EFFECT OF AlTi3B1 INOCULANT ON THE MICROSTRUCTURE AND HARDNESS OF THE AlCu4Mg1 ALLOY

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Abstract

This paper focuses on the effect of AlTi3B1 grain refiner on the microstructure and mechanical properties of AlCu4Mg1 alloy. Inoculant in the form of a master alloy with a titanium content of 3 [wt%] and boron content of 1 [wt%] was added to the alloy in various amounts. Boron is completely bound in the insoluble TiB_2 phase, which is usually very finely precipitated in the master alloy. The residue of the titanium content is precipitated in the form of polyhedral particles of the melt-soluble intermetallic phase $TiAl_3$. Inoculation of aluminium alloys is performed in order to improve the mechanical and technological properties of the material. In the case of inoculation, this improvement is mainly due to an increase in chemical and structural homogeneity and a decrease in the tendency to segregate individual elements. Optical microscopy was used to observe the microstructure, which evaluated the structure and its changes depending on the content of the inoculant and also the effect of heat treatment. In this experiment, we further focused on the hardness of the AlCu4Mg1 alloy after heat treatment and the addition of various amounts of inoculant. Hardness was evaluated from both a macro and a micro perspective using the Brinell and Vickers method. This is because the microhardness of the alloy can be significantly affected by the occurrence of chemical heterogeneity in the solidified casting.

Keywords: AlCu4Mg1, inoculation, AlTi3B1, heat treatment, microhardness, microstructure

1. INTRODUCTION

Grain size is one of the key factors that affect microstructure and metal/alloy properties. Fine structure leads to a more dispersed and refined porosity distribution, to a decreasing tendency to hot tearing, improve directional feeding characteristics, cause defects in hot spots, although it can also reduce fluidity. Reducing of the grain size has also influence on properties, e.g. strengthening through the Hall-Petch relationship, improved wrought processing by reducing the recrystallized grain size and achieving a fully recrystallized microstructure more easily [1]. In the early studies, it was found that the addition of other elements to the metal before solidification leads to a transition from columnar to equiaxed grain morphologies, although the effects of second phases and eutectic were also observed. The modern grain refiners for Al-based alloys are described in the work [2,3]. Inoculation of hypoeutectic aluminium alloys is performed with Ti or a combination of Ti and B. These elements are added to the melt using inoculum salts. (e.g. K_2TiF_6, KBF_4), inoculum tablets or in the form of Al-Ti or Al-Ti-B master alloys. During the titanium inoculation, Ti reacts with Al to form an intermetallic phase $TiAl_3$. This phase has the same grid as Al (K12) with parameters close to the grid of Al. That is why it is such an ideal crystallization nucleus. Phase $TiAl_3$ reacts with melt Al in a peritectic reaction at temperature 665 °C. Around the particle, $TiAl_3$ an envelope of solid solution forms α(Al) and further growth of aluminium dendrites continues. $TiAl_3$ form relatively coarse formations with a size of up to 100 μm. The finer the dispersion of $TiAl_3$, the more effective is the inoculant effect of the master alloy. Nuclei of $TiAl_3$ gradually dissolve in the melt and the inoculum effect fades over time. Effective action is about 30 to 45 minutes. As for effect B, this element itself does not act as an inoculant in the alloy. With Al, it forms intermetallic phases AlB_2,

TiB_2 or $(Al,Ti)B_2$ with size 0,5-2 μm. These particles then serve as nuclei. Due to B, the fading rate is significantly slowed down and the inoculum effect even lasts even after remelting [4,5].

The mechanical properties of aluminium alloys depend mainly on the type and properties of the base metal, on the dispersion of the structural components, on the presence and shape of the intermetallic phases and the heat treatment. The fine-grained structure improves all mechanical properties as well as many technological properties of alloys. From the point of view the mechanical properties, the tensile strength at normal temperature, yield strength ($R_{p0,2}$), ductility and hardness is usually monitored. In some cases, properties at elevated temperatures, fatigue properties or dimensional stability are important. The mechanical properties are also greatly influenced by the cooling rate, therefore they must be evaluated on samples solidifying under comparable conditions. The strength limit of common aluminium alloys in the cast state (depending on the alloy, casting method and wall thickness) is in the range of about 150-250 MPa. The strength properties are greatly increased by hardening. To hardenable alloys belongs the alloys type Al-Si-Mg, Al-Si-Cu and Al-Cu. High-strength alloys include in particular alloys of the type Al-Cu. These alloys reach a tensile strength of up to 350 MPa at high ductility. However, the disadvantage of these alloys is the very unfavorable foundry properties [4,6,7]. The Al-Cu-Mg alloy belongs to the group of alloys with higher and high strength, but with low corrosion resistance. The most used materials of this group are mainly dural AlCu4Mg, AlCu4Mg1 and AlCu4Mg1Mn, achieving considerable strength after curing by heat treatment (R_m to 530 MPa). The maximum solubility of copper in solid aluminium solution is under equilibrium conditions is 2.48 at% (~ 5.7 wt%) Cu at the eutectic reaction temperature 548.2 °C (**Figure 1**) [4].

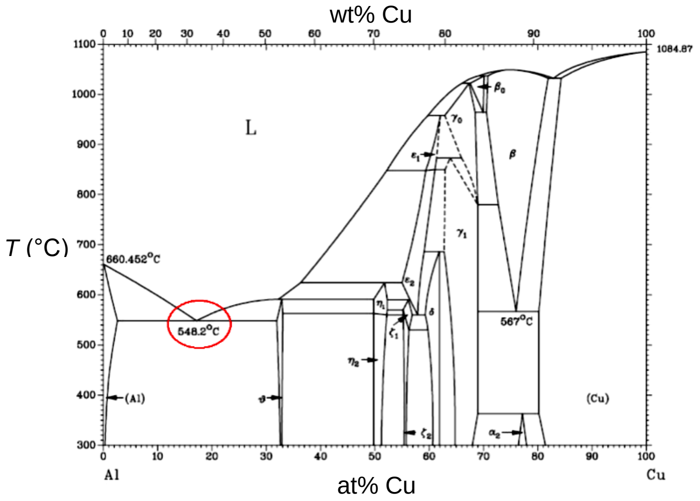

Figure 1 Binary diagram Al-Cu [4]

Al – Cu alloys mechanical properties depends on whether Cu is present in a solid solution in the form of a spheroidal or dispersed particle, or whether it forms a network at grain boundaries [4,6,7].

The article deals with the influence of AlTi3B1 inoculant on the microstructure and mechanical properties (hardness) of AlCu4Mg1 alloy (EN AW 2024) depending on the inoculant content AlTi3B1 from 0 to 5 wt% and heat treatment. The structure analysis also included image analysis of the fraction (phase analysis) and grain size analysis performed on an optical microscope with PC and software. Stream Essentials. It is a solution of a specific case of alloy application, inoculation and heat treatment parameters in practice. This experiment is part of wider research, which is focused on increasing the mechanical properties of this particular alloy by inoculating AlTi3B1 and AlTi5B1 in various concentrations with subsequent heat treatment. In the paper we present the results AlCu4Mg1, inoculated with AlTi3B1 in concentrations 0; 0.1; 0.3; 0.5; 1; 3; 5 (wt%) (the experiment is, of course, more extensive, but for the purposes of this paper only these concentrations are presented).

2. EXPERIMENTAL PART

AlCu4Mg1 alloy was used for the preparation of castings. This alloy was supplied by the manufacturer with a chemical composition according to the standard CSN EN 573-1 Aluminium and aluminium alloys - Chemical composition and types of wrought products - Part 1: Numerical designation. The aluminium alloy was melted in an induction furnace at 720 °C, the furnace temperature was captured using a digital thermometer with an accuracy of ± 2 °C. The melt was treated during melting by refining salt and the smear was shut down from the melt surface. At the end of the melting process, AlTi3B1 wire was added to the alloy in concentrations 0; 0.1; 0.3; 0.5; 1; 3; 5 (wt%). Graphite crucibles were used for casting. The heat treatment parameters were set by heating the castings 500 °C, withstanding at the temperature of 360 min. and cooling to water, subsequent ageing was unaffected.

2.1. Optical microscopy analysis and image analysis

Metallographic experimental samples were prepared from castings by the classical procedure of preparation of metallographic samples (cutting, grinding, and polishing). Thus prepared samples were observed and scanned by confocal laser microscope. The binary eutectic α+$CuAl_2$ mainly occurs in the alloy AlCu4Mg1 (type AlCuMg) and a small amount of ternary eutectic α+$CuAl_2$+S ($Cu_2Mg_2Al_5$). In addition to these basic phase components, there may be other phases, namely: Mg_2Si, $FeAl_3$, AlFeMnSi, AlCuFeMn etc., **Figure 2**.

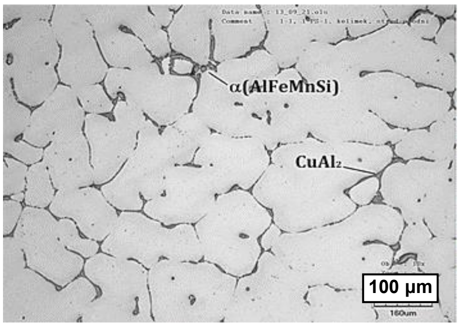

Figure 2 Structure of the AlCu4Mg1 alloy in the cast state, one of the most common phases of the duralumin type is $CuAl_2$ (creates cross-linking along grain boundaries), without heat treatment, sample 1, 0 wt% AlTi3B1

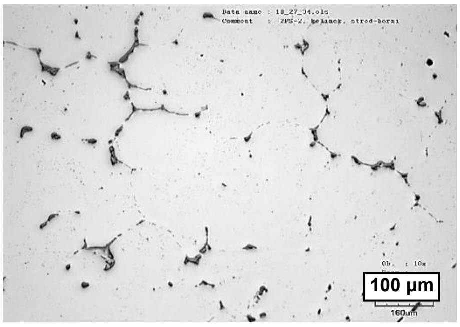

Figure 3 Structure of the AlCu4Mg1 alloy, after heat treatment, dissolution of phases, sample 2, 0 wt% AlTi3B1

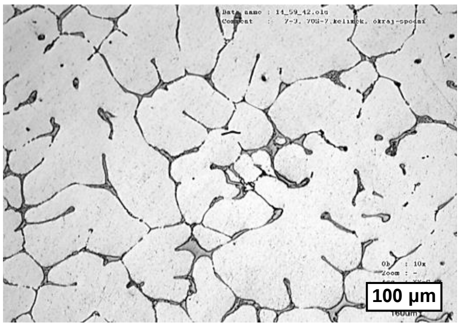

Figure 4 Structure of the AlCu4Mg1 alloy, without heat treatment, sample 7, 0.5 wt% AlTi3B1

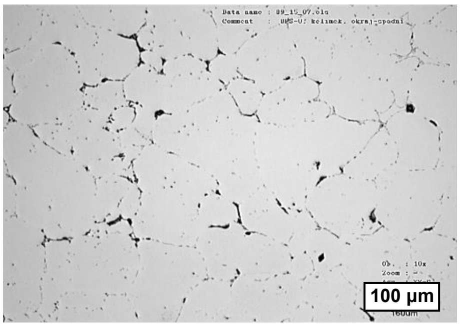

Figure 5 Structure of the AlCu4Mg1 alloy, after heat treatment, sample 8, 0.5 wt% AlTi3B1

Figures 2 to **5** document the structure of the material before and after heat treatment and with different addition of AlTi3B1 inoculant. Thanks to the heat treatment, the phases dissolved in the α matrix. A favorable effect of the inoculant on grain refinement was also observed when examining the structure. For the purposes of this paper, only representatives from all experimental samples (samples 1, 2, 7 and 8) are listed. Grain size measurements were performed on metallographic sections by image analysis of Grain Intercept, resp. measurement Grain size number G according to ASTM.

The influence of heat treatment can be monitored by means of phase analysis, from which we obtain, for example % fraction. In the phase analysis, we measure the proportion that the phase area occupies in the experimental samples. The results of the phase analysis are shown in **Table 1**, the analyzed area with the drawn phases that were analyzed is shown in **Figure 6**. The results are plotted in **Figure 8** (samples after heat treatment are marked in yellow).

Table 1 The image analysis results – fraction %, Grain size number G

Sample	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Fraction %	7.01	3.71	4.58	3.35	5.13	4.49	9.60	4.77	7.51	3.98	6.74	4.18	7.88	6.37
Heat treatment	no	yes	no	yes	no	yes	no	yes	no	yes	no	yes	no	yes
% AlTi3B1	0	0	0.1	0.1	0.3	0.3	0.5	0.5	1	1	3	3	5	5
Grain size number G	1.76	-	2.26	-	3.32	-	2.93	-	2.48	-	2.93	-	3.41	-

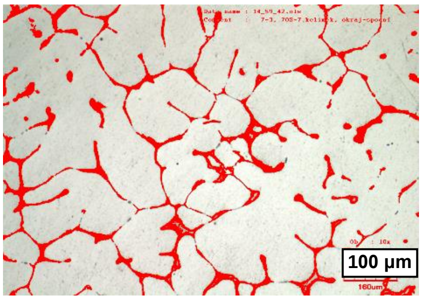

Figure 6 Phase analysis – fraction %, sample 7

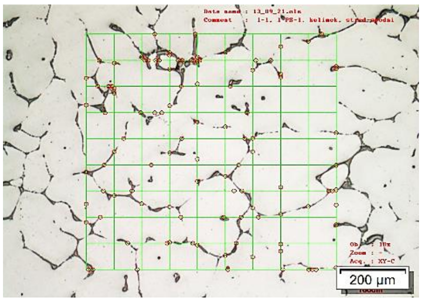

Figure 7 Image analysis grain size, sample 1

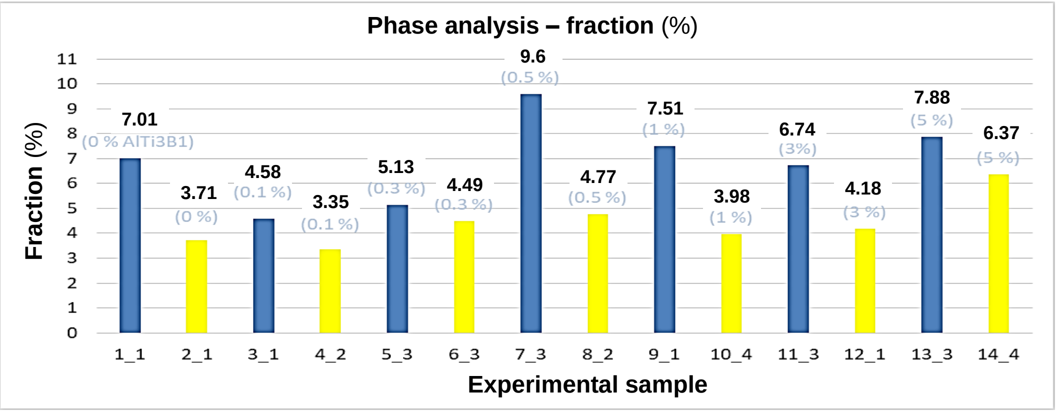

Figure 8 Graphical representation of the phase analysis result

The ASTM E112-13 grain size number, G, is defined as: NAE = 2G-1 where NAE is the number of grains per square inch at 100X magnification. Grain boundary intersection count: Determination of the number of times a test line cuts across, or is tangent to, grain boundaries, **Figure 7**.

Phase analysis showed the effect of heat treatment on a number of phases. Due to the heat treatment of the experimental samples, the phases dissolved and this corresponds to the proportion of the fraction before and after the heat treatment. After heat treatment, the fraction is always lower. In this analysis, the results are greatly influenced by the quality of the metallographic sample and also by the place from which the observed sample was taken, resp. from which part of the casting. Grain size measurements confirmed the effect of the inoculum on grain size. The grain size was measured on unheated samples. The results are shown in **Table 1**. ASTM grain size number increases with decreasing grain size.

2.2. Hardness and toughness measurement

The hardness test of castings made of AlCu4Mg1 alloy was performed according to the standard CSN EN ISO 6506-1 on a hardness tester Ernst Harrteprufer AT 250 at nominal load value HBW5 (F = 1 225 N, 125 kgf), which acted on the test specimen (ball with a diameter of 5 mm) for 10 s. The results of the Brinell hardness measurements are plotted in **Figure 9** (samples after heat treatment are marked in yellow). The microhardness test of castings from AlCu4Mg1 alloy was performed according to the standard CSN EN ISO 6507-1 on the microhardness tester Mitutoyo HM-220 at the nominal value of load HV 0,2 (F = 1,961 N, 200 g), which acted on the test specimen for 10 s. **Table 2** shows the Brinell and Vickers hardness measurements and the calculated (orientation) value R_m. The table shows the arithmetic mean of 20 measurements for all experimental samples ($\bar{x}$), standard deviation (σ) and variance of values (σ^2).

Table 2 Hardness measurement HBW, HV

HBW/Sample	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**
$\bar{x}$	71.1	82.0	74.2	98.6	77.7	102.0	77.9	88.1	76.4	84.1	75,9	87.5	74.8	85.9
σ	1.2	2.1	1.2	3.4	11.6	5.7	3.0	2.6	3.5	6.8	3.8	2.3	4.8	2.7
σ^2	1.5	4.4	1.4	11.6	2.6	32.4	8.9	6.9	12.2	46.1	14.5	5.2	23.0	7.4
HV/Sample	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**
$\bar{x}$	93.6	94.0	68.6	99.2	86.8	109.5	92.5	100.9	84.7	111.9	69.3	109.9	90.4	97.0
σ	21.9	5.4	9.7	12.5	13.5	10.7	7.4	3.7	8.3	3.9	7.2	7.3	11.3	8.3
σ^2	479.8	29.4	94.3	155.2	182.5	113.8	55.3	13.4	68.7	14.9	51.8	52.7	127.2	69.4
R_m (MPa) **(calc.)**	184.9	213.2	192.9	256.4	202.0	265.2	202.5	229.1	198.6	218.7	197.3	227.5	194.5	223.3
AlTi3B1 (wt%)	0	0	0.1	0.1	0.3	0.3	0.5	0.5	1	1	3	3	5	5

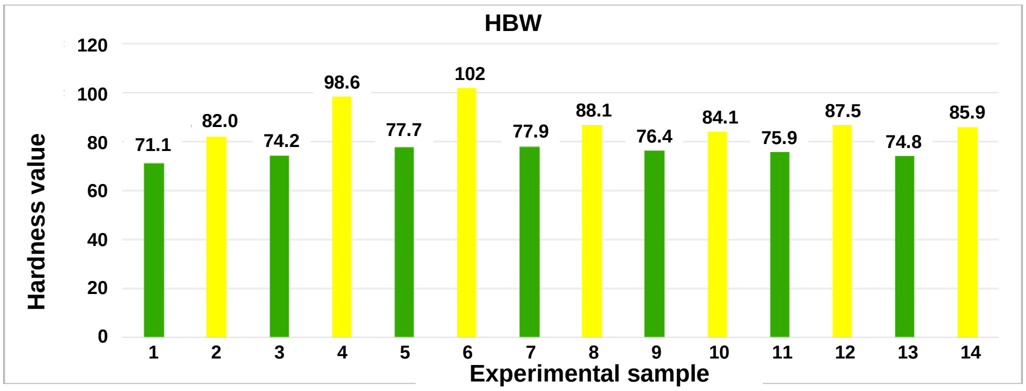

Figure 9 Graphical representation of the hardness measurement result HBW

The results of the measurements show a favorable effect of the inoculant and heat treatment on the hardness of the AlCu4Mg1 alloy. The hardness values were found to increase with increase of AlTi3B1 content, which is mainly attributed to the refinement of grains. Brinell measurements indicate that due to the AlTi3B1 inoculant content is the most preferred concentration 0.3 a 0.5 wt%. At this content, the hardness was highest. We achieved the increase in hardness by heat treatment. The results of Vickers measurements were significantly affected by the occurrence of chemical heterogeneity in the solidified casting. Defections in variance values and standard deviation are caused by structural inhomogeneity, where especially in the case of Vickers measurements, hardness was affected with the hard brittle intermetallic particles, whose hardness is different from the primary α dendritic structure. Due to the technology of gravity casting into graphite crucibles in laboratory conditions, there were pores in the structure, which are not a favorable defect in terms of mechanical properties.

3. CONCLUSION

In the present work, the effect of AlTi3B1 grain refiner on microstructure and mechanical properties of AlCu4Mg1 allow was studied. The following conclusions can be drawn based of the experimental results:

- the addition of AlTi3B1 master alloy reduced the grain size of AlCu4Mg1 alloy,
- the heat treatment was evaluated by means of phase analysis and its favorable effect on the structure was evaluated by means of phase analysis
- the proportion of fraction was lower in the samples after heat treatment,
- due to the favorable results of the image analysis (reduction of grain size, dissolution of intermetallic phases after heat treatment) a favorable effect of the inoculant on the mechanical properties was expected
- mechanical properties of AlCu4Mg1 alloy were improved by the addition of AlTi3B1 master alloy.
- at a content 0.3 wt% of AlTi3B1 the hardness increased from 71 HBW to 78 HBW and from 82 HBW to 102 HBW after heat treatment.
- the results are significantly affected by the sampling site for metallographic sample,
- however, the results can be used to assess the beneficial effect of the addition of inoculant on the microstructure and mechanical properties of the contents 0.3 and 0.5 wt% AlTi3B1 for specific AlCu4Mg1 alloy processing conditions.

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