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# Diplomarbeit

# Dislocation substructure evolution of an AI-6016-T4 alloy during cold defomation

Ausgeführt zum Zwecke der Erlangung des akademischen Grades eines Diplom-

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# Abstract

Industrial demands on materials are permanently growing. In order to meet the growing demands of strength, ductility and hardness, it is necessary to understand the deformation behavior of materials. The plastic deformation behavior depends on the movement of dislocations. Contingent on the heat treatment and plastic deformation conditions, movement of dislocations is hindered by precipitates, grain boundaries or other particles in the material. This resistance against deformation is essential for the macroscopic deformation behavior.

The aim of this thesis is to report dislocation substructure evolution during cold deformation and deepen the understanding of deformation mechanisms. To achieve this, a solution heat treated, quenched and naturally aged 6016-aluminum-alloy is deformed at room temperature to different final strains by plain strain compression. The microstructural evolution due to cold deformation is observed by high-resolution electron microscopy (TEM and EBSD). The typical inhomogeneous deformation is responsible for the elongated cell band structure within the grains, containing band walls (GNBs) and internal cell walls (IDBs). Due to the coarse grained material more and more high angle boundaries are generated during deformation.

# Kurzfassung

Um den steigenden Anforderungen an Festigkeit, Zähigkeit und Härte, die an heutige Materialien gestellt werden, nachkommen zu können, ist das physikalische Verständnis von plastischer Verformung unumgänglich. Das plastische Verhalten wird dabei durch die Bewegung von Versetzungen gesteuert. Ist diese Bewegung durch Ausscheidungen, Korngrenzen oder im Material gelöste Legierungselemente behindert, so hat dies einen gravierenden Einfluss auf das makroskopische Verformungsverhalten. Diese Einflüsse sind stark von den Wärmebehandlungszuständen und von den Verformungsparametern abhängig.

Das Ziel dieses Projektes, ist der Nachweis einer von Versetzungen gesteuerten Substrukturentwicklung und die Untersuchung des Verformungsverhaltens einer kaltverformten A6016 Aluminiumlegierung, welche vorher lösungsgeglüht, abgeschreckt und bei Raumtemperatur ausgelagert wird. Dabei werden die Legierungsproben bei gleichbleibender Umformrate unterschiedlich stark verformt. Das Verformungsverhalten wird mit Hilfe von hochauflösenden Transmissionselektronenmikroskopen oder mittels EBSD Aufnahmen nachgewiesen und beschrieben. Die Verformung verlief dabei typischerweise inhomogen. Zu Beginn der Verformung bildet sich eine blockartige Struktur im Inneren der Körner. Diese Blöcke sind durch Kleinkorngrenzen (GNBs) voneinander getrennt. Im Inneren dieser Blöcke befinden sich einzelne Zellen mit Zellgrenzen (IDBs). Während zunehmender Verformung werden immer mehr Blöcke mit sehr hohen Korngrenzunterschieden generiert.

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# **1** Introduction

In this day and age, properties of materials are constantly optimized and adapted to the demands of industry. These include mechanical properties, such as yield strength, tensile strength, elongation and fatigue strength, as well as chemical properties, for example corrosion resistance, and technological features, like formability and weldability. In order to fulfill the requirements of high strength levels, a metallurgical process, rather known as strain or work hardening, is often applied [1].

Increasing the strength values of a material due to plastic deformation is called strain hardening or work hardening. Plastic deformation performed at room temperature or at temperatures below the recrystallization temperature is termed cold working or cold rolling and it is sometimes economical and cheaper than hot working, particularly if close tolerances and good surfaces are required. This deformation leads to a macroscopic change of shape of the material on the one hand, and a special arrangement of the components within the material in the other hand. [2] In the material science such an arrangement is rather known as microstructure and might evolve different for each material. The basic components responsible for this microstructural evolution are the defects within the material, in particular the dislocations. [1]

In order to be able to cope also with future industrial demands, understanding of the mechanisms behind microstructural evolution during cold working is increasingly becoming a vital factor for success. Therefore this work deals with the arrangement and movement of dislocations during deformation at room temperature in order to understand hardening processes. For this purpose a solution heat treated common aluminum alloy of the 6xxx-series, more precisely a 6016-alloy, plastically deformed by plain strain compression to different final strains is investigated in high-resolution microscopy, such as EBSD and TEM, to observe and describe mechanisms and dislocation substructure evolution within the material.

# 2 Objectives

In this thesis a solution heat treated and naturally aged 6061-alloy is investigated. Specimens are deformed plastically with a constant strain rate to final low strains at room temperature. Afterwards cold deformed specimens are examined in highresolution electron microscopy, via TEM and EBSD.

The major objective of this work is to gain an insight in the dislocation substructure evolution during cold deformation. Therefore each state of deformation is observed, described and compared to states of higher or lower deformation. Spacing diameters between high or long angle grain boundaries, cell size and boundary misorientations are the parameters used mostly for the comparison.

An equally important aim is to find and understand the environmental conditions for such an investigation. As an example, what is required to observe cold deformed aluminum alloys by high-resolution microscopy?

A further objective is to obtain an overview of the possibilities and difficulties observing a cold deformed microstructure by EBSD and TEM.

## 3 State of the art

In this chapter an introduction to dislocations and their behavior during plastic deformation is given. Specifically, this section will deal with the dislocation in aluminum and their arrangement and movement during cold deformation. Before talking about dislocations, different types of crystalline defects are described. The class of line defects known as dislocations is assessed in more detail afterwards. Further on the techniques and methods used for the observation of dislocation are discussed. At the end of this chapter the expected dislocation substructure evolution in aluminum alloys during cold deformation is characterized. For a better understanding this will also include a detailed description of this particular type of deformation.

#### **3.1 Crystal Defects**

Many industrial materials, such as metals and non-metallic materials are crystalline. Atoms are arranged in a pattern, which repeats itself periodically in all three dimensions. The specific arrangement can be described by the crystal structure using a space lattice. The space lattice is formed by three set of lines, which divide the lattice into equal sized cells – the unit cells. The solid object formed by stacking equal unite cells next to each other in three dimensions is called crystal. [1]



Figure 1: Space lattice with unit cells [1].

A space lattice is pictured in Figure 1, where *a*, *b* and *c* are the basis vectors of the unit cell and any point, plane or direction can be characterized by reference to this vectors. A unit cell can be described by the three lengths of the basis vectors, the lattice parameters *a*, *b* and *c*, and the angles between them  $\alpha$ ,  $\beta$  and  $\gamma$ . [2] The crystal structure of a metallic or non-metallic material is reliant on the arrangement of the atoms within the unit cell, as already mentioned. Typical crystal structures are the body-centered cubic (bcc), face-centered cubic (fcc) and close-packed hexagonal (hcp), as pictured in Figure 2. In the bcc crystal structure the atoms are placed at the corners and in the center of the unit-cell. Aluminum has a fcc crystal structure, in which the atoms are placed at the corners and in the center of each unit-cell wall. The hcp crystal structure is rather complex and will not be discussed further in the scope of this work. For a better understanding, see [1-3].



Figure 2: Crystal structures: a) bcc, b) fcc and c) hcp. [2]

However, in reality, crystals are not perfect in structure but include defects or imperfections. These defects are classified pursuant to their spatial extension into zero-, one-, two- and three-dimensional defects. Zero-dimensional defects, also known as point defects, are single missing atoms (vacancies) in the crystal, where an atom is removed, or self-interstitial atom, when an atom is placed in a non-lattice site. This is shown in Figure 3. [3]

The defect, which arises if some of the atoms of the crystal lattice are disturbed along a line, is called linear or one-dimensional defects. In scientific terminology these defects are also called dislocations. Dislocations are the main topic of this work and will be explained in more detail afterwards. [2]



Figure 3: Point defects: Vacany (v) and interstitial atom (i). [2]

Two-dimensional defects extend in two directions over a relative large surface. Examples are stacking faults or grain boundaries. A crystal consists of atomic layers, which are stacked up according to an established step. If this stacking sequence is interrupted, this is called stacking fault or planar defect. Two possible types of stacking faults for a face-centered cubic structure are demonstrated in Figure 4 a) and b). An intrinsic fault arises, if an atomic layer is removed, for example de C layer in a), and the stacking sequence continues regularly above or below the missing plane. By inserting an extra plane between a stacking sequence, for example the A plane in b), an extrinsic stacking fault arises. The total energy of a crystal with a perfect atomic layer stacking is lower than the energy of a crystal with stacking faults and the differences between these energies is known as stacking fault energy  $\gamma_{SFE}$ . [3]

Polycrystalline metals are made of a large number of crystals. These crystals are rather known as grains and the areas between the grains are called grain boundaries (see Figure 4 c)). These areas contain a highly strained and distorted crystal lattice. For that reason grain boundaries are regions of high energy. According to the angle of misorientation  $\theta$  between the grains, high angle and low angle boundaries can be distinguished. If the angular misorientation is lower than 15° low angle boundaries are formed. This sort of boundary exerts a much lower resistance again plastic deformation and has fewer defects in contrast to high angle boundaries. The areas surrounded by low angle boundaries are called subgrains. [2]



Figure 4: Two-dimensional defects: a) - b) stacking faults and c) grain boundaries. [2]

There are many other types of two-dimensional defects such as phase boundaries, twin boundaries etc. Furthermore also three-dimensional or volume defects, as precipitates or inclusions, may be present within the crystals. A detailed discussion of all these types of defects is beyond the context of this work. Therefore these defects are not discussed further here (see [1-3] for further reading). However, all these defects are decisive for the properties and the deformation behavior of the material and dependent on the way the material is formed or processed. The most important ones are probably dislocations, because dislocations are the decisive factor for the plastic deformability of the material. This deformability is essential for the industrial use of the material. Therefore the next section will focus in greater detail on this type of defects.

#### 3.2 Dislocations

Dislocations are, as already stated, line defects within the crystal. These defects are essential for the mechanism, which is required for the plastic deformation of a material. If a material is under the influence of shear stress and gets plastically deformed, two lattice planes shift in relation to each other. This is only possible, if the material contains line defects – dislocations. This mechanism is demonstrated in Figure 5.



Figure 5: Plastic deformation by the motion of a dislocation. [3]

The lattice is under the influence of shear stress  $\tau$  and a step on the left side of the lattice is created. The dislocation line is seeded on the line D, where the extra half lattice plane ends. The lattice planes are represented by the vertical lines. Under further stress the dislocation moves through the lattice and leaves it by creating a new step on the right side. The material is sheared along the plane on which the dislocation moved. The vector *b*, rather known as Burgers vector, indicates the shift of the lattice and the intensity of the strain field around the dislocation. The vector is required to complete the Burgers circuit, which is a very helpful definition for a dislocation. [3]



Figure 6: Burgers Circuit a) around an edge dislocation and b) in a perfect crystal. [2]

Figure 6 b) shows the atom to atom path, called Burgers circuit, in a perfect crystal. Drawing the same closed loop in the distorted lattice on the left is only possible with the help of the Burgers vector b. It is the vector, which is needed to close the loop from the end of the Burgers circuit Q to its starting point M. Figure 6 a) also shows an example of an edge dislocation. It is one of the two basic types of a dislocation line and can be displayed by inserting a half extra plane above or below the dislocation line. In this example the dislocation line runs into the image plane and the Burgers vector is normal to the dislocation line, which is typical for an edge dislocation. An edge dislocation always creates a zone of elastic deformation. Above the dislocation line a compressive stress field is created and below the line a field of tension arises. [4] The other type of dislocation line is called screw dislocation. An example of a screw dislocation line is demonstrated in Figure 7. This line defect has its name of the helical surface created by the atomic planes around the screw dislocation line. The Burgers vector is, in contrast to an edge dislocation, parallel to the screw dislocation line and the resulting path of the Burgers circuit looks like a screw, hence its name. [4]



Figure 7: Burgers Circuit a) around a screw dislocation and b) in a perfect crystal. [2]

Nearly all crystals contain dislocations. In the order to measure the amount of such line defects and to compare the quantity with other materials, a unit of measurement is introduced: the dislocation density  $\rho$ . It is the number of dislocations intersecting a unit area  $[m^{-2}]$  or the total length of dislocations lines in a unit volume  $[m/m^3]$ . In general non-metallic or annealed materials have a lower dislocation density than metallic or heavily cold deformed materials. [3]

It has been mentioned already that dislocation movement is essential for plastic deformation. There are two types of movement: glide and climb. Glide is the type of dislocation movement, which occurs in the plane containing both the dislocation line and the Burgers vector. During each glide or slip step, a plane of atoms changes position by sliding or displacement over another plane, rather known as slip plane. The glide or slip of many identical atoms under an applied stress is the typical mechanism of plastic deformation in metals, which has already been shown in Figure 5. As mentioned, shear stress is required for slip and steps on the surface of the crystal are created during slip. This characteristic shear stress  $\tau$ , resolved on the slip plane in slip direction, is:

$$\tau = \frac{\mathrm{F}}{\mathrm{A}} \, \cos \phi \, \cos \lambda$$

#### 3.2-1

where  $F \cos \phi$  is the component of the force in slip direction and  $A/\cos \lambda$  the area of the slip surface. It is also possible that certain crystals between two slip planes may be undistorted, which is pictured in Figure 8 a).



Figure 8: Slip geometry: a) Slip in a cylindrical crystal, b) fcc unit-cell c) fcc slip systems.[2]

Slip or glide is depending on the slip system of the crystal. A slip system consists of a slip plane and a slip direction. The primary slip plane is the plane with the highest density of atoms. The slip direction is the direction in the slip plane corresponding to the shortest lattice translation vectors, usually the direction in which the atoms are closely spaced. In fcc-metals slip often occurs on {111} planes. Four {111} planes exist in a face-centered unit-cell and one of these planes is pictured in Figure 8 c).

Each {111} plane has three (110) slip directions, directions with the shortest lattice vectors. Therefore fcc-metals have twelve slip systems available, which lead to a high capacity for plastic deformation. [2] If dislocations would only move on a single slip plane, obstacles would soon hinder their motion. Therefore, screw dislocations in face-centered metals can switch from one (111) plane to another if it contains the direction of the Burgers vector. This is called cross slip and shown in Figure 9. A screw dislocation line moves in the (111) plane. The (111) plane also contains the direction of the Burgers vector, so if the screw dislocation expands and reaches S in b), the dislocation is able to move in both planes. Double cross slip is shown in d). Cross slip is not possible for edge dislocations, but they often transform into screw dislocation in order to do cross slip. [3]



Figure 9: Cross slip and double cross slip of a screw dislocation. [2]

The other type of dislocation movement, called climb, subsist only at elevated temperature, because it is dependent on diffusion. A dislocation can climb out of its slip plane if a large amount of vacancies is present. During climb vacancies can be created or destroyed. If a dislocation moves from one atomic slip plane to the other, due to the diffusion of individual vacancies or clusters of vacancies and creates steps, than these steps are called jogs. Examples for jogs in edge or screw dislocations are demonstrated in Figure 10 c) and d). Jogs are sources and sinks for vacancies. If steps are created in a dislocation line on the same slip plane, as pictured in a) and b) for edge and screw dislocations, than these steps are called kinks. [2]



Figure 10: Kinks in a a) edge and b) screw dislocation, jogs in a c) edge and d) screw dislocation. [2]

To produce macroscopic slip, large-scale deformation, a high amount of dislocations must be present in the material. Since this is not always the case, it is possible for dislocations to multiply and to act more effective on deformation. One of these possibilities is rather known as Frank-Read spiral mechanism and it says that if a dislocation line is pinned at its ends, it will bow out under the influence of shear stress. This continuous further until a circular loop is created and close itself at the ends. This process is repeated over and over, because the pinned dislocation line inside of the loop starts again to bow out due to the shear stress and another circular loop is created.

Dislocations may also be influenced by other dislocations present in the material. They will interact and if they have the Burgers vector of the same sign they will repulse each other. If they have the Burgers vector of the opposite sign they will annihilate each other or if they are not on the same slip plane, they will create a row of vacancies. All these interactions happen because the system will reduce it internal energy. Dislocation may also interact with other defects in the crystal. For example if a dislocation is pinned by an obstacle, than the dislocation may be immobile and it's called a sessile dislocation. A mobile dislocation is called glissile dislocation.

#### 3.3 Methods of Dislocation Observation

Understanding the behavior of dislocations in crystals described in the section above requires observing und studying them in the material. There are many methods of observation and they may be roughly distinguished by the different scale of magnification during the examination. In this chapter three methods of dislocation measurement are described and for nearly all higher resolutions as used in standard light microscopy are required. Those resolutions are obtained by electron microscopy; therefore at first a short overview of the electron microscopy is given.



Figure 11: Two basics operations of TEM imaging systems involving projecting (a) the diffraction pattern and (b) the image on to the viewing screen [2].

#### **3.3.1 Electron microscopy**

The transmission electron microscope (TEM) was developed for imaging higher resolutions than in light microscopy techniques. Visible light, which is used in light microscopes, has a limited resolution imposed by the wavelength of the light. The small de Broglie wavelength of electron waves utilized in TEMs does not have such a limit and fulfill the geometrical conditions of Bragg's law [5]. A beam of electrons emerges and is transmitted through a thin specimen, as seen in Figure 11. This specimen is situated in a vacuum and has to be stable during high voltage electron beam. Electrons interact with the crystalline specimen by passing through it. Due to the interaction electron beams are diffracted on parallel crystal planes in different directions (Figure 12) and focused in the back focal plane of the objective lens. In the figure above two different application methods are shown. Image b) shows the imaging mode, where the beam projects an image of a magnification in the nanometer range. Image a) pictures the diffraction mode, which is used to analyze defects, noticed in the imaging mode. Furthermore bright- or dark-field images can be pictured. Bright-field images result by a transmitted beam through the specimen. By selecting a diffracted beam a dark-field image is displayed [7].



The increase of the yield and tensile strength depends primarily on fine scaled features in the material. Age hardening precipitates, dislocations and other crystal defects pertain to these features and can only be studied by using transmission electron microscopy or other high resolution techniques. The visible contrast in TEM images is caused by differences in the diffraction and absorption of the electron beam. These contrast differences arise due to variations in orientation of individual grain boundaries or lattice defects, see Figure 12. Strain fields are responsible for it. The decisive factor for strain fields are dislocations and precipitates. Also analysis of the chemical composition of very small features is possible by using an energydispersive x-ray detector [5].

Transmission electron microscopy is surely one of the methods with the biggest magnification and, therefore, the most accurate to investigated dislocation in deformed materials. However, it is only possible to investigate a very small sample area, which is disadvantageous to obtain a representative picture of the material to be examined.

#### 3.3.2 Stress Pulse Double Etching Technique

The stress pulse double etching technique is used for the determination of the stress dependence of the dislocation velocity. Therefore at first, the specimen is annealed at high temperatures for a long time to decrease the dislocation density. Then a fresh dislocation is introduced and the starting position of the dislocation is marked by chemical or electrochemical etching. In Figure 13 etch pits are demonstrated. By applying a stress pulse the dislocation leaves the starting etch pit. Due to a second etching the final position of the dislocation is marked. The starting etch pit does not grow in depth due to second etching (left and central pit) and the final etch pit is created in the end position of the stress pulse and the distance between start and end point. However, this technique only works in materials with a very low dislocation density; because a high dislocation density may cause an overlapping of etch pits. [3]



Figure 13: Selective etching of a dislocation moved in two steps from left to right. [3]

#### 3.3.3 Electron backscatter diffraction

A further method to study defects, grain structures and orientation of the crystals within the material is the electron backscatter diffraction technique (EBSD). Inside the scanning electron microscope (SEM) [8] accelerated electrons are focused onto the surface of the crystalline material. Atomic layers in crystalline materials scatter the electron beam of the SEM. The scattered electron beam satisfying the Bragg's law produces visible lines by impingement on a phosphor screen. These lines are called electron backscatter patterns, rather known as Kikuchi patterns. Kikuchi patterns give therefore direct information of the geometry of the lattice planes in the specimen. For this reason crystallographic properties of the material can be analyzed.

As seen below the specimen is placed in SEM. The specimen is inclined at an angle of 70° towards the CCD camera. The electron beam hits the specimen and the diffraction pattern on the phosphor screen is collected and processed digitally. This is done to identify the single Kikuchi lines and further on to analyze the crystallographic structure, orientation and phases [8].



Figure 14: Schematic diagram of a typical EBSD installation [9].

The resolution of EBSD can be in micrometer range and a wider area, in contrast to TEM analyses, is accessible. Therefore analyses of very small microstructural features are more representative. EBSD is also a very fast and reliable method for quantitative metallography. Particularly in regard of automated EBSD a high number of grains can be analyzed in hours. The results of EBSD investigations are microstructural maps, which contain a high amount of further details about the microstructure within the material [9]. Using several tools within the EBSD-map evaluating software, information about the crystal orientation of the grains, the grain size, the grain boundary misorientation as well as subgrain size and boundaries can be calculated. A map, which describes the crystal orientation of the investigated area, is rather known as orientation map. The crystal orientation is represented by a specific color. This means that every grain or subgrain in the map has its own crystal orientation and, therefore, its own color. This is visible in Figure 15, where an orientation map of a hot deformed aluminum specimen is shown. Normally an inverse pole figure, as visible in the bottom-right of Figure 15, is used to plot the orientation of a given specimen direction, typically the sample normal, with respect to the crystal axes. The white lines in the map characterize high distorted areas between two grains with different crystal orientations, rather known as boundaries. These areas contain dislocations and other defects as described in chapter 3.1. Therefore, they have a higher misorientation angle than the black lines, which indicate subgrain boundaries. [9]

EBSD analysis does not reach the high resolution as achieved in TEM investigations, but the results may be more representative because of the much larger examined area. This is advantageous, especially if the material contains large grains.



Figure 15: Orientation map of a investigated hot rolled aluminum specimen [10].

## 3.4 Dislocations during cold deformation of Aluminum-Alloys

The previous chapter briefly describes dislocation behavior in general. However, dislocation behavior during plastic deformation is different for several materials. Since this thesis reports on the dislocation evolution in a deformed aluminum alloy, this chapter, therefore, describes the arrangement and movement of dislocations in these alloys. Furthermore dislocations act different if deformation is performed at elevated temperatures or at low temperatures. Since this thesis reports on cold deformation, the interaction and accumulation of dislocations during cold deformation of aluminum alloys is stated as well. First an introduction of the investigated sort of aluminum alloy is given and its production process is described briefly. Afterwards the researched and expected dislocation evolution is explained in detail.

#### 3.4.1 Aluminum Alloys

Aluminum alloys are created by alloying aluminum with other metals. In this way, required mechanical properties or values can be reached. The most significant alloying elements are magnesium, silicon, copper, manganese and tin. The principal classifications are casting alloys and wrought alloys, further subdivided into heat-treatable and non-heat-treatable alloys. In this work a heat-treatable wrought alloy of the Aluminum – Magnesium - Silicon alloys is used, which are also known as Al-6xxx series. By the existence of Magnesium and Silicon, this Aluminum alloy has an excellent precipitation-hardening capability. This leads to higher strength values as non-heat-treatable alloys and a good corrosion resistance [11].

As discussed in the introduction, the mechanical properties of the AI-6xxx series represent a good compromise for a wide range of applications. These properties depend on various influencing factors along a complex processing chain. The processing chain is shown in Figure 16. The alloy is inhomogeneous after casting and further processing like hot rolling is only possible if a homogenization step is done between. During hot rolling the material gets his final thickness. Often this step is followed by cold rolling to get better strength values. The elements of the alloy must

be solved in the matrix to allow further precipitation hardening. This is done by solution heat-treatment and rapid quenching. The mentioned steps are explained in more detail in the following literature [11-15].



Figure 16: Industrial processing chain of AI-Mg-Si-alloys based on a quantitative temperature – time diagram.

#### 3.4.2 Work hardening

Increasing the strength values of a material due to deformation is called strain hardening or work hardening. If this plastic deformation, which leads to a change of shape, is done at room temperature or at temperatures below the recrystallization temperature, then it is termed cold working or cold rolling. During cold working interaction and accumulation of dislocations within the crystal structure occurs. Thereby more and more obstacles for the dislocation movement are created and act against further deformation. This results in better mechanical properties, like an increase of the yield strength of the material. Basically deforming a metal means a macroscopic shape change. This shape change has different impacts on the microstructure of the metal. On the one hand the grains change their shape in a way correlated to the macroscopic change. In case of rolling the surface area of a grain increases during the reduction. This leads to a continuously creation of a new grain boundary area during deformation. Responsible for this creation are dislocations, which develop during the deformation process [15].



Figure 17: Rate of grain boundary growth for different deforming types [15].

The total grain boundary area of the system increases therefore and this rate of increase is strongly dependent on the way of the deformation. Figure 17 shows the rate of increase of grain boundary area per volume unit  $S_V$  for different deforming types adopting an initial grain size  $D_0$  as a function of the true strain  $\varepsilon$ . The grains change their shape depending on the deformation type. By compression grains become disc shaped, by rolling grains become laths and by wire drawing grains become needles [15]. On the other hand several structures are formed within and between the grains created by boundaries during the deformation of a metal. These boundaries result due to the accumulation of dislocations. In cubic metals sub-structures are formed by two different deformation mechanisms, slip or twinning. Polycrystalline metals with high stacking fault energy  $\gamma_{SFE}$  like aluminum alloys deform by slip and deformation usually takes place heterogeneous. The high stacking fault energy is also responsible for the decrease of strength values due to the recovery after cold deformation. Low stacking fault energy leads to a better separation of unit dislocations into partial dislocation and this prevents climbing and cross slip of dislocations, which is responsible for recovery. Slip in cold deformation of aluminum occurs on the activated {111} <110> slip systems, as already stated in chapter 3.2. Slipping might occur different in several grains of the material and this may lead to a microstructural inhomogenity, because regions with different orientation are formed [16]. The microstructural hierarchy of the substructure deforming by slip is shown in Figure 18.



Figure 18: Microstructure deforming by slip in a polycrystalline metal. [15]

In low deformed materials dislocations (a) appear in accidental structures or appear as tangles. Predicted by Taylor the activation of five slip systems in one grain would be energetically favorable, but during cold deformation within one grain several slip systems might be active. This leads to a subdivision of the structure within the grain. Depending on material and deformation temperature, subgrains or cells are formed out of dislocation boundaries (b). Even if the slip pattern of one subdivided fragment is different to the other, all together meet the Taylor requirement of strain adjustment. Cells are typical for cold deformed aluminum. Normally they organize them in a structure of elongated blocks a little more than one cell in width, rather known as cell bands [15]. This is shown in the TEM micrograph (a) of Figure 19. The exerted strain within the cell bands is approximately equidistant, but unlike between them due to the difference of the slip activity between the cell bands. The thicker boundaries, which bundle up the cells into elongated cell bands, have a higher misorientation angle than the boundaries within the cell bands. In b) of Figure 19 the misorientation angle of these boundaries is shown for 10 % cold rolled aluminum and these boundaries have been identified as dense dislocation walls (DDW's) or cell band walls. In this work the designation geometrically necessary boundaries (GNB's) of [17] is adopted, due to the fact that they result because of different strain levels between the cell blocks. The cell boundaries within the cell blocks are termed as incidental dislocation boundaries (IDB's), which are the thinner lines in b) of Figure 19. They result by random trapping of dislocation and the size, form and misorientation do not significantly change during deformation [17]. The misorientation angle of the IDB's in Figure 19 is about 0.5 - 1 degree and the cell width about  $0.5 - 1 \mu m$ .



Figure 19: a) cell block structure in 10 % cold rolled aluminum, b) sketch of GNB's (thick lines) and IDB's [11.]

If deformation continues another sort of aligned boundaries might appear in the microstructure. These aligned boundaries are designated as microshear bands. Microshear bands are features, which occur due to the deformation, like GNB's. Therefore these features are called persistent features of the microstructure. The fact that IDB's do not basically change during deformation, is the reason why they are termed as transient features of the microstructure. An example of a microshear band (S) is shown in Figure 20, which shows a TEM micrograph and an EBSD map of the microstructure of a 50 % cold rolled AI-0.1%Mg alloy.



Figure 20: a) Microshear bands (S-S) in 50 % cold rolled AI-0.1%Mg (TEM micrograph), b) EBSD map with cell bands (C) and microshear bands (S) [15].

The boundaries of these persistent features resulting during compression are tilted at an angel of 25 - 40 degrees to the rolling direction. The angle decreases on further compression. Normally one set of bands is formed, but in areas of high stresses it might happen that two sets are formed during deformation [15]. Relating on Figure 18 large areas with significantly different orientations might be formed in grains during cold deformation and are termed as deformation bands (c). In Figure 21 a grain (A) of an Al-1%Mg alloy is pictured and severed by deformation bands (B). Activation of various slip systems in the grain during deformation is one reason for the occurrence of deformation bands. The other reason is that the stress in the grain varies and some regions withstand higher stresses. Deformation bands mostly occur in materials with a coarse grain size. [15] Shear bands (Figure 18 d) in contrast to deformation bands pass through several grains and are formed due to the plastic instability of the material [15].



Figure 21: Deformation bands (B) in an Al-1%Mg alloy grain (A) [15].

Overall, one can say that all these mechanisms, resulting due to the accumulation and generation of dislocations, lead to a change of the stored energy within the material, but this change implies the strengthening that results during deformation [16]. Basically a system always tries to minimize its energy, thus either by organizing itself in a structure, as described previously, or by dislocation annihilation due to dynamic recovery [16].

To describe the microstructural evolution during cold deformation parameters like the cell size, rather known as boundary spacing, the misorientation angle of boundaries and the dislocation density are particularly suitable. The cell size is the spacing between the boundaries. The development of cell size during deformation is plotted in Figure 22 a). It can be seen that the cell size  $\delta$ , normalized to the cell size at a strain of 1.5  $\delta_{\varepsilon=1.5}$ , is decreasing by increasing strain and the inverse cell size (dashed line) increases linearly at a strain of 1 [16]. The misorientation angle  $\theta$  of the boundaries, reported in Figure 22 b) by several authors, however increases during deformation.



Figure 22: a) Evolution of cell size over the strain, b) Evolution in subgrain misorientation. [16]

In case of a microstructure subdivided by GNB's and IDB's the evolution of the cell size or the spacing between the IDB's and GNB's also decreases during cold deformation. In Figure 23 the evolution of boundary spacing and misorientation angle is plotted for an Al-1%Mg alloy is shown. The misorientation angle of IDB's increases more slowly than the angle of GNB's and stabilizes at a misorientation of 3°. The misorientation angle GNB's at high strains changes into a high angle misorientation and no stabilization is observed.



Figure 23: Evolution of boundary spacing and misorientation angles in aluminum for a) IDB's and b) GNB's [16].



Figure 24: Total dislocation density  $\rho_{tot}$  in deformed copper, (b) dislocation density stored within the cell and subgrains  $\rho_i$  [15].

As already mentioned during deformation a change in the dislocation density of the system occurs. This total dislocation density  $\rho_{tot}$  can further be divided into a dislocation density stored within the cell and subgrains  $\rho_i$  and in the cell or subgrain boundaries  $\rho_b$ . The increase of  $\rho_{tot}$  for copper during deformation is demonstrated in Figure 24 (a) and it is visible that after a strain of 0.5  $\rho_{tot}$  increases linearly with the strain. Dislocation density in aluminum within cell and subgrains does not strongly change after a greater deformation than 0.2 and is pictured in Figure 24 (b) [15].

This increase of strength may be demonstrated in a stress – strain curve, rather known as flow curve. In Figure 25 a qualitative example of a flow curve for different temperatures is given. Observation of the flow curves indicates that the stress level at any strain value is higher for T1 compared to the other because the deformation temperature of T1 is lower than the temperature of T2 or T3. Therefore, it can be said the lower the temperature, the higher the applied stress within the material. For polycrystalline FCC metals the flow-curve at low temperature might be divided into several deformation stages. To show the different stages of work hardening a strain hardening vs. flow stress curve, also known as Kocks - Mecking diagram is pictured underneath. In Stage II linear work hardening occurs and is sparsely dependent on temperature and strain rate, which is the change in strain with respect to time. Stage

III and IV are temperature dependent, whereby in Stage III parabolic hardening with a decreasing strain hardening rate occurs. The effect of strain hardening increases by decreasing deformation temperature (T1<T2<T3). Stage IV is similar to stage II, but linearly dependent on the flow curve. The differences between each stage are rather complex and are more accurate explained in [18].



Figure 25: a) Qualitative stress – strain curve at different temperatures, b) strain hardening vs. stress curve of fcc metals [18].

# **4 Experimental**

In this work an industrial AA6016 wrought alloy in the T4 temper conditions is examined. In the following chapter the composition of the alloy and the aging treatment to reach the T4 conditions are described. After the heat treatment cold deformation test are carried out and shortly pictured in this section. Subsequently specimens are prepared to investigate the deformed samples by means of TEM and EBSD. At the end the utilized equipment for the microstructure measurement is listed.

#### 4.1 Sample Production

According to the EN 573-3:2009 norm [19], the AA6016 wrought alloy used in this investigation has the chemical composition in weight percent as listed in Table 1.

AI	Mg	Si	Fe	Ti	Cu	Mn	Zn	Cr	Residuals	
96.4-98.8	0.25-0.6	1-1.5	0-0.5	0-0.15	0-0.2	0-0.2	0-0.2	0-0.1	0-0.15	

Table 1: Chemical composition of AA6016 in wt. %. [19]

The industrial casted and homogenized material has been hot rolled into 60 mm thick sheets. Afterwards 20 specimens are cut out along the Normal Direction (ND), perpendicular to the Rolling Direction (RD). Figure 26 shows the hot rolled 300x300x60 mm AA6016 plate and the rolling as well as the sample extraction direction (ND). The cylindrical samples with the diameter of 10 mm have a final length of 15 mm.



Figure 26: AA6016 plate and sample dimension.

#### 4.2 T4 Temper

To realize the T4 temper condition, solution heat treatment, quenching and naturally aging to a substantially stable condition has to be done [11]. The samples are solution heat treated by 560°C for 1 hour in a high temperature chamber furnace (HTF 1700). Solution heat treatment is performed to dissolve the atoms and disperse them finely in the aluminum matrix. Subsequently rapid water quenching to room temperature ensures that the atoms stay dissolved in the matrix and build a super saturated solution. The specimens are than naturally aged for 14 days by room temperature. In this period the alloy hasn't reached its maximum strength values but it is enough time to complete most of the natural aging mechanisms. [20-21]

#### 4.3 Deformation Experiments

The cold deformation experiments are performed on the *Gleeble 1500*, a servo hydraulic mechanical strain/compression testing machine. The schematic structure of the testing machine is shown in Figure 27 a), where a specimen (2) is fixed between two compression stamps (1-3). The deformation force *F* acting on the stamps deforms the specimen, with the initial length  $h_0$  and diameter  $d_0$ . To prevent a non-uniaxial compression test due to the friction force  $F_R$ , 0.1 mm thin molybdenum foils are applied between the tungsten stamps and the specimen as lubrication. This guaranties no barreling of the specimen, as displayed Figure 27 b).



Figure 27: a) Schematic Structure of the compressing machine [22]. b) Influence of Iubrication during deformation [23].

The strain rate  $\dot{\varphi}$  affects the flow stress and the time- and temperature depending recovery and recrystallization processes during deformation. Therefore it is necessary to ensure a constant deformation rate for comparing the results of the experiments. In case of a constant  $\dot{\varphi}$ , strain  $\varphi$  increases linearly with time *t*. This correlation is explained in equation 4.3-1, where  $h_0$  is the length of the specimen at the beginning and h(t) the length during deformation.

$$\dot{\varphi} = \frac{d\varphi}{dt} = \frac{1}{h(t)} \frac{dh_0}{dt}$$

4.3-1

To measure the microstructural development during cold deformation of the alloy, tests are carried out by a constant strain rate of 0.01. The tests are performed for 4 different final strains, as seen in Figure 28. This figure shows a qualitative stress-strain curve. The strain is pictured on the horizontal and the stress on the vertical axis. All experiments are performed three times.



Figure 28: Quantitative stress- strain curve, numbers show experiments.

The "U" specimen is investigated in TEM and EBSD without deformation, directly after solution heat treatment, quenching and natural aging for 14 days. In Table 2 the designation of the samples, their heat treatment condition and the realized strains during cold deformation are listed.
Sample-Nr.	Temper-Condition	Final strain		
U	Τ4	-		
1	Τ4	0.15		
2	Τ4	0.25		
3	Τ4	0.4		
4	T4	0.55		

Table 2: Schedule of samples, temper-condition and realized strains.

The data recorded by the *Gleeble 1500* are evaluated using *Matlab*, a mathematical software package. For a better understanding of deformation experiments utilizing the *Gleeble 1500* and evaluating data with the help of *Matlab*, see [23-24].

Roughly summarized, there are 15 samples in T4 temper state, 12 of them are deformed and three of them are not deformed. The remaining five samples would have been used in case of unexpected results.

## 4.4 Microstructure investigations

In chapter 3.3 examples of methods to investigated dislocations in aluminum are listed and described. Since it is expected that the dislocation density increases during plastic deformation, the stress pulse double etching technique, therefore, would not lead to meaningful results, as described in chapter 3.3. Because it is possible that the industrially produced sheet material contains large grains, EBSD is used to investigate a larger and more representative sample area as well as TEM investigations are done to get a high resolvedly picture of the examined material.

The quality of the results of TEM and EBSD investigations strongly depend on the specimen preparation. Due to the tremendous importance of this step and great difficulties involved in it, this section reports the way to realize optimal preparation.

#### 4.4.1 TEM

The deformed samples are cut in half, as pictured in Figure 29 (a) (red plane shows cutting plane), and a 1 mm thin sheet is cut-off carefully 8 (b), to not bring more dislocations and defects into it. This sheet is grinded to a thickness of 0.1 mm (c). An abrasive paper with a grit of 2000 is used. Foils with 3 mm in diameter are stamped out of this thin disk (d).



Figure 29: TEM-Foil preparation.

Foils are than electro-polished in *Struers TenuPol-5* to a final thickness of 3  $\mu$ m. A mixture of methanol and nitric acid is utilized as electrolyte (Ratio 2:1). The parameter settings are as follows:

- Voltage: ~ 9V,
- Current: ~ 90 mA,
- Temperature: ~-20°C,

TEM investigations are carried out using the following equipment:

- FEI Tecnai F20 TEM,
- Energy-dispersive x-ray (EDX) device,
- High-angle annular dark field (HAADF),
- Scanning transmission electron microscope (STEM) detector,
- Evaluation Software: TEAM EDS Analysis System for TEM.

# 4.4.2 EBSD

The one half of the cut-off specimen not used for TEM investigations is utilized for EBSD analysis, as shown in Figure 30 (a) and (b) (red plane shows cutting plane). The specimen is cold mounted in a conductive mounting material *CEM 3070*, as displayed in c). In order to ensure good adhesion of the mounting resin to the specimen material it is essential to degrease the sample with alcohol and to dry it thoroughly and carefully with a blow dryer. During cold mounting the specimen is cooled by water too. This is done to prevent any kind of annealing during mounting.



Figure 30: EBSD-Sample cutting and mounting.

The *Struers TegraForce-5* grinding and polishing machine is used to prepare the specimen for the EBSD analysis. The polish and grinding steps are listed in the following table.

Step	Polishing Disc	Grit [µm]	Time [min]	
Grinding	Abrasive Paper	500	2	
Polishing	Struers MD Largo	9	15	
Polishing	Struers MD Dur	6	25	
Polishing	Struers MD Mol	3	30	
OPS	Struers MD-Chem	1	15	
Cleaning	Struers MD-Chem	1	3	

 Table 3: Grinding and polishing steps for EBSD.

Between each step the specimen and sample holder are cleaned in an ultrasonic bath. For *OPS* a *Colloidal Silica 50nm alkaline* suspension is used (1:1 thinned with water). Directly after the *OPS*-step the specimens are polished with the same polishing disc but instead of using the suspension only ethanol, water and standard liquid soap are utilized. Afterwards the specimens are cleaned in an ultrasonic ethanol bath. At the end the specimens are dried with a blow dryer. Between the OPS, the cleaning and the drying step draining of the specimen surface is prevented.

EBSD investigations are carried out using the following equipment:

- FEI Quanta 200 FEG,
- Energy dispersive X-ray analysis (EDAX Pegasus XM4),
- OIM System with DigiView III EBSD Camera and integrated Forward Scatter Detector,
- Evaluation Software: EDAX Orientation Imaging Microscopy (OIM) Data Analysis.

# **5** Results

In this section the results of the experiments described in chapter 4 are analyzed. First the relevant results of the deformation tests are given. Afterwards the results of TEM and EBSD investigations are summarized.

# 5.1 Deformation Experiments

In Figure 31 the flow curves of four compression experiments are plotted. Each flow curve in the Figure is representative for the other two experiments performed to achieve this deformation state, because the realized strain and stress level for each state have nearly the same value. Even the slope of the flow curves has been approximately equal for each state. However, these four deformed samples, with their flow curve as seen in Figure 31, have been chosen for TEM and EBSD investigations, as well as one of the non-deformed samples with a T4 temper state. It can be seen that achieved final strain of 0.15 in sample 1 is located in the parabolic area, stage III, of the stress – strain curve. The reached strain of 0.25 in the sample 2 is located at the transition area between parabolic and linear hardening (Stage III – IV), as well as the final strains of 0.4 (sample 3) and 0.5 (sample 4). Furthermore it is visible that the yield strength  $R_e$  of the alloy reaches a value of ~135 [MPa]. The respective outputs of the experiments are separately listed in Table 4.

Sample	Strain	Stress [MPa]	Stage
1	0.15	288	=
2	0.25	321	III-IV
3	0.4	381	III-IV
4	0.55	402	III-IV

Table 4: Output values of compression experiments.



Figure 31: Stress-strain diagram of the experiments.

# 5.2 **TEM** investigations

In this section the results obtained by TEM investigations are presented. Investigations of the TEM-foils cut out in the TD-RD plane, as described in chapter 4.4.1, are carried out via TEM-bright field and dark field imaging, as well as EDX analysis to examine the chemical composition of the investigated particles. At first TEM foils of the non-deformed specimen and afterwards of the deformed specimen have been examined.

### 5.2.1 Non-deformed State

In the non-deformed sample dislocations have been found. Their arrangement is accidentally, but it is visible that the dislocation density is fairly high. If the investigated area is tilted about a small angle, the arrangement of the dislocations as well as the density changes completely.



Figure 32: Accidental dislocation structure in the non-deformed sample.

Moreover, two particles are found and analyzed in Figure 33. The particles have a size of about 100 nm. By analyzing them via EDX within the marked spots, pictured on the left side of the Figure, the chemical composition has been detected. On the right side the spectrum of the elements is plotted. The upper diagram shows the spectrum of spot 2 and the lower diagram the spectrum of spot 1. A high aluminum, as well as smaller silicon, iron and manganese peak is detected. This indicates that the investigated particles are AIFeMnSi-dispersoids.



Figure 33: TEM-image and EDX analysis of the non-deformed sample containing two AIFeMnSi-dispersoids.

#### 5.2.2 Deformed State

In the specimen deformed up to a strain of 0.55 an elongated cell band substructure has been documented via TEM. An example of this sighted substructure is pictured in Figure 34. This specific dislocation arrangement was found in all TEM-foils of this deformation state. However, it was not possible to find grain boundaries, which leads to the assumption that the grains in the investigated specimen are very large. To obtain a representative statement for the investigated material, therefore a detailed examination of these cell bands has not been carried out via TEM but via EBSD, as described in the following chapter. Also investigations of the lower de-

formed samples have not been carried out because these would not lead to a representative statement for the investigated material due to large grains.

EDX analysis has also been carried out in this sample and only dispersoids, containing elements as manganese, iron, copper and silicon, are detected. The results are listed in the appendix.



Figure 34: TEM-image of the sample deformed up to a strain of 0.55. Elongated cell bands separated by cell band boundaries are visible.

## 5.3 EBSD investigations

In this section characterization of the non-deformed and deformed microstructure for each strain state by EBSD is presented. EBSD observations are made on the longitudinal (TD-RD) plane, as described in chapter 4.4.2. For each of the four deformed samples an area of 500 x 500 pixels has been scanned during the investigation. Afterwards orientation mapping involving automatic beam scanning is used with a step sizes of  $0.5 - 1 \mu m$ . The pattern solving efficiencies is around 95 % for low strain compression and 90 % for higher strain. The way of mapping orientations to colors is based on inverse pole figures. The orientation map assigns a color to each direction of the fundamental sector of the inverse pole figure, as described in chapter 3.3.3. In this thesis the inverse pole figure is always pictured on the bottom right corner of the EBSD maps and the directions plotted are the stereographic projection of crystal directions parallel to the normal direction (ND or [001]).

In the orientation map of each deformation state four grains have been investigated in detail. At first the average inclination of the elongated cell bands to the rolling direction for each deformation state has been measured. The angle between the direction of the intersection of a grain due to a cell band and the rolling direction was measured for five cell bands per grain and then the average was calculated. The rolling direction (RD) is plotted in the upper right corner of the orientation map. Afterwards for each grain the misorientation within three different elongated cell bands, as well as the spacing between each misorientation change, has been measured. Misorientation change occurs if the crystallographic orientation of a measured pixel is different from the orientation of the adjacent pixel. Misorientation measurement has been realized to obtain the average misorientation of the cell boundaries (IDBs) and the average cell size. Furthermore, the misorientation perpendicular to the cell bands across the grain has been measured in three different positions, as well as the spacing between the misorientation changes. This has been done to obtain the average cell band boundary misorientation (GNB) and the average spacing between the boundaries. For this only misorientation angles higher than the average IDB misorientation angle have been used.

The position and misorientation of the dislocation boundaries in the following EBSD observations are determined by the linear intercept method. Applying this method the user defines a misorientation limit (for example 15°) and a dislocation boundary appears on the EBSD map, when a pixel has a greater misorientation than the limit. Later on dislocation boundaries of low misorientation angles may be reconstructed. This is realized with the methods described by F.J. Humphrey [15, 25, 26 and 27] and P. Hurley [26-27].

# 5.3.1 Non-deformed state

The microstructure of the non-deformed sample (U) is plotted in Figure 35. In this state no measurement within the grains has been performed. The orientation map confirms the assumption that the microstructure is very coarse grained, showing grains thicker than 400  $\mu$ m and longer than 800  $\mu$ m. The black lines indicate grain boundaries with a misorientation of 15° and higher.



Figure 35: Backscattered image showing the microstructure of the non-deformed specimen.

#### **5.3.2 Deformed state:** $\varphi = 0.15$

As seen in Figure 36 the microstructure (grain boundaries > 15°) of the low deformed sample is still coarse – grained. In most of the grains set of parallel bands (A-A) are observed after a compression up to a true strain of 0.15. This is visible due to the small variation in color between each band within the grain. A small variation in color means a small crystallographic orientation change because of dislocations or other defects. These parallel set of bands are inclined to the rolling direction (RD) by an average angle of 31°. The line A-A shows the direction of such an elongated cell band.



Figure 36: Backscattered image showing the microstructure of the specimen compressed up to a true strain of 0.15. A-A shows the direction of the grain intersecting bands. B shows the enlarged area pictured in Figure 37.

Figure 37 shows the enlargement of the map detail B pictured in Figure 36. Within this enlarged grain similar sets of bands are observed with different misorientations, recognizable by the change of the colors in the orientation map. Line C marks an example of a misorientation measurement perpendicular to the cell bands across the grain and line D displays a misorientation measurement within the cell band.



Figure 37: EBSD map of parallel set of bands in grain. Thick black lines show boundaries with a misorientation >15°, thin lines >2°.

In the lower right an enlargement of these cell bands is plotted by using methods described in the introduction of this chapter. It is visible that the grain is made up with a structure consisting of elongated blocks of cells bounded by dense dislocation walls (thick black lines). Between these cells, divided by internal boundaries, an area of a relatively low internal dislocation density is visible, It can be assumed, that these dense dislocations walls, rather known as geometrical necessary boundaries (GNBs), are of the same type as described in section 3.4.2. The boundaries of the cells within the bands are known as incidental dislocation boundaries (IDBs).By plotting the misorientation as a function of the distance along the line C and D, a certain repetition of the misorientation change is visible in Figure 38. Each peak in the Figure marks a boundary. High misorientation peaks indicate the dense dislocation walls of these set of bands (GNBs). The result of the measurement for GNBs is a mean boundary misorientation angle of approximately 5.9° and the spacing between the boundaries is approximately 5.5 µm. Low misorientation peaks indicate boundaries within the bands (IDBs). It is visible that the misorientation change is small but the peaks indicate that there are dislocation boundaries and relative dislocation-free zones within the bands. The mean boundary misorientation angle of these IDBs is around  $0.9^{\circ}$  and the spacing between them around  $3.6 \,\mu$ m.



Figure 38: Misorientation measurement of the sample 1.

#### **5.3.3 Deformed state:** $\varphi = 0.25$

In Figure 39 the still very coarse - grained microstructure (grain boundaries > 15°) of the compressed specimen is pictured. The elongated cell bands (A-A), which are now inclined to the RD in an average angle of approximately 29°, are still visible but some of them are intersected by secondary more diffuse bands (B-B).



Figure 39: EBSD-map of sample 2. A-A shows the direction of the set of bands. B-B shows the direction of the more diffuse set of secondary bands. Detail C and D is pictured in Figure 41.

The misorientation perpendicular to the cell bands across the grain is plotted in Figure 40. The peaks show the boundaries of the elongated cell bands. The valley area shows the distance between them. The same holds true for the misorientation of the cell interior. The mean misorientation of the elongated cell band walls (GNBs) and the cell internal boundaries (IDBs) as well as the spacing between each cell band wall and between each internal cell are calculated like in the analysis of the lower deformed sample. The results for GNBs are a mean misorientation of approximately 7.1° and a spacing of 4.8  $\mu$ m, for IDBs 1° and 3  $\mu$ m.



Figure 40: Misorientation measurement of the sample 2.

As discussed above in the display detail C blurred bands, which shear the elongated cell bands, are visible. This display detail is pictured in Figure 41. In the figure the direction of the elongated cell bands and of the microshear bands, intersecting the cell bands, is demonstrated. Microshear band misorientation measurement is done along line B repeated for five different bands within this grain, resulting in a mean misorientation of approximately  $4.7^{\circ}$  and a boundary spacing of approximately  $6.6 \,\mu$ m.



Figure 41: Detail C: Microshear bands in deformed specimen. Detail D: Misorientation measured along E. Black lines indicate boundaries higher than 15°.

In some grains such parallel sets of elongated cell bands visible at a lower strain have been replaced by thicker deformation bands with boundaries of high misorientation angles. Normally, as the name suggests, such high angle boundaries (HABs) or high angle grain boundaries (HAGBs) appear as boundaries of grains, but in this state they also occur within the grain or in the near of "usual" grain boundaries. As an example, this is visible in the detail D. Inclination to the RD as well as misorientation measurement has been done for HAB under the same conditions as for IDBs and GNBs. These bands are inclined in an average angle of approximately 25° to the RD. Within the grain only low misorientation angles have been measured in several directions. The amount is in the range of misorientation angles measured for IDBs. The mean misorientation of HABs is around 20° and the spacing between them is approximately 8.1  $\mu$ m. This is shown in Figure 42.



Figure 42: Misorientation measurement along the line E of detail D.

#### **5.3.4 Deformed state:** $\varphi = 0.4$

The microstructure of the sample deformed with a strain of 0.4 is pictured in the backscattered image above. The grains are more elongated compared to lower strain deformation (grain boundaries >  $15^{\circ}$ ). In this state nearly all grains are intersected by two different set of bands. It is not comprehensible which bands are the elongated cell bands appearing at a strain of 0.15 and which one are the secondary bands developing at a strain of 0.25.



Figure 43: EBSD-map of sample 3. A-A and B-B shows the direction of the grain intersecting bands. Detail C is pictured in Figure 45.

Misorientation measurement along A-A and B-B and repeated under the same conditions as described in the introduction of this chapter leads to a mean boundary misorientation of 1.7° and a mean boundary spacing of 2.8  $\mu$ m. This is shown in Figure 44.



Figure 44: Misorientation measurement of sample 3.

Generally higher and lower deformed areas can be distinguished as well as a larger amount of HABs than at lower strains. In the higher deformed areas grains seem like merging together or breaking up at their boundaries. An example of a high deformed area intersected by bands with high angle boundaries is shown in C) and magnified in Figure 45. Between these boundaries the occurrence of a cell- or subgrain structure can be distinguished. This substructure is displayed by using the methods described in the introduction of this chapter and combining it with an inverse pole figure orientation map. The thin black lines indicate low angle boundaries with a misorientation higher than 1°. Within this substructure areas of a lower misorientation is visible, which indicates low internal dislocation density.



Figure 45: Detail C: High deformed area of sample 3.

Misorientation measurement is done along line D and repeated in three different places within this area, resulting in a mean misorientation of approximately 21° and a boundary spacing of approximately 8.2  $\mu$ m. Measurement along the line E has been repeated in four different bands of this area, resulting in a mean misorientation of 1.4° and a spacing of 3  $\mu$ m. These values are approximately the same as measured within the grain intersecting bands above.

The high angle misorientation bands and cell bands are inclined in an angle of 27° to the rolling direction. This is measured under the same conditions as stated in the introduction of this chapter.

#### **5.3.5 Deformed state:** $\varphi = 0.55$

The microstructure of the sample deformed with a strain of 0.55 is pictured in Figure 46. Grains are elongated parallel to the rolling directions (grain boundaries > 15°). Some grains contain bands with low angle boundaries and some bands with high angle boundaries. The observed bands, whether bands with high or low angle boundaries, are inclined to the RD in an angle of approximately 19.5° (measured under the same conditions as described in the introduction of this chapter).



Figure 46: EBSD-map of sample 4. A shows set of bands within the grain. B shows grain breaking up.

Figure 47 shows the detail A of the figure above. Parallel set of bands intersecting each other may be observed. Misorientation measurement has been realized under the same conditions as described in the introduction of this chapter and the angles of boundaries are in both directions (C and D) approximately  $3^{\circ}$  and the spacing between the boundaries about 2.7  $\mu$ m. The misorientation change along the line C and D is plotted in Figure 48.



Figure 47: Detail A: parallel set of bands in a grain of sample 4.





In general it can be said that high stressed areas can be observed nearly everywhere in picture, indicated by the color gradient and by the high amount of high angle grain boundaries compared to the amount measured at lower strains. In Figure 49 a high stressed area within a grain is plotted. The results are thick deformation bands nearly parallel to the rolling plane. The misorientation along the line E is plotted in Figure 50. Misorientation measurement has been done under the same conditions as stated in the introduction of this chapter, which leads to a mean misorientation of 40° and a spacing of 4  $\mu$ m of the HAB of this deformation state.



Figure 49: Detail B: high stressed area with HAB.



Figure 50: Misorientation measurement in a high stressed area of the sample 4.

# 6 Discussion

In this section the results of chapter 5 are analyzed. Initially the temper state is discussed and afterwards the results of the deformation experiments, divided into a low deformation part, from 0 up to strain of 0.25, and a high deformation part (0.25 to 0.5), are investigated.

## 6.1 T4 temper state

This work is focused on the microstructural evolution of an AI-6xxx alloy during cold deformation. To be sure to discuss only the effects of cold working and not the effects of precipitation hardening a temper state with a stable amount of precipitations is selected. This is realized by choosing the T4 temper state, which is composed out of a solution heat treatment, rapid water quenching and natural aging. It should be noted that even in this temper state precipitation appear in the form of Mg- and Si clusters, co-clusters and finely dispersed precipitates, rather known as dispersoids,. Clusters and co-clusters are hardly visible in TEM investigations and surely affect the microstructural evolution by acting as weak obstacles for the dislocation movement [1-3]. Dispersoids are examined in TEM investigations, as shown in Figure 33, Figure 57, Figure 58 and Figure 59. All examined dispersoids have a size of approximately 100 – 200 nm in diameter and contain traces of manganese, iron, silicon and copper, which are the accompanying elements of this alloy. These dispersoids are formed during the casting and homogenization process and do not dissolve during the solution heat treatment. The occurrence of dispersoids and their size in diameter is typical for the 6xxx alloy series and comparable with other literature [13-14].

### 6.2 Deformation Experiments

Basically it can be said that the shape of the resulting stress – strain curves are in good connection with the shape of the flow curves plotted in Figure 25 a). A distinct parabolic hardening area is visible at the beginning of the resulting flow curves until to a strain level of between 0.1 and 0.3. The increase of the flow stress by increasing strain in in the linear hardening area (Stage IV) is very typical for a low deformation temperature and this is also in good connection with Figure 25 a). It is hard to say where stage III ends and stage IV starts, but what can be said is that the measured final strains are all in the area of stage III or in the transition area of stage IV [31]. The stress values of the experiments are not readily comparable with other experiments in literature. One reason may be measurement and evaluation inaccuracy. The elastic deformation of the deformation testing machine "Gleeble 1500" during the experiments has been taken into account but may be responsible for the minor deviations between the stress – strain curves of the experiments.

Remarkable is the fact that the sample material is very coarse grained, which is visible in Figure 35. Coarse grain size is not uncommon in industrial sheet material, due to high temperature gradients [12] but in literature, dealing with microstructure development of cold deformed aluminum, samples with a finer grain size are examined [15, 16, 17, 26, 27 and 29]. Therefore, comparisons between results of this work and results described in literature are only possible until a certain point of deformation. An important remedy against grain coarsening would be the addition of grain refining elements such as titanium during casting. Also annealing at a higher temperature to recrystallize the grain size would be a possibility.

A further point for discussion is the investigation made via transmissions electron microscopy. Dislocations in accidental structures or appearing as tangles have been documented successful in Figure 32, which shows a TEM-micrograph of the non-deformed specimen. These results compare well with those reported in Figure 18. Further on, at the different stages of deformation substructure evolution described in chapter 3.4.2 has been proven as well. The elongated cell block structure recorded in Figure 34 is similar to that substructure evolution pictured in Figure 19. These structures have not been further investigated via TEM due to the fact very large

grains within the material (see Figure 35). Using the small area of 4  $\mu$ m<sup>2</sup> examined via TEM compared to an average grain size of 600  $\mu$ m to make accurate statements about the material would not have been representative enough. Therefore, the specimens have been further investigated via EBSD. As already mentioned investigations to ensure the correct performance of the solution heat treatment, quenching and natural aging have been successful due to the identification of a precipitation-free aluminum matrix, except of dispersoids (see Figure 33).

In the following section the results of the experiments are separated into a low strain and higher strain deformation part. The reason for this separation is the fact that the results of lower strain deformation compare approximately with those reported in other literature [15, 16, 17, 26, 27 and 29] in contrast to the results of higher strain deformation.

#### 6.2.1 Low Strain Deformation $0 \le \phi \le 0.25$

Parallel bands are observed in Figure 36, showing the coarse grained microstructure of the  $\varphi = 0.15$  deformed sample. This cell band structure is in good comprehension with the substructure recorded in the small area investigated via TEM in Figure 34 and described in chapter 3.4.2. F.J. Humphreys and P.J. Hurley measured the orientation of these grain-intersecting set of bands to the rolling direction during cold rolling of a single-phase Al–0.1 Mg alloy for different strains and found out that the orientation to the RD decreases with increasing deformation [32]. A comparison between the average orientations measured by F.J. Humphreys and P.J. Hurley and the results of the experiments is set out in Figure 51. A more detailed listing is plotted in Table 6 in the appendix.



Figure 51: Comparison between the orientations of cell bands to RD measured in the experiments and by F.J. Humphreys and P.J. Hurley [32].

The slope of both functions is in good accordance until a strain of 0.4. After this strain the slope of the function of the experiment results decreases faster than the other. Possible reasons for this phenomenon are stated in section 6.2.2. The change in orientation of the parallel bands to the rolling direction, with increasing strain, indicates that these dislocation walls are dynamic or transient features of the

microstructure. Dislocations are produced due to crystallographic slip during deformation and dynamic recovery organizes them into boundaries. These boundaries or band walls remain active and are continuous disbanded, reformed and reorganized. Reorganization always follows the geometry of deformation. The mechanisms behind this permanent reorganization is discussed by several authors [15, 16, 17, 26, 27 and 29], but are still largely not understood.

Returning to Figure 51, remarkable is the deviations from approximately 6° between the two data lines. This may have several reasons: F.J. Humphreys and P.J. Hurley investigated an alloy of the 5xxx-series with a much smaller grain size of 82 µm in contrast to the investigated 6016 alloy with a grain size of approximately 700 µm. Other reasons may be different settings during the EBSD investigations, like smaller step sizes in a range of 0.10 - 0.15 µm and higher pattern solving efficiencies. A representative statement via EBSD can only by given by investigating an area containing several grains. Due to the large grain sizes in these experiments, this was only possible by investigating a huge area of more than 1000 x 1000 µm. Using a step size of 0.1 - 0.15 µm would lead to a very long investigation time, which has not been feasible in this work. A good compromise between step size, investigation time and resolution was made, by choosing a step size of 0.5 - 1 µm. Higher pattern solving efficiencies may also be achieved by reducing the step size and by a better sample preparation.

This microstructural transformation into a pancake-like cell band structure is very common for the stage III of work hardening, where the deformation becomes more and more inhomogeneous [31]. These parallel bands are cell blocks bounded by dislocation boundaries. Neighboring cell blocks may deform by different amount of shear or may use varying slip systems for the deformation, therefore these boundaries, separating the neighboring cell blocks, contain the lattice misorientation between them and are called geometrical necessary boundaries (GNBs) [25, 32]. As already mentioned in chapter 3.4.2, also within the cell blocks, cells are bounded by boundaries. The formation of these boundaries occurs due to statistical trapping of glide dislocations [16, 29]. Therefore they are called incidental dislocation boundaries (IDBs). Both types of boundaries were found in EBSD investigations of lower strain deformation and are equivalent with those described in chapter 3.4.2. It has

also been possible to reconstruct them with the methods described by F.J. Humphrey [15, 25, 26 and 27] and P. Hurley [26- 27]. To compare the obtained results of both types of boundaries with literature, mean misorientation and boundary spacing of GNBs as well as mean boundary misorientation and cell size diameter of IDBs were measured during EBSD investigations. The experimental results for IDBs, plotted in Figure 52 and listed in the appendix, and the results for GNBs is listed in Table 5.



Figure 52: Misorientation and cell size of IDBs vs. strain.

Strain	Misorientation [°]	Spacing [µm]		
0.15	5.945082	5.5		
0.25	7.125544	4.8		

Table 5: Misorientation and spacing of GNBs over the strain.

Figure 52 demonstrates that the misorientation of the cell boundaries increases slowly and the cell size decreases slowly with increasing strain. This is in good agreement with the experimental results of Q. Liu and N. Hansen [29], pictured in Figure 53. Also the increasing misorientation and the decreasing spacing of the GNBs with increasing strain are in good agreement with the literature. The misorientation of GNBs is higher and grows faster compared to IDBs. According to the re-

sults of Q. Liu and N. Hansen [29], these investigations indicate that major part of the dislocations formed during deformation is stored in GNBs. In the experiments of F.J. Humphreys and P.J. Hurley [32] misorientation and spacing remains relatively constant with increasing strain, listed in Figure 54. It is visible that the spacing between GNBs in the results of F.J. Humphreys and P.J. Hurley [32] is only one or two cells thick. This is in good agreement with the experimental results of this work.



Figure 53: Misorientation and spacing of GNBs and IDBs for high-purity aluminum with a average grain size of about 300 µm [29].

True strain	Cell band walls		Cell band interior		Microshear band walls		Microshear band interior	
	deg.	μm	deg.	μm	deg.	μm	deg.	μm
0.2	5.2	1.6	1.2	1.2	_	_	_	_
0.7	5.2	1.1	1.2	0.9	4.2	4.5	1.9	0.8
1.4	5.3	0.9	1.1	0.7	5.0	3.5	1.6	0.7
1.8	5.6	0.8	1.4	0.7	18.7	3.2	1.6	0.7
2.3	5.7	0.7	1.4	0.7	53.5	1.6	2.1	0.7

Figure 54: Mean misorientation angle and boundary spacing data for different boundary types each reduction measured by F.J. Humphreys and P.J. Hurley [32].

In the  $\varphi = 0.25$  deformation state even microshear bands with a mean misorientation of approximately 4.7° and a boundary spacing of about 6.6 µm have been sighted. In F.J. Humphreys and P.J. Hurley [32] investigations such microshear bands do not occur until a strain of 0.7. GNB-measurement was only possible up to a strain of 0.25 due to the reason described in the following section. Differences between experimental and reported values may be arisen due to the different investigated aluminum alloys. As mentioned, F.J. Humphreys and P.J. Hurley [32] observed an alloy of the 5xxx-series; Q. Liu and N. Hansen [29] investigated high-purity aluminum and both had a smaller grain size than the material used in this thesis. Q. Liu and N. Hansen [29] observed a large amount of boundaries by using TEM, which is more accurate but more complex and time-consuming in contrast to EBSD. As already stated above, F.J. Humphreys and P.J. Hurley [32] applied step size were smaller and more precise, than the step size applied in this work.

#### 6.2.2 Higher Strain Deformation $0.25 \le \phi \le 0.55$

In general, deformability of the material has been proven due to the experiments. The reason for this is the dislocation substructure evolution, which is taking place during deformation. At low deformation an elongated band structure has been formed to ensure the deformability of the material. During further deformation the elongated band structure has been intersected more and more by secondary more diffuse bands, called microshear bands (see Figure 41). Such an intersection of secondary bands is in good accordance with experiments reported in other literature [15, 16, 17, 25, 26, 27 and 29] and described in chapter 3.4.2. The origin of these bands is not clear, but according to D. A. Hughes and N. Hansen [17], they arise due to the disability of the alloy to endure further work hardening. After a higher deformation (0.25  $\leq \phi \leq$  0.55) it is no more possible to distinguish cell bands and microshear bands. In most of the grains cell bands are completely intersected by microshear bands and boundary observation of these bands leaded to misorientation and spacing diameter comparatively small as the values measured for IDBs. Other grains are more and more intersected by bands of high angle boundaries, visible in Figure 45. These high angle boundaries seem to be subject to the same laws as GNBs measured at lower deformations. Figure 55 shows the misorientation and spacing diameter change over the strain of these high angle boundary bands (see also appendix). As can be seen, the misorientation of this sort of bands increases with increasing strain, especially after a strain of  $\varphi = 0.4$ . The spacing diameter follows the converse behavior.



Figure 55: Misorientation and spacing of high angle boundary bands vs. strain.

The origins of this behavior at such low strains as reported in this work are unclear. In literature [32] this behavior occurs sometimes in very fine grained material at very high strains ( $\phi \ge 2$ ), where the boundary misorientation of GNBs increases over 15°. It seems that grains containing high angle boundary bands are breaking up and endure more strain and deformation as grains without these bands. They may accommodate the lattice misorientation between the neighboring bands. Due to the large change in misorientation and spacing during deformation they may accommodate more dislocations than IDBs. This may indicate that these sorts of boundaries are dynamic or transient features of the microstructure and undergo the same mechanisms as GNBs. For that reason it may be assumed that high angle boundaries within grains at these strains occur due to the large grain size.


Figure 56: Total length of high angle boundaries per map area over the strain.

Noticeable is also, that the amount of these bands increases significantly with strain, especially after a strain of 0.25.as shown in Figure 56 (see also appendix). This might lead to the conclusion that more and more dislocations are getting stored in these high angle boundaries and therefore, the further applied strain will almost exclusively been absorbed within these boundaries and this will lead to an additional increase of the total high angle boundary length.

### 7 Summary and Conclusion

An Al-6016-alloy was cold deformed by plain strain compression up to different strains and investigated via TEM and EBSD. Accidental dislocation arrangement in the non-deformed state and dislocation substructure evolution during cold deformation as described in previous works [15, 16, 17, 25, 26, 27, 29 and 32] has been documented successfully. Correlations between the different states of deformations have been recognized. As a basic principle, it can be maintained that the microstructure is organized by an elongated band structure, bounded by cell band walls (GNBs) and containing cells with internal cell walls (IDBs). Both boundaries increase their misorientation and decrease their spacing between each other with increasing strain. GNBs increase their misorientation faster than IDBs. This is in good accordance with results in literature and indicates that both types of boundaries are dynamic or transient features of the microstructure. During higher deformation more and more bands with high angle misorientation were generated due to a very large grain size. The high angle boundaries undergo the same mechanisms as low angle boundaries. Unfortunately comparison of this sort of results with conclusions reached in the scientific literature was difficult. The selection of a finer grained material would be advantageous for a better comparison of higher strain experiments with other reports. Previous works [15, 16, 17, 25, 26, 27, 29 and 32] have shown that EBSD and TEM lead to exceptional results for the investigation of cold rolled microstructure, especially in combination. This combination has been essential in this work as well. Accidental dislocations structures for the non-deformed state and cell block structures for the deformed state have been reported with TEM. A more accurate evaluation of the dislocation substructure evolution during cold deformation has been performed via EBSD. However, there were great difficulties in sample preparation, especially for EBSD investigations. Even if preparation methods have been constantly improved with time, for example by hydrofluoric acid (HF) etching, a good preparation quality was rarely achieved. The only remedy may be the use of an ion mill/slicer.

All in all it can be said that microstructural investigations of cold deformed material via EBSD and TEM is an excellent method, if certain framework conditions are respected.

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### 11 Appendix

### Nomenclature

γ\_\_\_\_\_shear strain

 $\gamma_{SFE}$ \_\_\_\_\_stacking fault energy [J/m<sup>2</sup>]

Γ\_\_\_\_\_line tension of a dislocation [J/m]

δ\_\_\_\_\_cell size [m]

 $\delta_{\varepsilon=1.5}$  \_\_\_\_\_\_ cell size at a true strain of 1.5 [m]

ε\_\_\_\_\_engineering strain

θ\_\_\_\_\_misorientation [°]

*θ*\_\_\_\_\_work hardening rate

 $\theta_{av}$ \_\_\_\_misorientation avarage [°]

 $\rho$ \_\_\_\_\_dislocation density [m/m<sup>3</sup>]

 $\rho_b$ \_\_\_\_\_\_dislocation density in cell or subgrain boundaries [m/m<sup>3</sup>]

 $\rho_i$ \_\_\_\_\_\_dislocation density within cells or subgrains [m/m<sup>3</sup>]

 $\rho_{tot}$ \_\_\_\_\_total dislocation density [m/m<sup>3</sup>]

τ\_\_\_\_\_shear stress [N/mm<sup>2</sup>]

 $\dot{\varphi}$ \_\_\_\_\_strain rate [s<sup>-1</sup>]

d<sub>0</sub>\_\_\_\_\_initial sample diameter [m]

*D*<sub>0</sub>\_\_\_\_\_initial grain size [m]

*D*<sub>*av*</sub>\_\_\_\_boundary spacing average [m]

E\_\_\_\_\_stored energy [J/m<sup>2</sup>]

F\_\_\_\_\_deformation force [N]

*F<sub>R</sub>*\_\_\_\_\_friction force [N]

h<sub>0</sub>\_\_\_\_\_initial sample length [m]

h(t)\_\_\_\_\_sample length during deformation [m]

S<sub>v</sub>\_\_\_\_\_unit volume [m<sup>3</sup>]

T\_\_\_\_\_temperature [°]

t\_\_\_\_\_time [s]

DDW	dense dislocation walls
EBSD	electron backscatter diffraction
GNB	_geometrical necessary boundary
HAB	high angle boundary
HAGB	high angle grain boundary
IDB	_incidental dislocation boundary
SEM	_scanning electron microscopy
TEM	_transmission electron microscopy

### **Results TEM:**



Figure 57: TEM-image marking the spots of the deformed sample up to a strain of 0.55.



Lsec: 29.3 0 Cnts 0.000 keV Det: Apollo XLT2 Windowless Det

Figure 58: EDX-analysis of Spot 1 = AIFeMnSi-dispersoid.



sec. 20.3 0 Chts 0.000 kev Det. Apolio XE12 Windowiess Det

Figure 59:EDX-analysis of Spot 2 = AIFeMnSi-dispersoid.

### **Results EBSD:**

Strain	Angle of bands to RD [°]
0.15	31.0
0.25	28.9
0.40	26.9
0.50	19.5

Table 6: Inclination angle of the cell bands to RD.

Strain	Misorientation [°]	Cell size [µm]
0.15	0.95	3.6
0.25	1.03	3.0
0.4	1.65	2.8
0.55	2.96	2.7

Table 7: Misorientation and cell size of IDBs over the strain.

Strain	Misorientation [°]	Spacing [µm]
0.25	19.9	8.1
0.4	21.2	8.2
0.55	39.7	4.0

Table 8: Misorientation and spacing of high angle band boundaries over the strain

Strain	High angle boundary length / map area [µm/µm <sup>2</sup> ]
0.15	0.00307
0.25	0.00837
0.4	0.03750
0.55	0.11065

 Table 9: High angle boundary length per map area over the strain.