

The deformation behavior and microstructure evolution of duplex Mg-9Li-1Al alloy during superplasticity tensile testing

Meiduo Liu^{1,*}, Haipeng Zheng², Tianlong Zhang², Ruizhi Wu²

¹Department of Environmental Engineering, Heilongjiang University of Technology, Jixi 158100, China

²College of Materials Science & Chemical Engineering, Harbin Engineering University, Harbin 150001, China

*Corresponding author e-mail: meiduo1979@163.com

Abstract. The superplastic mechanical properties and microstructure evolution of the duplex Mg-9Li-1Al alloy were investigated. The tensile testing results show that, the elongation of the as-extruded Mg-9Li-1Al alloy reaches 510% at 573 K with a strain rate of 2×10^{-4} s⁻¹. During the deformation process, the strips of α phase break into equiaxed structure. This phenomenon can be attributed to a particular dynamic recrystallization, which suggests that the β phase can recrystallize in the α phase due to the small misfit degree between α phase and β phase.

1. Introduction

Magnesium alloy is one of the lightest metallic engineering materials, and it has promising potential applications in the fields of aerospace, automotive and portable electronic devices. When Li content is 5.7-10.3 wt.%, Mg-Li alloy is composed of two phases, namely α phase and β phase. The duplex Mg-Li alloy possesses good comprehensive mechanical properties with a good plasticity and a moderate strength [1]. Therefore, the duplex Mg-Li alloy is suitable to be used to fabricate some complicated structures through superplastic forming process [2].

The superplasticity of duplex Mg-Li alloys has been researched by many researchers. The duplex Mg-8Li alloy possesses an elongation of 960% at 573K with a strain rate of 5×10^{-4} s⁻¹, and the dominant deformation mechanism is the grain boundary sliding accommodated by the controlled lattice diffusion [3,4]. Kawasaki et. al. [5] researched the flow and cavitation behavior of duplex Mg-9.5Li-1.0Zn alloy in quasi-superplasticity deformation. In our previous research, the superplasticity deformation mechanism of the duplex Mg-8Li-2Zn alloy is grain boundary sliding controlled by grain boundary diffusion [6,7].

Presently, many Mg-Li alloys contain Al due to the good solution strengthening effect of Al in Mg-Li alloys [8]. However, the superplasticity of duplex Mg-Li alloy containing Al is seldom researched. In this paper, the mechanical properties and microstructure evolution of the Mg-9Li-1Al alloy during superplastic tensile testing are researched. A particular dynamic recrystallization mechanism is found.

2. Experimental procedure

The materials used to prepare Mg-Li alloy were commercially pure Mg, Li and Al. The melting and casting processes of Mg-9Li-1Al alloy were conducted under the protection of argon atmosphere in a



median-frequency induction furnace. The detailed processing can be known from our previous literature [9]. The obtained ingot was homogenized at 300°C for 48 hours. Then it was extruded. The temperature during the extrusion is 573 K. The extrusion ratio is 32.11, from a diameter of 85 mm to a diameter of 15 mm.

The microstructure of the sample was observed with optical microscope. XRD pattern of the sample was measured with X-ray diffractometer. The superplastic tensile testing was performed with electronic universal testing machine. The gauge size of the tensile sample was 5 mm × 3 mm with a thickness of 2 mm. The superplastic tensile testing was conducted at the temperature of 423 K-623 K with the strain rate of 2×10^{-4} - $1 \times 10^{-2} \text{ s}^{-1}$.

3. Results and Discussion

The XRD pattern of the Mg-9Li-1Al alloy is shown in Fig.1. The alloy is composed of two phases, α phase and β phase. In the microstructure of the as-cast specimen, Fig. 2(a), the white part is α phase, and the black part is β phase [10]. The α phase in the as-cast specimen disperses within β phase with the shape of polygon. The volume fraction of α phase is obviously less than that of β phase. After extrusion, the α phase exists in the form of elongated strips along the extrusion direction, shown as Fig.2 (b).

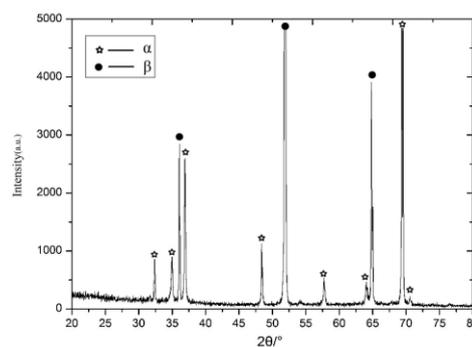


Fig. 1 The XRD pattern of Mg-9Li-1Al alloy.

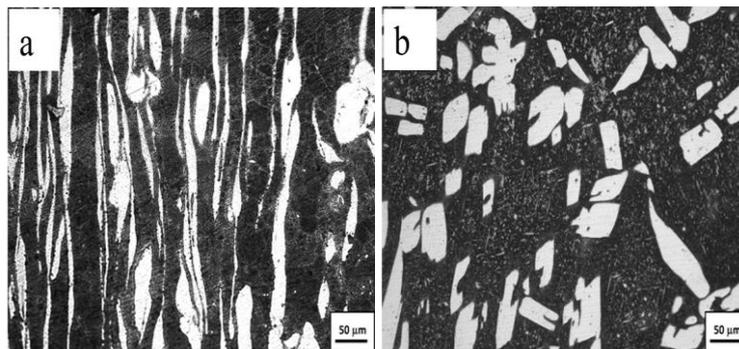


Fig. 2 Microstructure of Mg-9Li-1Al alloy (a) as-cast, (b) as-extruded.

The superplastic tensile elongation values of the as-cast and as-extruded Mg-9Li-1Al alloys are listed in Table 1. The as-cast Mg-9Li-1Al alloy shows a somewhat ability of super plasticity, 153-213%. The super plasticity ability is significantly improved after extrusion, 288-510%. The extruded specimen possesses a much larger elongation with a lower temperature and a larger strain rate.

After superplastic tensile testing (573 K , $1.0 \times 10^{-3} \text{ s}^{-1}$), the microstructural evolution of the as-extruded Mg-9Li-1Al with different distances from the fracture places is shown in Fig.3. The characteristic of the original elongated strips before tensile testing cannot be observed any longer. It

shows that, the long strip of α phase breaks into equiaxed structure, and it distributes randomly within the β phase. There is almost no difference between Fig. 3 (a) and Fig.3 (b), indicating that a uniform microstructure exists along the tensile direction in the specimen after superplastic tensile testing.

Table 1. Superplastic tensile elongation of as-cast and as-extruded Mg-9Li-1Al alloys.

| As-cast | | | As-extruded | | |
|------------------|---------------------------|-----------------|------------------|---------------------------|-----------------|
| Temperature/ (K) | Strain rate/ (s^{-1}) | Elongation/ (%) | Temperature/ (K) | Strain rate/ (s^{-1}) | Elongation/ (%) |
| 573 | 2×10^{-4} | 153 | 573 | 1×10^{-3} | 446 |
| 623 | 2×10^{-4} | 213 | 573 | 2×10^{-4} | 510 |
| 573 | 1×10^{-2} | 288 | 523 | 2×10^{-4} | 288 |

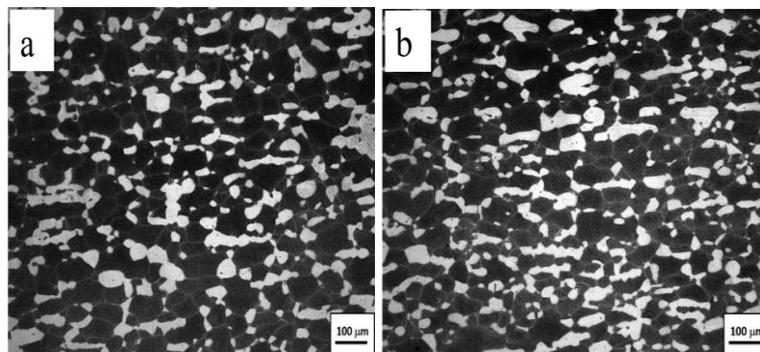


Fig. 3 Microstructural evolution of the as-extruded Mg-9Li-1Al with different distances from the fracture places (573 K, $1.0 \times 10^{-3} s^{-1}$) (a) 0 mm, (b) 7 mm.

In order to research the microstructural evolution during superplastic tensile process of the as-extruded Mg-9Li-1Al, the microstructure of the sample at different stages during the tensile test was observed, shown as Fig.4. The samples were taken from the tensile testing machine at different tensile stages and quickly quenched in cold water. At the stage of the stress rising, the α phase keeps the shape of strip, but the boundaries between α and β become wave-like from straight, and some of them become shape of beads, shown as Fig.4(a). At this stage, recrystallization fully happens in the β phase, most of the grains grow to fully occupy the space between the two elongated strips of α phase. Accordingly, the strips of α phase severely restrict the grain size of β phase. When the deformation reaches the stage of largest stress, α phase begins to fracture, and some β phases with a small size appear within the α phase, shown as Fig. 4(b). When the deformation proceeds to a steady-state, α phase entirely fractures into little polygons with a size of 10 microns or so, and the α phase is gradually scattered by the β phase, shown as Fig.4(c). This process lasts until the final fracture of specimen, shown as Fig.3. Finally, most of the α phases distribute uniformly within the β phase.

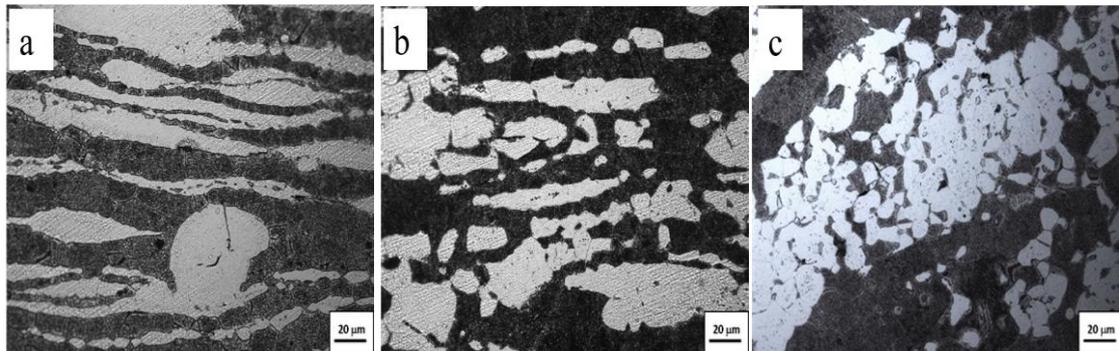


Fig. 4 Microstructure of the as-extruded Mg-9Li-1Al alloy at different superplastic deformation stages (573 K, $1.0 \times 10^{-3} \text{s}^{-1}$): (a) the stage of stress rising, (b) the stage of largest stress, (c) the stage of stable stress.

From the microstructure of the specimen during superplastic tensile testing, it can be observed that some β grains nucleate within the α phase through dynamic recrystallization, shown as Fig.5 (a) (b) (c). This kind of dynamic recrystallization causes the fracture and refining of α phase during deformation.

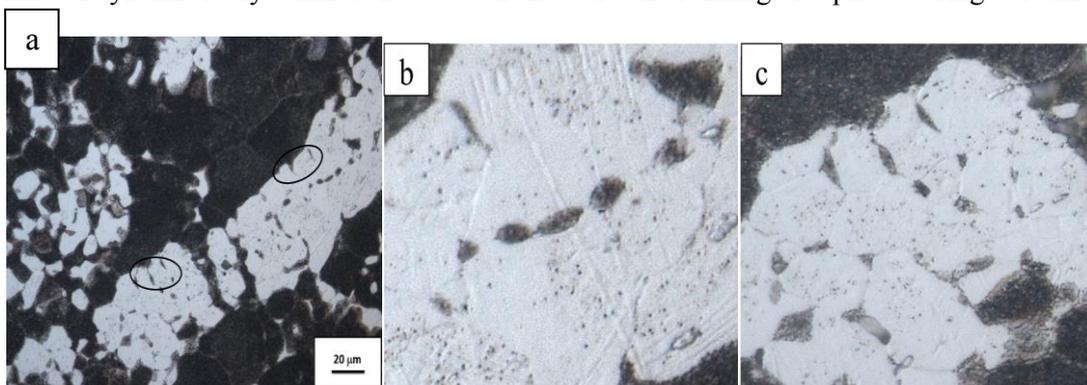


Fig. 5 Microstructure of specimen during superplastic tensile indicating the β nucleation within α phase through dynamic recrystallization.

α phase and β phase are different solid solution. According to the Mg-Li alloy phase diagram, there is almost no change in solid solubility at 573K. Therefore, there is no re-absorption phenomenon because of the composition difference of α phase and β phase. Strain can be gradually accumulated during thermal deformation process. Dynamic recrystallization will occur in the grain boundary under the action of heat activation and enough strain.

Recrystallization process generally cannot occur within a different phase because a relative large value of distortion energy usually exists at the interface between different phases. Therefore, to judge whether the β grain can nucleate in α phase through recrystallization, the critical factor is the energy state at the interface between the two phases. The interfacial energy mainly depends on the lattice matching degree at the phase boundary, namely interface mismatch.

The atomic phase radius refers to the atomic distance of α phase and β phase in the direction of paralleling to interface orientation, respectively. We measured the lattice parameters by X-ray diffraction data. We used the software to obtain the lattice parameters by fitting the unimodal of diffraction pattern with the least square method by MDI jade 6.0. Theoretically, the fitting results of different unimodal of the same phase in the same pattern should be completely equivalent. To reduce the errors of data collection and fitting process, we use the average data of different peaks as the final data. In the lattice of α phase: $a=0.322 \text{ nm}$, $c=0.520 \text{ nm}$; In the lattice of β phase: $a=0.352 \text{ nm}$. Then, the atomic radius of the α phase and β phase can be calculated separately through the lattice parameter

data with formula $r_{\alpha}=\frac{a}{2}$ and $r_{\beta}=\sqrt{\frac{3}{4}}\frac{a}{4}$. α phase: $r=0.160$ nm, β phase: $r=0.152$ nm. According to the definition of mismatch, $\delta=(d_1-d_2)/d_1$, d_1 and d_2 represent the atomic distance in the direction of paralleling to interface orientation. When fully coherent and fully incoherent, the value of δ is 0 and 1, respectively. When $\delta < 0.05$, a coherent interface can be formed. When $0.05 < \delta < 0.25$, there is the tendency to form semi-coherent interface, and when $\delta > 0.25$, an incoherent interface is generally formed^[11,12]. Assuming that the exposed external interface of the phase should be close-packed plane to reduce the interface energy^[13]. Considering that the orientation the two phase grains are almost always distributed along the tensile direction during the tensile testing, for the close-packed plane of hcp and bcc, the d_1 and d_2 will equal to the values of atomic radius of the two phases. Based on the values calculated above, the mismatch between the two phases can be obtained as: $\delta=0.0463$, which indicates that the two phases can form coherent interface. The interfacial energy of coherent interface is very low. This provides the energy basis to achieve the aforementioned recrystallization.

The dynamic nucleation process of dynamic recrystallization is mainly based on the classical liquid phase and nonuniform nucleation theory of solid phase. Although the coherent interface can greatly reduce the energy of nucleation, but the basic conditions of nucleation required are still necessary, that are, energy condition and composition condition.

Firstly, Considering the difference of gibbs free energy, we calculated $\Delta G(\beta \rightarrow \alpha) > 0$ and $\Delta G(\alpha \rightarrow \beta) < 0$ at the temperature of 573K. It can be said that the transformation from α -phase to β phase is a spontaneous process from the perspective of free energy at the temperature of 573K.

Distortion energy is the main source of energy during the recrystallization process from the perspective of energy condition. Distortion energy can be easily accumulated at grain boundaries during deformation process. For duplex Mg-Li alloy, distortion energy is more easily accumulated at the α -phase boundary because of the poor deformation ability. Therefore, as shown in Fig. (a) (b), a large number of new β phase grains were nucleated at the β grain boundary.

From the component condition, the difference between the α phase and the β phase is also the main obstacle that the nucleation needs to overcome. The study shows *that* [14] the lattice diffusion coefficient of β phase is more than 50 times to α phase because of the migration of Li atoms. It can be approximated that the high mobility of lithium atoms *remains* the “flowing state” in high temperature environment. The state provides the mechanism of the components to meet the condition required for nucleation similar to the nucleation process of liquid metal solidification.

4. Conclusion

The as-cast alloy possesses certain superplastic property, and it will be improved after extrusion significantly. The largest elongation of the as-extruded Mg-9Li-1Al alloy is 510%. During the superplastic tensile testing, the strip shape of α phase breaks into equiaxed microstructure. The phase boundary between α phase and β phase is coherent due to the low misfit degree. Therefore, the β phase grains can nucleate in the α phase through dynamic recrystallization, causing the break of α phase.

References

- [1] R. Z. Wu, Y. D. Yan, G. X. Wang, L. E. Murr, W. Han, Z. Zhang, M. L. Zhang, Recent progress in magnesium-lithium alloys, *Inter. Mater. Rev.* 60 (2015) 65-100.
- [2] M. Furui, H. Kitamura, H. Anada, Influence of preliminary extrusion conditions on the superplastic properties of a magnesium alloy processed by ECAP, *Acta Mater.* 55 (2007) 1083-1091.
- [3] F. R. Cao, H. Ding, Y. L. Li, The superplasticity, microstructure evolution and deformation mechanism of duplex superlight Mg-Li alloy, *Chin. J. Nonferr. Metals.* 19 (2009) 1908-1916.
- [4] X.J. Wang, D.K. Xu, R.Z. Wu, What is going on in magnesium alloys, *J. Mater. Sci. Tech.* Doi: 10.1016/j.jmst.2017.07.019.
- [5] M. Kawasaki, K. Kubota, K. Higashi, T. G. Langdon, Flow and cavitation in a quasi-superplastic two-phase magnesium–lithium alloy, *Mater. Sci. Eng. A.* 429 (2006) 334-340.
- [6] X. H. Liu, G. J. Du, R. Z. Wu, Z. Y. Niu, M. L. Zhang, Deformation and microstructure

- evolution of a high strain rate superplastic Mg–Li–Zn alloy, *J. Alloys. Compd.* 509 (2011) 9558-9561.
- [7] X. H. Liu, R. Z. Wu, Z. Y. Niu, J. H. Zhang, M. L. Zhang, Superplasticity at elevated temperature of an Mg–8%Li–2%Zn alloy, *J. Alloys. Compd.*, 541 (2012) 372-375.
- [8] T. C. Xu, X. D. Peng, J. W. Jiang, G. B. Wei, B. Zhang, Microstructure and Mechanical Properties of Superlight Mg-Li-Al-Zn Wrought Alloy, *Rare. Metal. Mat. Eng.* 43 (2014) 1815-1820.
- [9] T. Z. Wang, T. L. Zhu, J. F. Sun, R. Z. Wu, Influence of rolling directions on microstructure, mechanical properties and anisotropy of Mg-5Li-1Al, *J. Magnes. Alloy.* 3 (2015) 345-351.
- [10] Z. K. Qu, X. H. Liu, R. Z. Wu, M. L. Zhang, The superplastic property of the as-extruded Mg–8Li alloy, *Mater. Sci. Eng. A.* 527 (2010) 3284-3287.
- [11] P. A. Pluchino, X. Chen, M. Garcia, L. M. Xiong, D. L. McDowell, Y. P. Chen, Dislocation migration across coherent phase interfaces in SiGe superlattices, *Comp. Mat. Sci.*, 111 (2016) 1-6.
- [12] R. Dingreville, A. Hallil and S. Berbenni, From coherent to incoherent mismatched interfaces: A generalized continuum formulation of surface stresses, *J. Mech. Phys. Solids.* 72 (2014) 40-60.
- [13] T. O. Owolabi, K. O. Akande, Estimation of surface energies of hexagonal close packed metals using computational intelligence technique, *Appl. Soft. Comput.* 31 (2015) 360-368.
- [14] F. R. Cao, H. Ding, Y. L. Li, The superplasticity, microstructure evolution and deformation mechanism of duplex superlight MgLi alloy, *Chin. J. Nonferr. Metals.* 19 (2009) 1908-1916.