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Mechanism of heterogeneous distribution of Cr-containing dispersoids in DC casting 7475 aluminum alloy

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Abstract: The actual effective partition coefficients of Mg and Cr in a cross-section of a dendrite arm in a direct-chill (DC)-casting ingot of 7475 aluminum alloy are obtained. Meanwhile, by analyzing the microstructure, the mechanism of the heterogeneous distribution of E (Al₁₈Mg₃Cr₂) dispersoids in this DC ingot is revealed. The results show that the actual effective partition coefficients of Mg and Cr are 0.650 and 1.392, respectively, and they describe the heterogeneous distributions of Mg and Cr along the direction of radius of the cross-section of the dendrite arm of the alloy. After homogenization treatment at 470 °C for 24 h, Mg diffuses uniformly, but Cr hardly diffuses. Both the concentrations of Mg and Cr and the sites of heterogeneous nucleation in the alloy are the determinants of the formation of E dispersoids simultaneously. The heat treatment at 250 °C for 72 h provides a large number of the sites of heterogeneous nucleation of the cross-section during the subsequent heat treatment at higher temperature.

Key words: 7475 aluminum alloy; direct-chill-casting ingot; Al₁₈Mg₃Cr₂ dispersoids; heterogeneous distribution; effective partition coefficient

1 Introduction

7475 aluminum alloy is extensively used in the aerospace industry as a very important structural material due to its excellent properties, such as low density and high strength, fracture toughness [1], stress corrosion cracking resistance [2,3] and superplasticity [4,5]. The alloying characteristic of 7475 aluminum alloy is the addition of trace Cr to inhibit recrystallization and refine grains [6,7]. Although the addition of Cr tends to increase the quenching sensitivity of alloy plates [8,9], it can form fine Al₁₈Mg₃Cr₂ dispersoids in the alloy [10],

which can inhibit recrystallization and keep fibrous grains for the alloy plate after solution treatment. It is beneficial to improve the fracture toughness and stress corrosion resistance of alloy plates by increasing the crack propagation path [11,12].

In order to decrease the microsegregation of the ingot of the alloy and dissolve the eutectics in it, the homogenization treatment is necessary before subsequent processing [13,14]. The *E* dispersoids in 7475 aluminum alloy are usually formed during the homogenization treatment [15]. The size and distribution of the *E* dispersoids in alloys are always heterogeneous [16] due to the segregation of solutes inside the dendrite arms caused by selective

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crystallization during the solidification of solutes [17,18]. Precipitation of the secondary phase during the cooling process after solidification has a strong influence on the formation and distribution of the *E* dispersoids [19]. Besides, there are also many studies on the control of precipitation behavior of the other dispersoids in 7xxx aluminum alloy. Appropriate homogenization conditions can make the dispersoids in Al-Zn-Mg-Cu alloy finer and more dispersed [20]. By prolonging the homogenization time, the dispersoids with smallsize will dissolve into the matrix of the alloy, but those with large-size will further grow [21]. The high-density fine dispersoids formed during homogenization treatment have significant impacts on the grain size and mechanical properties of the alloy. Their main role is to stabilize the grain structure of alloy by pinning grain boundaries and subgrain boundaries and inhibit recrystallization during hot rolling. According to the Zener pinning formula, the decrease of Zener pinning force (ZPP) is due to the coarse dispersoids after a long time of homogenization treatment [22,23]. Among the dispersoids in Al-Zn-Mg-Cu alloy, the dispersoids containing Zr have been systematically studied. It has been proven that the size and distribution of dispersoids containing Zr in Al-Zn-Mg-Cu alloy can be effectively controlled by pretreatment at 250 °C [24]. But there are few studies on the forming mechanism of the heterogeneous distribution of dispersoids containing Cr in Al-Zn-Mg-Cu alloy.

In the present work, the concentration distributions of Mg and Cr in the dendrite arms of DC ingot of 7475 aluminum alloy are investigated to reveal the forming mechanism of the heterogeneous distribution of $Al_{18}Mg_3Cr_2$ dispersoids in it. It is expected to lay a foundation for the development of heat treatment process of further controlling the distribution of dispersoids containing Cr of 7475 aluminum alloys in the future.

2 Experimental

The experimental material was a DC ingot of 7475 aluminum alloy with a thickness of 300 mm, and its composition was as follows (wt.%): 5.52 Zn, 2.20 Mg, 1.60 Cu, 0.22 Cr, 0.20 Fe, 0.02 Si, <0.1 others, and balance Al. Specimens with a thickness

of 25 mm were cut from a quarter of the thickness of the alloy ingot. Four kinds of heat treatments were used: (1) single-stage homogenization of 470 °C, 24 h followed immediately by water quenching (for short, 470 °C, WQ), 24 h, (2) double-stage heat treatment including 470 °C, 24 h, WQ, and then 250 °C, 72 h, WQ (for short, (470 °C, 24 h, WQ) + (250 °C, 72 h, WQ)), (3) triple-stage heat treatment including 470 °C, 24 h, WQ, and then 250 °C, 72 h, WQ and again 470 °C, 24 h, WQ (for short, (470 °C, 24 h, WQ) + (250 °C, 72 h, WQ) + (470 °C, 24 h, WQ)), (4) (470 °C, 24 h, WQ) + (250 °C, 72 h, WQ) + (400 °C, 0.5 h, WQ). All of specimens were heated to 470 °C for 1 h from room temperature in a circulating air furnace. The configuration of second-phase particles was determined by an Olympus GX71 optical micro- scope (OM), a JEOL JEM-7100F scanning electron microscope (SEM), and a JEOL JEM-2100F transmission electron microscope (TEM). The observed surface of microstructure was the cross-sections of the ingot perpendicular to the casting direction.

A JEOL JXA-8530F electron probe microanalyzer (EPMA) was used to quantitatively analyze the actual elemental distributions of Mg and Cr from the center to the boundary of the cross section of dendrite arm of the alloy ingot. The analysis voltage is 15 kV, the beam current is 1×10^{-8} A, the beam spot size is 2 µm, and the step length is 2 µm. In a field of microstructure of SEM, a cross-section of a coarser dendrite arm about 80 µm from the center to its boundary for a total of 40 points was selected, which was measured each with a diameter of 2 µm. It was assumed that the center of the cross-section of the dendrite arm is the initial crystallization vicinity, and its corresponding crystallization solid fraction (f_s) is equal to zero (i.e. $f_s=0$). The corresponding crystallization solid fraction at the boundary of the cross-section of the dendrite arm is the ultimate crystallization vicinity (i.e. $f_s=1$). Because the excitation area of characteristic X-ray is larger than the actual measurement area, the two results near the boundary among the 40 measurement points were affected by the eutectic structure. The element contents of eutectic structure on the boundary cannot be described by the one-dimensional Scheil formula. Therefore, when fitting the experimental data according to the one-dimensional Scheil

formula, it is necessary to discard the two measurement points near the eutectic products and retain the other 38 measurement points. The corresponding solid fraction of the 38 measurement points is from 0 for the center of the cross-section of the dendrite arm of the alloy to 0.95 for near its boundary. Then, by combining the results of the EPMA quantitative analysis with the onedimensional Scheil-Gulliver model, the actual effective partition coefficients $K_{\rm e}$ of Mg and Cr across the cross-section were fitted. Finally, on the basis of the solid solubilities of Mg and Cr at 470 °C in 7475 aluminum alloy calculated by JMatPro, the relationships were calculated between the dispersoid volume fraction $(V_{\rm f})$ and the crystallization solid fraction (f_s) along the radial direction across the cross-section after heat treatment at 470 °C for 24 h.

Specimens for OM were etched by Keller's reagent (2 mL HF + 3 mL HCl + 5 mL HNO₃ + 250 mL H₂O). Specimens for TEM observation were cut from the ingot, mechanically thinned to approximately 90 μ m, and then electropolished in a twin-jet polishing unit operating at 15 V and -25 °C by a solution of 25% nitric acid and 75% methanol.

3 Results and discussion

3.1 Heterogeneous distribution of Al₁₈Mg₃Cr₂ dispersoids

Figure 1 shows the OM and SEM micrographs of the DC ingot of 7475 aluminum alloy. There are many strip-like constituents and lamellar eutectic structures (i.e. eutecticum) at the dendrite arm boundary or grain boundary (GB) in the ingot of the alloy. The results of energy dispersive spectrometer (EDS) show that the white lamellar eutectic structures in Fig. 1(b) mainly contain Al, Zn, Mg, and Cu. Some strip-like or clump-like constituents are Al₃Fe phase, the same as the research by FAN et al [14]. A shell-like structure distributes in grains or dendrite arms in the as-cast alloy with thickness about 15 µm near the grain boundary and the secondary phase cannot be seen in their center.

Figure 2 shows the stitching TEM images of the as-cast 7475 aluminum alloy from the grain boundary to the center of the grain. It can be observed that a large number of coarse strip-like or clump-like secondary phases and needle-like secondary phases with high density near the grain



Fig. 1 OM (a) and SEM (b) micrographs of DC ingot of 7475 aluminum alloy

Table 1 Chemical compositions obtained from EDSmeasurements in Fig. 1 (wt.%)

Phase	Zn	Mg	Cu	Cr	Fe	Si	Al
Eutecticum	27.60	18.56	21.93	0.03	0.03	_	31.84
Al ₃ Fe	2.40	1.27	7.97	0.72	27.72	0.01	59.91

boundary precipitated during the cooling process of the alloy after solidification. The EDS results show that the coarse strip-like or clump-like secondary phases mainly contain Al, Zn, Mg and Cu (namely, the T phase), and the needle-like secondary phases with specific directivity mainly contain Al, Cu and Mg (namely, the S phase), as given in Table 2, which is consistent with the result of REZNIK and OVSYANNIKOV [25]. The zone of the microstructure in Fig. 2(g) is the center of the grain of the alloy. In that zone, there is no coarse strip-like or clump-like secondary phase, but small needle-like precipitates are seen. The zone of the microstructure in Fig. 2(f) is approximately 25 µm away from the grain boundary, where there are a few coarse strip-like or clump-like secondary phases and some small needle-like precipitates.





Fig. 2 TEM images of distribution of secondary phases in ingot of alloy: (a) GB; (b-e) Away from GB in turn; (f) 25 µm away from GB; (g) Center of grain

Table 2 Chemical compositions obtained from EDS measurements in Fig. 2 (wt.%)

Phase	Zn	Mg	Cu	Cr	Fe	Si	Al
Т	22.91	6.60	1.98	0.04	0.26	0.03	68.18
S	4.65	5.01	10.66	0.12	0.27	0.21	79.08

The OM and SEM micrographs of the DC ingot of 7475 aluminum alloy after 470 °C, 24 h, WQ are shown in Fig. 3. Almost all the lamellar eutectic products containing Al, Zn, Mg, and Cu at the boundary are dissolved into the matrix of the alloy after homogenization at 470 °C for 24 h. It can be observed that there are only a small amount of clump-like constituents remaining on the grain boundary, which are proven to be Al₇Cu₂Fe by the EDS, the same as the research by PENG et al [26]. Many submicron dispersoids are also observed in the matrix of the alloy after 470 °C, 24 h, WQ, as shown in Fig. 3. Their distribution has a shell-like feature similar to Fig. 1. This distribution with a shell-like feature may also be seen as an egg-looking distribution. In the microstructure with an egg-looking distribution, the dispersoids form "egg white" with thickness about 25 µm and the dispersoid-free zone (DFZ) with large area in the center of the grain form "egg yolk" are observed. The submicron particles in Fig. 3 mainly contain Al, Mg, and Cr. They are Al₁₈Mg₃Cr₂ dispersoids proven by subsequent EDS results, which were precipitated during the homogenization treatment at 470 °C for 24 h. Significantly, in the microstructure of the shell-like structure, there are two kinds of



Fig. 3 OM (a) and SEM (b) micrographs of 7475 aluminum alloy after 470 °C, 24 h, WQ

DFZs in the homogenized alloy: one is a narrow strip at the both sides of the grain boundary, and the other is in the center of the grain with a large area.

Figure 4 shows TEM images of microstructure



Fig. 4 TEM images of dispersoid distribution in alloy after 470 °C, 24 h, WQ: (a) GB; (b–e) Away from GB in turn; (f) 25 µm away from GB; (g) Center of grain

from the grain boundary to the center of the grain of the 7475 aluminum alloy subjected to 470 °C, 24 h, WQ. It can be observed that there are many dispersoids with different shapes and sizes in the alloy. They are E phase particles proven by the EDS results in Table 3, which is consistent with the result of HE et al [9]. Figure 4(f) shows the microstructure of the zone about 25 μm away from the grain boundary, which is the transitional zone between the large DFZ in the center of grain and the precipitation zone of dispersoids in the shell. In this zone, there are a few of coarse dispersoids. Above all, it can be summarized that the size and distribution of the E phase in the 7475 aluminum alloy are heterogeneous after single-stage homogenization treatment at 470 °C for 24 h.

Table 3 Chemical compositions of E phase obtained from EDS measurements in Fig. 4 (wt.%)

				0	,			
Point	Zn	Mg	Cu	Cr	Fe	Si	Al	
1	7.46	3.61	3.84	1.68	0.21	0.08	83.11	
2	8.30	9.48	6.47	11.80	0.04	0.21	63.70	

Although the distribution of secondary phase particles in the specimens of 7475 aluminum alloy both as-cast and homogenized at 470 °C for 24 h, show similar shell-like features, the type of secondary phase in them are different. Secondary phase particles in the former are coarse T phase

particles formed during the cooling process of the alloy after solidification, while those in the latter are E dispersoid particles precipitated during homogenization treatment at 470 °C for 24 h. From the similar distribution of T and E phases, the formation of E dispersoids in the 7475 alloy during the homogenization treatment at 470 °C for 24 h might be directly related to the T phase particles in the DC ingot of 7475 aluminum alloy. That is to say, T phase formed during cooling process of the alloy after solidification might be beneficial to the heterogeneous nucleation of E phase, and then E phase particles grow up during the single-stage homogenization treatment at 470 °C for 24 h.

By comparing Fig. 2(a) with Fig. 4(a), it can be seen that the precipitate-free zone (PFZ) of the as-cast alloy near the boundaries of grain or dendrite arm is narrower and the dispersoid-free zone (DFZ) of the homogenized alloy is wider. That is to say, after cooling of DC ingot of the alloy, there are more secondary phases near the boundaries of its grain or dendrite arm, but the precipitates in a certain region close to those boundaries do not become the sites of forming Ephase during the homogenization treatment of the alloy ingot at 470 °C for 24 h. It can be deduced that the formation and distribution of the *E* phase in the grain of the alloy subjected to single-stage homogenization treatment at 470 °C for 24 h are not completely determined by the distribution of the existing secondary phases in it.

3.2 Effective partition coefficients of Mg and Cr in 7475 aluminum alloy

Because the distribution characterization of E dispersoids might be decided by the concentration of Mg or Cr which might influence the formation of the E dispersoids during the subsequent homogenization treatment at 470 °C, it is necessary to reveal the concentration distribution of Mg and Cr in the dendrite arms or grains in the DC ingot.

As the center of the cross-section of a dendrite arm or a grain is the zone of initial crystallization, while its boundary is the zone of final crystallization, it is assumed that the crystallization solid fractions (f_s) in its center and boundary are 0 and 1, respectively. Much nonequilibrium eutecticum, mainly containing Al, Zn, Mg, and Cu, is formed at the boundaries of grains and dendrites arm during the rapid cooling of DC ingot of the alloy, resulting in the segregation of Mg on the boundaries of the grains or dendrite arms. This phenomenon causes the actual concentration of Mg in the grain or dendrite arm to be relatively low and a sudden change of composition occurs at the interfaces between the matrix and the nonequilibrium eutecticum. Because the size of the excitation region of EPMA is larger than the size of beam spot, the composition of the alloy matrix near the eutecticum will be affected. So, in the calculation, the maximum value of the crystallization solid fractions is only taken equal to 0.95.

In the present work, three cross-sections of coarser dendrite arms in the as-cast alloy are selected to be measured by EPMA. The concentration distributions (c_1 , c_2 and c_3) of Cr and Mg from the center ($f_s=0$) of three cross-sections of the dendrite arms to their nearby boundaries ($f_s=0.95$) are shown in Fig. 5. The average concentration of Cr at $f_s=0.95$ near the boundaries of the dendrite arms is less than 0.1 wt.% and that of Mg is more than 2.5 wt.%, and in the center of the cross-sections of Cr and Mg are greater than 0.3 wt.% and less than 1.0 wt.%, respectively.

The average concentrations of Cr and Mg from each cross-section of coarser dendrite arm calculated by all of experimental data are given in Table 4. The average concentrations (\bar{c}) of the Cr and Mg in all of three cross-sections of dendrite arms of the alloy are 0.238 wt.% and 1.262 wt.%, respectively.



Fig. 5 Concentration distributions of Cr and Mg from center to nearby boundary of three cross-sections of dendrite arms in DC ingot of alloy and their fitted curves of one-dimensional Scheil model: (a) Cr; (b) Mg

 Table 4 Average concentrations of Mg and Cr calculated

 from three dendrite arms (wt.%)

Element	c_1	<i>C</i> ₂	Сз	\overline{c}
Mg	1.118	1.376	1.293	1.262
Cr	0.271	0.241	0.201	0.238

The coarsening process of the dendrite arm during the solidification is considered to be from the center to the boundary of the cross-section of a dendrite arm, which can represent the selective crystallization process in a single direction of the dendrite arm of the alloy during solidification. As noted before, the solid fractions in the dendrite arm center and on the dendrite arm boundary are 0 and 1, respectively. The distribution of solute atoms in the single direction should satisfy the one-dimensional Scheil–Gulliver model:

$$c = k_0 \overline{c} (1 - f_s)^{(k_0 - 1)} \tag{1}$$

where *c* is the concentration of the solute at different sites from the center to the boundary in a cross-section of a dendrite arm, k_0 is the equilibrium partition coefficient of the solute replaced by the effective partition coefficient k_e , \overline{c} is the average concentration of the solute replaced by the average value of the measured data, and f_s is the solid fraction.

The average concentrations of Mg and Cr in Table 4 are substituted into Eq. (1). Three groups of experimental data are fitted, as shown in Fig. 5. The effective partition coefficients k_e of Cr and Mg in three dendrite arms of the 7475 aluminum alloy during the crystallization process are calculated, as listed in Table 5. The average values of k_e of Cr and Mg are 1.392 and 0.650, respectively. This is also close to the result calculated by the weighted interval rank sort method by CHEN et al [27].

Table 5 Effective partition coefficients (k_e) of Mg and Cr in three dendrite arms and their average values

Element	k _{e1}	k_{e2}	ke3	$\overline{k}_{\mathrm{e}}$
Mg	0.625	0.680	0.646	0.650
Cr	1.321	1.292	1.564	1.392

The curves of the concentration distribution of Cr and Mg along the radial direction of the cross section of the dendrite arm in the DC ingot of the 7475 aluminum alloy can be described by Eqs. (2) and (3) by substituting the corresponding k_e =1.392 for Cr and k_e =0.650 for Mg into Eq. (1):

$$C_{\rm Cr} = 0.331 (1 - f_{\rm s})^{0.392} \tag{2}$$

$$C_{\rm Mg} = 0.82(1-f_{\rm s})^{0.35}$$
 (3)

The curves of the actual concentration distribution of Cr and Mg from the center (f_s =0) in the cross-section of the dendrite arm in the DC ingot of the alloy to its nearby boundary (f_s =0.95) are shown in Fig. 6(a) according to Eqs. (2) and (3). The actual concentrations of Mg and Cr change significantly from the center of the dendrite arm to its boundary due to selective crystallization during DC casting of the alloy. The concentration of Mg increases significantly near the boundary and that of Cr is very low near the boundary of the cross-section. The solid solubilities (*S*) of Mg and Cr calculated by JMatPro in 7475 aluminum alloy at 470 °C are 1.29% for Mg and 0.044% for Cr, respectively, which are the critical solubilities of



Fig. 6 Actual concentration distributions of Cr and Mg (a) and theoretical volume fraction of E dispersoids (b) in dendrite arm of DC ingot of 7475 aluminum alloy

Mg and Cr to form E dispersoids in this alloy at 470 °C (the two horizontal lines in Fig. 6(a)). E dispersoid particles might be formed in the DC ingot of 7475 aluminum alloy during the homogenization at 470 °C only when the concentrations of Mg and Cr exceed the critical solubility of 1.29% and 0.044% respectively. Figure 6(a) shows that the zone of forming *E* phase in 7475 aluminum alloy at 470 °C should be in the range between 0.726 and 0.994 of corresponding solid fraction of crystallization f_s . When f_s is less than 0.726, the concentration of Mg is less than 1.29%, and E dispersoids cannot be formed. When f_s is larger than 0.994, the concentration of Cr is less than 0.044%, and E dispersoids cannot be formed yet. That is to say, when f_s is less than 0.726 or greater than 0.994, E phase cannot be formed during the first homogenization.

The volume fraction of *E* dispersoids corresponding to solid fraction in the dendrite arm of the DC ingot of 7475 aluminum alloy homogenized at 470 °C can be calculated by [24] 4)

$$V_{\rm f} = \frac{c_{\rm s} \rho_{\rm a}}{w_{\rm d} \rho_{\rm d}} \tag{6}$$

where $c_{\rm s}$ is the supersaturation of the solute in the alloy, equal to the actual concentration minus the critical solubility calculated by JMatPro. w_d is the mass fraction of the solute in the dispersoid particles, and those of Mg and Cr are 11.01% and 15.69% in the E phase, respectively. $\rho_{\rm d}$ is the density of the dispersoid particles, equal to 2.87 g/cm³ for E phase [28]. ρ_a is the density of alloy, equal to 2.7 g/cm³ for 7475 aluminum alloy. Figure 6(b) shows the calculation result of the volume fraction of E dispersoids from the center of the cross-section of the dendrite arm of the alloy to its boundary. The distribution of E dispersoids is heterogeneous after the first homogenization of the DC ingot of 7475 aluminum alloy at 470 °C. Only in the zone where the corresponding solid fraction of crystallization is between 0.726 and 0.994 can the E phase be formed, and the volume fraction of Edispersoids has the maximum value of 0.836% corresponding to crystallization solid fraction of 0.778.

Figure 7 shows the concentration distributions of Cr and Mg measured by EPMA from the center $(f_s=0)$ of the cross-section of the dendrite arm in the 7475 aluminum alloy to the nearby boundary ($f_s=0.95$) after 470 °C, 24 h, WQ. Compared with Fig. 5, it can be seen that the distribution of Cr remains unchanged, that is, Cr in the dendrite arm of 7475 aluminum alloy does not diffuse uniformly after homogenization treatment at 470 °C for 24 h. However, Mg in the dendrite arm of the ingot diffuses almost uniformly after homogenization. The concentration of Mg from the center ($f_s=0$) to the nearby boundary ($f_s=0.95$) of the cross-section of dendrite arm is almost consistent, and it is close to the average composition of the alloy, which equals 2.2%. It is evident that there are enough Cr and Mg in the center of dendrite arm of the ingot after 470 °C, 24 h, WQ, which exceed the critical concentrations of Cr and Mg for the formation of the *E* phase. But, the *E* dispersoid could still not be formed in the center of dendrite arm, as shown in Figs. 3 and 4. This phenomenon shows that it is difficult to form E phase by homogeneous nucleation even if there is sufficient concentration of Mg and Cr during homogenization at 470 °C.



Fig. 7 Concentration distributions of Cr (a) and Mg (b) from center to nearby boundary of cross-section of dendrite arm in alloy after $470 \,^{\circ}$ C, 24 h, WQ

3.3 Heterogeneous nucleation of Al₁₈Mg₃Cr₂ dispersoids

Figure 8 shows the OM and SEM micrographs of DC ingot of 7475 aluminum alloy after (470 °C, 24 h, WQ) + (250 °C, 72 h, WQ). Figure 9 shows TEM images from the grain boundary to the center of the grain. The position of the microstructure in the grain shown in Fig. 9(f) corresponds to that in Fig. 4(f) (approximately 25 µm away from the grain boundary), which is the transitional zone between the large DFZ of E phase in the center of grain of the alloy and its precipitated zone in the shell only homogenized after 470 °C, 24 h, WQ. The coarse precipitates in Fig. 8(b) are E phase particles precipitated during the first homogenization treatment at 470 °C for 24 h, and the fine precipitates are MgZn₂ (η) and T phase precipitated during the following treatment at 250 °C for 72 h.

After the homogenization treatment at 470 °C for 24 h and WQ, the concentrations of Zn, Mg and Cu in the grain of the alloy should be homogeneous and supersaturated. During the subsequent heat treatment at 250 °C for 72 h, in the center of the



Fig. 8 OM (a) and SEM (b) micrographs of alloy after (470 °C, 24 h, WQ) + (250 °C, 72 h, WQ)



Fig. 9 TEM images of precipitates distribution in alloy after (470 °C, 24 h, WQ) + (250 °C, 72 h, WQ): (a) GB; (b–e) Away from GB in turn; (f) 25 μ m away from GB; (g) Center of grain

Table 6 Chemical compositions obtained from EDSmeasurements in Fig. 9 (wt.%)

Phase	Zn	Mg	Cu	Cr	Fe	Si	Al
Ε	5.57	4.01	2.00	4.76	0.40	_	83.26
Т	63.28	16.11	13.07	0.12	0.53	_	6.89

grain of the alloy, a large number of η and T precipitates are formed. These precipitates might provide the sites of heterogeneous nucleation of E dispersoids during the heat treatment at higher temperature.

In order to reveal that heat treatment after $250 \,^{\circ}\text{C}$ for $72 \,\text{h}$ can provide the sites of heterogeneous nucleation of *E* dispersoids, the alloy after heat treatment of (470 $^{\circ}\text{C}$, 24 h, WQ) + (250 $^{\circ}\text{C}$, 72 h, WQ) was homogenized again at 470 $^{\circ}\text{C}$ for 24 h, and then water quenched. The

TEM images of the precipitates distribution from the boundary to the center of grain of the 7475 aluminum alloy after the heat treatment of (470 °C, 24 h, WQ) + (250 °C, 72 h, WQ) + (470 °C, 24 h, WQ) are shown in Fig. 10. There are many ultrafine particles in the center of the grain of the alloy, where there is no *E* dispersoid precipitated only after 470 °C, 24 h, WQ. EDS results show that these ultrafine particles in the center zone of the grain are also *E* dispersoids, as given in Table 7. This indicates that a large number of η and *T* precipitates in the center of grain promote the heterogeneous nucleation of *E* dispersoids during the subsequent heat treatment at 470 °C for 24 h.

In order to observe the sites of heterogeneous nucleation of *E* dispersoids, the heat treatment at 400 °C for 0.5 h followed by water quenching is adopted after (470 °C, 24 h, WQ) + (250 °C, 72 h,

WQ) to prevent the particles precipitated at 250 °C for 72 h from dissolving back into the matrix. Figure 11 shows the STEM micrographs and EDSmapping results of the 7475 aluminum alloy

after (470 °C, 24 h, WQ) + (250 °C, 72 h, WQ) + (400 °C, 0.5 h, WQ). It can be observed that there are some small *E* phase particles on the interface of the *T* phase. The distributions of Cr, Cu, Mg and Zn



Fig. 10 TEM images of precipitates distribution in alloy after (470 °C, 24 h, WQ) + (250 °C, 72 h, WQ) + (470 °C, 24 h, WQ): (a) GB; (b–e) Away from GB in turn; (f) 25 µm away from GB; (g) Center of grain

Table 7 Chemical compositions obtained from EDS measurements in Fig. 10 (wt.%)								
Point	Zn	Mg	Cu	Cr	Fe	Si	Al	
1	7.82	4.99	3.74	5.03	0.26	_	78.16	
2	6.81	3.49	3.29	2.17	0.25	0.01	83.98	



Fig. 11 STEM micrograph (a) and EDS mappings (b-f) of alloy after (470 °C, 24 h, WQ) + (250 °C, 72 h, WQ) + (400 °C, 0.5 h, WQ): (b) Cr; (c) Cu; (d) Mg; (e) Zn; (f) Al

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corresponding to the microstructure in Fig. 11(a) are shown in Figs. 11(b–e), respectively. The EDS results of E and T phase in Fig. 11(a) are given in Table 8. These experimental results prove that the precipitates formed during the heat treatment at 250 °C for 72 h can provide the sites of heterogeneous nucleation of E dispersoids during the subsequent heat treatment. The E dispersoids will gradually grow bigger during the heat treatment at treatment at higher temperature.

Table 8 Chemical compositions obtained from EDSmeasurements in Fig. 11 (wt.%)

Phase	Zn	Mg	Cu	Cr	Fe	Si	Al
Ε	11.62	3.30	2.97	1.99	0.47	0.16	79.48
Т	24.44	6.24	9.29	0.06	0.54	0.09	59.35

In summary, it is proven that the role of the first heat treatment at 470 °C for 24 h is to accelerate the diffusion of Mg and the concentration of Mg and Cr in the center of grain or dendrite arm can meet the critical concentration for the formation of E dispersoids. Although there are sufficient Mg and Cr in the center of cross-section of dendrite arm in DC ingot of 7475 aluminum alloy after homogenization treatment at 470 °C for 24 h, it is difficult to form E dispersoids only by homogeneous nucleation. The role of the second heat treatment at 250 °C for 72 h is to precipitate η and T phase particles to provide the sites of the heterogeneous nucleation of E dispersoids. The role of the third heat treatment at 470 °C for 24 h is to promote the growth of the *E* dispersoids.

4 Conclusions

(1) The actual effective partition coefficients of Mg and Cr across the dendrite arm section in the DC ingot of 7475 aluminum alloy during the solidification process are 0.650 and 1.392, respectively. In the center of cross-section, the concentrations of Mg and Cr are less than 1.0 wt.% and greater than 0.3 wt.%, respectively.

(2) After homogenization treatment at 470 °C for 24 h, Mg in the grain of 7475 aluminum alloy diffuses uniformly, but Cr hardly diffuses. The distribution of the *E* dispersoids in its grain is heterogeneous, with a shell-like feature. Both the concentrations of Cr and Mg and the heterogeneous nucleation sites are the determinants for the

formation and distribution of E dispersoids simultaneously.

(3) Although there are sufficient Mg and Cr in the center of cross-section of dendrite arm in DC ingot of 7475 aluminum alloy after treatment at 470 °C for 24 h, it is difficult to form *E* dispersoids by homogeneous nucleation. The heat treatment at 250 °C for 72 h provides a large number of the sites of heterogeneous nucleation for *E* dispersoids, which will form during the subsequent heat treatment at higher temperature.

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DC 铸造 7475 铝合金中含铬弥散相非均匀分布的机理

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摘 要: 研究 7475 铝合金半连续铸锭中枝晶臂横截面上镁元素和铬元素的实际有效分配系数。同时,通过微观 结构分析,揭示 *E* (Al₁₈Mg₃Cr₂)弥散相在半连续铸锭中非均匀分布的机理。结果表明,镁和铬的实际有效分配系 数分别为 0.650 和 1.392,这些系数表明合金枝晶臂横截面半径方向上镁和铬的非均匀分布。经 470 °C 均匀化处 理 24 h 后,镁元素扩散均匀,而铬元素几乎不扩散。镁和铬的浓度以及合金中的异质形核位置同时是形成 *E* 弥散 相的决定因素。250 °C 热处理 72 h 为大量细小的 *E* 弥散相在随后高温热处理过程中合金枝晶臂横截面心部的形 成提供异质形核位置。

关键词: 7475 铝合金; 半连续铸锭; Al18Mg3Cr2 弥散相; 非均匀分布; 有效分配系数