Effect of Zr on Selected Properties and Porosity of AlSi9Cu1Mg Alloy for the Purpose of Production of High-precision Castings

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The effect of Zr was assessed on changes in the mechanical properties of the alloy under investigation by the addition of Zr with a graduated increase by 0.05 wt. % Zr. Experimental samples were cast into ceramic molds, half of which were hardened by precipitation after casting. The measured values indicate an improvement in the mechanical properties, especially in experimental alloys containing Zr ≥ 0.20 wt. %. In the evaluation of R_m, the most significant improvement occurred in the experimental alloy with the addition of Zr 0.25 wt. % after heat treatment and E in the experimental alloy with the addition of Zr 0.20 wt. % after heat treatment, as well. The effect of Zr in the AlSi9Cu1Mg alloy investigated was also analyzed for porosity. It can be concluded that the addition of Zr into alloys is manifested by an increase in gassing of experimental alloys, especially in variants containing Zr ≥ 0.15 wt. %. At a higher Zr content there was a significant increase in the DI density index and pore size. For higher amounts of Zr ≥ 0.20 wt. %, long acicular phases with slightly cleaved morphology are visible in the metal matrix. Exclusion of the Zr phase nuclei with regard (also) to the change of exclusion temperature can cause capture of hydrogen bubbles (pockets) and thus increase the gassing rate.

Keywords: AlSi9Cu1Mg, Ceramic mold, Al_3Zr, Investment casting, Improvement of mechanical properties

1 Introduction

Al-Si alloys have experienced significant progress in the foundry industry, especially in the production of high-precision castings. AlSi9Cu1Mg is also an important representative of such alloys. A higher Si content in the AlSi9Cu1Mg alloy positively affects properties, contraction, shrinkage, and also reduces the susceptibility to hot cracking. On the contrary, due to Mg and Cu content, Mg_2Si and Al_2Cu phases are excluded, allowing precipitation hardening. The AlSi9Cu1Mg alloy is defined as sub-eutectic, preferably used for thin-walled castings, which can be subsequently welded and used at temperatures around 200 °C. For the needs of the aerospace and automotive industries, there is a constant pressure, often triggering innovations in the materials originally used. One way of improving the properties of the original Al-Si alloys is also by alloying with elements such as Mo, Cr, Ce, Ni or Zr. The aim is to improve the mechanical properties and heat resistance of the alloys, thereby increasing the usability of Al castings for applications above 250 °C. As a result, it is possible to produce Al castings with significant weight reduction, for higher thermal loads, or castings of more complicated structures and designs. An important area of application, mainly utilising the above-mentioned properties, is for example for engine block castings, gearboxes, cylinder heads, etc. Here, the emphasis is on the ability to work at high performance while bearing high operating loads. The consequent savings in the form of a reduction in dimensions or an improvement in the environmental aspect (reduction of fuel consumption) are significant positives that increase the priority of the development of such treated alloys. The experimental part follows the work of the authors Vončina and Medved [1, 2] pointing to improvement of mechanical properties of hardenable alloys AlSi9Cu3 and AA2050 after alloying with Zr and Mo. The improvement in mechanical properties in the experiment is attributed to the effect of the excluded Zr phases. Zr is preferably excluded in the form of Al_3Zr during the peritectic reaction, with a Zr content of ≥ 0.10 wt. %. Al_3Zr is excluded in two significantly different crystallographic morphologies, either as a tetragonal DO_{23} system or a coherent metastable system $L1_2$. The negative effect on Zr phase exclusion and the efficiency of these phases is closely related to the Mg and Cu content. Several works declare a visible exclusion of Zr phases with an addition of Zr ≥ 0.08 wt. %. At the same time, a positive effect on mechanical properties is reported, especially at a Zr content of 0.15 wt. %. No Zr content higher than 0.15 wt. % manifested any significantly positive effect Zr and its phases. The issue of the effect of Zr on increasing the gassing rate for the AlSi9Cu1Mg alloy and, overall, for the Al-Si system is still not sufficiently investigated. Several works offer a partial explanation of the effect of Zr on increasing the gassing rate for alloys or pure metals, but these are ternary systems of Al, Mg or Zr alloys. Therefore, the work itself builds on the enrichment of knowledge in the issue. Possibilities of the effect of alloying additions such as Zr on mechanical properties or heat resistance of the AlSi9Cu1Mg alloy show the possibility of developing a more sophisticated alloy. There are possibilities to utilise such modified Al alloy in foundry industry also in the technology of investment casting. This enables to produce more complicated designs and operationally more resistant castings for the automotive and aviation industries. [1-11]

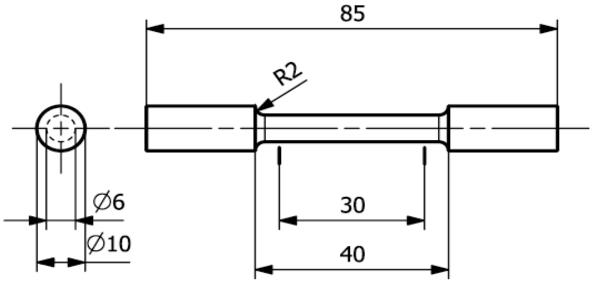
2 Experimental part

The process of manufacturing the ceramic mold consisted of gradual dipping of the waxy tree in the ceramic slurry followed by sprinkling with refractory (heat resistant) grains (opening material). The ceramic mold consists of three basic layers ensuring the formation of contact, insulation and reinforcing cover layers. The material composition of the individual cover layers is given in Table 1. The STN EN 1706 standard does not define the minimum mechanical properties of the AlSi9Cu1Mg alloy for casting into ceramic molds. It is an alloy used extensively for ingot casting, eventually for sand casting. Comparison of the measured results of the mechanical properties was made with the values for casting into sand molds. These are characterized by a similar heat dissipation, i.e. by similar crystallization.

***Tab. 1** Material composition of ceramics layers*

	1. Layer	2. Layer	3. Layer	4. Layer	5. Layer
Binder	Primcot cote plus	SP-Ultra 2408	MatriXsol 30	MatriXsol 30	MatriXsol 30
Grains	Cerabed DS 60	Rancosil A	Molochite 30-80 DD	Molochite 30-80 DD	Molochite 30-80 DD

a)



b)

***Fig. 1** a. Ceramic mold after casting, b. Scheme of testing sample*

The finished ceramic mold with an approximate thickness of 3mm was fired (burned) at 750 °C for at least 1.5 hours. The mold temperature was in the range of 510 to 540 °C. The samples were cast at a rate of 0.3 kg/s from a casting height of 500 mm. The casting temperature was 750 ± 10 °C. After casting, the ceramic mold was cooled in air for 1 hour. The AlSi9Cu1Mg primary alloy was delivered by the manufacturer in a pre-vaccinated and pre-modified state (Tab. 2)

The metallurgical process consisted in melting the AlSi9Cu1Mg primary alloy with the aim of producing the so-called reference alloy (without addition of Zr), and subsequent alloying it with AlZr15 foundry (master) alloy. Zr was added in graduated amounts by 0.05 wt. % Zr (from 0.05 wt. % to 0,30 wt. % Zr). During the experiment, neither the reference alloy nor the experimental alloys were degassed throughout the melting process. The AlZr15 foundry alloy was characterized by impaired melt solubility, therefore the melt had to be preheated to a temperature of 770 ± 10 °C. This led to the creation of conditions for increasing the gassing by hydrogen and the emergence of porosity or other defects. After casting of experimental alloys with an addition of Zr from 0.00 wt. % to 0.30 wt. %, the process continued by precipitation hardening (curing) T6. TS was performed on 5 total of 10 pieces of experimental samples (Fig. 1.) from each experimental variant.

***Tab. 2** Chemical composition of primary alloy AlSi9Cu1Mg*

Element	Si	Fe	Cu	Mn	Mg	Cr	Ni	Zn	Pb	Sn	Ti
[wt. %]	8.04	0.13	0.65	0.07	0.39	-	-	0.004	-	0.01	0.18

2.1 Effect of Zr on mechanical properties

Ten experimental samples were cast from each experimental variant (Fig. 1.), half of which were heat treated by precipitation hardening (curing) T6. When evaluating R_m (STN EN 1706 – min.135 MPa), the highest tensile strength without heat treatment, namely 172 MPa, and 260 MPa after heat treatment was achieved in experimental samples with addition of Zr 0.25 wt. % (Fig. 2.). The best agreed yield strength $Rp_{0.2}$ (STN EN 1706 – min. 90 MPa) was achieved in samples without heat treatment (137 MPa) and after heat treatment 207 MPa in experimental samples with addition of Zr 0.15 wt. % (Fig. 2.). The modulus of elasticity E (STN EN 1706 – min. 70 GPa) in experimental samples without heat treatment manifests a gradually increasing characteristics up to the value of 0.15 wt. % Zr. This is followed by a decrease to 62 and 61 GPa, and then there is a re-increase to 66.8 GPa noticeable only at the addition of 0.30 wt. % Zr. This is also the highest E value for samples without heat treatment. For heat treated samples, the highest value of 81.8 GPa was measured at the addition of Zr 0.20 wt. % (Fig. 3.). When evaluating the ductility A_M (STN EN 1706 – min. 1 %) (Fig. 3.), the best results for samples without heat treatment (2.0 %) and after heat treatment (3.2 %) were achieved in the reference sample.

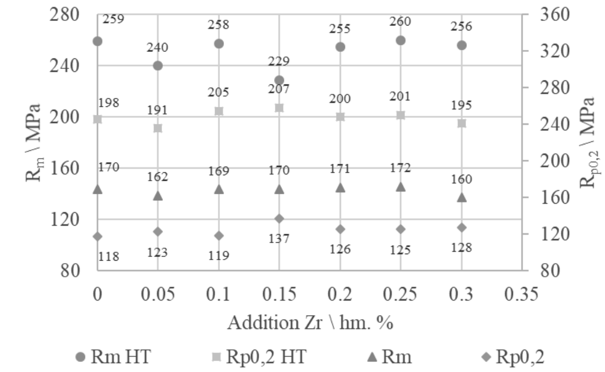


Fig. 2 *Dependence Rm a Rp0.2 before and after heat treatment*

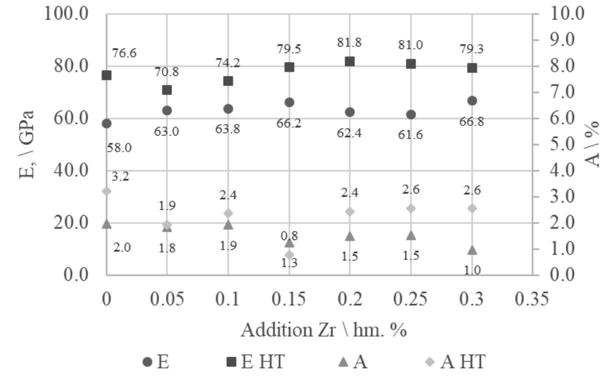


Fig. 3 *Dependence AM, E before and after heat treatment*

When evaluating HBW hardness (STN EN 1706 – min. 60 HBS) of experimental alloys without heat treatment, the highest value of 71 HBW was obtained in the variant with the addition of Zr 0.15 wt. % (Fig. 3.). Hardness 95 HBW was obtained in heat treated samples of variants with the addition of 0.05 wt. % Zr. Based on the measured values, already with Zr content ≥ 0.10 wt. % Zr has a reinforcing effect on the metal matrix of the AlSi9Cu1Mg alloy. Increasing R_m results in an AlSi9Cu1Mg alloy which is able to withstand, even when the functional cross-sections of the casting walls are narrowed (for example engine blocks), at least the same operating loads as in the case of casting walls with additional additions, because of ensuring the endurance after exposure to operating pressures

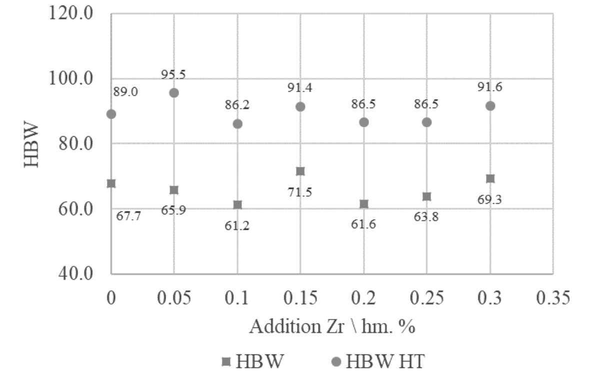
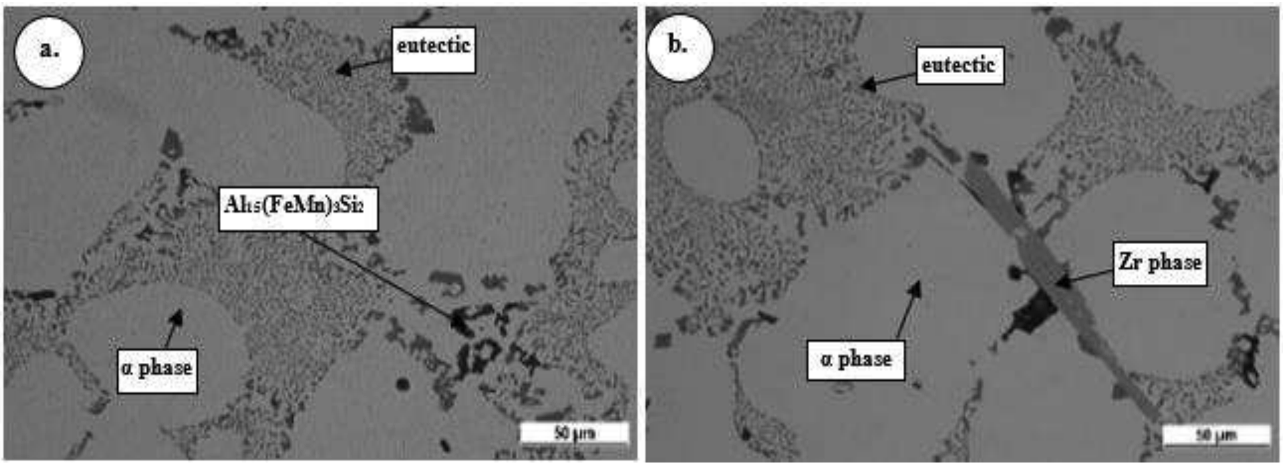


Fig. 4 *Dependence HBW before and after heat treatment*

To more precisely define the effect of Zr phases on the characteristics of the metal matrix and intermetallic phases of AlSi9Cu1Mg experimental alloys, microhardness measurements were performed on variants with Zr additions of 0.20, 0.25 and 0.30 wt. % (Fig. 5.). Indeed, in the given variants, sufficiently large Zr phases were excluded, in which it was possible to measure the hardness without the risk of extrusion of the indentation beyond the evaluated Zr phase. The parameters of microhardness measurement are given in Table 3. The phases evaluated were preferably excluded in the form of acicular structures with a smooth surface and split ends (Fig. 5c.). The measurement was performed on an area prepared for metallographic evaluation. For detailed resolution and Zr phases, samples were etched by ferrous phase H_2SO_4 etching agent. Observable Zr phases in microstructures are probably intermetallic phases of the Al_3Zr type or AlSiZr type. Similar identification of Zr phases is described also by Vončina in his work [1]. Preferably, however, Zr is excluded because of the smallest diffusion against the α matrix than Al_3Zr, as confirmed by Mahmudi's work [4]. With increasing content of Zr, we observed suppression of Fe phases and/or their common exclusion in interaction with Zr phases (Fig. 5c.).



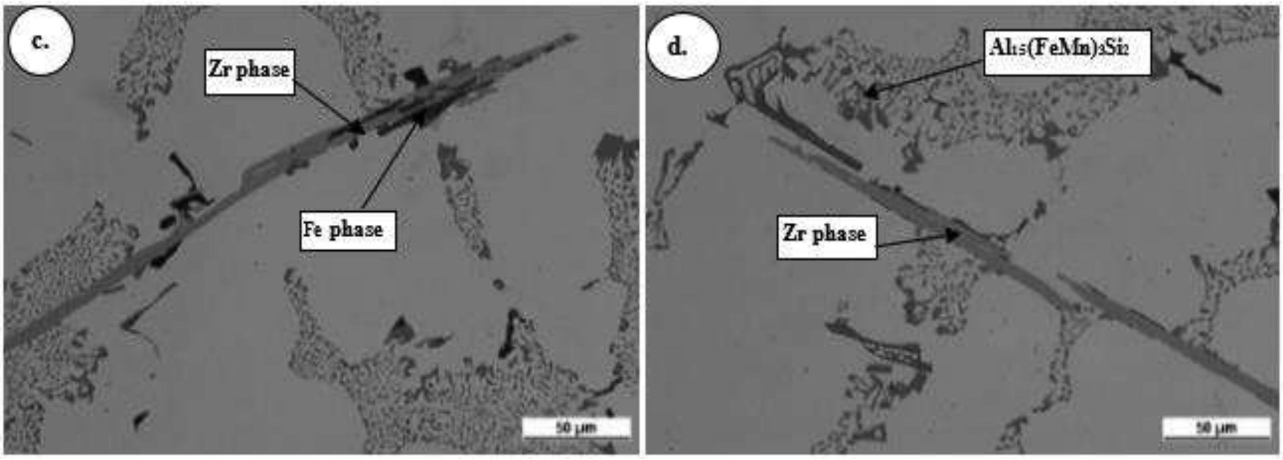


Fig. 5 *Microstructure of experimental alloys AlSi9Cu1Mgbefore heat treatment a. 0.00 wt. % Zr, b. 0.20 wt. % Zr, c. 0.25 wt. % Zr, d. 0.30 wt. % Zr, etchant H_2SO_4*

Tab. 3 *Parameters of measuring microhardness Zr phases at experimental alloys AlSi9Cu1Mg*

Measuring device	Hanemann typ Mod 32	Load time	10 sec
Optical microscope	Neophot 32	Eyepiece	15
Load	5 p	Temperature	15 °C
Converter	8	Numbers of punctures	2 punctures on phases, 10 on sample

Thanks to the evaluation of microhardness, it was possible to define the effect of Zr phases on the hardness increase of the AlSi9Cu1Mg alloyed metal matrix under investigation. Unlike the macrohardness, which is evaluated based on puncture focused over a larger area, the resulting microhardness defines the real hardness of the structural component to be evaluated. On this basis it is then possible to define the effect of the individual alloying additions on the characteristics of the emerging intermetallic phases in the metal matrix. In this case, the aim was to determine the hardness of the Zr phase relative to the basic structural components such as α phase and eutectic silicon. The measurement was carried out with Hanemann Mod 32 microhardness tester. The device uses the Vickers method, with the measuring body embedded in the front lens of the objective. The advantage of the hardness tester was the direct measurement of the created indentation. The calculation of the resulting microhardness was performed by substituting the measured values into the relation (1).

$$HVM = \frac{1854{,}4\ .\ p}{F^2}, \qquad (1)$$

Where:
p ... Load [N]
F^2 ... Length of diagonal [mm]

The highest average microhardness 268 HVM 5 was measured in an alloy with the addition of Zr 0.20 wt. %. In contrast, the lowest average microhardness value of 195 HVM 5 was measured in an alloy with the addition of Zr of 0.30 wt. %. The difference between the highest and lowest average microhardness values is almost 37 %. For an alloy with the addition of 0.25 wt. % microhardness was achieved close to an alloy containing 0.20 wt. % Zr. As mentioned above, a strong interaction of Zr and Fe phases is observed at higher Zr contents. This may result in a decrease in the microhardness of the excluded Zr phases due to disruption of phase homogeneity or changes in the phase chemical composition. The idea builds on the measured microhardness values of the Zr phases in the variant with the addition of 0.20 wt. % Zr (Fig. 6.). The punctures 3, 4 and 5, 6 were carried out into the Zr phases where no interaction with the Fe phases occurred, nor the exclusion of the Fe phases near the measured Zr phases. The remaining punctures were performed into the Zr phases that interacted with the Fe phases. After comparing the values of punctures into the Zr phases with and without interaction with Fe phases, the resulting difference between them is approximately 11 %. The average values of microhardness of Zr phases and basic structural components are given in Table 4.

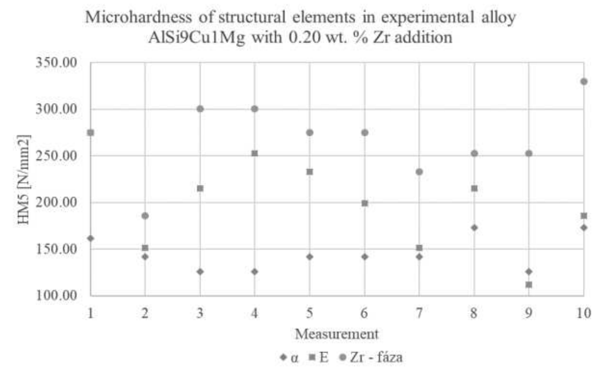


Fig. 6 *Microhardness values of experimental alloy AlSi9Cu1Mg with addition 0.20 wt. % Zr*

Tab. 4 *Average values of microhardness HVM 5 of structural components experimental alloy AlSi9Cu1Mg*

Addition Zr [wt. %]	0.20	0.25	0.30
α [HVM 5]	145.37	156.34	119.66
E [HVM 5]	199.12	202.58	143.05
Zr – phase [HVM 5]	268.18	264.22	195.32

2.2 Effect of Zr on porosity

Due to the deliberate absence of degassing of experimental AlSi9Cu1Mg alloys, it was possible to observe a sharp increase in the density index (DI) with increasing Zr content in the alloy. The resulting mechanical properties of Al alloys are closely related to the solubility rate of hydrogen in the melt. The most significant increase in the DI density index is observed for variants with a Zr content > 0.10 wt. % (Fig. 7.). The absence of melt degassing and the alloying process contributed significantly to the gassing increase in AlSi9Cu1Mg experimental alloys. The DI density index increase is already observed in the experimental alloy with the addition of Zr 0.05 wt. % where the DI density index is 14.5 %. This represents an increase by 32 % over the reference alloy, which reached a DI density index of 11 %. A sharp increase occurs in alloys at a Zr content from ≥ 0.15 wt. % up to 0.25 wt. %, where the DI density index ranges from 17 to 19 %. This represents a 57 to 80 % increase over the reference alloy. With the given the Zr contents, large Zr phases are excluded, as can be seen in Figure 5. In the macrostructural evaluation of the porosity of individual experimental samples (Fig. 8.), the samples with the addition of Zr 0.20 wt. % and 0.25 wt. % (Fig. 8c. and 8d.) manifested exclusion of hydrogen in the form of larger pores than in the reference samples or samples with added 0.10 wt. % Zr (Fig. 5a. and 5b.). The total area of the pores formed on the observed macrostructure surface was 2.08 % for experimental alloys with the addition of 0.20 wt. % Zr, and in the variant with 0.25 wt. % Zr the total area was 2.04 %. This represents an increase by approximately 0.5 % over the reference sample. Compared to an alloy with the addition of 0.10 wt. % Zr it represents an increase by 30 %. At the same time, as can be seen for alloys with the addition of 0.20 to 0.25 wt. % Zr (Fig. 8c. and 8d.), the formation can be observed of larger pores than that of the reference alloy (Fig. 8a.). It can be concluded that the addition of Zr promotes an increase in gassing in the AlSi9Cu1Mg alloy. Hydrogen bubble entrapment may also be associated with the shape of the excluded Zr phases. Larger phases can act as "catchers" of excluded hydrogen bubbles. The Zr phases are already excluded at 630 °C, where the formation of germs could trap excluded hydrogen bubbles.

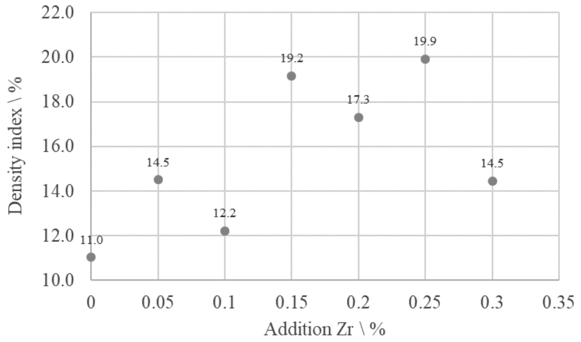


***Fig. 7** Dependence of DI density index on Zr concentration in AlSi9Cu1Mg alloys*

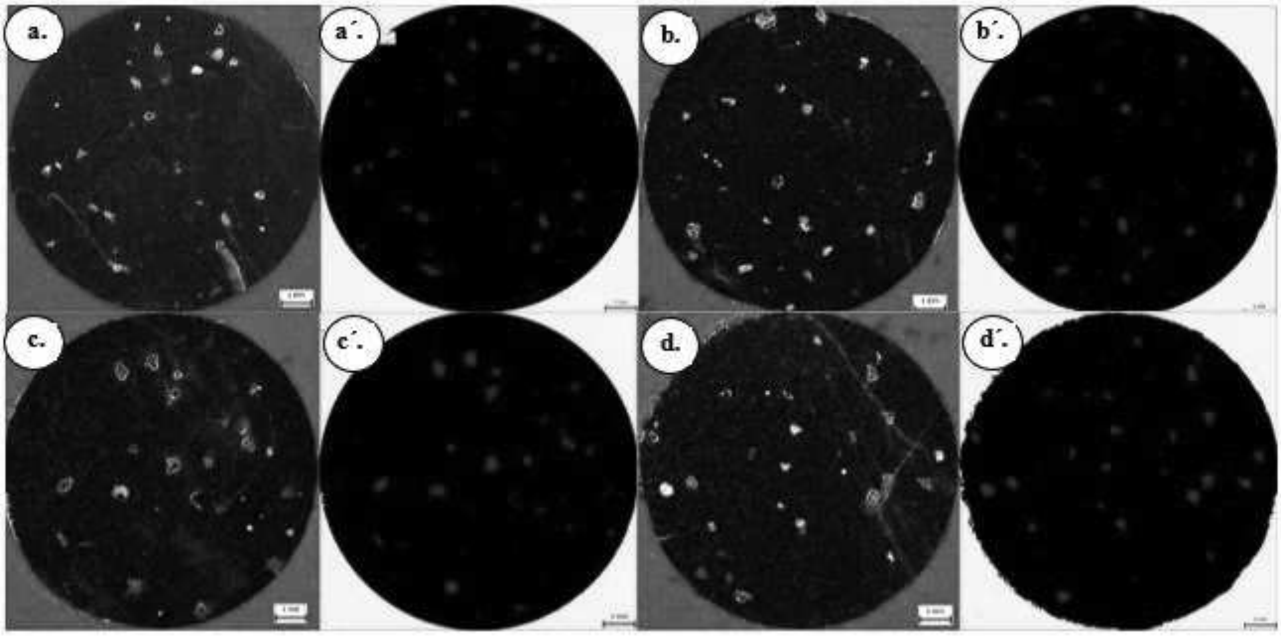


***Fig. 8** Macrostructure of AlSi9Cu1Mg a. 0.00 wt. % Zr, b. 0.10 wt. % Zr, c. 0.20 wt. % Zr, d. 0.25 wt. % Zr and analytical evaluation of total pore area using Quick photo Industry software a´. 0.00 wt. % Zr, b´. 0.10 wt. % Zr, c´. 0.20 wt. % Zr, d´. 0.25 wt. % Zr*

When observing the cooling curves (Fig. 9.) of the evaluated experimental alloys, the exclusion changes occur mainly of Zr and α phases. Table 5. clearly shows that up to 0.10 wt. % Zr added, the exclusion temperatures of the α phase and eutectics are stable. The Zr phase is excluded at 630 °C with Zr 0.10 wt. % added. The change occurs when Zr 0.25 wt. % and 0.30 wt. % are added. The exclusion of Zr phases at given contents shifted from 630

°C to 619 °C. The α phase exclusion shifted to a temperature of 621 °C, representing an increase by 5 °C over the reference alloy. From the above, it can be concluded that the effect of Zr on the exclusion temperatures of individual structural components in the experimental AlSi9Cu1Mg alloy is significant. Similar conclusions have been confirmed by Hermandez-Sandoval's work [9]. The assumption of Zr phase exclusion at given temperatures is based on the results of microstructure evaluation, where the occurrence of individual Zr phases was confirmed (Fig. 5.). For experimental alloys with the addition of Zr from 0.20 wt. % to 0.30 wt. %, there is a higher degree of supercooling than in experimental alloys with the addition of Zr 0.05 wt. % and 0.10 wt. %. A higher degree of hypothermia represents the exclusion of a large number of Zr phases of larger dimensions. On this basis, hydrogen bubbles may be trapped during Zr phase germ exclusion. [8, 9]

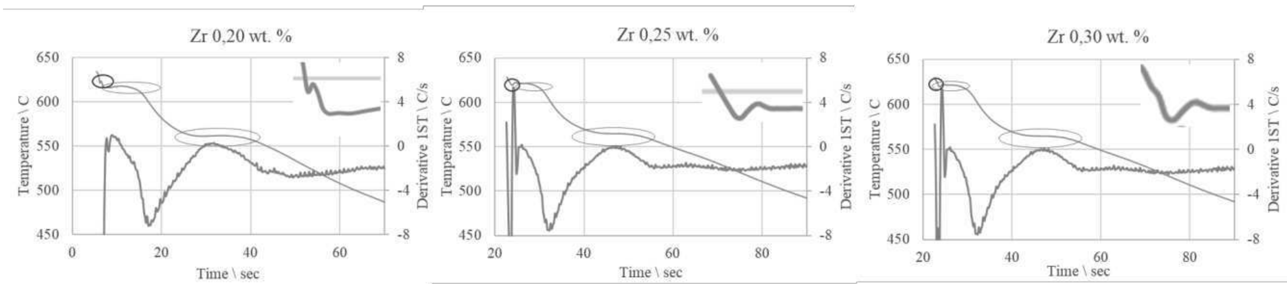


***Fig. 9** Graphs of cooling curves of experimental alloys AlSi9Cu1Mg with graded amount of Zr*

***Tab. 5** Base phase deposition temperatures for individual AlSi9Cu1Mg experimental alloys*

Variant	0.00 wt. %	0.05 wt. %	0.10 wt. %	0.20 wt. %	0.25 wt. %	0.30 wt. %
α phase (°C)	616	618	618	616	620	621
Eutectic Al-Si (°C)	565	564	564	561	564	564
Zr phase (°C)	-	630	630	623	619	619

3 Evaluation

The increase in the mechanical properties values occurs mainly in experimental alloys with the addition of Zr 0.20 wt. % and 0.25 wt. %. Comparison of mechanical properties of experimental samples cast into ceramic molds with STN EN 1706 standard for samples cast in sand found significantly higher values. The improvement of R_m and $Rp_{0.2}$ in reference alloys without heat treatment is by approximately 35 % compared to STN EN 1706. After alloying the Zr alloy, these values increased by about 15 %. Improvement also occurs in A_M, where there is an increase by up to 90 %. In terms of HBW hardness, the improvement is in the range of 13 to 20 %. After the T6 precipitation curing heat treatment, the mechanical properties of the experimental alloys reached values close to those when cast into metal molds. Even when Zr 0.10 wt. % is added, Zr phase exclusion can be observed. Especially at higher contents of Zr ≥ 0.20 wt. % Zr phases are excluded in the form of large smooth acicular needles intersecting the α phases and the eutectic. By eliminating the Zr phases, there is a noticeable slight increase in R_m. Thus, the Zr phases may have a reinforcing effect on the metal matrix of the AlSi9Cu1Mg alloy under investigation. After heat treatment, at higher Zr contents, large acicular phases break down into smaller plate-like phases. By breaking down into smaller plate-like phases, the E value increased by approximately 3 % compared to the experimental variant with an addition of 0.15 wt. % Zr and 6 % compared to the reference alloy (without Zr addition). Based on the measured results, the metal matrix of the experimental AlSi9Cu1Mg alloy is strengthened by the Zr phase breakdown into smaller plate-like phases. The hardness of the excluded Zr phases reaches values in the range of 200 to 260 HVM 5. In particular, the experimental variants containing 0.20 wt. % Zr achieve an average microhardness of Zr phases of about 268 HVM 5. A decrease in microhardness was observed mainly in those Zr phases in which there was an interaction with Fe phases. Thus, the Fe phases may have a negative effect on reducing the hardness of the individual Zr phases excluded. The measured hardness of the Zr phases is at the interface of the hardness of phases such as Al_4Ca and Al_6CuMg_4. Based on structural analyzes it can be concluded that Zr acts as a corrector in suppressing the negative effect of Fe phases, i.e. it affects the morphology of Fe particles. Increasing the amount of skeletal formations or clusters of Zr and Fe phases influences mechanical properties. When evaluating the gassing of experimental alloys with hydrogen, a higher gassing rate was found for alloyed alloys. For experimental variants with a Zr content > 0.10 wt. % there was a sharp increase in the DI density index (Fig. 7.). The worst results were achieved in variants with the additions of 0.15 wt. % and 0.25 wt. % Zr (Fig. 4.). When evaluating the metallographic sample macrostructure, an increased concentration of larger pores was observed than in the reference alloy and the variant with 0.10 wt. % Zr added. With a theoretical area of 100 mm^2, a 0.5 % increase would represent an area of 5 mm^2. Also interesting is the change in the cooling curve with a significant degree of supercooling during the Zr phase exclusion. For experimental alloys with Zr ≥ 0.20 wt. % addition with a significant degree of supercooling during the Zr phase exclusion, there was a sharp increase in the DI density index. Zr can increase the gassing rate either in the form of a hydrogen solubility enhancing element in the experimental alloy, or the excluded Zr phase nuclei trap the excluded hydrogen bubbles above the Zr content ≥ 0.10 wt.%.

4 Conclusion

It can be concluded that the addition of Zr to the alloy under investigation significantly affects the mechanical properties of the alloy. Hardness and microhardness values were closely related to the formation of Zr phases in interaction with Fe phases. Zr acted as a corrector in the AlSi9Ci1Mg alloy, i. e. it influenced the morphology of acicular iron phases. When evaluating the gassing, the experimental alloys with a higher content of Zr ≥ 0.15 wt. % reached a higher DI density index. On this basis, it is believed that Zr increases the gassing rate and contributes to the formation of porosity in the castings. Positive is achievement of mechanical properties after casting into ceramic molds, which are close to values of samples cast into metal molds for and after heat treatment defined by STN EN 1706.

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