

## HIGH STRAIN RATE SUPERPLASTICITY OF WE43 MAGNESIUM ALLOY

<sup>1</sup>Tomáš VÁVRA, <sup>1</sup>Robert KRÁL, <sup>2</sup>Jiří KUBÁSEK, <sup>1</sup>Mária ZEMKOVÁ, <sup>1</sup>Peter MINÁRIK

<sup>1</sup>Charles University, Department of Physics of Materials, Prague, Czech Republic, EU, tomvavra1@gmail.com

<sup>2</sup>University of Chemistry and Technology, Department of Metals and Corrosion Engineering, Prague, Czech Republic, EU, <u>Jiri.Kubasek@vscht.cz</u>

https://doi.org/10.37904/metal.2019.760

#### **Abstract**

Superplastic behavior of ultrafine-grained (UFG) magnesium alloy Mg-4Y-3RE (wt%) prepared by equal channel angular pressing (ECAP) was investigated at high strain rates. Eight passes through ECAP resulted in grain refinement down to  $\sim$ 340 nm and formation of a high volume fraction of fine secondary phase particles located at the grain boundaries and triple-points. Dense distribution and high thermal stability of these particles provided excellent thermal stability of the UFG microstructure even at high temperatures. The microstructure of the material was investigated by transmission electron microscopy. Superplastic behavior was investigated in the temperature range of 350-450 °C and strain rate range of  $10^{-2}$  s<sup>-1</sup> -  $10^{-1}$  s<sup>-1</sup>. The results revealed high strain rate superplasticity in the investigated material. Deformation to fracture exceeded 1000 % for several deformation conditions, even at the strain rate of  $10^{-1}$  s<sup>-1</sup>.

Keywords: Superplasticity, magnesium alloy, equal channel angular pressing

# 1. INTRODUCTION

Magnesium alloys offer some unique features such as the lowest density of all structural metals, good castability or high specific strength. For these qualities, magnesium alloys are used as construction materials in automotive aerospace and electronics industry. Furthermore, it is used as a material for energy absorption and vibration damping. For their nontoxicity, Mg alloys have potential in medicine as biodegradable implants [1].

WE43 - Mg-4Y-3RE (rare-earth elements) (wt%) is a well-known commercial alloy. This alloy has exceptional qualities such as high strength, creep resistance and good corrosion resistance [2]. It is suitable for usage even at elevated temperatures up to 300 °C. Owing to these properties, this alloy is used especially in aviation and automotive industry. It has also potential in medicine for biodegradable implants manufacture [3-5]. The previous works showed that the WE43 alloy has good superplastic behavior after ECAP processing [6,7]. However, recently, significantly higher grain refinement was achieved due to the ECAP parameters optimization resulting in an average grain size of ~340 nm [8]. Moreover, the microstructure exhibited a high density of fine Mg $_5$ RE particles. Consequently, the thermal stability of the ultrafine-grained microstructure was improved [9]. Since the thermally stable microstructure with remarkably small grain size is a key factor for superplasticity, superior superplastic behavior is expected to be achieved in WE43 alloy with the optimized UFG microstructure.

#### 2. EXPERIMENTAL MATERIAL AND METHODS

The microstructure and ECAP processing parameters of the WE43 (Mg -3.8 wt% Y -2.6 wt% RE -0.45 wt% Zr -0.01 wt% Mn) alloy used in this study is well described in [8, 9]. It was shown that microstructure with an average grain size of ~340 nm and with a high density of fine  $Mg_5RE$  particles was achieved, see **Figure 1**. These particles having 150 nm in diameter were predominately found at grain boundaries and triple points,



and thus help to stabilize the microstructure. Annealing for 1 hour showed that the UFG microstructure was stable up to 280 °C [9].

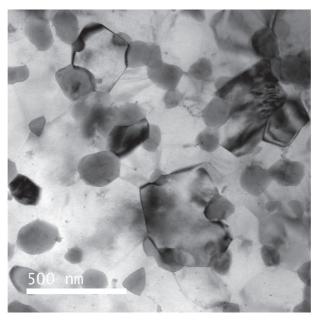


Figure 1 TEM micrograph of the investigated material [9]

The tensile deformation tests were carried out by Instron 5882 device using the selected deformation conditions. Dog-bone-shaped samples were machined out from the ECAP billets parallel to the processing direction. The samples were 1mm thick with the active zone of 6 x 4 mm.

Microstructure observation was performed by transmission electron microscope Jeol FX200. The samples were mechanically thinned and subsequently ion-milled by Leica EM RES102.

# 3. RESULTS

Firstly, the m-parameter was measured in the temperature range of 300-500 °C. For each temperature, the strain rate was step-wisely changed in the range from  $10^{-5}$  s<sup>-1</sup> to  $10^{-1}$  s<sup>-1</sup>. The changes in true stress were analyzed and the m-parameter was calculated according to (1). The dependence of m-parameter on the strain rate is shown in **Figure 2 (a)**. The additional line showing m = 0.3 is shown in **Figure 2(a)** because m > 0.3 is a necessary condition for superplastic deformation [10].

$$m = \frac{\delta \ln(\sigma)}{\delta \ln(\dot{\varepsilon})} \tag{1}$$

where  $\sigma$  is the stress (Pa) and  $\varepsilon$  is the strain rate (s<sup>-1</sup>) [11].

The m-parameter of the investigated material was sufficiently high in the whole investigated range (**Figure 2**) for almost all temperatures. The results showed that the m-parameter was much higher than 0.3 even at the strain rate of 10<sup>-1</sup> s<sup>-1</sup>. Nevertheless, the highest values of m-parameter at the highest strain rates was found for the temperature of 400 °C, which is considered as an ideal candidate for further investigation.

For the lower temperatures (from 300  $^{\circ}$ C to 400  $^{\circ}$ C), the *m*-parameter maximum is lower and/or shifted to the lower strain rates. It is expected that the average grain size remained relatively small and superplastic deformation can proceed by grain boundary sliding and diffusion creep. However, these thermally activated processes are not as fast as in the case of 400  $^{\circ}$ C. On the other hand, the increase in the deformation temperature to 450  $^{\circ}$ C and 500  $^{\circ}$ C led to a decrease of m-parameter as well. This deterioration can be



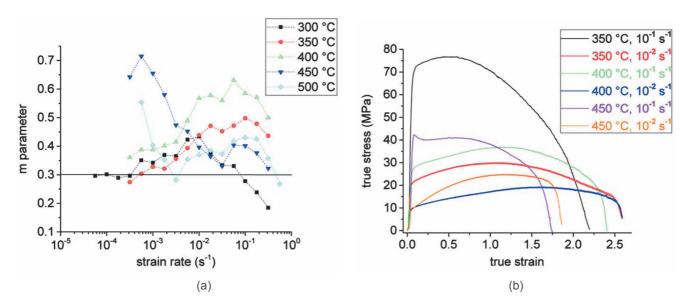
explained by a grain growth, which probably occurred during the temperature stabilization before the execution of the test. Therefore, the atoms have to travel a longer distance in larger grains during diffusion creep, which is slower than in the sample measured at 400 °C.

For the subsequent deformation tests, the strain rates of 10<sup>-2</sup> s<sup>-1</sup> and 10<sup>-1</sup> s<sup>-1</sup> were chosen, because the aim of this study was the investigation of the high strain rate superplasticity. As mentioned, the highest m-parameter in this strain rate range in question was measured for the temperature of 400 °C, therefore, the best superplastic behavior was expected for this temperature. In addition, one higher and one lower temperature was selected as well.

Deformation to failure tensile tests were performed using the aforementioned conditions. The resulting true stress-true strain curves are shown in **Figure 2** (b), Maximal elongation achieved during superplastic deformation is shown in **Table 1**. For the strain rate of  $10^{-1}$  s<sup>-1</sup>, the maximal elongation of 1013 % (2.41 true strain) was achieved at the temperature of 400 °C. For the strain rate of  $10^{-2}$  s<sup>-1</sup>, the maximal elongation of 1232 % (2.59 true strain) was achieved for two temperatures 350 °C and 400 °C. These results correspond with the m-parameter measurement, where the highest m-parameter for these strain rates was achieved at 400 °C followed by 350 °C (**Figure 2** (a)).

Grain boundary sliding and diffusion creep were not as active at 350 °C as at 400 °C, leading to lower deformation to failure 799 % (2.19 true strain). On the other hand, the results showed that the temperature of 450 °C was too high for superplastic deformation. According to [9], significant grain growth occurs at this temperature. Increase in the grain size during temperature stabilization (prior to the begin of the test) resulted in higher yield stress due to the decreased activity of grain boundary sliding, as depicts **Figure 2** (b). Consequently, maximal elongation was lower than the one measured at 400 °C for both strain rates. Moreover, at a strain rate of 10<sup>-1</sup> s<sup>-1</sup> sharp yield point appeared, which suggest that the dislocation deformation mechanism was the most active.

Upon the literature review, such excellent high strain rate superplasticity, as measured in this study, hasn't been reported in the WE43 alloy yet [6,7,12-14]. The highest elongation achieved so far has been 860 % and 960 % at  $10^{-1}$  s<sup>-1</sup> and  $2 \times 10^{-2}$  s<sup>-1</sup>, respectively [7].



**Figure 2** Effect of temperature to the evolution of m-parameter as a function of strain rate (a) and results of tensile deformation tests for selected conditions (b).



| Table 1  | Maximum       | alangation | roochod | durina | ouporplantia | deformation |
|----------|---------------|------------|---------|--------|--------------|-------------|
| i abie i | IVIAXIIIIUIII | elondation | reached | aurina | Superplastic | deformation |

| Temperature (°C) | Strain rate (s <sup>-1</sup> ) | Elongation (%) |
|------------------|--------------------------------|----------------|
| 0.50             | 10 <sup>-1</sup>               | 799            |
| 350              | 10 <sup>-2</sup>               | 1233           |
|                  | 10 <sup>-1</sup>               | 1013           |
| 400              | 10-2                           | 1232           |
|                  | 10 <sup>-1</sup>               | 475            |
| 450              | 10 <sup>-2</sup>               | 542            |

### 4. CONCLUSION

Superplastic behavior of ultrafine-grained (UFG) magnesium alloy Mg-4Y-3RE (wt%) prepared by equal channel angular pressing (ECAP) was investigated at high strain rates. It was shown that achieving grain refinement down to ~340 nm and formation of a high volume fraction of fine secondary phase particles located at the grain boundaries significantly enhanced superplastic behavior due to the enhanced grain boundary sliding and high thermal stability of the ultrafine-grained microstructure. Consequently, high strain rate superplasticity was observed in the investigated material. Deformation to failure of 1013 % at the strain rate of  $10^{-1} \, \text{s}^{-1}$  and  $1132 \, \%$  at the strain rate  $10^{-2} \, \text{s}^{-1}$  was significantly higher than previously reported in the investigated alloy using comparable deformation rates.

#### **ACKNOWLEDGEMENTS**

The authors are grateful for the financial support of the Czech Science Foundation under the project 19-00270S. M.Z. acknowledges financial support by the Charles University under the project GA UK no. 410119.

### **REFERENCES**

- [1] ZHENG, Y.F., GU, X.N. and WITTE, F. Biodegradable metals. *Materials Science and Engineering: R: Reports*. 2014. vol. 77, pp. 1-34. 10.1016/j.mser.2014.01.001
- [2] MORDIKE, B.L. Creep-resistant magnesium alloys. *Materials Science and Engineering: A.* 2002. vol. 324, no. 1, pp. 103-112. 10.1016/S0921-5093(01)01290-4
- [3] DRYNDA, A., DEINET, N., BRAUN, N. and PEUSTER, M. Rare earth metals used in biodegradable magnesium-based stents do not interfere with proliferation of smooth muscle cells but do induce the upregulation of inflammatory genes. *Journal of Biomedical Materials Research*. *Part A*. 2009. vol. 91, no. 2, pp. 360-369.
- [4] FEYERABEND, F., FISCHER, J., HOLTZ, J., WITTE, F., WILLUMEIT, R., DRÜCKER, H., VOGT, C. and HORT, N. Evaluation of short-term effects of rare earth and other elements used in magnesium alloys on primary cells and cell lines. *Acta Biomaterialia*. 2010. vol. 6, no. 5, The THERMEC'2009 Biodegradable Metals, pp. 1834-1842.
- [5] DING, Y., WEN, C., HODGSON, P. and LI, Y. Effects of alloying elements on the corrosion behavior and biocompatibility of biodegradable magnesium alloys: a review. *Journal of Materials Chemistry B.* 2014. vol. 2, no. 14, pp. 1912-1933.
- [6] KANDALAM, S., SABAT, R.K., BIBHANSHU, N., AVADHANI, G.S., KUMAR, S. and SUWAS, S. Superplasticity in high temperature magnesium alloy WE43. *Materials Science and Engineering: A.* 2017. vol. 687, pp. 85-92.
- [7] KANG, Z., ZHOU, L. and ZHANG, J. Achieving high strain rate superplasticity in Mg-Y-Nd-Zr alloy processed by homogenization treatment and equal channel angular pressing. *Materials Science and Engineering: A.* 2015. vol. 633, pp. 59-62. 10.1016/j.msea.2015.03.011



- [8] MINÁRIK, P., VESELÝ, J., KRÁL, R., BOHLEN, J., KUBÁSEK, J., JANEČEK, M. and STRÁSKÁ, J. Exceptional mechanical properties of ultra-fine grain Mg-4Y-3RE alloy processed by ECAP. *Materials Science and Engineering: A.* 2017. vol. 708, pp. 193-198. 10.1016/j.msea.2017.09.106
- [9] MINÁRIK, P., VESELÝ, J., ČÍŽEK, J., ZEMKOVÁ, M., VLASÁK, T., KRAJŇÁK, T., KUBÁSEK, J., KRÁL, R. HOFMAN, D. and STRÁSKÁ, J. Effect of secondary phase particles on thermal stability of ultra-fine grained Mg-4Y-3RE alloy prepared by equal channel angular pressing. *Materials Characterization*. 2018. vol. 140, pp. 207-216. 10.1016/j.matchar.2018.04.006
- [10] (PDF) Superplasticity and its practical using. {Superplasticita a její praktické užití}. Pavel Lukac Academia.edu. [accessed. 5. April 2019]. In Czech. Retrieved from: https://www.academia.edu/32506105/Superplasticita a jej%C3%AD praktick%C3%A9 u%C5%BEit%C3%AD
- [11] LANGDON, T.G. Seventy-five years of superplasticity: historic developments and new opportunities. *Journal of Materials Science*. 2009. vol. 44, no. 22, pp. 5998-6010. 10.1007/s10853-009-3780-5
- [12] CAO, G., ZHANG, D., CHAI, F., ZHANG, W. and QIU, C. High-strain-rate superplasticity in Mg-Y-Nd alloy prepared by submerged friction stir processing. *Advanced Engineering Materials*. 2016. vol. 18, no. 2, pp. 312-318. 10.1002/adem.201500221
- [13] WATANABE, H., MUKAI, T., ISHIKAWA, K., MOHRI, T., MABUCHI, M. and HIGASHI, K. Superplasticity of a particle strengthened WE43 magnesium alloy. *Materials Transactions MATER TRANS*. 2001. vol. 42, pp. 157-162. 10.2320/matertrans.42.157
- [14] CAO, G., ZHANG, D., CHAI, F., ZHANG, W. and QIU, C. Superplastic behavior and microstructure evolution of a fine-grained Mg-Y-Nd alloy processed by submerged friction stir processing. *Materials Science and Engineering:* A. 2015. vol. 642, pp. 157-166. 10.1016/j.msea.2015.06.086