5.1 Introduction

Superplasticity is the ability of a polycrystalline material to undergo very large uniform tensile deformation prior to failure, at a temperature well below its melting point. Typical values of the elongation to failure in uniaxial tension under superplastic conditions are of the order of a few hundred percent, and in some alloys can even exceed 1000%. Although initial experimental observations of superplasticity in metals date back to the 1920s, for a long time the phenomenon was mainly regarded as a laboratory curiosity [1]. However, research interests in superplasticity greatly increased in the 1960s [2,3], when it was demonstrated that in this regime metal sheets could easily be formed to complex shapes.

Superplastic forming (SPF) presents a potentially attractive alternative to other forming techniques. Due to the low flow stress characteristic of superplastic deformation, the tooling costs are minimal; blow forming is commonly used to form metal sheets under superplastic conditions. Furthermore, the exceptionally high ductility allows for a large freedom of design. At present, the main limitation towards mass application of SPF is the relatively low strain rate that is associated with conventional fine-structure superplasticity, which is discussed in Section 5.2.1. Forming of a typical component can take up to one hour at these strain rates. For this reason, SPF has mostly been restricted to the production of prototypes or small series of metallic components so far. However, research efforts are increasingly being directed towards new classes of superplastic materials, some of which exhibit superplastic behavior at considerably higher forming rates [4,5]. This so-called high-strain-rate superplasticity is expected to receive broad industrial interest and may replace existing forming techniques if such materials can efficiently be produced on a large scale. The present chapter is concerned with the high-strain-rate superplastic behavior of coarse-grained Al-Mg alloys, which are a promising candidate in this category.

The hallmark of superplastic deformation is a low flow stress $\sigma$ that shows a high strain-rate sensitivity $m$ as defined by
\[ \sigma = k \dot{\varepsilon}^n \]  
(5.1)

in which \( k \) is a constant and \( \dot{\varepsilon} \) is the strain rate. A high strain-rate sensitivity is necessary to stabilize the plastic flow so as to avoid necking during tensile deformation. The incipient formation of a neck leads to a local increase of the strain rate, which, in the case of a positive strain-rate sensitivity, leads to an increase of the flow stress in the necked region. If the strain-rate sensitivity is sufficiently high, the local flow stress increases to such an extent that further development of the neck is inhibited. Most common metals show a strain-rate sensitivity exponent lower than 0.2, whereas values around 0.3 or higher are needed to delay necking long enough to produce the strains characteristic of superplasticity. Besides a high strain-rate sensitivity, a low rate of damage accumulation (e.g. cavitation) is required to allow large plastic strains to be reached.

5.2 Superplasticity in fine-grained Al-Mg alloys

5.2.1 Conventional fine-structure superplasticity

The type of superplasticity most known and studied to date is that presented by fine-structured superplastic materials. Extensive reviews on this form of superplasticity are available [4,6-8]. The purpose of this section is to give an introduction to the microscopic mechanisms involved and the structural prerequisites for fine-grained superplastic alloys.

It is commonly accepted that fine-grain superplasticity relies on grain boundary sliding as the primary deformation mechanism. In order to avoid cavity formation, the grain boundary sliding must be aided by an accommodation mechanism that ensures rearrangement of the grains during deformation. This accommodation mechanism is generally believed to be the rate-controlling process. Numerous models have been proposed to describe the accommodation process [6], most of which involve diffusion-controlled dislocation motion, e.g. dislocation climb along the grain boundaries as shown in Figure 5.1. Provided that dislocation climb is rate-controlling, the strain-rate sensitivity exponent predicted by these models is equal to 0.5, which is in good agreement with experimental observations.

The diffusional nature of the accommodation process explains why conventional fine-grained superplasticity is restricted to fairly low strain rates. This is well illustrated by the characteristic sigmoidal shape of the curve that results when the flow stress is plotted versus the strain rate on a double-
logarithmic scale as depicted in Figure 5.2. The superplastic regime with $m \approx 0.5$, as indicated by region II in the figure, typically extends around $10^{-4}$ to $10^{-3} \text{ s}^{-1}$. The physical mechanisms responsible for the deformation in region I are not thoroughly understood. Traditionally, diffusional creep mechanisms are associated with such low strain rates; for fine-grained materials however, these mechanisms often do not correctly predict the low strain-rate sensitivity observed in this regime [7,10]. This has been attributed to the presence of a threshold stress [11]. Region III is associated with conventional dislocation creep and is well described by five-power-law constitutive equations [7] leading to a strain-rate sensitivity of $m \approx 0.2$. At the transition between regions II and III, two competing

![Figure 5.1: Ball-Hutchison model [9] of grain boundary sliding accommodated by dislocation movement [7].](image1)

![Figure 5.2: Typical sigmoidal strain-rate dependence of the flow stress for a conventional fine-grained superplastic material.](image2)
processes can control the deformation behavior: grain boundary sliding (region II) and dislocation slip (region III). Grain boundary sliding is promoted by small grain sizes and high temperatures; therefore, efforts to extend the superplastic regime towards higher strain rates are mostly made by refining the microstructure and improving the stability of the microstructure at higher temperature [12]. In general, a fine, equi-axed grain size of the order of 10 µm or smaller is required for the grain boundary sliding mechanism to operate and to be accommodated at the strain rates at hand. Since grain growth is very rapid in single-phase alloys at the temperatures where grain boundary sliding occurs, most superplastic alloys contain a finely dispersed distribution of second-phase particles to maintain a stable grain size throughout the deformation process.

5.2.2 Fine-grained superplastic Al-Mg alloys

Fine-grained Al-Mg alloys are commonly used in conventional SPF operations for light-weight applications. They present good corrosion resistance and weldability combined with a reasonable strength. At the superplastic forming temperature, the Mg is in solid solution and the resulting solute drag on gliding dislocations (also termed viscous glide) may benefit the strain-rate sensitivity of the alloys at strain rates both inside and outside the superplastic regime. Under superplastic conditions, viscous glide in the accommodating grains may become the rate-delimiting factor for grain boundary sliding. As illustrated in Figure 5.1, the accommodation by dislocation movement requires both glide and climb. Since these processes are interdependent, the slower of the two is rate-controlling. If (viscous) dislocation glide controls the accommodation rate, the strain-rate sensitivity exponent may increase from 0.5 up to unity [13]; experimental maximum values are reported to be between 0.5 and 0.7 [14-16]. At temperatures below or strain rates faster than superplastic conditions, viscous glide is commonly thought to be the primary deformation mechanism [15,17]. Viscous glide is often associated with a three-power-law regime, leading to a strain-rate sensitivity of $m \approx 0.3$. Although not as high as the values obtained under superplastic conditions, this value is significantly larger than shown in most metals during hot deformation. Al-Mg alloys therefore still show very good ductility even when grain boundary sliding is not predominant. The viscous glide mechanism will be more extensively discussed in the following sections as it forms the basis for the superplasticity of coarse-grained Al-Mg alloys investigated in the present work.
To maintain a stable grain size during deformation, fine-grained superplastic Al-Mg alloys contain alloying elements that form a fine dispersion of submicrometer-sized second-phase particles. The dispersoids should not coarsen appreciably at the superplastic forming temperature in order to remain capable of pinning grain boundaries and to avoid excessive cavity formation [18]. In the commonly used 5083 aluminum alloy, containing about 0.7% Mn, plate-like Al₆Mn-based particles accomplish this role [14,15,19]. In Al-Mg alloys containing Sc [20-22] or Zr [23-25], small coherent spherical Al₃Sc or Al₃Zr particles of typically less than 100 nm size are formed, which are particularly effective in inhibiting grain growth at higher temperatures. Moreover, such particles are thought to establish grain refinement in the initial stages of superplastic deformation by so-called continuous dynamic recrystallization [26]. In this process, the subgrains, which typically evolve during dynamic recovery, have their boundaries pinned by the particles. During deformation, the subgrain boundaries rearrange [27,28] and their misorientations continuously increase until they become typical of high-angle grain boundaries. The newly formed grains can subsequently participate in the grain boundary sliding mechanism. Thanks to this additional grain refinement and the stability of the coherent particles at high temperatures, Al-Mg-Sc and Al-Mg-Zr alloys can show superplastic properties at strain rates in excess of $10^2$ s⁻¹. However, the high costs of Sc and Zr and the thermomechanical processing required to produce an initially fine grain size pose significant barriers to the large-scale application of these alloys. The coarse-grained superplastic Al-Mg alloys do not suffer from these limitations and may therefore prove to be an attractive alternative within the range of high-strain-rate superplastic materials.

5.3 Superplasticity in coarse-grained Al-Mg alloys

5.3.1 Superplasticity based on viscous dislocation glide

The possibility to conduct high-strain-rate forming operations by deforming solid solution alloys in the viscous glide regime has received little attention compared to the vast amount of work on superplasticity based on grain boundary sliding. This is likely due to the fact that the tensile elongations obtained under solute-drag creep conditions are generally lower than in fine-structure superplasticity, owing to the difference in strain-rate sensitivity ($m \approx 0.3$ vs. $m \approx 0.5$, respectively). Nevertheless, for coarse-grained Al-Mg alloys deformed in the viscous glide regime, values for the maximum strain in excess of 300% can be
obtained [29-31]. Such elongations are close to those found in conventional superplasticity of fine-grained Al-Mg alloys and are sufficient for many practical applications. Moreover, forming by viscous-glide controlled creep has two important advantages over conventional superplastic forming: (i) the rate of viscous glide is not restricted by dislocation climb and consequently higher strain rates can be achieved, and (ii) since viscous glide is independent of grain size, the preparation of the materials is less complex. It should be noted that since the deformation under viscous-glide control does not follow the original definition of superplasticity in the strictest sense, the deformation behavior has also been referred to as “enhanced ductility” or “quasi-superplasticity” by some researchers [29,30,32]. In this chapter, we will use the designation “coarse-grain superplasticity” because of the low flow stress and relatively high strain-rate sensitivity associated with this regime, both of which are characteristic of superplastic deformation.

Viscous-glide creep, or solute-drag creep, results from the impediment of gliding dislocations by their interaction with solute atoms. There are two competing mechanisms in this regime, dislocation glide and climb; the slower of the two is rate-controlling. A physical interpretation of the empirically found three-power-law relation for viscous-glide creep is readily based on the Orowan equation, relating the macroscopic strain rate \( \dot{\varepsilon} \) to the mobile dislocation density \( \rho_m \) and the average dislocation velocity \( \overline{v_d} \):

\[
\dot{\varepsilon} = \chi b \rho_m \overline{v_d} \tag{5.2}
\]

Although no direct measurements of the relation between applied stress and dislocation velocity under solute-drag conditions are available, most models suggest that \( \overline{v} \propto \sigma \) in this regime, i.e. the stress exponent \( n_v = 1 \) [33]. Furthermore, experimental observations have shown that the stress exponent \( n_d \) of the mobile dislocation density \( \rho_m \propto \sigma^{n_d} \) lies between 1.6 and 1.8 for Al-Mg alloys [34,35]. This is in reasonable agreement with theoretical predictions [36] suggesting that \( \rho_m \propto \sigma^2 \) (see also Eq. (2.27)). The discrepancy between the theoretical and experimental values of \( n_d \) has been attributed to the fact that dislocations are increasingly located in subgrain boundaries at lower stresses, or alternatively to the incomplete relaxation of dislocation loops during unloading of specimens at room temperature [37]. Within the formulation of Eq. (5.2), the strain-rate sensitivity index depends critically on the stress dependence of the product \( \rho(\sigma)\overline{v_d}(\sigma) \). Assuming \( n_v \approx 1 \) and \( n_d \approx 2 \), it follows from Eq. (5.2) that \( \dot{\varepsilon} \propto \sigma^3 \), or as formulated in the original model by Weertman [36]:

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Superplastic behavior of coarse-grained Al-Mg alloys

\[ \varepsilon = \frac{0.35}{\xi} \mu \left( \frac{\sigma}{\mu} \right)^3 \]  

(5.3)

where \( \xi \) is a parameter that characterizes the interaction between the solutes and the dislocations. From Eq. (5.3) it follows that the stress exponent \( n \approx 3 \) and hence the strain-rate sensitivity \( m = 1/n \approx 0.33 \).

5.3.2 *Physical mechanisms of viscous-glide creep*

From the abovementioned considerations it is clear that the three-power law \((m = 0.33)\) is no more than an approximate relationship arising from the stress dependence of the dislocation density and the drag stress. In this section, we will evaluate the physical background of these phenomena. Based on experimental data the relation between the dislocation velocity and the stress is commonly described by \([38-40]\)

\[ \frac{n E}{k T} d v \sigma e^{-E/kT} = \]  

(5.4)

or, in simple form,

\[ \frac{n}{d v \sigma} \sigma = \]  

(5.5)

The difficulty with these expressions is that there is no upper limit for the dislocation velocity as a function of the applied stress. Returning to Newton’s second law and assuming the aforementioned linear proportionality \((n_v \approx 1)\) between the stress and the dislocation velocity, this would mean that the stress is proportional to the acceleration and likewise that the velocity is proportional to the acceleration. This in turn would imply that the dislocation accelerates at a constant velocity under a given stress, which is clearly a contradiction. To “hold back” the dislocation, there are basically two possibilities: (i) there is a drag damping the dislocation; this drag is considered viscous, in analogy with the movement of a body through a fluid; (ii) the effective mass \([33]\) increases with velocity causing the acceleration to decrease with increasing velocity.

To a first approximation we assume that the solid acts as a Newtonian viscous material:

\[ F_{\text{visc}} = -B v_d \]  

(5.6)

where \( B \) represents the damping coefficient, which in a Newtonian fluid is independent of the magnitude of \( v_d \). From the Peach-Köhler equation we have
where \( \tau_d b = B \nabla_d \) \( \quad (5.7) \)

where \( \tau_d b \) is the component of the shear force acting in the direction of the moving dislocation. The drag coefficient \( B \) consists of three basic contributions [41-43]:

\[
B = B_{PH} + B_{E} + B_{I}
\]

where the subscripts \( PH, E \) and \( I \) refer to phonon viscosity, electron viscosity and impurity effects, respectively. In the present case, the drag term \( B \) is dominated by the impurity contribution, which may be written as [44]

\[
B_{I,\text{glide}} = \frac{17}{4} \frac{A^2 c_0}{D_{Mg} kT \Omega b}
\]

\( (5.9) \)

where \( A \) is the elastic interaction energy term \( (3 \mu b \Omega / \pi) \) and \( c_0 \) the concentration of Mg in Al. If the velocity dependence is taken into account, the drag coefficient can be formulated as [45]

\[
B_{I,\text{glide}}' = \frac{A^2 c_0}{D_{Mg} kT \Omega b} \ln \left( \frac{k T D_{Mg}}{A \nabla_d} \right)
\]

\( (5.10) \)

which gives similar values to those obtained through Eq. (5.9). Analogously, a different relation can be derived for the climb contribution to the drag term. Assuming that dislocation climb is not affected by the impurities and depends only on self-diffusion through the vacancy mechanism, we have [33]

\[
B_{I,\text{climb}} = \frac{b kT}{2 \pi D_{\text{self}} \Omega} \ln \left( \frac{k T D_{\text{self}}}{A \nabla_d} \right)
\]

\( (5.11) \)

For solute drag on gliding dislocations to be the predominant mechanism, as is often claimed in literature, \( B_{I,\text{glide}} \) should be considerably larger than \( B_{I,\text{climb}} \):

\[
\frac{B_{I,\text{glide}}}{B_{I,\text{climb}}} \gg 1
\]

\( (5.12) \)

because glide and climb are sequential steps in the deformation process and therefore the largest drag coefficient corresponds to the rate-determining mechanism. For the present case however, the condition expressed by Eq. (5.12) depends very critically on the precise value of the activation energy \( Q \) for diffusion of Mg in Al as defined by

\[
D_{Mg} = D_0 e^{-Q/kT}
\]

\( (5.13) \)
Reported values for the activation energy range from 115 kJ/mol to 136 kJ/mol [46], depending on the experimental method used. From the often cited value of 115 kJ/mol follows $B_{\text{glide}} / B_{\text{climb}} = 0.15$ at a temperature of 700 K, which implies that the climb contribution to the drag term is more important than the viscous glide term. However, an activation energy of 136 kJ/mol leads to $B_{\text{glide}} / B_{\text{climb}} = 4.6$, in which case viscous glide predominates. It should therefore be concluded that both mechanisms may contribute significantly to the drag mechanism in the Al-Mg alloys at hand.

As a matter of course the solute drag on gliding dislocations only operates if the solutes are able to interact with moving dislocations; no solute atmosphere will be formed if the dislocations are moving too fast. In the case of a stationary edge dislocation in an isotropic medium, the flow lines of the solute atoms follow a circular trajectory. When the dislocation is moving with a velocity $\overline{v}_d$, an apparent flow relative to the dislocation has to be added to the flux equation [47-51]:

\[
J_d = -\frac{D_{\text{Mg}}}{k_BT} c_0 \nabla \left( W_{\text{static}} + \frac{k_BT\overline{v}_d}{D_{\text{Mg}}} \right)
\] (5.14)

Beyond a critical value of the dislocation velocity $\overline{v}_c$, defined by

\[
\overline{v}_c = \frac{3 + 2\sqrt{2}}{2} \frac{AD_{\text{Mg}}}{b^2 k_BT}
\] (5.15)

the flow lines are open and do not pass through the dislocation core. The reason for a solute atmosphere being formed is that the flow lines of the Mg atoms end at the dislocation core. Therefore, a solute atmosphere can only be formed if the dislocation is moving slower than the critical velocity. In this case, the formation of a solute atmosphere leads to a concentration gradient and consequently a drag force per unit length can be defined. For $\overline{v}_d > \overline{v}_c$, the drag term $B_l$ is equal to zero. Note that $D_{\text{Mg}}$ and thereby $\overline{v}_c$ depend strongly on temperature and activation energy through Eq. (5.13). The critical velocity as a function of temperature is displayed in Figure 5.3 for the present case of Mg in Al. It can be concluded that if the dislocation velocity $\overline{v}_d$ is larger than $5 \times 10^{-3}$ m/s at $T = 700$ K, Mg solutes in Al cannot reach the core of moving dislocations and therefore solute drag on gliding dislocations does not occur. The critical velocity poses an upper limit to the strain rates at which solute drag operates as given by Eq. (5.2). For the strain rates used in this study (around $10^{-3}$ s$^{-1}$), a maximum dislocation velocity of $5 \times 10^{-4}$
m/s requires a minimum mobile dislocation density of the order of $10^{11} \text{ m}^{-2}$. This value is substantially lower than to the total dislocation densities typically encountered and it may therefore be assumed that solute atmospheres are formed at these strain rates.

The dislocation density has some further significant consequences for superplastic behavior. The flow stress, i.e. the summation of the internal stress (Eq. (2.27)) and the drag stress (Eq. (5.7)), attains a minimum at a critical dislocation density of

$$
\rho_c = \left( \frac{B\dot{\epsilon}}{\alpha \mu b^3} \right)^{2/3} \tag{5.16}
$$

Considerable work softening, which is a prerequisite for superplastic behavior, may be expected in an alloy where $\rho_c$ is high. In this case, the initial dislocation density will be lower than $\rho_c$ and softening characterizes the first part of deformation [52]. For the present alloys, the critical dislocation density is about $10^{13} \text{ m}^{-2}$ at 700 K and a strain rate of $10^{-3} \text{ s}^{-1}$; this is indeed substantially higher than the aforementioned mobile density required to create solute atmospheres. For the critical dislocation density to be a maximum, the product $B\dot{\epsilon}$ should be maximized. In steady state, i.e. according to Eq. (5.2), we can reformulate Eq.
(5.16) in an expression that depends on the critical velocity and therefore we may state that for extended softening $Bv_c$ should be maximized.

The role of plasticity-induced vacancies in the climb and solute drag processes may be evaluated by recourse to references [53-55]. Figure 5.4 shows the total vacancy concentration, i.e. the sum of thermal and plasticity-induced vacancies, for the present case as a function of temperature for two strain rates at a final strain of 200%. From the plot it can be concluded that the thermal vacancies are dominant above 500 K. Since the superplastic temperature range for the coarse-grained Al-Mg alloys lies around 700 K, plasticity-induced vacancies are not expected to play a significant role in the climb and diffusional processes.

5.3.3 Reconstruction mechanisms

The plastic deformation of coarse-grained alloys under viscous-glide conditions is a complex phenomenon as grain-size independent and grain-size dependent mechanisms take place simultaneously. The solute drag on dislocations is accompanied by reconstruction mechanisms, which are activated by the high temperature and can contribute further to the improvement of plasticity [56]. Dynamic reconstruction in coarse-grained Al-Mg alloys is often evidenced by extensive grain refinement, which is attributed to the formation of subgrain boundaries and their conversion into low-angle and high-angle grain boundaries. This evolution of the microstructure in the solute-drag regime has been studied in
detail in torsion [57] and compression [58] modes; however, significant differences compared to these modes may arise in tensile deformation due to the anisotropy of the stress and the effect of strain localizations.

In the work presented in this chapter, we have measured the superplastic response of one fine-grained and two coarse-grained Al-Mg alloys under uniaxial tension in order to identify the main characteristics of superplastic deformation and determine the optimum deformation parameters. The microstructure and dislocation substructure of the coarse-grained alloys are analyzed as a function of strain, strain rate and temperature using electron backscatter diffraction (EBSD) and transmission electron microscopy (TEM). The results are discussed in relation to the dynamic reconstruction mechanisms and their influence on the ductility of the alloys [59,60].

5.4 Experimental procedure

5.4.1 Materials and tensile tests

The alloys used in this study are two coarse-grained Al-4.4%Mg and Al-4.4%Mg-0.4%Cu alloys with minor amounts of Ti, Mn and Cr (<0.1%) and an average initial grain size of 70 µm, and a fine-grained Al-4.7%Mg-0.7%Mn alloy (AA5083) with an average grain size of 10 µm.

Specimens for tensile testing were laser cut from 2 mm thick cold-rolled metal sheets with the gauge direction parallel to the rolling direction and subsequently annealed for 10 minutes at 450 °C before deformation. Tensile elongation measurements were performed at constant crosshead speed under controlled temperature conditions using an Instron (Instron Corp., Norwood, MA) tensile machine and a three-zone-split furnace. The strain-rate sensitivity index \( m \) and the activation energy \( Q \) were determined from strain-rate-change (SRC) tests. The activation energy was calculated using the method described in reference [15]. The strain distributions over the gauge length were determined from optical measurements of the cross-sectional area of the gauge of the deformed specimens.

To investigate the microstructure during deformation, the tensile tests were interrupted by water quenching at several elongations up to 170%. The specimen surfaces were prepared for EBSD by electrochemical polishing in a 5% perchloric acid solution in methanol at -20 °C and 10 V. TEM specimens were laser cut from the gauges and thinned by twin-jet electrochemical polishing using the same electrolyte at -30 °C and 20 V.
5.4.2 **In situ deformation experiments**

In order to directly observe the evolution of dislocation structures during superplastic deformation, the coarse-grained Al-Mg alloys were subjected to in situ tensile experiments at elevated temperature in a TEM. Such experiments require a specimen stage that is capable of straining TEM specimens while maintaining a controllable temperature of the order of 400 °C. At present, only one type of stage with combined heating and straining capability is commercially available (Gatan Inc., Pleasanton, CA). The design of this stage relies on direct physical contact between a heating element and the specimen to control the specimen temperature. The temperature of the specimen is tacitly assumed to be equal to the furnace temperature as measured by a thermocouple. This is approximately valid at high temperatures (~ 1000 °C) when the specimen is mostly heated by radiation. However, at the intermediate temperatures used in this study, radiation is negligible, and the specimen temperature can only reach the furnace temperature if the thermal contact between the two is very good. The requirement that the specimen be movable for tensile testing results in poor thermal contact; moreover, the degree of contact fluctuates during the course of a tensile experiment. This was confirmed by calibration measurements in low vacuum on TEM tensile specimens with a thermocouple spot-welded close to the electron-transparent area. Applying a thermally conductive paste between the heating element and the specimen greatly improved the performance of the holder. However, such viscous agents are not suitable for high vacuum systems such as TEMs.

A few homemade heating straining stages have been developed over the past two decades [61-64], most of which use a filament to heat the specimen by radiation [61,62]. The temperature is measured by a thermocouple that is positioned as close as possible to the observed area of the specimen. Since the specimen is heated exclusively by radiation, the thermocouple attains approximately the same temperature as the specimen and therefore provides a very accurate temperature measurement. The high-temperature in situ experiments reported in this chapter were partially conducted at the Institut National Polytechnique in Grenoble, France, using the double-tilt heating straining holder described in reference [62]. Calibration experiments have shown the measured temperature of this holder to be accurate to within 10 °C [65]. In the intermediate temperature range (~ 150 °C), in situ tensile experiments were also performed using the Gatan heating straining holder described above.
5.5 Macroscopic mechanical behavior

The dependence of the elongation to failure on strain rate and temperature for the two coarse-grained materials is presented in Figure 5.5. Both alloys show maximum elongation to failure at a temperature between 400 and 440 °C, which is considerably lower than the temperatures higher than 500 °C usually required to achieve maximum ductility in fine-grained Al-Mg alloys [14,16,21]. Furthermore, the optimum strain rate lies around $10^{-2} \text{s}^{-1}$, which is high compared to the strain rates generally associated with the grain boundary sliding mechanism in fine-grained materials. The fine-grained Al-Mg-Mn alloy (not shown in Figure 5.5) failed always at lower values of the maximum strain than the coarse-grained alloys when deformed under the same conditions of temperature and strain rate. Of the coarse-grained alloys, the Al-Mg-Cu showed on average slightly higher ductility than the Al-Mg alloy, the highest value for the tensile elongation being 357% for deformation at 440 °C at a strain rate of $10^{-3} \text{s}^{-1}$. The relationship between flow stress, as measured during steady-state deformation, and strain rate at several deformation temperatures for the coarse-grained Al-Mg-Cu and the fine-grained Al-Mg-Mn alloy is shown in Figure 5.6. The data for the coarse-grained Al-Mg alloy follow exactly the same pattern as the Al-Mg-Cu alloy and have therefore been omitted. For the coarse-grained alloys, the $\log \sigma$ vs. $\log \dot{\varepsilon}$ dependence is linear over the entire strain rate interval investigated, while for the fine-grained Al-Mg-Mn alloy the dependence resembles the aforementioned sigmoidal shape (see Figure 5.2).

![Figure 5.5: Maximum elongation to failure as a function of strain rate at two different temperatures for the two coarse-grained Al-Mg alloys. Each data point represents the maximum of three tensile tests.](image)
The strain-rate sensitivity obtained from the data from Figure 5.6 is plotted as a function of \( \log \dot{\varepsilon} \) in Figure 5.7. The fine-grained material deformed at 520 °C shows a peak for the strain-rate sensitivity of about 0.7 at \( 10^{-4} \text{s}^{-1} \), decreasing to a value of around 0.3 at \( 10^{-2} \text{s}^{-1} \). The coarse-grained alloys are characterized by a relatively constant value for \( m \) of about 0.3. At a stress value of 25 MPa, the activation energy \( Q \) for the deformation of these alloys is equal to 153 kJ/mol, which is close to the activation energies for diffusion of Mg in Al (136 kJ/mol) and for self-diffusion in Al (142 kJ/mol).

![Figure 5.6: Temperature dependence of the flow stress vs. strain rate relationship for the three aluminum alloys.](image1)

![Figure 5.7: Strain-rate sensitivity (m) as a function of strain rate at different temperatures.](image2)
The values of the strain-rate sensitivity index and the activation energy indicate that the deformation rate in the coarse-grained alloys is indeed controlled by solute drag, which may be aided by climb as discussed in Section 5.3. The strain-rate sensitivity of the drag mechanism is high enough to stabilize the plastic flow to such an extent that elongations of a few hundred percent can be attained. However, although the solute drag operates relatively constantly throughout the entire range of strain rates investigated, the maximum elongation changes appreciably as a function of the strain rate as illustrated in Figure 5.5. This shows that in addition to viscous glide, strain-rate dependent reconstruction mechanisms greatly affect the ductility of the alloys.

5.6 Dynamic reconstruction

5.6.1 Reconstruction mechanisms

The strain distribution over the gauge of two specimens, one fine-grained (Al-Mg-Mn) and one coarse-grained (Al-Mg-Cu), having the same value for the maximum tensile elongation (320%) is presented in Figure 5.8. For both materials, the strain varies over the length of the gauge. In the case of the fine-grained material, about 5% of the gauge is heavily affected by cavitation. This phenomenon is more extensively described in references [66,67]. The coarse-grained alloy shows prolonged necking over about 20% of the deformed gauge.

Because of the localization of strain, the strain rate in the necked region becomes much higher than the macroscopic strain rate of $10^{-2}$ s$^{-1}$. This leads to the

![Figure 5.8: Strain distribution over the gauge length of two deformed specimens. The fine-grained Al-Mg-Mn alloy was deformed 320% at 520 °C and $10^{-3}$ s$^{-1}$ and the coarse-grained Al-Mg-Cu was deformed 320% at 420 °C and $10^{2}$ s$^{-1}$.](image)
Superplastic behavior of coarse-grained Al-Mg alloys

appearance of dynamic recrystallization close to the fracture place, as shown in Figure 5.9a. Dynamic recrystallization is presumably triggered by the high local dislocation densities generated at strain rates in excess of $10^{-1} \text{s}^{-1}$. The mechanisms involved in its nucleation and propagation cannot be conclusively identified from the present results; in particular, the mechanisms of oriented nucleation versus selective growth are widely under debate in literature [26]. Oriented nucleation is often observed in the presence of second phase particles. This so-called particle stimulated nucleation is evidently not relevant in our coarse-grained alloys. Reports of texture development during dynamic recrystallization indicate that selective growth plays a significant role in fcc metals [68]. The texture observed in the recrystallized parts of the Al-Mg specimens is similar to that of the uniformly deformed gauge, suggesting that selective growth may indeed be responsible for the microstructure observed. The new grains coarsen rapidly, producing inhomogeneities of the microstructure and consequently a premature failure of the material. It should be noted that irregularities in the material lead to necking in the first place and therefore it may be argued that the coarse microstructure is the origin of necking rather than its consequence; however, large grains of the size shown in Figure 5.9a were not found in the material prior to deformation and their development is therefore positively attributed to dynamic recrystallization.

At intermediate strain rates ($10^{-2} \text{s}^{-1}$), the main restoration mechanism in the coarse-grained alloys is dynamic recovery. This mechanism is associated with annihilation of dislocations and with the formation of a subgrain structure [53]. The newly generated microstructure, as illustrated in Figure 5.9b, is characterized by a finer grain size and an increased grain boundary density as compared to the original material. The grain refinement is a result of the continuous increase in subgrain boundary misorientations so that they become typical of low-angle and in some cases even high-angle grain boundaries. Analysis of the misorientation
angles shows a significant increase of the ratio of low-angle to high-angle boundaries in this regime [69]. The dynamic recovery greatly improves the ductility of the coarse-grained materials leading to values of the maximum strain in excess of 300%.

A quantitative measure of dynamic recovery is provided by the extent of grain refinement as measured by EBSD. Table 5.1 shows the average grain size of the coarse-grained alloy deformed to failure at different strain rates and temperatures compared to the original grain size. The maximum grain refinement at a deformation temperature of 420 °C is attained at a strain rate of about $10^{-2}$ s$^{-1}$.

At lower strain rates, the formation of a subgrain structure is suppressed, while at higher strain rates or temperatures, dynamic recrystallization becomes predominant and leads to rapid grain growth. A similar trend is shown by the evolution of texture during deformation as shown in Figure 5.10. The cube texture that develops along the tensile axis becomes most pronounced at the optimum strain rate of $10^{-2}$ s$^{-1}$. The evolution of this texture is associated with the formation of equiaxed grains at the highest strain rates.

Table 5.1
Average grain size of Al-Mg-Cu alloy before deformation and as deformed at various strain rates and temperatures. The grain sizes are measured from EBSD scans, requiring a misorientation of at least 5° in order for a grain to be counted.

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Strain rate (s$^{-1}$)</th>
<th>Average grain size (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>before deformation</td>
<td></td>
<td>115</td>
</tr>
<tr>
<td>420</td>
<td>$10^{-3}$</td>
<td>64</td>
</tr>
<tr>
<td>420</td>
<td>$10^{-2}$</td>
<td>43</td>
</tr>
<tr>
<td>420</td>
<td>$10^{-1}$</td>
<td>95</td>
</tr>
<tr>
<td>450</td>
<td>$10^{-1}$</td>
<td>171</td>
</tr>
</tbody>
</table>

Figure 5.10: (001) Pole figures for the Al-Mg-Cu alloy deformed at 420 °C at (a) $10^{-3}$ s$^{-1}$, (b) $10^{-2}$ s$^{-1}$ and (c) $10^{-1}$ s$^{-1}$. Dynamic recovery leads to a pronounced cube texture at a strain rate of $10^{-2}$ s$^{-1}$.
of a substructure during dynamic recovery, allowing favorable orientations of the
subgrains with respect to the tensile axis. In tensile deformation of fine-grained
Al-Mg [70], it was found that at a strain rate of $10^{-4}$ s$^{-1}$, when grain boundary
sliding dominates, the texture is significantly reduced; at a strain rate of $10^{-2}$ s$^{-1}$,
when solute-drag creep is the rate-controlling mechanism, a strong cube texture
developed similar to our observations.

5.6.2 Dislocation substructure

Figure 5.11 shows three micrographs representative of the microstructural
evolution observed during superplastic forming of the coarse-grained Al-Mg
alloy. At a strain of a few percent, just beyond the yield point, random
configurations of dislocations are visible (Figure 5.11a). This stage of
deformation is characterized by a drop of the flow stress [69], which indicates
dislocation multiplication from an initially low dislocation density pinned by Mg
solute [19]. During further straining, subgrain formation occurs primarily along
the original grain boundaries, as in Figure 5.11b showing subgrain boundaries
near a high-angle boundary triple junction. At this stage, the substructure shows
many incomplete subgrain boundaries, i.e. boundaries with a very low
misorientation ($<1^\circ$) that do not fully enclose a subgrain. Only when a strain of
the order of 1 is attained, the subgrains completely fill the grain interior. Figure
5.11c shows the refined subgrain structure at a strain of 170% and an average
subgrain size of approximately 5 µm. Note that the size distribution is fairly
broad, with observed subgrain sizes ranging from 1 to 10 µm. The subgrain
boundaries have an average misorientation of the order of $2^\circ$, with some of the
boundaries having misorientations high enough to be detected by EBSD.
Essentially the same substructure evolution was found in the Al-Mg-Cu alloy.

Without any external stress applied, the subgrain boundaries are
relatively stable under annealing at superplastic forming temperature as illustrated
in Figure 5.12. At this temperature, most of the lattice dislocations are absorbed
into the subgrain boundaries, and some rearrangement of the dislocations in the
subgrain boundaries is observed. However, the subgrain structure as a whole
remains intact for at least 10 minutes, even in a thin TEM foil, where dislocations
can easily escape to the free surfaces of the specimen. Given the strain rate
around $10^{-2}$ s$^{-1}$, this is long enough a time to ensure that the observed recovery
mechanisms are dynamic rather than static.

Our observations of subgrain formation are similar to those of binary Al-
Mg alloys in torsion [57] and compression [58] in the solute-drag regime.
Figure 5.11: Dislocation substructure in Al-Mg deformed at 440 °C and $5 \cdot 10^3 \text{s}^{-1}$ to (a) 4%, (b) 20% and (c) 170%.
However, since the maximum achievable strain in tensile mode is considerably lower, the grains do not thin to such an extent that so-called geometric dynamic recrystallization [71] becomes relevant. In all deformation modes, the substructure formation in the Al-Mg alloys is more sluggish than in pure Al, presumably due to a lowering of the stacking fault energy by the solute Mg.

The effect of the Mg content on the tensile ductility is twofold. On the one hand, a higher Mg content increases the extent of solute drag, thereby stabilizing the plastic flow. However, beyond a few percent Mg, the effect on the strain-rate sensitivity becomes fairly marginal; Taleff et al. [30] reported an increase of $m = 0.29$ to $m = 0.32$ in going from 2.8% to 5.5% Mg. On the other hand, the presence of Mg significantly reduces dynamic recovery as evidenced by the slow formation of subgrains. As a result, Mg concentrations above 5% can easily give rise to dynamic recrystallization within a certain domain of temperature and strain rate, which in the absence of grain refining second phase particles leads to rapid coarsening of the microstructure. The currently used composition with 4.4% Mg appears to be a good balance between solute drag and dynamic recovery, leading to enhanced tensile ductility in excess of 300%. In torsional deformation, where a high strain-rate sensitivity to avoid necking is less important, the ductility benefits most from dynamic recovery (leading to geometric dynamic recrystallization at high strains) and is consequently higher for pure Al than for Al-Mg alloys [72].

The initially inhomogeneous formation of subgrains gives rise to a “core and mantle” microstructure, in which most deformation is concentrated along the grain boundaries. In fine-grained superplasticity, this type of microstructure has
been associated with grain mantle deformation processes as an accommodating mechanism for grain boundary sliding [73]. In the present case, dynamic recovery is initially confined to the mantle region, but extends throughout the microstructure at higher strains.

5.6.3 In situ observations of substructure evolution

The evolution of the dislocation substructure during superplastic deformation can be directly observed in in situ tensile experiments in a TEM. A difficulty inherently associated with this technique is presented by the image forces resulting from the proximity of free surfaces, which may significantly influence the dislocation dynamics compared to bulk behavior (e.g. [63]). In the present investigation we have attempted to minimize such effects by preparing tensile TEM specimens from macroscopically prestrained alloys and studying only the initial motion of dislocations from their starting configuration. However, for the present case of Al-Mg alloys, it turns out that surface diffusion of Mg severely limits the temperature range at which the in situ experiments can be conducted.

At temperatures in excess of 200 °C, the tensile TEM specimens were consistently found to fracture intergranularly at very low loads (typically ~ 30 gf). This is evidently not representative of the bulk behavior at high temperature showing very high tensile ductility. Below 200 °C, the specimens showed ductile transgranular failure at loads of the order of 300 gf. By EDS and electron diffraction analysis it was found that the intergranular fracture areas of the TEM specimens deformed at high temperature contained large amounts of Mg and MgO. Presumably, surface diffusion of Mg becomes appreciable at high temperature and leads to segregation of Mg to the grain boundaries and consequently to grain boundary embrittlement.

In the temperature range below 200 °C, dislocation climb is not activated and therefore extensive dynamic recovery is not to be expected. However, even at low temperature, some rearrangements of the substructure were observed that may be illustrative of those occurring during dynamic recovery. Figure 5.13 shows dislocation motion leading to the formation of a subgrain boundary, which is the initial stage of grain refinement in the coarse-grained alloys. The absorption of dislocations by a subgrain boundary is shown in Figure 5.14; this process contributes to the increase in grain boundary misorientation that is associated with dynamic recovery. In other words: although dislocation motion is solely due to glide at low temperatures, the observed rearrangements resemble the processes that contribute to dynamic recovery at high temperature.
Figure 5.13: Rearrangement of dislocations leading to the formation of a subgrain boundary (marked with arrows) in Al-Mg during in situ straining at ~150 °C.

Figure 5.14: Absorption of dislocations into a subgrain boundary in Al-Mg during in situ straining at ~150 °C. The subgrain boundary is marked by a dotted line in (a). The arrows in (a) and (b) indicate the dislocations that are absorbed by the boundary in the next image respectively.
5.7 Conclusions

We have investigated the superplastic behavior of coarse-grained Al-Mg alloys. Under optimum deformation conditions of temperature and strain rate, these Al-Mg alloys can have an elongation to failure in excess of 300%. The measured values of the strain-rate sensitivity index of the order of 0.3 and the activation energy close to that for the diffusion of Mg in Al indicate that solute drag on gliding dislocations is the principal mechanism of plasticity. An analysis of the physical prerequisites for solute drag reveals that the occurrence of this mechanism depends critically on the average dislocation velocity and the activation energy for diffusion of Mg in Al.

The plasticity of the coarse-grained alloys is strongly influenced by dynamic reconstruction mechanisms. Dynamic recrystallization is dominant at strain rates in excess of $10^{1} \text{ s}^{-1}$ and results in a strong coarsening of the microstructure and premature failure. Dynamic recovery prevails at a strain rate around $10^{2} \text{ s}^{-1}$, leading to great enhancement of the plasticity of the coarse-grained materials.

During dynamic recovery, grain refinement occurs by the formation of subgrain boundaries and low-angle grain boundaries. TEM observations show that subgrain formation proceeds slowly, presumably due to a relatively low stacking fault energy combined with a low density of second phase particles. During initial straining, subgrains are formed primarily along the original grain boundaries. This results in a “core and mantle” microstructure, with dynamic recovery mainly taking place in the mantle region. A uniform substructure is established at a strain of the order of 1.

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