Crossref **DOI**: 10.31643/2023/6445.37 **Engineering and technology**

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# **Investigation of the Technology of Introducing Li, Mg and Zr Alloys into Aluminum Alloy**

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#### **ABSTRACT**



#### **Introduction**

Among aluminum alloys, the alloys of the Al-Li system are characterized by high strength characteristics, high corrosion resistance, and are easily amenable to any type of welding. It is known that welded aluminum structures give a significant weight advantage compared to riveted structures. For example, welded aluminum structures reduce the weight of aircraft, up to 15-25%. Earlier, in [1] were considered alloys of the Al-Li system, their types, properties, and applications. The simplest alloy 1420, as well as the possibilities of its production at Kazakhstani plants are considered in more detail. This alloy, belonging to the high-level category, has the following chemical composition:

1.9-2.3% Li; 5-6% Mg; 0.09-0.15% Zr; 0.1-0.3% Si, res. Al.

This article is devoted to the study of the initial phase of obtaining alloy 1420, namely, obtaining a primary material with the desired chemical composition. To begin with, let's consider the effect of alloying magnesium, zirconium and lithium on the strength properties of the material.

Magnesium helps to increase the strength of aluminum alloys due to the formation of a solid solution of Al-Mg, as well as by deformation hardening by riveting or cold hardening (Figure 1,2)  $[[2],[3]]$ . As can be seen in Figure 1 and 2, magnesium significantly affects the strength of aluminum. With a magnesium content of 5% (wt.) the yield strength of aluminum increases from 25

MPa to 160MPa. With a magnesium content of 2.5% (wt.) the ultimate strength increases from 180 MPa to 320 MPa with cold rolling up to 80%.

In Al-Mg alloys containing up to 6% Mg, an Al3Mg<sup>2</sup> intermetallic compound is formed with a solid solution of magnesium in aluminum. An increase in the magnesium content by 1% increases the ultimate strength of the alloy by  $\approx$  30 MPa, and the yield strength by  $\approx 20$  MPa. At the same time, the relative elongation decreases slightly and is within the range of 30-35% [[4], [5]]. In addition, an increase in the magnesium content of more than 6% leads to a deterioration in the corrosion resistance of the alloy. At a temperature of 300 °C, 6.7% Mg is dissolved in the alloy. Magnesium, which has not dissolved, is in the structure and forms  $Al<sub>3</sub>Mg<sub>2</sub>$ , Mg5Al<sup>8</sup> compounds in solid solution. This does not add additional strength, but reduces the plastic properties of the alloy. To improve the plastic properties of the alloy, the percentage of silicon and iron in it is reduced, and zirconium and titanium are added [[6], [7]].



**Figure 1** - The effect of magnesium on strength of aluminum alloy

The influence of lithium on the properties of Al-Mg and Al-Mg-Si alloys is given in [[8], [9], [10]]. The authors found that in the process of artificial aging, the dispersion-hardening phases of Al3Li, Al2MgLi and Mg2Si are formed in the alloy. The fine phases of Al3Li are isolated by reducing the solubility of lithium in the solid state when added to the Al-Mg alloy. The Al2MgLi phase is formed at a high magnesium content >4%. The main hardening in Al-Mg and Al-Mg-Si alloys occurs precisely due to the Al3Li and Al<sub>2</sub>MgLi phases. Also, the Al<sub>2</sub>MgLi phase improves the corrosion resistance of the alloy itself.



### **Figure 2** - The effect of deformation on strength of aluminum alloy

The authors Shamas ud Din, Hasan Bin Awais [[11], [12]] investigated the effect of lithium on the properties of Al-Mg-Si alloys. The research results showed (Figure 3, 4) that when lithium is introduced up to 3%, the strength limit of Al-Mg-Si alloys increases from 60 MPa to 350 MPa and when lithium is introduced up to 2%, the microhardness increases from 50 HV in the cast state to 90 HV in the aged state.



**Figure 3** - The effect of lithium on the microhardness of Al-Mg-Si alloys

It is known [1] that the addition of 1% lithium reduces the density of the alloy by 3% and increases the modulus of elasticity by 6%. For example, Figures 5 and 6 show the effects of lithium, magnesium, and zirconium content on Young's modulus and aluminum density. With an increase in the lithium content to 1%, the density of aluminum decreases from 2690 mg/cm<sup>3</sup> to 2600 mg/cm<sup>3</sup> and the Young's modulus increases from 66-67 GPa to 70-71 GPa  $[[13],[14]]$ . With a lithium content of up to 1.8%, the aluminum alloy has a low resistance to stress corrosion, and at 1.9% the alloy becomes resistant to corrosion cracking. With a lithium content of 1.92.0%, the tensile strength and yield strength increases, but the plastic properties decrease. A further increase in the lithium content from 2.1 to 2.3% contributes to an increase in looseness and the appearance of cracks [[15], [16]]. As can be seen, lithium is the most effective alloying element, which significantly increases the Young's modulus and reduces the density of aluminum.



**Figure 4** - The effect of lithium on the strength of Al-Mg-Si alloys

Zirconium forms with aluminum and lithium a composite intermetallic Al3Zr–Al3Li, which improves the ductility of the alloy. In aluminum alloys, the Al3Zr phase is used as a grain shredder, which improves the impact strength [[17], 18], [19], [20]].



**Figure 5** - Effect of alloying elements on Young's modulus

Thus, in this paper, the methods of alloying lithium, magnesium and zirconium into aluminum are considered. Difficulties in this work are associated with the processes of introducing zirconium and especially lithium into the liquid-alloy. Information on the technology of obtaining alloy 1420 is not disclosed in open literature sources,

since this alloy with a strength of  $\sigma_B \ge 415$  MPa falls under the export control regime of goods and technologies for missile, dual-use and nuclear purposes.



**Figure 6** - Effect of alloying elements on aluminum density

#### **The experimental part**

To obtain the aluminum alloy of the Al-Mg-Zr-Li system, melting and casting were carried out in a vacuum induction furnace UIPV-0.001 (JSC "IMiO", Almaty). The unit is equipped with various mechanisms and devices that load metal into the crucible, electromagnetic mixing, casting into molds, measuring the liquid-alloy temperature and pressure in the chamber. The maximum heating temperature of the furnace reaches 2000 $^{\circ}$  C, the residual pressure is 100 Pa. The entire process of melting, aging, alloying, casting and cooling takes place in a vacuum or in an argon atmosphere.

To determine the chemical composition and study the microstructure of samples, samples with a diameter of 40 mm and a height of 15 mm were cut from cast bars. The samples were cut at the Brilliant 221 unit. The cut samples were ground and polished on a Rubin 500 - Sahpir 320e machine using sanding papers and polishing suspensions from 15 microns to 1 microns. To identify the structure, the surface of the samples was etched with 10% nitric acid solution and 30% hydrofluoric acid solution. The microstructure of the polished and etched samples was studied using a Zeiss Axiovert 200 Mat optical microscope. The chemical composition was studied using an X-ray fluorescence semi-quantitative spectrometer Axios 1kW PANalytical (JSC "IMiO"), an atomic emission spectrometer Optima 8300 DV (JSC

"IMiO") and an electron probe chemical analyzer Joel Superprobe 733 (Institute of Geological Sciences named after K. I. Satpayev). To study the mechanical properties, compression tests were carried out on Shimadzu AG testing machine (JSC "IMiO"), Brinell hardness on the HBV-30A hardness tester (JSC "IMiO") and microhardness on the PMT-3 microhardometer (JSC "National Center for Space Research and Technology"). For compression tests, cylindrical samples were manufactured according to GOST 25.503-97 with dimensions of 40/60 mm. To determine the Brinell hardness, samples were made according to GOST 9012-59 with the extrusion of a 2 mm ball of hardened steel up to 10 prints with a load of 5 kgf. To determine the microhardness, the samples were made according to GOST 9450-76 with the extrusion of a diamond pyramid up to 30 prints with a load of 10 g. The phase composition of the samples was determined using an X-ray diffractometer DRON-3. Polished samples in the form of square plates with dimensions of 10/10/1 mm were prepared for the study.

In the work, the following materials were used to obtain a cast aluminum alloy of Al-Mg-Zr-Li system: aluminum of technical purity A0 or A5, magnesium Mg95, lithium LE-1, zirconium E100, aluminum-zirconium ligature AlZr5, aluminumlithium ligature AlLi10. The composition of the charge materials from which the cast alloys of the Al-Mg-Zr and Al-Mg-Zr-Li systems were smelted is shown in Table 1.

Two methods were used to introduce zirconium into the Al-Mg liquid-alloy: the introduction of pure zirconium (series No. 1) and the introduction of zirconium in the form of a ligature (series No. 2). Two methods were used to introduce lithium into the Al-Mg-Zr liquid-alloy: the introduction of lithium in the form of a ligature (series No. 3) and the introduction of pure lithium (series No. 4). All series of experiments are given below:

*Experiment Series No. 1.* Aluminum A0, magnesium Mg95 and zirconium E100 were used to produce the Al-Mg-Zr liquid-alloy. Aluminum and zirconium charge was simultaneously introduced into the crucible. Since the melting point of zirconium is higher than the melting point of aluminum, melting was carried out in a vacuum of 100 Pa at a temperature of 800 $^{\circ}$  C, then kept for 5 minutes and cooled to 700 $^{\circ}$  C, after which argon was injected to prevent magnesium evaporation and magnesium was injected. After that, the melt was

kept for 5, 10, 20 minutes and cast into a graphite mold. Cooled in the chamber.

*Experiment Series No. 2.* Aluminum A5, magnesium, and zirconium ligature AlZr5 were used to produce the Al-Mg-Zr liquid-alloy. Aluminum and zirconium ligature were simultaneously loaded into the crucible, melted in a vacuum of 100 Pa at a temperature of 800 $^{\circ}$ C. At this temperature, they were kept for 5 minutes, after which the liquid-alloy was cooled to 700 °C, then argon was injected and a magnesium charge materials were introduced. After that, the liquid-alloy was kept for 5, 10, 20 minutes at a temperature of 700 $^{\circ}$ C. Then the liquid-alloy was poured into graphite molds and cooled in the chamber.

*Experiment Series No. 3.* Aluminum A5, magnesium Mg95, zirconium ligature AlZr5, lithium ligature AlLi10 were used to produce the Al-Mg-Zr-Li liquid-alloy. Aluminum, lithium ligature and zirconium ligature were melted at a temperature of 700 $\degree$ C in an argon medium, then a magnesium suspension was introduced and kept for 5, 10 or 20 minutes at the same temperature. After that, the finished liquid-alloy was poured into a graphite mold and cooled inside the chamber.

*Experiment Series No. 4.* Aluminum A5, magnesium Mg95, lithium LE-1 and zirconium ligature AlZr5 were used to produce the Al-Mg-Zr-Li liquid-alloy. Aluminum, lithium and zirconium ligature were melted at a temperature of 700 $\degree$ C in an argon medium and a magnesium charge materials were added, then kept for 5, 10 or 20 minutes at a temperature of 700 $\degree$ C in an argon atmosphere. After that, the finished liquid-alloy was poured into a graphite mold and cooled inside the chamber.

### **Results of the experiment series No. 1-4**

The chemical composition of the obtained cast samples of Al-Mg-Zr-Li with different sequences of magnesium, lithium, AlZr5 and AlLi10 ligatures is shown in Table 2.

As can be seen from Table 2, the samples obtained in Series No. 1 contain a significant silicon content of 0.269-0.346% and other impurities of 0.735-0.78%. The use of A0 grade aluminum with a high impurity content does not give the desired result in terms of purity of the chemical composition, the total impurity content of which reaches up to 1.14%. The impurity content of more



**Table 1** - Elemental composition of the charge materials in a series of experiments





 $=$  36  $=$ 

than 1% negatively affects the mechanical properties of the alloy. Therefore, in the subsequent series of experiments, a cleaner grade A5 aluminum was used. In the whole series of experiments, a decrease in the magnesium content is observed with an increase in the exposure time from 5 to 20 minutes. The zirconium content in series No. 1, 2 and 4 is kept in the range of 0.098-0.124%, which corresponds to the specified amount of suspension for alloying. In series No. 3, the magnesium content of the samples sharply decreases from the specified 5.3% to 4.63-4.74%, zirconium from 0.12% to 0.094- 0.112%. The decrease in magnesium in all series of experiments is explained by the fact that magnesium is an active element and over 560 $\,^0$ C interacts with oxygen, which remains after pumping in a vacuum chamber and is present in the composition of the injected argon. The chemical composition of the alloy obtained in the experiment series No. 4 with an exposure time of 5 minutes is optimal, since the chemical composition is close to the specified content of the charge materials components and contains fewer impurities.

The microstructures of the Al-Mg-Zr and Al-Mg-Zr-Li samples obtained with a Zeiss Axiovert 200 Mat optical microscope are shown in Figures 7 and 8.



**Figure 7** – Microstructures of the Al-Mg-Zr alloys (magnification x200). Series No. 1.



**Figure 8** – Microstructures of the Al-Mg-Zr alloys (magnification x200). Series No. 2.

As can be seen in Figure 7, the samples of Al-Mg-Zr series No. 1 consists of Al, a solid solution of Mg in Al and an intermetallic  $Al_3Mg_2$ . The alloy also contains a large number of clusters of primary crystals in the form of needle plates. The separation of primary Al3Zr crystals from the liquid-alloy in the studied temperature range of 700-800 °C is most likely due to the heterogeneity of the liquid-alloy, which is confirmed by the uneven separation of particles in the form of clusters whose sizes reached more than 100 microns (Figure 7). Thus, in the course of research, it was found that as parameters for melting and casting bars from the alloys of the Al-Mg-Zr system, it is advisable to use zirconium ligature instead of metal pieces of zirconium, since zirconium in this case dissolves in the aluminum liquid-alloy and separation of smaller primary crystals of Al<sub>3</sub>Zr  $\leq$  100 microns occurs (Figure 8). Introduction of zirconium ligature into the aluminum liquid-alloy is an optimal choice when melting the alloy, because the zirconium ligature is evenly and faster distributed over the entire volume of the liquid-alloy compared to metallic zirconium, as evidenced by the analysis of the micrograph of the Al-Mg-Zr alloy from series No. 2. In this regard, in subsequent experiments of series No. 3 and No. 4, a zirconium ligature was used to introduce zirconium into the liquid-alloy.

The following phases are present in the microstructure of the Al-Mg-Zr-Li alloys of series No. 3 and No. 4: Al, Mg solid solution in Al,  $Al_3Mg_2$ ,  $Al_3Zr$ . As can be seen in Figure 9, the Al-Mg-Zr-Li alloy has a dendritic structure with many different inclusions. As it turned out, these inclusions appeared when the AlLi10 ligature was added.



**Figure 9** – Microstructures of the Al-Mg-Zr-Li alloys (magnification x200). Series No. 3

As is already known, lithium is coated with an oxide film at room temperature. The introduction of such a ligature into the aluminum liquid-alloy leads to the

appearance of many oxide films. These captives complicate the merging into the mold and fall into the cast bar. Lithium could also react with hydrogen (present in argon) at 500-700  $^{\circ}$ C, with iron and silicon (present in aluminum) at 600-700 $\degree$ C and with carbon (graphite cast). When composing the charge materials, it is recommended to exclude the use of AlLi10 ligatures.

As can be seen in Figure 10, in the structure of the Al-Mg-Zr-Li alloy of series No. 4 there are also Al phases, Mg solid solution in Al,  $Al_3Mg_2$ ,  $Al_3Zr$ . The multitude of phases makes it difficult to visually identify each phase, including the lithium phases that are present in the alloy.



**Figure 10** – Microstructures of the Al-Mg-Zr-Li alloys (magnification x200). Series No. 4

To determine the phases with lithium, an additional X-ray diffractometric analysis was performed on a DRON-3 diffractometer. The results of the phase analysis are shown in Figure 11.



**Figure 11** – Diffractogram of the presence of phases of aluminum with lithium

Peak d=2.03281 corresponds to 100% aluminum content, peak d=2.34890 has an atomic content of 53.3% aluminum and 46.7% lithium. This atomic ratio corresponds to the AlLi phase. The peak d=1.43908 has an atomic content of 19.5% aluminum and 80.5% lithium. This atomic ratio corresponds to the phase Al4Li9.

 $=$  37  $=$ 



**Table 3** – Mechanical properties of Al-Mg-Zr and Al-Mg-Zr-Li alloy

Compression, hardness and microhardness tests were carried out to study the mechanical properties of the samples. The results of the mechanical properties of alloys of the Al-Mg-Zr and Al-Mg-Zr-Li systems are shown in Table 3.

As can be seen from Table 3, Al-Mg-Zr alloys of series No. 2, compared with alloys of series No. 1, show an increase in all characteristics: Brinell hardness from 80 to 82.14 HB, microhardness from 148 to 154 MPa, compressive strength from 69.8 to 138.7 MPa, modulus of elasticity from 3.5 to 10.5 GPa, modulus of plastic deformation from 1.1 to 1.2 GPa, yield strength under compression from 77.22 to 164.5 MPa. Such indicators are achieved due to the uniform distribution of zirconium in the liquidalloy by introducing zirconium ligature instead of metallic zirconium.

Al-Mg-Zr-Li alloys of series No. 4 compared with alloys of series No. 3 showed an increase in Brinell hardness from 69.1 to 85 HB, microhardness from 97.2 to 139 MPa, compressive strength from 99.5 to 149.6 MPa, elastic modulus from 9.5 to 12 GPa, compressive yield strength from 119 up to 175.2 MPa and with a slight decrease in plastic deformation modulus from 0.9 to 0.83 GPa. As noted earlier, the introduction of the AlLi10 ligature negatively affects the mechanical properties of the Al-Mg-Zr-Li alloy. It is advisable to use lithium in its pure form.

## **Conclusions**

Cast alloys of the Al-Mg-Zr-Li system with the following characteristics were obtained:

- chemical composition: Al-92.245%, Mg-5.00%, Zr-0.105%, Li-2.21%, Si-0.238%, impurities-0.202%

- mechanical properties: Brinell hardness 85 HB, microhardness 139 MPa, compressive strength 149.6 MPa, elastic modulus 12 GPa, compressive yield strength 175.2 MPa and plastic deformation modulus 0.83 GPa.

- production technology: melting aluminum, lithium and AlZr5 ligatures at a temperature of 700  $\mathrm{^{0}C}$  in an argon medium, then adding a magnesium sample to the liquid-alloy and holding for 5 minutes at a temperature of 700 $^{\circ}$ C in an argon atmosphere. Draining the finished liquid-alloy into the graphite cast and cooling inside the chamber.

**Conflict of interest.** On behalf of all the authors, the correspondent author declares that there is no conflict of interest.

**Gratitude.** This study is funded by the Science Committee of the Ministry of Education and Science of the Republic of Kazakhstan (Grant № 00037/GF)", where 00037/GF (№ 00037/ГФ) is the IRN of the project.

*Cite this article as:* Ablakatov IK, Ismailov MB, Mustafa LM, Sanin AF. Investigation of the Technology of Introducing Li, Mg and Zr Alloys into Aluminum Alloy. Kompleksnoe Ispolzovanie Mineralnogo Syra = Complex Use of Mineral Resources. 2023; 327(4):32-40[. https://doi.org/10.31643/2023/6445.37](https://doi.org/10.31643/2023/6445.37)

 $\equiv$  38  $\equiv$ 

# **Легірлеуші Li, Mg және Zr металдарын алюминий қорытпасына енгізу технологиясын зерттеу**

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#### **ТҮЙІНДЕМЕ**



# **Исследование технологии введения в алюминиевый сплав легирующих Li, Mg и Zr**

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#### **АННОТАЦИЯ**

Поступила: *12 августа 2022* Рецензирование: *06 сентября 2022* Принята в печать: *31 января 2023*

Получены литые сплавы системы Al-Mg-Zr-Li со следующими характеристиками: химический состав: Al-92,245%, Mg-5,00%, Zr-0,105%, Li-2,21%, Si-0,238%, примеси-0,202%. Механические свойства: твердость по Бринеллю 85 HB, микротвердость 139 МПа, предел упругости при сжатии 149,6 МПа, модуль упругости 12 ГПа, предел текучести при сжатии 175,2 МПа и модуль пластической деформации 0,83 ГПа. Технология получения: плавка алюминия, лития и AlZr<sub>5</sub> лигатуры при температуре 700°С в среде аргона, затем добавление навески магния в расплав и выдержка в течении 5 минут при температуре 700<sup>0</sup>С в атмосфере аргона. Слив готового расплава в графитовую изложницу и охлаждение внутри камеры. Для получения алюминиевого сплава системы Al-Mg-Zr-Li плавка и литье проводились в вакуумной индукционной печи УИПВ-0,001 (АО «ИМиО», Алматы). Настоящая статья посвящена исследованию начальной фазы получения сплава 1420, а именно получению первичного материала с нужным химическим составом, влиянию легирующих магния, циркония и лития на прочностные свойства материала.

*Ключевые слова:* алюминий**,** алюминий-литиевые сплавы, Al-Mg-Zr-Li, плавка, литье, лигатура



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