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Trans. Nonferrous Met. Soc. China 24(2014) 3866−3871
Transactions of Nonferrous Metals Society of China
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Effect of minor Sc and Zr addition on microstructure and properties of ultra-high strength aluminum alloy

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Received 17 October 2013; accepted 13 November 2014

Abstract: The Al−9Zn−2.8Mg−2.5Cu−*x*Zr−*y*Sc alloys (*x*=0, 0.15%, 0.15%; *y*=0, 0.05%, 0.15%), produced by low-frequent electromagnetic casting technology, were subjected to homogenization treatment, hot extrusion, solution and aging treatment. The effects of minor Sc and Zr addition on microstructure, recrystallization and properties of alloys were studied by optical microscopy (OM), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The results show that Sc and Zr addition can refine grains of the as-cast alloy by precipitation of primary $Al_3(Sc,Zr)$ particles formed during solidification as heterogeneous nuclei. Secondary $Al_3(Sc,Zr)$ precipitates formed during homogenization treatment strongly pin the movement of dislocation and subgrain boundaries, which can effectively inhibit the alloys recrystallization. Compared with the alloy without Sc and Zr addition, the Al−Zn−Mg−Cu−Zr alloy with 0.05% Sc and 0.15% Zr shows the increase in tensile strength and yield strength by 172 MPa and 218 MPa, respectively. Strengthening comes from the contributions of precipitation, substructure and grain refining.
Key words: aluminum alloy; low-frequent electromagnetic casting; inhibit recrystallization; primary $Al_3(Sc,Zr)$ particles; secondary $Al_3(Sc,Zr)$ particles; substructure strengthening; precipitation strengthening; grain refining

1 Introduction

As a novel lightweight structure material, Al−Zn−Mg−Cu alloy with high strength, high toughness, good corrosion resistance and ascendant welding performance was widely used in aerospace fields [1−5]. Scandium (Sc) is one of the most effective alloying elements for improving properties of aluminum alloys. Some investigations [6−8] show that minor Sc or Sc and Zr addition can modify the microstructure and remarkably improve properties of alloys. When Sc and Zr are both used in Al−Zn−Mg−Cu alloys, $Al_3(Sc,Zr)$ precipitates occur. These precipitates are more effective recrystallization inhibitors than Al_3Sc or Al_3Zr, and the distribution of $Al_3(Sc,Zr)$ is more homogeneous than Al_3Zr in Al−Zn−Mg−Cu alloys. HE et al [4] investigated the refining effect of Sc and Zr elements on the microstructure of 7A55 alloy ingot. The results indicate that complex addition of Sc and Zr can modify the microstructure of cast ingot of 7A55 alloy, which increases the tensile strength and elongation. JIA et al [6] studied the effect of the content of Sc on formation of precipitates and recrystallization resistance in Al−Sc−Zr alloys. The results show that the average radius of the precipitates decreases with the increasing Sc content, while the number density of the precipitates increases and the temperature of recrystallization elevates remarkably. While the high price of Sc is prohibitive for extended commercial applications of Sc-containing aluminum alloys. There are few reports on decreasing cost of ultra-high strength Al−Zn−Mg−Cu alloys with moderate Sc and Zr.

In this work, different contents of Sc together with constant Zr content were added into the Al−Zn−Mg−Cu alloy to study the influence on the microstructure and mechanical properties and to investigate the effects of different contents of Sc added into alloy, which intends

Foundation item: Project (0211002605132) supported by Institute of Multipurpose Utilization of Mineral Resources, Chinese Academy of Geological Sciences, China; Project (0211005303101) supported by the Fundamental Research Funds for the Central Universities, China; Project (2010BB4074) supported by Natural Science Foundation Project of CQ CSTC, China; Project (2010ZD-02) supported by State Key Laboratory for Advanced Metals and Materials, China
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DOI: 10.1016/S1003-6326(14)63544-5

to balance the high properties and low production cost of ultra-high strength aluminum alloy containing Sc and Zr.

2 Experimental

High-purity Al (99.96%), pure Zinc (99.8%), pure Mg (99.8%), pure copper (99.6%), Al−2.59Sc and zirconium salts (18% Zr) were used as raw materials. Ingots with *d*152 mm of Al−9Zn−2.8Mg−2.5Cu−0.15Zr−*x*Sc alloys (*x*=0, 0.05%, 0.15%) were prepared by low-frequent electromagnetic casting technology. The chemical composition of each alloy was analyzed and the results are shown in Table 1. The ingot was then subjected to homogenization treatment (450 °C, 24 h), hot extrusion (400 °C, 600 mm/min), solution (445 °C, 2 h + 465 °C, 2 h) and peak aging treatments (120 °C, 24 h).

Table 1 Chemical composition of three alloys (mass fraction, %)

Sample No.	Zn	Mg	Cu	Sc	Zr	Fe	Si	Al
Alloy 1	8.56	2.78	2.30	0.00	0.00	0.12	0.06	Bal.
Alloy 2	8.72	2.88	2.42	0.05	0.16	0.24	0.07	Bal.
Alloy 3	8.29	2.85	2.30	0.15	0.13	0.15	0.06	Bal.

The alloys were processed into tensile samples according to GB/T16865−97 after heat treatments. The tensile testing was carried out on a CMT5105 machine at 2 mm/min drawing speed. The as-cast and solid-solution state microstructures of the alloys were observed by an Axiovert 40 MAT optical microscope (OM) and SEM. SEM samples were investigated using backscattering model after grinding and polishing. TEM specimens were prepared by cutting thin slices from the ingot, and then mechanically thinned down to 50 μm, followed by electropolishing using a twinjet polisher with 30% nitric acid solution in methanol from −20 to −30 °C with an applied voltage of 20 V. A Zeiss LIBRA 200 field emission TEM with an acceleration voltage of 200 kV was used for microstructure investigation.

3 Results

3.1 Effect of minor Sc and Zr additions on as-cast microstructure of alloys

The metallographic microstructures of the as-cast alloys are shown in Fig. 1. The microstructures have obvious change after adding Sc. The microstructure of alloy 1 without Sc and Zr addition contains coarse dendrite and the average grain size is close to 80 μm. However, the average grain sizes of the alloys 2 and 3 are 40−50 μm and 20 μm, respectively. Addition of Sc plays a remarkable role in grain refining, and the refining effect is improved with increasing adding amount of Sc in the present work.

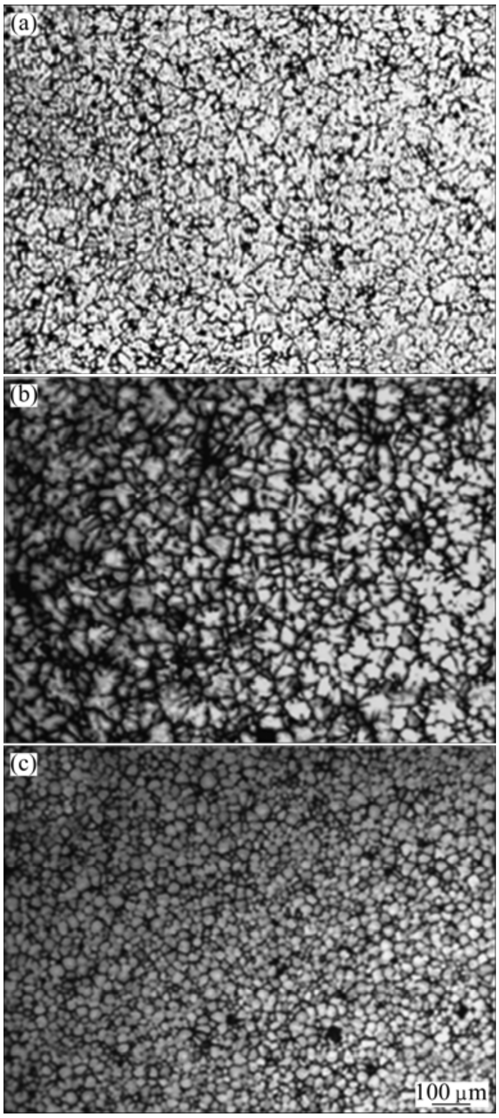

Fig. 1 Optical microstructures of as-cast alloys: (a) Alloy 1; (b) Alloy 2; (c) Alloy 3

Further microstructures were investigated by SEM. Besides the Al−Zn−Mg−Cu intermetallic phases at grain boundaries, the white particles with a size in micron level were often observed within the grains in the alloy 3, for instance, as seen in Fig. 2(a). SEM−EDS analysis shows that the particles are composed of Al, Sc, Zr elements (see Fig. 2(b)). DAI et al [8−10], SONG et al [11] and KNIPLING et al [12] also observed this kind of particles which belong to primary Al_3(Sc,Zr) formed during solidification. These primary Al_3(Sc,Zr) particles may act as the heterogeneous nuclei during solidification of α(Al), which promotes rapid nucleation and as a result, refines grains [8−10,13]. By comparing Figs. 1(b) and (c), the grain size of alloy 3 with 0.15% Sc is smaller than that of alloy 2 with 0.05% Sc. It is known from the Al−Sc binary phase diagram that the Sc solid solubility is about 0.03% in the aluminum solid solution at the solution temperature of 465 °C [14], while the addition of Zr element will decrease the maximum Sc solid solubility [15]. Therefore, the primary $Al_3(Sc,Zr)$ particles were formed in the alloy 3 with 0.15% Sc and 0.15% Zr, but not observed in the alloy 2 with 0.05% Sc and 0.15% Zr.

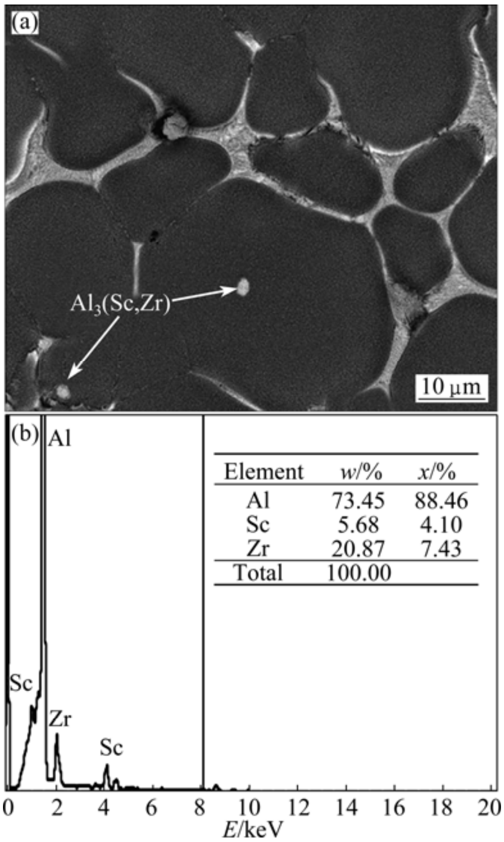

Fig. 2 Analysis of primary $Al_3(Sc,Zr)$ particle in as-cast alloy 3: (a) SEM micrograph showing intermetallic phases at grain boundaries and primary $Al_3(Sc,Zr)$ particles within grain; (b) EDS spectrum and chemical composition of primary $Al_3(Sc,Zr)$ particle

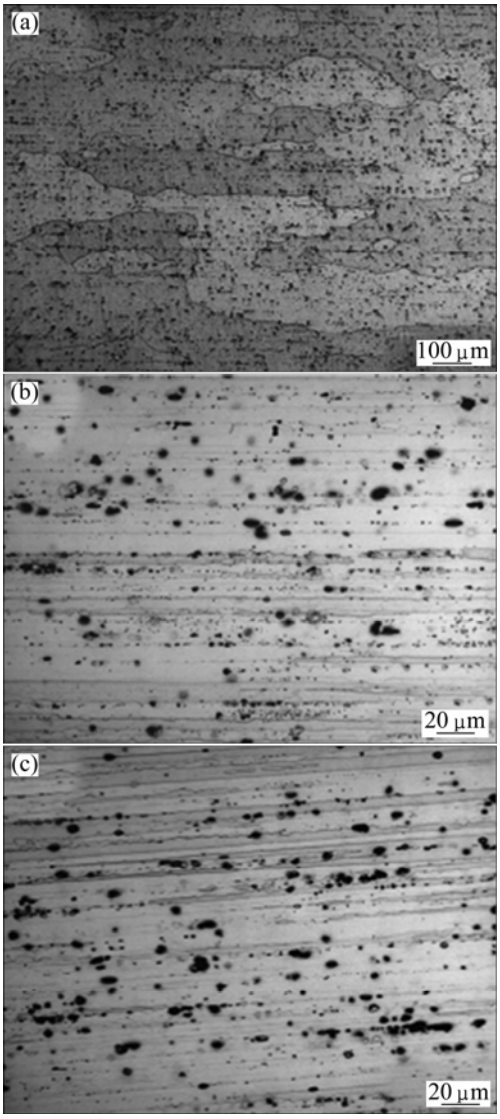

Fig. 3 Microstructures of extruded alloys after solid solution (445 °C, 2 h + 465 °C, 2 h) and aging (120 °C, 24 h) treatments: (a) Alloy 1; (b) Alloy 2; (c) Alloy 3

3.2 Effect of minor Sc and Zr on recrystallization of alloys

The microstructures of three extruded alloys after solid solution and peak aging treatment are shown in Fig. 3. It can be seen that the grains of the alloy 1 without Sc and Zr become significant coarsening after solid solution and aging treatment, which indicates that the recrystallization of alloy 1 occurs. However, the microstructures of the alloys 2 and 3 are unchanged and keep slender strips, which manifests that addition of Sc and Zr inhibits the occurrence of recrystallization.

The coffee bean-like $L1_2$-$Al_3(Sc,Zr)$ and dispersive black dot-like $\eta'(MgZn_2)$ precipitates in the alloy 3 in T6 state were observed in the bright-field TEM images as shown in Figs. 4(a) and (b). Both diffractions from the Al matrix and the precipitates are also shown in the insert of Fig. 4(a). Figure 4(b) shows that a large number of small dispersive $Al_3(Sc,Zr)$ precipitates are also formed at the grain boundaries and dislocations. Figures 4(c) and (d) are the dark-field TEM images of the alloys 2 and 3 in T6 state, respectively. It is detectable that alloy 3 has higher density of $Al_3(Sc,Zr)$ precipitates compared with alloy 2.

3.3 Effect of minor Sc and Zr on mechanical properties of alloys

The tensile properties of three alloys after solid solution and peak aging treatment are shown in Table 2. The tensile strength and yield strength of alloys 2 and 3 are obviously superior to those of alloy 1. The tensile strengths of alloys 2 and 3 after the solution treatment are enhanced by 172 MPa and 162 MPa, respectively,

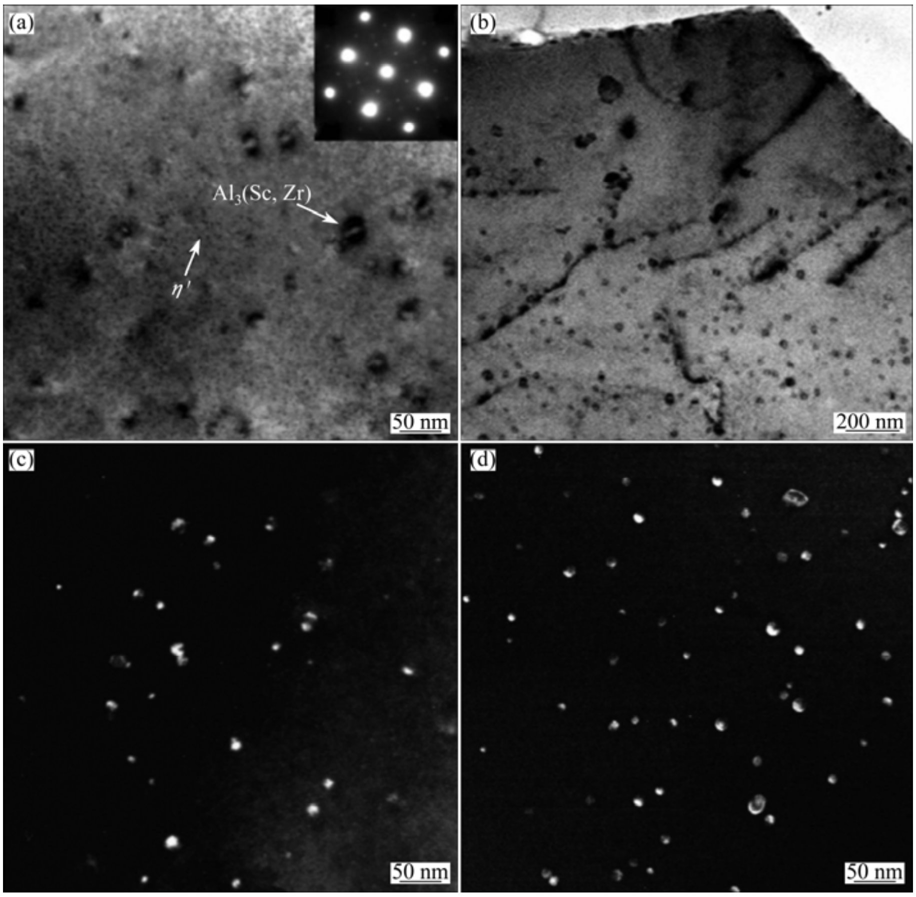

Fig. 4 TEM images of alloys in T6 state: (a, b) Bright-field TEM images of alloy 3; (c) Dark-field TEM image of alloy 2; (d) Dark-field TEM images of alloy 3

Table 2 Tensile properties of alloy 1, alloy 2 and alloy 3

Heat treatment	Alloy 1			Alloy 2			Alloy 3		
	σ_b/MPa	$\sigma_{0.2}$/MPa	δ/%	σ_b/MPa	$\sigma_{0.2}$/MPa	δ/%	σ_b/MPa	$\sigma_{0.2}$/MPa	δ/%
Solid solution treatment	498	407	14	650	515	12	660	523	13
T6 state	618	501	8.8	790	719	7.8	750	700	7

compared with that of alloy 1. In addition, the improvement of the tensile strength of alloys 2 and 3 in T6 state is also obvious. The elongation of the alloy 2 or 3 is slightly lower than that of the alloy 1. This indicates that the strength of the alloy can be improved by adding Sc and Zr with an expense of a little ductility.

It is worth to point out that the tensile strength of alloy 2 (650 MPa) is somewhat lower than that of alloy 3 (660 MPa) after solid solution treatment. The tensile strength of alloy 2 (790 MPa) is 40 MPa higher than that of the alloy 3 (750 MPa) in T6 state.

4 Discussion

4.1 Strengthening mechanism of Sc and Zr additions on aluminum alloy

The mechanical properties of aluminum alloys can be remarkably improved by adding Sc and Zr [7] due to the formation of $Al_3(Sc,Zr)$ phases, including primary and secondary $Al_3(Sc,Zr)$ particles. It is believed that the morphology and effect of primary and secondary $Al_3(Sc,Zr)$ particles should be different because the diffusions of Sc and Zr atoms are different during solidification and annealing [6,11,16]. The primary $Al_3(Sc,Zr)$ particles are formed during solidification by strong affinity of Sc and Zr atoms, which are interacted with the Al matrix because of their similar diffusions in liquid [11]. This is benefit to refine grain size of the as-cast alloy by precipitation of primary $Al_3(Sc,Zr)$ particles from the melt during solidification as heterogeneous nuclei (see Figs. 1 and 2). Therefore, the improvement of the mechanical properties is realized by grain refining caused by the primary $Al_3(Sc,Zr)$ particles.

Secondary $Al_3(Sc,Zr)$ precipitates are formed during annealing. Due to the faster diffusion rate of Sc atoms compared with Zr atoms in the Al matrix, the core of the

Al_3Sc was firstly formed, and then Zr atoms were segregated to the Al_3Sc−matrix interfaces to form surrounding Zr-containing “shells” at later stage [7,17−20]. The core-shell structure of the $Al_3(Sc,Zr)$ precipitate has been modeled by ROBSON [21] and LEFEBVRE et al [22].

The core-shell structure formed by co-adding Sc and Zr exhibits better thermal stability by reducing the matrix-precipitate lattice mismatch and further, the driving force for coarsening [23]. Thus, secondary $Al_3(Sc,Zr)$ precipitates not only strongly pin movement of dislocations and subgrain boundaries (see Fig. 4(b)), which results in enhancement of the mechanical properties, but also remarkably elevates recrystallization temperature of aluminum alloys [6,13,22].

4.2 Effect of secondary $Al_3(Sc,Zr)$ precipitates on aging precipitates in Al−Zn−Mg−Cu−Zr−Sc alloy

It is well known that the main strengthening phase of Al−Zn−Mg−Cu alloy is $\eta'(MgZn_2)$. The generally accepted precipitation sequences for 7000 series alloys are as follows [4,5,24,25]: supersaturated solid solution (SSS)→coherent GP zones→semi-coherent intermediate $\eta'(MgZn_2)$→ incoherent stable $\eta(MgZn_2)$. Researchers have different views on the effect of secondary $Al_3(Sc,Zr)$ particles on aging precipitates in Al−Zn−Mg−Cu− Zr−Sc alloy. XIAO et al [26] thought that combined additions of Sc and Zr result in a dense and homogeneous distribution of thermally stable secondary $Al_3(Sc,Zr)$ precipitates during annealing, which should rise the nucleation rate of $\eta'(MgZn_2)$ and improve the mechanical properties of alloys. They also argued that [26] it has no effect on the mechanical properties but enhances the phase transformation driving force of GP zones to $\eta'(MgZn_2)$. In studying the effect of minor Sc on quenching sensitivity of 7085 aluminum alloy, QI et al [16] found that as the quenching rate decreases, the equilibrium $\eta(MgZn_2)$ precipitates nucleate heterogeneously on $Al_3(Sc,Zr)$ precipitates and precipitate at subgrain boundaries. The precipitation of η phase reduces the solutes available for aging hardening, leading to the decrease of the mechanical properties of alloys.

In this study, it was not found that the metastable or equilibrium $MgZn_2$ phases nucleate heterogeneously on $Al_3(Sc,Zr)$ precipitates. Better mechanical property of alloy 3 than alloy 2 after solution treatment is definitely attributed to higher density of $Al_3(Sc,Zr)$ precipitates in alloy 3 which is confirmed by TEM results (see Figs. 4(c) and (d)), since the main alloying elements of Zn, Mg and Cu, at this stage, are in the solid solution and no strengthening phases are formed. However, the increase of the mechanical properties of three alloys after T6 treatment mainly comes from the contribution of forming metastable $MgZn_2$ phases, because the formed $Al_3(Sc,Zr)$ precipitates at solution treatment stage are very stable at ageing stage. The actual amount of Zn in the alloy plays a very important role in forming $MgZn_2$ strengthening phase on quantity, thus improving mechanical properties. For instance, the tensile strength of alloy 2 (790 MPa) is better than that of alloy 3 (750 MPa) in T6 state, which is corresponded to the real content of Zn in alloy 2 (8.72 %) and in alloy 3 (8.29%).

5 Conclusions

1) Minor Sc and Zr additions can refine grains of the as-cast alloy by forming primary $Al_3(Sc,Zr)$ particles during solidification as heterogeneous nuclei. Secondary $Al_3(Sc,Zr)$ precipitates formed during homogenization treatment effectively depresses recrystallization of the alloys during high temperature treatment by pinning the movements of dislocation and subgrain boundaries.

2) Minor Sc and Zr additions can remarkably improve the mechanical properties of Al−Zn−Mg−Cu alloy. Compared with the alloy without Sc and Zr addition, the tensile strength (790 MPa) and yield strength (719 MPa) of the alloy, containing 0.05% Sc and 0.15% Zr, are enhanced by 172 MPa and 218 MPa, respectively, in T6 state.

3) The ultrahigh tensile strength and yield strength of the alloy are realized by grain refining and precipitation strengthening by minor Sc and Zr addition, and high amount of Zn forming $MgZn_2$ strengthening phase.

4) Addition of 0.05%Sc can efficiently improve the mechanical properties of the alloy mainly by forming coherent $Al_3(Sc,Zr)$ precipitates.

Acknowledgments

The authors would like to acknowledge Prof. Jian-zhong CUI and Associate Prof. Qing-feng ZHU (Key Laboratory of Electromagnetic Processing of Materials, Ministry of Education, Northeastern University) for helping casting and extrusion of the studied materials.

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添加微量 Sc、Zr 对超高强铝合金微观结构和性能的影响

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摘 要：采用低频电磁铸造技术制备 Al−9Zn−2.8Mg−2.5Cu−xZr−ySc (x=0, 0.15%, 0.15%; y=0, 0.05%, 0.15%)合金，借助金相显微镜、扫描电镜、透射电镜、力学性能测试等手段分别对其均匀化、热挤压态、固溶态和时效态的组织与性能进行对比分析。结果表明：添加微量 Sc 和 Zr，会在凝固过程中形成初生 $Al_3(Sc,Zr)$，可显著细化合金铸态晶粒；均匀化时形成的次生 $Al_3(Sc,Zr)$粒子可以强烈钉扎位错和亚晶界，有效抑制变形组织的再结晶，显著提高合金的力学性能。与不含 Sc、Zr 的合金相比，含 0.05% Sc 和 0.15%Zr 的合金经固溶处理和峰值时效处理后其抗拉强度和屈服强度分别提高 172 MPa 和 218 MPa，其强化作用主要来自含 Sc、Zr 化合物对合金起到的亚结构强化、析出强化和细晶强化。

关键词：铝合金；低频电磁铸造；抑制再结晶；初生 $Al_3(Sc,Zr)$；次生 $Al_3(Sc,Zr)$；亚结构强化；析出强化；细晶强化

(Edited by Xiang-qun LI)