The influence of thermal treatment on microstructure and mechanical properties of the Si-rich Al-Mg-Si-Sc-Zr alloy

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Abstract: The paper studies the Al-Mg-Si alloy that does not contain scandium and zirconium, as well as the siliconrich Al-Mg-Si-Sc-Zr alloy. Multistage thermal treatment was carried out for the Al0.3Mg1Si0.3Sc0.15Zr alloy, which included annealing at a temperature of 440 °C for 8 h, high-temperature annealing at 500 °C for 0.5 h, and artificial aging at a temperature of 180 °C with soaking for 5 h. The Al0.3Mg1Si alloy was annealed at 550 °C for 8 h, and then artificial aging was carried out similarly to the alloy with Sc and Zr additives. To study the fine structure, transmission electron microscopy was used. In the as-cast condition and after each stage of thermal treatment, the mechanical properties of the alloys were determined. It has been found that in an alloy doped with Sc and Zr, the formation of Al₃Sc particles occurs already at the stage of formation of the cast structure. During subsequent artificial aging, the supersaturated solid solution decomposes with the formation of β " (Mg₅Si₆) particles improving mechanical properties. It has been found that in the scandium-containing alloy, fewer β'' (Mg₅Si₆) particles are formed, as a result of which its strength properties are slightly worse than those of the base alloy are. Moreover, these particles are larger than in an alloy that does not contain scandium. This is explained by the fact that complete quenching is impossible for an alloy with scandium additives.

Keywords: Al-Mg-Si-Sc-Zr; excess Si; multistage thermal treatment; artificial aging; TEM; mechanical properties; Al₃Sc; Mg₅Si₆.

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INTRODUCTION

Aluminium alloys combine high ductility, acceptable strength, good weldability, and high corrosion resistance, which determines their high demand in various branches of modern industry. Some of the most widely used aluminium alloys are the Al-Mg-Si system alloys. They received the name "Avial" in Russian literature and found their main application in aviation and the automotive industry. These alloys are capable of strengthening during thermal treatment, due to the release of the Mg₂Si strengthening phase. Note that when the ratio Mg/Si<1.73, there is a silicon excess, and when Mg/Si>1.73, there is a silicon deficiency [1-3]. A silicon excess accelerates the process of formation of the β " phase (Mg₅Si₆) and contributes to its more even release [4].

Aluminium alloys are often additionally doped with Sc. It has a modifying effect on the cast structure, and increases its strength due to the release of highly dispersed Al₃Sc

particles [5-7]. Usually, zirconium is added together with scandium, which thermally stabilises Al₃Sc particles, and increases the efficiency of the cast structure grinding [8; 9]. At the same time, adding scandium to the Al-Mg-Si system does not always lead to an increase in strength properties. This is caused by the fact that scandium and silicon tend to combine into the Sc₂Si₂Al phase, which is not a strengthening phase. An increase in silicon content leads to an increase in the probability of the formation of this phase [10]. Despite this, strengthening Al₃Sc nanoparticles were discovered even in the Al-Mg-Si system alloys, with a high silicon content [11–13]. However, to simultaneously obtain Al₃Sc and β " (Mg₅Si₆) strengthening particles in alloys with excess silicon, multi-stage thermal treatment is required. The following sequence may be one of its options: annealing at 440 °C to precipitate Al₃Sc, quenching at 500 °C for 30 min for partial Mg and Si dissolution, and ageing at 180 °C for 5 h to form \beta" (Mg₅Si₆) [14]. At the same time, the influence of this thermal treatment on

the formation of mechanical properties and microstructure in alloys with the Mg/Si \leq 0.6 ratio has not been previously studied.

The purpose of the study is to investigate the influence of thermal treatment on the formation of the microstructure and mechanical properties of the Al–Mg–Si alloy with excess silicon and additional alloying with Sc and Zr.

METHODS

The work studied the Al0.3Mg1Si0.3Sc0.15Zr alloy in the as-cast condition, as well as after various thermal treatments according to the modes given in Table 1. To assess the influence of scandium and zirconium, similar studies were carried out on the base Al0.3Mg1Si alloy (without Sc and Zr).

The authors carried out casting to a steel mould to ensure the rate of crystallisation and cooling of the cast structure close to real industrial technology. The weight of the cast ingots was 4.5 kg. The following materials were used as a charge for the alloy: A85 aluminium, MG90 magnesium, Al₁₂Si alloy, Al–Sc₂ and Al–Zr₅ alloys. The casting temperature was 720–740 °C. Before pouring the molten metal into the mould, it was refined with carnallite flux, added at the rate of 5 g per 1 kg of charge. After this, scale was removed from the molten metal surface and the metal was poured into a steel mould with a uniform casting time of 40 s. Thermal treatment of the samples was carried out in a muffle electric furnace with water quenching, after which their mechanical properties were determined.

The sizes and morphology of fine particles were studied using transmission electron microscopy (TEM), for the thermal treatment modes given in Table 1. The study was carried out on a Tecnai G2 30 Twin high-resolution microscope equipped with an EDAX energy dispersive X-ray analysis system at an accelerating voltage of 300 kV using standard techniques: light-field, dark-field images and electron microdiffraction. The linear dimensions of the structural elements were determined by direct measurements in the observation plane.

Sample preparation was carried out on Metaserv 250, TenuPol-5, Ultratonic Disk Cutter, PIPS II devices using instrumental methods.

Mechanical properties were determined on a universal testing machine (Zwick/Roell Z050) according to ISO 6892-1 in the as-cast state, and for each thermal treatment step. The dimensions of the samples were selected in accordance with DIN 50125. The properties obtained

during the tests, such as the yield strength ($\sigma_{0.2}$) and the tensile strength (σ_B) were calculated in accordance with GOST 1497-84 and GOST 11150-84.

RESULTS

In the cast structure of the Al0.3Mg1Si0.3Sc0.15Zr alloy, the strengthening phase particles were discovered having different morphologies (Fig. 1). At the same time, in some grains, particles were found that had an equiaxial shape, while in others, they were needle-shaped. Equiaxial particles, the average size of which is 30-40 nm, precipitate relatively uniformly in the grain volume (Fig. 1 a). In the structure of grains where needle-shaped particles are present, zones free from precipitation are observed, their width is ~500 nm; i. e., with relatively dense aggregates of needle-shaped particles, the precipitation of equiaxial dispersoids in such zones is completely absent. Moreover, in the single grain volume, as a rule, only one orientation of needle-shaped particles is observed among all crystallographically equivalent ones (Fig. 1 b). From this, one can conclude that for favorable growth of such particles, an appropriate grain orientation (relative to the temperature gradient during crystallisation) is necessary. From the analysis of the TEM results, it follows that all the observed particles were formed during the discontinuous precipitation of a supersaturated scandium solution during the movement of grain boundaries.

Using TEM, Sc-based phases were detected in a sample after annealing the Al0.3Mg1Si0.3Sc0.15Zr alloy for 8 h at a temperature of 440 °C. They are represented in the form of needle-shaped precipitations with a diameter of up to 40 nm and a length of several microns. Their high volume fraction with a relatively low distribution density should be noted. Moreover, equiaxial particles with a diameter of 20 nm are observed (Fig. 2 c); they are arranged in chains, which, apparently, can be associated with the Al₃Sc phase heterogeneous nucleation on dislocations. Note that needleshaped precipitations, like equiaxial ones, are apparently previously discovered particles of the Al₃Sc type, released as a result of continuous precipitation during cooling of the cast blank (Fig. 2).

In the Al0.3Mg1Si0.3Sc0.15Zr alloy after annealing according to the mode (440 °C, 8 h) + (500 °C, 0.5 h) + + (180 °C, 5 h) (Fig. 3), rod-like precipitations with a length of 500 nm and a diameter of about 200 nm containing Al, Si, Sc, and Zr are observed. Considering the size and morphology of the particles mentioned above, one

Table 1. Scheme of thermal treatment of alloys Таблица 1. Схема термической обработки сплавов

Alloy	Thermal treatment
Al0.3Mg1Si0.3Sc0.15Zr	440 °C, 8 h
	(440 °C, 8 h) + (500 °C, 0.5 h)
	(440 °C, 8 h) + (500 °C, 0.5 h) + (180 °C, 5 h)
Al0.3Mg1Si	(550 °C, 8 h) + (180 °C, 5 h)



Fig. 1. Electron microscopic images of the microstructure of the Al0.3Mg1Si0.3Sc0.15Zr alloy in the as-cast condition: a, b – light-field images Puc. 1. Электронно-микроскопические изображения микроструктуры сплава Al0,3Mg1Si0,3Sc0,15Zr в литом состоянии: a, b – светлопольные изображения

could assume that these particles represent an equilibrium Sc_2Si_2Al phase, which is apparently capable of partially dissolving zirconium. Evidently, even short-term heating at a temperature of 500 °C is enough for its formation. Particles released during discontinuous precipitation after casting, are observed along the grain boundaries (Fig. 3 a, 3 b).

After artificial ageing at 180 °C for 5 h, the β " phase (Mg₅Si₆) becomes the main strengthening phase for an alloy of this composition, which precipitates in the form of rods up to 70 nm long (Fig. 4 a). The quite high volume fraction and the size of β " particle should be noted, as indicated by the pronounced reflexes of this phase in the corresponding electron diffraction patterns (Fig. 4 b). Moreover, despite the rather large sizes of the β " precipitates, they retain their coherence with the aluminium matrix.

In the Al0.3Mg1Si alloy, the precipitation of Si-based phases is observed in the form of irregular-shape polygons, with dimensions up to 300 nm, which are apparently formed during the process of heating for quenching (Fig. 5 a). During artificial ageing at 180 °C for 5 h, highly dispersed needle-shaped precipitations of the Guinier – Preston zone and β " phase are formed (Fig. 5 b, 6 a). In general, the pattern is similar to the Al0.3Mg1Si0.3Sc0.15Zr alloy after three-stage thermal treatment; however, one should note that in the base alloy, more Mg₅Si₆ strengthening particles were found.

Fig. 7 represents the mechanical properties of the alloys under consideration in the as-cast state and after thermal treatment. In the as-cast condition, scandium and zirconium additives can significantly increase the properties of the alloy: yield strength by 32 MPa, tensile strength by 60 MPa. Annealing of Al0.3Mg1Si0.3Sc0.15Zr alloy at 440 °C for 8 h does not lead to a significant change in the yield strength and causes a decrease in the tensile strength by 32 MPa. Three-stage annealing of an alloy doped with Sc and Zr according to the route (440 °C, 8 h) + + (500 °C, 0.5 h) + (180 °C, 5 h), increases the yield strength by 8 MPa and the tensile strength by 17 MPa relative to the as-cast state. However, the base alloy after heating for quenching followed by artificial ageing (550 °C, 8 h) + (180 °C, 5 h) has significantly higher indicators – the yield strength increased by 106 MPa, the tensile strength increased by 70 MPa.

DISCUSSION

It has been found that the Al0.3Mg1Si0.3Sc0.15Zr alloy in the as-cast state has higher strength indicators, which is primarily due to the appearance of the Al₃Sc type particles, both semi-coherent and fully coherent to the aluminium matrix. Note that silicon significantly accelerates discontinuous precipitation, and actually makes the appearance of such particles inevitable [7].

Further annealing (440 °C, 8 h) does not lead to a significant change in the yield strength, and causes a decrease in the tensile strength. This is firstly related to the fact that the essential part of scandium precipitates from the super-saturated solid solution, when the ingot cools during the casting process, after which new strengthening particles are no longer formed. A decrease in strength properties can be caused as well by the release of magnesium in the form of Mg₂Si particles from a supersaturated solid solution, which, according to calculations represented in [15], begins at temperatures below 500 °C.

After final artificial ageing, the strength indicators in the alloy with scandium additives increase slightly, primarily due to the formation of β " particles (Mg₅Si₆), the presence of which is confirmed by TEM data. At the same time, the increase in strength caused by them is small in comparison with the base alloy, and the reason for this is the large size of these particles. In the base alloy, a larger number of β " particles (Mg₅Si₆) are formed, during ageing compared





а – светлопольное изображение;

b, **c** – темнопольные изображения в рефлексе $(110)_{Al_1Sc}$;

d – микроэлектронограмма (ось зоны) $2\overline{15}|_{Al}$

to an alloy containing scandium, which causes a more significant increase in strength. This is related to the fact that in the base alloy, it is possible to carry out complete quenching at a temperature of 550 °C and holding for 8 h. This mode allows magnesium to dissolve and thereby contributes to the release of a much larger number of β " particles (Mg₅Si₆).

At the same time, it is impossible to carry out full quenching in an alloy containing scandium, since it will completely neutralise the strengthening effect of Al₃Sc particles. This is explained by the fact that when heating to a temperature of 550 °C and holding for 8 h, coagulation of particles occurs resulting in that the strengthening effect they cause is completely lost. Thus, scandium additives to Al-Mg-Si alloys cause a significant increase in strength, only in the as-cast state at the stage of the supersaturated solid solution decomposition. Subsequent stages of thermal treatment do not allow precipitating fully both types of strengthening particles. As a result, the mechanical properties of the base alloy are higher than the properties of the alloy containing scandium.



Fig. 3. Electron microscopic images of the Al0.3Mg1Si0.3Sc0.15Zr alloy microstructure after annealing at (440 °C, 8 h) + (500 °C, 0.5 h) + (180 °C, 5 h):
a – light-field image;
b – dark-field image in the (100)Al₃(Sc,Zr) phase reflex;
c – light-field image in transmission scanning mode (STEM);
d – spectrum of characteristic radiation at point 1 in Fig. 3 c
Puc. 3. Электронно-микроскопические изображения микроструктуры сплава Al0,3Mg1Si0,3Sc0,15Zr после отжига по режиму (440 °C, 8 ч) + (500 °C, 0,5 ч) + (180 °C, 5 ч):
a – светлопольное изображение в рефлексе фазы (100)Al₃(Sc,Zr);
c – светлопольное изображение в режиме сканирования на просвет (STEM);

d – спектр характеристического излучения в точке 1 на рис. 3 с



Fig. 4. Electron microscopic images of the Al0.3Mg1Si0.3Sc0.15Zr alloy microstructure after annealing at (440 °C, 8 h) + (500 °C, 0.5 h) + (180 °C, 5 h):

 a – dark-field image in phase reflexes;
 b – microelectronogram, zone axis [001]_{Al}

 Puc. 4. Электронно-микроскопические изображения микроструктуры сплава Al0,3Mg1Si0,3Sc0,15Zr nocлe отжига по режиму (440 °C, 8 ч) + (500 °C, 0,5 ч) + (180 °C, 5 ч):

 a – темнопольное изображение в рефлексах фаз;
 a – темнопольное изображение в рефлексах фаз;

b – микроэлектронограмма, ось зоны [001]_{Al}



Fig. 5. Electron microscopic images of the Al0.3Mg1Si alloy microstructure after annealing at (550 °C, 8 h) + (180 °C, 5 h): a, b – light-field images Puc. 5. Электронно-микроскопические изображения микроструктуры сплава Al0,3Mg1Si после отжига по режиму (550 °C, 8 ч) + (180 °C, 5 ч): a, b – светлопольные изображения



Fig. 6. Electron microscopic images of the Al0.3Mg1Si alloy microstructure after annealing at (550 °C, 8 h) + (180 °C, 5 h):
a – dark-field image in phase reflexes; b – microelectronogram
Puc. 6. Электронно-микроскопические изображения микроструктуры сплава Al0,3Mg1Si после отжига по режиму (550 °C, 8 ч) + (180 °C, 5 ч):
a – темнопольное изображение в рефлексах фаз; b – микроэлектронограмма



Fig. 7. Mechanical properties of the alloys under consideration after thermal treatment: a - yield strength; b - tensile strength

Рис. 7. Механические свойства рассматриваемых сплавов после термической обработки: *a* – предел текучести; *b* – предел прочности

CONCLUSIONS

1. In Al–Mg–Si series alloys with additional Sc and Zr alloying, improved mechanical properties are observed in the as-cast state compared to the base alloy. The main reason for this is the formation of strengthening Al_3Sc type particles precipitated during the cooling process of the cast structure. Solid solution strengthening with scandium and zirconium also contributes to improving mechanical properties.

2. Annealing at 440 °C does not lead to a significant increase in the number of Al_3Sc particles. This is related to the fact that the precipitation of the bulk of scandium from the solid solution occurs at the stage of cooling the ingot. The yield strength at this stage of thermal treatment does not change, but the tensile strength decreases. The decrease in this indicator is associated, primarily, with the magnesium release from the supersaturated solid solution.

3. Artificial ageing at 180 °C for 5 h leads to the formation of strengthening β " particles (Mg₅Si₆), which significantly increase the mechanical properties in both types of alloy. However, in the base alloy, the number of these particles is larger, and the strengthening effect they cause is higher. This is primarily related to the fact that for an alloy without Sc and Zr, complete hardening is possible (550 °C, 8 h) promoting the dissolution of Mg and Si.

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Влияние термической обработки на микроструктуру и механические свойства сплава Al–Mg–Si–Sc–Zr с избытком Si

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Аннотация: В работе исследовался сплав Al–Mg–Si, не содержащий скандия и циркония, а также сплав Al–Mg–Si–Sc–Zr с избытком кремния. Для сплава Al0,3Mg1Si0,3Sc0,15Zr была проведена многоступенчатая термическая обработка, включающая в себя отжиг при температуре 440 °C в течение 8 ч, высокотемпературный отжиг при 500 °C в течение 0,5 ч и искусственное старение при температуре 180 °C с выдержкой 5 ч. Сплав Al0,3Mg1Si подвергался отжигу при 550 °C в течение 8 ч, затем проводилось искусственное старение аналогично сплаву с добавками Sc и Zr. Для изучения тонкой структуры проводилось исследование при помощи просвечивающей электронной микроскопии. В литом состоянии и после каждой стадии термической обработки определялись механические свойства сплавов. Установлено, что в сплаве, легированном Sc и Zr, уже на стадии формирования литой структуры происходит образование частиц Al₃Sc. При последующем искусственном старении происходит распад пересыщенного твердого раствора с образованием частиц β'' (Mg₅Si₆), улучшающих механические свойства. Установлено, что в сплаве с содержанием скандия формируется меньше частиц β''' (Mg₅Si₆), в результате его прочностные свойства несколько хуже, чем у базового сплава. Кроме того, данные частицы крупнее, чем в сплаве, не содержащем скандий. Это объясняется тем, что для сплава со скандиевыми добавками невозможно проведение полноценной закалки.

Ключевые слова: Al–Mg–Si–Sc–Zr; избыток Si; многоступенчатая термическая обработка; искусственное старение; ПЭМ; механические свойства; Al₃Sc; Mg₅Si₆.

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