will be a 3D map of the shape of the grain.
- Next an optimisation algorithm is applied to determine the most likely orientation for each voxel within the grain interior (with the grain boundary found above). This step cannot be performed by traditional (scalar) tomography approaches, but requires vector tomography – a mathematical field that is much less developed.
Figure 7.9 shows the summed intensity for selected $\omega$ positions. The projections show that the grain outline is easily visible and an increased intensity in certain regions can give indications for the presence of two domains close in orientation within the grain. However, no major substructuring is visible. Those projections can already give a good first impression about the grain shape. Based on the projections the 3D appearance of the grain can be reconstructed. The center of mass is indicated by a red dot in and appears to be slightly misplaced due to a wrong centering during the measurement. For this reasons grain A from sample A was not well suited for a topo-tomography reconstruction. Nevertheless Figure 7.10 shows a preliminary reconstruction of the grain by W. Ludwig before extensive data cleaning as described above was done.

![Figure 7.9: Projections of grain A in sample A after cleaning of data. The center of mass is marked with a red dot (in the bottom right image) and appears to be slightly misplaced indicating a wrong centering of the grain during the measurement. Reproduced from [CER17].](image)
7.1.3.2 Sample B – AA1050 after cyclic deformation

Figure 7.11 is a collection of the projections recorded for grain B at different \( \gamma \) and \( \mu \) positions at a fixed angle \( \omega \). Each of the presented images corresponds to integration over an angular width of 0.0585° in \( \mu \). The shape changes significantly, since the Bragg condition is satisfied for different regions within the selected grain. In this way domains (examples are marked with a red circle) are revealed with a high spatial resolution. Being able to separate domains inside the grains in this way from each other, allows to follow the domain evolution. This demonstrates the potential of the DFXRM approach for this kind of studies.

In Figure 7.11 Figure 2.1 the grain can be observed within a range of approximately six sequential images (in \( \mu \) and as well in \( \gamma \)), where each of the images has an angular width of 0.0585°. This gives an approximation for the angular spread present in the sample of 0.35°. The total angular spread is much larger for the sample due to mechanical deformation. This way of illustrating the acquisition sequence points out, that DFXRM can provide a clear estimation about the spread in orientation for the different regions within the grain.

Figure 7.12 shows selected projections of the integrated intensity - integrated over all associated \( \phi \) and \( \chi \) cleaned images - for selected \( \omega \) position. These integrated projections give an impression of the grain shape of grain B. It is visible that the center of mass is more central for this grain than observed for grain A in sample A, the reconstruction work was therefore improved mainly based on this grain B.
Unfortunately, at some projections a second grain with coinciding diffraction conditions was observed. Figure 7.13 shows some projections, where both grain B and this neighbouring grain are visible. This overlap deteriorates the reconstruction.

Figure 7.11: A set of images acquired at a fixed $\omega$ angle and in a grid of tilt position for varying $\gamma$ and $\mu$ with a step size (and with this angular width) of $\Delta\gamma = \Delta\mu = 0.0585^\circ$. Reproduced from [CER17].
Figure 7.12: Example of the appearance of grain B in sample B in selected projections after cleaning. The center of mass is marked with a red dot (in the bottom right image) and appears to be in the center of the grain. The data set was therefore better suited for 3D reconstruction. Reproduced from [CER17].

Figure 7.13: Example of a neighbouring grain a (note, that it is different from grain A in sample A) passing by grain B visible at selected projections. Reproduced from [CER17].

Nevertheless a first low-quality 3D reconstruction was done using the Recon3D software. The result is shown in Figure 7.14. The reconstruction also shows that the outer surface corresponding to the grain boundary appears in a wavy manner partly caused by the data treatment, where the diffracted signal has to be separated from the surround matrix.
Figure 7.14: 3D reconstructions of Grain B in Sample B done by Recon3d. Reproduced from [CER17].

Finally the Recon3D software failed to provide a reliable mosaicity maps. Additional work performed by colleagues Alberto Cereser and supervisor Henning Friis Poulsen pointed to issues with interpolation as being at least one of the culprits. Simulations gave evidence to the importance of knowing the angular (or reciprocal space) resolution function well. Unfortunately this function was not determined with sufficient care during the beamtime.

7.1.4 Conclusion DFXRM

First results obtained for grains embedded within an undeformed and a cyclically deformed AA1050 needle were presented. The data was acquired in the so-called topo-tomography approach to allow a fast data acquisition and a three-dimensional reconstruction of the grain shape and later its mosaicity (internal orientation variations). In literature already various examples for the successful application of DFXRM on deformed and annealed commercially pure aluminium and individual dislocations are presented. Hence DFXRM is seen as a promising technique to reveal spatially localized phenomena for future investigations being complementary to HRRSM investigations. With DFXRM it will be possible to reveal the grain and subgrain structure of cyclically deformed samples with a high spatial resolution of up to 50 nm (after additional improvements of the optics such as the implementation of multilayer Laue
lenses [MUR19]) and investigate the orientations and stresses on several length scales in three dimensions. The observations can then be related to the phenomena observed in the reciprocal space. Future investigations might include in-situ deformation (as presented for HRRSM) using a load frame suited for the small sample sizes and the limited space available on the goniometer to follow the subgrain structure during cycling and during individual load cycles. As discussed for the HRRSM studies the localization of the grain within the investigated sample and the local neighbourhood can be of high relevance for the internal stresses. A multi-scale characterization of grains can thus be beneficial for relating the local environment to the local mechanical responses. The investigations of a high number of grains and subgrains is important to gain statistical information about the microstructure.
7.2 Annealing studies

The received material was a 90% cold-rolled AA1050 sheet, which required further heat treatment of the sample to obtain an undeformed microstructure. Figure 7.15 shows the grain structure of the cold-rolled material before annealing, where very small grain sizes (ca. 1 µm) and deformation bands from rolling are visible.

![Figure 7.15](image)

Figure 7.15: (a) SEM-BSE image of the grain structure in a 90% cold-rolled AA1050 sample, where small grain sizes are visible in all the investigated region. Some deformation bands are visible and marked with blue arrows. (b) SEM-BSE image of the microstructure showing small grains with sizes of around 1 µm.

For HRRSM experiments the desired grain sizes were 30-50 µm and grains free of deformation structure. The time and temperature for recrystallization of the material were investigated. AA1050 stripes were heat treated for different times at different temperatures in static laboratory air and cooled at air to form defect-free grains.

The annealed specimens were hardness tested with Vickers indent. Figure 7.16 shows the evolution of the hardness of the aluminium samples after isochronal annealing of one hour at different temperatures. In total 5 Hardness indents were made at random locations on the surface of each heat treated sample (without metallographic preparation) using a microhardness tester with 50 g load and Vickers indent. The initial hardness of the cold-rolled hardness is around 43 HV\textsubscript{0.05}. At 250 °C about half of the microstructure has recrystallized. A higher hardness with respect to the finally achieved hardness shows however, that the deformed microstructure is still at least partly present. A fully recrystallized
Figure 7.16: Measurements of the Vickers hardness (using a load of 50 g) of the AA1050 samples after annealing for one hour at different temperatures. Five measurements were done at random locations at the unprepared sample surface.

Figure 7.17: Light Optical Microscopy Image of the annealed grain structure after annealing for 1 hour at 600°C. The sample was etched with 5% Hydrofluoric acid after metallographic preparation of the cross section. It shows a homogeneous heat treatment throughout the cross section. The edges of the sample are just outside of the image.
8 References


9 Acronym List

AI – Aluminium
APS – Advanced Photon Source
BSE – Backscattered Electrons
cECCI – controlled Electron Channeling Contrast Imaging
Cu – Copper
DFXM – Dark Field X-Ray Microscopy
EBSD – Electron Backscatter Diffraction
ECCI – Electron Channeling Contrast Imaging
FWHM – Full Width at half of maximum intensity
HRRSM – High Resolution Reciprocal Space Mapping
ND – Normal Direction
Ni – Nickel
PSB – Persistent Slip Bands
RD – Rolling Direction
SE – Secondary Electrons
SEM – Scanning Electron Microscope
TA – Tensile Axis
TD – Transverse Direction
TEM – Transmission Electron Microscope
A major failure reason for structural materials is fatigue-related damage due to repeatedly changing mechanical loads. During cyclic loading, dislocations self-organize into ordered structures, which play a decisive role for the materials lifetime. The synchrotron technique High Resolution Reciprocal Space Mapping using high energy X-rays was successfully applied to characterize these heterogeneous deformation structures evolving during cyclic deformation of commercially pure, polycrystalline aluminium. Insight into the structural reorganization within single grains embedded in the bulk material is gained by in-situ monitoring of the microstructural evolution during individual tension-compression load cycles and after selected numbers of cycles along tension-tension or tension-compression cycling sequences. By High Resolution Reciprocal Space Mapping individual subgrains can be resolved in the bulk of polycrystalline specimens and their fate, their individual orientation and elastic strains, tracked during different loading regimes. With this approach, the evolution of the intragranular structure in selected grains was followed.