

The Effect of Mn and Homogenisation Procedure on Mechanical Properties and Grain Structure in Extruded AA6082

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Abstract

Extruded profiles of AlMgSi-alloys are widely used in various industries such as the automotive industry, aerospace and in the oil and gas industry. To tailor the alloys for specific needs and applications, more information about the behaviour of alloying elements is needed. In this study, extruded profiles of AA6082 have been examined. Seven different alloys with increasing content of Mn have been subjected to three homogenisation procedures. The purpose of the different homogenisation procedures was to obtain different densities of dispersoids, while still maintaining the same level of elements in solid solution. The study have examined the effect of Mn and homogenisation procedure on grain structure, strength and corrosion behaviour.

Results from this study showed that the density of dispersoids greatly affect the final grain structure after extrusion. Homogenisation variants that contained a high density of dispersoids showed low degree of recrystallisation. Increasing amount of Mn also contributed to a lower degree of recrystallisation. In as extruded condition variants with a high density of dipersoids were harder than variants with lower density, despite equal chemistry and elements in solid solution. At peak strength, increased content of Mn led to a reduction in strength.

Sammendrag

Ekstruderte profiler av AlMgSi-legeringer er utbredt i flere sektorer. De er ofte anvendt i bil- og transportindustrien, luftfart og olje- og gassindustrien. For å skreddersy legeringer til spesifikke oppgaver og behov er mer informasjon om legeringselementers påvirkning ønsket. I denne studien er ekstruderte profiler av AA6082 undersøkt. Totalt syv legeringer med økende innhold av Mn har blitt homogenisert på tre ulike måter. Formålet med dette var å oppnå ulik tetthet av dispersoider, men samtidig holde nivået av elementer i fast løsning det samme. Denne oppgaven har undersøkt påvirkningen av Mn og homogeniseringsparametre på kornstruktur, styrke og korrosjonsegenskaper.

Resultatene fra denne studien viste at tettheten av dispersoider påvirker kornstrukturen etter ekstrudering i stor grad. Homogeniseringsvariantene med høy tetthet av dispersoider viste liten grad av rekrystallisering. Økende innhold av Mn bidro også til en redusering av rekrystallisasjon. I som-ekstrudert tilstand viste variantene med høy tetthet av dispersoider seg å ha en noe høyere hardhet enn variantene med lav tetther av dispersoider, på tross av identisk kjemi og andel elementer i fast løsning. Ved utherding til maksimal styrke, førte økende andel Mn til redusert styrke.

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Abbreviations

H2	=	homogenisation procedure H2
H4	=	homogenisation procedure H4
H5	=	homogenisation procedure H5
β'	=	Hardening phase
β''	=	Hardening phase
SSSS	=	Super saturated solid solution
GP-zones	=	Metastable Guinier-Preston zones before the β' and β'' particles.
R_m	=	Tensile strength
$R_p 0.2$	=	Yield strength
HV	=	Vickers hardness
HV1	=	Vickers hardness, load 1 kg
HV5	=	Vickers hardness, load 5 kg
IGC	=	Intergranular corrosion
PC	=	Pitting corrosion
AE	=	As extruded
UA	=	Underaged
T6	=	Solution treated and artificially aged to peak strength
OA	=	Overaged
MJ	=	Mega Joule
kWH	=	kilo watt per hour
wt%	=	Weight percent
P_Z	=	Zener drag
P_D	=	Driving force caused by temperature and force
σ_i	=	Intrinsic contribution from pure aluminium
σ_{ss}	=	Contribution from alloying elements in solid solution
σ_p	=	Contribution from dispersoids and age hardening precipitates
SEM	=	Scanning electron microscope
BSE	=	Back-scatter detector

Chapter]

Introduction

Aluminium is one of the most used and versatile metals in the modern world. With applications spanning from the automotive, marine and aerospace industry to household appliances such as beverage cans and aluminium foil. This metal have a good strength to weigh ratio, great properties at lower temperatures, good formability and welding properties as well as excellent corrosion resistance. In the oil and gas industry, steel is mostly used both topside and subsea. Although aluminium does not have the strength of steel it should be considered as a candidate for some applications, especially where weight and corrosion resistance is of importance. In general, aluminium provides a lower maintenance cost and will also have a lower life cycle cost. In many cases it is also possible to shorten the gap in strength between steel and aluminium with clever design. With 1/3 of the weight of steel, structures of aluminium would be easier, faster and cheaper to hoist if the structure is to be placed on the seabed for subsea production.

Another aspect of aluminium is the outstanding recycling possibilities. To recycle, as little as 5% of the energy originally required for production is needed. In a world of decreasing energy resources as oil, coal and gas, the need for materials for a sustainable future becomes more and more important.

In this study, alloy variants of the 6000-series have been studied. This alloy shows a good combination of strength, formability and resistance to corrosion. It is frequently used in extrusion products and widely used in the transportation industry. In addition it shows good properties at lower temperatures. The objective of the thesis is to study the influence of Mn on extrudability, strength, grain structure and corrosion behaviour of AA6082. The effect of Mn in solid solution and in dispersoids will be given special attention.

Chapter 2

Theory and earlier work

2.1 The aluminium value chain

A principle figure of the aluminium life cycle is given in figure 2.1. The production of aluminium starts with bauxite. This aluminium ore mainly consist of aluminium oxides, iron and silicon. It was named by the french geologist Pierre Berthier who discovered it in 1821 in the village of Les Baux in southern France. The largest and most lucrative bauxite deposits are located around the equator [7]. Major producers include Australia, Brazil, Jamaica and Surinam. In close proximity to the mines, a refinery is often located. Here, the bauxite gets refined to alumina (Al_2O_3) . Large amounts of energy is required to reduce alumina to aluminium, and production plants are often located in countries with a good supply of electric power. Although as much as 47 MJ (approximately 13 kWh) is required for molten electrolysis of 1 kg aluminium, this investment gives excellent dividents [8]. By reducing weight in transportation, fuel consumption is lowered. By reducing weight of heavy structures, lifting procedure time is shortened. By lowering the weight of packaging transportation costs are lowered.

Aluminium has great recycling properties. With correct sorting, scrapped aluminium can in principle be recycled to produce the same products over and over again. This only requires 5% of the original energy input. In other words, aluminium is the perfect eco-metal, and as recycling becomes increasingly important, so does the life cycle of aluminium [8].



Figure 2.1: The aluminium value chain, from alumina to the recycling circle. [9].

2.2 Aluminium alloys

Although shiny and nice to look at, aluminium in its pure form is not very useful for applications where strength is required. There are some products that consists of over 99 % aluminium, such as aluminium foil and conductive products, but in general aluminium is alloyed with other elements to achieve a broad range of properties such as increased strength and toughness, increased corrosion resistance and thermal capabilities. The main alloying element will decide what group of alloys the product belongs to. Table 2.1 shows the main alloying groups, their main alloy elements and main uses.

 Table 2.1: Table showing the aluminium alloying series, their main alloying element and some properties.

Series	Main alloying element(s)	Typical properties
1000	Pure Al, 99.3-99.9 %	Due to high conductivity this is typically used
		in electrical and heat-power industry.
2000	Copper (Cu)	Applications with high requirements to tensile
		strength. This alloy can acheive up to 400 MPa on
		completion of hardening.
3000	Manganese (Mn)	This medium strength alloy have good formability
		and is suitable for surface treatments, but poor for
		welding.
4000	Silicon (Si)	
5000	Magnesium (Mg)	Medium strength and excellent corrosion resitance
		in aggressive atmosphere and seawater.
6000	Mg and Si	Medium strength alloy often used in extrusion and
		is easily anodized.
7000	Zinc (Zn)	Highest strength of all alloy series, but prone to
		stress corrosion, especially when welded.
8000	Other (Fe, Ni, Li)	Alloyed with various elements depending on
		purpose. Examples are lithium for high strength
		and stiffness and iron for increased strength
		but maintaining electrical conductivity.

2.3 AIMgSi-alloys

Extruded profiles accounts for about 50 % of the total aluminium production world wide, and AlMgSi-alloys covers 75 % of these products [11]. Excellent corrosion resistance, formability and ageing properties makes these alloy attractive for the industry for numerous applications in the automotive industry, aerospace, marine industry as well as suitable containers for food and beverages. Some key mechanical properties are given in table 2.2 [33]. In addition to Mg (0.5-1.3 wt%) and Si (0.4-1.4 wt%) Mn, Cr and Cu may also be added. A table showing typical alloying elements and their function is found in table 2.3

Table 2.2: Key mechanical properties for the 6000-series aluminium alloy [33].

$R_{p0,2}$	190-360 MPa
R_m	220-390 MPa
Elongation	12-17 %

Table 2.3: List of typical alloying elements in the 6000-series and their purpose [33].

Elements	Purpose
Si & Mg	Added for increased strength through precipitation hardening
Mn & Cr	Added for grain fining purposes
Cu	Added for additional strength, but can reduce corrosion resistance

2.4 Production

An illustration of the typical process route for wrought alloys of aluminium is presented in figure 2.2a. As seen from the figure, the alloys must undergo several steps before reaching its final condition. The aluminium, along with its alloying elements, are transferred from the molten state to the casting die, which is cooled by water (direct chill method). When cooled, the elements becomes distributed unevenly in the alloy and a process to homogenise the billet is required, this step is further investigated in section 2.5. The billet is then ready for extrusion.

Before extrusion, the billet is preheated. Then, a press mechanism forces the material through a die with the desired shape, usually a plate. The composition of the alloy will influence the force needed to press the bolt through the die, and ultimately the final microstructure of the profile. Alloying elements that results in precipitates that hinder dislocation will cause the force needed to press the bolt through the die to increase[30]. When the force increases, more turbulent conditions arise at the edges of plate. This causes more dislocations and initiation points for grain growth. A principal sketch of the extrusion process is found in figure 2.2 as well as a cross section image of the evolution of the grain structure during extrusion. After extrusion, the profile is cooled, either by air or water, and then cut or pressed into its final shape. The very last step is called ageing and are covered in greater detail section 2.8.



Furu.

Figure 2.2: Figures of the process route (a), principle sketch of an extrusion press (b) and the evolution of grains during extrusion (c).

2.5 Effect of homogenisation

Homogenisation is the high temperature heat treatment process performed after casting. The objective of the process is to homogenise the billet, improving workability, remove micro-segregation as well altering the microstructure. It consist of three steps; heat-up, soaking and cooling. Several parameters will effect the behaviour of the billet during extrusion. This includes holding time, holding temperature and heat/cooling rates. These parameters will also effect the mechanical and other properties of the metal. Figure 2.3 shows the thermal history of extruded aluminium, where homogenisation is the second step in the process.



Figure 2.3: Thermal history of extruded aluminium [30].

During casting intermetallic phases may form at the end of the solidification process by eutectic or peritectic reactions. Due to the low solubility of iron in aluminium, these phases often consist of Fe. In 6000-series alloys, particles consisting of $\beta - AlFeSi$ and $\alpha - AlFeSi$ as well as Mg_2Si and other non-equilibrium phases may form. Many proposed stoichiometric compositions of these phases have been suggested [16, 17, 27, 40]. These particles are often referred to as primary particles. In the 6000-series, the homogenisation is vital both to transform the $\beta - AlFeSi$ consisting particles to $\alpha - AlFeSi$ and to dissolute the Mg_2Si particles [32]. The $\beta - AlFeSi$ -compounds reduces the ductility of the extrusion and transformation to the α -phase is wanted. Studies have claimed that this transformation is the reason for increased ductility during extrusion [26, 41], while other reports suggests that it is rather the improvement in Mg_2Si -phase distribution that is the cause. Although not yet fully understood it has been pointed out that small additions of Mn enhances the $\alpha - \beta$ transformation as well improving the uniformity of the Mg_2Si phase distribution, so it is reasonable to believe that these mechanisms are connected. The α -phase have a diffuse connection to the aluminium-matrix, does not form a continuous network due to shape change and does not melt during extrusion. Also, this phase requires less Si leaving more Si to contribute to strengthening of the alloy [41].

Small particles may form during the homogenisation process. These are often referred to as dispersoids, and will generally have the same composition as the primary particles.

They typically form at temperatures between 400 and 460 °C [23]. The size of the dispersoids are usually less than 0.5 μm . The dispersoids are important for the control of recrystallisation during the thermomechanical processing. Addition of Mn or Cr may increase the formation of dispersoids in the 6000-series alloy [23]. The hardening phase $\beta' - Mg_2Si$ may act as nucleation sites for dispersoids during homogenisation and therefore influencing the distribution of the dispersoids [18].

The holding time for the homogenisation is usually 2-3 hours in the industry. In order to remove micro-segregation the holding time may be longer due to slow diffusion of elements such as Mn. Large billets may also require longer holding time so that the coldest location of the billet receives minimum holding time [32].

When the desired holding time is reached, the billet enters the cooldown-stage. In general it is cooled rapidly, but not too fast as this will raise the flow stress of the material in the further processing. A cooling rate of 300-500 °C/h is considered adequate.

The dispersoids formed during solidification and homogenisation are known to have a complicated influence on the overall recrystallisation characteristics. These small dispersoids can result in a more homogeneous distribution of dislocations, which reduces the number of nucleation sites for recrystallised grains. A high density of dispersoids also reduces the inter-particle-spacing and rearrangement of the sub-grains becomes difficult due to pinning by the disperoids [18, 39].

2.6 The effect of Mn on grain structure after extrusion

Alloying elements and production parameters greatly influence the final grain structure of the alloy. As mentioned, control and knowledge of the grain structure is vital to control the final properties of the product. It is important to understand what parameters that can influence the grain structure as well as what effect the resulting grain structure will have on the end product properties. As this study focuses on a 6082-alloy with increasing amounts of Mn, effect on grain structure will be presented with regards to Mn.

Figure 2.4 shows a side by side comparison of the grain structure of extruded profiles of a 6060-alloy and a 6082-alloy. The 6060-alloy is not alloyed with Mn, whereas a typical 6082- alloy contains 0,5 wt% Mn.

When addressing the important parameters of the grain structure, it is the final grain structure that is considered. That means accounting for the reasons for changes in the grain structure happening during the extrusion process and its origins. One of the important parameters controlling the strength and toughness of the product is the density of grains and the size of these. Small grains will influence the strength mechanical properties of the alloy. Concentration of dislocations can occur on the grain boundaries. With few and large grains, these concentrations will cause high amounts of tension at these spots, leading to a large amount of stress on the given location. Small and more grains reduces this by hindering large constellations of dislocations to be concentrated on the grain boundaries. This contributes to lower the local tension and distribute the concentration of dislocations



Figure 2.4: Microgaph of the grain structure of a 6060-alloy (a) and a 6082-alloy (b) [15]

throughout the alloy. Although small grains are beneficial towards the mechanical properties of the alloy, it is not considered optimal with a fine grain structure throughout the cross section of the alloy. The reason for this is due to anisotropy. This means that the grain displays poorer properties in some stress directions. Typically there exists at least one direction with poorer properties.

During extrusion the degree of recrystallisation is controlled by several factors, such as the laws of recrystallisation [19]:

- A minimum deformation is needed to initiate the recrystallisation.
- The temperature at which recrystallisation occurs decreases as the time of annealing increases.
- The temperature at which recrystallisation occurs decreases as strain increases.
- The recrystallised grain size depends primarily on the amount of deformation, being smaller for large amounts of deformation.
- For a given amount of deformation the recrystallisation temperature will be increased by a larger grain starting size and a higher deformation temperature.

In general, low degree of recrystallisation is sought after. As mentioned in section 2.5 the addition of Mn and certain homogenisation procedures can result in the growth of very small particles, dispersoids. During extrusion and recrystallisation, these particles may hinder recrystallisation by pinning and hindering the growth of grains. Already in 1946, Smith looked at a correlation between size, fraction and driving force for the recrystallisation. This is know as the Zener drag and is given in equation 2.1.

$$P_Z = \alpha \gamma_{GB} \frac{f}{r} \tag{2.1}$$

Giving the total driving pressure for recrystallisation $P = P_D - P_Z$. Where P_D is the driving force for grain growth caused by pressing force and temperature. As seen from the

formula, both the size (r) and the fraction (f) of dispersoids will influence the Zener-drag. α and γ_{GB} are constants [38]. For commercial supersatureted alloys primary particles will give way for particle stimulated nucleation (PSN) while dispersoids will contribute to pinning, as mentioned earlier.

2.7 Strengthening mechanisms for AlMgSi

In alloys where several strengthening mechanisms are operative in room temperature, it is reasonable to assume that the individual strength contributions can be added linearly, as shown in equation 2.2 [29]. Optimal strength are achieved by a combination of these contributions. The mechanisms are described in figure 2.5 and briefly presented in the following.

$$\sigma_y = \sigma_i + \sigma_{ss} + \sigma_p \tag{2.2}$$

The definitions for the parts of the equation are as follows:

Parameter	Definition
σ_i	Intrinsic contribution from pure aluminium
σ_{ss}	Contribution from alloying elements in solid solution
σ_p	Contribution from dispersoids and age hardening precipitates



Figure 2.5: Visual representation of the effects that contributes to strengthening of aluminium, solid solution hardening (A) and precipitation hardening (B). The movement of dislocations can be bypassing larger particles (B) or simply shearing smaller particles (C) [28].



Figure 2.6: Alloying elements in solid solution, substituted into the matrix, causing irregularities in the matrix and hinder the movement of dislocations.

Alloying elements in age hardenable alloys, such as Mn, Mg, Cu and Si, will have a considerable effect of the strength of the alloy, with regards to solid solution strengthening. Strengthening from particles in solid solution occurs when the alloying elements is mixed at an atomic level with the aluminium matrix. Depending on the size of the atoms the alloying elements will be dissolved either by substitutional solution (typical for aluminium) or by interstitial solution (typical for carbon in steel), as shown in figure 2.6. This causes tension in the matrix and creates irregularities in the lattice. As dislocations have trouble moving around these irregularities and therefore require more stress to accomplish this, an increase in strength is achieved.

Dispersoids in the microstructure may also contribute to strengthening of the metal. Proposals for this mechanism are based on slip distribution and grain size [10].

2.8 Precipitation hardening

In its base condition aluminium is relatively soft and weak. In order to increase the strength and durability of the material certain alloying elements can be added. The mechanism that contribute to the strength increase is called precipitation hardening. To achieve this improvement of strength the material must undergo some steps in the process route.

First, the alloy is heated to a temperature above the solvus temperature, but below the equilibrium temperature. A typical temperature for this is 540 °C. The alloy is held at this temperature until the alloying elements are dissolved into solid solution. The material is then cooled, either by water or air. Because of reduction in solubility as the temperature decreases, the aluminium alloy becomes saturated on alloying elements. This is called super saturated solid solution (SSSS). Finally a heat treatment on lower temperature is necessary. This is usually done in a temperature range of 150-200 °C. During this heat treatment metastable phases will precipitate and the material will undergo ageing. Below is the typical precipitation sequence:

 $SSSS \rightarrow solute clusters \rightarrow GP - zones \rightarrow \beta'' \rightarrow \beta' \rightarrow \beta(Mg_2Si)$

The β'' and β' are metastable phases of Al, Mg and Si. Studies shows that alloys that

are artificially aged to peak strength contains a mix of both β'' and β' [33]. A sketch of how ageing time effects strength is presented in figure 2.7.

The peak strength condition is usually the most interesting case for industrial applications. However, if the alloy is held at the ageing temperature for an extensive time the stable equilibrium phase Mg_2Si will form. This will cause the material to loose strength and hardness, and is called overageing.



Figure 2.7: Principle sketch showing how ageing times affects strength. Underageing, T6 and overageing areas are highlighted.

2.9 Corrosion of aluminium

Aluminium in general is considered to have good corrosion resistance, especially for alloys containing Mg. This due to the fact that Mg is included in the oxide layer and increase the protection of the material. However, in certain environments local corrosion attacks can occur. In the following a brief introduction to the corrosion types relevant to this study are presented, namely intergranular corrosion (IGC) and pitting corrosion, as well as their effect on the AlMgSi-alloy.

Studies show that alloys with some degree of copper can be susceptible to localized corrosion attacks. Mondolfo was the first to address this effect [24] and this has also been covered by El-Menshawy [13]. A series of papers released by Svenningsen et al. in 2006 and studies by Hurlen Larsen has also given this topic attention [20–22, 34–37]. This will be further discussed in section 2.9.1 and 2.9.2. In addition to the various effects of alloying elements, the thermomechanical history has an effect on corrosion type and susceptibility.

2.9.1 Intergranular corrosion (IGC)

This form of corrosion is related to potential differences between the ground boundaries adjacent grain bodies. It is is frequently found in 2000-, 5000-, 7000 and in some cases

in the 6000-series [2]. Some precipitates are anodic and some are cathodic, depending on what elements the precipitates consists of, and when these emerge at the grain boundaries during ageing a shift in potential arises. When the element migrates to the grain boundary, the surroundings gets depleted of the element and the local concentration is no longer uniform. This difference in concentration causes a potential difference at a microscopic level. A principle sketch of the mechanism is found in figure 2.8. In their studies, Svenningsen et. al [20–22, 34–37] found that AlMgSi-alloys containing copper tends to form a Cu-rich film along the grain boundaries, which can be the case of the more severe IGC attacks. Reports have shown IGC effects on alloys with Cu content as low as 0.17 wt% [37].



Figure 2.8: Principle sketch showing proposed mechanism for IGC [3].

Cu is not the only alloying element that seems to cause IGC. Especially the ratio between Si and Mg is crucial. If the alloys consist of more Si then is needed to form Mg_2Si studies show increased susceptibility to IGC [22]. In general the Mg/Si-ratio should be in the vicinity of 1.73. Studies have suggested that a Mg/Si-ratio that differ from this may cause IGC [22]. Although, Zhan reported that this effected was of lesser importance compared to the effect from Cu [42].

In addition, the cooling rate during production is also a contributing factor. Svenningsen et al. showed a relationship between cooling rate and IGC. Air cooled samples were more susceptible to IGC than rapidly cooled (quenched) samples, when subjected to IGC testing in as extruded condition [36, 37].

Mondolfo also suggested that Mn can be a reducing factor of IGC caused by hindering precipitation of elements to the grain boundary and thereby inhibiting corrosive conditions [25].

2.9.2 Pitting corrosion

Pitting corrosion is a severe form of corrosion. Small dents and openings in the outer layer of the material can be observed, but underneath a large crevasse can be found. Due to a small ratio of the anodic on the cathodic surface, the corrosion pits can grow quickly causing damage deep into the material. A surface surrounded by water or seawater can be subjected to pitting, but there have been indications that alloys with high content of Cu, such as the 2xxx-series, are more prone to this kind of corrosion [5]. Defects and flaws in the material surface are often initiation points for these kinds of attacks as well as a chlorinated environment, but this mechanism is not yet fully understood [14].

A closer look at the propagation process suggests the overall reaction equation, as shown in 2.3, for pitting corrosion.

$$2Al + 3H_2O + \frac{3}{2}O_2 \to 2Al(OH)_3$$
 (2.3)

The process can be further broken down into two steps. Firstly aluminium is dissolved and the Al^{3+} cations reacts with Cl^- anions to form $AlCl^{-4}$ which then gets hydrolyzed. This will in turn acidify the bottom of the pit due to formation of H^+ . This acid environment is highly aggressive and causes propagation to continue. The Al^{3+} cations concentrated at the bottom of the pit starts to diffuse out of the pit and reacts with hydrogen from either the hydrogen gas evolution or the water reduction and forms aluminium hydroxide which precipitates and deposits at the border of the pit. A principle sketch of the reaction is shown in figure 2.9 and a picture of a surface with deposits around a pit is found in figure 2.10.



Figure 2.9: A principle sketch of the suggested mechanism for pitting corrosion[4].



Figure 2.10: Image showing aluminium hydroxide deposited around a pit caused by pitting corrosion on the surface of an aluminium alloy containing Cu [4].

Previous studies have also showed a relationship between the ageing process and susceptibility to pitting corrosion. Several authors notes that as an alloy enters the overaged con-



Figure 2.11: An example of corrosion types found in various stages of ageing on an solution heat treated and air cooled alloy with 0.17wt% Cu [36].

dition the dominating corrosion mode is pitting corrosion [34–36]. Finally, an overview of what types of corrosion that can be expected in at different ageing times is presented in figure 2.11.

Chapter 3

Materials and experimental procedure

In this study, seven alloy variants of aluminium alloy 6082 have been studied. The content of Mn increases as the alloy sample number increases, from 0.006 wt% to 1.172 wt%. In addition all alloys were homogenised with three different schemes. The chemical composition of each alloy can be found in table 3.1.

Profiles from each billet were extruded at Gløshaugen NTNU and several studies were carried out at NTNUs metallurgical laboratories with the different profiles. This included artificial ageing, optical characterisation, measurements of hardness and electrical conductivity, IGC-testing and particle analysis. The experimental set up will be covered in this section. In addition a simulation with Alstruc was performed.

Prior to the laboratory work, a risk assessment was performed.

 Table 3.1: Chemical composition of the different alloys variants given in wt%.

Alloy	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	Mg/Si
A8	1.01	0.20	0.001	0.006	0.65	0	0.004	0.006	0.65
A9	0.98	0.19	0.001	0.204	0.62	0	0.004	0.006	0.63
A10	0.98	0.20	0.001	0.413	0.61	0	0.004	0.006	0.62
A11	098	0.19	0.001	0.521	0.64	0	0.004	0.006	0.65
A12	0.98	0.20	0.001	0.615	0.65	0	0.004	0.006	0.66
A13	098	0.20	0.001	0.800	0.65	0	0.004	0.006	0.66
A14	0.98	0.20	0.001	1.172	0.64	0	0.004	0.005	0.65
6082	0.8-1.3	0-0.5	0-0.1	0.4-1.0	0.6-1.2	0-0.25	0-0.1	0-0.2	

3.1 Material processing

3.1.1 Casting and homogenisation

The billets were direct chill cast at Hydro Research and Technology Development Center at Sunndalsøra. The billets were produced with a diameter of 95 mm. After casting, the billets were homogenised with three different schemes. A very short holding time (H4), an industry standard time (H2) and a variant with very long holding time (H5). The parameters for the homogenisation are given in tables 3.2, 3.3 and 3.4. The purpose of the homogenisation variant H4 was to encourage the formation of many small dispersoids, while still maintaining the same amount of alloying elements in solid solution as homogenisation variant H5. This was done by measuring the electrical conductivity of the alloy as it was homogenized and compare it to the H5 variant. As a result, the H4 variants required different holding times for the different alloy samples to maintain the corresponding level of elements in solid solution. A visual representation of homogenisation steps H4 and H5 is found in figure 3.1.

Table 3.2: Homogenisation step H4 for alloys A8-A14. Alloys 10 and 11 only required heating to the holding temperature, before reaching the desired level of elements in solid solution. WQ = Water quenched.

Alloy	Holding temp [$^{\circ}C$]	Holding time [min]	Heating rate $[^{\circ}C/h]$	Cooling
A8	555	20	200	WQ
A9	555	40	"	"
A10	555	0	"	"
A11	555	0	"	"
A12	555	15	"	"
A13	555	40	"	"
A14	545	60	200	"

Table 3.3: Homogenisation step H2 for alloys A8-A14

Alloy	Holding temp [$^{\circ}C$]	Holding time [min]	Heating rate $[^{\circ}C/h]$	Cooling
A8-A14	575	135	200	WQ

Table 3.4: Homogenisation step H5 for alloys A8-A14

Alloy	Holding temp [$^{\circ}C$]	Holding time [min]	Heating rate $[^{\circ}C/h]$	Cooling
A8-A14	585	480	200	WQ


Figure 3.1: Visual representation of homogenisation steps H4 and H5.

3.1.2 Extrusion

The samples were extruded at NTNU in Trondheim. The dimensions of the extruded plates were $500 \times 6.5 \times 0.29$ cm. Some extrusion parameters are given in table 3.5. All profiles were water quenched after extrusion. An image of the extrusion press is found in 3.2.

	N	Aax pres	S		Surface	Velocity	
	fo	orce $[kN$	7]	temp	erature	[mm/s]	
Alloy	H4	H2	H5	H4	H2	H5	All
A8	2703	2737	2700	540	537	541	5.2
A9	2939	2837	2703	543	539	547	"
A10	3285	2915	2798	553	544	546	"
A11	3472	2967	2888	552	546	546	"
A12	3369	2989	2897	555	547	548	"
A13	3416	3115	2944	552	548	549	"
A14	3580	3240	3055	561	554	559	"

Table 3.5: Extrusion parameters for all alloys and homogenisation variants.



Figure 3.2: Image showing the extrusion press.

3.2 Artificial ageing

Prior to the artificial ageing, the profiles were cut in samples with dimensions $2 \times 2 \times 0.29$ cm and held in room temperature for 24 hours. For this part of the study, all variations of the alloys were heat treated according to table 3.6, giving a total of 126 samples. These were later tested for hardness. In addition, a set from homogenisation variants H4 and H5 were cut with dimensions $3 \times 3 \times 0.29$ cm from all alloys. These were aged to peak hardness (5h) and later subjected to IGC testing.

Table 3.6: Ageing time and temperature. All samples were treated according to this table.

 Temp
 Ageing time

 185°C
 0 m
 30 m
 2 h
 5 h
 1 d
 1 w

3.3 Optical characterisation

To examine the grain structure of the variants of the alloys, a light microscope was used.

3.3.1 Sample Preparation

Prior to examination, the samples were cut, ground and polished. For grinding, waterproof grinding paper with silicon carbides were used in increasing fineness, from 120 μ m to 2000 μ m. For polishing, napless polishing cloths with 6-1 μ m diamonds and DiaPro Mol suspension was used. Between each step, the samples were washed with water and ethanol. The samples were then anodized. For this step, the a samples were immersed in a solution of 5 % *HBF*₄ and a potential of 20 V was then applied, with the current set to 1 A. The samples were held in the anodizing solution for 90 seconds and then rinsed with water and ethanol.

3.3.2 Optical analysis

After preparation, the samples were then studied in the light microscope at two magnification levels; 2.5x and 5x. To produce distinguishable polarized light were used. To capture the images the ccd-chip in the microscope was used along with an image capturing software. An illustration showing the view plane of for the optical analysis is given in figure 3.3.

3.4 Measurement of hardness

Hardness values were measured using a Struers Duramin A2500. A a load of 5 kg and a load time of 45 seconds was used. Each sample were tested 3 times at different positions of the sample.



Figure 3.3: Sketch showing the view plane of the optical analysis of the grain structure.

3.5 Measurement of electrical conductivity

With a Forester Sigmascope 2.069, the electrical conductivity was measured. The instrument was calibrated before use and the frequency was set to 120 Hz.

3.6 Intergranular corrosion test

The IGC test was performed in accordance with the BS 11846 Method B standard[1]. Homogenisation variants H4 and H5 were chosen for this part. For this part the samples with dimensions 3 x 3 x 0.29 cm were used. Every sample was degreased with acetone and ethanol and then air dried followed by alkaline etching. The etching was performed by dipping the samples in 7.5 % NaOH at $55 - 60^{\circ}$ C. Finally the samples were desmutted in sulfuric acid for about 1 minute.

The accelerated corrosion test was then performed in a solution of 30 g/L NaCl and 10 ml/L 39 % HCl for 24 hours. After the test the samples were washed with water and sulfuric acid and scrubbed with a plastic brush to remove any corrosion product. The samples were also weighted before and after the corrosion test.

To examine the results of the test the light microscopy was used. All samples were cold mounted, ground and polished as mentioned in section 3.3.1. Similar to the characterisation of the grain structure, the plane parallel to the extrusion direction were examined for the samples subjected to the IGC test.

3.7 Particle analysis

To analyse the details of the primary particles and dispersoids found in the alloys, a electron microscope was used in addition to the image analyse software iSolution DT. A stepby-step procedure in described in the following.

3.7.1 Electron microscope and BSE

Prior to examination in the electron microscope, the samples were prepared as described in section 3.3.1, except anodizing. The samples were then examined in the electron microscope with two magnification levels. One magnification level for analysis of the larger primary particles and a one magnification level to extract information about the smaller dispersoids. After focus was adjusted the backscatter electron detector was used to provide images with particles clearly distinguishable from the matrix. An overview of the parameters used is found in table 3.7.

Table 3.7: Table showing parameters used to acquire images for particle analysis

Parameter	Value
Detector	Backscatter, secondary
Acc. voltage	4 kV
Working distance	9 mm
Apperature	$120 \ \mu m$
Magnification	350x/5000x

3.7.2 Particle analysis with iSolution DT

After imaging in the electron microscope the images were analysed in iSolution DT. The levels of contrast, brightness and gamma were adjusted to highlight the particles of interest. Figure 3.4 shows an example of images before and after adjustment. Noise pixels in the size of 1-2 pixels were then removed from the image before finally counting and measurement of size was performed. It is important to note that the definition of a dispersoids in this study is defined as a particle with an equal circle diameter of less than 0.5 μm . By that definition, particles with a size smaller than this were removed from the analysis of primary particle, and particles with a size larger than this were removed from the analysis of dispersoids.



(a) Original image

(b) Adjusted image.

Figure 3.4: Example of image properties adjusted before analysis. In this case the A11-H2 variant at 5000x magnification.

3.7.3 Presentation of anyalysis

The measurement tool in iSolution DT provides a large amount of information concerning particles analysed. These key values were extracted and are presented in section 4.4:

- Particle size distribution
- Average praticle size
- Density
- Area fraction

3.7.4 Alstruc simulation

To simulate casting and homogenisation parameters relevant to this study, the simulation program Alstruc was used. Values for volume fraction, density and particle size were extracted. A.L. Dons describes Alstruc in Journal of Light Metals 1 (2001) 133:

"The Alstruc microstructure simulation code is built on standard solidification theory. The metal solidifies one part at a time with the concentration in the recent layer of solid-state aluminium almost proportional to the concentration in the liquid. The "constants" of proportionality are called distribution coefficients, and the values are found in the phase diagram. The microstructural input parameters are the composition, the dendrite arm spacing and the grain size. The main output parameters are the temperature as a function of the fraction solid, the concentration profile in the solid-state from the dendrite centre to the dendrite boundary, the volume fraction of each type of particle, the temperature interval in which they form, and tentative particle sizes, the temperature-dependent thermal conductivity, density, specific heat and heat of fusion." [12]

When examining the results of the simulation it is very important to remember that the simulation presents results expected before extrusion, while the samples examined in this study have been extruded.



Results

4.1 Grain structure

The alloys were examined in the light microscope to investigate the grain structure. This was performed prior to precipitation hardening and corrosion testing on the as-extruded profiles.

In the following the grain structure of the various samples is presented. Figure 4.4 shows alloys A8-A11 and figure 4.7 shows alloys A12-A14. The difference in grain structure as the content of Mn increases, should be noted. Also interesting is the difference in grain structure with respect to homogenisation temperature and holding time, despite identical chemistry and level of solid solution.

Alloy 8

Alloy 8 has the lowest amount of Mn. The grain structure is fully recrystallised and consists of fairly grains through the entire profile. This structure is somewhat similar in at all homogenisation temperatures, although the H4 variant displays somewhat coarser grains.

Alloy 9

Alloy 9 displays an increase in grain size for all homogenisation temperatures. In addition, the fibrous structure in homogenisation variant H4 now becomes prominent. A recrystallised layer is now visible at the outer edge of the extruded plate, near the surface.

Alloy 10

In alloy 10, the fully recrystallised grain structure observed at homogenisation temperature H2 in alloy 8 and 9, is gone. It is replaced by a fibrous structure with a relatively thick recrystallised layer near the surface containing large grains. For the H4 variant the same conditions are observed, but the recrystallised layer is much smaller. In contradiction, the H5 variant still holds a recrystallised grain structure through the entire profile. However, it is to be noted that the size of the grains have increased closer to the center of the sample.

Alloy 11

Alloy 11 shows further less degree of grains than the previous alloys. Especially the recrystallised layer in variant H4 is now even smaller. The H5 variant now shows a mix of fibrous structure and grains scattered around the center of the sample.



(a) A8-H4

(**b**) A8-H2

(c) A8-H5



(a) A9-H4

(**b**) A9-H2

(c) A9-H5



(a) A10-H4

(b) A10-H2

(c) A10-H5



(a) A11-H4

(**b**) A11-H2

(c) A11-H5

Figure 4.4: Optical micrographs showing the grain structure of alloys A8-A11 for homogenisation variants H4, H2 and H5. Images are taken perpendicular to the extrusion direction with 2.5x magnification.

Alloy 12

Alloy 12 shows similar characteristics as alloy 11. Fibrous structure with a recrystallised layer for the H2 variant, a very thin recrystallised layer in the H4 variant and then a fibrous structure with some grains in the H5 variant. It should be noted that a closer look at the recrystallised layer of the H2 variant reveals size differences in the recrystallised layer, with somewhat smaller grains close to the surface.

Alloy 13

A continuation of the trend with less and less grains and a smaller recrystallised layer is observed for this alloy. The recrystallized layer for both H2 and H4 variants appears smaller than previous samples. For the H5 variant there is still a mixture of large and small grains as well as a fibrous structure.

Alloy 14

The final alloy in the sample series shows a clear fibrous structure in all homogenisation variants. The H2 variant displays the thinnest recrystallised layer of all the H2 variants. Similar conditions are observed for the H4 variant, with the recrystallised layer almost invisible. The H5 variant has made the transition into a completely fibrous structure near the center of the plate with very large grains in a thick recrystallised layer towards the surface.



(a) A12-H4

(**b**) A12-H2

(c) A12-H5



(a) A13-H4

(b) A13-H2

(c) A13-H5



Figure 4.7: Optical micrographs showing the grain structure of alloys A12-A14 for homogenisation variants H4, H2 and H5. Images are taken perpendicular to the extrusion direction with 2.5x magnification.

4.2 Electrical conductivity

Figure 4.8 shows the measured values of electrical conductivity for homogenisation variants H4, H2 and H5. As seen from the figure, alloys with a lower amount of Mn displays higher conductivity throughout all homogenisation variants. The alloy with most Mn, alloy A14, displays the lowest electrical conductivity. A closer look at the evolution reveals difference between the homogenisation variants for each alloy. This is further discussed in section 5. A complete table of values for all alloys and homogenisation variants can be found in the appendix B.



Figure 4.8: Evolution of electrical conductivity plotted with respect to ageing time for homogenisation variants H4, H2 and H5.

4.3 Hardness measurements

Hardness values as a function of artificial ageing times were measured. The peak hardness values are presented in table 4.1 and in figure 4.9 which shows the evolution of hardness as function of ageing times for the respective homogenisation temperatures H4, H2 and H5. In the graphical representation the alloys are grouped with respect to homogenisation temperature.

As seen in table 4.1, the highest values of hardness were measured in alloys A8 through A10, i.e. the alloys with lowest Mn content. The homogenisation scheme seem to influence the hardness values to some extent where the H4 variants has the lowest average value of 100.7 HV5 and the H2 and H5 variants shows averages of 109.4 HV5 and 111.5 HV5 respectively. A more complete picture of the evolution of hardness as function of ageing time can obtained by studying figure 4.9. Although some variations depending on homogenisation procedure, most of the alloys maintain their relative position with respect to each other. Also, some variants (A11-H4, A11-H5, A12-H2, A12-H4, A12-H5) actually showed highest hardness after 2 hours ageing time. A complete chart of all hardness values can be found in appendix A.

Table 4.1: Table showing the peak hardness values [HV5] for all alloy variants. Standard deviation is presented in parenthesis for each value. Values in **bold** reached peak hardness after 2h ageing time.

Alloy	H4 [HV5]	H2 [HV5]	H5 [HV5]
A8	113.0 (3.5)	120.7 (5.0)	116.0 (2.7)
A9	117.3 (2.3)	115.0 (1.0)	117.3 (2.3)
A10	108.3 (2.3)	111.0 (4.6)	119.3 (4.0)
A11	105.7 (4.0)	110.7 (1.2)	115.3 (2.1)
A12	108.3 (2.1)	116.0 (6.5)	116.0 (3.5)
A13	98.7 (1.2)	105.3 (4.0)	105.0 (3.0)
A14	95.0 (1.7)	97.7 (2.9)	99.3 (1.5)



(c) Homogenisation procedure H5



4.4 Particle analysis

Particle size, size distribution, density and area fraction was measured as described in section 3.7. The results are presented graphically to best describe correlations and differences across the alloys and the homogenisation variants. Alloys A9, A11, A12 and A14 were selected for this experiment.

4.4.1 Particle size distribution

The particle size distribution displays the frequency of the equal circle diameter for each particle. Occurrences for each size interval is represented by the density of said size interval. A visual representation of the particles observed can be found in figure 4.11. This shows SEM-images for alloy A14 with homogenisation variant H4 and H5 respectivly. The image shows both the larger primary particles and the smaller dispersoids. Equivalent images for all alloys and homogenisation variants can be found in the appendix C.



(c) H5, primary particles, 350x

(d) H5, dispersoids, 5000x

Figure 4.10: SEM-iamges of alloy A14 in homogenisation variant H4 (a-b) and H5 (c-d). Images taken perpendicular to extrusion direction.

Effect of Mn on primary particles

The particle size distribution for primary particles with respect to the various alloys are shown in figure 4.11. At low amounts of Mn, the size distribution is centered around the lower sizes, 0.5-1.0 μm . From the figure it is also noted that the homogenisation variant H4 displays higher density of particles, regardless of Mn content. As content of Mn increases, the size distribution is shifted towards increasing size. This holds for homogenisation variants H2 and H5. However, H4 shows similar distribution of size for all alloys examined, except for alloy A14 where it is more similar to the other two homogenisation variants.



Figure 4.11: Particle size distribution for the different alloys for the primary particles.

Effect of Mn on dispersoids

The size distribution of dispersoids with regards to increasing content of Mn is found in figure 4.12. In alloy A9, with low content of Mn, a high density of particles in the 0.1 μ m-range is observed for homogenisation variant H4. As content of Mn increases, the distribution of size remains relatively unchanged for this homogenisation variant. Homogenisation variants H2 and H5 displays an increase in density of dispersoids in general, as the content of Mn increases. As the content of Mn increases, an increase in density of larger sized dispersoids is seen. Homogenisation variant H5 appears to have a slightly more even size distribution, especially for alloy A12, regardless of content of Mn. Homogenisation variant H2 also shows a size distribution that is similar, regardless of Mn content, except that the number of particles is higher as content of Mn increases.



Figure 4.12: Particle size distribution for the different alloys for the dispersoids.

4.4.2 Area fraction

Tables 4.2 and 4.3 shows the area fraction for the primary particles and dispersoids for alloys A9, A11, A12 and A14 for all homogenisation temperatures. As content of Mn increases area fraction of both primary particles and dispersoids increases. For the primary particles an increase of area fraction is also noted as homogenisation holding time increases. For the dispersoids however, the area fraction is relatively equal for alloys A9, A11 and A12 in all homogenisation variants, but increasing for alloy A14 only.

Table 4.2: Area fraction of primary particles in alloys A9, A11, A12 and A14 for all homogenisation temperatures presented in percentage. Standard deviation in parenthesis.

Alloy	H4 [%]	H2 [%]	H5 [%]
A9	0.522 (0.076)	0.540 (0.067)	0.664 (0.059)
A11	0.450 (0.034)	0.637 (0.188)	0.927 (0.053)
A12	0.724 (0.128)	0.871 (0.116)	1.007 (0.154)
A14	1.251 (0.291)	1.372 (0.184)	1.557 (0.093)

Table 4.3: Area fraction of dispersoids in alloys A9, A11, A12 and A14 for all homgenisation temperatures presented in percentage. Standard deviation in parenthesis.

Alloy	H4 [%]	H2 [%]	H5 [%]
A9	0.404 (0.060)	0.232 (0.058)	0.420 (0.019)
A11	0.805 (0.113)	0.790 (0.163)	0.782 (0.174)
A12	1.022 (0.144)	1.005 (0.175)	0.963 (0.019)
A14	1.106 (0.232)	1.091 (0.290)	1.608 (0.385)

4.4.3 Particle Density

The particle density for each alloy and homogenisation variant is presented in figures 4.13a and 4.13b. Both with respect to alloy and homogenisation temperature.

The effect of homogenisation on particle density

Figure 4.13a shows the density of primary particles with different homogenisation schemes. As seen from the figure, the density of primary particles is relatively constant for all alloys across the homogenisation scheme, although alloys 9 and 14 shows a slightly higher density in variant H4. The density in the named alloys are observed to be around 8000-10000 particles/ mm^2 . Alloy 12 on the other hand, displays a large variation in primary particle density. Almost 14000 particles/ mm^2 at maximum at H4 and down towards 6000 particles/ mm^2 for the H2 variant.

Figure 4.13b shows the density of dispersoids with different homogenisation schemes. The particle density of dispersoids remains relatively unchanged with respect to homogenisation for the H2 and H5 variant, while an increase in particle density is observed for the H4 variant. Alloy 9, with low content of Mn also displays an overall lower density of particles compared to A11, A12 and A14. It is also noted that alloy 14 shows smaller density at H4 than A11 and A12, but higher density at H5.



Figure 4.13: Particle density as N/mm^2 with respect to homogenisation for each alloy.

The effect of Mn on particle density

Figure 4.14a shows the density of primary particles for alloys 9-14. regardless of Mn content the density of primary particles does not show much variation and is in the 9000-11000 particles/ mm^2 -range. Alloy 12 on the other hand shows a significant increase in density for the homogenisation variants H4 and H5, and a decrease for the H2 variant.

Figure 4.14b shows the density of dispersoids for alloys 9-14. An increase increase in density is observed from alloy 9 to 11 and 12, especially for homogenisation scheme H4 which increases from 500000 particles/ mm^2 to over 1500000 particles/ mm^2 . For alloys 11 and 12 the difference between homogenisation scheme is greater than for alloys 9 and 14. It is also noted that the high density for alloys 11 and 12 for homogenisation variant H4 is reduced some in alloy 14.



Figure 4.14: Particle density as N/mm^2 with respect to content of Mn for each alloy.

4.4.4 Average particle size

Average particle sizes for both primary particles are presented in figure 4.15, with respect to homogenisation scheme (a-b) and with respect to content of Mn (c-d). With regards to homogenisation temperature a slight increase in the average particle size for the primary particles is observed. As with previous observations, the A12 variant displays somewhat unusual behaviour compared to the other alloys, here at the H2 variant.



Figure 4.15: Particle density as N/mm^2 with respect to homogenisation procedure (a-b) and content of Mn (c-d) for each alloy.

4.5 Alstruc simulation

The results of the Alstruc simulation is presented in the following. Volume fraction, average particle size and the density of particles is presented. Alloy A11 (0.5 wt% Mn) were not selected for this simulation as alloys A10 (0.4 wt% Mn) and A12 (0.6 wt%) were believed to provide sufficient information in this range of Mn-content. The simulated values and the values observed in this study will be further discussed in section 5.

4.5.1 Volume fractions

The volume fractions of dispersoids and primary particles are given in table 4.4. As content of Mn increases so does the volume fractions of the particles involved. The lack of Mn in alloys A8 and A9 causes formation of the β -phase. An increase of volume fraction for dispersoids for homogenisation variant H4 is also noted.

	Dispersoids			Primary particles α			Primary particles β		
Alloy	H4	H2	H5	H4	H2	H5	H4	H2	H5
A8	0	0	0	0	0	0	0.534	0.538	0.538
A9	0.037	0.043	0.039	0.279	0.523	0.555	0.322	0.038	0
A10	0.208	0.049	0.028	0.749	0.895	0.954	0	0	0
A12	0.442	0.255	0.121	0.978	1.086	1.169	0	0	0
A13	0.660	0.504	0.367	1.189	1.254	1.334	0	0	0
A14	1.140	0.968	0.842	1.617	1.643	1.709	0	0	0

Table 4.4: Volume fractions [%] for alloys A8, A9, A10, A12, A13 and A14

4.5.2 Average particle size

The average particle size, that is the equal circle diamter, is given in table 4.5. As with volume fractions, the particle size is increasing as content of Mn increases, but only in a very slightly manner. However, the size of the dispersoids in homogenisation variant H5 is quite much larger than in homogenisation variant H4, about 50 %. The primary particles seem to settle at at size between 5.2 and 6.0 μm . They also display a steady increase in size as homogenisation holding time increases, as is the case for H5.

	Dispersoids			Primary particles α			Primary particles β		
Alloy	H4	H2	H5	H4	H2	H5	H4	H2	H5
A8	0.022	0.024	0.024	0	0	0	1.438	1.442	1.442
A9	0.062	0.072	0.100	1.214	1.534	1.568	1.492	0.734	0
A10	0.084	0.064	0.032	5.238	5.560	5.678	0	0	0
A12	0.090	0.108	0.134	5.586	5.784	5.928	0	0	0
A13	0.096	0.114	0.140	5.728	5.830	5.950	0	0	0
A14	0.102	0.124	0.166	5.650	5.682	5.756	0	0	0

Table 4.5: Average particle size $[\mu m]$ for alloys A8, A9, A10, A12, A13 and A14

1

4.5.3 Particle density

Table 4.6 shows the density of dispersoids $[N/\mu m^3]$ and primary particles $[N/mm^2]$. A significant increase in density observed for both primary dispersoids and primary particles as content of Mn increasing. With regards to the dispersoids, it is noted that the homogenisation procedure affects the density by a substantial amount with a high density in homogenisation variant H4 and a decreasing density as homogenisation holding time and temperature increases. The density of primary particles seem to be affected only by content of Mn, and not by homogenisation procedure.

	Dispersoids $[N/\mu m^3]$		Primary particles α [N/mm^2]			Primary particles β [N/mm^2]			
Alloy	H4	H2	H5	H4	H2	H5	H4	H2	H5
A8	0.038	0.038	0.037	0	0	0	10358	10393	10393
A9	10.506	8.292	2.320	9434	11921	12187	5797	2852	0
A10	26.307	12.267	7.070	21836	23177	23667	0	0	0
A12	41.804	14.088	3.264	25079	25971	26617	0	0	0
A13	53.139	23.736	6.222	29005	29521	30138	0	0	0
A14	77.372	36.217	9.557	40512	40735	41270	0	0	0

Table 4.6: Density for alloys A8, A9, A10, A12, A13 and A14

4.6 IGC-testing

The samples were subjected to the IGC-test as described in section 3.6 and analysed. The results are presented as weight loss and as a qualitative analysis based on observations in the light microscope. Although all samples were examined, only locations with damage observed are presented.

4.6.1 Weight loss

Figure 4.16. shows the loss of material each sample experienced during the IGC-test. The weight loss is converted to micrometer. As seen, all but one sample shows weight loss in the 0.2 μ m-range.



Figure 4.16: Loss of material for each sample measured in micrometer.

4.6.2 Optical analysis

Attacks of various modes and degrees where observed when the samples were examined in the light microscope. An overview of damage observed can be found in table 4.7. Figure 4.17 shows examples of attacks observed when analysed. It should be noted that the most severe attacks were observed in sample 8-H5 and 11-H5. The homogenisation variants H4 showed more occurrences of pitting corrosion than H5, which most often displayed IGC attacks. It is important to note that Alloys A8 and A9 displayed IGC attacks in both homogenisation variants. Figure 4.17(a-d) shows an increase in degree of severeness with regards to pitting corrosion in the samples A8-A14. Overall, the degree of corrosion in these alloys is not alarming and will be further discussed in section 5.5.

Table 4.7: Overview of corrosion modes and degree of alloys A8-A14 for homogenisation variants H4 and H5.

Alloy	Comment
A8-H4	Some pitting and IGC observed
A8-H5	Severe IGC combined with pitting corrosion observed
A9-H4	IGC observed
A9-H5	Small IGC attacks observed
A10-H4	No damage observed
A10-H5	Small IGC attacks observed
A11-H4	Pitting corrosion observed
A11-H5	IGC and combined with pitting corrosion observed
A12-H4	Pitting corrosion observed
A12-H5	Small pitting corrosion attacks observed
A13-H4	Small pitting corrosion attacks observed
A13-H5	IGC combined with pitting corrosion observed
A14-H4	Pitting corrosion observed
A14-H5	Some IGC observed







(**g**) A11-H5

50 µn

(h) A13-H5

Figure 4.17: Examples of pitting corrosion attacks observed in some samples of homogenisation variant H4 (a-d), and examples of IGC attacks observed in homogenisation variant H5 (e-h). Images taken perpendicular to extrusion direction. Note the different scale bars.

50 µm

Chapter 5

Discussion

In the following the most important findings of the study are highlighted and discussed in further detail. The main focus will be on the effect of homogenisation and content of Mn on evolution of primary particles and dispersoids, and their effect on grain structure, strength and corrosion behaviour. This discussion is difficult due to the complicated correlation between the individual components and due to the fact that many of the contributing factors influence each other. A structural approach to explain the different factors is attempted, where the individual factors are discussed individually. This is hopefully simplified due to the structure of this study, where exaggerated parameters have been used to emphasize the different contributions and their effects.

5.1 Effect of homogenisation on particles and level of solid solution

Figure 5.1 shows the particle size distribution of primary particles and dispersoids. In contradiction to the figures 4.11 and 4.12 found in the results section, the particle density is here presented with respect to homogenisation variant. The goal of the homogenisation procedure for H4 variant was to encourage the formation of a large number of small dispersoids with the same amount of elements in solid solution as homogenisation variant H5. As seen in figure 4.8 from the results of the electrical conductivity, it can be stated that this was successful. The measurement of conductivity is indeed similar. As mentioned in 2.5, the presence of Mn may encourage the formation of dispersoids. Alloy A14 displayed highest density of dispersoids regardless of homogenisation procedure. This was the alloy with highest amount of Mn at 1.17 wt%. Alloy A9 showed the lowest density of dispersoids. Although different in density the alloys seem to share the same pattern in evolution of particle size distribution for the different homogenisation variants. That is increased density of large sized dispersoids, and a reduced density of smaller sized ones. Due to the slow diffusivity of Mn in the A1-matrix, it is likely to find more and smaller dispersoids when the homogenisation holding time is short and the temperature is low, such as in ho-

mogenisation variant H4. It would also be likely to find more of these in an alloy with higher Mn content, such as in alloy A14. As homogenisation holding time increases, Mn will diffuse into the matrix and will no longer be a part of the dispersoids, leading to a smaller amount of these. This statement seem to fit correctly with the results regarding particle size distribution found in this study.



Figure 5.1: Particle size distribution for all homogenisation variants. Distrubution of dispersoids and primary particles on the left and right hand side, respectively.

The primary particles, which is mainly formed during casting, displays a similar evolution. The particle size distribution starts out located around the portion of smaller sizes, then shifts towards a large particle size as homogenisation holding time and temperature increases. A particular situation is noted for A14 is noted in homogenisation variant H5. A disruption in the expected evolution compared to the other alloys, that is an increase in density around 0.5 μm . In this study the difference between dispersoids and primary particles is defined by particles of a size lesser or greater than 0.5 μm . A suggestion for the unexpected behaviour is that the sudden increase in density of small particles in fact is due to larger sized dispersoids. These may have, due to a high content of Mn in this alloy, grown in size reaching 0.5 μm or greater and therefore being classified as primary particles during the analysis. As such, this fake tail end of the graph could possibly be discarded. This would suggest that the trend line should start at a point of larger size. That would provide a more reasonable evolution across the homogenisation variants compared to the other alloys.

5.2 Grain structure

As an increase in dispersoids have been established for increasing content of Mn as well as short homogenisation holding time, it interesting to evaluate this effect on the grain structure. Figures 4.4 and 4.7 in section 4.1 shows this effect quite visually. As the density and size of Mn containing particles increases, there is a decrease in recrystallisation. This effect seems to be most dominating from around 0.5 wt% Mn. There are several mechanics that control the degree of recrystallisation. Both temperature and pressing force during extrusion as well as the presence of particles in the alloy. At low content of Mn, there is virtually no hinders for recrystallisation and a totally recrystallised grain structure is observed. But, already at a small increase of Mn, such as in alloy A9, a clear change can be noted. The presence of dispersoids will hinder recrystallisation as grain growth is stopped in its when encountering a particle. As the number of dispersoids increases, either as a result of homogenisation or as a result of increasing amount of content of Mn, the growing grains encounters an increasing number of hinders. This leads to a more fibrous structure, as seen in the alloy variants of homogenisation variant H4 or when the content of Mn increases. However, the presence of Mn will also increase the force required to extrude the profiles. This would normally suggest a large degree of recrystallisation as there is more turbulent flow along the edges of the profile when the force is increased. As seen from table 3.5 the force is indeed increased as the content of Mn increases. The expected greater degree of recrystallisation is not observed for the variants with high content of Mn and at homogenisation variant H4. This suggests that the high density of dispersoids found here effectively counter the grain growth, even with turbulent conditions. This can be related to the equation for total driving force for recrystallisation presented in 2.6, $P = P_D - P_Z$. Even though the driving force P_D increases due to higher pressing force, the small radius for dispersoids in the denominator of the expression for P_Z will negate the effect of an increased P_D .

5.3 Strength (hardness)

Examination of the hardness showed that the highest values were recorded in the alloy variants with low amounts of Mn. In addition the H4 homogenisation variant displayed significantly lower peak hardness. When the evolution of hardness is plotted with respect to increasing content of Mn for homogenisation variants H4 and H5 for the as extruded alloys and the peak hardness values an interesting development emerges, as shown in figure 5.2. The hardness values for the homogenisation values in each alloy is different from another. This even before the alloys have undergone artificial ageing. At this point, the only difference in the alloys are the amount of dispersoids. By examining the correlation further it is also notable that the difference for for each homogenisation variant appears to increase slightly as the content of Mn increases. For the variants of peak hardness, a similar pattern is observed. The difference in measured hardness between the homogenisation variants for each alloy is small for alloy variants A8 and A9, then increasing as the Mn content increases and approach the same value.



Figure 5.2: Evolution of hardness for the as extruded (AE) variants and for the variants aged to peak hardness (T6) for homogenisation parameters H4 and H5 with regards to increasing content of Mn.

For the as extruded variants, it is reasonable to believe that the dispersoids plays a key role in the variation in measured hardness. The mechanism involved here is referred to as Orowan strengthening, or bypassing. This means that the dislocations will be forced to move around the hard and small dispersoids. As the number and density of these increases, there will be more hinders for dislocations to move leading to a strengthening of the material as discussed in section 2.7. The results shows an increase of dispersoids in the homogenisation variant H4 and as the content of Mn increases more dispersoids are presumably formed. At lower content of Mn as seen in alloy A8 and A9 this is not enough to make an impact on the hardness, for alloy A10 and A11 the difference is significant.

A drop in the difference is also noted for alloys A12 and A13, and a large difference for A14. The non-linear nature of the evolution is not clear. Although the homogenisation procedure H4 was performed in a way that ensured equal conductivity compared to the H5 variant, the A10 and A11 variants were only brought up to holding temperature and then cooled, whereas the A12 and A13 were held for some time at this temperature. This could have affected the growth of dispersoids leading in the smaller difference in hardness observed for alloys A12 and A13 in their as extruded condition.

The decreasing hardness of the alloys for the peak hardness variants may be explained by the increasing amount of Mn in the alloys. As content of Mn increases the volume fraction of the primary particles also increases. As the primary particles consist of Si, there will be less Si available in solid solution to form MgSi-hardening precipitates during ageing. This will result in a decrease in peak hardness, as observed in the figure. The difference in peak hardness with regards to homogenisation variants can be explained by the difference in the density of dispersoids. A proposed mechanism is that the dispersoids may act as nucleation sites for MgSi-precipitates, affecting the strength contribution in a negative direction when the density of dispersoids is large. This could cause the difference in peak hardness observed between homogenisation variants H4 and H5, but should be investigated further.

Some of the alloy samples, namely A11 and A12, with 0.5 wt% Mn reached peak hardness after 2 hours of artificial ageing, rather than after 5 hours. This could indicate that the real peak hardness may be achieved somewhere in between 2 and 5 hours for these alloys.

5.4 Alstruc simulation

The simulated values extracted from Alstruc were quite different than the observed values observed in this study, although some parameters were more similar than others. When evaluating these results it is important to remember that the simulation parameters is calculated as a finished homogenisation whiles the alloys studied have been extruded in relatively thin profiles. In addition there is uncertainty regarding the actual measurement. There are several factors influencing the analysis of particles. Resolution and other parameters in the electron microscope, the representativeness of the images taken and the definition of a particle of a certain size.

The particle size simulated differ when compared to the observed values. Both for primary particles and dispersoids, although the primary particles displays the largest difference. That said, both the observed size of dispersoids and the simulated ones shows an increase in size as homogenisation holding time and temperature increases. The primary particles are in general much lower when observed, than simulated. A possible explanation to this is that these particles may be broken and shattered during extrusion, reducing their size. This is also observed when examined in the electron microscope. This may also influence the particle density. Although the density of primary particles observed is much lower than the simulated ones, this may be due to incorrect counting as a result of the definition of dispersoids used in this study, that is a particle with a size less than 0.5 μm . In that case, shattered primary particles or just primary particles with a size lesser than 0.5 μm , may be classified as dispersoids, wrongfully putting them in the dispersoid category.

When comparing the volume fraction of the simulated and observed values fits more correctly than for the other parameters. Although not entirely coherent, the values are in the same range. They also show the same trend as homogenisation holding time and temperature increase as well as content of Mn increases.

5.5 IGC test

The IGC stress test showed occurrences of both pitting and IGC. In general the attacks were more prominent and more often of the IGC type in homogenisation variants H5 than H4. As the H5 variants showed a thicker layer of recrystallised grain structure with large grains, it is reasonable to believe that a recrystallised layer may promote the susceptibility to IGC. Although this may be the case, the overall corrosion resistance of all samples and variants is considered low. Inter granular corrosion is often correlated with the presence of Cu. The alloys in this study have a very low amount of Cu and the lower susceptibility to IGC associated with low content of Cu is found here as well. The measured loss of weight found in this study is in fact of a such low amount that a major part may be contributed from the etching itself during sample preparation as described in section 3.3.1. Previous, unpublished work by the author showed increased corrosion loss as Cu content increased, as shown for sample A6 and A7, with a Cu content of 0.086 wt% and 0.091 wt%. Samples A1-A5 contained 0.001 wt% Cu in figure 5.3.



Figure 5.3: Corrosion loss during IGC test, fall 2013. Samples A6 and A7 with a Cu content of 0.086 wt% and 0.091 wt%, respectively. Samples A1-A5 with a Cu content of 0.001 wt%.

5.6 Further work

This study has covered the effect of Mn and homogenisation on a 6082-type alloy. Future work could look deeper into various variations of this alloy. This could be some of the following suggestions:

- The effect of dispersoids on precipitation hardening phases of MgSi
- Different homogenisation and/or extrusion parameters
- Different alloying elements, such as Cr and Cu
- Other precipitation hardening temperature
- Excess content of Si

It could also be of interest to further study the exact composition of the intermetallic phases and changes in this as effect of content of Mn or homogenisation scheme.

Chapter 6

Conclusion

In this study the effects of Mn and homogenisation have been studied on a AA6082. The main conclusions of the study is presented in the following.

- An increase in density of dispersoids and primary particles can be expected with an increase of content of Mn.
- An increase in density of dispersoids and primary particles can be expected with a short homogenisation holding time (0-1h) and low temperature (545 °C).
- The presence of dispersoids greatly effect the final grain structure of the extruded profile. High density of dispersoids leads to a low degree of recrystallisation.
- The formation of dispersoids associated with higher amounts of Mn may lower the final hardness of the profile as less Si is available to form the hardening Mg_2Si -phase during artificial ageing.
- The alloy variants studied displays low susceptibility to corrosion, as is associated with low amounts of Cu. However, it should be pointed out that the alloy variants with a higher degree of recrystallisation seemed more prone to intergranular corrosion than the alloys with a fibrous structure.
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Hardness values

Table A.1: Table showing hardness values [HV5] measured for all alloys in homogenisation variants **H4** with respect to ageing time. Standard deviation in parentheses.

H4	A8	A9	A10	A11	A12	A13	A14
0 min	77.3 (2.1)	79.3 (2.1)	79.7 (2.1)	85.3 (0.6)	85.0 (1.7)	86.3 (1.2)	84.3 (1.5)
30 min	81.7 (2.1)	77.0 (1.0)	83.7 (2.1)	85.0 (1.0)	84.0 (4.0)	82.7 (2.3)	80.7 (1.5)
2h	110.0 (3.0)	110.3 (1.5)	106.0 (2.0)	105.7 (4.0)	108.3 (2.1)	98.7 (1.2)	93.3 (2.9)
5h	113.0 (3.5)	117.3 (2.3)	108.3 (2.3)	100.7 (1.5)	102.0 (3.6)	96.7 (0.6)	95.0 (1.0)
1d	100.0 (7.0)	109.0 (3.6)	102.0 (3.5)	93.0 (2.6)	83.3 (5.1)	84.3 (4.2)	80.0 (1.2)
1 w	74.3 (2.3)	76.3 (0.6)	70.0 (1.0)	66.0 (1.0)	70.3 (2.5)	70.3 (2.1)	67.0 (1.0)

Table A.2: Table showing hardness values [HV5] measured for all alloys in homogenisation variants **H2** with respect to ageing time. Standard deviation.

H2	A8	A9	A10	A11	A12	A13	A14
0 min	79.0 (3.6)	76.3 (0.58)	78.0 (2.6)	76.3 (1.2)	80.3 (2.1)	82.3 (4.0)	79.0 (1.0)
30 min	91.0 (2.6)	77.0 (1.7)	79.7 (1.5)	76.5 (0.5)	87.3 (2.0)	77.3 (1.5)	81.0 (4.0)
2h	111.7 (3.8)	113.0 (3.0)	104.0 (2.0)	109.7 (1.5)	116.0 (6.6)	101.7 (1.5)	97.7 (2.5)
5h	120.7 (5.0)	115.0 (1.0)	111.0 (4.6)	110.7 (1.2)	110.0 (2.0)	105.3 (3.0)	93.0 (1.7)
1d	100.7 (5.0)	104.7 (5.0)	102.7 (3.8)	99.0 (1.0)	97.3 (0.6)	97.0 (3.6)	87.7 (2.9)
1w	76.3 (2.0)	75.0 (3.0)	77.3 (1.5)	75.7 (3.5)	77.3 (0.6)	73.3 (1.5)	70.3 (2.3)

а

H5	A8	A9	A10	A11	A12	A13	A14
0 min	75.0 (2.6)	80.3 (2.3)	76.3 (1.2)	79.7 (2.1)	82.7 (1.5)	83.0 (1.0)	74.7 (3.2)
30 min	86.7 (2.5)	88.7 (2.9)	91.7 (4.0)	78.7 (2.1)	89.0 (3.5)	89.3 (1.5)	79.3 (1.5)
2h	113.7 (2.1)	114.0 (3.6)	115.3 (5.5)	115.3 (2.8)	116.0 (3.5)	104.2 (2.0)	89.3 (0.6)
5h	116.0 (2.6)	117.3 (2.3)	119.3 (4.0)	112.3 (4.9)	111.0 (3.6)	105.0 (3.0)	99.3 (2.5)
1d	97.7 (3.8)	107.0 (1.0)	101.3 (3.5)	106.7 (4.0)	93.3 (2.9)	95.3 (3.0)	92.3 (1.5)
1w	76.7 (0.6)	78.0 (1.7)	75.7 (1.5)	76.0 (5.3)	79.7 (2.5)	78.3 (2.1)	72.7 (0.6)

Table A.3: Table showing hardness values [HV5] measured for all alloys in homogenisation variants **H5** with respect to ageing time. Standard deviation.

Appendix B

Electrical conductivity

 Table B.1: Table showing evolution of electrical conductivity [MS/m] measured for all alloys in homogenisation variants H4 with respect to ageing time.

H4	A8	A9	A10	A11	A12	A13	A14
0 min	24.93	24.19	23.80	24.00	24.02	24.01	23.42
30 min	26.46	25.20	24.88	24.88	24.77	24.27	24.17
2h	28.56	26.99	27.17	27.10	27.00	26.50	26.26
5h	29.29	27.96	27.92	27.77	27.88	27.66	27.63
1d	29.70	28.67	28.77	28.67	28.77	28.55	28.26
1w	32.48	30.70	30.26	30.16	30.07	29.56	28.74

 Table B.2: Table showing evolution of electrical conductivity [MS/m] measured for all alloys in homogenisation variants H2 with respect to ageing time.

H2	A8	A9	A10	A11	A12	A13	A14
0 min	25.06	24.29	23.87	23.97	24.00	23.88	23.46
30 min	26.47	25.25	24.96	25.03	24.86	24.40	24.33
2h	28.38	27.45	26.92	26.46	26.51	26.34	25.66
5h	29.17	28.03	27.77	27.74	27.67	27.78	27.52
1d	29.92	28.66	28.36	28.57	28.36	28.62	28.25
1 w	32.42	30.93	30.16	29.94	29.79	29.52	29.02

 Table B.3: Table showing evolution of electrical conductivity [MS/m] measured for all alloys in homogenisation variants H5 with respect to ageing time.

Н5	A8	A9	A10	A11	A12	A13	A14
0 min	25.14	24.15	23.72	23.59	23.69	23.48	23.13
30 min	26.39	25.38	24.80	24.52	24.45	24.32	24.15
2h	28.59	27.35	26.88	26.37	26.01	25.83	24.79
5h	29.13	27.61	27.19	27.44	27.20	27.17	26.71
1d	29.61	28.37	27.78	27.79	27.84	27.67	27.61
1w	32.32	30.48	29.47	28.93	29.05	28.51	28.35



SEM images



Figure C.1: SEM-pictures of alloy A9 for all homogenisation variants at 350x (a-c) and 5000x (d-f) magnification.



Figure C.2: SE-pictures of alloys A11 and A12 for all homogenisation variants at 350x (a-c, g-i) and 5000x (d-f, j-l) magnification.



Figure C.3: SEM-pictures of alloy A14 for all homogenisation variants at 350x (a-c) and 5000x (d-f) magnification.