

INFLUENCE OF TEMPERATURE AND STRAIN RATE ON SUPERPLASTICITY KINETICS OF COMPOSITE AZ61-F MAGNESIUM MATERIALS

Michal Besterčí^{1)*}, Song-Jeng Huang²⁾, Katarína Sülleiová¹⁾, Beáta Ballóková¹⁾

¹⁾ Institute of Materials Research of Slovak Academy of Sciences, Košice, Slovakia

²⁾ Department of Mechanical Engineering, National Taiwan University of Science and Technology, Taipei, Taiwan

Received: 28.08.2018

Accepted: 17.09.2018

*Corresponding author: e-mail:mbesterčí@saske.sk Tel.: +421 557922411, Institute of Materials Research of Slovak Academy of Sciences, Watsonova 47, 040 01 Košice, Slovakia

Abstract

Micromechanisms of fracture of AZ61-F composites in the zone of quasi-superplastic deformation were analyzed and quantified in this work. Deformation of AZ61-F magnesium alloys with 1 wt.% of Al₂O₃ phase was tested at a temperature of 473 K and different strain rates. It was shown that at the temperature of 473 K and the highest strain rate applied from 1×10^{-2} to $1 \times 10^{-4} \text{ s}^{-1}$, a significant growth of ductility was observed. The mean dimples diameter of the ductile fracture decreased with the decreasing strain rate. The grain size of 0.7 μm was reached by severe plastic deformation using equal channel angular pressing (ECAP). Secondary Mg₁₇Al₁₂ and Al₂O₃ phases were identified. The maximum strain was reached at the temperature of 473 K and strain rate of $1 \times 10^{-4} \text{ s}^{-1}$.

Keywords: Mg composite materials, superplasticity, temperature, strain rate, fractographic analysis

1 Introduction

Magnesium alloys are gaining more recognition as the lightest structural materials for lightweight applications, due to their low density and high stiffness-to-weight ratio. Even so, Mg alloys have not been used for critical performance applications because of their inferior mechanical properties, compared to other engineering materials. Nevertheless, there are some practical limitations for using magnesium alloys in industrial applications because these alloys are inherently brittle due to their hexagonal crystal structure and the consequently limited number of active slip systems. An important current requirement is therefore to develop procedures having the potential for improving the ductility of Mg-based alloys. Nowadays it is well established that the processing of fcc metals by the method of the equal channel angular pressing leads to ultrafine grain sizes that are typically within the submicrometer range [1]. If these small grain sizes are reasonably stable at the elevated temperatures, it is possible to achieve a superplastic ductility with elongations to the failure of $> 1000 \%$ [2, 3]. However, it is more difficult to apply ECAP processing to Mg-based alloys successfully. Miyahara et al. [4] experiments were conducted on a commercial AZ61 magnesium alloy to evaluate the potential for achieving the ultrafine grain size and superplastic ductility by the EX-ECAP two-step processing procedure of extrusion plus equal channel angular pressing. The results show that EX-ECAP gives excellent grain refinement with grain size of ~ 0.6 and $\sim 1.3 \mu\text{m}$ after pressing at 473 and 523 K, respectively. The alloy processed by EX- ECAP exhibits exceptional

superplastic properties including a maximum elongation of 1000 % after pressing through four passes when testing at 473 K with an initial strain rate of $3.3 \times 10^{-4} \text{ s}^{-1}$. Hence, many researchers attempt to fabricate Mg based metal matrix composites by different methods to obtain light-weight materials with excellent mechanical properties [5–8]. Magnesium based nanocomposite (AZ91-D Al_2O_3) was studied in [9, 10], where the effect of nano-alumina particles and heat treatment on microstructure, mechanical properties and failure mechanism were investigated. Superplasticity in polycrystalline materials is facilitated by dynamic modifications in the microstructure. Retrieval of the superplastic microstructure at elevated temperatures rests on the ability to maintain dynamic recovery. The strain rate is defined by Mukherjee equation, [11]:

$$\dot{\varepsilon} = K D G^2 b / k T (\sigma / G)^p (\sigma / G)^{1/m} \quad (1)$$

where: K - constant

D - diffusion coefficient

G [Pa] - shear modulus,

b - Burgers vector

k - Boltzmann's constant

T [K] - temperature

d [μm] - grain size

σ [Pa] - applied stress

p - grain size exponent (2 - 3)

1/m - stress exponent (0.4 – 0.7)

The primary deformation mechanism in superplastic materials is a grain boundary sliding with stress accommodation by diffusion or dislocation movement. According to eq. (1), the finer grains, the higher strain rate to obtain superplasticity at a given stress. Grains are smaller than 10 μm , and equiaxed grains with large angle boundaries are essential. Superplasticity is maintained if the dynamic growth of grains is minimized. However, superplasticity can be limited by cavitation on the grain boundaries, [12]. In Ref. [13], the main features of the high strain rate superplasticity are described:

1. superplasticity depends on the parameters of the microstructure – both matrix and secondary phase grain sizes,
2. temperature suitable for superplasticity is dependent on the matrix material,
3. thermo-mechanical treatment can significantly influence superplasticity.

Intensive R&D produced a variety of superplastic alloys and after thermo-mechanical treatment, superplastic behavior was reported in many conventional alloys, too. Research was focused first on Al, Ti and Mg base alloys. Superplasticity in Al base alloys is reported in [14–13].

The aim of this paper is to analyze kinetics of superplastic behavior of AZ61-F-Mg composites with 1 wt% of Al_2O_3 phase in dependence on the temperature and strain rate.

2 Experimental materials and methods

The matrix used in this work is magnesium alloy AZ61-F made by Metaltech Industrial Co, LTD, Taiwan, the chemical composition is shown in **Table 1**.

Table 1 Chemical composition of AZ61-F

Elements	Al	Mn	Zn	Si	Fe	Cu	Ni	Mg
wt. %	5.95	0.26	0.64	0.009	0.005	0.0008	0.0007	Balance

Particles Al_2O_3 with the weight fraction of 1 % are used as the reinforcement phase. The commercially available Al_2O_3 powder with a particle diameter about 20 nm with the purity of ≥ 99.8 % is added into AZ61-F to form Mg-based metal-matrix composites. The melt-stirring technique was used to fabricate the present Mg composites. The AZ61-F and Al_2O_3 particles were initially placed inside a graphite crucible and heated to 673 K in a resistance-heated furnace; then a stirring vane functioned; meanwhile, CO_2 and SF_6 gushed from the gas tank into the crucible to mix the melt. CO_2 and SF_6 also prevented the melt from oxidation. The crucible was continuously heated up to 973 K, and then the molten alloy was stirred with a vane operated at 350 revmin^{-1} for 3 min. Finally, the composite melt was poured into a metallic mould. The Mg composite containing 1 wt. % of Al_2O_3 was prepared for further mechanical testing. ECAP was carried out in a die with the die angle of $\Phi = 120^\circ$. The scheme of ECAP is shown in **Fig. 1**. The deformation temperature was set at 573 ± 10 K. During deformation, the plunger speed was about 1.0 mms^{-1} . After each extrusion pass, the billet was quenched into water. The billets were rotated counterclockwise around the exit extrusion axis by 90° between each pass, the so-called Bc route, and each bar was ECAPed by 4 passes. From these alloys, specimens ($\varnothing 3$ and 30mm long) were prepared for tensile testing.

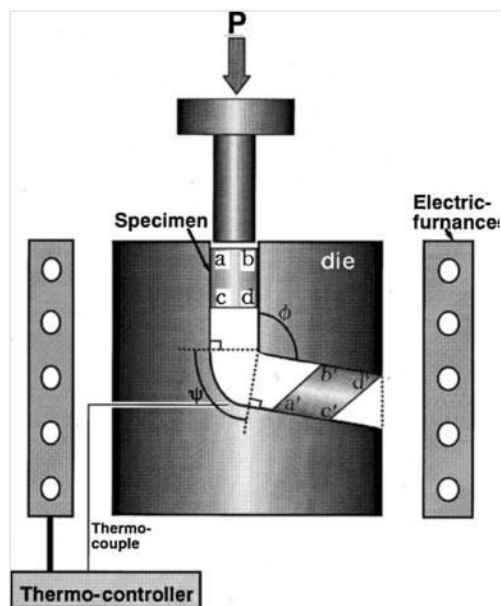


Fig. 1 Scheme of the equal-channel angular pressing (ECAP)

3 Results and discussion

Microstructure parameters of the system, namely the matrix grain size, mean size of the dispersed particles and their average distance before and after ECAP were evaluated. Microstructure of the starting material (before ECAP) was heterogeneous with grain size of 25–30 μm . The grains sizes of material after ECAP were observed by TEM, **Fig. 2**. In comparison with the grain size of starting experimental material, the grain sizes after ECAP decreased on the values of cca 0.7 μm . The mean size was estimated by measurement of 50–100 grains which had good contrast. Results are in a good agreement with [24]. Fine Al_2O_3 particles observed by TEM

were distributed randomly as well as in clusters. Al_2O_3 particles sized of cca 20 nm and their clusters are localized on the grain boundaries and in the grains. Transmission electron microscopy and selective electron diffraction showed the presence of spherical $\text{Mg}_{17}\text{Al}_{12}$ phase; the mean particles size was 0.5 μm , **Fig. 3**. These results are in a good agreement with [25, 26]. This phase has a body centered cubic crystal structure with the lattice parameter $a = 10.569 \text{ \AA}$. $\text{Mg}_{17}\text{Al}_{12}$ particles were distributed preferentially on the high angle grain boundaries and inhibit the grains growth.

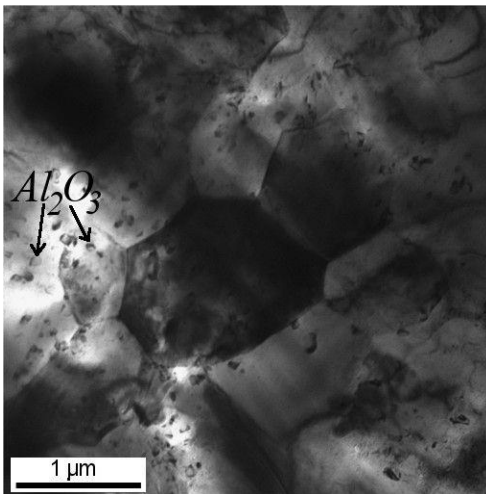


Fig. 2 TEM micrographs of grains sizes and distribution of Al_2O_3 particles, material after ECAP

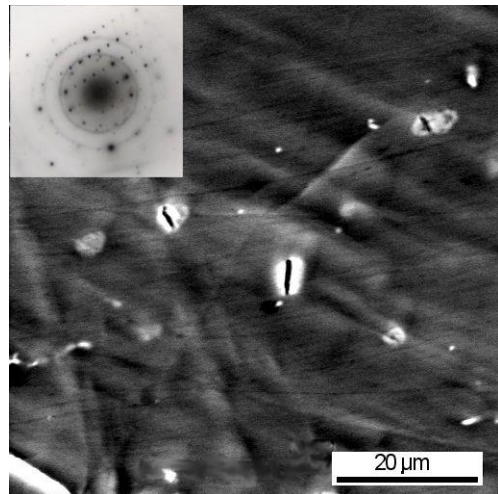


Fig. 3 TEM micrographs and diffractogram of $\text{Mg}_{17}\text{Al}_{12}$ phase, material after ECAP

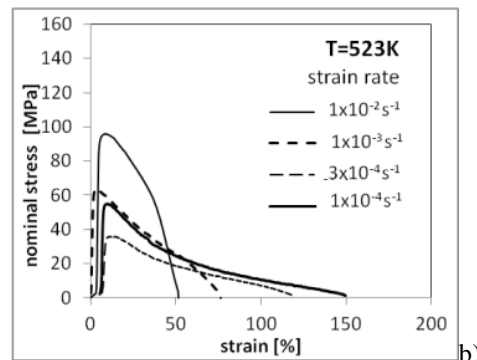
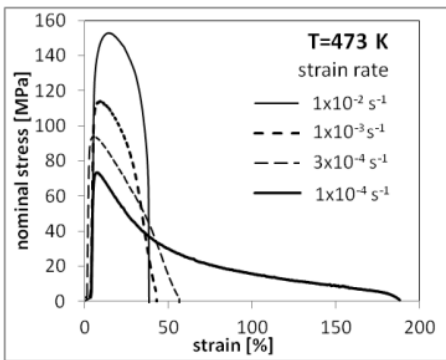


Fig. 4 Nominal stress vs. true strain at 473 K (a) and 523 K (b) for samples with 1% of Al_2O_3 phase pressed through four passes

Analyses of the deformation behavior were performed by tensile deformation at temperatures 473 and 523 K and strain rates of $10^{-2} \text{ s}^{-1} \times 10^{-4} \text{ s}^{-1}$, **Fig. 4**. It was found that experimental material had maximal superplasticity at temperature of 473 K and strain rate of $1 \times 10^{-4} \text{ s}^{-1}$, **Fig. 4(a)**, what is in agreement with [25, 26] for the commercial alloy AZ61Mg. A probable mechanism of superplasticity is the high angle grain boundary sliding. Strength properties

decrease with higher test temperature as expected, **Fig. 4(b)**. Dependences of nominal stress versus strain at different strain rates are identical in the both plots. **Fig. 5** shows the specimens tested at the slower rates pull out to a point. It is a proof of macroscopic necking within gauge lengths. It is well established that this behavior represents a true superplastic condition.

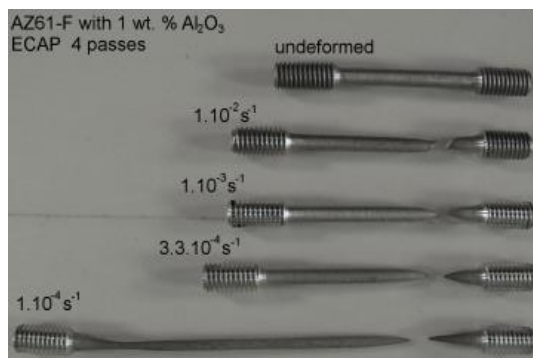


Fig. 5 Test specimens of AZ61-F with 1% of Al_2O_3 phase after deformation by different strain rates at 473 K

A fracture surface at optimal temperature of 473 K and strain rate of $1 \times 10^{-4} \text{ s}^{-1}$ is shown in **Fig. 6**. The fracture is a transcrystalline ductile with dimples of two size categories. Larger dimples ($10 \mu\text{m}$) were created on $\text{Mg}_{17}\text{Al}_{12}$ particles, smaller ones on dispersed Al_2O_3 particles (ca $0.7 \mu\text{m}$). According to Gurland–Plateau theory the ductile fracture had three stages: initiation, growth and coalescence of cavities. Cracks were created on the particle–matrix interphases and presumably in the triple junctions of the grains of the matrix, [26].

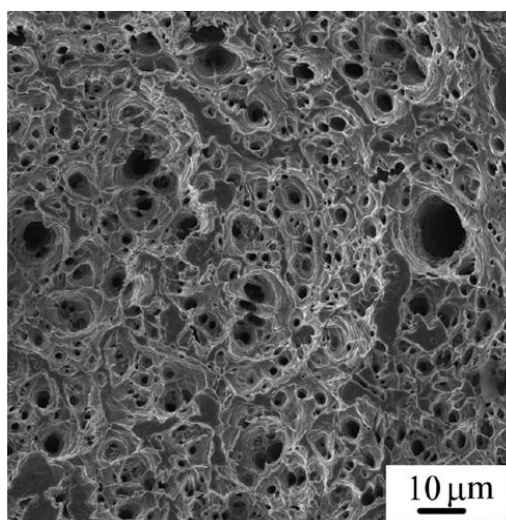


Fig. 6 Fracture at temperature of 473 K and strain rate of $1 \times 10^{-4} \text{ s}^{-1}$

Conclusions

Based on the extensive experimental studies of kinetics of superplastic deformation of AZ61 Mg- Al_2O_3 composites the following results were obtained:

1. The mean grain size of the experimental material with 1 wt.% of Al₂O₃ after ECAP by 4 passes was 0.6–0.8 μm. Mg₁₇Al₁₂ and Al₂O₃ particles of secondary phases were identified by TEM.
2. The max value of superplasticity cca 180% was reached at the temperature of 473 K and strain rate of $1 \times 10^{-4} \text{ s}^{-1}$. A probable mechanism of superplasticity is the high angle grain boundary sliding.
3. At maximum values of superplasticity the fracture was transcrystalline ductile with dimples of two size categories (10 μm and 0.7 μm) initiated by the particles of secondary phases (Mg₁₇Al₁₂ and Al₂O₃).

References

- [1] Y. Iwahashi, Z. Horita, M. Nemoto, T. G. Langdon: Acta Materialia, Vol. 46, 1998, No. 9, p. 3317-3331, DOI: 10.1016/S1359-6454(97)00494-1
- [2] C. Xu, M. Furukawa, Z. Horita, T. G. Langdon: Acta Materialia, Vol. 53, 2005, No. 3, p. 749-758, DOI: 10.1016/j.actamat.2004.10.026
- [3] M. Kawasaki, T. G. Langdon: Acta Materialia, Vol. 53, 2005, No.20, p. 5353-5364, DOI: 10.1016/j.actamat.2005.08.012
- [4] Y. Miyahara, Z. Horita, T. G. Langdon: Materials Science Engineering A, Vol. 420, 2006, No. 1-2, p. 240-244, DOI: 10.1016/j.msea.2006.01.043
- [5] G. Cao, H. Choi, J. Oportus, H. Konishi, X. Li: Materials Science Engineering A, Vol. 494, 2008, No. 1-2, p. 127-131, DOI: 10.1016/j.msea.2008.04.070
- [6] S. Ugandhar, M. Gupta, S. K. Sinha: Composite Structures, Vol. 72, 2006, No. 2, p. 266-272, DOI: 10.1016/j.compstruct.2004.11.010
- [7] S. F. Hassan, M. Gupta: Journal of Alloys and Compounds, Vol. 419, 2006, No. 1-2, p. 84-90, DOI: 10.1016/j.jallcom.2005.10.005
- [8] S. F. Hassan, M. Gupta: Materials Science Engineering A, Vol. 392, 2005, No.1-2, p. 163-168, DOI: 10.1016/j.msea.2004.09.047
- [9] M. Shanthi, K. S. Tun, R. S. Pandey, M. Gupta: Kovove Mater., Vol. 49, 2011, No. 3, p. 197-205, DOI: 10.4149/km_2011_3_197
- [10] P. Ch. Lin, S. J. Huang, P. S. Hong: Acta Metallurgica Slovaca, Vol. 16, 2010, No. 4, p. 237-244
- [11] A. K. Mukherjee, J. E. Bird, J. E. Dorn: Transactions of American Society for Metals, Vol. 62, 1969, p. 155-179
- [12] R.S. Mishra, A.K. Mukherjee: Materials Science Engineering A, Vol. 234, 1997, p. 1023-1025, DOI: 10.1016/S0921-5093(97)00321-3
- [13] R.S. Mishra, T.R. Bieler, A.K. Mukherjee: Scripta Metallurgica et Materialia, Vol. 26, 1992, p. 1605-1608, DOI: 10.1016/0956-716X(92)90264-F
- [14] K. Higashi: Materials Science Engineering A, Vol. 166, 1993, No. 15, p. 109-118, doi: 10.1016/0921-5093(93)90315-6
- [15] T. Kvackaj et al.: Archives of Metallurgy and Materials, Vol. 58, 2013, Issue 2, p. 407-412, DOI: 10.2478/amm-2013-0008
- [16] J. Bidulska et al.: Archives of Metallurgy and Materials, Vol. 58, 2013, Issue 2, p. 371-375, DOI: 10.2478/amm-2013-0002
- [17] J. Bidulska et al.: Acta Physica Polonica A, Vol. 117, 2010, Issue 5, p. 864-868, DOI: 10.12693/APhysPolA.117.864

- [18] T. Kvackaj et al.: Acta Physica Polonica A, Vol. 122, 2012, Issue 3, p. 557-560, DOI: 10.12693/APhysPolA.122.557
- [19] I. Jin, T.R. Bieler: Journal of Materials Science., Vol. 28, 1993, No. 9, p. 2413-2417
- [20] T.G. Nieh, J. Wadsworth, T. Imai: Scripta Metallurgica et Materialia, Vol. 26, 1992, No. 5, p. 703-708, DOI: 10.1007/BF01151673
- [21] K. Higashi, T. Okada, T. Mukai, S. Tanimura: Scripta Metallurgica et Materialia, Vol. 26, 1992, No. 5, p. 1605-1608
- [22] F. Li, W.T. Roberts, P.S. Bate: Acta Materialia, Vol. 44, 1996, No. 1, p. 217-233, DOI: 10.1016/1359-6454(95)00141-8
- [23] A. Urena, J.M. Gomez de Salazar, J. Quiñones, J.J. Martin: Scripta Materialia, Vol. 34, 1996, No. 4, p. 617-623, DOI: 10.1016/1359-6462(95)00551-X
- [24] A. El-Morsy, A. Ismail, M. Waly: Materials Science and Engineering A, Vol. 486, 2008, No. 1-2, p. 528-533, DOI: 10.1016/j.msea.2007.09.044
- [25] K. Matsubara, Y. Miyahara, Z. Horita, T.G. Langdon: Metallurgical and Materials Transactions A, Vol. 35A, 2004, No. 6, p. 1735-1744, DOI: 10.1007/s11661-004-0082-z
- [26] Y. Miyahara, K. Matsubara, Z. Horita, T.G. Langdon: Metallurgical Materials Transactions A, Vol. 36A, 2005, No. 7, p. 1705-1711, DOI: 10.1007/s11661-005-0034-2

Acknowledgements

Authors are grateful for the support of experimental works by the project VEGA 2/0080/17 supported by the Slovak National Grant Agency.