## 0.100//3113/3-016-1002-0

# Effect of Extrusion Ratio on the Microstructure and Mechanical Properties in an Al-Cu-Mg-Ag Alloy

XU Xiaofeng, ZHAO Yuguang\*, ZHANG Ming, NING Yuheng, WANG Xudong

(Key Laboratory of Automobile Materials, Ministry of Education, and Department of Materials Science and Engineering, Jilin University, Changchun 130025, China)

**Abstract:** In order to examine the effect of extrusion ratio on the microstructure and mechanical behavior in Al-Cu-Mg-Ag alloy, the Al-6.3Cu-0.48Mg-0.4Ag alloy was subjected to extruding with different extrusion ratios of 17, 30 and 67. The results indicate that the grains are refined and the strength is improved effectively with increasing extrusion ratio. However, further investigation shows that the extrusion ratio of 30 is more effective than the lower extrusion ratio (17) and the higher extrusion ratio (67) to refine the grains in the T6-temper alloy. Moreover, the sample with an extrusion ratio of 30 obtains more precipitates and superior mechanical properties after T6 treatment. This study supports the idea that there exists a critical extrusion ratio for grain refinement and improvement of mechanical properties for the T6-temper alloy. Recrystallization and precipitation during T6 treatment were introduced to explain the effects of extrusion ratio on the microstructure and mechanical properties of the Al-Cu-Mg-Ag alloys.

Key words: Al alloy; extrusion ratio; grain refinement; precipitation; microstructure

## **1** Introduction

It is well known that trace additions of Ag to Al-Cu-Mg alloys with high Cu:Mg ratios can accelerate the age hardening reaction and change the precipitation behavior<sup>[1,2]</sup>. The Ag-contained Al-Cu-Mg alloys facilitate the formation of the plate-like  $\Omega$  phase on the {111} planes of the matrix<sup>[3]</sup>. Due to the fine and uniformly distributed precipitate  $\Omega$ , the alloys exhibit excellent mechanical properties and creep resistance at elevated temperature<sup>[2,4]</sup>. Therefore, the Al-Cu-Mg-Ag alloy is recognized as the potential material for the aerospace applications and attracts considerable commercial interest<sup>[5]</sup>.

Severe plastic deformation (SPD) processing is effective for the production of materials with ultrafine grains<sup>[6-8]</sup>. In the past decades, various SPD methods

have been used to obtain the microstructural refinement in various alloys<sup>[6]</sup>. However, as is known, the ductility of the SPD materials is usually inadequate for practical applications<sup>[9]</sup>. For the age-hardenable high-strength Al alloys, a significant increase in strength and proper ductility can be achieved through a combination of deformation and precipitation hardening<sup>[10-12]</sup>.

As is known, the  $\Omega$  phase is more effective to strengthen the alloys and has the lower thickening rate compared with  $\theta$  phase in the Al-Cu-Mg-Ag alloys. Moreover, the precipitation of the two phases are competitive. It has been proved that the dislocations promote the precipitation of  $\theta$  phase while restrain that of  $\Omega$  phase<sup>[13]</sup>. Generally, the solution treatment consumes lots of dislocations and makes the recrystallized structure, which is beneficial for the precipitation of the  $\Omega$  phase. Therefore, the Al-Cu-Mg-Ag alloys show super strength and excellent creep resistance in the T6 heat treatment (solid solution and artificial aging). However, the long-time elevated temperature has a negative effect on the deformation strengthening. It is well known that grain refinement strengthening is recognized as an effective way to improve both the strength and ductility. As a result, the deformation process also plays an important role in strengthening the alloys<sup>[6]</sup>. In the Al-Cu-Mg-Ag alloy, the hot extruding is the common method. However, the

<sup>©</sup> Wuhan University of Technology and Springer-Verlag GmbH Germany, Part of Springer Nature 2018

<sup>(</sup>Received: Dec. 21, 2016; Accepted: Feb. 25, 2018)

XU Xiaofeng (徐晓峰): Ph D; E-mail: xuxiaofeng@jlu.edu.cn \*Corresponding author: ZHAO Yuguang (赵字光): Prof.; Ph D; E-mail: zhaoyg@jlu.edu.cn

Funded by the National Natural Science Foundation of China (No.51071075) and the Deep Continental Scientific Drilling Equipment Development (SinoProbe-09-05), as well as by the 985 Project-Automotive Engineering of Jilin University

effect of the deforming degree on the microstructure and mechanical properties of Al-Cu-Mg-Ag alloy has been little reported in the previous studies. In this paper, the effects of extrusion ratio on the microstructure and mechanical behavior of Al-Cu-Mg-Ag alloy will be investigated.

## 2 Experimental

The experimental material Al-Cu-Mg-Ag alloy was prepared with pure Al, pure Cu, pure Mg, pure Ag and Al-5Zr, Al-15Mn, Al-5Ti-B master alloy by ingot melting in a crucible furnace. Table 1 shows the nominal chemical composition of the present alloy. After casting, the ingots were homogenized at 500 °C for 24 h, scalped to 80 mm and then extruded to various-diameter rods with the extrusion ratio of 17, 30 and 67 at about 400 °C, then cooled in the air. To obtain the optimized solution temperature, the dissolving temperature of secondary phases of the extruding samples was determined using differential scanning calorimetry (DSC). All the measurements were done at heating rate of 10 °C/min.

Table 1 Nominal compositions of the alloy investigated/wt%

Cu Mg	Ag	Zr	Mn	Ti	Fe	Si	Al
6.3 0.48	0.4	0.2	0.3	< 0.1	< 0.1	< 0.05	Bal.

The tensile specimens were got parallel to the extruding direction. The tensile tests were conducted at the strain rate of  $10^{-3}$ s<sup>-1</sup> at room temperature. Specimens ground and polished for tensile testing were dog-bone shaped sheets with gauge length 30 mm, and cross section 4×2 mm. At least 3 specimens were tested for each condition. The specimens for transmission electron microscopic (TEM) observation were prepared by the standard twin-jet electropolishing method with a voltage of 10-15 V in 80% ethanol and 20% perchloric acid at -30 °C. The TEM observations were carried out on a JEM-2100F and operated at 200 kV.

## **3 Results and discussion**

#### **3.1 DSC**

Fig.1 presents the DSC traces of the alloys after hot extruding. Two exothermal peaks are apparent in each DSC thermogram, which indicates that two different reactions occur during the heating process. The peak temperatures correspond to the dissolving temperature of secondary phase and the melting temperature of the alloys. It can be seen that the extrusion ratio has few effects on the dissolving temperature. On account of the dissolution temperature of second-phase (Fig.1) and avoiding overburning the alloy, the samples will be solid solution treated at 520 °C. To ensure the secondary phase having enough time to dissolve in the matrix, the solid solution treated time was confirmed as 2 h. Hence, the samples were solution-treated at 520 °C for 2 h, water-quenched and then immediately aged at 185 °C for 4 h.



Fig.1 DSC curves of extruded Al-Cu-Mg-Ag alloy with different extrusion ratios

### **3.2 Mechanical properties at room temper**ature

The representative engineering tensile stressstrain curves of the extruded Al-Cu-Mg-Ag alloy for different extrusion ratios are presented in Fig.2(a). The ultimate tensile strength (UTS) of the alloy with



Fig.2 Typical engineering stress-strain curves of : (a) extruded alloys with various extrusion ratios; (b) T6-temper alloys with different extrusion ratios

extrusion ratio of 17 is 334 MPa, which is 50 MPa lower than that of the alloy with an extrusion ratio of 67, and it is clear that the yield strength (YS) and UTS increase with increasing extrusion ratio, while the elongation shows the different condition, which is the alloy with an extrusion ratio of 30 having the best elongation (24.7%). Fig.2(b) demonstrates the curves of the mechanical properties of the T6-temper alloys with different extrusion ratios. In the T6 condition, the alloy with the extrusion ratio of 30 presents the better mechanical properties (YS, UTS and the elongation to failure are improved to 342 MPa, 542 MPa and 22%) compared with the other samples, and the further improved extrusion ratio does not make the strength increase continuously. These phenomena will be explained by the corresponding change in microstructure.

#### **3.3 Microstructure**

Figs.3(a), (b) and (c) show the influence of extrusion ratio on the optical microstructure of the extruded alloys. It is easily understood that the larger degree of deformation makes the alloy more compact and finer fiber structures with increasing extrusion ratio, which will be beneficial for both the strength and elongation of the alloys. However, when the extrusion ratio exceeds the critical value, the strength of the alloy increases and the elongation decreases, which is typical worked metals, so the alloy with extrusion ratio of 67 has the worse elongation compared with the alloy with extrusion ratio of 30. Meanwhile, the higher extrusion ratio makes the secondary phases distribute more uniformly, which is beneficial for the secondphase dissolving in the matrix during heat treatment. In addition, it should be noted that the large plastic deformation can also make parts of the secondary

phases dissolve in the matrix during extruding<sup>[14]</sup>, and the heat induced by deformation can also accelerate the second-phase dissolution, which may also have contribution to the improvement of the mechanical properties.

Figs.3(d), (e) and (f) show the optical microstructures of the aged Al-Cu-Mg-Ag alloys in different conditions. The alloy with extrusion ratio of 30 has the finer grains compared with the other samples, and there are slightly fewer secondary phases in the alloy with higher extrusion ratio, which is beneficial for the elongation of the alloy. Regarding the strengthening mechanism of the materials, the strengthening effect usually is described by Hall-Petch equation:  $\sigma_s = \sigma_0 + \kappa d^{-1/2}$  where  $\sigma_s$  is the yield strength,  $\sigma_0$  reflects the intragranular resistance to deformation, K is a constant that evaluates the impact of grain boundaries on deformation and d is the grain diameter<sup>[15]</sup>. In addition, it is well known that the finer grains are also beneficial for the elongation of the alloy, which has contribution to the superior mechanical properties of the alloy with an extrusion ratio of 30. Meanwhile, it can be seen that recovery and recrystallization occur in the alloys, and parts of grains are still elongated along the extruding direction, as shown in Figs.3(d), (e) and (f). Generally, the larger extrusion ratio means that finer fiber structure will be produced, as shown in Figs.3(a), (b) and (c). However, the larger deformation makes the more rapid recovery and recrystallization at high temperature, and the grain growth rate will also be accelerated<sup>[16]</sup>. As a result, it is shown that the alloy with an extrusion ratio of 67 has larger grain size than the alloy with an extrusion ratio of 30, implying that the finer T6-temper structure will not always be promoted by the larger deformation.



Fig.3 Optical microstructures of the extruded Al-Cu-Mg-Ag alloys with extrusion ratio of (a) 17, (b) 30, and (c) 67; optical microstructures of the solution treated alloys with extrusion ratio of (d) 17, (e) 30, and (f) 67



Fig.4 Bright field TEM micrographs showing the microstructures with different extrusion ratios of (a) 17, (b) 30, and (c) 67 in T6 conditions, (d) the high resolution TEM (HRTEM) and fast fourier transformation (FFT) of the typical  $\Omega$ phase in the alloy with an extrusion ratio of 17

The TEM micrographs of the aged Al-Cu-Mg-Ag alloy under different conditions are shown in Fig.4. A uniform dispersion of fine and nano-scale precipitates was detected. The primary secondary phases are plate-like  $\Omega$  phase. For the plate-like  $\Omega$  phase on the {111} planes, the increase in strength can be given by the rela-

tion<sup>[17]</sup>:  $\Delta \sigma = \frac{\Gamma}{r} \ln \left( \frac{0.158r}{r_0} \right)$ , where  $\Delta \sigma$  is the strength increase, *r* is the radius of the plate-plane,  $r_0$  is the dislocations by passing radius,  $\Gamma$  is the parameter only concerning the volume fraction of the precipitation. In the same alloy system, the higher  $\Gamma$  and the smaller r make the larger  $\Delta \sigma$ . As shown in Fig.4, it can be seen that the number of the secondary phases in the alloy with an extrusion ratio of 30 is larger compared with the other two samples. After extruding and SST, the solute elements distribute uniformly, as the above discussion, and the severe deformation can promote the dissolution of the residual phases<sup>[14,18]</sup>. On one hand, the process makes more solute dissolve in the matrix (the number of secondary phases decreases with increasing extrusion ratio in Figs.3(d), (e) and (f)); On the other hand, the larger degree of dissolution will accelerate precipitation and reduce the effect of the residual phase on precipitation (the residual phase can serve as the core of the precipitates and weaken the precipitation strengthening). Besides, during artificial aging, the finer grains make the solute element distribute more uniformly, which plays a positive role in precipitation.

Moreover, during artificial aging, the finer grains provide more grain boundaries to constrain the motion of the solute, which is beneficial for hindering the growth of the precipitates<sup>[19,20]</sup>. Based on the disscusion above, the alloy with an extrusion ratio of 30 has the superior mechanical properties. Meanwhile, it should be noted that the large number of uniformly distributed precipitates will be the obstacle to the motion of the dislocations during deformation, which may result in the slightly decreased elongation of the alloy with an extrusion ratio of 30 compared with that of higher extrusion ratio alloy.

## **4** Conclusions

a) The A1-6.3Cu-0.48Mg-0.4Ag alloy was subjected to different extrusion ratios of 17, 30 and 67 at about 400  $^{\circ}$ C. With increasing extrusion ratio, the size of the fibrous structure decreases and the strength increases, but the elongation decreases when the extrusion ratio reaches 67.

b) After T6 treatment, the sample with an extrusion ratio of 30 has the superior mechanical properties compared with the other specimens, which is attributed to the finer grains and the more precipitates. The excessive deformation accelerates the recrystallization and grain growth during heat-treatment.

c) There exists a critical extrusion ratio for improving the mechanical properties in the T6-temper Al-Cu-Mg-Ag alloy. When the extrusion ratio is higher than the critical value, it has a negative effect on the mechanical properties of the T6-temper alloy.

#### References

- Hou Y H, G u YX, Liu Z Y, *et al.* Modeling of Whole Process of Ageing Precipitation and Strengthening in Al-Cu-Mg-Ag Alloys with High Cu-to-Mg Mass Ratio[J]. *Trans. Nonferrous Met. Soc. China*, 2010, 20 (5): 863-869
- [2] Lumley R N, Morton A J, Polmear I J. Enhanced Creep Performance in An Al-Cu-Mg-Ag Alloy through Underageing[J]. *Acta Mater.*, 2002, 50 (14): 3 597-3 608
- [3] Hutchinson C R, Fan X, Pennycook S J, et al. On the Origin of the High Coarsening Resistance of Omega Plates in Al-Cu-Mg-Ag Alloys[J]. Acta Mater., 2001, 49 (14): 2 827-2 841
- [4] Skrotzki B, Shiflet G, Starke E. On The Effect of Stress on Nucleation and Growth of Precipitates in an Al-Cu-Mg-Ag Alloy[J]. *Metall. Mater. Trans. A*, 1996, 27 (11): 3 431-3 444
- [5] Polmear I J, Pons G, Barbaux Y, et al. After Concorde: Evaluation of Creep Resistant Al-Cu-Mg-Ag Alloys[J]. Mater. Sci. Technol., 1999, 15 (8): 861-868

- [6] Estrin Y, Vinogradov A. Extreme Grain Refinement by Severe Plastic Deformation: A Wealth of Challenging Science[J]. *Acta Mater.*, 2013, 61 (3): 782-817
- [7] Orlov D, Raab G, Lamark T T, et al. Improvement of Mechanical Properties of Magnesium Alloy ZK60 by Integrated Extrusion and Equal Channel Angular Pressing[J]. Acta Mater., 2011, 59 (1): 375-385
- [8] Cheng S, Zhao Y H, Zhu Y T, et al. Optimizing the Strength and Ductility of Fine Structured 2024 Al Alloy by Nano-precipitation[J]. Acta Mater., 2007, 55 (17): 5 822-5 832
- [9] Zhao Y L, Yang Z Q, Zhang Z, et al. Double-peak Age Strengthening of Cold-worked 2024 Aluminum Alloy[J]. Acta Mater., 2013, 61 (5): 1 624-1 638
- [10] Kim J K, Jeong H G, Hong S I, et al. Effect of Aging Treatment on Heavily Deformed Microstructure of A 6061 Aluminum Alloy after Equal Channel Angular Pressing[J]. Scripta Mater., 2001, 45 (8): 901-907
- Wang Z C, Prangnell P B. Microstructure Refinement and Mechanical Properties of Severely Deformed Al-Mg-Li Alloys[J]. *Mater. Sci. Eng. A*, 2002, 328 (1): 87-97
- [12] Shaeri M H, Salehi M T, Seyyedein S H, et al. Microstructure and Mechanical Properties of Al-7075 Alloy Processed by Equal Channel Angular Pressing Combined with Aging Treatment[J]. Mater. Des., 2014,

57: 250-257

- [13] Liu X Y, Pan Q L, Zhang X L, et al. Effects of Stress-aging on The Microstructure and Properties of An Aging Forming Al-Cu-Mg-Ag Alloy[J]. Mater. Des., 2014, 58: 247-251
- [14] Sha G, Wang Y B, Liao X Z, et al. Influence of Equal-channel Angular Pressing on Precipitation in an Al-Zn-Mg-Cu Alloy[J]. Acta Mater., 2009, 57 (10): 3 123-3 132
- [15] Barnett M R. A Rationale for the Strong Dependence of Mechanical Twinning on Grain Size[J]. Scripta Mater., 2008, 59 (7): 696-698
- [16] Rofman O V, Bate P S. Dynamic Grain Growth and Particle Coarsening in Al-3.5Cu[J]. Acta Mater., 2010, 58 (7): 2 527-2 534
- [17] Zhang G J, Liu G, Ding X D, et al. Experiment and Modeling Study of Aged Aluminium Alloys Strengthening Response[J]. Acta Metall. Sinica, 2003, 39 (8): 803-808
- [18] Liddicoat P V, Liao X Z, Zhao Y, et al. Nanostructural Hierarchy Increases the Strength of Aluminium Alloys[J]. Nat. Commun., 2010, 63 (1): 1-6
- [19] Hu T, Ma K, Topping T D, et al. Precipitation Phenomena in An Ultrafine-grained Al Alloy[J]. Acta Mater., 2013, 61: 2 163-2 178
- [20] Ma K, Wen H, Hu T, et al. Mechanical Behavior and Strengthening Mechanisms in Ultrafine Grain Precipitation-strengthened Aluminum Alloy[J]. Acta Mater., 2014, 62: 141-155