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Effects of solution treatment on microstructure and mechanical properties of Al−9.0Zn−2.8Mg−2.5Cu−0.12Zr−0.03Sc alloy

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Abstract: Effects of additions minor contents of 0.03% Sc and 0.12% Zr and solution treatment on microstructure and mechanical properties of Al−9.0Zn−2.8Mg−2.5Cu alloy were studied by metallographic microscopy, differential thermal analysis (DSC) and transmission electron microscopy (TEM), in order to obtain high-performance Al alloys. The minor additions of Sc (less than 0.1%) were carried out. The results show that with the additions of 0.03% Sc and 0.12% Zr, the petaloid $Al_3(Sc,Zr)$ precipitated phases occur in Al−9.0Zn−2.8Mg−2.5Cu alloy, and $Al_3(Sc,Zr)$ particles obviously hinder the recrystallization of Al−9.0Zn−2.8Mg−2.5Cu alloy during homogenizing and extruding processes due to their strong pinning effect on dislocation. Multi-stage solution is better than single solution, for it can avoid recrystallization of Al−9.0Zn−2.8Mg−2.5Cu alloy with the minor contents of Sc (less than 0.1%). The proper solution treatment is (420 °C, 3 h)+(465 °C, 2 h) under which Al−9.0Zn−2.8Mg−2.5Cu−0.12Zr−0.03Sc alloy obtains a tensile strength of 777.29 MPa and a elongation of 11.84%.

Key words: Al−Zn−Mg−Cu Alloy; solution treatment; structure; mechanical properties

1 Introduction

With the development of aviation industry, the requirements for the performance of 7xxx Al alloy are becoming increasingly high [1, 2]. Aluminum alloy with additions of Sc is considered a new kind of high performance alloys [3]. Sc in these Al alloys exhibits obvious grain refining which serves to improve strength, inhibit recrystallization in Al alloys, strengthen welds and eliminate hot cracking from welds [4−6]. Zr can also inhibit recrystallization in Al alloys [7]. Among these alloys, Al−Zn−Mg−Cu−Zr−Sc alloy has attached much attention in recent years. Nevertheless, researches showed that a refined microstructure and preferable mechanical properties of alloys could be obtained if Sc was up to 0.1% to inhibit recrystallization [8−11].

However, due to its high market price, the more the addition of Sc increases, the higher the cost rises. Thus, the effects of different solution treatments on the structure and mechanical properties of Al−9.0Zn−2.8Mg−2.5Cu−0.12Zr−0.03Sc alloy were investigated in this work, so as to explore whether high-performance alloys with can be obtained with the minor contents of Sc (<1%).

2 Experimental

The compositions of two kinds of alloys are shown in Table 1. The experiment was carried out by preparing alloy ingot, homogenization annealing of (200 °C, 2 h) + (440 °C, 6 h) + (460 °C, 12 h), extruding, solution, quenching and aging at 120 °C for 12 h. The solution treatment conditions are shown in Table 2.

DSC analysis was carried out by a DSC system (MDSC Q100, U.S.) with a heating rate of 10 °C/min. The mechanical properties were tested on SANS CMT−5105 microcomputer controlled electronic universal testing machine. The microstructure of sample was observed by TEM (TECNAI G220 FEI).

Table 1 Compositions of Al alloys (mass fraction, %)

Alloy	Zn	Mg	Cu	Sc	Zr	Al
1	9.0	2.8	2.5			Bal.
2	9.0	2.8	2.5	0.03	0.12	Bal.

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3 Results and discussion

3.1 Structure

Figure 1 offers the microstructures of Alloys 1 and 2 before hot working along the extruding direction. It can be seen that the grains are elongated along the extruding direction, and fibrous structure forms after extruding. The recrystallization of Alloy 1 occurs imperfectly perpendicular to the extruding direction, and its grain is not lathy fibrous, with an average diameter of about 23 μm. Alloy 2 exhibits fibrous grain, with an average diameter of about 4 μm, which indicates that the addition of Sc and Zr can inhibit the recrystallization.

Table 2 Solution treatment for extruding bar of alloy

No.	Solution treatment	Aging treatment
a	465 °C, 1 h	120 °C, 12 h
b	465 °C, 2 h	120 °C, 12 h
c	465 °C, 3 h	120 °C, 12 h
d	(445 °C, 2 h) + (465 °C, 1 h)	120 °C, 12 h
e	(445 °C, 2 h)↗(1 h) + (465 °C, 1 h)	120 °C, 12 h
f	(420 °C, 3 h) + (445 °C, 2 h)↗(1 h) + (465 °C, 1 h)	120 °C, 12 h
g	(420 °C, 6 h) + (465 °C, 2 h)	120 °C, 12 h
h	(420 °C, 3 h) + (465 °C, 3 h)	120 °C, 12 h
i	(420 °C, 3 h) + (465 °C, 2 h)	120 °C, 12 h
j	(200 °C, 3 h) + (420 °C, 3 h) + (465 °C, 2 h)	120 °C, 12 h

Notion: Marks↗show heating with the furnace

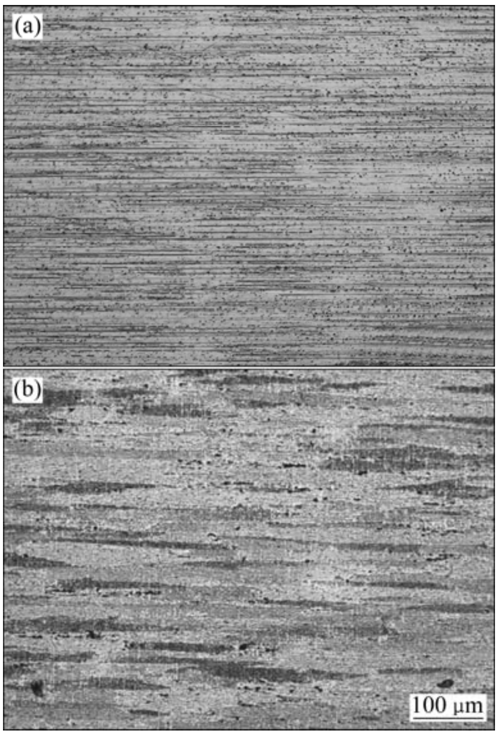

Fig. 1 Microstructures of extruded alloy bars along longitudinal direction: (a) Alloy 1; (b) Alloy 2

Figure 2 shows the TEM micrographs of Alloy 2. It can be seen that the petaloid precipitated phases exhibit fine and distribute dispersedly in α(Al) matrix. They are determined to be Al_3(Sc,Zr) particles through the [111]Al zone axis analysis of selected area electron diffraction (SAED) pattern. They are coherent with the matrix, and exhibit small coherency misfit and large dispersion. They are not easy to be dissolved back, gather and grow up. They always keep the deformation of substructure to a higher temperature due to their good thermal stability, strongly pin the dislocation and grain boundary, in order to hinder the dislocation movement and grain boundary migration, and restrain the recrystallization of Al alloy. As a result, the alloy still keeps its fibroid structure after solution treatment so as to improve its comprehensive mechanical property. Al_3(Sc,Zr) particle has strongly pinning effect on dislocation, as shown in Fig. 2(c).

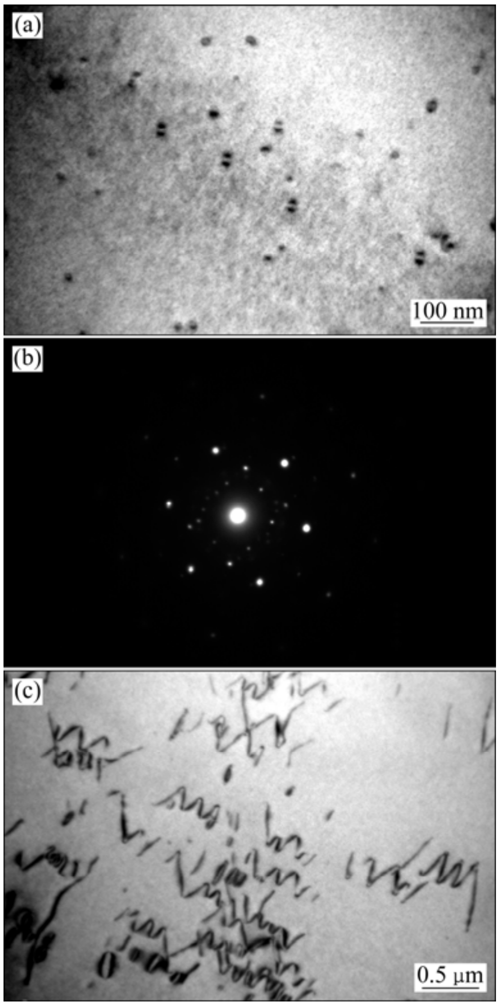

Fig. 2 TEM images of Alloy 2: (a) Bright field image; (b) SAED pattern with [111]Al zone axis; (c) Pinning of the second phase grain on dislocation

3.2 Effect of solution treatment on structure of alloy

Figure 3 reveals the DSC curves of two kinds of as-extruded Al alloys. It can be seen that the endothermic peak of Alloy 1 is bigger than that of Alloy 2, indicating that Alloy 1 has larger number of the second phases than Alloy 2 at a corresponding temperature. Because the endothermic peaks of two alloys start from 479 °C, their overheated temperature can be initially set at about 479 °C. Thus, their solution temperatures are set at less than 475 °C.

Figure 4 shows the metallograph of Alloy 1 after (465 °C, 2 h) + (120 °C, 12 h) treatment. It can be seen that Alloy 1 has fully recrystallized, and thus recrystallization is apt to occur in such alloy if without additions of Sc and Zr.

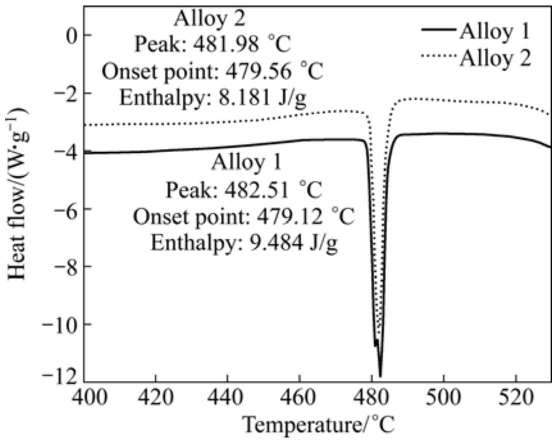

Fig. 3 DSC curves of two kinds of as-extruded alloys

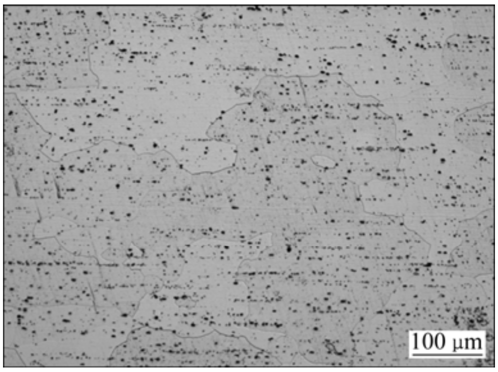

Fig. 4 Microstructures of Alloy 1 after hot working at (465 °C, 2 h) + (120 °C, 12 h)

Figure 5 indicates the longitudinal microstructures of Alloy 2 after the hot working at conditions Nos. a, b and c in Table 2. It is clear that the single solution will cause imperfect recrystallization even if Sc and Zr are added to Alloy 2, and the degree of recrystallization does not vary with the elongation of time.

During multi-stage solution, the first-stage prompts the Mg, Zn, Cu, Zr, Sc and other atoms to distribute finely and uniformly at grain boundary, and then the recrystallized grains are hindered to grow up after the second-stage solution, which avoids the atoms to grow up and recrystallize before the atoms disperse uniformly due to the high temperature during single-solution.

Figure 6 illustrates the microstructures of Alloy 2 after hot working at conditions Nos. h, i and j in Table 2. It can be seen that recrystallization occurs unobviously in Alloy 2 after 2 h-holding at 445 °C. If the temperature rises quickly to 465 °C, recrystallization will occur in Alloy 2. Thus, such processes can be avoided by gradually heating. Pre-treatment at 420 °C will not cause the grain to grow up obviously and will have little effect on the precipitation of the second phase.

Figure 7 shows the microstructures of Alloy 2 after hot working at conditions Nos. g, h, i and j in Table 2 in longitudinal direction. It is clear that recrystallization occurs obviously in Alloy 2 after 6 h-holding at 420 °C and its grains grow up rapidly, whereas in Table 2, there

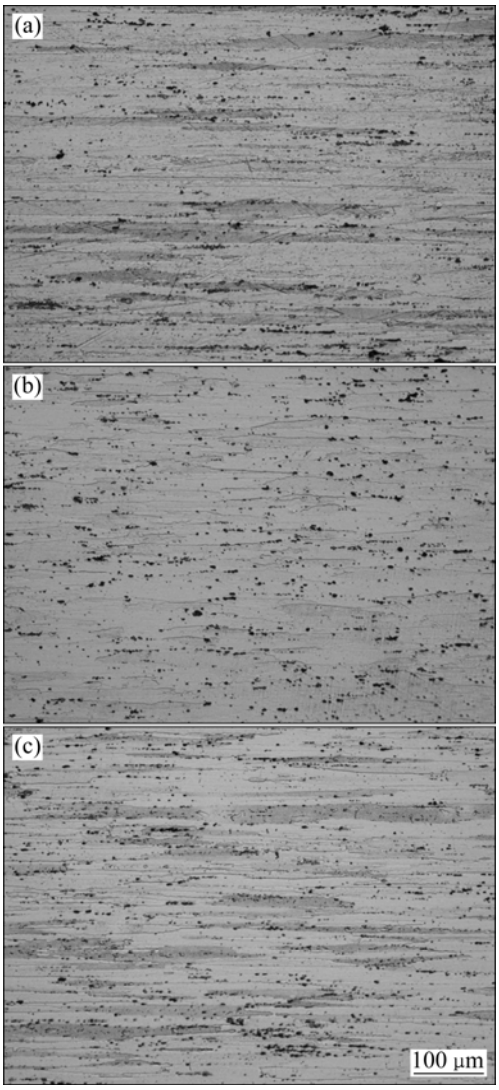

Fig. 5 Microstructures of Alloy 2 after hot working at different conditions: (a) (465 °C, 1 h) + (120 °C, 12 h); (b) (465 °C, 2 h) + (120 °C, 12 h); (c) (465 °C, 3 h) + (120 °C, 12 h)

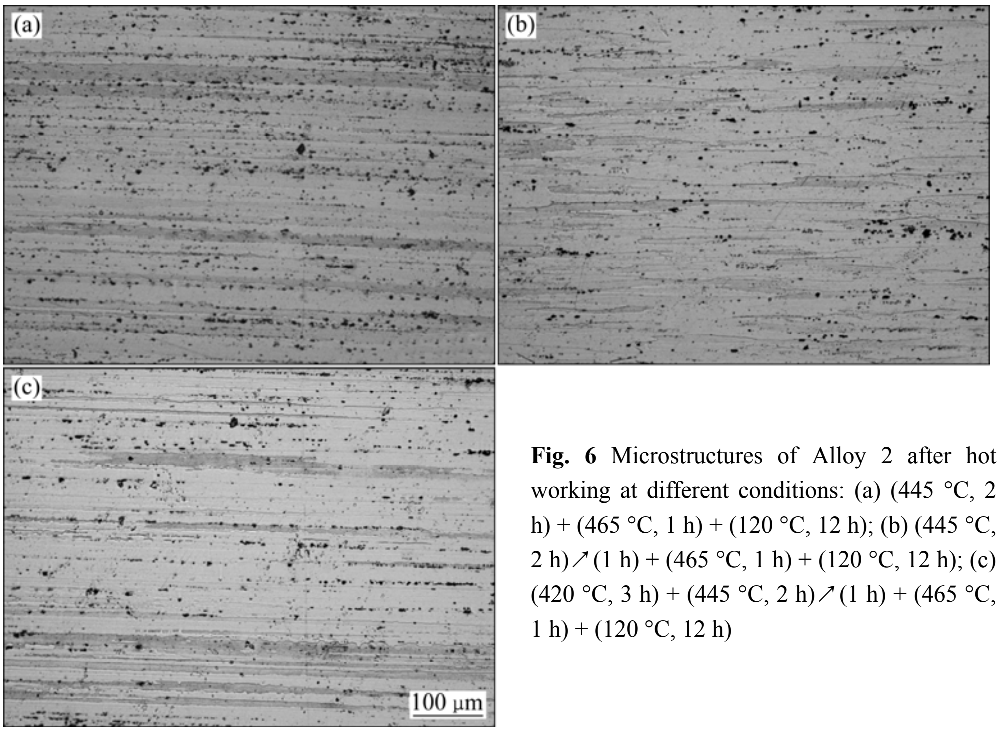

Fig. 6 Microstructures of Alloy 2 after hot working at different conditions: (a) (445 °C, 2 h) + (465 °C, 1 h) + (120 °C, 12 h); (b) (445 °C, 2 h)↗(1 h) + (465 °C, 1 h) + (120 °C, 12 h); (c) (420 °C, 3 h) + (445 °C, 2 h)↗(1 h) + (465 °C, 1 h) + (120 °C, 12 h)

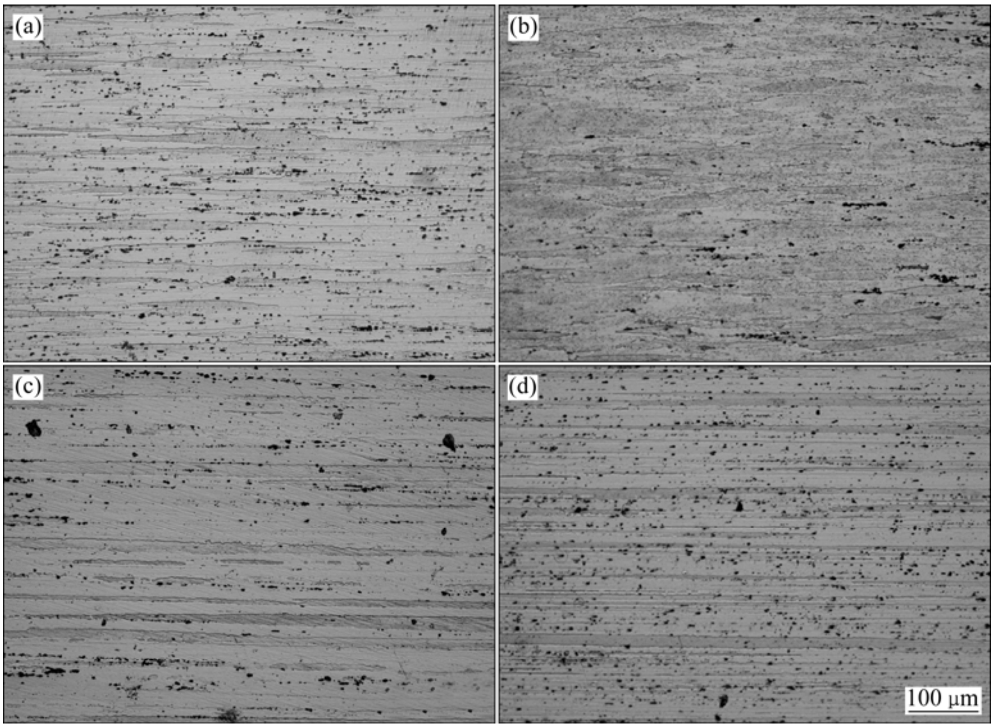

Fig. 7 Microstructures of Alloy 2 after hot working at different conditions: (a) (420 °C, 6 h) + (465 °C, 2 h) + (120 °C, 12 h); (b) (420 °C, 3 h) + (465 °C, 3 h) + (120 °C, 12 h); (c) (420 °C, 3 h) + (465 °C, 2 h) + (120 °C, 12 h); (d) (200 °C, 3 h) + (420 °C, 3 h) + 465 °C, 2 h) + (120 °C, 12 h)

is little difference of hot working between conditions No. g and h, and thus, the microstructures of Alloy 2 under the two hot working conditions are similar, and both present fully recrystallization. The grain size of condition No. e is about 21 μm after a short time of hot working, while that of condition No. f is about 27 μm and there are more precipitated phases in condition No. i. Recrystallization occurs partly in Alloy 2 after treatment at condition No. j in Table 2, and its grains become short significantly.

In the isothermal transversal chart, the solubilities of Sc and Zr below 550 °C are 0.06% and 0.03%, respectively. As the temperature drops, the three-phase zone enlarges and two-phase zone reduces, and while the contents of Sc and Zr are 0.03% and 0.12%, respectively, in Alloy 2. Thus, the temperature for $Al_3(Sc,Zr)$ particles to precipitate is 420 °C, which basically accords with the results in Ref. [12] that $Al_3(Sc,Zr)$ was formed in the homogenizing or hot working processes.

While the driving force P for the growth of alloy grain containing dispersed particles can be expressed as follows:

$$P=P_D-P_Z=P_D-(3\varphi\gamma_b)/d \quad (1)$$

where P_D is the driving force caused by the decline of recrystallized grain interfacial energy; P_Z is the Zener resistance caused by the pinning of the second phase particles; φ and d are the volume fraction and diameter of the second phase particles, respectively; γ_b is the interfacial energy of wide angle grain boundary [13].

As reported in Ref. [13], φ is usually enlarged with the increment of additions of Sc (>0.1%), and thereby P_D is less than P_Z, which hinders the recrystallization of alloy but increases its cost severely and then impedes its industrial application.

However, in this work, after pre-treatment at 420 °C, $Al_3(Sc,Zr)$ can precipitate fully from Alloy 2, and the precipitated phase particles of $Al_3(Sc,Zr)$, which are coherent with the matrix, are usually finer, thus the recrystallization does not occur after treatment No. i in Table 2, while when the holding time of Alloy 2 at 420 °C after treatment No. g is too long, and its holding temperatures after treatments Nos. a−d are too high, P_D is greater than P_Z, and therefore, recrystallization is apt to occur.

As a result, multi-stage solution expresses as the equilibrium point between P_D and P_Z when the content of Sc is smaller (<0.1%). When P_D is greater than P_Z, recrystallization occurs. On the contrary, recrystallization does not occur so it can continue to conduct solution treatment prompt $Al_3(Sc,Zr)$ to precipitate and Mg and Zn to dissolve into the matrix, thereby improving the mechanical properties of alloy.

3.3 Effects of solution treatment on mechanical property of alloy

Table 3 indicates the data sheet of mechanical properties of two kinds of alloys after different solution treatments. It is clear that Alloy 1 has a lower strength but higher elongation than Alloy 2 because the dispersed and coherent second phase $Al_3(ScZr)$ particles strongly pin the dislocation and subgrain boundary, resulting in the fine grain strengthening, sub-structure strengthening and dispersion strengthening. This is consistent with the studies of NIE et al [14], LIU et al [15], WANG et al [16], and the time of single solution has litter effect on the mechanical properties.

Pre-treatment is conducted at 445 °C before solution at 465 °C in order to increase the solubility of alloy. If the temperature rises directly to 465 °C, recrystallization occurs in the alloy. Gradually heating can keep the grain not to grow up, and at this time, the strength of Alloy 2 can reach 742.34 MPa. On the basis, if compound pre-treatment is conducted at 420 °C, the effect is not apparent.

When pre-treatment is conducted at 420 °C independently, the strength of alloy goes up significantly. When solution treatment is conducted at condition of (420 °C, 3 h)+(465 °C, 2 h), the tensile strength of Alloy 2 can reach the peak value of 777.29 MPa and elongation of 11.84%. If the time of pre-treatment or solution is prolonged, the strength decreases. On the basis, if pre-treatment at 200 °C is conducted, recrystallization occurs partly in Alloy 2 and the strength decreases.

Table 3 Effects of different solution treatments on mechanical properties of alloy

No.	Solution treatment	σ_b/MPa	δ/%
1a	465 °C, 1 h	602.32	15.47
2a		734.27	11.85
1b	465 °C, 2 h	593.40	17.44
2b		727.09	14.22
1c	465 °C, 3 h	588.31	18.14
2c		730.63	12.96
1d	(45 °C, 2 h)+(465 °C, 1 h)	592.66	15.81
2d		725.31	13.01
1e	(445 °C, 2 h)↗(1 h)+(465 °C, 1 h)	586.67	15.60
2e		742.34	13.80
1f	(420 °C, 3 h)+(445 °C, 2 h)↗	594.49	16.60
2f	(1 h)+(465 °C, 1 h)	735.23	13.04
1g	(420 °C, 6 h)+(465 °C, 2 h)	580.77	16.93
2g		683.35	15.77
1h	(420 °C, 3 h)+(465 °C, 3 h)	622.61	15.46
2h		755.49	12.40
1i	(420 °C, 3 h)+(465 °C, 2 h)	603.37	14.22
2i		777.29	11.84
1j	(200 °C, 3 h)+(420 °C, 3 h)+	601.29	16.54
2j	(465 °C, 2 h)	737.87	11.60

4 Conclusions

1) The additions of 0.03% Sc and 0.12% Zr can obviously hinder the recrystallization of Al−9.0Zn−2.8Mg−2.5Cu alloy during homogenizing and extruding processes.

2) The petaloid precipitated phase of $Al_3(Sc,Zr)$ occurs in Al−9.0Zn−2.8Mg−2.5Cu alloy with the additions of 0.03% Sc and 0.12% Zr. $Al_3(Sc,Zr)$ grain has

strong pinning effect on the dislocation.

3) Multi-stage solution obviously excels single solution, which can avoid the recrystallization of Al−9.0Zn−2.8Mg−2.5Cu−0.12Zr−0.03Sc with minor additions of Sc (<0.1%).

4) The proper solution treatment is (420 °C, 3 h)+(465 °C, 2 h) when Al−9.0Zn−2.8Mg−2.5Cu−0.12Zr−0.03Sc alloy acquires its tensile strength of 777.29 MPa and elongation of 11.84%, while Al−9.0Zn−2.8Mg−2.5Cu alloy acquires that 603.37 MPa and 14.22%, respectively.

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固溶处理对 Al−9.0Zn−2.8Mg−2.5Cu−0.12Zr−0.03Sc 铝合金组织性能的影响

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摘　要：采用金相显微镜、差热分析(DSC)和透射电镜(TEM)研究复合添加 0.03%Sc 与 0.12%Zr 及固溶处理对 Al−9.0Zn−2.8Mg−2.5Cu 合金组织性能的影响，以及添加少量(小于 0.1%)的 Sc 是否能得到高性能铝合金。结果表明：添加 0.03% Sc 与 0.12% Zr 可以使 Al−9.0Zn−2.8Mg−2.5Cu 合金出现“花瓣状”的 $Al_3(Sc,Zr)$析出相；$Al_3(Sc,Zr)$粒子对位错有强烈的钉扎作用，明显抑制 Al−9.0Zn−2.8Mg−2.5Cu 合金在均匀化和挤压过程中的再结晶；多级固溶明显优于单级固溶，可以在添加少量 Sc(小于 0.1%)时，避免 Al−9.0Zn−2.8Mg−2.5Cu 发生再结晶：(420 °C，3 h)+(465 °C，2 h)为最佳固溶条件，此时 Al−9.0Zn−2.8Mg−2.5Cu−0.12Zr−0.03Sc 合金的抗拉强度为 777.29 MPa，伸长率为 11.84%。

关键词：Al−Zn−Mg−Cu 合金；固溶处理；组织；力学性能

(Edited by Chao WANG)