LICENTIATE THESIS

As-cast AZ91D Magnesium Alloy Properties- Effect of Microstructure and Temperature

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Effect of Microstructure and Temperature
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“The journey of a thousand miles begins with one step.”

-Rumi

To my parents,
who had given me dreams to look forward to
Magnesium and magnesium alloys are used in a wide variety of structural applications including automotive, aerospace, hand tools and electronic industries thanks to their light weight, high specific strength, adequate corrosion resistance and good castability. Al and Zn are the primary alloying elements in commercial Mg alloys and commonly used in automotive industries. AZ91 is one of the most popular Mg alloys containing 9% Al and 1% Zn. Hence, lots of research have been done during last decades on AZ91D. However, the existing data concerning mechanical properties and microstructural features showed large scatter and is even contradictory.

This work focused on the correlation between the microstructure and the mechanical properties of as-cast AZ91 alloy. An exhaustive characterization of the grain size, secondary dendrite arm spacing (SDAS) distribution, and fraction of Mg17Al12 using optical and electron backscattered diffraction (EBSD) was performed. These microstructural parameters were correlated to offset yield point (R_p0.2), fracture strength and elongation to fracture.

It was understood that the intermetallic phase, Mg17Al12, plays an important role in determining the mechanical and physical properties of the alloy at temperature range from room temperature up to 190°C. It was realized that by increasing the Mg17Al12 content above 11% a network of intermetallic may form. During deformation this rigid network should break before any plastic deformation happen. Hence, increase in Mg17Al12 content resulted in an increase in offset yield point. The presence of this network was supported by study of thermal expansion behaviour of the alloy containing different amount of Mg17Al12.

A physically-based model was adapted and validated in order to predict the flow stress behaviour of as-cast AZ91D at room temperature up to 190°C for various microstructures. The model was based on dislocation glide and climb in a single-phase (matrix) material containing reinforcing particles. The temperature dependant variables of the model were quite well correlated to the underlying physics of the material.

Keywords: Magnesium alloys, As-cast, AZ91D, Mechanical properties, Microstructural scale effect, Physical modelling
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Hoda Dini
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SUPPLEMENTS

The following supplements constitute the basis of this thesis:

**Supplement I**  
H. Dini, N. Andersson, A.E.W Jarfors; Effects of Microstructure on Deformation Behaviour of AZ91D Cast Alloy. TMS 2014, 143rd ANNUAL MEETING & EXHIBITION February 16-20, San Diego, CA, USA.  
*Dini was the main author. Andersson and Jarfors contributed with supervision and advice on method of analysis.*

**Supplement II**  
*Dini was the main author. Andersson and Jarfors contributed with supervision and advice on method of analysis. Ghassemali helped with the EBSD results.*

**Supplement III**  
H. Dini, N. Andersson, A.E.W. Jarfors; Effect of Mg17Al12 content on mechanical properties of AZ91D cast alloy, submitted to the Scripta Materialia.  
*Dini was the main author. Andersson and Jarfors contributed with supervision and advice on method of analysis.*

**Supplement IV**  
*Dini was the main author. Svoboda and Lindgren contributed in adaption and optimization of the model. Ghassemali helped with the EBSD results. Andersson and Jarfors contributed with supervision and advice on method of analysis.*

The following supplement is not included in this thesis:

**Supplement V**  
*Zamani was the main author. Dini, Svoboda and Lindgren contributed in development and optimization of the model. Seifeddine, Andersson and Jarfors contributed with supervision and advice on method of analysis.*
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CHAPTER I

INTRODUCTION

CHAPTER INTRODUCTION

The background of present study with special focus on AZ91D alloy is described. General microstructural features and mechanical properties of the alloy are presented. A constitutive model based on the evolution of immobile dislocation which has been used for describing plastic flow of the alloy is introduced.

1.1 MAGNESIUM ALLOYS

Magnesium alloys have the lowest density (<1.8 g/cm³) of all the structural metals. Increasing their use in automobile and aircraft parts would increase energy efficiency by weight saving [1]. Magnesium is also the 8th most abundant element on the Earth, is easy to machine and is potentially recyclable [2]. The poor creep properties above 120°C is the main challenge with utilizing more Mg alloys in automotive stems [3]. Enhancing the creep resistance of magnesium alloys has therefore been the subject of various researches with the focus on influence of alloy composition with a view of improving creep resistance [4-6]. The most widely alloying element is aluminium, which is relatively not expensive, has a low density and strong strengthening effect on magnesium. Hence, most commercial Mg alloys are based on Mg-Al system, with small addition of other alloying elements, such as Zn, Mn, Si and rare earth to reach certain application requirements.

Although wrought magnesium alloys have higher mechanical strength than as-cast Mg alloys, they exhibit higher mechanical anisotropy which is detrimental to forming processes. Moreover, the processing of wrought Mg alloys into their final shape is more complex [7]. Hence, currently, Mg components are mostly produced by casting [8, 9] since this is the most cost-efficient process. However, poor mechanical performance of as-cast Mg alloys is the main concern in their application. The poor mechanical behaviour is mainly due to the presence of porosity and microstructural inhomogeneities such as variation in grain size and phase distribution [10-15]. Hence, various attempt has been carried out to relate microstructure to mechanical performance in Mg casting both by experimental [11-17] and modelling [11, 18-21] approaches. However, despite all of the studies in this area, a large scatter in data has been observed which means that a significant effort is still required to establish a quantitative relations between the microstructure and mechanical properties of magnesium alloys.
In conclusion, further works are required in order to i) understand the clear correlation between the microstructural feature and mechanical behaviour of Mg alloys and accordingly have a modelling tool to predict these behaviour ii) optimize manufacturing technique and thus improve the quality of magnesium cast parts.

1.2 MG-AL BASED ALLOYS

Aluminium is alloyed with magnesium to increase strength, castability and corrosion resistance. A maximum solid solubility of aluminium in magnesium is 12.7 wt% at 437°C (Figure 1) and then its solubility decreases to around 2% at room temperature. Hence, after a precipitation hardening treatment, an incoherent, coarse precipitates of the equilibrium Mg₅Al₁₂ phase will formed [22].

In order to improve fluidity and room temperature strength Zink is often added to Mg-Al alloys. Zinc also assists to reduce the corrosive effect of iron and nickel. However, addition of Zn in concentration higher than 1.0-1.5% to magnesium alloys containing 7 to 10% Al leads to hot cracking effect [22].

Manganese is another element that always alloyed with both Mg-Al and Mg-Al-Zn alloys in order to reduce impurities from iron and other heavy metal and turn them to Al-Mn-Fe intermetallic compounds. These intermetallic may settle down during alloy production. The concentration of Mn strongly depends upon Al concentration in the Mg-Al-Zn alloy because solubility of Mn in liquid magnesium sharply decreases with increasing Al content [22].

![Mg-Al phase diagram](image)

**Figure 1. Mg-Al phase diagram after reference [23].**

AZ91D (9% Al and 1% Zn) is one of the most common alloy in the Mg-Al-Zn alloying system. The microstructure and phase transformation taking place during solidification of AZ91D
alloy is studied in depth through the literatures [24-26]. It has been revealed that microstructure of all as-cast AZ91D constituents of hypoeutectic Mg-Al solid solution, eutectic Mg-Al supersaturated solid solution, and Mg<sub>17</sub>Al<sub>12</sub> phase. These phases are obtained from non-equilibrium solidification conditions.

Under equilibrium conditions, AZ91D alloy should solidify with the formation of only hypoeutectic Mg-Al solid solution (Figure 1). However, the slow diffusion of aluminium under non-equilibrium solidification conditions leads to the formation of eutectic Mg<sub>17</sub>Al<sub>12</sub> phase and Al-rich eutectic Mg-Al solid solution [27].

Under non-equilibrium solidification it is assumed that at the interface of the liquid/solid each phase has the chemical composition identified from the equilibrium phase diagram, but diffusion is not allowed in the solid, but in the liquid composition is uniform due to convection [28].

In addition, the morphology of eutectic structure formed under non-equilibrium cooling conditions is different from typical eutectic morphology formed under eutectic solidification [29]. Here, a divorced eutectic structure is formed instead of the typical lamellar eutectic morphology [30].

In rapid solidification, the eutectic phase of Mg<sub>17</sub>Al<sub>12</sub> can form as a separate large particles with different shapes around the primary Mg-Al solid solution. It was indicated that the aluminium content of the Mg-Al solid solution can vary from 3-4% in the bulk to more than 10% in the vicinity Mg<sub>17</sub>Al<sub>12</sub> phase. In ingot casting due to slow cooling rate discontinuous precipitation can also take place in solid state. However, no discontinuities precipitation occurs in die casting due to high cooling rate [28]. The microstructure of die cast AZ91D alloy consists of hypoeutectic Mg-Al solid solution grains along with eutectic Mg<sub>17</sub>Al<sub>12</sub> particles surrounded by islands of eutectic Mg-Al supersaturated solid solution [28, 31, 32]. Figure 2 shows the solidification sequences of AZ91D alloy with ingot casting and die casting.

![Solidification sequences of AZ alloys after reference [31].](image)

The solidification sequences of ingot casting AZ91D alloys will be described as follows: Solidification of hypoeutectic magnesium solid solution from molten liquids starts at 595°C and
followed by the formation of the first Mg-Al solid solution crystals having about 1.5% Al. As the alloy cools down, crystals of hypoeutectic Mg-Al solid solution increases in size, with each layer of atoms added to the crystal surface being richer in aluminium than the preceding layer. When temperature of 470°C is reached (at which freezing would be complete according to the equilibrium diagram) the crystals have about 1.5% Al at the centre, but approximately 9% at the surface. Thus, the average composition is somewhere between 1.5 and 9% Al, although freezing is not yet complete. Further cooling increases the size of hypoeutectic Mg-solid solution crystals and the chemical composition of the outside of the crystals reaches about 12% Al when the temperature reaches the eutectic temperature about 437°C. At this temperature the growth of hypoeutectic Mg-solid solution is terminated and grains exhibit Al-rich and Al-poor regions [22]. Hence, the growth of Mg-Al solid solution is completed at 437°C and liquid reaches eutectic temperature solidifying as two phases: eutectic Mg-Al solid solution and Mg17Al12 intermetallic. Depending on the subsequent cooling rate in the solid state, the eutectic Mg-solid solution can experience a discontinuous decomposition with formation of Mg17Al12 intermetallic in lamellar form due to decreasing aluminium solubility in magnesium-aluminium solid solution or remains as supersaturated solid solution at room temperature due to slow diffusion rate of aluminium in magnesium. Accordingly, cooling rate has effect on grain size, secondary dendrite arm spacing (SDAS) and the fraction of Mg17Al12. Figure 3 shows the typical microstructure of AZ91D.

![Microstructure of AZ91D](image)

**Figure 3. Microstructure of AZ91D.**

### 1.3 DEFORMATION MECHANISMS OF MAGNESIUM ALLOYS

According to von Mises criterion [9] more than 5 independent slip systems are needed for metals to deform uniformly and without failure at grain boundaries. Magnesium alloys have a hexagonal closed pack (HCP) structure. Therefore, only a limited number of slip systems are available to accommodate plastic deformation. At room temperature, magnesium alloys have four independent slip systems, and the remaining deformation is accommodated by twinning. However, at elevated temperatures additional slip systems become active, providing sufficient independent systems and hence ductility at elevated temperatures will be improved.
1.3.1 Role of Slip in Deformation

Figure 4 shows the slip planes (basal, prism and pyramidal) in magnesium alloys [9, 33]. Slip occurs when the resolved shear stress on the slip plane reaches the critical value for that particular system. The critical resolved shear stress (CRSS) for non-basal slip system is much greater than basal slip system at room temperature. Hence, magnesium can be deformed easily within their basal planes at room temperature. Only two independent slip systems exist along slip direction (11̅20), see Table 1. The pyramidal (a) slip produces identical shape change as produced by combined basal slip and prismatic (a) slip, the resulting number of independent slip systems from all three deformation modes are still 4 [9] and thus cannot accommodate deformation along (c) direction to fulfil criterion stated by von Mises. Therefore, some other non-basal slip systems having a component in c-direction (Table 1) should be activated [34] or the deformation should occur by twinning [33].

At elevated temperature above 200°C, the critical resolved shear stress for non-basal system rapidly decreases, and secondary slip systems {1100}(11̅20) prismatic and {1101}(11̅20) pyramidal (see Table 1) become active. The larger burger vectors of (c + a) and small pyramidal plane distance at room temperature makes the activation of pyramidal slip system quite difficult but increasing the temperature will ease the activation process of pyramidal slip system [34]. These additional slip planes explain the increasing ductility at elevated temperature.

### Table 1. Independent dislocation systems in HCP metals [33].

<table>
<thead>
<tr>
<th>Direction</th>
<th>Plane</th>
<th>Notation</th>
<th>Number of independent modes</th>
</tr>
</thead>
<tbody>
<tr>
<td>⟨a⟩</td>
<td>Basal</td>
<td>(0002)(11̅20)</td>
<td>2</td>
</tr>
<tr>
<td>⟨a⟩</td>
<td>Prismatic</td>
<td>(11̅00)(11̅20)</td>
<td>2</td>
</tr>
<tr>
<td>⟨a⟩</td>
<td>Pyramidal, 1st order</td>
<td>(1101)(11̅20)</td>
<td>4</td>
</tr>
<tr>
<td>⟨a⟩ + ⟨c⟩</td>
<td>Pyramidal, 2nd order</td>
<td>(1011)(1123)</td>
<td>4</td>
</tr>
<tr>
<td>⟨a⟩ + ⟨c⟩</td>
<td>Pyramidal, 2nd order</td>
<td>(2111)(1123)</td>
<td>4</td>
</tr>
<tr>
<td>⟨a⟩ + ⟨c⟩</td>
<td>Pyramidal, 2nd order</td>
<td>(1122)(1123)</td>
<td>4</td>
</tr>
</tbody>
</table>

1.3.2 Deformation by Twinning

Twinning is an important deformation mechanism in HCP (hexagonal close packed) materials at room temperature. Twinning is a deformation mechanism where a portion of the original crystal will take up a new orientation under shearing and produce a mirror image of the parent
crystal [35]. However, it is more dominant in wrought alloys with presence of texture in microstructure or mainly under compression flow stress. Hence, twinning is not a major deformation mechanism in as-cast AZ91D under conditions of practical interest of this study.

1.4 MECHANICAL PROPERTIES OF MG-AL ALLOYS

As mentioned earlier, the microstructure of as-cast Mg-Al in initial state is consist of solid solution and Mg17Al12 phases. The Mg17Al12 phases precipitates at grain boundaries during deformation in the form of fine precipitates which are known to increase the strength by suppressing basal slip [36]. These precipitated Mg17Al12 phases are perpendicular to the basal plan [37] (main slip system of α-magnesium matrix) and make the dislocations movement more difficult and hence increases the strength of the alloy [38].

The main challenge in application of Mg-Al–based alloys is the poor mechanical properties at high-temperature and particularly creep resistance at temperature above 120°C. It is indicated by literatures [6,32,39-41] that the low creep resistance of Mg-Al–based alloys is attributed to the poor thermal stability of Mg17Al12 phase. Zhu et al.[16] reported that Mg17Al12 precipitates tend to coarsen at elevated temperatures, thus losing their strengthening effect. Discontinuous precipitation can result to grain boundary sliding at elevated temperature. The Mg17Al12 phase at grain boundaries may decompose at elevated temperatures which will increase the content of aluminium in these regions. Consequently, the failure will happen in these regions [38]. Moreover, it is reported that at the interface of Mg17Al12/magnesium matrix micro cracks may nucleate due to not compatible bcc structure of Mg17Al12 and hcp structure of matrix. This leads to a limited ductility of the alloy [38]. However, it is reported that temperatures lower than 120°C are not high enough to soften Mg17Al12 particles or to weaken solid-solution hardening and hence the contribution of grain-boundary sliding is not noticeable [42, 43].

Regev et al.[44-46] indicated that for pressure die cast alloys (with grain size of 15 µm) and ingot cast alloys (with grain size of 300 µm) at the temperature range from 70°C to 200°C, low stress creep deformation is dominated by grain boundary sliding. However, at higher stress levels (\( \sigma / E > 10^{-3} \)), dislocation climb becomes the governing creep mechanism. The deformation behavior of the AZ91D at stress levels higher than what is required for creep with a relatively low temperature has not been extensively studied. At this condition, the diffusivity is limited and the main deformation mechanism is dislocation glide [47].

Moreover, changing the grain size influences the deformation map of the material [47]. Although the mechanical behaviour of Mg-Al produced by die casting has been extensively investigated and discussed [44, 45, 48-50], some studies [44, 49-53] have shown that alloys of similar composition produced with different cooling rates or different technologies can exhibit a quite different behaviour due in particular to microstructural features such as volume fraction, distribution and coarseness of the Mg17Al12, α- grain sizes and Al content in solid solution. However, to the best knowledge of author, the correlation between microstructural features and mechanical behaviour has not been clearly established for the AZ91 cast alloy. This knowledge is essential for selecting manufacturing techniques. Moreover, detailed description of material behaviour in a wide range of temperature and strain rate is crucial for simulation of deformation process. For this particular purpose, using physically-based models capable of correlating microstructural features to the mechanical response is preferred [54]. The physically-based model which has been used for describing plastic flow of AZ91D alloy is introduced in following section.
1.5 DEFORMATION MODELLING OF MAGNESIUM ALLOYS

Constitutive models adequately representing the deformation behaviour of engineering materials under combination of thermal and mechanical loading are crucial for obtaining accurate results in simulations of manufacturing and service. The constitutive models are mathematical description of physical phenomena and hence, different modelling approaches exist.

Commonly, the deformation behaviour of metals is represented by an empirical relationship, mostly in terms of the power law of strain and strain rate [55-57]. The empirical relations are usually lacking in predictive capabilities beyond the derived range of experimental conditions at which they were curve-fitted. Models which are related to the underlying physics of the deformation are therefore preferable. The advantage of using physically-based models is an expected larger domain of validity since these models have predicting capability outside the range of experimental data used for calibration, provided that the deformation mechanisms included in the models are describing the dominating deformation behaviour correctly [58].

The physical model which will be presented in following section includes a coupled set of evolution equations for the internal state variables, dislocation density and vacancy concentration. The concept of the dislocation density is the amount (length) of dislocations for a representative volume element divided by its volume. The model considers two different densities, a mobile and an immobile dislocation density. Change in density of immobile dislocation is related to slip system and the thermally activated annihilation by the climb of the dislocations. The immobilization rate of mobile dislocations is a function of microstructure, strain rate and temperature. The recovery process occurs with climb [59] and glide of dislocations [60]. The diffusion of vacancies, which usually takes place at elevated temperature, is a dominant factor in the recovery process of dislocations. The high concentration of vacancies near grain boundaries enhances creep controlled by dislocation glide and climb processes [61]. These internal state variables are used instead of accumulated effective plastic strain, commonly used in phenomenological models. The concept of the dislocation density is the amount (length) of dislocations for a representative volume element divided by its volume. The model considers two different densities, a mobile and an immobile dislocation density.

1.5.1 Modelling of flow stress

The flow stress is consist of two part, i) a component due to long-range barriers, $\sigma_G$, that cannot be assisted by thermal energy and ii) a component due to short-range barriers, $\sigma^*$, which is thermally activated. Hence, the flow stress in Eq.1 is defined as the combination of components of resistance to the motion of dislocations [62-64].

$$\sigma_y = \sigma_G + \sigma^*$$  \hspace{1cm} (1)

Long range flow stress contribution is related to the interaction of immobile dislocations in substructure and it can be written as [62]:

$$\sigma_G = m\alpha Gb\sqrt{\rho_i}$$  \hspace{1cm} (2)
where $m$ is the Taylor orientation factor and $\alpha$ is a proportionality factor and $\rho_i$ is the immobile dislocation density. The shear modulus, $G$, can be computed from the Young’s modulus $E$ and Poisson ratio $\nu$ as,

$$G = \frac{E}{2(1+\nu)}$$

(3)

The short-range term in Eq.1 is the thermally activated flow stress component. It is the stress needed for a dislocation to pass short-range obstacles and to move it through the lattice. The total transient time taken by a dislocation to move over a distance between two obstacles consists of a waiting time and a travel time. The moving dislocation has the waiting time in front of an obstacle before it manages to pass the obstacle and then moves to the next one. The travel time is small compared to the waiting time and is assumed to be negligible. The waiting time is the inverse of the frequency of the successful jumps to overcome the obstacles. This frequency is related to the probability, defined by the Boltzmann law of energy distribution, that the dislocation has an energy that exceeds the needed activation energy to overcome the obstacles. The waiting time and thereby the average velocity is assumed to depend on the Gibbs free-energy of activation $\Delta G$ for cutting or by-passing of obstacles [65], and on the temperature $T$. The average velocity is defined as:

$$\bar{v} = \nu_a \exp\left(-\frac{\Delta G}{kT}\right)$$

(4)

where $\lambda$ is the mean free path, $\nu_a$ is the attempt frequency, $\Delta G$ is the activation energy, $k$ is the Boltzmann constant and $T$ is the temperature in Kelvin. The dislocation density and velocity is related to plastic strain rate via the Orowan equation [66]:

$$\dot{\varepsilon}^p = \frac{\rho_m b \bar{v}}{m}$$

(5)

where $\bar{v}$ is the average velocity of mobile dislocations having a density $\rho_m$. The Eq.5 relations can be written as:

$$\dot{\varepsilon}^p = \frac{\rho_m \Lambda b \nu_a}{m} \exp\left(-\frac{\Delta G}{kT}\right)$$

(6)

The motion of dislocations is facilitated by thermal energy. If the stress is insufficient to drive a dislocation pass a barrier having activation energy $\Delta G$, the probability that a dislocation will jump over the barrier increases with increased temperature. Different shapes of barrier energy distribution results in different constitutive equations. A generalized equation for these shapes was proposed by Kocks et al [67] with two parameters, $p$ and $q$, and is given by:

$$\Delta G = \Delta F \left[1 - \left(\frac{\sigma^*}{\sigma_{ath}}\right)^p\right]^q$$

(7)
Here, \( \Delta F \) is the total free energy required for a dislocation to overcome the lattice resistance or obstacles. The quantity \( \sigma_{\text{ath}} \) is the athermal flow strength that must be exceeded in order to move dislocations across the lattice without the aid of thermal energy. The exponents \( 0 < p \leq 1 \) and \( 0 < q \leq 2 \) are related to the shape of energy barriers. The pre-exponential term in Eq.6, which is approximated following Frost and Ashby [65] to be constant, is expressed as

\[
\dot{\varepsilon}_{\text{ref}} = \frac{\rho_m Ab v_a}{m}
\]  

(8)

where \( \dot{\varepsilon}_{\text{ref}} \) is the reference strain rate. Combining (6) with (7) and (8) yields to:

\[
\dot{\varepsilon}^p = \dot{\varepsilon}_{\text{ref}} \exp \left\{ -\frac{\Delta F}{k T} \left[ 1 - \left( \frac{\sigma^*}{\sigma_{\text{ath}}} \right)^p \right]^q \right\}
\]

(9)

Here, \( \Delta F = \Delta f_0 G b^i \) is the activation energy necessary to overcome lattice resistance in the absence of any external force and \( \sigma_{\text{ath}} = \tau_0 G \) is the shear strength in the absence of thermal energy. Some guidelines regarding of \( \Delta f_0 \) and \( \tau_0 \) are given in Table 2 [65]. Here, \( L \) is the mean spacing of the obstacles, precipitates or solutes.

<table>
<thead>
<tr>
<th>Obstacle Strength</th>
<th>( \Delta f_0 )</th>
<th>( \tau_0 )</th>
<th>Example</th>
</tr>
</thead>
<tbody>
<tr>
<td>Strong</td>
<td>2</td>
<td>( &gt; \frac{b}{L} )</td>
<td>Strong precipitates</td>
</tr>
<tr>
<td>Medium</td>
<td>0.2-1.0</td>
<td>( \approx \frac{b}{L} )</td>
<td>Weak precipitates</td>
</tr>
<tr>
<td>Weak</td>
<td>&lt;0.2</td>
<td>( \ll \frac{b}{L} )</td>
<td>Lattice Resistance,</td>
</tr>
</tbody>
</table>

Accordingly, the short-range stress component in Eq.1 as a function of the effective plastic strain rate, can be derived as [67-69]:

\[
\sigma^* = \tau_0 G \left[ \ln \left( \frac{\dot{\varepsilon}_{\text{ref}}}{E^p E^r} \right) \right]^{l/p}
\]

(10)

1.5.2 Evolution of immobile dislocation density

The equation for the flow stress in Eq.1 requires evolution equations for internal state variables which are the dislocation density and the vacancy concentration. The mobile dislocation density is assumed to be much smaller than the immobile one [70, 71]. The evolution of the immobile dislocation density is expressed in two terms of hardening (+) and recovery (-) [72]:

\[
\dot{\rho}_i = \dot{\rho}_i^{(+)} + \dot{\rho}_i^{(-)}
\]

(11)
**Hardening Process:**

Mobile dislocations move over a mean free path $\Lambda$ before they are immobilized or annihilated. The immobile dislocation density is assumed to increase proportional to the plastic strain rate, which is related to the density of mobile dislocations, shown in Eq. 5, and inversely to the mean free path:

$$\dot{\rho}_m^{(+)} = \frac{m}{b} \frac{1}{\Lambda} \dot{\varepsilon}^p$$

(12)

where $m$ is the Taylor orientation factor. The mean free path can be related to SDAS ($\lambda$) in cast alloys and also dislocation subcell or subgrain diameter ($s$) as

$$\frac{1}{\Lambda} = \frac{1}{\lambda} + \frac{1}{s}$$

(13)

The effect of grain size on flow stress known as Petch-Hall relation, is accounted via this term. The formation and evolution of subcells is related to the immobile dislocation density by the parameter $K_c$ [73]:

$$s = K_c \frac{1}{\sqrt{\rho_i}}$$

(14)

**Recovery Processes:**

The dislocation density may be reduced by different processes. Recovery, remobilization and/or annihilation of dislocations are proportional to the current dislocation density and controlled by dislocation climb and glide. Recovery by dislocation glide is described as [67, 74]:

$$\dot{\rho}_{i,glide} = \Omega \rho_i \dot{\varepsilon}^p$$

(15)

where $\Omega$ is a recovery function dependent on temperature. This model accommodates only the dynamic recovery due to the strain rate. Static recovery controlled by diffusion climb is assumed to be [75]:

$$\dot{\rho}_{i,climb} = 2c_v D_v \frac{c_v}{c_v^{eq}} \frac{Gb^2}{kT} \left( \rho_i^2 - \rho_i^{eq} \right)$$

(16)

where $c_v$ and $c_v^{eq}$ are current and equilibrium vacancy concentrations respectively and $c_f$ is a material coefficient related to the stacking-fault energy. The dislocation density decreases towards an equilibrium value of $\rho_i^{eq}$. The self-diffusion coefficient is given according to Reed-Hill and Abbaschian [76]:

$$D_v = a^2 v_j e^{\frac{\Delta S_{vm} + \Delta S_{sf}}{k}} e^{\frac{Q_{vm} + Q_{sf}}{kT}} = D_ve^{\frac{Q_v}{kT}}$$

(17)

where $\Delta S_{vm}$ is the increase in entropy due to motion of vacancy, $\Delta S_{sf}$ is the increase in entropy when forming a vacancy, $Q_{vm}$ is the energy barrier for vacancy motion and $Q_{sf}$ is the activation energy for vacancy formation.
1.5.3 Evolution of excess vacancy concentration

Calculation of the excess vacancy concentration is required for the solution of Eq.16. The generation and motion of vacancies are coupled with the recovery of dislocations and diffuse solute atoms. The model presented here is only concerned with mono-vacancies. When a crystal is retained a sufficient time at a given temperature, an equilibrium level of vacancies is reached. Deforming the material or changing the temperature generates the excess vacancies. The effect of excess vacancies on the diffusion can be written as [77]:

\[
\dot{c}_v^e = \dot{c}_v - \dot{c}_v^e = \left( \frac{\sigma_y b}{Q_{vf}} + \zeta \frac{c_j}{4b^2} \right) \frac{Q_{ef}}{b} \dot{\varepsilon}^p - D_{im} \left( \frac{1}{s^2} + \frac{1}{g^2} \right) \left( c_v - c_v^e \right)
\]

(18)

The stress \(\sigma_y\) in Eq.18 is equal to the flow stress during a plastic deformation, the factor \(\chi \sigma_y \dot{\varepsilon}^p\) is the fraction of the mechanical work needed for the vacancy formation, \(Q_{vf}\) is the activation energy for forming a vacancy, \(Q_0\) is the atomic volume and \(c_j\) is the concentration of thermal jogs. The parameter \(\zeta\) describes the neutralisation effect by vacancy emitting and absorbing jogs, \(c_v^e\) is the equilibrium concentration of vacancies at a given temperature, \(c_v\) is the non-equilibrium vacancy concentration and \(D_{im}\) is the vacancy migration. The equilibrium concentration of vacancies at a given temperature is [76, 78]:

\[
c_v^e = \exp \left( \frac{\Delta S_{vf}}{k} \right) \exp \left( - \frac{Q_{ef}}{kT} \right)
\]

(19)

The rate of change in the vacancy equilibrium concentration is related only to the temperature change

\[
\dot{c}_v^e = c_v^e \left( \frac{Q_{vf}}{kT^2} \right) \dot{T}
\]

(20)

Details of the model for vacancies and diffusion are to be found in Militzer et al [77] and Lindgren et al [72].

1.5.4 Stress-Update and Optimization of the model

The radial return operator for the integration of constitutive equations is used for updating the flow stress. are commonly used [79, 80]. The computation of the increment of effective plastic strain, which fulfils the consistency condition, requires calculation of the yield stress and hardening modulus for the current iteration of the plastic strain and internal state variables. The evolution of the internal state variables is governed by the coupled differential equations. The rate of total change in immobile dislocation density is derived by (21) and the rate of change in vacancy concentration is derived by (22):

\[
\dot{\rho}_i = \left( \frac{m}{b} \frac{1}{\Lambda} - Q \rho_i \right) \dot{\varepsilon}^p - 2 c_j D_v c_v \frac{Gb^4}{kT} \left( \rho_i^2 - \rho_i^2 \right)
\]

(21)
\[
\dot{\varepsilon}_v = \left[ \chi \frac{\Omega_0}{Q_{ef}} (\sigma_e + \sigma^v) + \frac{c_j}{4b^2} \frac{\Omega_0}{b} \right] \dot{\varepsilon}^{pl} - D_{\text{vm}} \left( \frac{I}{s^2} + \frac{I}{g^2} \right) (c_v - c_v^{eq}) + c_v^{eq} \left( \frac{Q_{ef}}{T^2} \right) T
\]  

(22)

Once the dislocation density and vacancy concentration are known, the hardening modulus and the flow stress can be evaluated. During the increment iteration the plastic strain rate is assumed constant. The hardening modulus in the incremental form is given by:

\[
H' = \frac{d\sigma_e}{d\varepsilon^{pl}} = \frac{\partial \sigma}{\partial \varepsilon} \left( \frac{\partial \rho_{\theta}}{\partial \varepsilon^{pl}} + \frac{\partial \rho_{\theta}}{\partial c_v} \frac{\partial c_v}{\partial \varepsilon^{pl}} \right) + \frac{\partial \sigma^v}{\partial \varepsilon^{pl}}
\]

(23)

The parameters for the model can be obtained by using an in-house Matlab based toolbox. And then, a physical based model for the evolution of flow stress of alloy depending on temperature range, strains and strain rates should be developed. One set of experiments should be used for model calibration and another more complex set of tests for its validation.
CHAPTER 2

RESEARCH APPROACH

CHAPTER INTRODUCTION

This chapter describes the research methodology used in this thesis. The purpose and aim of the work are first described. It is then followed by description of material used and experimental procedure.

2.1 PURPOSE AND AIM

This work constitutes an attempt to correlate the microstructural features (e.g. SDAS, grain size and intermetallic phases) and the mechanical behaviour of as-cast AZ91D alloys at temperatures from room temperature up to 190°C. It is then followed by adaption and optimization of a physically based model which enables prediction of the flow stress behaviour of the alloy covering different microstructures. As a benefit, the optimized parameter of the model can be employed in FEMs simulation of the behaviour of cast components at different working temperatures.

2.2 RESEARCH DESIGN

2.2.1 Research approach

The research approach applied in this thesis is the traditional positivist research design suggested by Williamson [81]. It is schematically illustrated in Figure 5. The positivist approach represents the traditional approach to natural sciences; it is often related to experimental research designs and quantitative data and is a rather linear and fixed research design [81]. In this thesis the approach starts with a definition of the topic of interest. The literature review of the research area was performed. And that, together with a work on the theory behind mechanical behaviour and microstructural features of magnesium alloys formed the basis for a more detailed definition of the research problem and the research questions and a definition of the desirable physical modelling process. Now, a hypothesis is created and research is performed to collect data, which is analysed and interpreted to see if the hypothesis is supported. This leads to a framing of general laws. In this work, to analyses the method and the effects of its parameters, different sets of numerical experiments were then defined, performed and evaluated. Eventually, the conclusion of the result thus made.

First step in information gathering process is to plan the search. Then, the search is performed and all results of possible relevance to the problem are retrieved and evaluated. If a record is
still considered relevant after the evaluation, it is saved and managed. Based on the results of the search, the search plan may need to be modified and some or all of the steps of the search process repeated. If no new relevant records are retrieved, the search process may be considered completed. In the analysis of the problem, the topic of interest was defined as “correlation between mechanical properties and microstructural features of AZ91D”. The scope of the literature review was divided into three parts; the first part concerns itself with manufacturing technique and microstructural features which then provide important contributions to the mechanical behaviour in as-cast AZ91D. The third part compares different modelling methods used for modelling of the mechanical behaviour of AZ91D alloys. The information resources used were online databases (mainly ScienceDirect1, Scopus2 and SpringerLink3, but also e.g. journal specific websites and e-books) and library resources (books and journals).

**Figure 5. Schematic illustration of the research approach for present work.**

### 2.2.2 Research questions

The industrial partner of this project is Husqvarna AB and the studied component is crank case of Husqvarna chainsaw. The crank case is being made of AZ91D and the in service temperature of this component is from RT to 190°C. In order to have a sustainable optimization design of cast component the closed chain of design (Figure 6) need to be fulfilled. In present work, part of this chain has been defined as study scope. The first step is manufacturing technique followed by microstructure and mechanical characterization. The focus will be then on the correlation between the microstructural features and the mechanical behaviour of the alloy. The logic of current research activities (mechanical performance during use) and future work (mechanical performance in process and alloy development) are presented in Figure 7. Several research questions have been raised and answered fulfilling each step. The main research questions are listed below and subsequently are addressed in the indicated supplements:

- **Research question 1:** *(Supplement I & III)*
  What is the effect of microstructure (size, morphology and distribution of different phases) on mechanical behaviour of as-cast AZ91D alloy?

- **Research question 2:** *(Supplement II)*
  What is the correlation between microstructural features and thermal expansion of the alloy?

- **Research question 3:** *(Supplement IV)*
  Is it possible to extend a dislocation density based model to predict the flow stress of as-cast AZ91D at temperature range from RT to 190°C?
Research question 4: (Supplement IV)
Does the model have sufficient validity for different as-cast microstructures?

Figure 6- The closed chain of design optimization sustainability.

Figure 7. Schematic illustration research activities of the project.
2.3 MATERIAL AND EXPERIMENTAL PROCEDURE

2.3.1 Materials
Commercial ingots of AZ91D alloy with composition shown in Table 3 was used as the base alloy. To minimize the amount of oxides films and inclusions during casting ingots were melted in mild steel crucibles under vacuum filling process and gas protection of dry air with 0.5 % SF6, see Figure 8. The melt was sucked up to the mild steel rods of 1000 mm long. The solidified rods then were cut into 170 mm long bars and re-melted in Bridgman furnace under protective gas (0.5% SF6 in dry air). The melt was kept for 25 minutes at 650°C to fully melt. The cooling rate of the specimen could controlled by drawing rate of the furnace. In this study, to generate different microstructures commonly found in slow cooled sand cast materials up to rapidly cooled high pressure die cast materials, furnace pull-rates from 0.3 mm/s to 6 mm/s were used. The schematic of Bridgman furnace device used in experiments is shown in Figure 9.

Table 3 -The chemical composition of the AZ91D alloy (wt.%)

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<tr>
<th></th>
<th>Mg</th>
<th>Al</th>
<th>Zn</th>
<th>Mn</th>
<th>Fe</th>
<th>Ni</th>
<th>Cu</th>
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<tr>
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Figure 8. Schematic drawing of vacuum filling device used in the experiments.
2.3.2 Mechanical testing
Tensile bars were prepared according to ASTM B577 [82] and tested from room temperature (RT) up to 190°C, with strain rates ranging from $10^{-4}$ up to $10^{-1}$/s. A ZWICK-ROLL™ Z100 Laser extensometer was used to measure strain. The geometry of the test specimen is shown in Figure 10. The tensile test schedule is shown in Table 4 together with the results. To facilitate an effective optimization the number of data point were reduced using a specially devised MATLAB™ code.

2.3.3 Microstructure characterization
Samples were prepared following standard procedures for microstructural characterization [83]. Sectioned samples were etched at room temperature using 10 ml HF (48%) for 1-2s to etch the Mg$_{17}$Al$_{12}$ phase [84]. It should mentioned that all optical characterization collected from at least five images per sample for assuring the internal validity.

The SDAS was quantified by identifying and measuring small groups of well-defined secondary dendritic arms on the optical micrographs. The value of SDAS was then practically determined using $SDAS = L/n$, where $L$ is the length of the line drawn from edge to edge of the measured arms, and $n$ is the number of dendritic arms. The mean values of SDAS for each sample are the average of more than 50 dendrite arms.

To assess actual grain size and subcell size of samples before and after deformation EBSD technique was employed with scanning step size of 0.8 µm. Low angle grain boundaries (LAGBs) having misorientations between 2° and 15° were depicted as white lines, and high
angle grain boundaries (HAGBs) with misorientations greater than 15° as black lines. In order to minimize errors in measurement, misorientations below 2° were not considered. Grain size on an image was measured with "Planimetric" method described by ASTM E112 [85] where the grain size determined by calculating the number of grains per unit area. The kernel average misorientation (KAM) maps were retrieved directly from EBSD data.

The area fraction of phases was measured using Olympus Stream Motion v. 1.8, based on image contrast. Following ASM specialty handbook of magnesium and magnesium alloys [86], the shape factor of Mg17Al12 were obtained from $4\pi A/p^2$ relation, where $A$ and $p$ are area and perimeter of particles in a cross-section, respectively. A perfect globule is characterized by a shape factor of 1.

2.3.4 Coefficient of thermal expansion

The coefficients of thermal expansion (CTE) of AZ91D was determined following of DIN 51045-1 [87] standard. A NETZSCH 402PC dilatometer at 5 K/min heating rate and with 1.2 min$^{-1}$ Ar gas flow was employed. Displacement of the 12 mm long AZ91 samples as a function of temperature (50°C to 190°C) was measured using an alumina probe. CTE was then calculated from the displacement data.

2.3.5 Differential scanning calorimetric

Disk shaped samples having 42.1 mg weight were prepared for differential scanning calorimetric (DSC) measurements with five times temperature cycle from RT to 190°C. Heating rate was 5 K/min. and cycles included static or dynamic temperature steps in order to achieve desired temperature. The measurements were carried out with NETZSCH 404 C calorimeter and following DIN 51007:1994-06 [88].
CHAPTER 3

SUMMARY OF RESULTS AND DISCUSSION

CHAPTER INTRODUCTION

In this chapter, the main results of the appended supplements are summarized and discussed. The supplements address the research questions to various degrees.

3.1 MICROSTRUCTURE CHARACTERIZATION

The gradient solidification set-up was utilized to manufacture samples having desirable microstructure. Analysis of EBSD orientation mapping of all samples revealed the random orientation of the grains suggesting that the sample manufacturing did not result in any texture and grains have no preferred directionality. Hence, the assumption of manufacturing samples with isotropic microstructure confirmed.

The typical EBSD maps for two different solidification speed before deformation is shown in Figure 11. LAGBs between $2^\circ$ and $15^\circ$ were depicted as white lines, HAGBs $> 15^\circ$ as black lines. It should be mentioned that the possible twin boundaries are resulted from mechanical polishing. Grain sizes of all samples obtained from different solidification speed are presented in Table 4.

It was clearly understood that increasing in drawing rate assisted to formation of finer SDAS, (see Figure 12 and Table 4) as well as formation of divorced eutectic (Figure 13). This results are in good agreement with literature findings [84, 89].

The observed equiaxed of fine and coarse microstructures of AZ91D (Figure 12-a and b) contain $\alpha$-Mg (white phase) surrounded by $\text{Mg}_17\text{Al}_{12}$ particles (dark phase). Intermetallic precipitated in different shapes at grain boundaries as well as inter-dendritic regions. The sizes of the $\text{Mg}_17\text{Al}_{12}$ particles are measured to be about several micrometres. For high solidification rate (6 mm/s) the range of microstructural scale for $\text{Mg}_17\text{Al}_{12}$ ranged from 0.8 $\mu$m up to 16.5 $\mu$m. For low solidification rate (0.3 mm/s) this range was from 1.3 $\mu$m up to 24.2 $\mu$m. The shape factor of $\text{Mg}_17\text{Al}_{12}$ for different solidification speed appeared to be independent of the solidification rate (almost 0.60 ± 0.2) but area fraction showed a significant variation. This fairly large scatters suggests the possibility of a continuous network of particles at the intragranular regions. Increasing the solidification speed results to lower area fraction of $\text{Mg}_17\text{Al}_{12}$.
intermetallic. The area fraction of all tested samples are presented in Table 4. In agreement with Nagasekhar et al. [90], measuring the connectivity of Mg$_{17}$Al$_{12}$ was not possible by optical nor by Scanning Electron Microscopy (SEM). The Mg$_{17}$Al$_{12}$ precipitated along triple junction grain boundaries and cross-section based analysis cannot capture the degree of their connectivity accurately.

Furthermore, the relationship between grain size and Mg$_{17}$Al$_{12}$ fraction was studied. In Figure 14 the Mg$_{17}$Al$_{12}$ fraction is plotted versus grain size and it is realized that there is a relation between the precipitated fraction and grain size. The samples displaying a higher fraction indicates a strong relation. The lower fraction samples also display a strong relation but with a significantly lower slope. The intersection between these two classes occurs around 9% Mg$_{17}$Al$_{12}$. The outlier showed a low Mg$_{17}$Al$_{12}$ fraction at a large grain size.

![IPF maps for illustrating grain orientation](image)

Figure 11. IPF maps for illustrating grain orientation. LAGBs between 2° and 15° were depicted as white lines, HAGBs > 15° as black lines (a) for furnace pulled at 6 mm/s with grain size of 93 µm ± 4.4 µm and (b) for furnace pulled at 0.3 mm/s with grain size of 254 µm ± 3.7 µm.
Figure 12. Optical micrograph illustrating SDAS (4.2 μm ± 1.2 μm) for samples drawn at 6mm/s, (b) Optical micrograph illustrating SDAS (25.0 μm ± 1.6 μm) for samples drawn at 0.3mm/s (Supplement I).

Figure 13. Illustration of the divorced eutectic and the Mg$_{17}$Al$_{12}$ fraction and morphology. (a) Mg$_{17}$Al$_{12}$ fraction of 7.1% ±0.4% with a shape factor of 0.60 ± 0.2 for samples drawn at 6mm/s, (b) Mg$_{17}$Al$_{12}$ fraction of 11% ±0.9% with a shape factor of 0.60 ± 0.2 for samples drawn at 0.3mm/s (Supplement I).

Figure 14. Fraction of Mg$_{17}$Al$_{12}$ versus grain size (Supplement III).
Table 4. Experimental conditions and mechanical results of relevant microstructures obtained from a variety of cooling rates between 0.3mm/s and 6mm/s. The (*) indicating the Runs selected to illustrate the modeling optimization results in Figure 26. The sample numbering is identified as: R(run number): sample replication number (total run replications).

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<td>190</td>
<td>0.0001</td>
<td>132</td>
<td>0.06</td>
<td>103.4±0.3</td>
<td>93.4</td>
<td>5.3</td>
<td>7.8</td>
</tr>
<tr>
<td>R7(5)</td>
<td>3</td>
<td>190</td>
<td>0.0001</td>
<td>132</td>
<td>0.06</td>
<td>103.4±0.3</td>
<td>93.4</td>
<td>5.3</td>
<td>7.8</td>
</tr>
</tbody>
</table>

3.2 MECHANICAL CHARACTERIZATION

Examples of typical stress-strain curves are shown in Figure 15 at room temperature and 190°C. In general there was scatter in the results as previously suggested by literatures [44, 48-50, 91, 92]. Figure 15 illustrates that the finer microstructure has lower yield strength than the coarser structure suggesting that there are other factors determining the yields strength than SDAS. On the other hand, the strain rate dependence at 190°C is as expected where a lower strain rate is showing less hardening than at a higher strain rate. In summary, Figure 15 illustrates some points i) the offset yield strength (Rp0.2) appears strongly dependent on the Mg17Al12 fraction, ii) at higher fraction of Mg17Al12 the offset yield strength (Rp0.2) also showed a higher dependence of strain rate, iii) the hardening appears higher for lower Mg17Al12 fraction and smaller SDAS, iv) a higher Mg17Al12 fraction appears to reduce the elongation to failure.

In order to understand the significance of the experimental parameters an Analysis of Variance (ANOVA) was made using the DesignExpert™ software. Table 5 shows analysis of variance of the offset yield strength (Rp0.2) for different factors and their interactions. The model in annotated view is labelled as “significant”. "F-value" column and associated probability
(“Prob>F”) shows that there is a very small probability, near 0.81% chance (P=0.0081), that the differences model terms (A, B, D and BD) could occur due to noise. Lack of fit is not significant which is relative to “Pure Error”. In other words, it appears at this stage the difference between terms of models is significant suggesting that the model is fit. Interesting to note is that temperature (A) and Mg_{17}Al_{12} fraction (D) are significant. Strain rate (B) is marginally significant but very close to significance. The interaction between the strain rate and Mg_{17}Al_{12} fraction (BD) is also marginally significant. Any suitable fit with SDAS (or C) in the analysis could not be obtained. The analysis confirmed that temperature reduce yield strength as expected. The result of yield strength model is shown in Figure 16. It reveals an interaction between two factors of Mg_{17}Al_{12} fraction and strain rate which effect on yield strength of specimens. It is conceived that at high strain rate the effect of Mg_{17}Al_{12} fraction on yield strength is more dominant. While, no significant effect was observed at lower Mg_{17}Al_{12} fraction. Higher strain rates leads to increased yield strength. It means that higher fraction of Mg_{17}Al_{12} results in increased initial strength of the alloy.

![Figure 15. Stress–strain curves for specimens deformed at a) Room temperature and b) 190°C and strain rate of 0.0001 1/s (Supplement III).](image-url)
Table 5. Analysis of variance of the $R_{p0.2}$ for different factors and their interactions with R-squared of 0.90 (Supplement I).

<table>
<thead>
<tr>
<th>Source</th>
<th>Sum of Squares</th>
<th>df</th>
<th>Mean Square</th>
<th>F-value</th>
<th>P-value Prob &gt; F</th>
<th>Significance</th>
</tr>
</thead>
<tbody>
<tr>
<td>Model</td>
<td>7162.69</td>
<td>4</td>
<td>1790.67</td>
<td>6.38</td>
<td>0.0081</td>
<td>Significant</td>
</tr>
<tr>
<td>A-Temp</td>
<td>1412.76</td>
<td>1</td>
<td>1412.76</td>
<td>5.04</td>
<td>0.0487</td>
<td>Significant</td>
</tr>
<tr>
<td>B-Strain rate</td>
<td>1382.93</td>
<td>1</td>
<td>1382.93</td>
<td>4.93</td>
<td>0.0507</td>
<td>Marginally significant</td>
</tr>
<tr>
<td>D- Mg$<em>{17}$Al$</em>{12}$ fraction</td>
<td>4122.91</td>
<td>1</td>
<td>4122.91</td>
<td>14.69</td>
<td>0.0033</td>
<td>Significant</td>
</tr>
<tr>
<td>BD</td>
<td>1190.40</td>
<td>1</td>
<td>1190.40</td>
<td>4.24</td>
<td>0.0664</td>
<td>Marginally significant</td>
</tr>
<tr>
<td>Residual</td>
<td>2805.77</td>
<td>10</td>
<td>280.58</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Lack of Fit</td>
<td>822.00</td>
<td>4</td>
<td>205.50</td>
<td>0.62</td>
<td>0.6641</td>
<td>Not significant</td>
</tr>
<tr>
<td>Pure Error</td>
<td>1983.77</td>
<td>6</td>
<td>330.63</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cor Total</td>
<td>9968.46</td>
<td>14</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Figure 16. Interaction of strain rate and Mg$_{17}$Al$_{12}$ fraction on yield strength. Centre line is the actual trend. Dash lines are confidence intervals (Supplement I)

Furthermore, the average value of offset yield strength ($R_{p0.2}$) obtained for tensile tests at room temperature and 190°C is plotted against area fraction of Mg$_{17}$Al$_{12}$ in Figure 17 a). It was revealed that the content of Mg$_{17}$Al$_{12}$ was strongly contributing to the offset yield strength of AZ91D, the higher the area fraction of Mg$_{17}$Al$_{12}$ the higher the $R_{p0.2}$ value. In addition, the linear regression analysis was carried out in order to quantify the relationship between the Mg$_{17}$Al$_{12}$ content and the $R_{p0.2}$ magnitudes. The analysis revealed that there is a different linear slope fit for Mg$_{17}$Al$_{12}$ content below 8.8% and above 11%. When the Mg$_{17}$Al$_{12}$ content is ≥11% the $R_{p0.2}$ at both RT and 190°C increases with sharper slope in comparison to lower content of Mg$_{17}$Al$_{12}$. Jarfors et al. [93] suggested that the intermediate connectivity of particles starts at 8% and by 11% particle fraction there is a full connectivity between particles. Hence, one can conclude
that by increasing the fraction of Mg$_{17}$Al$_{12}$ (above 11%), these particles start to impinge on each other, forming a network like a rigid scaffold. During tensile testing, the plastic deformation can only occur when the entire Mg$_{17}$Al$_{12}$ network is deformed. It seems reasonable to assume that this network should be able to withstand higher loads before it starts to deform, because it is difficult for the continuation of slip and dislocations to move across the Mg$_{17}$Al$_{12}$ network during deformation [94]. Hence, a higher fraction of Mg$_{17}$Al$_{12}$ leads to higher interconnectivity and a more rigid and harder material. Consequently, the offset yield strength of AZ91D alloy is mainly determined by the continuous network of Mg$_{17}$Al$_{12}$. The effect of SDAS and grain size on offset yield point showed no physical meaning. Hence, it is confirmed that there are other parameters that have effect on offset yield point of AZ91D rather than SDAS or grain size, see Figure 18 a).

Furthermore, analysing the elongation to failure, Figure 17 c), showed a clear reduction with an increasing Mg$_{17}$Al$_{12}$ fraction which is related to brittle nature of Mg$_{17}$Al$_{12}$. Above 10% Mg$_{17}$Al$_{12}$ the elongation to failure appears to be in the order of 4% with a fair scatter. Obviously, the SDAS and grain size have inverse relationship with elongation to failure, see Figure 18 c).

Once the material starts to yield, it is important that which parameters affect the stress-strain behavior. It was realized that Mg$_{17}$Al$_{12}$ content, SDAS and grain size have no obvious effect on fracture strength, $R_m$, of the material, see Figure 17 b) and Figure 18 b). However, as it was expected the temperature decrease the fracture strength. Furthermore, the hardening was defined as:

$$\frac{\Delta \sigma}{\Delta \varepsilon} = \frac{\sigma_F - \sigma_Y}{\varepsilon_F - \varepsilon_{YS}} \quad (24)$$

Table 6 shows the ANOVA results for hardening. The model is significant and there is only a 0.02% chance that a "Model F-Value" could occur due to noise. Lack of fit is not significant suggesting that the model is fit. Interesting to note is that only temperature (A) is significant. Surprisingly strain rate is not significant nor is SDAS (C) and Mg$_{17}$Al$_{12}$ fraction (D). What stands out is that the interaction between temperature and strain rate (AB) is marginally significant. In the evaluation Strain rate (B) is kept to keep model hierarchy intact. This suggests, not unexpectedly, that temperature dependent dislocation mobility has a dominant role in the deformation behavior of AZ91D. It should here, again, be noted that there is a fairly large scatter in data and the results should be seen as qualitative.
Figure 17. Variation of a) $R_{p0.2}$, b) fracture strength, and c) elongation to fracture with respect to fraction of $\text{Mg}_{17}\text{Al}_{12}$ at room temperature and 190°C and linear regression plot (Supplement III).
Figure 18. Variation of a) $R_{p0.2}$, b) fracture strength and c) elongation to fracture with respect to fraction of with respect to SDAS and grain size value at room temperature and 190°C (Supplement III).
Table 6. Analysis of variance of the hardening for different factors and their interactions with R-squared of 0.87 (Supplement I).

<table>
<thead>
<tr>
<th>Source</th>
<th>Sum of Squares</th>
<th>df</th>
<th>Mean Square</th>
<th>F value</th>
<th>P-value</th>
<th>Prob&gt;F</th>
</tr>
</thead>
<tbody>
<tr>
<td>Model</td>
<td>3.39</td>
<td>3</td>
<td>1.13</td>
<td>16.59</td>
<td>0.0002</td>
<td>Significant</td>
</tr>
<tr>
<td>A-Temp</td>
<td>0.66</td>
<td>1</td>
<td>0.66</td>
<td>9.65</td>
<td>0.0100</td>
<td>Significant</td>
</tr>
<tr>
<td>B-Strain rate</td>
<td>0.11</td>
<td>1</td>
<td>0.11</td>
<td>1.55</td>
<td>0.2393</td>
<td>Hierarchy</td>
</tr>
<tr>
<td>AB</td>
<td>0.28</td>
<td>1</td>
<td>0.28</td>
<td>4.12</td>
<td>0.0673</td>
<td>Marginally significant</td>
</tr>
<tr>
<td>Residual</td>
<td>0.75</td>
<td>11</td>
<td>0.068</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Lack of Fit</td>
<td>0.32</td>
<td>5</td>
<td>0.063</td>
<td>0.88</td>
<td>0.5478</td>
<td>Not significant</td>
</tr>
<tr>
<td>Pure Error</td>
<td>0.43</td>
<td>6</td>
<td>0.072</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cor Total</td>
<td>4.14</td>
<td>14</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Figure 19 illustrates the interaction between temperature and strain rate on hardening. It is shown that temperature primarily contributes to softening at low strain rate and at 0.1 1/s the hardening effect is rendered temperature independent. The fact that there was no influence from the Mg17Al12 fraction on the hardening suggests that there is none or little hardening effect from the Mg17Al12 particles and that there is only effects from glide and climbing as more glide systems are engaged in the α-phase.

![Figure 19. Interaction of temperature and strain rate on hardening. Center line is the actual trend. Dash lines are confidence intervals (Supplement I).](image-url)

To study more about the effect of SDAS on rate of hardening an apparent toughness index was calculated as Rm*εf and plotted against SDAS, Figure 20. It was realized that hardening rate in the tensile tests appeared to be higher for smaller SDAS while no such relation could be seen for the ultimate tensile strength. Analyzing the influence of SDAS as apparent toughness showed a strong inverse relation to SDAS. It should also be noted that temperature did not show significant effect and all measured values fall on the same line.
Figure 20. Apparent toughness as function of SDAS at both room temperature and at 190°C (Supplement III).

### 3.3 COEFFICIENT OF THERMAL EXPANSION

Figure 21 shows the relative elongation of alloy from 323 to 423 K (50°C to 150°C) for five cycles. It can be seen that the cast alloy with low fraction of intermetallic expanded more than alloy containing higher intermetallic phase fraction.

Figure 21. Relative elongation of AZ91D cast alloy containing different level of intermetallic fraction (Supplement II).
The coefficient of thermal expansion (CTE) at a particular temperature is calculated as follows:

\[ \alpha = \frac{\partial }{\partial T} \left( \frac{\Delta l}{l} \right) \] (25)

where \( l \) is the actual length of specimen at \( T \). The mean linear thermal expansion coefficient can be derived as follows:

\[ \bar{\alpha} = \frac{l}{l_0} \left( \frac{\Delta l}{\Delta T} \right) \] (26)

where \( l_0 \) is the initial specimen length, \( \Delta l \) is the change in length over a temperature interval \( \Delta T \).

The differential linear thermal expansion coefficient of AZ91D for different microstructure in the temperature range of 323 to 420 K is represented in Figure 22. It is indicated that the value of CTE increases with increasing temperature. In the beginning of temperature range of 323 to 348 K the rate of thermal expansion increased faster, which can be attributed to the residual stress. Residual stress due to the phases in and around grains is released by increasing temperature \([95]\).

It is revealed that CTE values for sample with lower fraction of Mg_{17}Al_{12} are higher than the value for samples with higher fraction of intermetallic (see Figure 22). As it was showed earlier by optical microstructure investigation the fast solidification speed (6 mm/s drawing rate) has lower amount of intermetallic in microstructure (7.1±1.1%). Hence, it is reasonable to assume that there is limited interconnectivity of intermetallic in this microstructure. This assumption is in a good agreement with the fact indicated by Jarfors et al.[93] that intermediate connectivity of particles starts at 8% and by 11% particle fraction there is a full connectivity between particles. Hence, one can assume that the effective CTE upper bound (when there is a limited particle connectivity) is suitably equals to CTE value for microstructure with low level of intermetallic fraction. The upper bound of linear thermal expansion coefficient, \( \bar{\alpha}_{\text{eff}} \), of an isotropic two-phase composite can be obtained by Eq.27, and lower bound, \( \bar{\alpha}_{\text{eff}} \), Eq.28, can be obtained if indices 1 and 2 are interchanged \([96]\).

\[ (\alpha_{\text{eff}})_{\text{up}} = \alpha_2 - f_2(\alpha_2 - \alpha_1) \frac{K_2(3K_2 + 4G_2)}{K_1(3K_1 + 4G_1) + 4f_2G_2(K_1 - K_2)} \] (27)

\[ (\alpha_{\text{eff}})_{\text{low}} = \alpha_1 - f_1(\alpha_1 - \alpha_2) \frac{K_1(3K_1 + 4G_1)}{K_2(3K_2 + 4G_2) + 4f_1G_1(K_2 - K_1)} \] (28)

where, \( \alpha, f, K \) and \( G \) are linear thermal expansion coefficient, phase fraction, bulk modulus and shear modulus of phase 1 and phase 2 respectively. In Eq.27 indices 1 is representing intermetallic properties and 2 is representing matrix properties. While, in Eq.28 the indices 1 and 2 are representing matrix and intermetallic properties respectively.
Figure 22. Illustration of CTE for AZ91D cast alloy applying 6 mm/s and 0.3 mm/s drawing speed a function of temperature. The error bars are 95% confidence intervals. The line shows the upper bound fit for calibration of AZ91D thermal expansion coefficient considering no connectivity between intermetallic. The upper bound fit obtained from actual CTE value by 6 mm/s drawing rate leading to low fraction of intermetallic (supplement II).

The effective CTE upper bound fit is shown in Figure 22. If in Eq. 27 the \( (\alpha_{\text{eff}})_{up} \) equals to actual experimental value of linear thermal coefficient obtained for 6 mm/s drawing rate (microstructure with lower value of intermetallic), and having value of \( \alpha_1, K_1, G_1 \) and \( f_1 \) corresponded to CTE, bulk, shear and area fraction of intermetallic the \( \alpha_2, K_2, G_2 \) corresponding to matrix values are obtainable. The temperature dependency of intermetallic elastic modulus (bulk and shear modulus) were applied after work of Zhang et al. (2010) [97] where the elastic stiffness are obtained from First-principles calculations based on the stress–strain method. For more detail the reads are referred to the original reference [97]. Accordingly, the actual matrix’s CTE value of the upper bound fit as a function of temperature can be derived as a model with second order equation. The graphs showing the changing of CTE for matrix and intermetallic are present in Figure 23. Furthermore, the temperature dependency of bulk modulus and shear modulus for matrix was derived in the same way and compared with the available results for intermetallic modulus [97]. The elastic modulus of matrix and intermetallic are represented in Figure 24.

The calculated values of CTE, bulk and shear modulus for matrix and intermetallic were applied in order to obtain corresponding CTE lower bound of AZ91D with high fraction of intermetallic. The lower bound of linear thermal expansion coefficient \( (\alpha_{\text{eff}})_{\text{low}} \) of an isotropic two-phase composite can be obtained by Eq.28. In Eq. 28 \( \alpha_1, K_1, G_1 \) and \( f_1 \) are matrix value for CTE, bulk modulus, shear modulus and phase fraction respectively and \( \alpha_2, K_2 \) and \( G_2 \) are corresponding to intermetallic CTE and stiffness modulus, accordingly the value of CTE lower bound was obtained. Experimental results for this alloy including correlated upper and lower bound of CTE are presented in Figure 25.
Figure 23. Illustration of temperature dependency of thermal expansion coefficient of matrix phase and intermetallic phase. Matrix CTE value are obtained using the CTE upper bound fit. Intermetallic values are obtained after reference [97] (supplement II).

Figure 24. Comparing the temperature dependency of bulk and shear modulus for matrix phase of AZ91D and intermetallic (Mg_{17}Al_{12}) as a function of temperature (supplement II).

The results strongly confirm the idea that there is a correlation between the connectivity of intermetallic in the microstructure and the thermal expansion of the alloy. It is revealed that higher fraction of intermetallic phase results in decreasing CTE of the alloy. By increasing the fraction of intermetallic, Mg_{17}Al_{12} particles start to impinge each other and forming a network.
The CTE of the alloy results from interaction of CTE of matrix and intermetallic phase. Thermal expansion coefficient of intermetallic is lower than matrix leading to less expansion by increasing temperature. Consequently, the material with higher fraction expands less than alloy having a lower fraction of Mg$_{17}$Al$_{12}$.

In conclusion, the assumption was that AZ91D CTE upper boundary corresponds to material with lower amount of Mg$_{17}$Al$_{12}$ and therefore having disjointed one. The assumption was then validated from the mathematical point of view where the CTE upper boundary was perfectly fitted by experimental results for high fraction of Mg$_{17}$Al$_{12}$. Hence, having the network of intermetallic in the slow speed solidification samples which was previously realized through mechanical behavior study is supported by thermal expansion behavior.

![Figure 25](image-url)

**Figure 25.** The upper and lower bound of thermal expansion for AZ91D cast applying 0.3 mm/s furnace drawing rate. The experimental results are shown with 95% confidence interval (supplement II).

### 3.4 PHYSICAL MODELLING OF AZ91D FLOW CURVES

#### 3.4.1 Calibration of the model

The calibration parameters used in the present model are collated in Table 7. The Poisson’s ratio was assumed constant, $\nu = 0.35$ [86]. The tensile test revealed the value of Young’s modulus (E) at room temperature is almost 45 GPa, which linearly decreased with increasing temperature following Eq.10 which is obtained after reference [98] for temperature below 400°C. Besides, shear modulus can be obtained knowing the Young’s modulus as [86]:

$$E = -0.0208T + 50.516$$ \hspace{1cm} (29)

For hexagonal close-packed (hcp) metals, in which the basal slip is activated at a low stress, the Taylor factor has been suggested to be 6.5 [99]. Caceres et al. [100] estimated the Taylor factor for random Mg polycrystals to be 4.5. For polycrystalline Mg having a specific texture that
inhibit basal and prismatic slip while favoring pyramidal polyslip, the Taylor factor is between 2.1 and 2.5. On the other hand, for the AZ91D alloy, using EBSD technique, Yuan et al. [101] obtained value of 2.3 and 2.1 for processing direction (PD) and transverse direction (TD), respectively. Other reports [94, 102] assumed Taylor factor of 3 while basal slip defined as slipping system. In this work, the Taylor factor was considered as 3 which is the intermediate value from literatures [94, 100-102]. Besides, it is stated that in the temperature range of RT to 190°C Taylor factor can be assumed as a constant value, since the same slip systems are active during deformation [18]. However, if there is a possibility of increasing number of slip systems, the temperature dependency of $K_c$ could compensate the choice of having a constant Taylor factor. An increase of $K_c$ at elevated temperatures would be equivalent to a decreased Taylor factor due to increasing number of slip systems.

The increase in entropy corresponding to the formation of vacancy, $\Delta S_{vf}$, (related to recovery by climb [72]) is in order of $0.5k-2k$ ($k=\text{Boltzmann's constant}$) for temperatures higher than the Debye temperature. In this study it was assumed $\Delta S_{vf}=k$ [77].

The Mg lattice self-diffusion activation energy is 135 KJmol$^{-1}$ [65] and activation energy for diffusion of Al in Mg is 143 kJmol$^{-1}$ [103]. The average Q was assumed to be 138 kJ mol$^{-1}$ [47]; a value that is reasonably close to the activation energy for self-diffusion in Mg. The activation energy for vacancy migration, $Q_{\text{vm}}$, was reported to be in the range from 38 kJ mol$^{-1}$ to 76 kJmol$^{-1}$ [104]. Besides, the activation energy for vacancy formation, $Q_{vf}$, was reported to be in the range from 55 kJ mol$^{-1}$ to 85 kJ mol$^{-1}$ [104, 105]. This study adapted data from Frost et al. [47] with $Q_{vf} = 81$ kJ mol$^{-1}$ and $Q_{\text{vm}} = 54$ kJ mol$^{-1}$. The value for initial vacancy diffusivity ($D_{\text{vm}}$) corresponds to pure Mg [65].

3.4.2 Optimized model parameters

The parameters optimized and their values are collated in Table 8. The comparisons between typical experiments and predictions of the flow stress curves are plotted in Figure 26. Although the model applied for all test conditions, given in Table 4 only selected curves (identified in Table 4 by *) were plotted here. Markers are experimental data and lines are the flow stress model prediction. The overall fitting showed good agreement between the computed model and measured tensile stress-strain curves especially for deformation at temperature below 190°C. The model at temperature 190°C for some test conditions showed a slight deviation.

It should be mentioned that the model was built up on assumption of having single-phase material, containing precipitation hardening particles. Moreover, as it was mentioned earlier, a correlation with SDAS was achieved through the Eq.13 where SDAS size has influence on the initial mean free path, before the subcell structure will be the main hinder for dislocation motion. Hence, the range of parameters shown in Table 8 is applicable for all microstructures.

The parameter, $\Delta F$, is characterized as the strength of a single obstacle and $\Delta F = \Delta f_0 G b^2$ [106]. Obstacles were classified by their strength as suggested by Frost et al. [47]. The optimized $\Delta f_0$ value was obtained in the range of 0.2 at room temperature, which increased to 0.5 at 190°C. In this temperature range the obstacles are precipitates that can be characterized as week suggesting that the intermetallics have only limited effect on hardening [47].
Table 7. AZ91D parameters for model calibration (supplement IV).

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Notation</th>
<th>Value</th>
<th>Dimension</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Boltzmann’s constant</td>
<td>$k$</td>
<td>$1.38 \times 10^{-23}$</td>
<td>J/°C</td>
<td>---</td>
</tr>
<tr>
<td>Melting temperature</td>
<td>$T$</td>
<td>470-595</td>
<td>°C</td>
<td>---</td>
</tr>
<tr>
<td>Taylor factor</td>
<td>$m$</td>
<td>3</td>
<td>---</td>
<td>intermediate value from references [94, 100-102]</td>
</tr>
<tr>
<td>Atomic volume</td>
<td>$\Omega_0$</td>
<td>$2.33 \times 10^{-28}$</td>
<td>m$^3$</td>
<td>[65]</td>
</tr>
<tr>
<td>Burger’s vector</td>
<td>$b$</td>
<td>$3.21 \times 10^{-10}$</td>
<td>m</td>
<td>[102]</td>
</tr>
<tr>
<td>Young’s modulus at RT</td>
<td>$E$</td>
<td>45</td>
<td>GPa</td>
<td>[86]</td>
</tr>
<tr>
<td>Shear modulus</td>
<td>$G$</td>
<td>17</td>
<td>GPa</td>
<td>[86]</td>
</tr>
<tr>
<td>Poisson’s ratio</td>
<td>$\nu$</td>
<td>0.35</td>
<td>---</td>
<td>[86]</td>
</tr>
<tr>
<td>Activation energy for forming a vacancy</td>
<td>$Q_{vf}$</td>
<td>81</td>
<td>kJmol$^{-1}$</td>
<td>[65]</td>
</tr>
<tr>
<td>Energy barrier for vacancy migration</td>
<td>$Q_{vm}$</td>
<td>54</td>
<td>kJmol$^{-1}$</td>
<td>[65]</td>
</tr>
<tr>
<td>Self-diffusion activation energy</td>
<td>$Q_s = Q_{vf} + Q_{vm}$</td>
<td>135</td>
<td>kJmol$^{-1}$</td>
<td>The average from references [65, 103]</td>
</tr>
<tr>
<td>Initial vacancy diffusivity</td>
<td>$D_{vac}$</td>
<td>$1.0 \times 10^{-4}$</td>
<td>m$^2$s$^{-1}$</td>
<td>[47, 65]</td>
</tr>
<tr>
<td>Reference strain rate</td>
<td>$\dot{\varepsilon}_{ref}$</td>
<td>$10^6$</td>
<td>s$^{-1}$</td>
<td>[72]</td>
</tr>
<tr>
<td>Entropy increased when creating a vacancy</td>
<td>$\Delta S_{vf}$</td>
<td>$k = \text{Boltzmann’s constant}$</td>
<td>J/K</td>
<td>[77]</td>
</tr>
<tr>
<td>Friction of mechanical work for vacancy formation</td>
<td>$\chi$</td>
<td>0.1</td>
<td>---</td>
<td>[77]</td>
</tr>
<tr>
<td>Formation energy of thermal jogs</td>
<td>$Q_i$</td>
<td>$6.88 \times 10^{-20}$</td>
<td>J</td>
<td>[77]</td>
</tr>
<tr>
<td>Neutralization effect by vacancy emitting and absorbing jogs</td>
<td>$\zeta$</td>
<td>10</td>
<td>---</td>
<td>[77]</td>
</tr>
<tr>
<td>Initial immobile dislocation density</td>
<td>$\rho_{0i}$</td>
<td>$10^{13}$</td>
<td>m$^2$</td>
<td>[72]</td>
</tr>
</tbody>
</table>
The quantity \( \tau_0 \), as a short-range component in Eq. 10, is the strength needed in order to move the dislocation across the barrier in the absence of thermal energy [106]. Besides, this term is not only reflecting the strength but also density and arrangement of the obstacles. The \( \tau_0 \) is proportional to \( b/l \) where \( b \) is magnitude of burger vector and \( l \) is the obstacles spacing [47].

The obtained optimized \( \tau_0 \) showed slight decreasing trend by increasing temperature from room temperature up to 190°C. It can be concluded that by increasing temperature the obstacles space will increase and hence \( \tau_0 \) will decrease. As it is mentioned earlier in the current model optimized values for \( \Delta f_0 \) and \( \tau_0 \) obtained independent of microstructures. In order to verify that is a reliable result the effect of reinforcing particles during the hardening process for different SDAS must be studied. Hence, the differential scanning calorimetry (DSC) measurement was carried out in order to rule out non-equilibrium effects that may occur during heating from RT to 190°C. The samples were selected from the same samples which models were build up on (test conditions on Table 4 are identified by *). A negligible difference in the shape of the DSC signal between different materials were detected, which implied that there is no different phase precipitation at this temperature range Figure 27 and it can be concluded that the variation of \( \Delta f_0 \) and \( \tau_0 \) may not be influenced by changing microstructure.

The \( \Omega \) parameter describes the recovery process induced by dislocation glide and annihilation, see Eq.8. The optimized \( \Omega \) values were increased by raise in temperature. The formulations by Bergström et al. [70] and Estrin [71] showed that \( \Omega \) has an exponential relationship with temperature as \( \Omega = \Lambda \exp(-Q_{\text{dislocation glide}} / RT) \), where \( \Lambda \) is an empirical constant, \( R \) is the gas constant and \( Q_{\text{dislocation glide}} \) is the activation energy for dislocation glide. The corresponding activation energy for dislocation glide was calculated to be in the order of estimated 5 kJmol\(^{-1}\).

It is indicated that the activation energy for dislocation glide could be expected to be lower than the activation energy for forest dislocation cutting which is in the order of \( Gb^2/4\pi \) [107]. This value at room temperature equals to 26.48 kJmole\(^{-1}\) in present study.
Figure 27. DSC curves of the AZ91D alloy with different SDAS and Mg17Al12 fraction, shows that no phase transformation or other significant non-equilibrium events happen during heating.

\( K_c \) is a temperature dependent parameter related to the dislocation subcell diameter (Eq.14). Considering the mean free path from Eq.13, it can be concluded that a higher \( K_c \) value introduces larger subcell size and mean free path, resulting in less hardening effects. However, the subcell size and therefore, the mean free path are also strongly dependent on the rate of recovery of dislocation. Since, the rate of recovery increases with temperature this also lead to an increase of the subcell size.

Table 8. Optimized parameters for AZ91D as temperature dependent.

<table>
<thead>
<tr>
<th>T(°C)</th>
<th>20</th>
<th>89</th>
<th>133</th>
<th>190</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \tau_0 )</td>
<td>0.03817</td>
<td>0.0365</td>
<td>0.03657</td>
<td>0.026</td>
</tr>
<tr>
<td>( \Delta f_0 )</td>
<td>0.2985</td>
<td>0.2702</td>
<td>0.33746</td>
<td>0.5612</td>
</tr>
<tr>
<td>( K_c )</td>
<td>18.66</td>
<td>20.609</td>
<td>20.9291</td>
<td>24.368</td>
</tr>
<tr>
<td>( \Omega )</td>
<td>27.83</td>
<td>23.7823</td>
<td>24.3643</td>
<td>74.445</td>
</tr>
<tr>
<td>( \alpha )</td>
<td>Constant = 0.7</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>( c_\gamma )</td>
<td>Constant = 0.0029</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>( \Gamma )</td>
<td>Constant = 0.55</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>( q )</td>
<td>Constant = 1.68</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

3.4.3 Validation of the model using microstructural information

The model validity was confirmed by comparing the predicted dislocation density with the measured values from the tensile samples. The obtained values of computed dislocation density identical to three of the fitted curves (see Figure 26) are given in Figure 28. On the basis of the EBSD data, the kernel average misorientation (KAM), was mapped for measuring the local misorientations. KAM quantifies the average misorientation around a measurement point with respect to a defined set of second nearest neighbor points. Typical KAM maps are shown in
Accordingly, the geometrically necessary dislocations (GNDs) density were calculated as follows [108]:

$$\rho_{GND} = \frac{\phi}{bL}$$  \hspace{1cm} (30)

where $\phi$ is the rotation angle of the crystal which obtained from KAM data for the unit length of $L$ which is here equals to the double times of EBSD step size (2×0.4μm, since the KAM was obtained based on second nearest neighboring pixels) and $b$ is the magnitude of burgers vector. The calculation of the GND densities were made based on KAM data which defines the rotation angle, $\phi$ at each specified x and y point on the EBSD map. The 2nd neighbor rank, corresponding to a distance of 1.6 μm, is most suitable to obtain scatter-free information [108]. The typical corresponded GND densities maps of deformed samples are shown in Figure 28. The average $\rho_{GND}$ were calculated from EBSD data of 5 different scanned fields on each sample. The average $\rho_{GND}$ for specimens is comparable with final dislocation density value predicted by the model (Figure 28). The predicated values were in a good agreement with experimental results and thereby confirmed the model validation. It is worth mentioning that the calculated GND densities (Figure 28) were in a good agreement with available data for material with hcp crystallographic [109, 110] and Mg [111].

In addition, the model predication revealed that under all deformation conditions in the present study, the computed dislocation densities were increased by strain. Besides, corresponding to the effect of dynamic recovery, at typical strain rate, the immobile dislocation density increased by decreasing deformation temperature (Figure 29). Moreover, the model showed that dislocation density increased with increasing strain rate (see Figure 30). Dislocation density is determined from the balance between strain hardening and dynamic recovery [63]. Once the strain rate increases, a higher value of dislocation density at steady state is formed, which leads to achieve larger saturation stress level.
Tensile test condition

<table>
<thead>
<tr>
<th>Temperature</th>
<th>Strain rate</th>
<th>SDAS</th>
<th>R</th>
</tr>
</thead>
<tbody>
<tr>
<td>89°C</td>
<td>0.06 1/s</td>
<td>4.2μm</td>
<td>3(8)</td>
</tr>
<tr>
<td>133°C</td>
<td>0.03 1/s</td>
<td>6.4μm</td>
<td>5(5)</td>
</tr>
<tr>
<td>190°C</td>
<td>0.0001 1/s</td>
<td>12.5μm</td>
<td>5(5)</td>
</tr>
</tbody>
</table>

Average \( \rho_{GND} \) (m\(^{-2}\))

<table>
<thead>
<tr>
<th>Temperature</th>
<th>( \rho_{GND} ) (m(^{-2}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>89°C</td>
<td>( 3.17 \times 10^{14} )</td>
</tr>
<tr>
<td>133°C</td>
<td>( 2.85 \times 10^{14} )</td>
</tr>
<tr>
<td>190°C</td>
<td>( 0.50 \times 10^{14} )</td>
</tr>
</tbody>
</table>

Model \( \rho_{i} \) (m\(^{-2}\))

<table>
<thead>
<tr>
<th>Temperature</th>
<th>( \rho_{i} ) (m(^{-2}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>89°C</td>
<td>( 3.41 \times 10^{14} )</td>
</tr>
<tr>
<td>133°C</td>
<td>( 3.24 \times 10^{14} )</td>
</tr>
<tr>
<td>190°C</td>
<td>( 0.54 \times 10^{14} )</td>
</tr>
</tbody>
</table>

Figure 28. Typical KAM, corresponded GND maps and comparing experimental dislocation density with model predictions. EBSD data collected with respect to the 2nd neighbor rank, corresponding to a distance of 1.6 μm which is most suitable to obtain scatter free information.
Figure 29. Dependency of computed dislocation density on microstructure and temperature.

Figure 30. Dependency of computed dislocation density on strain rate.

The model predicated that the specimens deformed at room temperature have a higher dislocation density compared to 190°C (Figure 29). Hence, there is a high density of randomly-tangled dislocations at room temperature. Besides, it is well known that a high density of straight-edge dislocations and distribute of these dislocation will form subcells or dislocation cells [112]. The formation of a subcell, is accompanied by high stacking-fault energy, which aids cross-slip and thereby cell formation [113]. Subcell sizes can be predicated through Eq.14 for different test conditions, where the optimized K_c values are available in Table 8 at relevant temperature.
On the other hand, it should be mentioned that measuring the subcell size through LAGB measurements was not tried but the subcell structure of the maps do not seem to be fully developed, making estimation of subcell sizes difficult and not accurate. This was expected since the material is in as-cast conditions and the deformations are not as large as wrought conditions where the subcells are more easily detectable. A typical LAGB maps obtained from EBSD analysis is shown Figure 31.

![Figure 31. Typical LAGB maps for sample deformed under tensile test at 89°C and 0.0001 s⁻¹ strain rate.](image)

Model predication (Figure 29) reveled that annihilation of dislocation occurred when material deformed at 190°C and subsequently the predicted size of the cell is larger (2.7 ±0.5μm) than that predicted at room temperature (1.11±0.3 μm). It was reported that the dislocation density in the cell walls is considerably lower compared with that for room temperature [114]. Indeed, in the presence of cells, the immobile dislocations in the cell walls act as pinning obstacles, whilst the mobile dislocations within the cells can travel relatively without obstacle [112]. Under high-temperature deformation, the immobile dislocations are released across the cell from one cell wall to another localized on the opposite side and the material contains a large size cell, which allows the mean free path of the mobile dislocations to be wider [114]. This may lead to annihilation and decrease of the hardening rate. From the macroscopic viewpoint, this phenomenon can be confirmed by comparing the true flow stress–strain curves at room temperature and 190°C, already presented in Figure 26.
Moreover, the predicted subcell size and dislocation density of specimens will be assisted to understand the quantitative correlation between these microstructural aspects and the macroscopic behavior at different deformation temperatures. Figure 29 illustrates the computed immobile dislocation density at various temperatures and SDAS values versus strain for selective test conditions at strain rate of $0.0001 \text{ s}^{-1}$. The model presented that dislocation density content after deformation to the same strain at temperature from RT to 190°C is clearly smaller in material with coarser SDAS and higher intermetallic $\text{Mg}_{17}\text{Al}_{12}$ phase fraction. It seems that the grain boundaries and SDAS are the dominant obstacles against dislocation motion. Dislocations piled up against the grain boundary during tensile deformation and by increasing dislocation density their arrangement in microstructure network became more complex. In general, by preset results one can conclude that the flow stress can be linked quantitatively to the dislocation cell size. In other words, there is an inverse relationship between the cell size and the flow stress. The more the cell size decreases, the more the flow stress increases. This founding is in a good agreement with results from Wang et al. [114] Although, in current study a similar relationship has not observed for offset yield stress ($R_{p0.2}$). This can be correct assuming that there is no size effect; for which different behavior might be expected [115]. The model and experimental results has shown that the finer coarseness of microstructure gives higher deformation hardening and earlier investigation showed that a coarse microstructure yielded at higher offset yield strength.
CHAPTER 4

CONCLUSIONS

CHAPTER INTRODUCTION

This chapter summarizes the highlighted conclusions which were made from this study.

In this thesis, an attempt was made to correlate the microstructure and mechanical properties of AZ91D. Accordingly, different solidification speed were applied to generate variety of microstructure resembling different casting states from slow cooling to high cooling. Moreover, a physically based model was adapted in order to describe flow stress curves of the as-cast alloy including effect of microstructure and temperature. The following conclusions are highlighted from the work:

- **Sample manufacturing technique**
  Samples having controlled microstructures and showing isotropic microstructure, were prepared concluding that the gradient solidification set-up was a reliable technique for manufacturing.

  It was observed that both grain size and SDAS decreases with decreasing solidification speed. The shape factor of Mg$_{17}$Al$_{12}$ was independent of the solidification rate (0.60 ± 0.2). The large scatter of the shape factor of Mg$_{17}$Al$_{12}$ suggesting the possibility of a more or less continuous network However, Area fraction of Mg$_{17}$Al$_{12}$s increased with decreasing solidification speed.

  Moreover, it was realized that there is a relation between the precipitated fraction and the grain size. The samples displaying a higher fraction indicates a strong relation.

- **Correlation between microstructure and mechanical properties**
  It was observed that the offset yield point ($R_{p0.2}$) was strongly contributed to the Mg$_{17}$Al$_{12}$ content of the alloy and appears to be independent of the SADS value. It was clearly understood that two different groups of behavior could be observed. Both the magnitude of $R_{p0.2}$ and the dependence on the fraction of Mg$_{17}$Al$_{12}$ changed between 9 and 11%. The higher fraction of Mg$_{17}$Al$_{12}$ (≥11%) directed to a formation of rigid network which increased the offset yield point.
Furthermore, analysing the elongation to failure showed a clear reduction with an increasing \( \text{Mg}_{17}\text{Al}_{12} \) fraction. Above 10% of \( \text{Mg}_{17}\text{Al}_{12} \) the elongation to failure appears to be in the order of 4% with a fair scatter.

On the other hand, ultimate tensile strength did mainly depend on temperature and no statistically significant dependence on the grain size, SDAS nor fraction \( \text{Mg}_{17}\text{Al}_{12} \) could be established.

In addition, hardening rate appeared to be higher for smaller SDAS and analyzing the apparent toughness showed a strong inverse relation to the SDAS.

- **Correlation between microstructure and thermal expansions**
  The results demonstrated that the connectivity of intermetallic phase was contributing strongly to the thermal expansion of the alloy. The lower CTE bound suggests the possibility of \( \text{Mg}_{17}\text{Al}_{12} \) network formation for the high fraction of intermetallic (\( \geq 11\% \)). The CTE curve on the upper bound corresponded to low fraction of intermetallic (\(<8\% \)) and possibility of network formation was considered to be almost zero. The lower CTE of intermetallic causes the reduced sensitivity to increasing temperature. Hence, possibility of having a continuous network of intermetallic by increasing and impinging of the \( \text{Mg}_{17}\text{Al}_{12} \) particles is supported.

- **The modelling flow stress behaviour and validity**
  A dislocation density model was optimized for describing the flow stress of cast AZ91D with limited number of adjustable parameters which all have a transparent physical meaning. The presented model includes the effect of the microstructural scale on the deformation behavior of the alloy. It should mentioned that the model validity fairly confirmed by comparing the predicted dislocation density and the GND dislocation density obtained through the EBSD data. The uniqueness of current modeling is that the predicted tensile curves obtained with sufficient accuracy whereas the obtained optimized parameter are applicable for all microstructure range. This enables the usage of current model in future simulation coding where the mechanical properties predication of selected manufacturing technique is desirable.
CHAPTER 5

FUTURE WORK

CHAPTER INTRODUCTION
In this chapter the work prospects concerning better understanding of mechanical properties of the AZ91D is presented. This empirical approach founded from mechanical behavior characterization and physical modelling can be extended to the predicting of behavior in other magnesium alloy with network of Mg-Al. Hence, alloy development for high temperature usage is considered as one of the future work.

Compression flow behaviour and twinning
Understanding the behaviour of material under compression test condition would be beneficial. Besides, twinning as one of the deformation mechanisms under compression conditions should be considered as a topic of research interest. Accordingly, physical modelling for predicting compression flow curves is required.

Creep behaviour
Significant research is required to correlate the microstructure with creep properties of AZ91D. The controlling creep mechanisms of the alloy at different stress and temperature conditions should be understood. Modelling of the creep behaviour including the effect of microstructure is the significant of interest.

Stress relaxation behaviour
The behavior of screw fitted (fasted) in a component (crank case of hand chain saw) made of AZ91D at high temperatures can be studied. This part is joined together with an aluminum part and the fastener is a M5 bolt made of steel. These three parts form the model which is simulated using Finite Element Analysis (FEA) procedures. Creep behavior and thermal expansion are included in the simulation.

Residual stress in cast component
The quality of a manufactured product is sensitive to residual stress, and warping is a consequence of residual stress. If warping exceeds a given tolerance, product assembly can be hindered. In order to improve, it is important to understand the magnitude, distribution, and effects of process induced residual stresses (and warping). In order to determine how these residual stresses affect the final shape and performance of the product, a detailed structural analysis that accounts for the residual stresses is necessary. Abaqus can be used for simulation purposes.
**Alloy development**
Mg–Al–Ca alloys have shown good creep resistance, good castability and are cost effective. MRI 153M and MRI 230D with Al and Ca as the major alloying elements are capable for operations at temperature above 150°C. In future investigation, one can study and improve the creep-resistant of the MRI 153M and MRI 230D alloys. The effect of microstructure on mechanical properties and modelling of them need to be demonstrated.
REFERENCES

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[58] B. Babu, Physically based model for plasticity and creep of Ti-6Al-4V, Division of Material Mechanics, Luleå University of Technology, 2008.
APPENDED PAPERS

**Supplement I**
H. Dini, N. Andersson, A.E.W. Jarfors; Effects of Microstructure on Deformation Behaviour of AZ91D Cast Alloy. TMS 2014, 143rd ANNUAL MEETING & EXHIBITION February 16-20, San Diego, CA, USA.

**Supplement II**

**Supplement III**
H. Dini, N. Andersson, A.E.W. Jarfors; Effect of Mg_{17}Al_{12} content on mechanical properties of AZ91D cast alloy, submitted to the Scripta Materialia.

**Supplement IV**