Iranian Journal of Materials Forming 6 (2) (2019) 2-9

IJMF

Online ISSN: 2383-0042

Iranian Journal of Materials Forming

Journal Homepage: http://ijmf.shirazu.ac.ir

Grain Refinement and Hall-Petch Strengthening of Magnesium Alloy Via Alloying and Hot Extrusion

M. Razzaghi, H. Mirzadeh* and M. Emamy

College of Engineering, University of Tehran, Tehran, Iran

ARTICLE INFO

Article history:

Received 19 January 2019
Revised 14 March 2019
Accepted 29 March 2019

Keywords:

Magnesium alloys
Tensile properties
Grain size
Hall-Petch strengthening

ABSTRACT

In this paper, the effects of the addition of Al, Zn and Mn along with the application of the hot extrusion process on the microstructural refinement and enhancement of the mechanical properties of magnesium alloy have been studied. Based on Mg-2Al alloy, it was found that the addition of 0.5 wt% Zn to form Mg-2Al-0.5Zn alloy or 0.5 wt% Mn to form Mg-2Al-0.5Mn alloy is the effective way for grain refinement of α-Mg in the as-cast state. Moreover, further remarkable refinement of the grain size can be achieved by the extrusion process in such a way that the average grain size of the extruded Mg-2Al-0.5Mn alloy was determined to be 1/165 that of the as-cast Mg. The obtained refined alloys showed significant enhancement of the yield stress and tensile strength, where the former was successfully related to the average grain size by the Hall-Petch relationship with the slope of ~ 309 MPa/$\mu m^{0.5}$. The use of the grain refinement process showed that at first the yield ratio did not change considerably, while the tensile strength, the work-hardening exponent, and the uniform elongation increased. However, after a transition grain size (~ 32 µm), the yield ratio increased sharply due to the large increase in the yield stress, and hence it was not possible to further enhance the uniform elongation using the grain refinement despite obtaining higher yield and tensile strengths.

1. Introduction

The low strength and poor ductility of Mg alloys have effectively negated their desirable attributes for automotive, aerospace, and medical applications [1-3]. The response of the scientific community and related industries has been the development of special alloys and application of various processing routes.

Alloying elements such as Al, Zn, and Mn have been extensively used in this respect. Among the developed alloys, AZ (Mg-Al-Zn) and AM (Mg-Al-Mn) alloys, i.e. Mg-Al alloys with the addition of Zn and Mn below 1 wt%, are among the most widely used ones. In fact, Al, Zn, and Mn are effective grain refiners of α-Mg in the as-cast condition, where the grain refinement is known as one of the most effective means for improving the strength and ductility of the as-cast Mg alloys [4-6].

The famous Hall-Petch relationship of $\sigma = \sigma_0 + k/\sqrt{D}$ can be used to relate the yield stress (σ) to the grain size (D) through the Hall-Petch slope (k). Yu et al [7] have recently summarized the Hall-Petch data for Mg alloy by considering the effects of the texture, temperature and stain, and grain size range on k. A compilation of Hall-Petch data for Mg alloys has been shown in Table 1, which reveals that the grain size range has some effects on the values of k [7].

Table 1: Hall-Petch slope for different Mg alloys [7].

Alloy	Pure Mg	AZ31	AZ31	AZ31	AZ61	AZ91	Mg-1Zn	Mg-1Y
Range of D (µm)	11–140	2.5–8	3–23	13–140	8–150	1–100	10–218	11–190
k (MPa/$\mu m^{0.5}$)	294	304	291	281	344	244	273	252

* Corresponding author
E-mail address: hmirzadeh@ut.ac.ir (H. Mirzadeh)

Severe plastic deformation (SPD) techniques have gained significant attention due to their remarkable grain refinement and possibility of attaining superplastic forming capability [8-13]. However, among the various processing routes, the hot extrusion is the simplest one, which can induce intense grain refinement toward favorable effects on the mechanical properties of Mg alloys [14-16].

The studied AZ and AM alloys usually have high Al contents. While Al is one of the best strengthening elements in Mg alloys, its addition at high levels is unfavorable in many applications [17-19]. In other words, if the good mechanical properties are satisfied, Mg alloys with Al < 3 wt% might find wider applications. The present work aims to deal with this subject by adding small amounts of Zn and Mn (~ 0.5 wt%) and applying the hot extrusion process to induce grain refinement.

2. Experimental Details

Weight percentages being considered, molten Mg, Mg-2Al, Mg-2Al-0.5Zn (AZ20), and Mg-2Al-0.5Mn (AM20) were prepared in an electric furnace and poured from 750 °C in a metallic mold. The as-cast ingots were subjected to the homogenization treatment (400 °C for 20 h) followed by the extrusion (385 °C with ratio of 12:1). Optical and scanning electron microscopes and X-ray diffraction (XRD) were used for microstructural and phase analyses, respectively. Finally, the tensile testing (crosshead speed of 1 mm/min at room temperature) was applied to the subsize ASTM E8-04 round samples with a gauge length and diameter of 16 mm and 4 mm, respectively. The reproducibility of the results was also verified by the repetition of some tests. While the error bars were not included for the sake of brevity, it is declared that the standard deviation of the grain size and yield/tensile strength measurements were below 9.5 µm and 8 MPa, respectively.

3. Results and Discussion

3.1. Microstructures

Representative micrographs of the as-cast alloys are shown in (Fig. 1). While large α-Mg grains (~ 528 µm) can be seen for pure Mg (Fig. 1a), the addition of 2 wt% Al has resulted in a remarkable grain refinement (~ 106 µm) based on the known growth restriction effect of Al (Fig. 1b) [20]. Similarly, the addition of 0.5 wt% Zn (Fig. 1c) or 0.5 wt% Mn (Fig. 1d) to Mg-2Al to form Mg-2Al-0.5Zn and Mg-2Al-0.5Mn alloys can contribute to further grain refinement, where the average grain sizes of 56 µm and 32 µm were determined for Mg-2Al-0.5Zn and Mg-2Al-0.5Mn alloys, respectively. The arrows shown on the micrograph of Mg-2Al-0.5Mn alloy (Fig. 1d) point to a second phase, which can be better identified in the corresponding SEM image (Fig. 1e). This phase was characterized as Al_8Mn_5 intermetallic based on the XRD patterns shown in (Fig. 2). The latter fig also reveals that the other alloys just have the α-Mg matrix, which can be also verified based on their microstructures (Fig. 1).

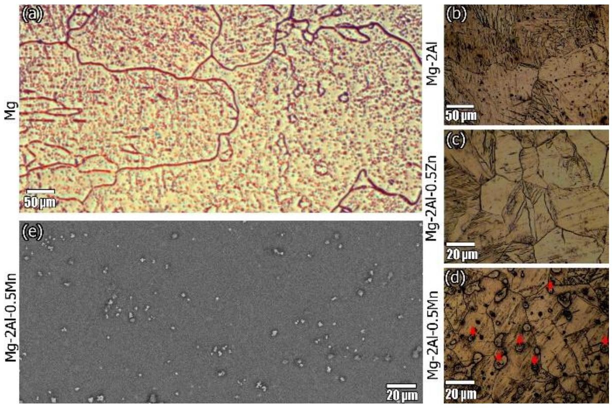


Fig. 1. Microstructures of the as-cast alloys.

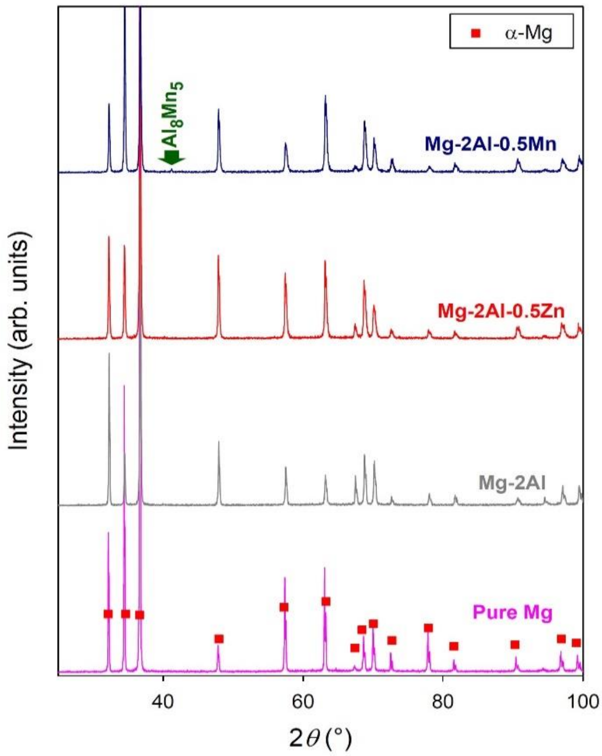
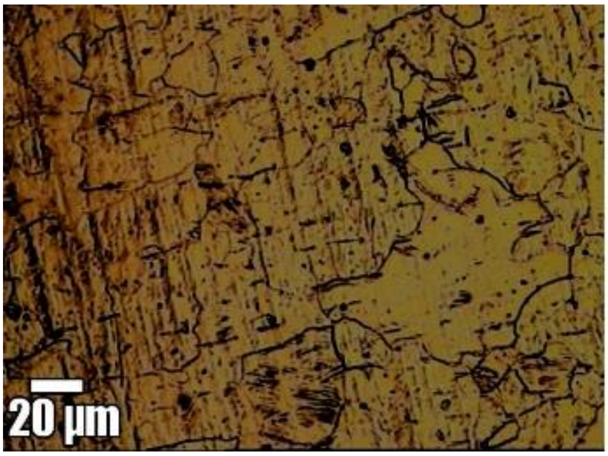
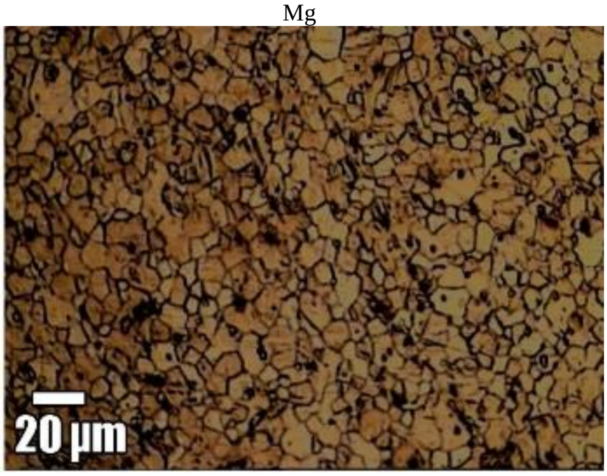


Fig. 2. XRD patterns of the as-cast alloys.

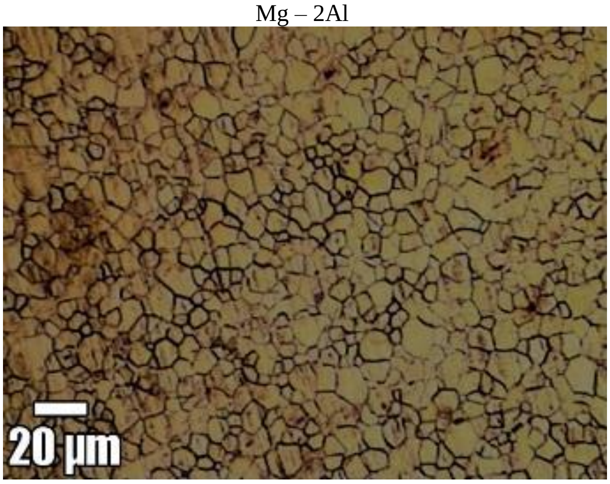
Representative extruded microstructures are shown in (Fig. 3). For all alloys, an intense grain refinement can be seen compared with the as-cast counterparts (Fig. 1), which is related to the recrystallization processes induced by hot extrusion. For instance, the finest grain size has been achieved for the extruded Mg-2Al-0.5Mn alloy with the average grain size of 3.2 μm corresponding to the ratio of $D_{as\text{-}cast}/D_{extruded} = 10$.



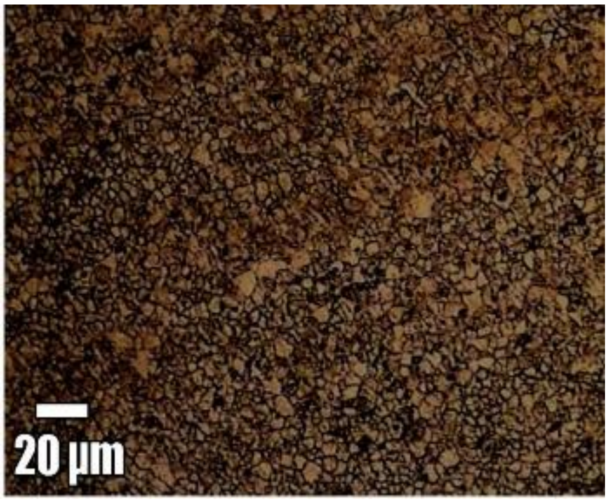
Mg



Mg – 2Al



Mg-2Al-0.5Zn



Mg-2Al-0.5Mn

Fig. 3. Representative microstructures of the extruded alloys.

3.2. Tensile properties

A summary of the tensile properties based on the tensile tests is shown in Fig. 4. It can be seen that, generally, by using the grain refinement (Fig. 4a), the yield stress (YS, Fig. 4b) and the tensile strength (UTS, Fig. 4c) increase. For instance, among the extruded alloys, Mg-2Al-0.5Mn alloy with the finest grain size shows the highest yield stress (205 MPa) and tensile strength (283 MPa). Compared with the as-cast Mg (yield stress of 38 MPa and tensile strength of 92 MPa), these values show ~ 440 % and 207 % enhancement, respectively. This reveals that by proper alloying and with the aid of hot extrusion process, it is possible to enhance the mechanical strength of Mg alloys significantly.

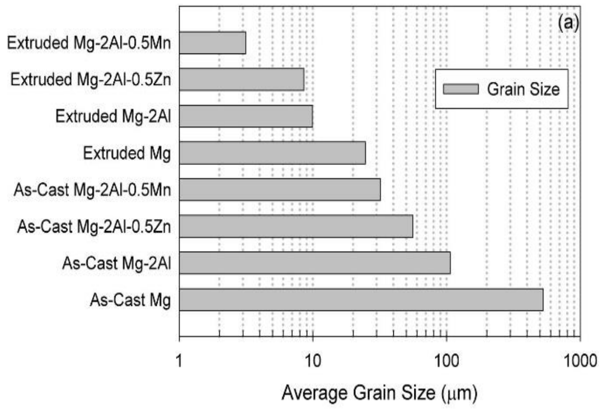


Fig. 4. Summary of the tensile properties of the alloys studied in this work.

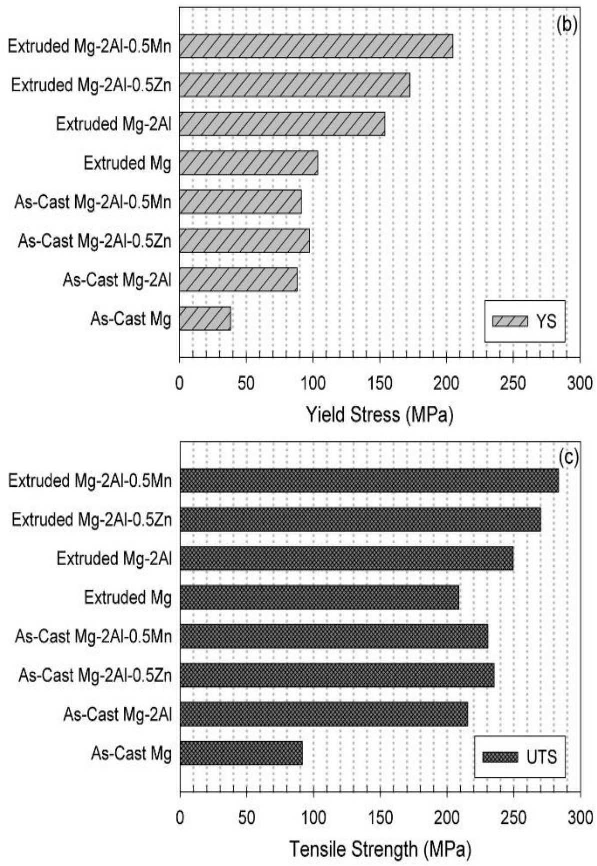


Fig. 4. Continue

The effect of the grain size can be quantitatively shown based on the Hall-Petch plot (Fig. 5), where it can be seen that all data can be represented by a linear line with a slope of ~ 309 MPa/μm$^{0.5}$. This indicates that the grain size is the main variable to control the mechanical properties of these essentially single-phase alloys. However, the solid solution strengthening and particle hardening effects due to the presence of Zn and Mn can be responsible for the significant scatter in the H-P plot. The Hall-Petch slope of 309 MPa/μm$^{0.5}$ is consistent with the results reported for pure Mg (294 MPa/μm$^{0.5}$ for the grain size range of 11–140 μm [21]), and the nearly similar AZ31 alloy (304 MPa/μm$^{0.5}$ [22], 291 MPa/μm$^{0.5}$ [23], and 281 MPa/μm$^{0.5}$ [24] for the grain size ranges of 2.5–8 μm, 3–23 μm, and 13–140 μm), respectively**.**

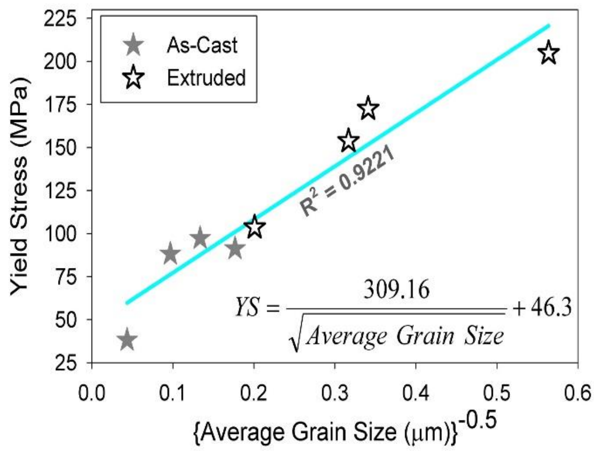


Fig. 5. Hall-Petch plot applied to the yield stress of the alloys studied in this work.

3.3. Work-hardening behavior

The yield ratio (YS/UTS) is a good indication of the work-hardening capability of the engineering alloys [25, 26], where reduction in its value is an indication of a better work-hardening capacity. The yield ratio of the present alloys versus the grain size is shown in Fig. 6a. It can be seen that refining the grain size can change the yield ratio in such a way that two trends can be identified: firstly, by grain refinement, the yield ratio does not change considerably, and second, it increases sharply by further grain refinement. By curve fitting on the parts of the graph corresponding to these two regions, the transition grain size separating these two trends was determined as 31.75 μm.

The work-hardening curves corresponding to different grain sizes are shown in Fig. 6b: Coarsest (As-Cast Mg), transition (~ As-cast Mg-2Al-0.5Mn with an average grain size of 32 μm), and finest (Extruded Mg-2Al-0.5Mn). Based on the Considere's criterion [27], the plastic instability occurs when the strain-hardening rate coincides with the flow stress. Moreover, by considering the Hollomon equation [27], the value of true strain at the coincidence point determines the work-hardening exponent (*n*-value). Based on Fig. 6b, the *n*-value increases up to the transition grain size, and then, it does not change considerably. In fact, the work-hardening curves of the as-cast and extruded Mg-2Al-0.5Mn alloys are similar except for the level of the flow stress that is higher for the extruded condition.

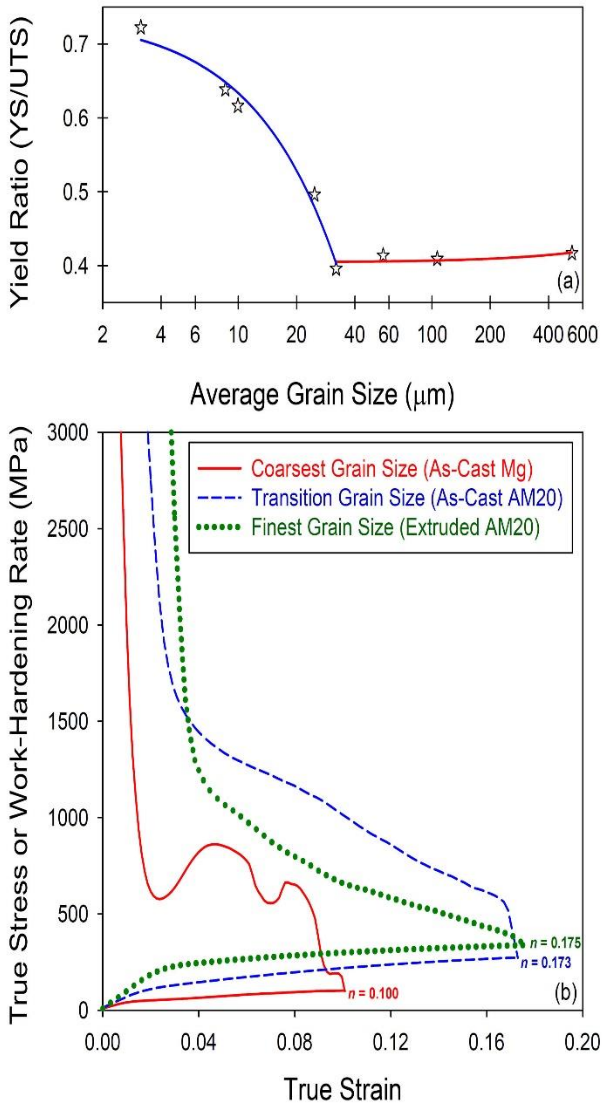


Fig. 6. Analyzing the work-hardening behavior.

Therefore, the work-hardening exponent does not reduce by grain refinement beyond the transition grain size (Fig. 6b). If the value of true strain at the coincidence point is expressed as engineering strain, the obtained value determines the uniform elongation. Therefore, by employing the grain refinement process toward the transition grain size, the uniform elongation as well as the tensile strength enhances whereas at lower grain sizes, due to the increased yield ratio (related to the sharp increase in the yield stress) and the consequent leftward shift of the coincidence point (Considere's criterion), it is not possible to further enhance the uniform elongation.

4. Conclusion

In this study, the effects of the addition of Al, Zn and Mn along with the application of the hot extrusion process on the microstructural refinement and enhancement of the mechanical properties of magnesium alloy have been examined. The following conclusions can be drawn from this study:

(1) Based on Mg-2Al alloy, it was found that the addition of 0.5 wt% Zn to form Mg-2Al-0.5Zn alloy or 0.5 wt% Mn to form Mg-2Al-0.5Mn alloy is the effective way for the grain refinement of α-Mg in the as-cast state. For instance, the average grain size of the as-cast Mg-2Al-0.5Mn alloy was determined to be 1/16.5 that of the as-cast Mg. Moreover, further remarkable refinement of the grain size can be achieved by the extrusion process in such a way that the average grain size of the extruded Mg-2Al-0.5Mn alloy was determined to be 1/10 that of its as-cast counterpart.

(2) The obtained refined alloys showed significant enhancement of the yield stress and tensile strength. For instance, the extruded Mg-2Al-0.5Mn alloy with the finest grain size shows the highest yield stress (205 MPa) and tensile strength (283 MPa). Compared with the as-cast Mg (yield stress of 38 MPa and tensile strength of 92 MPa), these values show ~ 440 % and 207 % enhancement, respectively. The yield stress was also successfully related to the average grain size by the Hall-Petch relationship with a slope of ~ 309 MPa/$\mu m^{0.5}$.

(3) By using the grain refinement, firstly the yield ratio did not change considerably, while the tensile strength, the work-hardening exponent, and the uniform elongation increased. However, after a transition grain size (~ 32 μm), the yield ratio increased sharply due to the large increase in the yield stress, and hence it was not possible to further enhance the uniform elongation using the grain refinement despite obtaining higher yield and tensile strengths.

5. References

[1] S. You, Y. Huang, K.U. Kainer, N. Hort, Recent research and developments on wrought magnesium alloys, *Journal of Magnesium and Alloys*, 5 (2017) 239-253.

[2] H. Mirzadeh, Constitutive analysis of Mg–Al–Zn magnesium alloys during hot deformation, *Mechanics of Materials*, 77 (2014) 80-85.

[3] H. Mirzadeh, M. Roostaei, M.H. Parsa, R. Mahmudi, Rate controlling mechanisms during hot deformation of Mg–3Gd–1Zn magnesium alloy: Dislocation glide and climb, dynamic recrystallization, and mechanical twinning, *Materials and Design*, 68 (2015) 228-231.

[4] M.A. Easton, M. Qian, A. Prasad, D.H. StJohn, Recent advances in grain refinement of light metals and alloys, Current Opinion in Solid State and Materials Science, 20 (2016) 13-24.

[5] B. Pourbahari, H. Mirzadeh, M. Emamy, Toward unraveling the effects of intermetallic compounds on the microstructure and mechanical properties of Mg–Gd–Al–Zn magnesium alloys in the as-cast, homogenized, and extruded conditions, *Materials Science and Engineering*, A 680 (2017) 39-46.

[6] Y. Ali, D. Qiu, B. Jiang, F. Pan, M.X. Zhang, Current research progress in grain refinement of cast magnesium alloys: a review article, *Journal of Alloys and Compounds*, 619 (2015) 639-651.

[7] H. Yu, Y. Xin, M. Wang, Q. Liu, Hall-Petch relationship in Mg alloys: A review, *Journal of Materials Science and Technology*, 34 (2018) 248-256.

[8] M. Kawasaki, R.B. Figueiredo, T.G. Langdon, The Requirements for Superplasticity with an Emphasis on Magnesium Alloys, *Advanced Engineering Materials*, 18 (2016) 127-131.

[9] R. Alizadeh, R. Mahmudi, P.H.R. Pereira, Y. Huang, T.G. Langdon, Microstructural evolution and superplasticity in an Mg–Gd–Y–Zr alloy after processing by different SPD techniques, *Materials Science and Engineering*, A 682 (2017) 577-585.

[10] R. Zheng, T. Bhattacharjee, A. Shibata, T. Sasaki, K. Hono, M. Joshi, N. Tsuji, Simultaneously enhanced strength and ductility of Mg-Zn-Zr-Ca alloy with fully recrystallized ultrafine grained structures, *Scripta Materialia*, 131 (2017) 1-5.

[11] M. Sarebanzadeh, R. Roumina, R. Mahmudi, G.H. Wu, H.R. Jafari Nodooshan, Enhancement of superplasticity in a fine-grained Mg–3Gd–1Zn alloy processed by equal-channel angular pressing, *Materials Science and Engineering*, A 646 (2015) 249-253.

[12] N. Bayat Tork, S.H. Razavi, H. Saghafian, R. Mahmudi, Superplasticity of a fine-grained Mg–1.5 wt% Gd alloy after severe plastic deformation, *Iranian Journal of Materials Forming*, 3 (2016) 65-74.

[13] E. Bagherpour, M. Reihanian, N. Pardis, R. Ebrahimi, T.G. Langdon, Ten Years of Severe Plastic Deformation (SPD) in Iran, part I: Equal-Channel Angular Pressing (ECAP), *Iranian Journal of Materials Forming*, 5 (2018) 71-113.

[14] B.P. Zhang, L. Geng, L.J. Huang, X.X. Zhang, C.C. Dong, Enhanced mechanical properties in fine-grained Mg–1.0 Zn–0.5 Ca alloys prepared by extrusion at different temperatures, *Scripta Materialia*, 63 (2010) 1024-1027.

[15] B. Pourbahari, H. Mirzadeh, M. Emamy, The effects of grain refinement and rare earth intermetallics on mechanical properties of as-cast and wrought magnesium alloys, *Journal of Materials Engineering and Performance*, 27 (2018) 1327-1333.

[16] A. Kumar, G.K. Meenashisundaram, V. Manakari, G. Parande, M. Gupta, Lanthanum effect on improving CTE, damping, hardness and tensile response of Mg-3Al alloy, *Journal of Alloys and Compounds*, 695 (2017) 3612-3620.

[17] J. Walker, S. Shadanbaz, T.B.F. Woodfield, M.P. Staiger, G.J. Dias, Magnesium biomaterials for orthopedic application: a review from a biological perspective, Journal of Biomedical Materials Research Part B: Applied Biomaterials 102 (2014) 1316-1331.

[18] S. Candan, E. Candan, Comparative study on corrosion behaviors of Mg-Al-Zn alloys, Transactions of Nonferrous Metals Society of China 28 (2018) 642-650.

[19] H. Feng, S. Liu, Y. Du, T. Lei, R. Zeng, T. Yuan, Effect of the second phases on corrosion behavior of the Mg-Al-Zn alloys, *Journal of Alloys and Compounds*, 695 (2017) 2330-2338.

[20] D.H. StJohn, M.A. Qian, M.A. Easton, P. Cao, Z. Hildebrand, Grain refinement of magnesium alloys, *Metallurgical and Materials Transactions*, A 36 (2005) 1669-1679.

[21] Y.N. Wang, C.I. Chang, C.J. Lee, H.K. Lin, J.C. Huang, Texture and weak grain size dependence in friction stir processed Mg–Al–Zn alloy, Scripta Materialia 55 (2006) 637-640.

[22] L. Guo, Z. Chen, L. Gao, Effects of grain size, texture and twinning on mechanical properties and work-hardening behavior of AZ31 magnesium alloys, *Materials Science and Engineering*, A 528 (2011) 8537-8545.

[23] Y. Chino, M. Mabuchi, R. Kishihara, H. Hosokawa, Y. Yamada, C. Wen, Apos, K. Shimojima, H. Iwasaki, Mechanical properties and press formability at room temperature of AZ31 Mg alloy processed by single roller drive rolling, *Materials Transactions*, 43 (2002) 2554-2560.

[24] Y. Wang, H. Choo, Influence of texture on Hall–Petch relationships in an Mg alloy, *Acta Materialia*, 81 (2014) 83-97.

[25] R. Kuziak, R. Kawalla, S. Waengler, Advanced high strength steels for automotive industry, Archives of Civil and Mechanical Engineering 8 (2008) 103-116.

[26] M. Nouroozi, H. Mirzadeh, M. Zamani, Effect of microstructural refinement and intercritical annealing time on mechanical properties of high-formability dual phase steel, *Materials Science and Engineering*, A 736 (2018) 22-26

[27] G.E. Dieter, *Mechanical Metallurgy*, 3rd ed., McGraw-Hill, (1988).

ریزدانه‌سازی و استحکام‌بخشی هال-پچ برای آلیاژ منیزیم از طریق آلیاژسازی و اکستروژن داغ

مرجان رزاقی، حامد میرزاده، مسعود امامی

دانشکده مهندسی متالوژی و مواد، دانشکده فنی، دانشگاه تهران، تهران، ایران.

چکیـــده

تأثیر افزودن آلومینیم، روی و منگنز در کنار استفاده از فرآیند اکستروژن داغ بر ریزدانه‌سازی و بهبود خواص مکانیکی آلیاژ منیزیم بررسی شد. براساس آلیاژ Mg-2Al، مشخص شد که افزودن نیم درصد وزنی روی و یا نیم درصد وزنی منگنز راه مناسبی برای ریزدانه‌سازی منیزیم آلفا در حالت ریختگی است. همچنین، ریزدانگی قابل توجهی از طریق فرآیند اکستروژن داغ قابل دستیابی است به شکلی که برای آلیاژ Mg-2Al-0.5Mn، نسبت اندازه دانه ریختگی به‌اندازه دانه اکسترود شده به ۱۶۵رسید. بهبود شدیدی در تنش تسلیم و استحکام کششی نهایی به‌دست آمد که بهبود تنش تسلیم توسط اثر هال-پچ با شیب ۳۰۹ $MPa/\mu m^{0.5}$ به متوسط اندازه دانه ربط داده شد. با ریزدانه سازی، نسبت تسلیم ابتدا تغییر چندانی از خود نشان داد، درحالی‌که استحکام نهایی، توان کارسختی، و ازدیاد طول یکنواخت افزایش یافتند. اما با ریزدانه‌سازی زیر اندازه دانه انتقالی (حدود ۳۲ µm)، نسبت تسلیم به شدت افزایش یافت که به افزایش تنش تسلیم نسبت داده شد. بنابراین، امکان بهبود بیشتر ازدیاد طول یکنواخت با ریزدانه‌سازی وجود نداشت درحالی‌که بهبود در تنش تسلیم و استحکام نهایی قابل دستیابی یود.

واژه‌های کلیدی: آلیاژهای منیزیم، خواص کششی، اندازه دانه، استحکام‌بخشی هال-پچ