Effect of cooling rate on the size fluctuation of V-containing phases in Al-V master alloys

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Abstract. To investigate the relationship between the solidification cooling rate and the size fluctuation of different V-containing phases, Al-4 wt. % V master alloys with various V-containing constituents were prepared at different cooling rates carried out by a wedge-shaped water-cooled copper mold, a graphite mold and a refractory mold at 1050℃, respectively. The variance of lognormal density function was used to characterize the size fluctuation of V-containing phases quantitatively. The results show that the largest size difference of both $Al_{10}V$ phases and Al_3V phases is simultaneously present in the ingot prepared by the average solidification cooling rate of 2℃·s^{-1}, while the smallest difference of $Al_{10}V$ phases in size is present in the ingot prepared by the average solidification cooling rate of 271℃·s^{-1}. The size fluctuation of the Al_3V phases first increases slightly and then decreases with increasing average solidification cooling rate in the range of 30~195℃·s^{-1}.

Keywords: master alloy, V-containing phase, cooling rate, size fluctuation.

1 Introduction

Minor addition of vanadium (V) inhibits the recrystallization behaviors of Al-Mg-Si alloys. The number of reports about the effects of V content on the mechanical properties of aluminum alloys was large [1-2], but the focus on the evolution of V-containing phases should be discussed in details. Further, V-containing constituents with different types, morphologies, sizes, size fluctuations *etc.* in Al-V master alloys brought about different addition effects [3]. Most of these parameters, such as size and size fluctuation, were influenced by the cooling rates largely during the preparation of Al-V master alloys [4].

In this paper, various Al-4 wt. % V master alloys with different cooling rates were prepared, and the size and the size fluctuation of V-containing phases were characterized quantitatively. Then, the relationship between the solidification cooling rate and the size fluctuation of different V-containing phases was established to determine the preparation parameters for the Al-V master alloys with the V-containing phases in uniform size.

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2 Experiment

Al-50 wt. % V alloy particles were added into high purity aluminum (99.99 wt.%) at 800℃ to produce the Al-4 wt. % V master alloys. Then, the melt was heated to 1050℃, and mechanical stirring was used to promote dissolution. After being kept at 1050℃ for 5 min, the melt was poured into a wedge-shaped water-cooled copper mold, a graphite mold and a refractory mold, respectively.

To obtain the cooling rates at different positions of the wedge-shaped water-cooled copper mold, four K-type linear thermocouples were arranged at different depths along the centerline of the cavity, as shown in Fig. 1(a). Fig. 1(b) shows a schematic diagram of both the refractory mold and graphite mold. One K-type linear thermocouple was arranged 15 mm from the bottom of the mold along the centerline of the cavity. All these thermocouples were connected to the HIOKI data collector for the temperature recording.

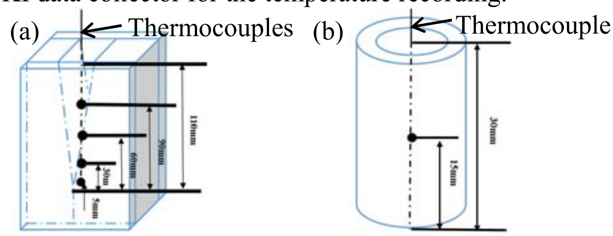

Fig. 1. Diagram of casting molds with the locations of thermocouples: (a) Water-cooled copper mold, (b) Graphite mold and refractory mold.

The metallographic samples prepared by both the graphite mold and refractory mold were cut at the center of the cylindrical ingots. For the wedge-shaped ingot prepared by the mold shown in Fig. 1(a), 7 metallographic samples were cut every 10 mm from the tip of the wedge-shaped ingot along its centerline. The microstructures were observed by three-dimensional laser confocal microscopy (OLYMPUS LEXT OLS4100). The size of different V-containing constituents were calculated by ImagePro Plus software.

3 Results and discussion

Fig. 2 shows the as-cast microstructures of the center of the ingots prepared by the graphite mold and refractory mold and the microstructures of the wedge-shaped ingot at 70 mm, 60 mm, 50 mm, 40 mm, 30 mm, 20 mm and 10 mm away from the tip prepared at 1050℃. The Al_3V and $Al_{10}V$ phases [3-4] are present in the ingots shown in Fig. 2, and the cooling rates have significant effects on their type, size and size fluctuation in different Al-4 wt. % V master alloy ingots.The average solidification cooling rates (v_e) at positions 5 mm, 30 mm, 60 mm and 90 mm away from the tip of the "wedge-shaped" mold can be obtained first according to the temperature dependence on time measured by the thermocouples shown in Fig. 1(a) during solidification at 1050℃. Consequently, the dependence of v_e on the distance away from the tip of the "wedge-shaped" mold along its centerline at 1050℃ can be obtained by fitting calculation, as a result of equation (1). Therefore, the value of v_e at positions of 70 mm, 60 mm, 50 mm, 40 mm, 30 mm, 20 mm and 10 mm away from the tip of the "wedge-shaped" mold at 1050℃ can be obtained by Eq. (1) and shown in Table 1.

The size distribution of the $Al_{10}V$ phases in Figs. 2 (a), (b) and (i) can be induced by a normalization method [5] as shown in Fig. 3. That is, the horizontal coordinate-axis in Fig. 3 is the ratio of the area of each $Al_{10}V$ phase (A) to the average area of $Al_{10}V$ phases (A_{evg}). Then, the interval length of the abscissa is taken as 0.1. Accordingly, the ratio of the number of $Al_{10}V$ phases (N) in the corresponding interval to the total number (N_{tol}) of $Al_{10}V$ phases is determined the ordinate in Fig. 3. As a result, according to Fig. 3, the size distribution of the $Al_{10}V$ phase conforms to a lognormal distribution [5]. The corresponding lognormal distribution probability density function can be described by equation (2) [5].

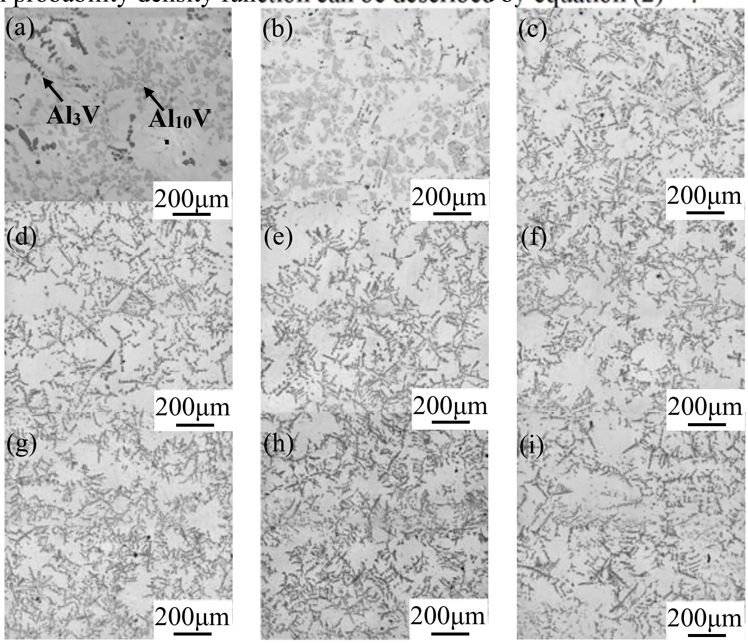

Fig. 2. As-cast microstructures of ingots prepared by different molds at 1050℃: (a) Refractory mold (Center), (b) Graphite mold (Center), and "wedge-shaped" copper mold with different distances away from the tip: (c) 70 mm, (d) 60 mm, (e) 50 mm, (f) 40 mm, (g) 30 mm, (h) 20 mm, (i) 10 mm.

Table 1. Mean solidification cooling rates inside the cavities of casting molds at different locations after the melt was poured at 1050℃.

Casting mold	Distance from the tip of the wedge-shaped copper mold along the centerline of the cavity/mm							Graphite mold	Refractory mold
Location of temperature testing	10	20	30	40	50	60	70	Center	Center
Mean solidification cooling rate/℃·s⁻¹	271	195	139	100	71	51	36	30	2

$$v_e = -2.412 + 379.299 \times e^{(-d/30.498)} \quad (1)$$

in which, v_e is the average solidification cooling rate, ℃·s^{-1}; and d is the distance from the tip of the "wedge-shaped" mold along its centerline, mm.

$$y = y_0 + \frac{B}{\sqrt{2\pi}wx} e^{\frac{-[\ln\frac{x}{x_c}]^2}{2w^2}} \quad (2)$$

in which, x is the ratio of A to A_{evg} (A/A_{evg}); y is the ratio of N value in the corresponding interval to N_{tol} (N/N_{tol}); y_0 is the position parameter; w is the scale; B is the concentration; and x_c is the median of lognormal distribution.

Correspondingly, the variance (D(X)) of the lognormal distribution density function can be described as equation (3) by comparing Eq. (2) with the standard lognormal distribution density function [6]. D(X) can considered to characterize the size distribution of $Al_{10}V$ phases. The smaller its value is, the smaller the size difference of the $Al_{10}V$ phase.

$$D(X) = B^2(e^{w^2} - 1)e^{2\ln x_c + w^2} \quad (3)$$

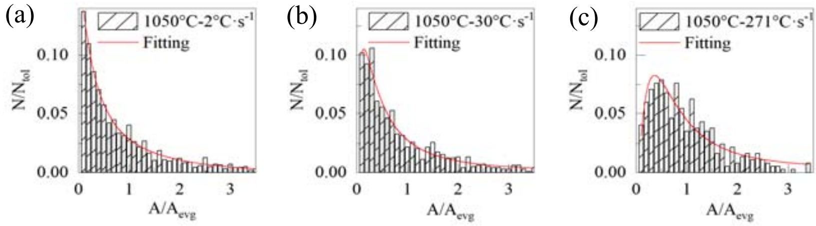

Fig. 3. Dependence of size distribution of $Al_{10}V$ phases on v_e in the ingots prepared at 1050℃: (a) 2℃·s^{-1}, (b) 30℃·s^{-1}, (c) 271℃·s^{-1}.

These parameters (y_0, w, B, x_c, D(X)) can be obtained by fitting the dependence of N/N_{tol} on A/A_{evg} shown in Fig. 3 and summarized in Table 2. As a result, the influence of v_e on the size fluctuation of $Al_{10}V$ phases can be illustrated by the value of D(X) in Table 2. That is, the size difference of the $Al_{10}V$ phase is largest when v_e is as low as 2℃·s^{-1} based on the largest D(X) value in Table 2. With increasing v_e to 30℃·s^{-1}, the size difference of the $Al_{10}V$ phase largely decreases. After the value of v_e increases to 30℃·s^{-1}~195℃·s^{-1}, no $Al_{10}V$ phase is present in the ingot. However, when v_e continues to increase to 271℃·s^{-1}, $Al_{10}V$ phases are present again. Their size difference decreases continuously, which might be because the large solidification cooling rate results in insufficient time for the growth of $Al_{10}V$ phases after their nucleation.

Table 2. Parameters of the lognormal distribution function of $Al_{10}V$ phases in size in the ingots prepared by different mean solidification cooling rates (v_e) at 1050℃.

Average solidification cooling rate/℃·s^{-1}	y_0	x_c	w	B	D(X)	R^2	RMSE
2	-0.003	0.79	1.62	0.13	1.862	0.99	0.004
30	0.001	0.58	1.20	0.09	0.037	0.96	0.006
271	0.004	0.80	0.89	0.09	0.014	0.90	0.009

Similarly, the effect of v_e on the size fluctuation of Al_3V phases can be analyzed in the same way, and thus, the corresponding parameters can be summarized in Table 3. Based on D(X) value shown in Table 3, it can be found that the largest size difference of the Al_3V phases is present when v_e is as low as 2℃·s^{-1} as well. The reason why the size differences of both the $Al_{10}V$ phase and Al_3V phase are present in the ingot prepared at v_e value of 2℃·s^{-1} might be that there should be a long time for the growth of both the $Al_{10}V$ phase and Al_3V phase due to the low cooling rate (2℃·s^{-1}) based on Al-V binary phases diagram [7], and thus, the concentration fluctuation and temperature difference during solidification should influence the growth of both the $Al_{10}V$ phase and Al_3V phase in size largely, leading to the largest size difference at last.

When v_e is in the range of 30℃·s^{-1}~195℃·s^{-1}, the size difference of the Al_3V phase slightly increases first and then decreases with increasing v_e, and the maximum size difference of the Al_3V phase is present at the v_e value of 139℃·s^{-1}. The reasons should be divided into two parts: (1) v_e is in the range of 30℃·s^{-1}~139℃·s^{-1}: There still should be some time for the growth of the Al_3V phase, as a result of the difference in the size distribution for the Al_3V phase in the growth process. Meanwhile, the increase in v_e promotes the number of Al_3V phases increasing according to Fig. 2. As a result, the number of the Al_3V phases with different sizes increases to a certain extent based on the above two reasons. (2) v_e increases to as large as 195℃·s^{-1}: Although the number of Al_3V phases will increase further due to the larger v_e, the larger v_e value should lead to no time for the growth of Al_3V phases at the same time. As a result, the size difference of the Al_3V phase decreases. When v_e continuously increases to 271℃·s^{-1}, the size difference of Al_3V phases is slightly larger than that of Al_3V

phases present in the ingots prepared under the condition of v_e among 30℃·s^{-1}~195℃·s^{-1}. The reason might be that non-homogeneous distribution of temperature caused by the large v_e (271℃·s^{-1}) during solidification should bring about the different length of time for the growth of Al_3V phases in different locations, as a result, the size difference of the Al_3V phase increases to some extent finally.

Table 3. Parameters of the lognormal distribution function of the Al_3V phase in size in the ingots prepared by different mean solidification cooling rates (v_e) at 1050℃.

V_e /℃·s^{-1}	2	30	36	51	71	100	139	195	271
y_0	-0.002	0.006	-0.001	-0.000	-0.000	-0.002	-0.004	-0.000	-0.003
x_c	0.80	0.51	0.78	0.78	0.77	0.85	0.80	0.71	0.88
w	1.54	0.72	0.95	0.87	0.94	0.95	0.99	0.82	0.98
B	0.13	0.08	0.11	0.10	0.10	0.11	0.12	0.10	0.12
D(X)	1.126	0.002	0.027	0.015	0.020	0.032	0.041	0.010	0.047
R^2	0.99	0.97	0.91	0.90	0.94	0.92	0.96	0.98	0.96
RMSE	0.002	0.006	0.008	0.009	0.008	0.008	0.006	0.005	0.006

4 Conclusion

The largest size fluctuation of both the $Al_{10}V$ phase and Al_3V phase is present in the ingot prepared by the average solidification cooling rate of 2℃·s^{-1} at 1050℃. The size of the $Al_{10}V$ phases is most uniform in the ingot prepared by the average solidification cooling rate of 271℃·s^{-1}. While, to produce the Al-V master alloys with Al_3V phases in uniform size at 1050℃, the average solidification cooling rate should be determined as 36℃·s^{-1}~195℃·s^{-1}.

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