Review
Processing and mechanical properties of fine-grained magnesium alloys

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Magnesium alloys are promising light structural materials. The present paper focuses on fine-grained magnesium-based materials. Grain refinement is attained by hot working without additional treatments. Also, a very small grain size of less than 1 μm is obtained by equal channel angular extrusion. A good combination of high strength and high ductility at room temperature is attained by grain refinement. Furthermore, fine-grained magnesium-based materials exhibit superplastic behavior at high stain rates ($\geq 10^{-1}$ s^{-1}) or low temperatures ($\leq$473 K). These point out the importance of grain refinement to process magnesium-based materials with excellent mechanical properties.

1. Introduction

Magnesium alloys have high potential as structural materials because of their low density. Furthermore, Mg resources are abundant, and also Mg products can be recycled more easily, compared to polymers. To date, most of Mg products have been fabricated by casing [1–3], in particular, diecasting and thixo-casting. On the other hand, there are few applications using forging, rolling, extrusion and so on in fabrication of Mg products. However, it is important to increase Mg applications using plastic forming of forging, rolling and extrusion in order to fabricate a variety of Mg products and increase the consumption of Mg products. Control of precipitation and grain refinement can be attained by thermo-mechanical treatment consisting of hot working and heat treatment [4–9], resulting in significant improvement of mechanical properties.

In general, the yield strength as a function of grain size can be represented as Hall-Petch equation;

$$\sigma = \sigma_0 + K d^{-1/2} \quad (1)$$

where σ is the yield stress, σ_0 is the yield stress of a single crystal, K is a constant and d is the grain size. A value of K increases with increasing the Taylor factor [10]. The Taylor factor generally depends on the number of the slip systems. Because the slip systems are limited and the Taylor factor is larger for *h.c.p.* metals than for *f.c.c.* and *b.c.c.* metals, *h.c.p.* metals exhibit the strong influence of grain size on strength. Therefore, it is suggested that high strength can be attained in fine-grained Mg-based materials. The relationship between the yield stress and the grain size is shown in Fig. 1 for AZ91 Mg alloy [11] and 5083(H321) alloy, where the yield stress of a single crystal and a value of K for 5083(H321) alloy are taken to be 230 MPa [12] and 63 MPa $\mu m^{1/2}$ [13]. It can be seen that the yield stress of the Mg alloy is lower than that of the Al alloy in a large grain size range $\geq$2 μm, however, the yield stress of the Mg alloy is higher than that of the Al alloy in a small grain size range <2 μm.

Grain refinement leads to not only high strength at room temperature, but also superplasticity at high temperatures. In general, a large elongation of more than 300% is attained for superplastic metallic materials. Such a large elongation is enough for near-net-shape forming of a product with complicated shapes. To date, superplastic forming has been used mainly in aerospace industries in Al alloys, Ti alloy and so on [14–16]. However, there are few applications using superplastic

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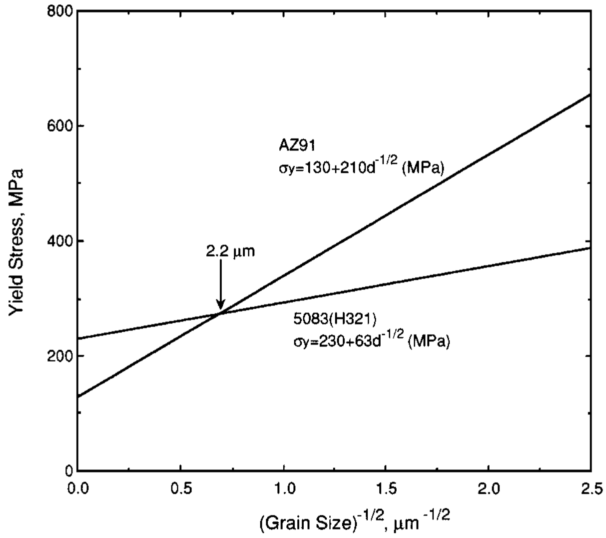

Figure 1 The relationship between the yield stress and the grain size for AZ91 Mg alloy and 5083(H321) alloy.

forming in Mg alloys. Because Mg alloys have poor workability because of the h.c.p. structure, superplastic forming is expected to be applied in the processing in practical Mg applications.

In general, large elongation is attained only in a very low strain rate range, typically $\sim 10^{-3}\,s^{-1}$, for conventional superplastic materials. One of the drawbacks in current superplastic forming technology is the low forming rate, resulting in poor productivity. Therefore, commercial applications using superplastic forming has been limited. Recently, however, it has been demonstrated that some aluminum-based materials exhibit superplastic behavior at high strain rates above $10^{-2}\,s^{-1}$ [17–22]. High strain rate superplasticity is very attractive for commercial applications. For example, the mechanically alloyed Al materials showed large elongation of about 1000% in a high strain rate range of 10–300 s^{-1} [19]. High strain rate superplasticity is attributed to a very small grain size [22].

In the present paper, grain refinement and mechanical properties, in particular, tensile properties at room temperature and superplasticity, of fine-grained Mg alloys are reviewed. The present paper points out that grain refinement significantly improves the mechanical properties of magnesium-based materials.

2. Grain refinement by hot extrusion

Amorphous or nanocrystalline Mg specimens have been processed by a rapid solidification method [23–29]. Recently, Mg alloys with a very small grain size of about 1 μm have been produced by a powder metallurgy method [30]. These processes give rise to a very small size, but high costs. It is noted that Mg alloys with a small grain size of less than 10 μm can be processed from cast ingots with a large grain size by hot working without additional treatments. Microstructure of an as-extruded AZ91 Mg alloy is shown in Fig. 2. It can be seen that grain refinement is attained by hot extrusion. It should be noted that a large bulk sample with a small grain size can be processed from ingots by hot extrusion without additional treatments for

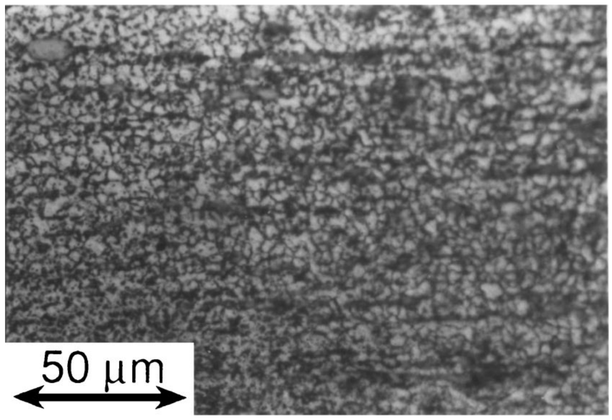

Figure 2 Microstructure of as-extruded AZ91, showing that grain refinement is attained by hot extrusion.

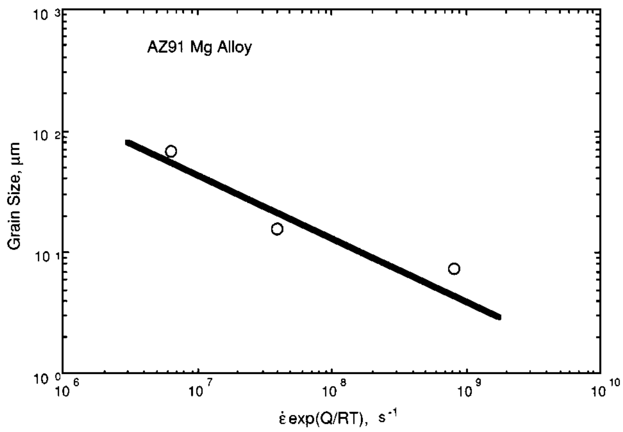

Figure 3 The relationship between the grain size and the *Z* parameter for AZ91.

magnesium-based materials. This is attractive for commercial applications.

The grain size of as-extruded specimens strongly depends on the extrusion temperatures. AZ91 alloy ingots were extruded at three temperatures of 573, 673 and 753 K to investigate effects of the extrusion temperature [31]. The grain size of the materials extruded at 573, 673 and 753 K were 7.6, 15.4 and 66.1 μm, respectively. This suggests that the grain size decreases with decreasing extrusion temperature. The grain size of the extruded materials can be expressed as a function of the Z parameter of the extrusion condition ($=\dot{\varepsilon}\exp(Q/RT)$, where $\dot{\varepsilon}$ is the extrusion strain rate, Q is the activation energy for lattice diffusion of magnesium (=135 kJ/mol [32]), R is the gas constant and T is the extrusion temperature). The relationship between the grain size and the Z parameter of the extrusion condition is shown in Fig. 3. It can be seen that the grain size decreases with decreasing Z parameter. It has been reported that high strength of more than 500 MPa is attained in the ZK60 alloy extruded at a relatively low temperature of 423 K [33]. The high strength for the extruded ZK60 is probably attributed to a very small grain size.

3. Mechanical properties

3.1. Mg-Al-Zn-Mn and Mg-Zn-Zr alloys

Mg-Al-(Zn)-Mn alloys (for example, AM60 and AZ91) and Mg-Zn-(Zr) alloys (for example, ZK60 and ZK61)

TABLE I Tensile properties at room temperature for Mg alloys

Materials		UTS (MPa)	0.2% Proof Stress (MPa)	Elongation (%)
As cast	AZ91 (F)	131	72	1–3
	AZ91 (T6)	235	108	3
As cast	ZK60 (F)	275	196	5
	ZK60 (T5)	314	265	4
Extruded	AZ91	341	244	13
Extruded	ZK60	371	288	18
P/M	AZ91	432	376	6
P/M	ZK61	400	383	7

are typical commercial Mg alloys. As mentioned above, grain refinement is attained by extruding ingots for these Mg alloys. Tensile properties at room temperature of the cast ingots and the extrusions in AZ91 and ZK60 are listed in Table I. The extruded materials showed much higher strength than the cast ingots. The high strength for the extruded materials is attributed to a small grain size. Furthermore, large elongations of more than 10% were attained for the extruded materials, showing that ductility is increased by hot extrusion. It should be noted that both high strength and high ductility are attained by grain refinement. Intergranular fracture occurs in a Mg alloy with a large grain size; however, intergranular fracture is limited in a Mg alloy with a small grain size [34], indicating that the fracture mechanism is changed by grain refinement. This is because the critical stress for crack propagation at grain boundaries increases with decreasing grain size [34].

Grain refinement gives rise to not only a good combination of high strength and high ductility at room temperatures, but also superplasticity at elevated temperatures. Recently, it has been reported [30] that an extruded AZ91 showed a maximum elongation of 425% at $3 \times 10^{-4}\,s^{-1}$ with 523 K, and an extruded ZK60 showed a maximum elongation of 730% at $4 \times 10^{-4}\,s^{-1}$ with 573 K. The superplastic properties strongly depend on the grain size. The variation in flow stress (top figure) and elongation to failure (bottom figure) at 573 K as a function of strain rate is shown in Fig. 4 for AZ91 with different grain sizes of 5.0, 9.1 and 16.4 μm. In general, the logarithmic stress-logarithmic strain rate relation is sigmoidal for superplastic metals. The strain rate sensitivity of stress is high ($>$0.3) and large elongation is attained in an intermediate strain rate region, which is the superplastic region. However, in both low and high strain rate regions, the strain rate sensitivity is low and large elongation is not attained. For the extruded Mg alloys, the logarithmic stress-logarithmic strain rate curves were sigmoidal, as has been observed for superplastic metals. The high strain rate sensitivity of about 0.5 was attained in an intermediate strain rate region. It should be noted that the superplastic strain rate region shifts to a higher strain rate range with decreasing grain size.

A powder metallurgy (P/M) method can give rise to a smaller grain size, compared to an ingot metallurgy (I/M) method. It has been reported [35] that AZ91 and ZK61 alloys with a very small grain size of about 1 μm were processed through a P/M route. Further-

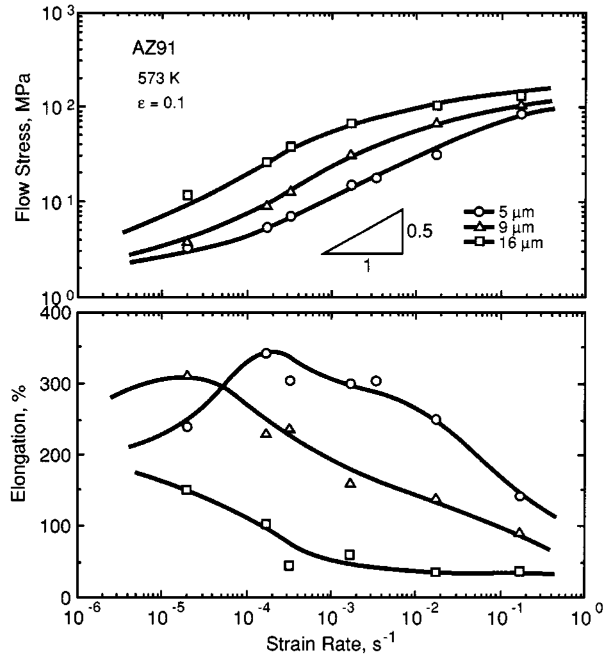

Figure 4 The variation in flow stress (top figure) and elongation to failure (bottom figure) at 573 K as a function of strain rate for AZ91 with different grain sizes of 5.0, 9.1 and 16.4 μm.

more, the grains were more stable at elevated temperatures and grain growth was limited for the P/M Mg alloys, compared to the I/M Mg alloys [36]. The tensile properties at room temperature for the P/M AZ91 and ZK61 alloys are listed in Table I. The P/M AZ91 and P/M ZK61 showed high strength of 432 and 400 MPa, respectively. The strengths of the P/M Mg alloys are higher than those of the extruded Mg alloys. This is attributed to a smaller grain size for the P/M alloys.

The P/M alloys exhibit high strain rate superplasticity [30]. The variation in elongation to failure at 573 K as a function of strain rate is shown in Fig. 5 for P/M ZK61 and I/M ZK60, where the grain size is 1.2 μm for the P/M alloy and 2.4 μm for the I/M alloy, respectively. The P/M alloy exhibited a maximum elongation of 432% at a high strain rate of $10^{-1}\,s^{-1}$. It can be seen that the P/M alloy exhibited superplastic behavior at higher strain rates than the I/M alloy. High strength and high strain rate superplasticity for the P/M Mg alloys are attributed to the very small grain size of about 1 μm [30]. It is of interest to note that the maximum elongation of 432% for the P/M alloy is smaller than that of 730% for the I/M alloy. This is probably because the oxide on the surfaces of powder causes cavitation during superplastic deformation.

3.2. Mg-Y-Re alloy

High strength, high ductility, and high strain rate superplasticity can be attained by grain refinement, as shown above. However high creep resistance can not be attained for fine-grained materials. In general, it is difficult to attain high strength at elevated temperatures for magnesium-based materials because of the fast diffusion rate. Recently it has been pointed out [37] that the Orowan mechanism is effective to attain high strength at elevated temperatures. Therefore a dispersion of fine

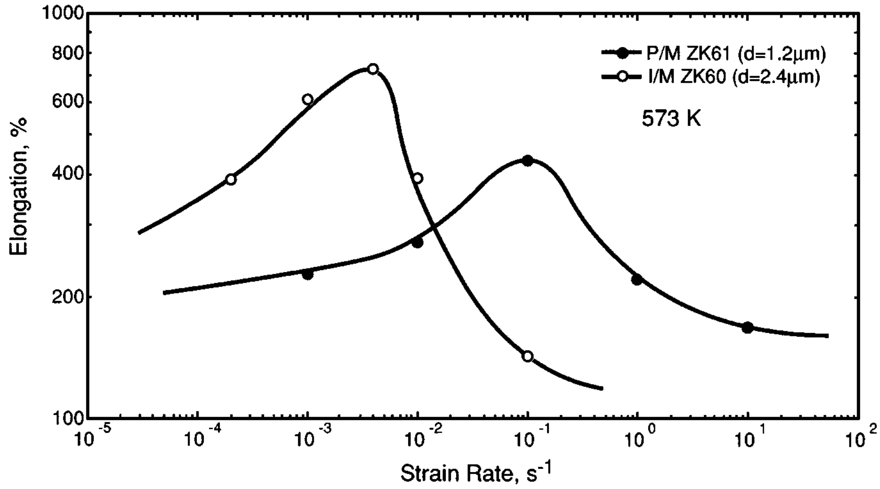

Figure 5 The variation in elongation to failure at 573 K as a function of strain rate for P/M ZK61 and I/M ZK60.

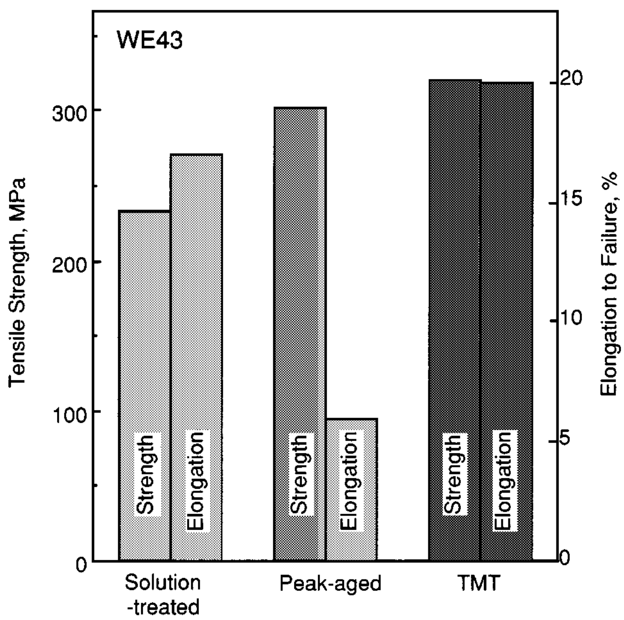

Figure 6 Tensile strength and elongation to failure at room temperature for WE43, showing that a good combination of high strength and high ductility is attained by thermo-mechanical treatment (TMT).

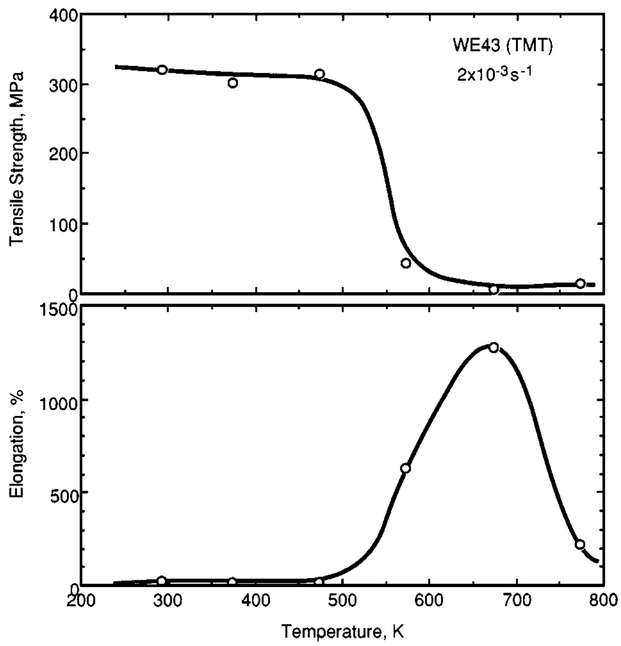

Figure 7 The variation in tensile strength (top figure) and elongation to failure (bottom figure) as a function of temperature for WE43 processed by thermo-mechanical treatment.

and stable particles is required for high creep resistance. It is known that Mg-Y-Re alloys exhibit high creep resistance [38, 39] because fine and stable particles consisting of Y and Re are dispersed.

Control of precipitation and grain refinement is attained by thermomechanical treatment (TMT) consisting of hot working and heat treatment. Therefore, multi-performances with high strength & high ductility & high creep resistance & high strain rate superplasticity can be obtained by thermomechanical treatment for Mg-Y-Re alloys. Recently Mohri *et al.* [34] conducted TMT consisting of hot extrusion and artificial aging to control microstructure of a cast Mg-4Y-3Re (WE43) alloy. In the TMT, hot extrusion was carried out at 673 K with an extrusion ratio of 100 : 1, and then artificial aging was conducted at 473 K for 7.2 ks. Tensile strength and elongation to failure at room temperature are shown in Fig. 6 for solution-treated, peak-aged and TMT WE43. It should be noted that a good combination of high strength (=320 MPa) and high ductility (=20% elongation) is attained for the TMT material. High ductility for the TMT material is because intergranular fracture is limited by grain refinement [34].

The variation in tensile strength (top figure) and elongation to failure (bottom figure) as a function of temperature is shown in Fig. 7 for WE43 processed by thermo-mechanical treatment. It should be noted that the TMT WE43 showed high strength of more than 300 MPa to an elevated temperature of 473 K. Also, it is of interest to note that elongation increases rapidly at 573 K and a very large elongation of 1274% is attained at 673 K. It has been reported that the TMT WE43 showed a large elongation of 358% at a high strain rate of $4 \times 10^{-1}\ s^{-1}$ with 673 K [34]. Therefore, it is demonstrated that the TMT WE43 exhibit multi-performance with high strength & high ductility & high creep resistance & high strain rate superplasticity. The multi-performance is attributed to fine precipitates and the small grain size (=about 1 μm [34]).

TABLE II Mechanical properties at room temperature for Mg-Li alloys[a]

Materials	UTS (MPa)	0.2% Proof Stress (MPa)	Elongation (%)	Hardness (Hv)
Mg-5.5Li	131.5	70.0	52.3	46.6
Mg-8.5Li	121.2	85.8	65.2	43.2
Mg-8.5Li-1Y	121.2	90.4	64.0	46.6

[a]The Mg-Li alloys are annealed at 623 K for 1.8 ks after worm rolling at 473 K.

3.3. Mg-Li alloy

Mg-Li alloys are ultra-light metals. Mg-Li alloys with Li wt% > 5.5 consist of two phases of a α phase (Mg solid solution) and a β phase (Li solid solution). It has been reported that two phase Mg-Li alloys exhibit superplasticity [40–43]. Three Mg-Li alloys; Mg-5.5 wt % Li, Mg-8.5 wt % Li and Mg-8.5 wt % Li-1 wt % Y, were investigated. The Mg-5.5Li is a α single phase, and the Mg-8.5Li and the Mg-8.5Li-1Y are a $\alpha + \beta$ phase (approximately, 40% α and 60% β). The Mg-Li alloys were annealed at 623 K for 1.8 ks after warm rolling at 473 K. The microstructures are shown in Fig. 8, where the rolling direction is horizontal. For the two phase alloys, α and β phases developed a banded structure parallel to the rolling direction. The band spacing is of the order to 20 μm for the Mg-8.5 Li and less than 10 μm for the Mg-8.5Li-1Y. It is noted that the addition of yttrium plays a vital role in grain refinement.

The mechanical properties at room temperature for the Mg-Li alloys are listed in Table II. It should be noted that the Mg-Li alloys exhibit large elongation >50% at room temperature.

The variation in elongation to failure at 623 K as a function of strain rate is shown in Fig. 9 for the Mg-Li alloys. Superplastic behavior was not observed for the Mg-5.5Li. However, the Mg-8.5Li showed a large elongation of 590% at $2 \times 10^{-4}\ s^{-1}$ and the Mg-8.5Li-1Y showed a large elongation of 390% at $4 \times 10^{-3}\ s^{-1}$. The Mg-8.5Li-1Y showed larger elongations in a high strain rate range of more than $10^{-3}\ s^{-1}$, compared to the Mg-8.5Li. This is because of the smaller grain size for the Mg-8.5Li-1Y.

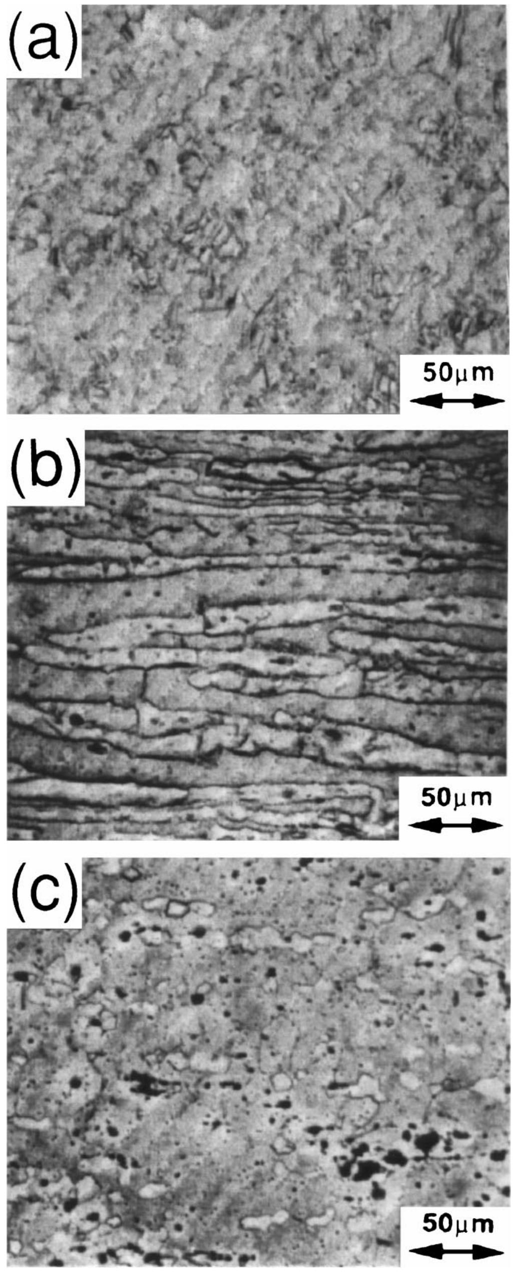

Figure 8 Microstructures of Mg-Li alloys, (a) Mg-5.5 wt % Li and (b) Mg-8.5 wt % Li, (c) Mg-8.5 wt % Li-1 wt % Y, where the specimens are annealed at 623 K for 1.8 ks after warm rolling at 473 K. The rolling direction is horizontal.

3.4. Mg matrix composites

Grain refinement largely contributes to high strength at room temperature for Mg matrix composites as well as Mg alloys [37]. However, it is difficult to attain high creep resistance for Mg matrix composites, compared to Al matrix composites, because the grain boundary diffusion rate of Mg is much higher than that of Al. Mabuchi *et al.* [37] have reported that Mg matrix composites reinforced with Mg_2Si particles exhibit a large decrease in strength at 473 K. However, it may be easy to attain superplasticity for Mg matrix composites, compared to Al matrix composites, because the stress concentrations caused by the presence of reinforcements are relaxed easily by diffusion. Recently, many instances of superplasticity in Mg matrix composites have been reported [44–52]. The superplastic properties of Mg matrix composites reinforced with

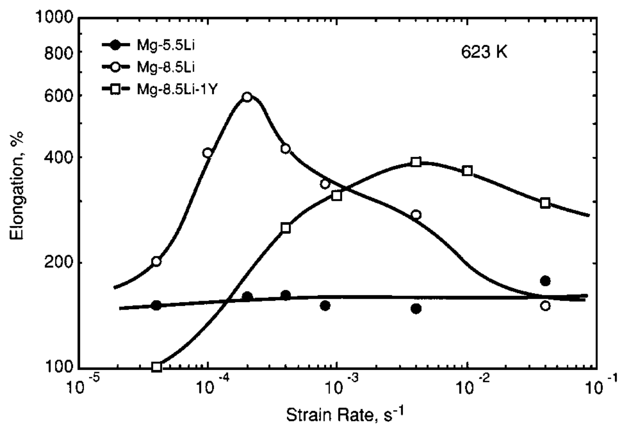

Figure 9 The variation in elongation to failure at 623 K as a function of strain rate for Mg-Li alloys.

TABLE III Superplastic properties of magnesium matrix composites reinforced with particles

Composites	Volume fraction of reinforcements (%)	Elongation (%)	Strain rate (s^{-1})	Temperature (K)	Strain rate sensitivity	References
Mg-9Li/B_4C	5	355	10^{-3}	473	0.5	[44]
Mg-6Zn(ZK60)/SiC	17	360	1.3	723	0.33	[45, 46]
Mg-5Zn/TiC	20	340	7×10^{-2}	743	0.43	[47]
Mg-4Al/Mg_2Si	28	370	10^{-1}	788	0.5	[48, 49]
Mg-4Zn/Mg_2Si	28	290	10^{-1}	713	0.5	[48]
Mg-4Zn/Mg_2Si	10	210	2×10^{-5}	723	0.5	[50]
Mg-5Al/AlN	15	300	10^{-3}	723	0.25	[51]
Mg-6Zn(ZK60)/SiC	17	450	10^{-1}	623	0.5	[52]

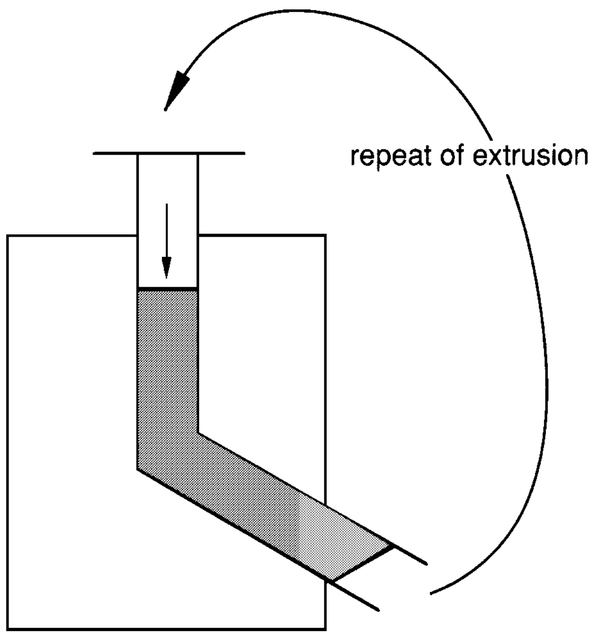

Figure 10 Schematic illustration of ECAE.

particles are summarized in Table III. Nieh *et al.* [45] showed that superplasticity is attained at a high strain rate of $1.3\,s^{-1}$ for the ZK60/SiC_p composite. For Al matrix composites, a liquid phase is required to attain high strain rate superplasticity in order to relax the stress concentrations caused at the reinforcements [53–57]. However high strain rate superplasticity is attained without the presence of a liquid phase for Mg matrix composites [48]. This is because the stress concentrations at the reinforcements are relaxed only by diffusion for Mg matrix composites.

4. Low temperature superplasticity

Equal channel angular extrusion (ECAE) is a new process to attain large shear strain [58–63]. A schematic illustration of ECAE is shown in Fig. 10. Through this technique large bulk samples with submicrometer grain size have been processed for Al alloys [59–63]. Recently, it has been reported that the fine-grained AZ91 with a very small grain size of less than 1 μm is processed by ECAE [64]. The microstructure of the AZ91 processed by ECAE is shown in Fig. 11. It should be

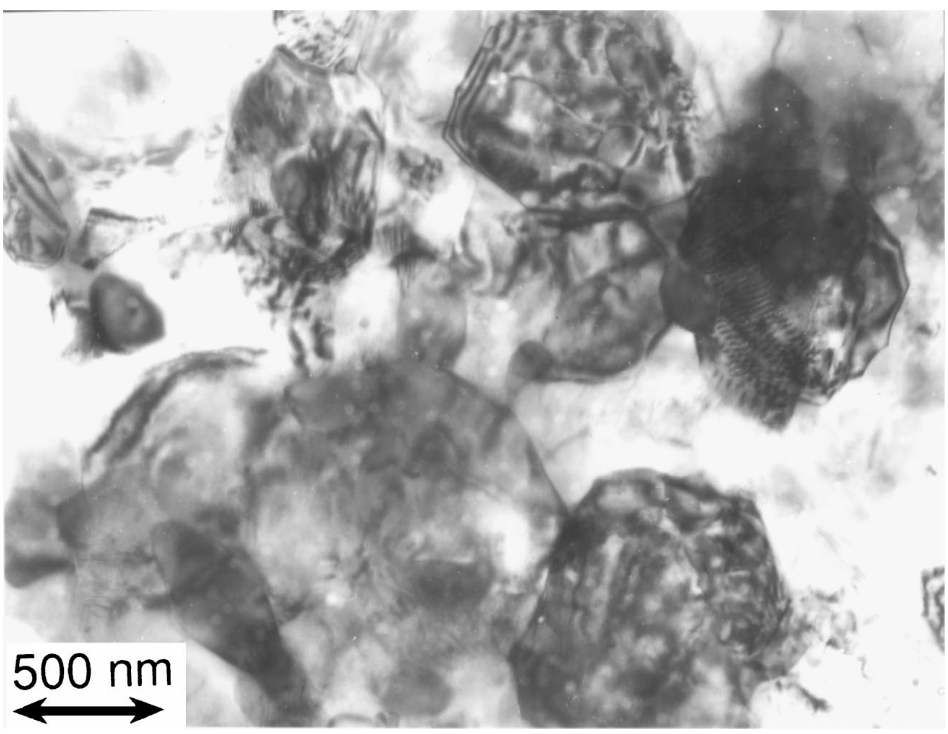

Figure 11 Microstructure of AZ91 processed by ECAE.

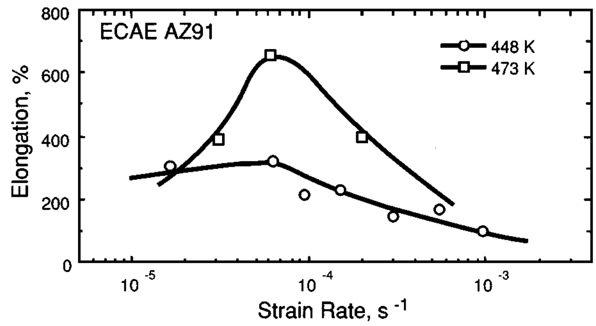

Figure 12 The variation in elongation to failure as a function of strain rate for AZ91 processed by ECAE.

noted that a very small grain size of less than 1 μm was attained from an as-cast ingot by ECAE. Therefore, ECAE is expected to be a new processing method by which a very small grain size is attained from as-cast ingots.

The AZ91 processed by ECAE shows superplastic behavior at low temperatures of 448–473 K [64]. The temperature range is about $0.5T_m$, where T_m is the melting temperature of pure magnesium (=924 K). The variation in elongation to failure as a function of strain rate is shown in Fig. 12 for AZ91 processed by ECAE. It should be noted that a large elongation of 660% is attained at about $0.5T_m$. In general, superplasticity is attained at elevated temperatures $>0.5T_m$, where T_m is the melting temperature of the material. For example, a fine-grained 7075 Al alloy, which is a typical superplastic Al alloy, shows superplastic behavior at 793 K. The temperature of 793 K is $0.85T_m$, where T_m is the melting temperature of pure aluminum (=933 K). It should be noted that the AZ91 processed by ECAE exhibits superplastic behavior at low temperatures of about $0.5T_m$. The low temperature superplasticity is attributed to a very small grain size [64].

5. Summary

For magnesium-based materials, grain refinement can be attained by hot extrusion without additional treatments. A good combination of high strength and high ductility at room temperature is attained by grain refinement. Furthermore, control of precipitation and grain refinement can be attained by thermo-mechanical treatments (TMT) consisting of hot working and heat treatment. The WE43 alloy processed by TMT showed high strength, high ductility, high creep resistance and high strain rate superplasticity. Also, a very small grain size of less than 1 μm is attained by equal channel angular extrusion. The fine-grained AZ91 exhibited superplastic behavior at low temperatures ($\leq$473 K). Thus, grain refinement significantly improves mechanical properties for magnesium-based materials.

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