Synergistic effects of composition and heat treatment on microstructure and properties of vacuum die cast Al-Si-Mg-Mn alloys

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Abstract: The purpose of this study was to prepare high-quality Al-Si-Mg-Mn alloy with a good combination of strength and ductility employing the vacuum-assisted high-pressure die cast process. An orthogonal study of heat treatments was conducted to design an optimized T6 heat treatment process for both Al-10%Si-0.3%Mg-Mn and Al-11%Si-0.6%Mg-Mn alloys. The results demonstrate that no obvious blisters and warpage were observed in these two alloys with solid solution treatment. After the optimal T6 heat treatment of 530°C×3h + 165°C×6h, Al-11%Si-0.6%Mg-Mn alloy has better mechanical properties, of which tensile strength, yield strength and elongation reached 377.3 MPa, 307.8 MPa and 9%, respectively. The improvement of mechanical properties can be attributed to the high density of needle-like β'' (Mg₂Si₆) precipitation after aging treatment and the fine and spherical eutectic Si particles uniformly distributed in the α -Al matrix.

Key words: heat treatment; vacuum die casting; Al-Si-Mg-Mn alloy; microstructure; mechanical properties

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High pressure die casting has been widely applied in the manufacturing of high-quality aluminum alloy components in the automotive industry. However, conventional HPDC components have an inherent porosity defect that limits a further solid solution treatment at high temperature. Previous works^[1-5] focus on the application of low-temperature and short-time solid solution treatment in order to improve the mechanical properties without the surface blisters. S. Shivkumar et al.^[1] investigated the solution treatment of an Al-Si-Mg alloy at 540 °C in a time range from 10 min to 100 h and found that eutectic silicon could be spheroidized within ten minutes. Lumley et al.^[2] reported that A360 die castings were obviously strengthened even if the solution treatment stage was carried out at 450 °C for 15 min. Furthermore, Srivastava et al.^[3] proposed that the homogeneity in compositions could be achieved with a solution treatment at 480 °C for only 4 min in a Al-9%Si-Mg-Mn die casting alloy, and the yield strength increased to 200 MPa.

To meet the industrial demand for the higher-property

HPDC components, vacuum assisted high pressure die casting (VA-HPDC) has been developed. By evacuating air from the injection chamber and die cavity, VA-HPDC can considerably reduce the porosity defects, which makes the castings capable of higher-temperature and long-time heat treatment. However, few studies have investigated the heat treatment effect on the mechanical properties of vacuum assisted high pressure die castings.

This work investigated two kinds of Al-Si-Mg-Mn aluminum alloy, i.e. Al-10%Si-0.3%Mg-Mn and Al-11%Si-0.6%Mg-Mn, for the complex automotive components requiring high strength and ductility. The orthogonal experiment method was applied to optimize the heat treatment procedure. Moreover, the microstructures and tensile properties of the designed alloys under the optimized heat treatment conditions were also studied.

1 Experimental procedure

The chemical compositions of the experimental alloys measured by an optical emission spectroscopy (OES) are presented in Table 1. The ingots were melted in an electromagnetic induction furnace (ALM-200) maintaining a melt temperature of 720 °C, and then degassed for 15 min by Argon (Ar). Afterwards, the temperature of the melt was kept at 700 °C for casting.

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Table 1: Chemical composition of alloys (wt.%)

Alloy	Si	Mg	Mn	Fe	Cu	Sr	Al
A1	9.45	0.30	0.72	0.09	0.02	0.02	Bal.
A2	11.15	0.66	0.66	0.11	0.02	0.02	Bal.

The mold preheat temperature was 120 °C.

Tensile specimens (ASTM B 557-06), as shown in Fig.1, were cast in a horizontal cold chamber die casting machine (BD-350V5) with a vacuum system. The liquid metal was filled in the injection chamber at 0.2 m·s⁻¹, and then injected into the die cavity at 2.75 m·s⁻¹. The vacuum system started when the feed inlet was completely closed by the piston until the metal liquid fully filled the cavity. During the rapid solidification of the molten metal, the average vacuum was maintained at 90 kPa and the casting pressure was 13.7 MPa.

Fig. 1: Shape and dimensions of tensile specimen

The heat treatment was carried out in an electrical resistance furnace with an accuracy of 1 °C. The orthogonal experiment was designed to study the four factors of T6 heat treatment process including solution temperature (A), solution time (B), aging temperature (C) and aging time (D). Three levels were selected for each factor, as shown in Table 2. The orthogonal test

scheme was based on L₉ (3⁴) table and the repeated experiments were designed to analyze the random error. The quality value of Q was taken as the evaluation standard of mechanical properties, as shown in Eq. (1):

$$Q = \sigma_{UTS} + k \log(E_f) \quad (1)$$

where Q is the quality index in MPa; σ_{UTS} refers to ultimate tensile strength in MPa; E_f refers to elongation in pct.; and k is a material constant which is taken as 150 in this work [6].

Range analysis and variance (ANOVA) analysis were used to investigate the influence of the chosen heat treatment factors on the mechanical properties and to determine the optimal heat treatment process.

The values of $\overline{K}_{jm}$ and R for each factor at different levels are depicted in Table 3. In general, the range R reflects the relative influence on the quality value of Q . Furthermore, the optimal level of each factor

Table 2: Factors and levels of orthogonal experiment

Alloy	A Solution Temperature (°C)	B Solution Time (h)	C Aging Temperature (°C)	D Aging Time (h)
1	490	1	165	2
2	510	2	180	4
3	530	3	195	6

Table 3: Orthogonal design analysis of quality value of Q index

Alloy	Factors	$\overline{K}_1$	$\overline{K}_2$	$\overline{K}_3$	R	f	S	V	F	Significance
A1	A	821.8	825.6	885.1	63.3	2	3774.8	1887.4	133.8	**
	B	827.4	846.1	859.0	31.6	2	756.0	378.0	26.8	**
	C	878.7	834.1	819.7	59.1	2	2844.7	1422.3	100.8	**
	D	834.7	846.5	851.4	16.7	2	221.1	110.5	7.8	*
	Error					9	126.9	14.1		
	Total					17	7723.6	454.3		
A2	A	784.3	857.0	913.8	129.5	2	12635.2	6317.6	329.4	**
	B	835.7	850.9	868.5	32.7	2	805.0	402.5	21.0	**
	C	876.8	846.8	831.6	45.2	2	1587.0	793.5	41.3	**
	D	814.5	846.8	893.8	79.3	2	4765.7	2382.8	124.2	**
	Error					9	172.6	19.2		
	Total					17	19965.6	1174.4		

Note: $F_j > F_{0.05}(2,9) = 4.256$, the significance of the factor j marked as *; $F_j > F_{0.01}(2,9) = 8.022$, the significance of the factor j marked as **.

can be determined by comparing K_{jm} , which is the sum of the indicators (the quality value of Q) of the repeated experiments corresponding to factor j and level m . The range R of each factor j ($j=A, B, C, D$) is calculated by Eq. (2):

$$\overline{K_{jm}} = K_{jm}/3$$

$$R_j = \max\{\overline{K_{jm}}\} - \min\{\overline{K_{jm}}\} (m=1,2,3) \quad (2)$$

where $\overline{K_{jm}}$ is the average of K_{jm} in MPa; R is the difference between the maximum and the minimum of $\overline{K_{jm}}$ in MPa.

Variance (ANOVA) analysis was conducted on the basis of the degree of freedom (f), the sum of the squares (S), variance (V), and F-value (F). In these parameters, the difference between the values of each factor represents the relative influence degree. The larger these parameters, the stronger the influence. The formulae applied in the study are:

$$T = \sum_{i=1}^n \sum_{t=1}^s Q_{it}$$

$$S_T = \sum_{i=1}^n \sum_{t=1}^s Q_{it}^2 - \frac{T^2}{ns}; f_T = ns - 1; V_T = \frac{S_T}{f_T}$$

$$S_e = \sum_{i=1}^n \sum_{t=1}^s Q_{it}^2 - \frac{1}{s} \sum_{i=1}^n (\sum_{t=1}^s Q_{it})^2; f_e = n(s-1); V_e = \frac{S_e}{f_e}$$

$$S_j = \frac{1}{r} \sum_{i=1}^m K_{ji}^2 - \frac{T^2}{ns}; f_j = m-1; V_j = \frac{S_j}{f_j}; F_j = \frac{V_j}{V_e} \quad (3)$$

where n , which is 9 in this work, is the experiment number in each repeated test; m is the number of the levels; s is the number of the repeated tests' times; V_j and V_e are the variance of factors and error, respectively. The last column of Table 3 exhibits the result of the significance test (F-test) of each factor.

Tensile testing for both the as-cast and the T6-treated specimens was carried out at a strain rate of $1 \text{ mm} \cdot \text{min}^{-1}$ on an electronic universal testing machine (CMT5105, SANS) at

room temperature. The specimens for the optical microscope (OM, BX-60M) and the scanning electron microscope (SEM, XL30, Philips) to analyze the microstructure were polished and etched with 0.5% HF. The precipitates in the T6-treated samples were also analyzed using a transmission electron microscope (TEM, G2 T20, Fei Tecnai) equipped with Energy Dispersive X-ray Spectrometer (EDS).

2 Results and discussion

The variance (ANOVA) analysis and the orthogonal results of the repeated tests are listed in Tables 3 and 4, respectively. A more distinct presentation of the effects of these factors for each alloy is shown in Fig. 2. It is evident that all the selected four factors have a significant effect on the mechanical properties at a 95% confidence limit through the F-test in Table 3, while a random error plays a negligible role. The most important influence factor on the mechanical properties of the two alloys is the solution temperature, accounting for the obviously higher variance than that of other factors in the experimental data. Therefore, it can be concluded that the influence sequence of these factors for Al alloy is $A > C > B > D$, while for A2 alloy it is $A > D > C > B$. Meanwhile, these influence sequences on mechanical properties of two alloys are consistent with the R value's order.

From Fig. 2, it can be easily seen that the difference between the values at different levels of solution temperature is greater than that of the other three factors in these two alloys. All of the four chosen factors in the two alloys exhibit the same variation trend in R value. According to the range analysis, the optimal T6 heat treatment parameters combination of the two alloys should be $A_3B_3C_1D_3$, that is $530^\circ\text{C} \times 3\text{h} + 165^\circ\text{C} \times 6\text{h}$.

The macroscopic metallographic observation in Fig. 3 proved that no obvious blisters and warpage appeared in the two alloys with $530^\circ\text{C} \times 3\text{h}$ solid-solution treatment. There is no doubt that with the assistance of the vacuum system, less air was trapped into the melt during the HPDC process, which

Table 4: Results of orthogonal tests of two designed alloys

No.	Factors				A1(Al-10%Si-0.3%Mg-Mn)		A2(Al-11%Si-0.6%Mg-Mn)	
	A ($^\circ\text{C}$)	B (h)	C ($^\circ\text{C}$)	D (h)	Q_1 (MPa)	Q_2 (MPa)	Q_1 (MPa)	Q_2 (MPa)
1	490	1	165	2	414.0	416.1	372.1	384.1
2	490	2	180	4	404.2	411.8	386.8	386.9
3	490	3	195	6	414.8	404.5	408.4	414.6
4	510	1	180	6	402.5	403.5	441.2	437.0
5	510	2	195	2	396.0	397.6	396.8	402.1
6	510	3	165	4	441.1	436.2	443.7	450.3
7	530	1	195	4	425.6	420.5	436.9	435.9
8	530	2	165	6	462.1	466.7	488.9	491.2
9	530	3	180	2	438.5	441.8	440.1	448.3

means the VA-HPDC components can be heat treated at the high temperature with a relatively long period.

The microstructures of the as-cast and the optimized T6-treated alloys are shown in Fig. 4. The as-cast microstructure of these two alloys mainly consisted of four phases: α -Al grains, eutectic silicon particles, Mg_2Si precipitates and Al-(Mn,Fe)-Si particles (Figs. 4a and 4b), confirmed by EDS in SEM. The rosette-like α -Al grains were surrounded by the continuous network of eutectic regions (darker area) and the precipitated phases were mostly observed in the eutectics. Due to the rapid cooling rate, the eutectic silicon showed a fine and rod-like morphology, and the α -Al dendrites were significantly suppressed. Also, the size of Mg_2Si and Al-(Mn,Fe)-Si precipitates were considerably reduced, but the Mg_2Si precipitation was more easily observed with the increased addition of Si and Mg content in A2 alloy. After the optimized T6 treatment (Figs. 4c and 4d), the eutectic regions became less distinct with the spheroidization of eutectic silicon particles and the dissolution of the Mg_2Si precipitates, but the Al-(Mn,Fe)-Si particles could still be found in the Al matrix. With the addition of a sufficient amount of Mn, blocky α -type Al-(Mn,Fe)-Si instead of plate-like β -type Al_3FeSi has been

reported to be beneficial to the mechanical properties due to the decrease of stress concentration^[7].

The tensile properties of the as-cast and the optimized T6-treated alloys are summarized in Table 5. It is evident that the tensile properties are better than that of others in the orthogonal tests (Table 4), which further confirms that the process of $530^\circ C \times 3h + 165^\circ C \times 6h$ is the optimal T6 treatment for these two alloys. In addition, although there is almost no difference in the tensile strength of both alloys in the as-cast condition, the UTS and YS of A2 alloy after T6 treatment are 377.3 MPa and 307.8 MPa, respectively, which are much higher than that of A1 alloy. Meanwhile, the elongation of T6-treated A2 alloy can maintain about 9%, which is comparable to the A1 alloy. Obviously, through the regulation of Si and Mg content and the optimized T6 treatment, the tensile property of the A2 alloy, especially the yield strength, was significantly increased and the Q value could reach 519 MPa.

To further study the strengthening mechanism, TEM analysis (Fig. 5) of the A2 alloy was performed. Figure 5(a) shows that lots of needle-like precipitates exist in the α -Al matrix and the contents of Mg and Si of these precipitates are nearly equal in EDS. In Fig. 5(b), it is observed that the length of these

Fig. 2: Effects of solution treatment temperature, solution treatment time, aging temperature and aging time on Q index of alloys

Fig. 3: Macroscopic structure of two alloys with $530^\circ C \times 3h$ solid-solution treatment: (a) A1 alloy; (b) A2 alloy

Fig. 4: Optical microstructure of as-cast and T6 heat treated (530°C×3h+165°C×6h) two alloys and EDS of Al-(Mn,Fe)-Si particles and Mg₂Si precipitate: (a) as-cast A1 alloy; (b) as-cast A2 alloy; (c) T6 A1 alloy; (d) T6 A2 alloy; (e) EDS results of Al-(Mn,Fe)-Si particles; (f) EDS results of Mg₂Si precipitate

Table 5: Mechanical properties of as-cast and T6 optimized heat treatment of both alloys

Alloy	Condition	R_m (MPa)	$R_{p0.2}$ (MPa)	A (%)	Q (MPa)
A1 (Al-10%Si-0.3%Mg-Mn)	As-cast	302.7	156.5	8.2	439.8
	T6	330.2	255.8	10.6	483.9
A2 (Al-11%Si-0.6%Mg-Mn)	As-cast	302.9	178.5	5.7	416.3
	T6	377.5	307.8	8.9	519.9

needle-like precipitates is 30–100 nm and the width is 3–5 nm. It is well known that the precipitation sequence of the Al-Si-Mg alloy is SS(super-saturated solid solution)→GP →β'' (needle)→β' (rod)→β (plate) [8–12]. The shape and size of these precipitates are consistent with the β'' (Mg₅Si₆) phase reported in previous studies [12–15]. Moreover, β'' phase was considered to be the dominant strengthening phase responsible for the peak-hardening effect [16]. From Fig. 5(a), with the increased addition of Si and Mg content in A2 alloy, it was found that a large amount of β'' phase uniformly distributed in the α-Al matrix, which could be the main reason for the increase of the tensile strength after the optimized T6 treatment. The higher solution treatment temperature (530 °C) with longer solution time (3 h) promoted the dissolution and re-diffusion of the Mg₂Si precipitates and increased the number of vacancy-solute clusters in the aluminum lattice. This kind of heat treatment could not only obviously accelerate the precipitation of β'' phase, but also

enhance the uniform distribution of the precipitated particles, improving the strength of the alloys.

The fracture morphologies of the as-cast and the optimized T6-treated A2 alloy are shown in Fig. 6. The fracture feature of the as-cast A2 alloy is basically intergranular fracture. It could be observed that the α-Al grains in the fracture region maintained the rosette-like shape (Fig. 6a, arrow B). The crack propagated mainly along the brittle eutectic regions (Fig. 6a, arrow A) and bypassed the ductile α-Al grains. During tensile testing, the eutectic silicon phases were prone to crack under a high stress and instantaneously reduced the load-bearing area in the eutectic regions. Since these brittle eutectic regions are more or less interconnected, the crack could expand rapidly through the eutectic regions, which is detrimental to the plasticity of the alloy. After T6 treatment, however, the serrated edge of α-Al grains and the broken eutectic silicon particles were observed in the fracture region (Fig. 6b). The spherical eutectic silicon

Fig. 5: TEM image of A2 alloy after T6 optimal heat treatment: (a) distribution of precipitated particles in α-Al matrix; (b) needle-like β'' (Mg₅Si₆) precipitates observed by TEM from [001]_{Al} orientation

Fig. 6: Microstructures in near fracture of as-cast and T6 optimized heat treatment of A2 alloy: (a) as-cast A2 alloy fracture: eutectic region (arrow A), edge of α-Al grains (arrow B); (b) T6 A2 alloy fracture: eutectic Si particles (arrow A), edge of α-Al grains (arrow B)

particles considerably reduced the stress concentration. When the discontinuous spheroidized silicon particles are broken under the stress, the fracture process was less instantaneous, as the cracks mainly propagated through the more-ductile aluminum matrix. The spheroidization of eutectic silicon can compensate for the stresses created by the formation of β'' phase. These changes might be contributed to the improved strength and ductility for these two alloys after T6 heat treatment.

3 Conclusions

The synergistic effects of composition and heat treatment on the microstructures and mechanical properties of VA-HPDC Al-Si-Mg-Mn aluminum alloys were investigated. The following conclusions can be summarized based on the results obtained:

(1) A systematical analysis of optimal T6 heat treatment process was conducted by the means of the orthogonal experiment method, and an optimized T6 treatment process for VA-HPDC Al-Si-Mg-Mn alloys was determined: $530^{\circ}\text{C} \times 3\text{h}$ solid solution and $165^{\circ}\text{C} \times 6\text{h}$ artificial aging.

(2) For Al-Si-Mg-Mn die-casting alloy, the small adjustment of Si and Mg content coupled with the optimized T6 treatment resulted in remarkable improvement in the mechanical properties. The tensile strength, yield strength and elongation reached 377.3MPa, 307.8 MPa and 9%, respectively in Al-11%Si-0.6%Mg-Mn alloy.

(3) The needle-like $\beta''(\text{Mg}_5\text{Si}_6)$ precipitates uniformly distributed in the α -Al matrix and the eutectic silicon turned to fragment and spheroidized, which were the main reason for the two alloys to enhance the mechanical properties.

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