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**HYBRID SUPERPLASTIC FORMING OF
AZ31 MAGNESIUM ALLOY**

GUO MEILING

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Guo Meiling

School of Mechanical and Aerospace Engineering

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Abstract

Hybrid superplastic forming (SPF) has recently been developed as a sheet forming technique, combining a hot drawing (mechanical pre-forming) process and a gas forming process. The hot drawing stage greatly enhances the formability and the forming efficiency of the metal sheet. The blank sheet with a desired amount of material is drawn into the die to form an intermediate hollow shape by a punch. During the gas forming stage, the mechanical pre-formed part is further formed at a target strain rate by applying the gas pressure. It is considered as an efficient way to form components with complex shapes in automotive and aerospace industries.

In this work, non-superplastic grade AZ31B magnesium sheets were successfully formed by hybrid SPF at 400 °C. The dome height of formed part was 60 mm under gas pressure cycle S1, approximately 60 % of the die diameter. In contrast, the part formed by conventional SPF cracked at the same height. The influence of punch shape on the formability of AZ31B was also investigated. The minimum thickness of 1.31 mm after hybrid SPF was found at location where the material first came into contact with the punch, and the corresponding thickness reduction was 59%. Compared with the conventional SPF process, the thickness distribution of part formed by hybrid SPF was significantly improved.

Additionally, the microstructure evolution of AZ31B in uniaxial tensile tests and hybrid SPF were examined by electron backscatter diffraction. The static and dynamic grain growth at grip region and gauge region were also

investigated at various strain levels. Many subgrains with low misorientation angle were observed in the coarse grains during SPF process. The average misorientation angle and fraction of high angle grain boundaries decreased with increasing strain. This explains well the generation of subgrain via dislocation motion during SPF. Therefore, the main deformation mechanisms during hybrid SPF was determined to be a combination of recrystallization, dislocation climb and grain boundary sliding (GBS).

Based on the tensile test results, parameters of hyperbolic sine creep law model and two-term material constitutive model were determined at 400 °C. The hybrid SPF behavior of non-superplastic grade AZ31B was predicted by ABAQUS using these two material forming models. The FEM results of thickness distribution, thinning characteristics and forming height were compared with the experimental results. For the hot drawing process, the simulation results of both two models agree well with the experimental results. However, neither the hyperbolic sine creep law material model nor the two-term material model could predict the dome height and thickness distribution well during hybrid SPF. There is an increase in deviation between the prediction and experimental results when the material undergoes larger strain deformation during SPF.

As a result, the part formed by hybrid SPF was done faster with higher dome height and a more even thickness distribution than conventional SPF. The forming time for hybrid SPF was significantly shortened as the hot drawing step formed the pre-formed component rapidly before the final more time-

consuming gas blow forming. Accordingly, AZ31B part formed by hybrid SPF is structurally stronger due to the presence of fewer and smaller cavities as compared to conventional SPF.

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Chapter 1 INTRODUCTION

1.1 Background

Magnesium alloys have been widely used for low weight structures in the aerospace and transportation industry due to their low density; only two thirds that of aluminum alloys. To decrease fuel consumption and CO₂ emissions in the automotive industry, the application of Mg is proved as an effective option for its reduced weight. Thus, it is possible for the automotive industry to manufacture lighter, more environmentally friendly cars by using improved alloying and processing techniques of magnesium. Besides the major advantage of being light, the other benefits of choosing magnesium for engineering designs include high specific strength and stiffness, relatively low melting temperature and efficient thermal conductivity, all of which enable it to be cast at high productivity rate.

However, magnesium alloys have limited ductility and poor formability at room temperature due to the limited slip systems available in hexagonal close packed (HCP) crystal structures. It only exhibits a ductility of 20% at room temperature, so press forming of magnesium alloy sheet is a challenging task. Because of the HCP structure, it is easier to manufacture the complicated parts by casting rather than by plastic deformation processes. To date, the demands for wrought magnesium alloy products such as rolled sheets, plates and forgings are still very limited compared to magnesium casting alloys. However, magnesium alloys fabricated by mechanical forming have higher ductility, improved specific strength and better mechanical properties compared to the cast parts [1-

4]. Moreover, the formability of wrought magnesium can be improved by deforming the alloy through warm and hot working at elevated temperatures.

On the forming process, the research effort focuses on the processing technique, formability of material, and finite element modeling. Hybrid superplastic forming (SPF) is recently developed as a sheet forming technique, combining a hot drawing (mechanical pre-forming) process and a gas forming process [5, 6]. It is an efficient way to form sheet metals into complex-shaped parts for the automotive and aerospace industries. Compared with the conventional SPF process, the forming time for hybrid SPF can be significantly shortened as the hot drawing step produces a pre-formed component before gas blowing forming.

Nevertheless, literature on the hybrid SPF of magnesium alloys is quite limited since the majority of research is concentrated on aluminum alloys and titanium alloys [5, 7]. Magnesium alloys have a number of engineering advantages, such as light weight, high specific strength and stiffness. For material cost, the superplastic grade material can be up to three times cost of commercial non-superplastic alloys. Both the total time taken to form part by hybrid SPF and raw material determine the cost. Thus, commercial non-superplastic magnesium alloy is chosen in this research.

1.2 Objective

In this study, the primary objective is to form non-superplastic grade magnesium alloy with uniform thickness by employing a proposed hybrid SPF technology which combines hot drawing with gas forming. This hybrid SPF process together with better control of gas pressure cycle obtained from Finite

Element Modeling (FEM) will possess the capability to deform the non-superplastic grade magnesium alloy with better thickness distribution.

In order to achieve this goal, it is essential to investigate the superplastic deformation behavior, deformation mechanisms, evolutions of microstructures and their effects on the hybrid SPF of AZ31 magnesium alloy.

1.3 Scope of Work

The scope of this research is as follows:

- To design an efficient hybrid SPF process which combines hot drawing (mechanical pre-form) and gas forming process and investigate the forming capability of this forming process. The specialized die and punch need to be designed and fabricated to form AZ31 magnesium alloy sheets by hybrid SPF.
- To study the effect of forming parameters (punch shape, temperature, strain rate, gas pressure, etc.) on the forming height and thickness distribution of part formed by hybrid SPF, so as to achieve a forming process with faster forming rate and better thickness distribution.
- To investigate the deformation behavior, the relationship between the flow stress and strain rate, the microstructure evolution and deformation mechanisms during the proposed hybrid SPF process. Macro and micro analyses, ductility, grain boundary and micro-defects (void or grain growth), will be conducted and the comparison between the hybrid SPF and the conventional SPF process (pure gas forming) will also be performed.

- To establish a material model for analysis of the thinning characteristics and the optimization of the process design. The suitable rheological models will be reviewed, investigated and selected to represent the flow behavior of materials. The simulation results will be compared with those from experiments to verify the feasibility and effectiveness of the FEM modeling.

1.4 Thesis Outline

This thesis is outlined as follows:

Chapter 1 gives an introduction to the background of magnesium alloys, and highlights the objective and scope of my research.

Chapter 2 presents the literature review related to magnesium alloy and its properties, requirements of superplasticity, deformation mechanisms that are needed to achieve superplasticity and finite element modeling.

Chapter 3 describes the experimental procedure for hybrid SPF, including tooling design, forming process and EBSD specimen preparation.

Chapter 4 presents a series of experiments and investigations to establish the suitable punch diameter and radius, forming temperature and gas pressure-time profile. The thickness distribution of the final part was measured by splitting the part into two equal halves.

Chapter 5 analyzes the microstructure evolution for static and dynamic grain growth conditions in uniaxial tensile specimen, conventional SPF formed part and hybrid SPF formed part.

Chapter 6 presents the FEM simulation for hybrid SPF process by ABAQUS. The FEM results of thickness distribution, thinning characteristics and forming height were compared with the experimental results.

Chapter 7 concludes this thesis with the highlights of the recommended future work.

Chapter 2 LITERATURE REVIEW

2.1 Magnesium Alloys

2.1.1 Background

The use of lightweight alloys in aerospace and automotive industries in building lighter parts with enhanced performance holds great promise [8-10]. Magnesium is the third most commonly found element in the sea (0.14%) and sixth on earth (2.5%). The most notable advantages of magnesium are its light weight (1.7g/cm^3) and high strength to weight ratio [11]. This low density not only allows a potential weight reduction of 22-70% in overall product design, it also provides a great opportunity for a better fuel economy as with a lighter transportation device, less fuel will be consumed [12].

Besides the major advantage of being light, some other benefits of magnesium are listed as follows.

1. High specific strength and stiffness.
2. Relatively low melting temperature and efficient thermal conductivity.
3. High damping capacity.
4. Good weldability.
5. Good machinability.
6. High recycling potential.

Magnesium is a material known for its low density and high strength. However, pure magnesium is mechanically weak in nature and requires alloying with other elements to attain better mechanical properties. As such, magnesium alloys in the market comprises of mixtures of magnesium and other metals such

as aluminum, copper, zinc and manganese to strengthen and refine the microstructure. The commercially available magnesium alloys that are commonly used include AZ31, AZ61 and AZ80. Magnesium alloys such as AZ31B are considered as suitable candidates for sheet metal forming processes by virtue of their high specific strength and low density [13, 14].

The range of wrought magnesium which is commercially available is very limited. The chemical compositions of typical wrought magnesium alloys are listed in Table 2-1.

Table 2- 1 Chemical composition (weight percentage) of wrought magnesium alloys [15].

Alloy	Al	Mn	Zn	Ca	Si	Cu	Ni	Fe	Mg
AZ31B	2.5-3.5	0.2 min	0.7-1.3	0.04 max	0.3 max	0.05 max	0.005 max	0.005 max	Bal.
AZ31C	2.5-3.5	0.2 min	0.6-1.4	0.04 max	0.3 max	0.1 max	0.03 max	-	Bal.
AZ61A	5.8-7.2	0.15 min	0.4-1.5	-	0.3 max	0.05 max	0.005 max	0.005 max	Bal.
AZ80A	7.8-9.2	0.15 min	0.2-0.8	-	0.3 max	0.05 max	0.005 max	0.005 max	Bal.

The disadvantage of magnesium alloys mainly lies in its low ductility at room temperature, hence limiting its ability to form complex parts. Magnesium alloys have a hexagonal closed packed (HCP) crystalline structure, and the lack of slip systems leads to low ductility and formability of this material at room temperature [16-20]. The mechanical properties of wrought magnesium alloy AZ31B sheet at room temperature are given in Table 2-2.

Table 2- 2 Mechanical properties of typical wrought magnesium alloy AZ31B sheet [21].

Condition	Tensile strength (MPa)	Yield strength (MPa)	Elongation (%)
Parallel to rolling direction			
Annealed (O)	225	150	21
Hard rolled (H24)	290	220	15
Perpendicular to rolling direction			
Annealed (O)	270	170	19
Hard rolled (H24)	295	235	19

Formability of magnesium is strongly dependent on the orientation of the basal plane. In the production of magnesium sheet, the rolling procedures will result in the development of a strong crystallographic grain orientation with the basal plane aligned parallel to the rolling direction.

As shown in Figure 2-1, the material fails easily if the stress direction is parallel to the basal plane as compared to 45° to the basal plane. This implies that the material ductility is improved if the stress direction is pointing out of the basal plane instead of parallel to the basal plane. Similar studies have also shown that a weaker basal texture generally results in improved formability and reduction of tensile-compressive anisotropy due to the suppression of twin generation [22, 23].

Figure 2- 1 Effect of texture orientation of tensile flow curve in AZ31 alloy (I: stress parallel to basal planes, II: stress at 45° with basal planes).

2.1.2 Crystal structure of magnesium

In a single crystal of magnesium, only three different slip systems are identified by critical resolved shear stresses (CRSS) [24]. In magnesium, the basal slip system has a much smaller CRSS than a non-basal slip system on pyramidal or prismatic planes at room temperature. Thus, basal slip is considered as the main slip system that results in most plastic deformation in polycrystalline magnesium alloys [25].

The planes, slip systems and crystal structure of magnesium are shown in Figure 2-2 [26]. At room temperature, there are only two independent slip systems, the (0001) $\langle 11\bar{2}0 \rangle$ basal slip and $\{10\bar{1}2\}\langle 10\bar{1}1 \rangle$ mechanical twinning that could accommodate plastic deformation to a small degree. The existence of only the two slip systems in magnesium results in strain incompatibility between grains, which allows failure to occur easily with little deformation, therefore leading to magnesium exhibiting low ductility.

Figure 2- 2 Slip Systems (a) Basal and prismatic slip planes (b) Pyramidal slip planes [26].

In order to satisfy von Mises criterion for homogenous shape change, at least five independent slip systems are necessary. Other slip systems such as $\{10\bar{1}0\}$ $\langle 11\bar{2}0 \rangle$ prismatic slip and $\{10\bar{1}1\}$ $\langle 11\bar{2}0 \rangle$ pyramidal slip requires a larger CRSS for activation and is difficult to operate at room temperature. These systems also have the same slip direction and do not lead to deformation out of the basal plane.

For improved formability, higher order pyramidal slip where the slip vectors are out of the basal plane is necessary. This slip mode offers five independent slip systems that could satisfy von Mises criterion. However, pyramidal slip has a much larger CRSS (~100 times) than the basal slip and can only be activated at elevated temperatures.

In order to make magnesium alloys viable for applications, a higher working temperature is required to activate more slip systems to improve formability [27, 28].

2.2 Superplasticity

The definition for superplasticity was initially proposed and accepted at the 1991 International Conference on Superplasticity in Advanced Materials (ICSAM-91) held in Japan. The capability to exhibit high tensile elongations of a polycrystalline material prior to failure is defined as superplasticity. The elongations of superplastic materials are generally at least 200 % with strain rate sensitivities close to 0.5 [29].

Superplasticity is an important field for scientific research. It lends strong theoretical support to the superplastic forming industry in which superplastic metal sheets are used to manufacture complex-shaped parts [30]. To date, thousands of tons of metallic parts are processed and widely used in various applications [31, 32].

The advantages of SPF are obvious:

1. By using this method, complicated and intricate shapes can be formed from a single piece of material using a single processing step, thereby contributing to cost saving.
2. The high forming temperature effectively relieves the stress formed during processing. As such, there is little or no residual stress present when the part is removed from the press. Therefore, there is excellent repeatability between pressings.

3. During sheet forming, this fabrication process leads to inexpensive tooling due to it requiring only a single die, as compared to conventional methods that usually require a matched pair of tooling.

It is reported that metallic, ceramic, intermetallic, or composite materials with relatively coarse to ultrafine grain sizes (20 μm -30 nm) and isotropic or anisotropic grain shape, size or orientation can exhibit superplasticity [33].

A variety of types of superplastic behavior are summarized in Figure 2-3. Lots of materials including commercial aluminum alloys, superplastic ceramics, nickel alloys have applied superplastic models to describe their flow characteristic.

Additionally, the consideration also includes superplastic behavior in nanoscale materials at a predicted range and superplastic behavior at ultra-high strain rates. All of the observations mentioned above indicate that superplasticity can be achieved by an operational deformation mechanism at a wide range of strain rates. Various polycrystalline materials have been included in the superplasticity category.

Figure 2- 3 Overview of superplasticity in a wide range of materials [34].

The research [34] dealt with the process of fine structure superplasticity, where the high tensile elongation was attained at constant temperature without a phase change. This process is different from transformation superplasticity, where the high ductility arises from periodically cycling the temperature of a material through the phase change [33]. Phase transformation during thermal cycling led to exceptional weakness in material when under small applied stress.

2.2.1 Requirements of structural superplasticity

Superplasticity is the ability that materials possess to experience large, neck-free, tensile deformation before fracture takes place. One of the most typical superplastic behaviors is fine structure superplasticity.

To develop superplastic materials, the structural prerequisites for metal based materials with fine grains have been studied and well established. Structural superplasticity requires small and equiaxed thermally stable grains (typically

grain size smaller than 10 μm) and high forming temperature (typically at or above 0.5 T_m , where T_m is the absolute melting temperature of material) [35].

It is reported the tensile elongations can reach as high as thousands of percent in certain metal alloys with fine grains. However, in practice, these two requirements mentioned above are usually incompatible since that the grains of pure metals and solid solution alloys tend to grow at elevated temperatures. To inhibit grain growth, superplastic materials are generally two phase or contain a fine dispersed second phase.

In the following sections, the requirements for the structural material to achieve superplasticity are discussed.

2.2.1.1 Grain size and grain shape

Concerning the grain size and shape, one important prerequisite for structural superplasticity is the material has fine grains with equiaxed grain structure. Typically, the grain size needs to be smaller than 10 μm . Nevertheless, a majority of wrought materials do not have the required equiaxed grains at the beginning stage. With a decreasing grain aspect ratio during superplastic deformation, a nearly equiaxed microstructure can be obtained after 30% strain [34].

Small grains have more grain boundaries and thus can better support grain boundary sliding during the superplastic deformation. Grain boundary sliding is normally demonstrated by the offset of scratch lines on a sample surface after

deformation, as illustrated in Figure 2-4. During this process, the grain boundary sliding acts as the rate controller by leading to increase of the superplastic strain rate when the grain size decreases.

Figure 2- 4 SEM images showing offsets of marker lines in FSP Al-Mg-Sc alloy specimens (a) ~ 20 % elongation (b) ~40 % elongation (c) ~80 % elongation [36].

The equiaxed grain shape guarantees that the grain boundary can experience a shear stress which impels the progress of grain boundary sliding. During the superplastic deformation process, grain rearrangement and rotation also occur besides grain sliding and migration. The equiaxed grains can rotate easily during superplastic flow, thus providing the grains with an additional degree of freedom. The grain rotation together with the grain boundary sliding in the deformation process leads to an overall reduction in texture.

Superplastic grade materials are also more expensive compared to their conventional counterparts due to the processes necessary to produce fine grain structures. This hinders the use of SPF in mass production due to the high costs of raw materials.

2.2.2 Grain boundary characteristics

High angle grain boundaries are better for grain sliding and grain rotation compared with low angle grain boundaries. Thus, high angle grain boundaries between matrix grains are preferred since grain boundary sliding generally dominates the deformation.

In contrast, low angle boundaries generally exist in components formed by warm working process, and they do not slide easily under the appropriate shearing stresses. Stress concentrations can be observed at triple points and other obstructions along the grain boundary during grain boundary sliding controlled deformation. Thus, high stress concentrations will be created at triple points during grain boundary sliding due to the lack of boundary mobility. Finally, cracks will be caused leading to early failure of materials.

2.2.1.3 Second phase

Single phase materials with fine grains, normally have rapid grain growth at elevated temperatures. Thus, a second phase material, or particles at grain boundaries are required to restrain the grain growth during the SPF process. Fine grain size and uniform distribution of the second phase materials can well

inhibit grain growth and normally the inhibition effects are enhanced as the quantity of the second phase increases.

2.2.1.4 Strain rate

Strain rate is considered as one of the most important parameters in superplasticity. As shown in Figure 2-5, the flow stress is particularly sensitive to strain rate in structural superplastic materials. As seen, the datum points tend to lie along an S-shaped curve which divides the behavior into three distinct regions, when the flow stress is logarithmically plotted against the imposed strain rate.

The curve in Figure 2-5(a) is generally illustrated by the relation

$$\sigma = K\dot{\epsilon}^m \quad \text{Equation 2-1}$$

where σ , $\dot{\epsilon}$ denote the flow stress, and strain rate respectively; K is a material constant that would vary with the test temperatures, the microstructure and defect structure of the specimen.

The strain rate sensitivity (m) value of a material is defined as

$$m = \frac{\delta \ln \sigma}{\delta \ln \dot{\epsilon}} \quad \text{Equation 2-2}$$

which is the slope of the line in Figure 2-5(a) at any specified strain rate. From previous studies, a constant strain rate is usually used to test the sample and the curve is plotted by describing the relationship between the steady flow stress and imposed strain rate on a double logarithmic scale. Thus, the slope of the plot is able to describe the strain rate sensitivity.

As shown in Figure 2-5 (a), regions I and III with strain rate sensitivity of $m \leq 0.3$ represent deformation at low strain rate and high strain rate respectively. Region II ($m \geq 0.3$) is the region where a material can deform superplasticity with large elongations. Maximum elongations are observed in region II comparing with the significant decreases in the total elongations in both regions I and III.

The mechanisms of grain boundary sliding working together with the relative displacements of individual grains in region II leads to the occurrence of superplasticity. In the superplastic region II, the grains move over each other showing that grain boundary sliding is the main flow process [36].

In region III, the deformation is dominated by recovery controlled dislocation creep and the strain rate exhibits grain size insensitivity. As described in a previous analysis of experimental data, the value of measured elongations to failure has an increased trend with the increase of the strain rate sensitivity m [37].

Figure 2- 5 Strain rate dependence of flow stress and strain rate sensitivity[38].

2.2.1.5 Forming temperature

Superplasticity often takes place at high temperatures, above approximately half of the melting point. The temperature has a great influence on the flow stress and strain rate. A wide range of materials were already systematically tested and the corresponding results were also well documented.

The changes with increasing deformation temperature are summarized as follows.

1. The overall level of the flow stress is decreased, and the high strain rates give rise to the least effect. Furthermore, the flow stress of superplastic material has greater temperature dependence than a material with coarse grains.
2. The maximum value of strain rate sensitivity m increase with increasing forming temperature.

3. The transition between the two different regions is displaced to higher strain rates.
4. In the superplastic region II, the value of m is more sensitive to temperature than strain rate.

2.2.2 Stress-Strain behavior

The curves of stress-strain can be related to the experimental process and the averaged view of the operating processes are well reflected. Figure 2-6 describes four types of stress strain curves collected from experiments. Figure 2-6 (a) shows a typical stress strain curve of superplastic material, during which the flow stress keeps almost constant in the superplastic flow. It is denoted as type 'F' (flat).

The continuous strain hardening up to substantial strain values can be seen from the curve in Figure 2-6 (b), and thus it is referred to as type 'H' (hardening). These curves are typical for materials subject to concurrent grain coarsening.

As can be seen, the flow stress increases as the grain size increase. In Figure 2-6(c), the curve shows continuous strain softening which is referred to as type 'S' (softening). This can be attributed to the loss of cross-section area from both internal cavitation and external reduction of area.

Comparatively, Figure 2-6(d) shows a more complicated stress-strain behavior, hereafter referred to as type 'C' (complex). The magnitude of strain hardening in the initial stage can be explained on one hand by the grain coarsening, and on

the other hand, by the subsequent strain softening. This phenomenon is considered to originate from the microstructure of the raw material.

Figure 2- 6 Examples of four different types of stress strain curves observed in superplastic PM aluminum alloys [39].

2.3 Superplastic Deformation

2.3.1 Fundamentals

As an attractive process in aerospace, chemical, transportation and biomedical fields, SPF enables complex parts to be completed in one forming step at elevated temperature through a controlled strain rate by gas pressure, with the required microstructures [40-47]. SPF is considered as an effective technique to fabricate hollow fan blades [48], honeycomb structures [49], bellows expansion joints [50] and large engine components [51, 52].

The major advantage of SPF is its capability to form complex and large parts with little springback and residual stress in single forming step. Therefore the final shape of the part is identical to the shape of mould and this process also improves the dimensional accuracy of the parts. This process helps to reduce manufacturing costs as it allows the production of a complex part in a single operation.

However, the drawbacks of SPF are in the high costs of materials due to its strict fine grain size requirements, as well as its forming conditions of high temperatures and low strain rates. The slow forming rate and high forming temperature will result in many problems, such as reduced tool life and high heating demands. Moreover, the high temperature requirement limits its application in forming of the alloys that are sensitive to oxidation at elevated temperatures, in particular for magnesium alloys [53].

2.3.2 Superplastic forming technology

Of great significance is that in 1970, Backofen [54] at the Massachusetts Institute of Technology showed a simple air pressure operation can be used to form the superplastic Zn-Al alloy into a practical shape as in glassblowing.

Figure 2-7 illustrates the schematic diagrams of the SPF process. Initially, a sheet is clamped and sealed on the die. The gas in die chamber at lower position is vented to atmosphere. Argon gas is normally imposed over the sheet for high pressure forming. Once the sheet contacts with the die corner, the material in this region is locked by friction and further deformation is therefore prevented.

The remaining free regions will continue to expand until the part is net-shape formed.

Figure 2- 7 Schematic of superplastic forming (a) Clamping and sealing (b) Blow forming (c) Fully formed part [55].

Nevertheless, it is rather difficult to prepare superplastic grade material, especially with ultra-fine grain structures. Comparing with other sheet forming methods, another disadvantage must be pointed out: the slow forming rate [56]. The optimum strain rate for the SPF using commercial alloy lies between 10^{-4} s^{-1} to 10^{-3} s^{-1} . Cycle times vary from thirty minutes to two hours in conventional SPF.

In addition, it is inevitable that the high temperatures in SPF would cause lots of drawbacks, leading to relative poor mechanical properties of the products. More microstructural defects and higher amounts of cavitation are induced. For alloys that are sensitive to oxygen, this high temperature requirement would severely limit the applications of SPF. The oxide film formed during forming is harmful to material properties, and it is easy to give rise to damages to the parent material.

Consequently, further research on the sheet forming should focus on increasing the forming rates and decreasing the forming temperatures, and thus alternative processing techniques and better process control need to be developed to improve productivity for higher production volume.

Quick plastic forming (QPF) technology was introduced as a blow forming process, and it is specifically useful for the high volume productions of aluminum panels in automotive application [57, 58]. It was reported that QPF for aluminum alloy components can be implemented at higher forming rates and lower forming temperatures than SPF. Comparatively, the major deformation mechanism of SPF is grain boundary sliding, while QPF benefits from GBS and solute drag creep [58, 59].

The procedure of this technique as shown in Figure 2-8 is to first load the blank onto the blank holder on the die setup and heat the setup to a suitable forming temperature which the material will exhibit good superplastic behavior. After that, the hot drawing step will begin with the hydraulics mechanism in the test rig engaging the punch to draw the blank and exert a compression force on the blank against the blank holder, forming a seal that prevents gas pressure loss during the gas forming stage. When the hot drawing step is completed, SPF will start and the gas blow forming process will complete the rest of the forming process. When the SPF step is completed, the product can be removed from the die cavity and be allowed to cool.

Figure 2- 8 Schematic of the hot draw mechanical pre-forming process (a)

Loading and sealing (b) Hot drawing (c) Superplastic forming [6].

Y. Luo [6] developed a die system that can couple SPF with mechanical pre-forming. This novel technology is able to produce a thickness profile with superior quality by reducing material thinning from a maximum of 53 % to 18% as shown in Figure 2-9. It also significantly decreases forming time of deep draw panels compared with conventional SPF from 20 to 3 min. Experimental results above mentioned also proved that this die design possessed capability to fabricate these non-SPF AA 5182 aluminum sheet and AZ31 magnesium sheet with less formability.

Figure 2- 9 Formed part of SPF 5083 (a) Hot draw mechanical pre-forming SPF

(b) Gas forming only.

Hybrid SPF is defined as a sheet forming process combining hot drawing (mechanical pre-forming [6]) with gas forming [60]. The aim of introducing deep drawing before gas forming is to eliminate the drawbacks of slow forming rate. With the use of a mechanical force in the form of a punch, a partial final shape can be produced on the part before subjecting it to gas forming. Since the part is closer to its final desired shape and dimension after the initial deep drawing, the overall time taken for the part to finish its forming with gas forming is reduced, hence lesser cycle time per part is needed.

Jun Liu [60] also designed a process, in which the stamping and gas blow forming were combined as one operation as shown in Figure 2-10. Compared with the conventional SPF process, the forming time of non-superplastic grade aluminum alloy 5083 was greatly reduced from 30 min to 8 min, together with a forming temperature decreased from 773 K to 673 K. Ti-6Al-4V alloy sheets were successfully formed at 800 °C in 16 minutes by this hybrid forming technique within a non-isothermal heating system [61]. To justify the formed part quality, a number of parameters including gas pressure cycles, material draw-in amount, bulge heights and thickness reduction were tested. It shows that the minimum thicknesses area with reduction of 54 % located at the outward corners. Compared with the conventional SPF process, the formability of non-superplastic grade aluminum alloy was improved by hybrid SPF process.

Figure 2- 10 Forming sequence of the hybrid SPF of non-superplastic AA 5083 sheet (a) Clamping stage (b) Drawing and sealing stage (c) Gas blow forming stage.

The advantages of the hot drawing and the superplastic gas forming are relatively complemented to achieve the aim of quick SPF. By adopting a mechanical pre-forming operation into SPF, hybrid forming can enhance formability and improve production efficiency for automotive and aerospace industries.

The other advantage of the hybrid SPF is it can run the forming process at a much lower temperature, indicating that superplasticity is not required at this condition. Since more materials are introduced in the forming area during hot drawing stage. The following gas forming temperature can be lower and the oxidation rate and grain growth can be limited.

The process also reduces the requirements of the conventional SPF on grain sizes of sheet materials, and allows for the use of ordinary commercial alloy sheet materials and reduces the material cost.

Nevertheless, the research work concerning the hybrid SPF of magnesium alloys is very limited, since the majority of research is concentrated on aluminum alloys and titanium alloys [5, 7].

2.3.3 Superplasticity of magnesium alloys

The early studies of superplastic behavior of magnesium alloys at elevated temperature were primarily on AZ31 and AZ61 alloys [62-65]. Superplastic deformation of AZ31B with an elongation of 475 % at $1 \times 10^{-4} \text{ s}^{-1}$ was proved to be attributed to GBS, while non-superplastic deformation with an elongation of 160 % at $1 \times 10^{-2} \text{ s}^{-1}$ was caused by the intragranular dislocation creep with some contribution from GBS [66].

J. C. Tan [67] enhanced the superplasticity of AZ31B with the grain size of 12 μm from 265 % to 360 % by a two stage deformation method. During stage one of the deformation, specimen deformed to strain of 60 % where 85 % of the homogeneous dynamic recrystallization grains with the grain size of 6 μm can be attained at 250 °C and strain rate of $1 \times 10^{-4} \text{ s}^{-1}$. Subsequently, stage two was performed at 450 °C and $2 \times 10^{-4} \text{ s}^{-1}$ till fracture. Stage one was aimed at refining the coarse grains to the fine equiaxed grains which deformed by GBS accommodated by intragranular slip. Stage two was performed at higher temperature and strain rate, hence dynamic grain growth occurred and the predominant deformation mechanism involved viscous glide accommodated by lattice diffusion.

AZ31 magnesium alloy can be processed into fine grains to achieve superplasticity by heat treatment. M. Cusick et al. [68] managed to control the microstructure of AZ31 with grain sizes ranging from 8 to 15 μm by different annealing treatments. The effects of multiple annealing temperatures and times on SPF were examined by using tensile specimens and bulge sheets. The randomly orientated texture of AZ31 alloy sheet was achieved by using an annealing process at 773 K before and after isothermal rolling at 298-573 K, reported by Masahide Kohzu [69].

Ivo Schindler [70] examined the softening process in hot forming of wrought magnesium alloy AZ31. As shown in Table 2-4, the grain size after recrystallization as a function of the initial grain size, deformation conditions, and annealing temperature is illustrated.

Table 2- 3 Grain size after recrystallization.

Temperature, K	Strain, -	Strain rate, s^{-1}	Initial grain size, μm	Recrystallized grain size, μm	
				Experiment	Calculation
523	0.20	0.1	30.3	10.6	10.9
523	0.20	0.1	38.2	11.9	11.9
523	0.20	0.1	45.6	13.8	12.7
523	0.25	0.1	38.2	10.8	11.2
523	0.15	1.0	38.2	11.2	10.3
523	0.20	1.0	38.2	10.1	9.6
523	0.15	10	38.2	8.3	8.3
523	0.20	10	38.2	7.9	7.8
573	0.10	0.1	45.6	17.8	19.1
573	0.10	1.0	38.2	13.5	14.4
573	0.15	1.0	38.2	12.1	13.0
573	0.15	10	38.2	10.2	10.5
623	0.10	1.0	30.3	16.2	16.0
623	0.10	1.0	38.2	18.2	17.5
623	0.10	1.0	45.6	20.1	18.7
623	0.15	1.0	38.2	16.3	15.8
623	0.15	10	38.2	12.2	12.0
673	0.10	1.0	38.2	20.6	20.6
673	0.15	10	38.2	14.9	15.0
673	0.20	10	38.2	13.6	14.0

K. F. Zhang [71] investigated the formability of AZ31 alloy sheets with grain size of 4.5 μm after hot rolling process. The maximum limit drawing ratio

reached 2.65 at a punch speed of 30 mm/min at 200 °C, whereas the maximum limit dome height was only 10.8 mm. Non-isothermal deep drawing with punch temperature range of 50 to 90 °C is preferred to achieve the best drawability.

2.3.4 Formability improvement of magnesium alloys by severe plastic deformation

A lot of studies have been conducted on magnesium alloy over the past decades to examine the mechanical properties achieved through severe plastic deformation (SPD) [72-74]. Roberto B. Figueiredo et al. [75] studied the influence of the rolling direction on the flow properties and the development of internal cavities in AZ31 alloy processed by equal-channel angular pressing (ECAP). The experimental results showed that the cavities were formed at grain boundaries instead of at precipitates proving that the rolling direction does not influence the flow and cavitation.

R. Lapovok et al. [76] produced a bimodal grain structure of AZ31 (elongation larger than 1200 %) processed by ECAP with a back pressure at low temperature. Zhang Datong et al. [77] reported the AZ31 hot rolled plate with an average grain size of 92 µm was refined to 11.4 µm after the friction stir processing (FSP). The FSP AZ31 alloy exhibited high strain rate superplasticity with the elongation of 268 % at 723 K and $1 \times 10^{-2} \text{ s}^{-1}$.

In these studies, SPD has been demonstrated as an effective way to refine microstructure of magnesium alloy which lead to both ductility and ultimate tensile strength improvements. Fine grain size also reduces mechanical

twinning, improves anisotropy and promotes additional deformation mechanism such as grain boundary sliding [23, 78].

Ghosh [79, 80] investigated the effect of grain refinement and texture on properties of wrought AZ31B Mg alloy plate using a process of alternate biaxial reverse corrugation with decreasing processing temperature from 250 to 170°C. Using this technique, a homogeneous average grain size of 1.4 μm was achieved at cumulative strain of 5.0. The tensile yield strength was found to increase from 160 to 280 MPa and ductility improved with tensile elongation increases from 13.0% to 22.6%. The differences in yield strength for in-plane tension (parallel to the rolling direction) and compression was found to decrease due to the grain refinement as shown in Figure 2-11. The improvement in the tensile-compressive anisotropy was attributed to the reduction of twinning activities in the in-plane compression of the fine grain alloy. However, the difference in true strain at failure between the SPD-processed in-plane tension and compression becomes large.

Figure 2- 11 Stress–strain curves in tension and compression for (a) As-received alloy (b) As-processed alloy (c) Stress vs. strain relationship for normal-to plane compression of the as-received and the as-processed materials.

Miura et al. [81] studied multidirectional forging of AZ31 Mg alloy with a decreasing temperature from 350 to 150 °C. Using this approach a final average grain size of 0.36 μm is developed at strain 4.8 with a final basal texture is approximately 90° to the final compression axis. Tension test were conducted using different orientation (0, 45, 90°) with respect to the final compression axis at 150 °C. It was found that the anisotropy in yield strength in the different orientation are lower at low strain rate of $5 \times 10^{-5} \text{ s}^{-1}$ as compare to higher strain rate $5 \times 10^{-3} \text{ s}^{-1}$ as shown in Figure 2-12.

Figure 2- 12 Effect of MDF on anisotropy at 423K at different strain rates.

In most of the SPD studies, the starting materials are mainly wrought Mg alloy and there are limited studies that examine the effect on cast material. There are also very few studies that investigate the combine effect of alloying and SPD on formability improvement. In one of such studies, M. Janecek [82] investigated the effect of ECAP on an initial squeezed-cast AZ31 alloy. It was reported that the room temperature compressive yield strength increased by 2.2 times. However, at temperature above 200°C the compressive yield strength became worse than the initial squeezed cast material as shown in Figure 2-13.

Figure 2- 13 Yield stress of as cast AZ31 and AZ31 after 0 and 4 passes at different temperature.

R. Mahmudi [83] investigated the effect of alloying using lanthanum-rich mischmetal (RE) and calcium addition followed by ECAP on AZ31 alloy. In comparison between ECAPed AZ31 and AZ31-0.3%Re-0.3%Ca, the effect of alloying elements only marginally increases the ultimate tensile strength (220.2 to 228.6 MPa) while tensile elongation decreases (45.7% to 41.3%) as shown in Table 2-5 . Although alloying reduces the ECAPed grain size from 1.6 to 1.2 μm , the yield strength decreases from 109.2 to 134.6 MPa. It is possible that the greatest ductility improvement came from the texture modification as a result of the ECAP process and rather than alloying with RE elements.

Table 2- 4 Tensile properties after 4 ECAP passes.

Alloy	Yield strength (MPa)	Tensile strength (MPa)	e_r(%)
AZ31 as-extruded	136	208.3	29.7
AZ31	109.2	220.2	45.7
AZ31-0.6%RE	129.8	211.8	35.4
AZ31-0.6%Ca	148.2	233.5	28.5
AZ31-0.3%RE-0.3%Ca	134.6	228.6	41.3

However, the size of magnesium specimen with fine grains processed by SPD method mentioned above is usually quite small, not suitable to deform part with complex shape.

2.4 Deformation Mechanisms

In this section, various mechanisms involved in the superplastic deformation process are analyzed. The most commonly accepted mechanism for superplastic flow is grain boundary sliding, which is accommodated by dislocation slip and

climb, grain boundary migration, grain rotation and recrystallization [66].

Figure 2-14 shows the superplastic deformation mechanism map for magnesium alloys at different strain rates and temperatures.

From various experimental results, it is well recognized that a couple of processes including grain boundary sliding, dislocation motion and diffusional processes take place during superplastic deformation. Meanwhile, in some cases, evidence for recrystallization was confirmed. The previous researches lend strong support to the presence of various mechanisms for superplasticity process and these mechanisms can be applied to a very wide range of materials. It can be deduced that different mechanisms predominate at various conditions and lead to particular combinations of mechanical performances and microstructural properties.

Figure 2- 14 Deformation mechanism maps for Mg alloys constructed on the basis of the modified constitutive equations at (a) 573K (b) 623K (c) 673K [84].

2.4.1 Creep mechanism

Diffusional creep is defined as the high-temperature deformation that results from the transport of matter by diffusion rather than dislocation motion. It usually takes place in materials with fine grains at a relatively high temperatures ($0.9 T_m$). In this process, the atom diffusion is rapid. It is believed that the nonhydrostatic stress together with atom flow from compression to tension sites contribute to activate the diffusion mechanism. Diffusional creep can be further divided into Nabarro-Herring creep and Coble creep.

In Nabarro-Herring creep, the matter transport is implemented in the form of diffusion through the grain lattice, as shown in Figure 2-15. Atoms perform the diffusion process through the lattice following the arrow direction, and a counter flux of vacancies can be seen correspondingly.

Figure 2- 15 Schematic illustration of Nabarro-Herring or diffusion creep under an applied stress σ [85].

The material transported in the diffusion is described as the dashed hexagonal grain which elongates following the direction of the shaded region r . In

polycrystalline pure metals with coarse grains, Nabarro-Herring creep controls the deformation rate at above $0.8 T_m$ with relatively low stresses.

The Coble creep is actually a specialized case of Nabarro- Herring creep. In this creep, the transport of matter is realized by grain boundary diffusion instead of lattice diffusion, as shown in Figure 2-16. The difference between Nabarro-Herring and Coble creep lies in that Coble creep depends more on the grain size than Nabarro-Herring creep. Moreover, Coble creep are usually tested at lower temperatures ($\sim 0.4 T_m$) though occurring in similar materials.

Figure 2- 16 Schematic illustration of Coble creep. Atomic flow occurs along grain boundaries [86].

In general, creep mechanism is realized by not only lattice diffusion but also the grain boundary diffusion. Both of them contribute a lot in the diffusion process [87]. The creep models generally considered stress exponent n as 1, thus the flow stress is proportional to strain rate. Nevertheless, n is appropriately 0.5 in the typical superplastic deformation. Then the grain will be elongated after deformation following the creep mechanism. In practice, the grain shape of many superplastic materials remains equiaxed during superplastic deformation.

Several coarse grained materials have been reported to exhibit enhanced tensile ductility [58, 59]. GBS is not the predominant mechanism due to the coarse grain size. This behavior is the result of solute drag (glide) controlled creep mechanism with strain rate sensitivity m of 0.33.

2.4.2 Grain boundary sliding with various accommodation processes

Grain Boundary Sliding is a deformation mechanisms in which fine grain particles slide past along each other, or in a zone immediately adjacent to their common boundary [88] without the creation of significant voids in the process. This phenomenon was first observed using electron microscopy, and is a process that occurs if there is deformation incompatibility in the structure while still having the necessary conditions for grain boundary sliding operation.

This mechanism has had much attention paid on it due to its dominant role in superplastic formation as well as being an origin of high temperature fracture at grain boundaries [89]. Specifically, there has been general agreement that this mechanism is responsible for more than 50% of the strain found in superplastic materials. For GBS to occur, high angle boundaries are usually required to allow sliding under shearing stresses. In addition, equiaxed small grains as well as presence of melt or fluid film along grain boundaries are also preferred in facilitating this mechanism.

As known, the relative movement of adjacent grains causes superplastic elongations within the polycrystalline matrix. Although the grain boundary sliding acts as the main flow process in superplasticity, this sliding must be

accommodated by intragranular slip so as to avoid the development of internal cavities [90].

The grain boundary sliding is defined as the process in which individual grains slide over each other without changing the grain shape, and meanwhile the deformation occurs either at or in a zone adjacent to their common boundary. To determine the contribution of grain boundary sliding ξ , experiment results concerning the three regions of flow show that region II contribute the greatest among the three regions. . ($> 50\%$) comparing with the other two regions, I and III [91].

Grain boundaries of superplastic materials are usually curved and contain run in dislocations. As a result, local accommodation at ledges and dislocation controlled the sliding rate. Accommodation at triple points is also of great importance.

2.4.2.1 Grain boundary sliding with accommodation by dislocation motion

Ball and Hutchinson proposed that groups of grains slide as a unit until obstructed by a blocking grain as shown in Figure 2-17. The stress concentration is relieved via dislocation motion in the blocking grain. These dislocations pile up against the opposite grain boundary. Further sliding of grains is enabled as the dislocation in the pile up climbs into and along the grain boundaries to annihilation sites.

Figure 2- 17 A schematic illustration of the Ball-Hutchison model grain boundary sliding between a group of grains leading to rate controlling slip in the blocking grain [92].

2.4.2.2 Grain boundary sliding with accommodation by diffusion creep

Ashby and Verrall proposed a much more complex model of grain boundary sliding with accommodation by diffusion creep at triple points, as shown in Figure 2-18. A group of four grains moves from the initial (Figure 2-18(a)), through the intermediate (Figure 2-18(b)), to the final state (Figure 2-18(c)). The grains slide over one another by accommodation strains in the arrow direction could achieve a true strain of 0.55.

Figure 2- 18 A schematic illustration of the grain switching mechanism [93].

The shape of grains temporarily changed in the diffusion process. The diffusive current of grain 1 and grain 2 during boundary diffusion are shown in Figure 2-

18(d) and Figure 2-18(e) respectively. The material effectively insert into the boundaries of two grains in diffusive process.

2.4.3 Recrystallization

It is well recognized that the formation of fine equiaxed grains in superplastic material during deformation is as a result of dynamic recrystallization. The energy which is stored within the material in the form of dislocations can be released by the formation and migration of high angle boundaries.

At temperatures above $0.5 T_m$, the strain hardening of material is counteracted by dynamic restoration processes during plastic deformation, such as dynamic recovery and dynamic recrystallization, as shown in Figure 2-19 [94-96].

Figure 2- 19 Schematic representations of the three types of as-deformed microstructure.

Recrystallization has been broadly defined as the nucleation and growth of stress-free grains in a metal and has been one of the most effective ways to change the microstructure and properties of materials. These new stress-free grain consume the original grains in the growth process while usually resulting in a reduction in strength and harness of the material though increasing their ductility in the process [97].

During recrystallization under the influence of heat, absorption of dislocations and point defects occur through the migration of high angle boundaries. High angle boundaries are high in energy and mobility, while low angle boundaries are low in energy and mobility. Hence, though the rearrangement under the influence of temperature, dislocations at the high angle boundary areas pile up at low angle boundaries. This process results in a reduction in the overall strain energy of the system as well as a lowered in total grain boundary area which is free from defects as shown in Figure 2-20.

Figure 2- 20 Recrystallization grain process (a) Subgrain initial substructure (b) Growth of initial subgrain over smaller neighboring grains (c) Large grain bigger angle boundary formed [98].

As recrystallization is considered an irreversible thermally activated process, it is also important to understand the kinetics of the reaction which tends to follow the profile. However, initial conditions should also be taken into account to accurately map the transition process.

Factors such as those below would have effects on the progress of the recrystallization.

1. Annealing temperature
2. Strain rate
3. Initial grain size
4. Purity of the sample

Dynamic Recrystallization refers to the process of growth and nucleation of new grains during a deformation process. This is done through two main mechanisms: subgrain rotation and grain boundary migration. The latter being a recovery mechanism which accommodates dislocation processes to allow large strains in the material.

2.5 Finite Element Modeling

In the past, studying of processes such as SPF used to be done through numerous experiments in an iterative approach, using various forming parameters which can be time-consuming and tedious. However, advancements in computer technology have provided the capability to use software simulations to aid the study of various manufacturing processes including SPF with knowledge of the relevant models. Finite element analysis (FEA) is

instrumental in the design of the SPF process due to the capability of predicting optimum gas pressure cycles and final thickness distribution profile with the help of accurate constitutive models that defines material characteristics and behaviors [99].

A constitutive model describes material behaviors mathematically with theoretical constitutive laws. A good constitutive model characterizes the relationships between a material's flow stress, strain and strain rate parameters while taking into account other factors such as temperature, deformation characteristics, grain growth, strain hardening, cavitation and deterioration of mechanical properties [100, 101]. An accurate constitutive model is useful in FEA to simulate and predict material deformation and damage reliably. Hence, the development of the right constitutive model is important for the study of processes that involves material behavior, especially for engineers who design manufacturing processes such as SPF. The model allows for optimization of parameters in materials processing as well as selection of suitable material processing methods to achieve the required output.

As the data required formulating the constitutive laws are usually obtained through uniaxial tensile test experiments at high temperatures, human errors produced in these experiments are also incorporated into the constitutive model, resulting in inaccuracy. The experimental data will be used to obtain the coefficients in the constitutive equations [99, 101-103]. Thus, the need to simulate the manufacturing process accurately will require accurate constitutive equations, allowing FEA to be utilized to optimize the process [99].

Friction is an important and complex feature during sheet deformation. Formation and thinning are strong functions of contact, friction and lubrication. The friction coefficients depend on many variables such as forming temperature, strain rate and applied pressure [104]. However, thickness variation due to the friction and springback during hot drawing can be reduced by SPF which is non-contact [105]. In this research, no direct measurement of friction between magnesium sheet and tool with graphite as the lubricant was conducted.

An ideal template for constitutive equations have been introduced that can characterize material behavior over a wide range of thermo-mechanical loads. It is known that microstructure strongly influences the inelastic behavior. The microstructure is largely affected by the chemistry of the material, as well as history of processing that the material have been subjected to [101]. In the case of superplasticity, the factors to be considered in the constitutive model would include temperature, strain hardening effects, grain growth, cavitation effects, as well as deterioration in post-formed thermo-mechanical properties.

It can be characterized by the constitutive equation at a constant temperature:

$$\sigma = f(\varepsilon, \dot{\varepsilon}, d) \quad \text{Equation 2-3}$$

Where σ is the flow stress, $\dot{\varepsilon}$ is the strain rate, ε is strain and d is grain size.

The function can be expressed at a given equilibrium state as

$$\sigma = K \dot{\varepsilon}^m \varepsilon^n d^p \quad \text{Equation 2-4}$$

where

$$m = \frac{\partial(\ln \sigma)}{\partial(\ln \dot{\varepsilon})}, n = \frac{\partial(\ln \sigma)}{\partial(\ln \varepsilon)}, p = \frac{\partial(\ln \sigma)}{\partial(\ln d)} \quad \text{Equation 2-5}$$

K is a material constant, m is the strain rate sensitivity value, n is the strain hardening exponent and p is the grain growth exponent.

It is possible to further simplify the equation above by neglecting grain growth, reducing to an extended power law model as shown

$$\sigma = K\dot{\epsilon}^m \quad \text{Equation 2-6}$$

Constitutive models based on the equations above have been used to produce relatively accurate results in finite element method simulation [106].

Table 2- 5 Superplastic models developed over the years.

Name	Year	Equations	Comments
Ball-Hutchison [92]	1969	$\dot{\epsilon} = K_1(b/d)^2 D_{gb}(\sigma/E)^2$	GBS accommodated by dislocation climb
Langdon [107]	1970	$\dot{\epsilon} = K_2(b/d)^1 D_L(\sigma/E)^2$	Movement of dislocations adjacent to GBs
Ashby-Verrall [93]	1973	$\dot{\epsilon} = K_7(b/d)^2 D_{eff}(\sigma - \sigma_0/E)^2$ $D_{eff} = D_L[1 + (3.3w/d)(D_{gb}/D_L)]$	Rate controlling diffusional accommodation
Gifkins [108]	1976	$\dot{\epsilon} = K_3(b/d)^2 D_{gb}(\sigma/E)^2$	Pile-up at triple points (core-mantle)
Gittus [109]	1977	$\dot{\epsilon} = K_4(b/d)^2 D_{IPB}(\sigma - \sigma_0/E)^2$	Pile-up at interphase boundary
Padmanabhan [110]	1980	$\dot{\epsilon} = K_8(b/d)^2 D(\sigma/E)^2$	Non-rate controlling diffusional accommodation D may differ from D_L and D_{gb}
Arieli and Mukherjee [111]	1980	$\dot{\epsilon} = K_5(b/d)^2 D_{gb}(\sigma/E)^2$	Climb of individual dislocations near GBs
Ruano and Sherby [112]	1984	$\dot{\epsilon} = 6.4 \times 10^9(b/d)^2 D_L/b^2(\sigma/E)^2$	Phenomenological, $T = (0.4 - 0.6)T_m$

Kaibyshev et al. [113]	1985	$\dot{\varepsilon} = K_6 / kT (b/d)^2 D_0 \exp(-Q/kT) (\sigma - \sigma_0/E)^2$	Hardening and recovery of dislocations at GBs
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The models in Table 2-5 have been developed throughout the years by various researchers and each of them can only be applied when certain conditions are present. K_1 to K_8 in the table are material constants, σ_0 denotes the threshold stress, T is the temperature in absolute scale, d is grain size, b is the Burger's vector, Q is activation energy, E is young's modulus and k is the Stefan-Boltzmann constant. D_{gb} , D_L , D_{IPB} and D_{eff} are grain boundary, lattice, interphase boundary and effective diffusion coefficient respectively, and their values can be determined experimentally for the material of interest.

There are also superplastic models developed for use with specific materials to account for difference in material properties and deformation mechanisms [114]. One relationship commonly used to describe AZ31 forming behavior at elevated temperature is

$$\dot{\varepsilon} = A \left(\frac{\sigma}{E} \right)^n \exp(-Q/RT) \quad \text{Equation 2-7}$$

Where A and n are constants, E is the elastic modulus, Q is the activation energy and R is gas constant.

The widely adopted models to analyze SPF processes are the visco-plastic model and the visco-elastoplastic model. The elastic deformation of the material is not very significant to the results and therefore the visco-plastic model is adopted [115].

Most of the SPF simulations are conducted using commercial FEM software, such as ANSYS, ABAQUS and MARC [6, 116-118]. The ability to develop optimum gas pressure cycle is a key factor during the part forming process. However, it is still a complicated work for SPF simulation taking account of deformation mechanisms.

Chapter 3 EXPERIMENTAL PROCEDURE

3.1 Materials

Commercial Mg-3Al-1Zn (AZ31B) hot rolled sheets with thickness of 3.2 mm were used for the high temperature tensile tests and hybrid SPF in this study. This material was supplied by Baoji Yide Metal Material Co., Ltd. The as-received sheets had been cross hot rolled and then annealed to obtain homogeneous mechanical properties. The chemical composition of this AZ31B-O magnesium alloy is provided by the supplier in Table 3-1.

Table 3- 1 Material composition (weight percentage) of AZ31B magnesium alloy.

Mg	Al	Zn	Mn	Cu	Ni	Si	Fe
Remainder	2.88	0.79	0.98	0.00075	0.00089	0.021	0.002

The range of wrought magnesium alloys which is commercially available is very limited. AZ31B is one of the very few commercial magnesium alloys that can be hot rolled into thin sheets with thickness varying from 0.05 mm to 6.0 mm and no special thermal treatment is required.

The hot rolling sequences for AZ31B which is carried out between 300 °C and 450 °C are summarized in Table 3-2.

Table 3- 2 Hot rolling sequences for AZ31B magnesium alloy.

Material	Operation	Temperature (°C)	Thickness reduction (%)
AZ31B	Slab breakdown rolling	425-450	10-20
	Coil rundown rolling	350-440	5-20
	Finishing step	<250	5

3.2 High Temperature Tensile Tests

3.2.1 Tensile test specimen

Figure 3- 1 Dog-bone shaped tensile test specimen (unit: mm).

The dog-bone shaped tensile test specimens were prepared by wire cutting as illustrated in Figure 3-1. Two sets of specimens with gauge length of 20 mm and gauge width of 4 mm were prepared, one set have rolling direction along the length of the specimen, while the other have the rolling direction perpendicular to the length of the specimen.

Tensile tests of these two sets of specimens were performed under the same temperature and strain rate conditions to investigate the material mechanical properties, as well as the effect of rolling direction have on the formability of AZ31B-O sheets. This is important as the blanks will experience material deformation in all directions along its plane during the hot drawing stage and gas forming stage.

3.2.2 Tensile test procedures

Figure 3- 2 (a) Tensile test setup (b) Electrical resistance heater (c) Heater control unit (d) INCONEL high temperature tensile test rods.

The uniaxial tensile tests were conducted using Instron™ 8502 Dynamic Tensile Test Machine as shown in Figure 3-2. Tensile tests were done in a temperature range from 350 °C to 450 °C at 25 °C intervals. Temperature fluctuations were maintained within 5 °C margin from the preset temperature via the Heater Control Unit to ensure test results are of sufficient accuracy.

Due to the difference in test temperatures for each specimen, the time required to heat up to their respective required temperatures varies between 10 minutes to 30 minutes. To prevent possible effects on microstructure due to prolonged heat exposure, the specimen was loaded on to the machine only when the desired temperature was achieved. The specimens were given 5 minutes to

reach thermal equilibrium before the start of each test. Each of the tests were done at constant crosshead displacement velocity, with corresponding initial strain rates from $3 \times 10^{-4} s^{-1}$ to $1 \times 10^{-2} s^{-1}$.

The specimen was quenched in water in order to retain the microstructure of the AZ31B alloy in its deformation stage after the completion of tensile test.

Using the experimental results and data from the series of tensile tests, the material were characterized via graphical plots of parameters such as percentage elongation, true strain, strain rate, temperature and flow stress to gain an insight into the formability of AZ31B alloy.

3.2.3 True stress and true strain

The true stress and strain of the tensile tests results can provide more accurate data to understand the deformation characteristics of AZ31B, as it accounts for the effects of decreasing cross-sectional area as elongation increases. Thus, using instantaneous measurement of actual cross-sectional area instead of using initial cross-sectional area is important in the tensile test study.

In the uniaxial tensile tests however, the raw data of the results generated in the computer program consists of engineering stress and strain measurements. Therefore, it is imperative that the results need to be converted to true stress and strain values via mathematical calculations. The following equations describe the definitions of these stress and strain parameters.

Engineering strain:

$$e = \frac{\Delta L}{L_0} = \frac{1}{L_0} \int_{L_0}^L dL \quad \text{Equation 3-1}$$

True strain:

$$\varepsilon = \int_{L_0}^L \frac{dL}{L} = \ln \frac{L}{L_0} \quad \text{Equation 3-2}$$

$$e = \frac{\Delta L}{L_0} = \frac{L-L_0}{L_0} = \frac{L}{L_0} - 1 \quad \text{Equation 3-3}$$

$$e + 1 = \frac{L}{L_0} \quad \text{Equation 3-4}$$

$$\varepsilon = \ln \frac{L}{L_0} = \ln(e + 1) \quad \text{Equation 3-5}$$

Engineering stress:

$$s = \frac{P}{A} \quad \text{Equation 3-6}$$

True strain:

$$\sigma = \frac{F}{A} = \frac{F}{A_0} \frac{A_0}{A} \quad \text{Equation 3-7}$$

$$\frac{A_0}{A} = \frac{L}{L_0} = e + 1 \quad \text{Equation 3-8}$$

$$\sigma = \frac{P}{A_0} (e + 1) = s(e + 1) \quad \text{Equation 3-9}$$

3.3 Tooling Design for Hybrid Superplastic Forming

In order to create a better and more efficient manufacturing process, the methodology of manufacturing components in a single operation through hybrid SPF is proposed. This technology combines a hot drawing process and a gas blow forming process as shown in Figure 3-3. The blank is first held in place between the clamp and the die cavity. Hybrid SPF starts with a deep hot drawing process, where the punch moves up to deform the blank, giving it a partial deformed shape. Lastly, similar to conventional SPF, pressurized gas

enters the chamber and exerts a pressure onto the blank, forcing it to take the shape of the die cavity.

Figure 3- 3 Schematic of the hybrid superplastic forming process.

Figure 3-4 shows the die setup for this hybrid SPF process. A view of the overall die setup inside the furnace is shown in Figure 3-5. By using this hybrid SPF process, it is possible to achieve better material draw in and allow faster forming of components in a single process.

Figure 3- 4 Schematic of hybrid superplastic forming die setup.

Figure 3- 5 Overall die setup in furnace.

3.3.1 Conical female die

The conical die as designed in Appendix A is used to demonstrate the different level of formability with various dome heights. The formability of material increases as the blank sheet is drawn into the die cavity deeper. The die cavity has a conical geometry with a rounded tip as shown in Figure 3-6.

Figure 3- 6 Conical die installed inside furnace.

3.3.2 Blank holder

The blank holder is used to hold the blank in place during the forming process as designed in Appendix B. As the punch is engaged, the disc springs are compressed, a force is exerted on the blank through the blank holder to hold the blank firmly. The guide pins on the other hand, assists the loading of blank quickly as they allow blank sheet to be positioned centrally on the setup. The disc-springs made from INCONEL material are used to apply the load on the blank holder.

3.3.3 Punch

The punch as in Appendix C is specifically designed for hybrid SPF. The punch is removable so that study on the benefits of the hybrid SPF process is possible by comparing it with results from conventional SPF methods without the use of the punch. It is also possible to study the effects of punch shape parameters on hot drawing results. The punch with flexible length from 25 mm to 50 mm can be achieved by placing a spatial plate with different thicknesses.

3.3.4 Sealing system

The sealing system as designed in Appendix D is used to ensure no gas leaking during the SPF process. The sealing purpose is achieved by a lock bead in this process. It forms a seal which allows for achieving the required gas pressure during SPF by preventing argon gas from escaping when pressure is applied on the blanks against it.

3.4 Hybrid Superplastic Forming

The hybrid SPF experiments of AZ31B were carried out in the Murdock 140-10000 SPF/DB machine as shown in Figure 3-7. The machine is capable of achieving a pressing force of 90.7 tons and a maximum working temperature of about 982 °C. The maximum gas pressure the machine can supply for the gas forming stage is 600 PSI, utilizing argon gas to carry out gas forming to avoid reaction at elevated temperatures. The test rig consist of a furnace and a control panel unit which controls the temperature in the furnace as well as the raising and lowering of the bottom platen in the furnace, which acts as the driving force for the hot drawing step.

Figure 3- 7 (a) Hybrid superplastic forming machine (b) Furnace (c) Control Panel (d) Programmable Logic Controllers.

The system is controlled by Programmable Logic Controllers (PLC), where the required parameters are set through the touchscreen display on the control panel of the test rig. The pressure cycle can be set via this method and the system will automatically adjust the internal conditions in the furnace so that the argon gas pressure would match the preset value at a specific time. The pressure cycle that would produce the required optimum strain rate is estimated through FEM simulations with suitable constitutive model using ABAQUS software package. The input parameters include material behaviors, interaction, load, strain rate and temperature.

During the forming process, the hot drawing first formed the part partially from the starting metal sheet rapidly in several seconds, and then a designed gas

forming process is applied to achieve the desired conical shape by high gas pressure.

3.4.1 Hot drawing

Based on the die design, the initial sheet size with an additional metal allowance of hot rolled AZ31B magnesium alloy was determined to be $165 \times 165 \text{ mm}^2$ for a good sealing condition as shown in Figure 3-8. The blank is allowed to be heated for 5 minutes in the furnace to reach thermal equilibrium before the hybrid SPF experiment begins.

During hot drawing, the amount of non-superplastic grade AZ31B material draw-in is related to the drawing temperature and the punch stroke. At the hot drawing stage, the sheet metal with Crown 8078 dry graphite lubricant on both sides was drawn into the conical die cavity by different punches at a constant speed until it reached a total displacement of 25 mm at 400 °C. The formed part after hot drawing is shown in Figure 3-9.

Figure 3- 8 AZ31B blank for hybrid superplastic forming.

Figure 3- 9 AZ31B formed part after hot drawing.

3.4.2 Gas forming

When the hot drawing step was completed, the gas blow forming process started and completed the rest of the forming process. To prevent oxidation, argon gas was then applied on the pre-formed sheet for gas blow forming. The pressure-time cycle, which was input into the Murdock 140-10000 SPF/DB forming press by minute, was predicted from simulation by ABAQUS to maintain the sheet to be formed at a target strain rate. Figure 3-10 shows a constant gas pressure cycle, the applied gas pressure can achieve 220 PSI after 10 mins in experiment.

Figure 3- 10 Pressure cycle applied on sheet.

The height of the formed part, thickness profile and signs of damage or rupture on the formed parts are indicators of the quality of the hybrid superplastic formed part. The formed part after gas forming is shown in Figure 3-11.

Figure 3- 11 AZ31B formed part after gas forming.

3.5 Microstructural Examinations

3.5.1 Electron backscatter diffraction (EBSD)

Electron Backscatter Diffraction is a Scanning Electron Microscope (SEM) based microstructural-crystallographic technique used to extract information of the crystallographic orientation of samples. This method can be used to index and identify the seven different crystal systems, and is commonly used in crystal orientation mapping, defect studies, and phase identification studies as it can abstract information regarding crystal orientation, misorientation statistics, grain size etc. As such, EBSD has become a well-established technique for SEM due to its ability to abstract crystallographic information with relative ease.

The physical properties of materials such as Young's modulus, hardness, plasticity, permeability are often anisotropic with respect to the crystallographic direction in which they are measured. EBSD can probe not only the proportions of each orientation component present in a specimen, but also the distribution of these components in the microstructure.

Traditional grain size measurements rely on observation of grain boundaries in an image of the microstructure. It has become apparent since the advent of EBSD that not all grain boundaries are revealed by the conventional etching route. This is especially true for the so-called 'special' boundaries, including twin and low angle boundaries. Since a grain is defined essentially as a unit of uniform crystallographic orientation, EBSD is an ideal tool for grain size measurement. Figure 3-12 illustrates the overall setup of EBSD.

Figure 3- 12 Experimental setup for EBSD [119].

The SEM fires a focused beam of electrons onto the well-polished surface of a specimen tilted angle at about 70° . The diffracted or backscattered electrons from the specimen forms a pair of Kossel cones for each reflecting plane, as shown in Figure 3-13. These Kossel cones then get projected on the EBSD detector screen, producing pairs of Kikuchi lines. These bands represent each of the lattice diffracting crystal planes and with them, the crystal structure of the specimen can be investigated.

Figure 3- 13 Formation of Kikuchi band [120].

3.5.2 EBSD specimen preparation

After hybrid SPF, the formed part was cut into two equal halves using wire cutting for further characterizations as shown in Figure 3-14. To investigate the deformation mechanism, the maximum thinning area during hot drawing and gas forming were examined by electron backscatter diffraction (EBSD). The samples were initially ground and mechanically polished, followed by electro-polishing in AC2 electrolyte solution which was provided by the supplier Struers at room temperature and 15 V for 20 s. The detailed grinding and polishing conditions were summarized in Table 3-3. The EBSD examinations were conducted using a JEOL 7600F field emission scanning electron microscope with an Oxford Instruments HKL EBSD system as shown in Figure 3-15.

Table 3- 3 Mechanical preparation method for AZ31B magnesium alloy.

Step	1	2	3	4	5	6
Surface	SiC paper #1200	MD-Largo	MD-Dac	MD-Dac	MD-Chem	MD-Chem
Speed (rpm)	200	150	150	150	150	150
Suspension		DP-Spray 9 μm	DP-Spray 3 μm	DP-Spray 3 μm	DP-Spray 1 μm	OP-S 0.04 μm
Lubricant	water	DP-Yellow	DP-Yellow	DP-Yellow	DP-Yellow	DP-Yellow
Force (N)	20	20	25	10	10	10
Time (min)	1:30	5:00	2:30	2:30	3:00	5:00

Figure 3- 14 Formed part cut into two equal halves.

Figure 3- 15 JEOL 7600F field emission SEM with EBSD system.

3.6 Finite Element Modeling

In this study, FEM was conducted to predict the hot drawing and gas forming process using commercial finite element analysis software ABAQUS. ABAQUS allows the user to define more complicated material constitutive in additional subroutines, which are written in the programming language FORTRAN.

The material model is first described in user subroutine by FORTRAN, and then implemented in ABAQUS. The data from uniaxial tensile tests were used to describing the deformation mechanisms of AZ31B during biaxial gas blow forming. At the hot drawing stage, the die was fixed and the punch moved down at a constant speed with displacement of 25 mm in 5 seconds. The thickness prediction of part was sensitive to friction. The friction coefficient of 0.2 was chosen to predict the interaction between the blank sheet and tool with graphite as the lubricant [104]. At the gas forming stage, the solution dependent gas pressure was applied on the metal sheet to maintain a target strain rate. Thus, the predicted pressure cycle curve can be obtained from history output results. The purpose of this FEM is to predict the SPF behavior of AZ31 magnesium sheet at 400 °C accurately.

Chapter 4 HYBRID SUPERPLASTIC FORMING OF AZ31B SHEETS

The hybrid SPF technique utilizes hot drawing process before the conventional SPF to form a part in one operation. During the forming process, the hot drawing first formed the part partially from the starting metal sheet rapidly in several seconds, and then a designed gas forming process is applied to achieve the desired conical shape by high gas pressure. The hybrid SPF process aims to achieve the advantages of both mechanical pre-forming (a relatively fast process) and SPF which allows for high formability.

4.1 Hot Drawing

4.1.1 Effect of punch shape on formability

In the hot drawing stage, the non-superplastic grade AZ31B magnesium sheet with size $165 \times 165 \text{ mm}^2$ and thickness 3.2 mm was drawn into the die cavity by punch in 5 seconds. Three different punches were used to investigate the effects of punch shape on the formability of AZ31B sheets in hybrid SPF as shown in Figure 4-1. The parameters of these three punches are listed in Table 4-1.

Figure 4- 1 Schematic of the punch (unit: mm).

Table 4- 1 Punches used in hybrid SPF.

	Punch 1	Punch 2	Punch 3
D_p	50	70	70
r	20	25	30

4.1.2 Hot drawing temperature

To investigate suitable temperature for AZ31B in hybrid SPF which consists of hot drawing and gas forming, the metal sheet was subjected to hot drawing at temperatures 250 °C and 400 °C.

At 250 °C, the metal sheet was ruptured due to the lack of slip systems at low temperature while on the other hand at 400 °C, the sheet metal formed well as shown in Figure 4-2.

Figure 4- 2 Part formed by hot drawing at (a) 250 °C (b) 400 °C.

4.1.3 Thickness distribution of part formed by hot drawing

Thickness profiling was carried out for components formed at 400 °C. In order to investigate the impact of the hybrid SPF process, thickness profiling was also done for components formed using only hot drawing and components formed with only gas forming under similar conditions. Using only gas blow forming will replicate the performance of conventional SPF process. This was done to provide a baseline comparison, and to highlight other impacts on the quality of formed components.

The AZ31B sheet was subjected to a pressure of 245 PSI by conventional SPF to the height of hot drawing at 400 °C. It took 6 minutes to form the part with same height as hot drawing, much longer than hot drawing which is 5 seconds.

The cross-sections of the formed AZ31B parts at 400 °C by conventional SPF and hot drawing using different punches are shown in Figure 4-3. The thickness profiling of each formed part was done through a numbering system where each point on a formed AZ31B will be compared with the similar point on another formed AZ31B paired. Each point is about 10 mm from each other. The locations chosen for thickness measurement are labelled by numbers from the dome center to the edge and therefore help identify the best hot drawing conditions for AZ31B.

Figure 4- 3 Part formed by different punches after hot drawing.

The thickness distribution of AZ31B parts formed by hot drawing using different punches is shown in Figure 4-4. It reveals that the shape of punch has great effect on the thickness profile of part formed by hot drawing. The initial thickness of the blank was 3.2 mm. The thickness profile of formed part under hot drawing shows a more uniform thickness distribution as compared to the part formed under conventional gas forming. The maximum thinning 33.8% occur at the die radius area where the material first pulling into the die cavity during conventional gas forming.

Figure 4- 4 Thickness distribution of AZ31 parts formed at different conditions.

Comparing with the conventional gas formed part, thickness distribution at the die radius area for the part formed by hot drawing is much better. As the punch radius increases from 20 mm to 25 mm, thickness at point 2 which is 10 mm from dome center can be significantly improved. However, the thickness decreases when the punch radius increases from 25 mm to 30 mm. Hence,

punch 2 with D70 r25 is chosen for further study as it has relatively uniform thickness distribution.

By integrating a drawing process to create this sheet metal forming procedure, drawbacks such as slow forming rates of conventional SPF technique can be addressed. This is important for industries which manufacture complex parts with SPF in large volumes. The hybrid SPF process significantly reduces cycle time through the partial formation of the required part through hot drawing, which can be done in 5 seconds. The gas forming time and the overall time required for the forming of a component is therefore reduced.

Since the part will be partially formed by hot drawing, the amount of superplastic deformation required will be smaller compared to conventional SPF and therefore materials used in this technique do not require the level of fine grain size needed for optimal superplastic characteristics. The production costs can thus be lowered as the materials used do not require special grain refinement processes to enhance their superplastic characteristics.

4.2 Gas Forming

4.2.1 Gas pressure cycle

4.2.1.1 Constant gas pressure

Traditional constant gas blow forming was carried out using gas pressure from 170 PSI to 320 PSI. Various attempts were taken to determine the suitable gas pressure range for hybrid SPF. From the forming results as summarized in Table 4-2, it is better to control the gas pressure between 200 PSI and 320 PSI to achieve high formability of AZ31.

Table 4- 2 Hybrid superplastic forming results using constant gas pressure.

Pressure (PSI)	Punch 1	Height (mm)	Forming time (min)
170	D_p 50 r 20	37	20
200	D_p 50 r 20	45	25
250	D_p 50 r 20	51 (crack at 16 min)	16
320	D_p 50 r 20	30 (crack at 6 min)	6

4.2.1.2 Gas pressure curve

Using numerous simulations by FEM software ABAQUS, an estimation of the different strain rates on AZ31B during gas forming can be obtained. The effects of varying strain rates from the simulation allow the narrowing down of a few pressure cycles that yield the optimal forming process on AZ31B, which then can be used for experimental study.

In this simulation, the material's creeping behavior is modeled after a hyperbolic sine law model. After inputting the necessary variables, the gas pressure amplitude can be obtained from the simulation of hot drawing and gas forming process as shown in Figure 4-5. The oscillations of load are due to the solution-dependent amplitude calculation method in software.

Figure 4- 5 Solution-dependent amplitude from simulation model.

Figure 4- 6 Gas pressure-time curve in experiment.

Figure 4-6 shows the pressure cycle used in the hybrid SPF experiment. The gas pressure forming time is about 60 minutes, and the entire hybrid SPF process can be completed within 65 minutes, much faster than conventional SPF techniques that require hours to form a component, even after including the

time for loading and unloading of the blank as well as the time needed for mechanical hot drawing step.

The rate of change of pressure is important as it influences the forming strain rate of blank sheet. From the tensile test results, it is evident that the strain rates have a major effect on the formability of AZ31B, hence rate of change of pressure can affect the quality of the formed component and the performance of the gas forming step.

During the actual experiment, Punch 2 was used for hot drawing and forming temperature 400 °C was kept constant throughout. Table 4-3 to Table 4-6 shows the four gas pressure cycles used in the experiment. All four formed AZ31B parts using different pressure cycle experienced cracks prior to their completion time of 60 mins as shown in Figure 4-7.

Table 4- 3 Gas pressure cycle setting 1, S1.

Time (min)	1	2	3	4	5	6	10	11	60
Pressure (PSI)	104	107	107	139	169	185	185	221	221

Table 4- 4 Gas pressure cycle setting 2, S2.

Time (min)	1	2	3	9	10	11	12	13	14	15	20	25	30	35	40
Pressure (PSI)	27	34	40	50	62	89	108	150	220	250	375	300	320	340	350

Table 4- 5 Gas pressure cycle setting 3, S3.

Time (min)	1	2	3	4	5	12	15	22	24	60
Pressure (PSI)	50	100	170	207	245	245	207	207	170	170

Table 4- 6 Gas pressure cycle setting 4, S4.

Time (min)	1	2	3	10	11	17	18	60
Pressure (PSI)	162	194	232	232	194	194	162	162

Figure 4- 7 Formed AZ31B part with gas pressure cycle (a) S1 (b) S2 (c) S3 (d) S4.

Hence in order to determine which pressure cycle settings yield the best formability, a comparison test is used base on the dome height of formed AZ31B part as shown in Table 4-7.

Table 4- 7 Experimental results of formed part with different gas pressure cycle.

Gas pressure cycle setting	Formed height (mm)	Time prior to failure (min)
S1	60	59
S2	51	29
S3	48	22
S4	50	29

4.2.2 Failure of formed part

Figure 4-8 shows some of the types of failure encountered while performing hybrid SPF with different parameters. These failures results in a loss of gas pressure during the gas forming stage, and as a result, the parts cannot be fully formed. Figure 4-8 (a) was a result of excessive blank holder force 80 PSI after the mechanical hot drawing step that restricts material flow and causes high degree of thinning at the die radii area. Figure 4-8 (b) shows a defect caused by cavitation effect.

Figure 4- 8 Failure due to (a) High blank holder force (b) Cavitation.

Figure 4- 9 Formed part after gas forming (a) AZ31B-H24 (b) AZ31B-O.

Figure 4-9 shows the parts formed by gas pressure cycle S1 using two different temper materials AZ31B-H24 and AZ31B-O. During the sealing stage, the

flange area of sheet was clamped firmly by sealing bead. Due to the existence of texture, AZ31B-H24 as shown in Figure 4-9(a) exhibited anisotropy of deformation. The effect of texture on the mechanical forming properties of AZ31B was attributed to the development of preferred grain orientation [121]. It is assumed that deformation of AZ31B-O sheet is isotropic at elevated temperature.

Through multiple attempts in an iterative approach, as well as with the aid of simulation software, it was possible to narrow down the optimal gas pressure range of hybrid SPF experiments for AZ31B. However, these parameters apply only to non-superplastic grade AZ31B magnesium alloys. If other materials are to be used in hybrid SPF, another study will have to be carried out to investigate the material characteristics and formability.

4.2.3 Thickness distribution of part formed by SPF

The cross-sections of the formed AZ31B parts at 400 °C by conventional SPF and hybrid SPF using gas pressure cycle S1 are shown in Figure 4-10. The dome height of the formed part, thickness profile and signs of rupture on the formed part are indicators of the quality of the part formed at their respective conditions.

Figure 4- 10 Cross sections of formed AZ31B parts at 400 °C (a) Hybrid SPF (b) Conventional gas forming.

The part was successfully formed by hybrid SPF with maximum bulge height 60 mm in 58 mins. The dome height of formed part was 60 mm under optimum conditions, approximately 60 % of the die diameter. In contrast, the part formed by conventional SPF without punch using the same gas pressure cycle cracked at height 60 mm in 27 mins.

Critical points were selected to be used as a basis of comparison for parts formed at different conditions to investigate the quality of the formed part. The

minimum thickness of 1.31 mm after hybrid SPF was found at location 3 where the material first came into contact with the punch as shown in Figure 4-11. The corresponding thickness reduction is 59%. The AZ31 sheet was stretched by the moving punch during hot drawing stage, resulting in thickness reduction at the contact area. The thickness was sensitive to friction constant as well as the gas pressure. The friction constant was quite high using graphite as the lubricant during hot drawing.

In contrast, without the influence of the mechanical punch, location 1 became the thinnest as it was located in the center of the blank, where experienced the highest deformation during SPF. The part formed by conventional SPF cracked at location 1 dome center.

The thickness comparisons reveal that the part formed by hybrid SPF presents more even material distribution, leading to better forming ability than the part deformed by conventional SPF. Therefore, the formability of non-superplastic grade AZ31 magnesium alloy was significantly improved by hybrid SPF and the cost of raw material required for SPF was reduced.

Figure 4- 11 Thickness distribution of AZ31 parts formed by hybrid SPF and conventional gas forming.

4.3 Summary

As compared to the conventional SPF process, the optimized hybrid SPF improved the formability of non-superplastic grade AZ31B alloy at 400 °C using gas pressure cycle S1 and punch 2. The conclusions can be made as follows:

1. The thickness profile of part formed by conventional SPF to the height of hot drawing shows a more uniform thickness distribution than the part formed by hot drawing. The maximum thickness reduction is 33.8% at the die radius area where the material first pulling into the die.
2. The influence of punch shape on the formability of AZ31B during hot drawing was investigated. As the punch radius increase from 20 mm to 25 mm, thickness distribution is greatly improved. Comparing with the

conventional gas formed part, thickness at the die radius area of the part formed by hot drawing is much better. Hence, punch 2 with D70 r25 is chosen as it is the best punch for further study.

3. Non-superplastic grade AZ31B magnesium alloy sheets were successfully formed with dome height 60 mm by hybrid SPF. The forming was performed at 400 °C by involving a hot drawing and gas forming stage. In contrast, the part formed by conventional SPF technology cracked at the same height.
4. The minimum thickness of 1.31 mm after hybrid SPF was found at location 3 where the material first came into contact with the punch, and the corresponding thickness reduction is 59%. Comparing with the conventional SPF process, the thickness distribution of formed part was significantly improved.
5. As compared to conventional SPF, hybrid SPF combines the use of a mechanical process (hot drawing) before the conventional SPF. This combination not only allows reduce the forming time, it also reduces the material cost by using the non-superplastic grade material. The formability of coarse grain AZ31B is significantly improved. It makes hybrid SPF a more ideal choice for future manufacturing methods.

Chapter 5 MICROSTRUCTURE EVOLUTION OF AZ31B

5.1 Microstructures of Tensile Specimen at 400 °C

5.1.1 As-received material

Specimens from longitudinal (rolling direction), transverse (perpendicular to rolling direction) and short transverse (thickness direction) direction were examined by EBSD. The average grain size, average misorientation angle and fraction of high angle grain boundaries (HAGBs) are summarized in Table 5-1. It is evident that the as-received material possessed similar microstructure in LS and TS direction.

Table 5- 1 Microstructure of as-received AZ31B in EBSD map.

Direction	Average grain size (μm)	Average misorientation angle ($^{\circ}$)	Fraction of HAGBs
LT	12.0	22.3	0.6
LS	11.4	14.4	0.37
TS	10.7	14.8	0.41

The typical microstructure of as-received commercial AZ31b-O is shown in Figure 5-1 to Figure 5-3. Low angle grain boundaries (LAGBs, between 2° and 15°) and high angle grain boundaries (HAGBs, $>15^{\circ}$) were depicted as white lines and blank lines in EBSD map respectively. The theoretical distribution for magnesium with random orientations was also displayed in a blank solid curve.

GBS is easy to take place in material with small grain size, high misorientation angle and even grain size distribution during forming process.

Non-uniform grain shapes and grain orientations are observed within the sheet material. It is clear that there are many subgrains with white lines in the coarse grains. The fine grains evolved as a result of static recrystallization during annealing treatment. And the coarse grains were formed due to the static grain growth.

The AZ31B-O sheet with grain aspect ratio lower than 2 is treated as a fully recrystallized microstructure during the high temperature forming process. Complete annealing would result to a homogeneous microstructure with equiaxed grains. The mean grain size of the as-received material is determined to be 11.1 μm .

Figure 5- 1 EBSD map of as-received AZ31B in LT plane.

Figure 5- 2 EBSD map of as-received AZ31B in LS plane.

Figure 5- 3 EBSD map of as-received AZ31B in TS plane.

5.1.2 Grip region

In order to elucidate the microstructure evolutions in hot rolled AZ31B, both static grain growth and dynamic grain growth were investigated. Specimens for static grain growth study were sectioned from the grip regions of tensile specimen which formed at strain rate 10^{-3} s^{-1} and temperature 400°C . The grip region revealed static grain growth while the gauge region exhibited dynamic grain growth.

The typical microstructures of AZ31B-O tensile specimen at grip region are shown in Figure 5-4 to Figure 5-7. The specimens were exposed in air at 400°C for 200 s, 600 s, 1000 s and 2155 s respectively. The average grain size, average misorientation angle and fraction of HAGBs are summarized in Table 5-2. It is obvious that the grains are thermally unstable and the grain size increases exponentially with forming time. For the specimen with true strain 1.15, the average grain size increased from $11.1 \mu\text{m}$ to $19.4 \mu\text{m}$. The fraction of HAGBs was approximately 0.75 at forming time 200 s, 600 s, and 2155 s.

Table 5- 2 Microstructure of specimen at grip region in EBSD map.

Time (s)	Average grain size (μm)	Average misorientation angle ($^\circ$)	Fraction of HAGBs
200	14.0	28.3	0.79
600	16.2	28.9	0.74
1000	16.5	24.4	0.61
2155	19.4	30.8	0.75

Figure 5- 4 EBSD map of tensile specimen at grip region (200 s) in LT plane.

Figure 5- 5 EBSD map of tensile specimen at grip region (600 s) in LT plane.

Figure 5- 6 EBSD map of tensile specimen at grip region (1000 s) in LT plane.

Figure 5- 7 EBSD map of tensile specimen at grip region (2155 s) in LT plane.

The static grain growth was found to following the Equation 5-1 [122].

$$d_s = (d_0^N + Ct)^{\frac{1}{N}} \quad \text{Equation 5-1}$$

Where d_s is the static grain size, d_0 is the initial grain size, C is constant for a particular material at certain temperature, t is the time at temperature and N is grain size exponent.

The relationship of grain size at grip region and time is shown in Figure 5-8. The curve fitting parameters describing the static grain size model is presented in Table 5-3.

Figure 5- 8 Static grain growth expressed as a function of time.

Table 5- 3 Parameters describing the static grain size of AZ31B at 400 °C.

Parameter	Static grain size model
d_0 (μm)	11.1
C (μm s ^{-N})	188770
N	6.7

The grain boundaries of AZ31B material gain additional energy at high temperature and hence driven to migrate into lower energy grains [123]. And the lack of dispersoid particles at the grain boundaries could not impede the migrating of grain boundary. This leads to the rapid grain growth in AZ31B at high temperature.

In this study, the blank sheet took approximately 5 minutes to reach thermal stabilization at elevated temperature, and then followed by hot drawing or conventional SPF.

5.1.3 Gauge region

In this section, the microstructure evolution and dynamic grain growth under the influence of stress at gauge region were examined. Specimens for dynamic grain growth study were tested at strain rate 10^{-3} s^{-1} at 400 °C. The microstructure of specimen in LS plane which contained the longitudinal and short transverse direction was examined by EBSD. The EBSD maps of AZ31B-O tensile specimen with different strain levels at gauge region are shown in Figure 5-9 to Figure 5-12.

Table 5-4 lists the average grain size, average misorientation angle and fraction of HAGBs. The fraction of HAGBs is approximately 0.6 at strain levels of 18%, 47%, 69% and 115%, much lower than the value at grip region which is about 0.75. HAGBs at adjacent grains are more prone to sliding when shear stress is applied. Fine grains with high angle boundaries are capable of GBS during straining at the optimum conditions.

Table 5- 4 Microstructure of specimen at gauge region in EBSD map.

Time (s)	Average grain size (μm)	Average misorientation angle ($^{\circ}$)	Fraction of HAGBs
200	13.7	22.9	0.57
600	14.5	20.1	0.56
1000	14.7	22.2	0.56
2155	15.3	23.2	0.59

Obvious difference of the grains can be observed as compare to the grip region, many subgrains with white lines are found in the coarse grains. In addition, the grains are slightly elongated along the tensile direction with increased grain aspect ratio which explains the grain morphology evolution during straining.

The grain boundaries become ‘serrated’ as the subgrain within the deformed grain grows into its neighbor to form a bulge free of dislocations [124]. The subgrains were observed in the coarse grains of AZ31B during deformation at various strain levels.

The grip region of tensile specimen was only subjected to static grain growth due to free from stress. In contrast, the gauge region sustained both static grain growth and dynamic grain growth under the influence of stress. Thus, the main deformation mechanism is dynamic recrystallization and GBS accommodated by dislocation climb during high temperature deformation.

Figure 5- 9 EBSD map of tensile specimen at gauge region with strain 0.18 in LT plane.

Figure 5- 10 EBSD map of tensile specimen at gauge region with strain 0.47 in LT plane.

Figure 5- 11 EBSD map of tensile specimen at gauge region with strain 0.69 in LT plane.

Figure 5- 12 EBSD map of tensile specimen at gauge region with strain 1.15 in LT plane.

The dynamic grain growth model is described by Sato et al. [125]. This model assumes that the contribution from static and dynamic grain growth can be added together to estimate the final grain size.

$$d = d_s \exp(\alpha \varepsilon) \quad \text{Equation 5-2}$$

Where d is the final grain size, α is a constant describing dynamic grain growth.

Thus, the grain growth law can be written as

$$d = (d_0^N + Ct)^{\frac{1}{N}} \exp(\alpha \varepsilon) \quad \text{Equation 5-3}$$

Figure 5-13 shows the ratio of average grain size between the gauge region and grip region after tensile test at 400 °C. The grain growth model was fit to the experimental data, the fitting parameters were listed in Table 5-5.

Figure 5- 13 The ratio of grain size between the gauge region and grip region plotted against true strain.

Table 5- 5 Parameters describing the grain size of AZ31B at 400 °C.

Parameter	Grain size model
$d_0 (\mu m)$	11.1
$C (\mu m s^{-N})$	188770
N	6.7
α	-0.21

5.2 Microstructures of Part Formed at 400 °C

As discussed in Section 4.2.3, the maximum sheet thinning after hybrid SPF was found in the vicinity of location 3 where the material first came into contact with punch. Thus, the microstructures of formed parts after hot drawing and gas forming at this location were examined by EBSD.

5.2.1 Part formed by hot drawing using punch 2

The EBSD map of part formed by hot drawing is shown in Figure 5-14. The average grain size is 5.1 μm with aspect ratio 3.2 and a high peak value of HAGBs around 87° is observed. It is widely accepted that the misorientation between the twinned and the untwinned lattice is 86.3° [126, 127]. Many lenticular type twins are observed near the big grains due to mechanical twinning.

The average misorientation angle increases to 40.7° and fraction of HAGBs increases to 0.52, indicating that the main deformation mechanism during hot drawing stage is viscous glide.

Figure 5- 14 EBSD map of part formed by hot drawing.

5.2.2 Part formed by hybrid SPF

At the hybrid SPF stage as shown in Figure 5-15, obvious differences can be observed when compared with the hot drawing sample. The dynamic recrystallized grains with equiaxed shape are distributed evenly throughout the whole zone. In comparison with the microstructure of as received material, the average grain size increases from 11.1 μm to 15.3 μm . It appears that most of the grains have grown larger. The twins generally diminished during gas forming due to the recrystallization [25].

The fraction of very low LAGBs with misorientation below 5° increases significantly to 0.68 due to the dislocation climb during deformation. In addition, the subgrains in white lines within the coarse grains seem more randomly distributed, which have not completely migrated into HAGBs during hybrid SPF.

Figure 5- 15 EBSD map of part formed by hybrid SPF.

5.2.3 Part formed by conventional SPF

Figure 5-16 shows the microstructure of part formed by conventional SPF to the height of hot drawing. As discussed in section 4.1, it took 6 minutes to form this

part. From the EBSD map, it can be observed the average grain size increases from 11.1 μm to 14.3 μm . The fraction of HAGBs is 0.54, similar to the value at gauge region during uniaxial tensile test.

Figure 5-17 shows the EBSD map of part formed by conventional SPF to the height of hybrid SPF. As the AZ31B sheet was further formed by argon gas, some small cavities depicted as black points in Figure 5-17 were observed in the specimen. Both the average misorientation angle and fraction of HAGBs decrease with increasing strain. This explains well the generation of subgrain via dislocation motion during SPF. Energy is stored within the material in the form of dislocations during plastic deformation. The stored energy can be released through recovery, recrystallization and grain coarsening [128].

Therefore, the main deformation mechanism of non-superplastic grade AZ31 magnesium alloy was a combination of recrystallization, dislocation climb and GBS during hybrid SPF.

Figure 5- 16 EBSD map of part formed by conventional SPF to the height of hot drawing.

Figure 5- 17 EBSD map of part formed by conventional SPF to the height of hybrid SPF.

5.2.4 Cavitation phenomenon

During GBS controlled high temperature deformation, the grain boundaries should be mobile to avoid the stress concentration at the triple points of grains. By grain boundary migration, stress concentration can be reduced and hence avoid the nucleation of cracks. However, the concentration of strain induced vacancy is significant in AZ31B due to low diffusivities. Such larger size cavity is formed due to the nucleating of smaller neighboring cavities into a single large one. With sufficient amount of such cavities will eventually lead to cracks and flaws appearing on the formed AZ31B part.

The captured images from SEM of hybrid SPF and conventional SPF are shown in Figure 5-18 and Figure 5-19 respectively. The cavities are highlighted in white boxes. It can be seen, not only the amount of cavitation in AZ31B part formed by conventional SPF significantly more than that by hybrid SPF, the size of the cavities is larger as well. The higher amount and larger size cavities reflects the reason why the formed AZ31B under conventional SPF experienced a much earlier failure as compare to hybrid SPF, given both using the same pressure cycle. The nucleation of several large cavities eventually formed cracks on the formed AZ31B, leading to failure during conventional SPF. The presence of cavities in the formed parts can adversely affect their mechanical properties.

Figure 5- 18 SEM image for hybrid SPF.

Figure 5- 19 SEM image for conventional SPF.

5.3 Summary

The microstructure evolutions and deformation mechanism of non-superplastic grade AZ31B in uniaxial tensile tests and hybrid SPF were investigated.

The conclusions are summarized as follows:

1. The microstructures of tensile specimens from the grip region and gauge region were investigated. The grains in the grip region appear to be coarser due to static grain growth during tensile test. The grains in the gauge region were refined due to the dynamic recrystallization and formation of equiaxed grains. The static and dynamic grain growth relationships were also investigated during high temperature deformation.
2. Many subgrains with low misorientation angle were observed in the coarse grains. The subgrains seem more randomly distributed within the material during SPF. Both the average misorientation angle and fraction of HAGBs decrease with increasing strain. This explains well the generation of subgrain via dislocation motion during SPF. The main deformation mechanisms during hybrid SPF was determined to be a combination of recrystallization, dislocation climb and GBS.
3. Due to the presence of fewer and smaller cavities, AZ31B part formed by hybrid SPF is structurally stronger as compared to conventional SPF. The few and smaller cavities together with better thickness distribution highly suggests that the mechanical properties of AZ31B part formed by hybrid SPF is improved.

Chapter 6 FINITE ELEMENT MODELING OF AZ31

High temperature tensile tests of non-superplastic grade AZ31B sheets were conducted in air at temperatures from 375 °C to 450 °C and initial strain rates from $3 \times 10^{-4} \text{ s}^{-1}$ to $3 \times 10^{-2} \text{ s}^{-1}$. Tensile tests are useful for establishing material models as the stress-strain data can be obtained from the load force and displacement. The hybrid SPF behavior of AZ31B magnesium sheet at 400 °C was studied by ABAQUS. The FEM results of thickness distribution, thinning characteristics and forming height were compared with the experimental results.

6.1 High Temperature Behavior of AZ31B

6.1.1 Tensile direction parallel to rolling direction

Figure 6- 1 True stress-strain curve of AZ31B parallel to rolling direction.

Figure 6-1 describes the relationship between the true stress and true strain of AZ31B specimens with rolling direction parallel to elongation at different temperatures. The plastic stress is dependent not only on the forming temperature but also on the strain rate [129, 130]. The experimental data from tensile tests show that the flow stress decreases with increasing testing temperature and slower strain rate. Generally, the material exhibits weak strain hardening at slow strain rate $3 \times 10^{-4} \text{ s}^{-1}$ and elevated temperature. The increased tensile stress may be because of the occurrence of dynamic grain growth.

Reasonably high elongations larger than 80% can be achieved over a wide range of strain rates at various temperatures. It is obvious that the elongation of AZ31B is highly dependent on the strain rate and forming temperature. The true stress – strain curves at 400 °C show similar profiles as that at 375 °C. The flow stress decreases with increasing strain, and a sharp decrease is found for strain

rate greater than $3 \times 10^{-3} \text{ s}^{-1}$. This is due to the dynamic recrystallization, the deformed grains are replaced by recrystallized grains during the hot deformation process. For the curve at 450 °C, the flow stress shows a wide range of steady state throughout the deformation.

The flow stresses (at true strain of 0.1) as a function of strain rate at various temperatures are plotted in Figure 6-2.

Figure 6- 2 Flow stress of AZ31B parallel to rolling direction at different temperatures.

In order to numerically characterize the behavior pattern of the sample as it undergoes the tensile test, the strain rate sensitivity index or strain-hardening exponent (m) is introduced. This value is dependent on the true stress, true strain and k , a strength coefficient of the material which is equal to the true stress at a true strain of 1.0. The relationship of the variables is based on the equation below

$$\sigma = k\dot{\epsilon}^m \quad \text{Equation 6-1}$$

Where σ is the true flow stress, k is a constant and $\dot{\epsilon}$ is the true strain rate.

This m value represents the material's ability to resist necking as well as characterizes the stability and overall deformation during superplastic flow, where a value of 1 corresponds to a Newtonian viscous flow which represents high formability. This value is dependent on factors such as strain rate and ambient temperature [38]. Superplasticity is based on the empirical condition of $m \geq 0.3$, and commonly lies in the range of 0.3-0.8 [131].

The m value can also be determined based on the true stress – true strain graph. This value is equal to the graphical slope of the log true stress vs. log true strain rate curve.

$$m = \frac{\ln(\sigma_2/\sigma_1)}{\ln(\dot{\epsilon}_2/\dot{\epsilon}_1)} \quad \text{Equation 6-2}$$

Where σ_1 , σ_2 and $\dot{\epsilon}_1$, $\dot{\epsilon}_2$ are flow stresses and strain rates before and after each jump at a particular strain level.

For this AZ31B material, the m value is about 0.22 to 0.26 throughout the entire strain rate range. Creep mechanisms active at lower forming temperature and faster strain rates, the typically m value of dislocation climb creep is approximately 0.2 [132]. The m value of solute drag creep is approximately 0.3, which falls in between the values for GBS and dislocation climb creep [66]. During uniaxial tensile test, any inhomogenities present within the cross section area of specimen will generate strain localization. The discontinuity growth rate

is controlled by the rate of strain hardening and strain rate sensitivity exponent m . When m is low, neck grows sharply leading to lower elongation to failure.

6.1.2 Tensile direction perpendicular to rolling direction

Due to the radial direction in which metal sheets are drawn into the die cavity when performing hybrid SPF, there is a need to investigate the tensile properties of AZ31B in the direction perpendicular to its rolling direction. Hence, a second set of tensile tests were carried out to study the material characteristics.

Figure 6- 3 True stress-strain curve of AZ31B perpendicular to rolling direction.

Figure 6-3 shows the true stress-strain curves at various strain rates for the second set of tests. The results show similar trends to that in the first set of experiments, where the elongation occurs in a direction parallel to the rolling direction of the material. The fundamental characteristics of AZ31B magnesium alloy does not change drastically with the rolling direction at high temperature.

Figure 6- 4 Flow stress of AZ31B perpendicular to rolling direction at different temperatures.

Figure 6-4 shows the trend of flow stress with varying strain rates at different temperatures for AZ31B perpendicular to rolling direction. The sheet orientation has little effect on the tensile test results. This indicated that AZ31B-O does not exhibit planar anisotropy under these forming conditions. This agrees well with the conclusions of Abu-Farha [133].

In general, greater elongation to failure of AZ31B magnesium alloy can be attained at higher test temperature. However, magnesium alloy is sensitive to oxidation at high temperature. It was observed that the fracture tensile test specimen had dark surface with serious oxidation at 450 °C. From the experimental tensile test results, AZ31B specimen exhibits good formability with little oxidation at 400 °C. Therefore, the temperature chosen for hybrid SPF was 400 °C to minimize oxidation problem.

6.2 Material Model

6.2.1 Hyperbolic sine creep law model

A hyperbolic sine creep law material model 1 is used to control the gas forming strain rate of AZ31B. The relationship of the flow stress with forming temperature and strain rate is used to describe the high temperature deformation behavior by Sellars and Tegart [134].

$$\dot{\epsilon} = A[\sinh(\alpha\sigma)]^n \exp(-Q/RT) \quad \text{Equation 6-3}$$

where $\dot{\epsilon}$ is the strain rate, A is the structure factor, α is the material constant, σ is the flow stress (takes peak value), n is the stress exponent, Q is the activation energy, R is the gas constant (8.31 J/mol), T is the deformation temperature.

To calculate the value of α in Equation 6-3, following two equations were adopted to describe the relationship between flow stress and strain rate at elevated temperature.

$$\dot{\epsilon} = A_1 \sigma^{n_1} \exp(-Q/RT) \quad \text{Equation 6-4}$$

$$\dot{\epsilon} = A_2 [\exp(\beta\sigma)] \exp(-Q/RT) \quad \text{Equation 6-5}$$

where A_1 , A_2 and n_1 are constants. The slope of curve $\ln\dot{\epsilon} - \ln\sigma$ represents $n_1 = \frac{\partial \ln\dot{\epsilon}}{\partial \ln\sigma}$ as shown in Figure 6-5. The relationship of $\ln\dot{\epsilon}$ and σ is shown in Figure 6-6, from the slope $\beta = \frac{\partial \ln\dot{\epsilon}}{\partial \sigma}$ can be obtained. Thus, the value of $\alpha = \beta/n_1$ is estimated to be 0.0363.

Figure 6- 5 The relationship between $\ln\dot{\epsilon}$ and $\ln\sigma$.

Figure 6- 6 The relationship between $\ln \dot{\epsilon}$ and σ .

Zener-Hollomon parameter is considered as a hyperbolic sine function of σ , which is given by [135]

$$Z = \dot{\epsilon} \exp\left(\frac{Q}{RT}\right) = A[\sinh(\alpha\sigma)]^n \quad \text{Equation 6-6}$$

By taking natural logarithms of both sides of Equation 6-6

$$\ln Z = \ln \dot{\epsilon} + \frac{Q}{RT} = \ln A + n \ln(\sinh(\alpha\sigma)) \quad \text{Equation 6-7}$$

$$\sigma = \frac{1}{\alpha} \ln \left\{ \left(\frac{Z}{A} \right)^{\frac{1}{n}} + \left[\left(\frac{Z}{A} \right)^{\frac{2}{n}} + 1 \right]^{\frac{1}{2}} \right\} \quad \text{Equation 6-8}$$

$$Q/RT = \ln A - \ln \dot{\epsilon} + n \ln(\sinh(\alpha\sigma)) \quad \text{Equation 6-9}$$

The activation energy Q was calculated through Equation 6-10:

$$Q = R \left[\frac{\partial \ln(\sinh(\alpha\sigma))}{\partial (1/T)} \right]_{\dot{\epsilon}} \left[\frac{\partial \ln \dot{\epsilon}}{\partial \ln(\sinh(\alpha\sigma))} \right]_T \quad \text{Equation 6-10}$$

in which the first item stands for the slope of curve $\ln(\sinh(\alpha\sigma)) - \frac{1000}{T}$ as shown in Figure 6-7, and the second item stands for the slope of curve $\ln \dot{\epsilon} -$

$\ln(\sinh(\alpha\sigma))$ as shown in Figure 6-8. The slopes of the curves are 5.534 and 3.969 respectively. As a result, the activation energy in Equation 6-10 is calculated to be 182515 J/mol.

Figure 6- 7 The relationship between $\ln(\sinh(\alpha\sigma))$ and $1000/T$.

Figure 6- 8 The relationship between $\ln\dot{\epsilon}$ and $\ln(\sinh(\alpha\sigma))$.

According to the value of Q , the Z parameter at different deformation conditions can be obtained from Equation 6-7. The relationship between Z and $\ln(\sinh(\alpha\sigma))$ is shown in Figure 6-9. The values of A and n are calculated to be 3.638×10^{11} and 3.968 respectively.

Figure 6- 9 The relationship between $\ln Z$ and $\ln(\sinh(\alpha\sigma))$.

When substituting all the above values into Equation 6-4, the constitutive equation of AZ31B magnesium alloy at 400 °C can be expressed as:

$$\dot{\varepsilon} = 3.638 \times 10^{11} [\sinh(0.0363\sigma)]^{3.97} \exp(-182515/RT) \quad \text{Equation 6-11}$$

6.2.2 Grain boundary sliding and dislocation climb creep model

Recent studies suggest that the high temperature forming behavior of AZ31 is controlled by two creep mechanisms. Taleff et al. [118] developed a model that accounts for both grain boundary sliding creep and dislocation climb creep at elevated temperature. At high temperatures and slow strain rates, grain boundary sliding creep controls deformation. At lower temperatures and faster strain rates, dislocation climb creep controls deformation.

$$\dot{\varepsilon} = \frac{A_{GBS}}{d^p} \sigma^{n_{GBS}} + A_{DC} \sigma^{n_{DC}} \quad \text{Equation 6-12}$$

where $\dot{\varepsilon}$ is true strain rate, A_{GBS} and n_{GBS} are constants that describe GBS in AZ31B, d is the average grain size, p is the grain size exponent, σ is true stress, A_{DC} and n_{DC} are constants that describe dislocation creep in AZ31B.

The first part of this two-term material model indicating the true strain rate due to GBS while the second part shows the true strain rate due to dislocation climb creep.

$$d = (d_0^N + Ct)^{1/N} \exp(\alpha \varepsilon) \quad \text{Equation 6-13}$$

Static grain growth and dynamic grain growth were studied in Section 6.1. Table 6-1 lists the parameters of the grain size model in Equation 6-13.

Table 6- 1 Parameters for grain size model of AZ31B at 400 °C.

Parameter	Grain size model
$d_0 (\mu m)$	11.1
$C (\mu m s^{-N})$	188770
N	6.7
α	-0.21

The deformation is almost completely due to dislocation climb creep at strain rate $10^{-2} s^{-1}$ and faster. The contribution from grain boundary sliding is ignored due to the fast strain rate. As a result, the parameters for dislocation climb model are described in Table 6-2. The data obtained from tensile test are used to fit the model parameters as shown in Figure 6-10.

Table 6- 2 The parameters of two-term material model 2.

Parameter	Grain size model
$A_{DC} (s^{-1}MPa^{-n_{DC}})$	5.4×10^{-12}
n_{DC}	6.16
$A_{GBS} (s^{-1}MPa^{-n_{GBS}} \mu m^p)$	1.4×10^{-3}
n_{GBS}	3.5
p	4

Figure 6- 10 The relationship between true stress and true strain rate.

$$\dot{\epsilon} = \frac{1.4 \times 10^{-3}}{[(11.1^{6.7} + 188770t)^{1/6.7} \exp(-0.21\epsilon)]^4} \sigma^{3.5} + 5.4 \times 10^{-12} \sigma^{6.16} \quad \text{Equation 6-14}$$

The two-term material constitutive model 2 was written in FORTRAN based on user-defined subroutine CREEP in Appendix E.

6.3 FEM Simulation

The FEM simulation model which comprises of punch, conical die and a quarter metal sheets is presented in Figure 6-11(a). All the tools are modeled

using analytical rigid elements as the geometry is quite simple. This reduces the CPU time and simplifies the contact definition. The edge node of the blank was fixed during simulation as shown in Figure 6-11(b). The meshed metal sheet with C3D8R element for hot drawing is shown as Figure 6-11(c). The number of elements through the thickness is increased to capture the deformation behavior through the thickness accurately. The widely adopted models to analyze SPF processes are the visco-plastic model and the visco-elastoplastic model [115].

For AZ31B material, Young's modulus is 45000 MPa and Poisson's ratio is 0.34 in elastic material property. Two material models as discussed in 6.2 are adopted in visco-plastic material property. Regarding the addition of keywords to the model for creep strain rate control, it can be done through CAE by using the tab model/edit keywords/model, which allows for the modification to be saved with the *.cae file.

Figure 6- 11 FEM model for hot drawing (a) Tools (b) Boundary condition (c) Meshed blank (d) Hot drawing results.

Figure 6-12 shows the FEM model for hybrid SPF. After hot drawing, the mechanical pre-formed part is imported from *.odb file and then remeshed for hybrid SPF. The mesh density is refined using partitions and edges seeding at locations where the large strains are expected to occur. Firstly several edges are drawn in a horizontal plane in the feature wire. Secondly the part is partitioned using partition/cell-Extrude/sweep edges by selecting one after the other one the different horizontal edges, then extruding along direction and selecting a vertical edge on the part as the extrusion direction. This operation is repeated for the 4 arcs drawn before deleting the horizontal edges to avoid meshing them.

Figure 6- 12 FEM model for hybrid SPF (a) Tools (b) Meshed blank (c) Hybrid SPF results.

Figure 6-13 shows the FEM model for conventional SPF. At the gas forming stage, the solution dependent gas pressure is applied on the metal sheet to maintain a target strain rate. Thus, the predicted load amplitude can be obtained from history output results as shown in Figure 6-14. Together with the gas pressure applied on the sheet, the gas pressure cycle for experiment can be determined. The decrease in gas pressure prior to complete is thought to be caused by the thinning of metal sheet.

Figure 6- 13 FEM model for conventional SPF (a) Tools (b) Meshed blank (c)

Conventional SPF results.

Figure 6- 14 Gas pressure amplitude obtained from history output results.

6.4 Discussion

The hybrid SPF and conventional SPF experimental results of AZ31B at 400 °C, such as dome heights and thickness profiles have been adopted in the comparison. The experimental and prediction results using the above two material models are compared as listed in Table 6-3 and Table 6-4. The gas load in simulation is modified according to the gas pressure cycle used in experiment which is adjusted by minute.

Table 6- 3 Dome height of experimental and prediction results.

Part	Model 1	Model 2	Experiment
Hot drawing	27.8	27.8	28
Hybrid SPF	75.3	72.9	60
Conventional SPF to the height of hot drawing	28.1	25.5	28
Conventional SPF to the height of hybrid SPF	68.9	44.1	60

Table 6- 4 Minimum thickness of experimental and prediction results.

Part	Model 1	Model 2	Experiment
Hot drawing	2.72	2.72	2.52
Hybrid SPF	0.33	0.29	1.31
Conventional SPF to the height of hot drawing	2.37	2.48	2.54
Conventional SPF to the height of hybrid SPF	0.27	1.12	0.75(crack)

Figure 6-15 shows the experimental and prediction results of thickness profile after hot drawing. The thickness distribution is plotted in Figure 6-16. The simulation results of both two models agree well with the experimental results with maximum error 4.6%. It reveals that the material constitutive models

provide relatively accurate predictions of hot drawing process. The visco-plastic material property ‘creep’ has little effect on the deformation results, as the hot drawing process is completed within 5 seconds.

Figure 6- 15 Thickness profile of part formed by hot drawing

(a) Experiment (b) Model 1 (c) Model 2.

Figure 6- 16 Thickness distribution comparison of part formed by hot drawing.

The thinning characteristics of hybrid SPF from experiment and simulation are shown in Figure 6-17 and Figure 6-18. Both two models predicted dome height of part formed by hybrid SPF much higher than the experimental result 60 mm. In addition, the maximum thickness reduction occurred at the dome pole in simulation. However, the thinnest area was at location 3 where the blank sheet came into contact with the punch by experiment.

For hybrid SPF simulation, the mechanical pre-formed part was imported from the *.odb file of hot drawing and then the part was remeshed without any material history data, which would result in inaccuracy. The AZ31B material experienced hardening with high stored energy by punch during the hot drawing stage. However, the gas forming simulation of blank with initial residual stress would have convergence issue using the creep law.

Figure 6- 17 Thickness profile of part formed by hybrid SPF

(a) Experiment (b) Model 1 (c) Model 2.

Figure 6- 18 Thickness distribution comparison of part formed by hybrid SPF.

Comparison results of part formed by conventional SPF to the height of hot drawing are shown in Figure 6-19 and Figure 6-20. The part was predicted with dome height error 0.4% and maximum thickness error 6.7% using hyperbolic sine creep law material model 1. In contrast, the part was predicted with dome height error 8.9% and maximum thickness error 2.4% by two-term material model 2.

Figure 6- 19 Thickness profile of part formed by conventional SPF to the height of hot drawing (a) Experiment (b) Model 1 (c) Model 2.

Figure 6- 20 Thickness distribution of part formed by conventional SPF to the height of hot drawing.

For the part further formed by conventional SPF to the height of hybrid SPF, the results are shown in Figure 6-21 and Figure 6-22. Neither the hyperbolic sine creep law material model 1 nor the two-term material model 2 could predict the dome height and thickness distribution well. There is an increase in deviation between the prediction and experimental results when the material undergoes larger strain deformation during the conventional SPF.

The two-term material model assumes GBS depends on average grain size. However, even the metal sheets with same nominal composition may have different initial grain size. Additionally, the material used in this experiment was commercial non-superplastic grade AZ31B-O with great deviation in grain size. From grain size statistics in EBSD, the standard deviation of part formed by conventional SPF to the height of hot drawing was 9.7 μm . Many fine grains

were observed around the coarse grains. The grain size can affect the strain rate as well as the magnitude of flow stress. The strain rate will decrease with increasing grain size during the GBS controlled SPF process [136]. Therefore, a material model accounts for grain size deviation may lead to a better simulation result.

Figure 6- 21 Thickness profile of part formed by conventional SPF to the height of hybrid SPF (a) Experiment (b) Model 1 (c) Model 2.

Figure 6- 22 Thickness distribution of part formed by conventional SPF to the height of hybrid SPF.

6.5 Summary

The hybrid SPF behavior of non-superplastic grade AZ31B at 400 °C was studied by ABAQUS using two different material models. The FEM results of thickness distribution, thinning characteristics and forming height were compared with the experimental results.

The conclusions are summarized as follows:

1. Base on the data from tensile tests, parameters of hyperbolic sine creep law model and two-term material constitutive model were determined at 400 °C.
2. FEM models for hybrid SPF and conventional SPF were established in ABAQUS. At the gas forming stage, the solution dependent gas pressure was applied on the metal sheet to maintain a target strain rate. Thus, the load amplitude curve was obtained from history output results.

The gas pressure cycle predicted from simulation was used for SPF experiment.

3. The hybrid SPF and conventional SPF experimental results of AZ31B at 400 °C, such as dome heights and thickness profiles have been adopted in the comparison. For the hot drawing process, the simulation results of both two models agree well with the experimental results.
4. Both two models predicted the dome height of part formed by hybrid SPF much higher than the experimental result 60 mm. In addition, the maximum thickness reduction occurred at the dome pole in simulation. However, the thinnest area was at location 3 where the blank sheet came into contact with the punch by experiment.
5. For conventional SPF, neither the hyperbolic sine creep law material model 1 nor the two-term material model 2 could predict the dome height and thickness distribution well. There is an increase in deviation between the prediction and experimental results when the material undergoes larger strain deformation during the conventional SPF.

Chapter 7 CONCLUSIONS AND SUGGESTIONS FOR FUTURE WORK

7.1 Conclusions

In the current scope of study, a series of uniaxial tensile tests were conducted to gain an insight into the mechanical properties of non-superplastic AZ31B magnesium alloy. Investigations were carried out for the formability, thickness profile and microstructure evolution of AZ31B magnesium sheets formed by hybrid SPF. Finite element simulations were performed by ABAQUS software package to obtain the pressure cycle during the hybrid SPF, which plays a significant role for achieving high formability of the target material.

From the results, the conclusions can be deduced as follows:

1. Magnesium alloy sheets of non-superplastic grade AZ31B were successfully formed by hybrid SPF at 400 °C. The dome height was 60 mm under the optimum conditions, approximately 60 % of the die diameter. In contrast, the part formed by conventional SPF technology cracked at the same height. The fewer and smaller cavities together with better thickness distribution highly suggest that the mechanical properties of AZ31B part formed by hybrid SPF are improved.
2. The part formed by conventional SPF shows a more uniform thickness distribution as compared to the hot drawing part with the same forming height. The maximum thickness reduction of 33.8% was found at the die radius area where the material initially pulling into the die.
3. As compared to conventional SPF, hybrid SPF combines the use of a mechanical process (hot drawing) before the conventional SPF. This combination not only allows reduce the forming time, it will also reduce

the material cost by using the non-superplastic grade material. The formability of coarse grain AZ31B is significantly improved. It makes hybrid SPF a more ideal choice for future manufacturing methods.

4. Some of the damages that occurred during the experiments can be observed near the tip of the conical shaped as well as near the base of the component. The damaged observed near the tip is likely due to cavitation phenomenon and the damage near the base is likely related to excessive blank holder force.
5. EBSD results indicate the main deformation mechanisms during hybrid SPF include recrystallization, dislocation climb and GBS. This is evident by the presence of subgrains with low misorientation angle observed in the coarse grains during SPF. The average misorientation angle and fraction of HAGBs decrease with increasing strain. This explains well the generation of subgrains via dislocation motion during SPF.
6. The elongation of the AZ31B magnesium alloy is a function of strain rate and temperature. Higher elongation is achieved at higher temperature and lower strain rate, indicating that higher ductility is achievable as temperature increases and strain rate decreases.
7. Based on the tensile test results, parameters of hyperbolic sine creep law model and two-term material constitutive model were determined at 400 °C. The hybrid SPF behavior of non-superplastic grade AZ31B was predicted by ABAQUS using these two material models. The FEM results of thickness distribution, thinning characteristics and forming height were compared with the experimental results.

8. The hybrid SPF and conventional SPF experimental results of AZ31B at 400 °C, such as dome heights and thickness profiles have been adopted in the comparison. For the hot drawing process, the simulation results of both two models agree well with the experimental results.
9. Both two models predicted dome height of part formed by hybrid SPF much higher than the experimental result. From simulation, the maximum thinning area occurs at the dome pole while in experiment the blank sheet near the punch radius has the maximum thickness reduction.
10. For conventional SPF simulation, the deviations between the simulated and experimental results increase with increasing strain.

7.2 Suggestions for Future Work

The future work on hybrid SPF should involve the application of more materials at various temperatures. More applications of FEM simulations can also be applied to optimize of the process. Details of the suggested improvements are as follows:

1. Currently the mechanical punch with constraint stroke has been used in the hot drawing process. Future work can use longer or shorter lengths by placing the special plate to explore the effects of different hot drawing parameters. More detailed studies of hot drawing are worth carrying out as it has great effect of the thickness distribution of pre-formed part.
2. Establishing material constitutive model account for the differences between uniaxial tensile tests and biaxial gas pressure forming experiments. As the non-superplastic grade AZ31B material used in this study has great deviation in grain size, the two-term material model depends on grain size should be modified.
3. In this study, the use of FEM simulations is limited to deriving the pressure cycle to be used in hybrid SPF experiments at 400 °C. In future, FEM can be applied to a more wide range of applications. Impact of different pressure cycles, temperatures and blank shapes can be studied with the use of software simulations.

Appendix A: Drawing of conical female die

Appendix B: Drawing of blank holder

Appendix C: Drawing of punch

Appendix D: Drawing of sealing system

Appendix E: User-defined code for two-term material constitutive model

3 SUBROUTINE

 CREEP(DECRA,DESWA,STATEV,SERD,EC,ESW,P,QTILD,

1

 TEMP,DTEMP,PREDEF,DPRED,TIME,DTIME,CMNAME,LEXIMP,LEND,

2 COORDS,NSTATV,NOEL,NPT,LAYER,KSPT,KSTEP,KINC)

C

 INCLUDE 'ABA_PARAM.INC'

C

 CHARACTER*80 CMNAME

C

 DIMENSION DECRA(5),DESWA(5),STATEV(1),PREDEF(*),DPRED(*),

1 TIME(2),COORDS(*),EC(2),ESW(2)

C

C DEFINE CONSTANTS

C Real A1, A2, AN1, AN2, p, d, GS1, GS2, GS3, GS4

C

 AN1=3.5

 A2=5.4E-012

 AN2=6.16

 p=4

 GS1=TIME(2)*188770

 GS2=11.1**6.7+GS1

 GS3=EXP(-0.21*EC(2))

GS4=0.1492537

d=GS2**GS4*GS3

A1=0.0014/(d**p)

STATEV(1)=d

C

DECRA(1) = A1*(QTILD**AN1)/(d**p)*DTIME

1 +A2*(QTILD**AN2)*DTIME

IF(LEXIMP.EQ.1) THEN

DECRA(5) = AN1*A1*(QTILD**(AN1-1.))/(d**p)*DTIME

1 +AN2*A2*(QTILD**(AN2-1))*DTIME

write(7,*) 'Grain Size=', d

END IF

C

RETURN

END

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