Chapter 5

# Superplastic Behaviour of Selected Magnesium Alloys

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Additional information is available at the end of the chapter

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**Abstract**

Superplastic materials exhibit anomalous plasticity, achieving strain until several thousand per cent. The phenomenon of plasticity is limited on special microstructure, temperatures and strain rates. Magnesium and magnesium alloys are known as materials with limited plasticity. This is due to their hexagonal structure of these materials. Finding the superplasticity conditions has a crucial importance for applications of magnesium alloys. In this chapter, we will deal with the superplastic behaviour of AZ91, QE22, AE42 and EZ33 magnesium alloys. Materials were prepared by various techniques: thermomechanical treatments, equal channel angular pressing, hot extrusion, rolling, friction stirring and high-pressure torsion. Strain rate sensitivity and elongation to fracture were estimated at various temperatures. Mechanisms of superplastic flow are discussed. Grain boundary sliding and diffusional processes were depicted as the main mechanisms responsible for high plasticity of these alloys. On the other hand, cavitation at elevated temperatures deteriorates the superplastic properties.

**Keywords:** AZ91 magnesium alloy, QE22 magnesium alloy, AE42 magnesium alloy, EZ33 magnesium alloy, superplasticity, strain rate sensitivity, activation energy

## 1. Introduction

A stable tensile deformation is realised when no localisation (necking) takes place and the cross-sectional area, A, decreases uniformly along the sample length. In such case, the following condition must be fulfilled [1]:

$$\frac{dln\dot{A}}{dlnA} = 0 \tag{1}$$

Materials, which show linear (Newtonian) viscosity, have the strain rate sensitivity parameter m

$$m = \frac{\mathrm{dln}\,\sigma}{\mathrm{dln}\,\dot{\varepsilon}} = 1. \tag{2}$$

Here, $\sigma$ is the true stress and $\dot{\varepsilon}$ is the strain rate (SR). The materials exhibit no necking and they deform to large extension before fracture. Such behaviour is typical for molten glass and polymers where $m$ approaches unity. Metals and alloys may have similar $m$ values under certain regimes of temperatures and strain rates. This phenomenon of 'unusual' deformation of materials up to several thousand per cent is known as *superplasticity*. Superplasticity is being investigated both for its scientific merit in the context of physical mechanisms operating during plastic deformation and fracture and for its technological significance in the industry. Several review articles summarise both results concerning to superplasticity of various materials and models explaining superplastic flow on the basis of physical mechanisms, e.g., [2–4]. Hexagonal alloys came into the focus of researcher later together with the increasing demand of the industry for magnesium alloys [5, 6].

Three general requirements for the occurrence of superplasticity are necessary:

- Fine microstructure of equiaxed grains with the grain size typically less than 10 μm; reasonably stable during deformation;
- Temperature which is higher than 0.5 $T_M$ ($T_M$ is the melting point temperature in K);
- Strain rate which is typically not too high (higher than $10^{-2}$ $s^{-1}$) and not too low (less than $10^{-6}$ $s^{-1}$).

Magnesium and magnesium polycrystalline alloys exhibit low plasticity because of their hexagonal structure. For compatible deformation of polycrystals, the activity of five independent slip systems is necessary [7]. Magnesium and magnesium alloys deform in many possible glide systems with dislocations of Burgers vector <***a***> = $1/3\langle 11\bar{2}0\rangle$ in basal, prismatic and first-order pyramidal planes and with dislocations of Burgers vector <***c*** **+** ***a***> = $1/3<11\bar{2}3>$ in first- and second-order pyramidal planes. The main deformation mode in magnesium is basal slip of <***a***> dislocations in (0001) planes. The secondary conservative slip may be realised by the <***a***> dislocations on prismatic and pyramidal planes of the first order. These slip systems are not crystallographic equivalent with the basal slip system. The critical resolved shear stress for deformation in these systems at lower temperatures is much higher. This shortage of slip systems with easy movable dislocations is the reason for small plasticity of hexagonal metals. Finding the conditions for superplastic deformation may help to overcome this disadvantage. As it will be shown later, superplastic deformation is realised not only by dislocation motion but also by other mechanisms as grain boundary sliding and diffusional transport contributing to the forming of a material. Conditions for the superplastic flow are different for various magnesium alloys depending on their melting point, microstructure, preparation method and diffusional characteristics. The crucial problem is to develop the fine-grained polycrystals with low cost, low forming stress, low forming temperature and high forming rate. In order to reduce grain size, several processes have been used: severe plastic deformation, powder metallurgy techniques, rapid solidification, friction stirring or hot rolling. In this chapter, superplasticity of four commercial magnesium alloys is reported. QE22 and EZ33 alloys contain besides Ag and Zn, respectively, rare earth elements and zirconium. AE42 alloy contains only Al and rare earth elements without Zr. A most frequently used magnesium alloy AZ91, usually prepared as the cast alloy, exhibits very low plasticity. All these alloys exhibit potential of a thermal treatment. Possible mechanisms of the superplastic flow are discussed.

## 2. Materials and experiments

### 2.1. AZ91 magnesium alloy

Cast magnesium alloy AZ91 (nominal composition Mg-9Al-1Zn-Mn0.3, in wt%) has a very low ductility especially at low temperatures. A thermal treatment of the alloy may improve the mechanical properties. Formed intermetallics $Al_{12}Mg_{17}$ and $Al_8Mn_5$ contribute to both high strength and lower plasticity. Various methods were used to achieve grain refinement and to discover the superplastic region.

#### *2.1.1. Hot extrusion*

Superplasticity of AZ91 magnesium alloys prepared by hot extrusion (HE) was observed by several authors. Solberg et al. [8] found an excellent superplastic behaviour of rapidly solidified (RS) and extruded alloy. Tensile experiments of a fine-grained AZ91 alloy processed by powder metallurgy (PM) and ingot metallurgy (IM) techniques [9] were carried out at a constant strain rate. The superplastic behaviour was estimated in PM samples at higher strain rates compared with samples prepared with IM route [10]. Results of experiments performed on samples prepared by HE are summarised in **Table 1**.

| Preparation route | Grain size (μm) | $T$ (°C) | SR ($s^{-1}$) | $m$ | $\varepsilon_f$ (%) | Remark | References |
|---|---|---|---|---|---|---|---|
| RS + HE | 1.2 | 300 | $2.5 \times 10^{-3}$ | 0.60 | 1480 | | [8] |
| PM + HE | 1.4 | 300 | $1 \times 10^{-2}$ | 0.50 | 280* | $T/T_M = 0.62$ | [9] |
| PM + HE | 4.1 | 250 | $3 \times 10^{-3}$ | 0.50 | 430* | $T/T_M = 0.57$ | [9] |
| IM + HE | 5 | 300 | $3 \times 10^{-3}$ | <0.3 | 425* | | [10] |

*Tensile test at constant strain rate.

**Table 1.** Superplastic characteristics and conditions estimated for AZ91 alloy prepared by hot extrusion.

#### *2.1.2. Thermomechanical treatment*

An alternative route to prepare superplastic alloy is the thermomechanical treatment (TMT) consisted from the heat treatment in two stages (homogenisation at 470°C for 10 h and ageing at temperatures from 200 to 380°C). Aged samples were hot extruded at 350°C. [11–13]. The grain size of samples and superplastic behaviour depends on the ageing temperature. This method is relatively simple and big bulks of the material may be prepared. It is an advantage against other methods, for example, high-pressure torsion technique, which is able to manufacture only small samples. Results of the superplastic behaviour of AZ91 alloys prepared by the TMT are summarised in **Table 2**.

| Preparation route | Grain size (μm) | *T* (°C) | SR ($s^{-1}$) | *m* | $\varepsilon_f$ (%) | Remark | References |
|---|---|---|---|---|---|---|---|
| TMT | 17 | 420 | $1 \times 10^{-4}$ | 0.44 | 413* | Aged 200°C | [11] |
| TMT | 17 | 380 | $3 \times 10^{-4}$ | | 285 | Aged 200 °C | [12] |
| TMT | 10.5 | 420 | $1 \times 10^{-4}$ | 0.54 | 484* | Aged 300°C | [11] |
| TMT | 10.5 | 380 | $3 \times 10^{-4}$ | | 280 | Aged 300°C | [12] |
| TMT | 10.5 | 340 | $3 \times 10^{-4}$ | | 291 | Aged 300°C | [12] |
| TMT | 11 | 420 | $1 \times 10^{-4}$ | 0.54 | 584* | Aged 380°C | [11] |
| TMT | 11 | 380 | $3 \times 10^{-4}$ | | 251 | Aged 380°C | [12] |
| TMT | 11 | 340 | $3 \times 10^{-4}$ | | 260 | Aged 380°C | [12] |
| TMT | 6.4 | 420 | $1 \times 10^{-4}$ | 0.45 | 250* | Aged 350°C | [13] |
| TMT | 6.4 | 420 | $3 \times 10^{-4}$ | 0.40 | 260* | Aged 350°C | [13] |
| TMT | 11.2 | 400 | $3 \times 10^{-4}$ | 0.35 | 250 | Aged 300°C | [14] |

*Deformed at a constant strain rate.

**Table 2.** Superplastic characteristics and conditions estimated for AZ91 alloy prepared by thermomechanical treatment.

### *2.1.3. Equal channel angular pressing*

Excellent superplasticity of samples prepared by equal channel angular pressing (ECAP) was observed by Chuvil'deev et al. [15] at a relatively high temperature of 300 °C. Mussi et al. [16] found the superplastic behaviour after ECAP at a constant temperature (eight passes) of 265°C (CT) and at decreasing temperature in each following pass from 265 down to 150°C in the last one. A substantial grain refinement was observed for both ECA extrusion techniques. ECAP technique with decreasing temperature caused a decrease in the grain size up to sub-microcrystalline region. Matsubara et al. [17] used (first time) a new method for preparation of fine-grained materials involving sequential application of hot extrusion and ECAP. This method is in the literature designated as EX-ECAP. Mabuchi et al. [18] determined the super-plastic behaviour of an AZ91 alloy after hot extrusion and ECAP. Very small grain size (about 1 μm) was very probably the reason for the occurrence of superplastic flow at a relatively low temperature of $0.48T_M$. Superplasticity of fine-grained AZ91 alloy prepared by ECAP technique and annealing was studied by Mabuchi and co-workers [19]. Subsequent annealing for 12 h at 225°C improved the superplastic behaviour of the alloy although the grain size increased 4.4 times. The authors explained this finding to the transformation of non-equilibrium grain boundaries in the as ECAPed material to equilibrium grain boundaries which were detected in the annealed alloy by means of the transmission electron microscopy. The superplastic behaviour of AZ91 alloys prepared by the hot extrusion and ECAP or by a combination of both techniques is summarised in **Table 3**.

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| Preparation route | Grain size (μm) | *T* (°C) | SR ($s^{-1}$) | *m* | $\varepsilon_f$ (%) | Remark | References |
|---|---|---|---|---|---|---|---|
| ECAP-6p. | 0.8 | 300 | $3 \times 10^{-3}$ | 0.3 | 570 | Q = 78 kJ/mol | [15] |
| ECAP-6p. | 0.8 | 250 | $3 \times 10^{-2}$ | 0.3 | 375 | Route $B_C$ | [15] |
| ECAP-8p. | 1.0 | 300 | $2 \times 10^{-4}$ | | 340 | CT | [16] |
| ECAP-8p. | 1.0 | 250 | $1 \times 10^{-3}$ | | 190 | CT | [16] |
| ECAP-8p. | 0.3 | 250 | $1 \times 10^{-3}$ | | 490 | DT | [16] |
| HE-ECAP | 1.0 | 200 | $6 \times 10^{-5}$ | 0.30 | 661 | $T/T_M = 0.51$ | [18] |
| HE-ECAP | 1.0 | 175 | $6 \times 10^{-5}$ | 0.30 | 326 | $T/T_M = 0.48$ | [18] |
| ECAP | 0.7 | 200 | $6 \times 10^{-5}$ | 0.5 | 650 | Q = 89 kJ/mol | [19] |
| ECAP | 0.7 | 150 | $6 \times 10^{-5}$ | 0.5 | 620 | Non-eq. GB | [19] |
| ECAP + ann.* | 3.1 | 200 | $7 \times 10^{-5}$ | 0.5 | 990 | Q = 89 kJ/mol | [19] |
| ECAP + ann.* | 3.1 | 250 | $8 \times 10^{-5}$ | 0.5 | 980 | Eq. GB | [19] |

*ECAP with subsequent annealing.
Non-eq. GB: non-equilibrium grain boundaries; Eq. GB: equilibrium grain boundaries.

**Table 3.** Superplastic characteristics and conditions estimated for AZ91 alloy prepared by the ECAP technique.

### *2.1.4. Hot rolling*

Wei et al. [20] prepared AZ91 sheet by repeated rolling (RR) at 400°C. The rolls were annealed between rolling passes at 130°C. An initial grain size of 78 μm decreased after rolling down to 11 μm. Resulting grains were equiaxed and no twins were observed in the microstructure. Rolling and heating were applied 11 times. Samples for tensile deformation at elevated temperature have the tensile axis parallel to the rolling direction. Interesting results were obtained by Mohri et al. [21] on hot-rolled (HR) sheets. High-ratio differential speed rolling (HRDSR) was used to refine the microstructure of the cast alloy [22]. Very fine particles of the γ-phase hinder the grain growth during the high-temperature straining. An increase in temperature from 300 to 350°C reduced substantially the plasticity. The superplasticity characteristics of samples prepared by hot rolling are given in **Table 4**.

| Preparation route | Grain size (μm) | *T* (°C) | SR ($s^{-1}$) | *m* | $\varepsilon_f$ (%) | Remark | References |
|---|---|---|---|---|---|---|---|
| RR (400°C) | 11 | 350 | $1.5 \times 10^{-3}$ | 0.64 | 455 | *Q* = 92.7 kJ/mol | [20] |
| HR (400°C) | 39.5 | 300 | $1.5 \times 10^{-3}$ | | 601 | sub-grains~1μ | [21] |
| HRDSR | 1–2 | 300 | $5.0 \times 10^{-4}$ | | 550 | | [22] |
| HRDSR | 1–2 | 300 | $1.0 \times 10^{-3}$ | 0.5 | 830 | | [22] |
| HRDSR | 1–2 | 300 | $1.0 \times 10^{-2}$ | | 450 | | [22] |
| HRDSR | 1–2 | 350 | $1.0 \times 10^{-3}$ | | 250 | | [22] |

**Table 4.** Superplastic characteristics and conditions estimated for AZ91 alloy prepared by the hot rolling.

### 2.1.5. Friction stirring

Friction stir processing (FSP) is usually used for the friction stir welding purposes. Mishra and Ma [23] showed that this method may be applied as a novel grain refinement technique. In this case, the stirring action of the tool on the bulk material is used for a forging/extrusion treatment at very high temperatures. Dynamic recrystallization and grain rotation leads to a very fine microstructure. The superplastic behaviour of AZ91 magnesium alloy and on FS-processed AZ91 produced by high-pressure die casting was observed by Cavaliere and Marco [24]. Raja and coworkers [25] estimated that the microstructure depends sensitively on the FS conditions. The highest elongation to fracture was found for samples processed on one side up to a depth of 5 mm. An excellent superplasticity behaviour was observed by Zhang et al. [26]. They estimated very fine $\gamma$-$Mg_{17}Al_{12}$ and Al-Mn intermetallics which effectively hinder the grain growth at elevated temperatures. The variation of this method consists in some additional cooling during FSP. Submerged friction stir processing (SFSP) is realised underwater and it has a great potential in the preparation of ultrafine-grained materials [27]. Chai et al. [28] by applying the SFSP on AZ91 alloy obtained a substantial refinement of the grain structure and a considerably enhanced superplastic behaviour. Elongations to fracture of samples prepared with the conventional FSP and SFSP are compared in **Table 5**. The grain size of samples prepared with the SFSP decreased to 1.2 μm compared with FSP performed on air. An excellent superplastic behaviour of SFSP samples especially at higher strain rates was ascribed to finer structure and larger fraction of grain boundary [28]. A combination of the high-pressure die casting (HPDC) with the FSP resulted into ultrafine material with the grain size lower than 1 μm and excellent superplastic properties at 330°C and at relatively high strain rates [29]. Superplastic behaviour of AZ91 alloys prepared with routes exploited FSP is summarised in **Table 5**.

| Preparation route | Grain size (μm) | $T$ (°C) | SR ($s^{-1}$) | $m$ | $\varepsilon_f$ (%) | Activation energy (kJ/mol) | References |
|---|---|---|---|---|---|---|---|
| FSP | | 300 | $5 \times 10^{-4}$ | 0.68 | 1050 | | [24] |
| FSP | | 350 | $5 \times 10^{-4}$ | 0.6 | 680 | | [25] |
| FSP | 3 | 300 | $3 \times 10^{-3}$ | | 240 | 175.05 | [26] |
| FSP | 3 | 300 | $4 \times 10^{-4}$ | | 517 | 175.05 | [26] |
| FSP | 3 | 300 | $1 \times 10^{-4}$ | | 1604 | 175.05 | [26] |
| FSP | 7.8 | 350 | $4 \times 10^{-4}$ | 0.36 | 554 | 125.91 (148.75)* | [28] |
| FSP | 7.8 | 350 | $3 \times 10^{-3}$ | 0.36 | 402 | 125.91 (148.75)* | [28] |
| FSP | 7.8 | 350 | $2 \times 10^{-2}$ | 0.25 | 158 | | [28] |
| SFSP | 1.2 | 350 | $4 \times 10^{-4}$ | 0.43 | 990 | 88.30 (168.12)* | [28] |
| SFSP | 1.2 | 350 | $3 \times 10^{-3}$ | 0.43 | 1202 | 88.30 (168.12)* | [28] |
| SFSP | 1.2 | 350 | $2 \times 10^{-2}$ | 0.30 | 990 | | [28] |
| HPDC + FS | 0.5 | 330 | $1 \times 10^{-2}$ | | 1251 | | [29] |
| HPDC + FS | 0.5 | 330 | $3 \times 10^{-2}$ | | 827 | | [29] |

*Activation energy was estimated for the temperature ranges of 200–350°C (lower value) and 350–375°C (higher value).

**Table 5.** Superplastic characteristics and conditions estimated for AZ91 alloy prepared by the friction stir processing.

### 2.1.6. High-pressure torsion

Samples processed by high-pressure torsion (HPT), (10 turns), have a fine-equiaxed grain structure with a significant thermal stability [30]. The thermal stability was due to high volume fraction of nanosized $\gamma$-phase particles. The main mechanism is grain boundary sliding accommodated by dislocation creep process and diffusional process. The fine fibrous microstructure resisted cavitation and linked the disconnected grains [30]. The experimental results concerning to the superplastic behaviour of AZ31 alloy prepared by HPT are given in **Table 6**.

| Preparation route | $T$ (°C) | SR ($s^{-1}$) | $m$ | $\varepsilon_f$ (%) | References | Remark |
|---|---|---|---|---|---|---|
| HPT | 200 | $1 \times 10^{-2}$ | 0.42 | 590 | [30] | |
| HPT | 200 | $1 \times 10^{-3}$ | 0.46 | 660 | [30] | |
| HPT | 200 | $1 \times 10^{-4}$ | 0.46 | 670 | [30] | |
| HPT | 300 | $1 \times 10^{-1}$ | 0.41 | 410 | [30] | |
| HPT | 300 | $1 \times 10^{-2}$ | 0.42 | 860 | [30] | |
| HPT | 300 | $1 \times 10^{-3}$ | 0.43 | 1050 | [30] | 10 turns |
| HPT | 300 | $1 \times 10^{-4}$ | 0.52 | 1308 | [30] | 10 turns |

**Table 6.** Superplastic characteristics and conditions estimated for AZ91 alloy prepared by high-pressure torsion.

## 2.2. QE22 alloy

Mg-2Ag-2RE-0.7Zr alloys were developed for applications at elevated temperatures. Further improvement of mechanical properties is possible using some heat treatments producing thermally stable precipitates [31, 32].

### 2.2.1. Thermomechanical treatment

Thermomechanical processing for the production of fine-grained materials used by several authors [13, 14, 33] consists of subsequent steps: solution treatment at 470°C for 10 h quenched on air; and aged for 10 h followed by hot extrusion at a temperature of 350°C. Three ageing temperatures were applied with the aim to find optimum conditions for the superplastic deformation. Microstructure of the alloy consists from $\alpha$-grains decorated in grain boundaries by the chain of intermetallic particles containing Nd and Ag. Tiny Zr particles (100–200 nm) were not dissolved and affected by the thermal treatment. The activation energy was calculated to be Q = 114.8 kJ/mol [34]. **Table 7** summarises superplastic characteristics.

| Preparation route | Grain size(μm) | $T$ (°C) | SR ($s^{-1}$) | $m$ | $\varepsilon_f$ (%) | Remark | References |
|---|---|---|---|---|---|---|---|
| TMT | 6.1 | 420 | $2 \times 10^{-4}$ | 0.65 | 490 | Ageing 380°C | [34] |
| TMT | 1.0 | 450 | $3.3 \times 10^{-4}$ | 0.71 | 880* | Ageing 350°C | [33] |
| TMT | 1.0 | 480 | $3.3 \times 10^{-4}$ | 0.66 | 910* | Ageing 300°C | [33] |
| TMT | 0.7 | 420 | $1 \times 10^{-4}$ | 0.62 | 720 | Ageing 300°C | [13] |

| Preparation route | Grain size(μm) | $T$ (°C) | SR ($s^{-1}$) | $m$ | $\varepsilon_f$ (%) | Remark | References |
|---|---|---|---|---|---|---|---|
| TMT | 0.7 | 420 | $3 \times 10^{-4}$ | 0.75 | 780 | Ageing 300°C | [13] |
| TMT | 0.7 | 420 | $5 \times 10^{-4}$ | 0.50 | 450 | Ageing 300°C | [13] |
| TMT | 0.7 | 420 | $1 \times 10^{-3}$ | 0.42 | 360 | Ageing 300°C | [13] |
| TMT | 0.7 | 420 | $5 \times 10^{-3}$ | 0.38 | 300 | Ageing 300°C | [13] |
| TMT | 0.7 | 420 | 0.01 | 0.32 | 240 | Ageing 300°C | [13] |
| TMT | 1.9 | 400 | $3 \times 10^{-4}$ | 0.75 | 750* | Ageing 380°C | [14] |

*Straining at a constant crosshead speed.
Tensile tests were performed at a constant strain rate.

**Table 7.** Superplastic characteristics and conditions estimated for QE22 alloy prepared by TMT.

### *2.2.2. Friction stir process*

Samples for tensile tests were friction stir processed in a temperature-controlled atmosphere [35]. In order to increase the efficiency of the FS, a device external cooling set-up was used. The FSP was performed in two passes with different tool rotation rates. Refinement of the microstructure was really significant from 38 μm down to 630 nm (**Table 8**). Conditions for superplasticity and elongation to fracture of samples are reported in **Table 8**.

| Preparation route | Grain size (μm) | $T$ (°C) | SR ($s^{-1}$) | $m$ | $\varepsilon_f$ (%) | Remark | References |
|---|---|---|---|---|---|---|---|
| FSP | 0.63 | 450 | $1 \times 10^{2}$ | | 1630 | Q = 170 kJ/mol | [35] |
| FSP | 0.63 | 425 | $1 \times 10^{2}$ | | ~1000 | | [35] |
| FSP | 0.63 | 350 | $3 \times 10^{3}$ | 0.5 | 850 | | [35] |
| FSP | 0.63 | 350 | $1 \times 10^{2}$ | 0.5 | 450 | | [35] |
| FSP | 0.63 | 300 | $5 \times 10^{-4}$ | | 420 | Q = 142 kJ/mol | [35] |

**Table 8.** Superplastic characteristics and conditions estimated for QE22 alloy prepared by FSP.

## 2.3. AE42 alloy

In AE42 alloy, fine $Mg_9RE$ precipitates along the grain boundaries are formed. Aluminium improves castability and room temperature mechanical properties [36].

The AE42 magnesium alloy (Mg-4Al-2RE) casting was subjected to the subsequent two-stage thermal treatment: homogenisation (10 h at 470°C) and ageing at 300 °C and 350 °C, followed hot extrusion at 350 °C. Thermomechanical treatment refined the microstructure under 10 μm [13] and increased plasticity of samples [13, 14] as it is shown in **Table 9**.

| Preparation route | Grain size (μm) | $T$ (°C) | SR ($s^{-1}$) | $m$ | $\varepsilon_f$ (%) | Remark | References |
|---|---|---|---|---|---|---|---|
| TMT | 6.2 | 420 | $1 \times 10^{-4}$ | 0.43 | 235 | Ageing 300°C | [13] |
| TMT | 6.2 | 420 | $3 \times 10^{-4}$ | 0.37 | 220 | Ageing 300°C | [13] |
| TMT | 6.2 | 420 | $5 \times 10^{-4}$ | 0.36 | 180 | Ageing 300°C | [13] |
| TMT | 6.2 | 420 | $1 \times 10^{-3}$ | 0.30 | 150 | Ageing 300°C | [13] |
| TMT | 13.5 | 400 | $1 \times 10^{-4}$ | 0.4 | 220 | Ageing 350°C | [14] |

Tensile tests were performed at a constant strain rate.

**Table 9.** Superplastic characteristics and conditions estimated for AE42 alloy prepared by TMT.

### 2.4. EZ33 alloy

The Mg-RE-Zn alloys have additional rare earth elements to improve the creep resistance and to refine the grain size. Further increase of strength occurs if zinc is added [37]. The continuous networks of intergranular phases in Mg-Zn-Nd-Zr alloys significantly deteriorate the ultimate tensile strength and elongation [38] Superplastic samples of the EZ33 (Mg-2.5Zn-3RE-Zr) magnesium alloy were prepared with the same thermomechanical treatment as AZ91, QE22 and AE42 alloys. Precipitation ageing was realised at 470°C for 10 h with a subsequent cooling on the air. As it follows from **Table 10**, the tensile elongations to fraction for all samples exceeded 200%, which represents a substantial improvement over the poor room-temperature ductility; typical for magnesium alloys [13, 34].

Similar alloy Mg-3Gd-1Zn (GZ31) was processed by EX-ECAP and deformed by shear punch testing [39]. Samples with very fine microstructure (grain size 1.7 μm) were deformed in shear punch tests in the temperature interval from 300 to 500°C. A strain rate parameter of 0.51 and activation energy of 73 kJ/mol indicate superplastic behaviour — see **Table 10**.

| Preparation route | Grain size (μm) | $T$ (°C) | SR ($s^{-1}$) | $m$ | $\varepsilon_f$ (%) | Remark | References |
|---|---|---|---|---|---|---|---|
| TMT | 1.2 | 420 | $1 \times 10^{-4}$ | 0.65 | 700 | Ageing 350°C | [13] |
| TMT | 1.2 | 420 | $3 \times 10^{-4}$ | 0.73 | 710 | Ageing 350°C | [13] |
| TMT | 1.2 | 420 | $5 \times 10^{-4}$ | 0.50 | 420 | Ageing 350°C | [13] |
| TMT | 1.2 | 420 | $1 \times 10^{-3}$ | 0.48 | 340 | Ageing 350°C | [13] |
| TMT | 1.2 | 420 | $5 \times 10^{-3}$ | 0.35 | 280 | Ageing 350°C | [13] |
| TMT | 1.2 | 420 | 0.01 | 0.30 | 230 | Ageing 350°C | [13] |
| TMT | 2.2 | 420 | $4 \times 10^{-4}$ | 0.36 | 473 | Ageing 300°C | [34] |
| TMT | 2.2 | 420 | $2 \times 10^{-4}$ | 0.38 | 490 | Ageing 300°C | [34] |
| EX-ECAP | 1.7 | 400 | $1 \times 10^{-3}$ | 0.51 | | GZ31 73 kJ/mol | [39] |

Tensile tests were performed at a constant strain rate

**Table 10.** Superplastic characteristics and conditions estimated for EZ33 alloy prepared by TMT.

## 3. Discussion

### 3.1. Grain refinement

Results obtained for various materials prove that the grain size has the key role for the occurrence of superplastic flow. It follows from relationship (3), (the inverse dependence on grain size in a power of 2). The second necessary condition which should be fulfilled to achieve superplastic deformation is the thermal stability of grains.

If superplastic materials are prepared by some thermomechanical treatment, the processing route should introduce (produce) a number of recrystallization nuclei in the alloys through homogenisation, ageing with a successive hot extrusion process. Comparing resulting grain sizes of samples prepared by the TMT, it is to see that the grains in the alloys containing Zr are much finer. The finer grains in the magnesium alloys QE22 and EZ33 alloys may be attributed to small Zr particles and RE-rich precipitates formed in both materials during the thermal treatment.

It should be mentioned that the grain refinement in hexagonal alloys is different from that in fcc metals [40]. The grain refinement in hexagonal alloys subjected to ECAP starts by the nucleation of tiny grains in pre-existing grain boundaries [41, 42]. The stress concentration in the grain boundaries may activate non-basal slip. A co-operation of basal and non-basal slip is the prerequisite for formation of three-dimensional net of equiaxed grains. Reducing number of passes and increasing the processing temperature may shift the superplasticity region to higher temperatures [43].

Fragmentation of second-phase particles was observed in QE22 alloy after multi-pass FSP [44]. Very high plastic deformation is introduced into the stirred zone during FSP. Disintegrated particles serve as nuclei in the dynamic recrystallization. At high strain rate superplasticity, a rapid growth of cavities is due to a significant stored plastic deformation and lattice diffusion. Similar mechanisms of grain refinement may be considered in the high-pressure torsion producing: disintegration of intermetallics followed by dynamic recrystallization (DXR) observed by Száraz et al. [45] in Mg-based nanocomposites subjected to high-pressure torsion.

### 3.2. Mechanisms of superplastic flow

Superplastic flow of metallic materials may be described using constitutive equation [46]:

$$\dot{\varepsilon} = A\left(\frac{Gb}{kT}\right)\left(\frac{\sigma - \sigma_0}{G}\right)^n\left(\frac{b}{d}\right)^p D, \tag{3}$$

where A is a dimensionless material constant, $G$ is the shear modulus, $b$ is the Burgers vector, $\sigma$ is the applied stress, $\sigma_0$ is the threshold stress, $d$ is the grain size, n is the stress sensitivity exponent (n = 1/m), p is the grain size exponent and $kT$ has its usual meaning. $D$ is the diffusion coefficient (=$D_0 \exp(-Q/RT)$, where $D_0$ is the frequency factor, $Q$ is the activation energy for the diffusion process and $R$ is the gas constant).

Plastic deformation of coarse-grained materials at temperatures $T < 0.4T_M$ is realised by dislocation motion and storage of dislocations on obstacles. These obstacles may be of two types: dislocation and non-dislocation type (grain boundaries, twins´ boundaries and incoherent precipitates). Storage of dislocations contributes to strain hardening of a material. On the other hand, recovery processes like cross-slip and dislocation climbing play an important role especially at elevated temperatures and contribute to softening. Steady-state character of the stress-strain curves is a consequence of a dynamic equilibrium between hardening and softening processes. In fine-grained materials, two additional deformation mechanisms—grain boundary sliding accommodated by slip and diffusion flow—should be considered [46, 47].

Steady-state plastic flow of coarse-grained metals at higher temperatures, above $0.4T_M$, is usually described by dislocation motion and storage on obstacles. Each mechanism—dislocation slip, grain boundary sliding and diffusional flow—has its characteristic values of stress sensitivity, n, grain size exponent, p and the activation energy, Q. Plastic materials have typically high values of the stress sensitivity parameter $n = 1/m$. For $n \geq 5$, we may consider that the main mechanism of plastic deformation is the dislocation motion. The activation energy is done by the strength of local obstacles in the thermally activated dislocation motion. If this dislocation motion is non-conservative—climb of dislocations (for example, in the case of the high-temperature creep)—the characteristic activation energy is close to the activation energy for the lattice diffusion or pipe diffusion. The grain size exponent has very low value approaching zero [47]. As it was shown above, high values of the strain rate sensitivity parameter (in the case of ideal superplasticity $m = 1$) are characteristic for the superplastic flow, i.e., the stress sensitivity parameter must be from the interval $1 < n < 3$. The apparent activation energy may help to identify the deformation mechanism(s) occurring in the superplastic flow [47].

$$Q = \frac{1}{m} R \frac{\Delta ln\,(\sigma/G)}{\Delta(1/T)}, \tag{4}$$

where $\sigma$ is the applied stress, $R$ is the gas constant and $T$ is the absolute temperature. The activation energy estimated for lattice and grain boundary diffusion in magnesium is 135 and 92 kJ/mol, respectively [48]. The activation energies reported in **Tables 3–5**, **8** and **10** have values which are between both values estimated for the lattice and grain boundary diffusion. It indicates that very probably both mechanisms are present. Activation energies introduced in **Table 8** for QE22 alloy are higher than the values measured for lattice diffusion. Arzt and co-workers [49] have shown that the overcoming of intergranular precipitates by grain boundary dislocations leads to a higher activation energy [49].

It is interesting to note that mechanical twinning as an additional deformation mechanism was observed in some cases.

**Figure 1** shows scanning electron micrographs (SEM micrographs) of AZ91 and QE22 samples deformed in the superplastic region. Deformation twins are visible in both micrographs 1a and 1b. They provide evidence that in Mg alloys, twinning may serve as an additional accommodation mechanism for grain boundary sliding at straining temperatures which do not allow for

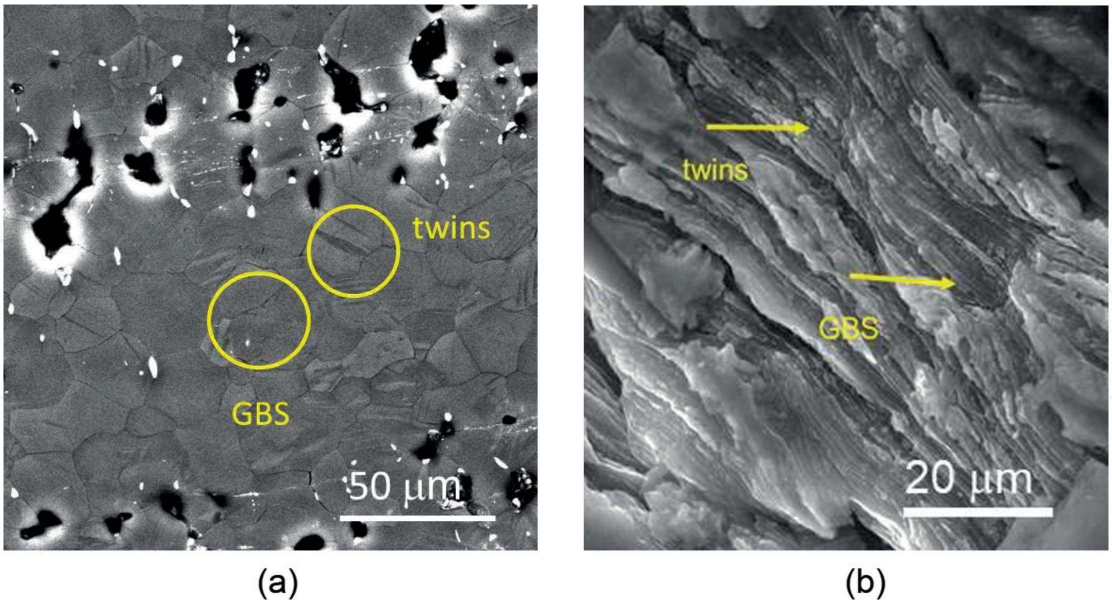


**Figure 1.** (a) Twins and grain boundary sliding in the AZ91sample deformed at 340 °C and (b) surface wrinkles estimated after deformation of QE22 alloy at 450°C.

sufficiently fast diffusion-related accommodation. On the other hand, transmission electron microscopy revealed in QE22 and EZ33 alloys clear grains, without dislocation in deformed samples at 420°C as it has been reported in our previous paper [50].

### 3.3. Cavitation during superplastic flow

Cavitation in the AZ91 alloy occurred during superplastic deformation at 420°C. Similarly, chains of cavities are obvious in the micrograph of the AZ91 alloy deformed at 340°C.

The formation and growth of cavities relaxes the stress concentration in triple junctions caused by grain boundary sliding (GBS). Stress concentrations are likely developed at sites which impede grain boundary sliding such as grain boundary particles, ledges and triple points. Cavities created by vacancy clustering may nucleate if the stress concentration is not relieved sufficiently rapidly. The AZ91 alloy with intermetallic inclusions can be considered as a natural composite. The stress concentrations are formed at the particles on sliding grain boundaries. Local tensile stress caused by sliding at interfaces may be expressed in the following form [19]:

$$\sigma_{slid} = \frac{0.92kTd_p\dot{\varepsilon}dV_f}{\Omega D_L\left(1+5\frac{\delta D_{GB}}{d_pD_L}\right)} \tag{5}$$

where $d_\mathrm{p}$ is the particle diameter, $\dot{\varepsilon}$ is the strain rate, $D_\mathrm{L}$ is the lattice diffusion coefficient and $D_\mathrm{GB}$ is the grain boundary diffusion coefficient, $\delta$ is the grain boundary width, $\Omega$ is the atomic volume and $V_\mathrm{f}$ is the volume fraction of particles. Other symbols have the same meaning as in Eq. (3). Mechanical twinning observed in AZ91 alloy deformed at 340°C and in QE22 alloy deformed at 430°C may serve as an additional accommodation mechanism for grain boundary sliding at straining temperatures which do not allow sufficiently fast diffusion-related accommodation. The insufficiently accommodated GBS process is documented in **Figures 1a** and **2**, where cavities formed during deformation of AZ91 alloy are visible. Chains of cracks are oriented in the extrusion direction; they show that this process is also influenced by the basal texture of materials formed during the hot extrusion. The cracks were developed from small voids created as a result of the grain boundary sliding. The influence of texture is also visible in **Figure 1b**, where observed wrinkles are traces of basal planes which were oriented, in the textured sample, parallel to the extrusion direction and sample axis.

The presented results allow us to conclude that grain boundary sliding is very probably the main deformation mechanism during the superplastic deformation in the reported alloys. The different thermomechanical treatment influenced the samples microstructure and also different contributions to the superplastic flow: grain boundary sliding, lattice/grain boundary diffusion, dislocation deformation (slip of basal and non-basal dislocations, creep and climb of dislocations) and mechanical twinning.

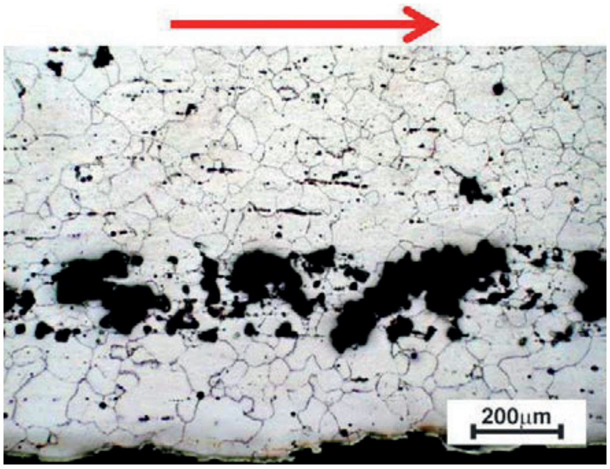


**Figure 2.** Cavities in AZ91 sample deformed at 420 °C and constant strain rate of $1 \times 10^{-4}$ $s^{-1}$. Arrow indicates tension direction.

## 4. Concluding remarks

The superplastic behaviour of four magnesium alloys has been reported. Occurrence of superplasticity requires a special microstructure containing small equiaxed grains and stable grain boundaries. Various methods were used for preparation of materials corresponding to these requirements. Thermomechanical treatment is the method which is relatively simple and gives acceptable results. Bulks of materials may be processed and used for possible applications in the industry. On the other hand, methods, exploited severe plastic deformation or friction stir processing, are still an academic issue. The main problem of superplasticity is the transfer of results achieved in the laboratory into the industry. Applications of magnesium alloys in various industrial branches continuously increase. Owing to small plasticity of these alloys, the finding of the conditions for superplastic flow plays a very important role.

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## Conflict of interest

Authors declare no conflict of interest.

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