

# Heat Treatment of AlSi7Mg0.3 Aluminium Alloys with Increased Zirconium and Titanium Content

E. Kantoríková \*, M. Kuriš, R. Pastirčák

 <sup>a</sup> Department of Technological Engineering, University of Žilina in Žilina, Univerzitná 8215/1, 010 26 Žilina, Slovakia
\* Corresponding author. E-mail address: elena.kantorikova@fstroj.uniza.sk

Received 09.06.2020; accepted in revised form 07.06.2021

#### Abstract

The paper compares changes in the structure and mechanical properties due to the synergistic effect of alloying elements Zr and Ti. It is assumed that by increasing the content of Zr and Ti in the aluminium alloy, better mechanical properties will be achieved. Paper focuses on description of the differences between the samples casted into the shell mold and the metal mold. Main difference between mentioned molds is a different heat transfer coefficient during pouring, solidification and cooling of the metal in the mold. The main goal was to analyse the influence of Zr and Ti elements and compare the mechanical properties after the heat treatment. Curing and precipitation aging were used during the experiment. The effect of the elements on AlSi7Mg0.3 alloy created differences between the excluded Zr phases after heat treatment. Evaluation of the microstructure pointed to the decomposition of large predominantly needle Zr phases into smaller, more stable formations.

Keywords: Heat treatment, Aluminum alloys, Zirconium, Titan, Mechanical properties

# 1. Introduction

At present, it is impossible to imagine the automotive industry without light aluminium alloys. Science and research are still engaged in ways of innovation. Aluminium alloys are easy to process and have a wide range of applications. The future of aluminium alloys depends on further development, especially production and heat treatment technology. In case of the aluminium casting alloys, alloys with silicon and other additive elements have developed very rapidly in a wide range of economic favourable applications. Alloying is defined as a planned increase of the selected element in the experimental alloy with positive effect on the selected properties and at the same time, they can suppress the negative effect of harmful elements such as Fe [1]. Therefore alloying opens new opportunities in the structural optimization of lightweight parts [2]. For example, an increase in the ultimate tensile strength (Rm) ensures, in practice a significant weight reduction of the casting without losing the strength properties of the functional cross-section of the casting.

The development of new and more sophisticated Al alloys consists in a change of chemical composition and technological processes. By adding uncommon elements such as Zr to the aluminium alloys such as Al-Si-Mg-Cu, it is possible to change the selected properties by altering the microstructure (exclude of new intermetallic phases, grain refinement or Fe fixation) and the effect on heat treatment - annealing and artificial aging [3].

Al-based alloys with zirconium show excellent mechanical properties. Zirconium is added to the Al-Si alloys to increase the alloy strength (strengthening effect being initiated by the



elimination of the intermetallic phase of Al<sub>3</sub>Zr or AlSiZr). Zr is characterized by the lowest diffusion in Al compared to other elements such as Mn, V or Sc. Zr atoms have high binding energy with unoccupied Al sites [4]. The Al3Zr-based phases are resistant to dissolution and coarsening at temperatures above 250 °C, affecting the size and shape of the grains and sub-grains in the Al-Si metal matrix of the alloy. Thereby they allow to increase and maintain the strength of the Al-Si alloy even after precipitation hardening. This is particularly advantageous for Al-Si-Mg-Cubased alloys where the hardening phases of Al2Cu or Mg2Si lose stability. Excluded phases based on Zr does not achieve a strengthening effect like those of Al<sub>2</sub>Cu or Mg<sub>2</sub>Si, but they are characterized by higher stability at temperatures above 250 °C [5]. The efficiency of Zr and its phases is closely related to the Mg and Cu content since the elements reduce the potency of Zr in Al alloys. Zirconium forms fine precipitates that prevent recrystallization, resulting in a fine cast structure. Zirconium improves strength properties by grain refining and improves corrosion resistance [6]. Mechanical properties are the decisive criterion for the practical use of the material. A new challenge in the field of aluminium alloy research is their resistance to working temperatures above 300 °C where Zr also shows positive results when combined with Ti. Acquired benefits are important especially for castings of gearboxes and engine blocks. Al-Si-based alloys represent an important group of alloys used to produce Al alloy castings. Conventional Al-Si alloys contain a eutectic with lamellar or filamentous morphology. This is attributed to a strong anisotropy of Si growth and lower interfacial energy between Si and Al. The amount, size and shape of the free Si are related to the mechanical properties of the Al-Si binary alloy. The eutectic gives Al-Si alloys good standard quality, but also reduces linear shrinkage and the tendency for hot cracking or micro-porosity [7].

Heat treatment is used to increase the mechanical properties such as the strength and ductility of the Al alloy. Adding a trace element increases the heat treatment effect. The curing of Al alloys with the addition of Zr is often carried out by precipitation hardening and subsequent artificial aging.

The paper describes differences in mechanical properties of Al alloy with addition of zirconium after heat treatment. It describes the differences of the samples cast into the shell mold and the metal mold, since each is characterized primarily by a different heat dissipation after casting and by a different grain and sub grain growth mechanism in the Al alloy metal matrix. Each technology has its advantages, in the case of ingot mold casting there is a sharp heat dissipation and the formation of finer grains, and due to the solid mold the stresses in the casting are created. On the other hand, the casting technology on the smelting model ensures better running property and a more stable solidification process due to the casting into the heated shell, especially in the production of complex and precise castings. The disadvantage is non-regular texture in terms of grain size [7, 8].

Light metals such as aluminium alloys have given the automotive industry a new impulse. New materials triggered the development of new production technologies and led to new material properties [8, 9].

AlSi7Mg0.3 alloy is used for the production of castings where good casting properties, corrosion resistance, pressure tightness and weldability are required. It is therefore often used in the production of components for internal combustion engines. It is therefore important to find possibilities of using AlSi7Mg0.3 aluminium alloy for current applications such as electric cars. It is also used in the production of components for the aerospace and defence industries. In order to achieve the positive parameters mentioned above, it is necessary to ensure a high purity of the primary alloy, because iron results in a decrease in the quality of the production of castings and an increase in costs due to nonconforming production [9]

#### 2. Methods and goals

An alloy commonly used in the automotive industry AlSi7Mg0.3 was selected for the experiment, which normally contains (in wt.%) 6.7 Si, 0.13 Fe, 0.5 Cu, 0.34 Mg and 0.1 Ti [10]. Six types of samples were produced, which were cast into a shell mold and six types into a metal mold (sample mold). Three experimental melts were produced with a constant addition of 0.2% Zr followed by a gradual addition of Ti in 0.1, 0.2 and 0.3 wt. %. The aim was to evaluate the effect of the combination on grain refinement, elimination of new intermetallic phases and their common synergy in improving mechanical properties. The heat treatment procedure was carried out according to STN EN 1706. Hardening T6, solution annealing 540 °C, 12 hours / water 20 ° C. Artificial aging 150 °C, 3 hours on the air. The heat treatment was carried out in an electric chamber furnace. The selected mode is used for castings from which good castability, corrosion resistance, pressure tightness and weldability are required. The use of such castings is, for example, in nuclear power plants, aircraft pumps or engine cylinder blocks. Brinell hardness, tensile and standard optical metallography were performed on the samples.

Chemical composition of finished castings was verified on the spectrometer. It can be read from Table 1 that the content of Zr and Ti in the shell form did not sufficiently dissolve compared to the metal form, where there is a sufficient amount of Zr, but the titanium did not appear. The Ti inoculant was added due to the greater number of Zr grains. With Ti we wanted to influence the size of Zr grains and thus their number.

The hardness of the cast and heat treated samples was measured according to Brinell, ball diameter 5 mm, load 250 kg, time 10 s. Each value is an average of at least five separate measurements taken randomly. For the tensile test, samples with a circular diameter of 8 mm and a length of 65 mm were produced. The heat treatment was already performed on the finished samples. The samples were drawn on an electronic universal testing machine with a capacity of 20 kN. Three samples were tested for each condition. Mechanical properties such as tensile strength and elongation were derived from the machine data collection system.





Chemical analysis of experimental material (Content of the element weight %)								
Type of sample	Element	Si	Fe	Mg	Cu	Zr	Ti	Al
Shell (in wt. %)	0.2Ti	6.90	0.113	0.395	0.54	0.0019	0.155	91.8
	0.3Ti	6.86	0.116	0.374	0.55	0.0939	0.161	91.7
	0.4Ti	6.80	0.120	0.360	0.54	0.2190	0.133	91.7
Steel mold ( in wt. %)	0.2Ti	6.94	0.125	0.07	0.55	0.126	0.22	91.5
	0.3Ti	6.73	0.128	0.07	0.54	0.15	0.27	91.7
	0.4Ti	6.55	0.127	0.07	0.5	0.122	0.23	92.0

Table 1.

# 3. Results and discussion

Changes in hardness of samples before and after heat treatment are presented in Table 2. The heat treatment increased the hardness of all samples compared to as-cast condition. In the case of samples cast into a metal mold, the specified value according to AC421 90 HBW is prescribed after heat treatment. The hardness of the material increases as the amount of titanium is added, but by heat treatment the values have stabilized at the same value of 90 HBW. In terms of the hardness of the material, the increased Zr content in the metal form did not occur. In the shell form, the hardness difference is clear because the prescribed value is 75 HBW. Hardness increased by 30% after heat treatment. It is assumed that the increased hardness is related to a different heat transfer than the metal mold.

#### Table 2.

Brinell hardness

HBW 5/250/10									
	Sh	nell	Metal mold						
	Non HT	With HT	Non HT	With HT					
0.2Ti	66	86	75	91					
0.3Ti	61	87	80	90					
0.4Ti	64	92	84	87					

The mean values from the tensile test Rm and the ductility A are shown in Figure 1. The highest tensile strength of the shell mold was 258 MPa for the sample with 0.3% Ti, prescribed in the standard is 260 MPa. For the metal mold, this is a sample with 0.4% Ti value of 280 MPa, prescribed is 290 MPa. None of the samples reached the reference, conventional maximum tension. In the elongation evaluation, the best results were obtained with a sample of 0.4 Ti after heat treatment, die casting, A = 8%. the reference value is set at 4%. A standardized result was achieved on a sample with 0.3% Ti mold and 0.4% Ti shell. The results are shown in the graph Figure 2. For aluminium alloys, ductility is an important indicator of mechanical properties. It expresses possible changes in the crystal lattice and changes in dislocations in materials.



Fig. 1. Ultimate tensile strength, Mm NHT - metal mold non heat treatment, S NHT - Shell non heat treatment, Mm HT - metal mold heat treatment, S HT - Shell heat treatment

The basic microstructure of the AlSi7Mg0.3 alloy is hypoeutectic, containing primary dendrites and eutectic consisting of  $\alpha$  phase and Si. Due to the content of elements such as Fe and Mn in the alloy, they can be deposited in the form of ferric intermetallic phases in various morphological forms. The effect on the resulting structure is closely related to the chemical composition and casting technology. In the instance of mold casting technology, quicker cooling and a fine dendritic structure can be expected, in comparison to conventional casting [11, 12].



Fig. 2. The ductility of examined samples

www.czasopisma.pan.pl





Fig. 3. Microstructure of AlSi7Mg0.3 with 0.2% Zr before HT, etchant 0.5% HF

The heat treatment affected the entire structure and changes the Zr phases. The number of Zr phases also increased according to the inoculated Ti content. However, there was a difference between the samples cast into the shell mold, where Zr appeared as sharp needles, distributed individually throughout the cross-section of the sample (Fig. 4). In contrast to the samples cast into the metal mold, where the phases decomposed into tiny clusters of small needles with a rounded end (Fig. 5).



Fig. 4. Microstructure of AlSi7Mg0.3 with 0.2% Zr, with HT cast into shell mold, etchant 0.5% HF.



Fig. 5. Microstructure of AlSi7Mg0.3 whith 0.2% Zr, with HT cast into metal mold, etchant 0.5% HF.

By adding various elements to the AlSi7Mg0.3 alloy, it is possible to provide an improvement in the mechanical properties of the alloy. Zr provides increased strength, hardness and high temperature resistance. The increase in the above-mentioned properties, such as strength or hardness, is due to the exclude of the hardened Al3Zr phases which intersect the AlSi phases and the eutectic. These can be secreted as single needles or a group of needles. The difference in structure is due to the casting method, but also the heat treatment technology. Thus, the Zr phases may have an enhancing effect on the metal matrix of the studied AlSi9Cu1Mg alloy. After the heat treatment, at higher Zr contents, the large needle phases disintegrate into smaller plate phases. The work shows that the casting mold also affects the resulting properties.

# 4. Conclusions

Samples that were cast into the shell mold with the addition of Ti by 0.1 wt. % to an AlSi7Mg0.3 alloy with a constant Zr content of about 0.20 wt. % have a higher hardness than the samples cast into the metal mold. After heat treatment, the increase in hardness was 30%. The tensile strength test was done according to standard AC121 for both sample types. Ductility increased by 1% in shell and 4% in metal molds. In the structure, the zirconium phases were much more clear in shell form. The casting from the metal mold presents a larger number of small Zr phases. Corresponding results indicate the possibility of developing a more sophisticated alloy that would be able to better withstand changes during working condition, especially in internal combustion engines.

#### Acknowledgements

This study has been supported by the Scientific Grant Agency of the Ministry of Education of the Slovak Republic VEGA 1/0494/17. The authors acknowledge the grant agency for support.

www.czasopisma.pan.pl



# References

- [1] Bolibruchová, D., Tillová, E. (2005). Al-Si foundry alloys. Žilina.
- [2] Michna, Š., Lukáč, I. (2005). et al. Encyclopedia of aluminum.
- [3] Bechný, L. (1990). Foundry metallurgy and technology. ALFA Bratislava.
- [4] Bolibruchová, D., Kuriš, M. & Matejka, M. (2019). Effect of Zr on selected properties and porosity of AlSi9Cu1Mg alloy for the purpose of production of high-precision castings. Manufacturing Technology. 19(4), 1213-2489.
- [5] Bolibruchova, D., Macko, J. & Bruna, M. (2014). Elimination of negative effect of Fe in secondary alloys AlSi6Cu4 (EN AC 45 000, A 319) by nickel. *Archives of Metallurgy and Materials*, 59, 717-721
- [6] Mahmudi, R., Sepehrband, P. & Ghasemi, H.M. (2006). Improved properties of A319 aluminum casting alloy modified with Zr. *Materials Letters*. 2606-2610. DOI 10.1016/j.matlet. 2006.01.046
- [7] Peng, G., Chen, K., Fang, H. & Chen, S. (2012). A study of nanoscale Al3(Zr,Yb) dispersoids structure and thermal stability in Al–Zr–Yb alloy. *Materials Science and Engineering*. Volume 535, 311-315.

- [8] Sha, G. & Cerezo, A. (2004). Early-stage precipitation in Al–Zn–Mg–Cu alloy (7050). Acta Materialia. 52(15), 4503-4516.
- [9] Lü, X., Guo, E., Rometsch, P. & Wang, L. (2012). Effect of onestep and two-step homogenization treatments on distribution of Al3Zr dispersoids in commercial AA7150 aluminium alloy. *Transactions of Nonferrous Metals Society of China*. 22, 2645-2651. Science Direct.
- [10] STN EN 1706. AC–42100. Aluminium alloy for general purpose castings.
- [11] Liu, S., Zhang, X.M. & Chen, M.A. & You, J. H. (2008). Influence of aging on quench sensitivity effect of 7055 aluminium alloy. *Materials Characterization*, 59(1), 53-60.
- [12] Pourkia, N., Emamy, M., Farhangi, H. & Seyed, E. (2010). The effect of Ti and Zr elements and cooling rate on the microstructure and tensile properties of a new developed super high-strength aluminium alloy. *Materials Science and Engineering A*. 527, 5318-5325.
- [13] Tillova, E., Chalupova, M. (2009). Structural analysis of Al-Si alloys. Žilina: EDIS ŽU UNIZA.