EFFECT OF AlTi5B1 INOCULANT ON THE MICROSTRUCTRE AND HARDNESS OF THE AlCu4Mg1 ALLOY

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https://doi.org/10.37904/metal.2021.4267

Abstract

Aluminum alloy EN AW-2024 (AlCu4Mg1 - dural) – Al-Cu-Mg group is one of the most used alloys from this group, which achieves considerable strength after heat treatment. It is an alloy with higher and high strength, but low corrosion resistance. The mechanical properties of Al-Cu alloys depend on whether Cu is present in the aluminum solid solution α in the form of a spheroid, possibly as dispersed particles or if it is forming a network at grain boundaries. The paper is focused on the evaluation (microstructure, grain size, phase fraction, hardness) of this particular alloy using AlTi5B1 inoculant in various concentrations to refine the alloy grain. The heat treatment of the alloy was also performed in the experiment to increase the mechanical properties. Inoculation of aluminum alloys is performed 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 and image phase analysis was used to observe the microstructure, which evaluated the microstructure and its changes depending on the content of the inoculant and also the effect of heat treatment in the combination of different amount of inoculant. It is generally known that AlTi5B1 acts in certain amounts to refining the alloy grain. Our work is based on the knowledge of the behaviour of different concentrations of inoculant in this particular AlCu4Mg1 alloy and responds to the requirement from practice. We achieved the best results with a concentration 3 wt% of AlTi5B1. For the other concentrations, the results were almost comparable and not much worse than for the concentration with the best results. It doesn't make sense to add more than 3 wt% of inoculant in this case.

Keywords: Metallurgy, inoculation, AlCu4Mg1, heat treatment, microstructure

1. INTRODUCTION

Aluminum and its alloys are materials that have been tested for many years and are used for various applications. The most characteristic property of aluminum is its low weight (3 times lower than Fe) and low melting point (about 2.5 times lower than Fe). Another advantage of aluminum and its alloys is good corrosion resistance due to the formation of a thin and resistant (passive) Al_2O_3 layer. Aluminum and its alloys are therefore suitable for use in the construction of aircraft, automobiles, various building structures, etc. AlCu4Mg1 is an aluminum alloy for forming with higher and high strength but low corrosion resistance. There is mainly a binary eutectic α + $CuAl_2$ and a small amount of ternary eutectic α + $CuAl_2$ + $Cu_2Mg_2Al_5$ in the alloy AlCu4Mg1 (type AlCuMg). In addition to these basic phase components, there may be other phases, namely: Mg_2Si, $FeAl_3$, AlFeMnSi, AlCuFeMn, etc. This type of alloy is used for various applications from the aerospace, automotive and construction industries. The structure and properties of these materials can be influenced by several factors, such as a change in chemical composition, the addition of grain refining agents, minimization of inclusions and impurities, or the use of heat treatment [1,2].

When we use high concentrations of alloying elements than inhomogeneity in the structure and strong segregation of the secondary phase can occur. For castings, mechanical properties may vary from location to location due to changes in grain size, amount of eutectic phase, and amount of precipitates. Much attention has been paid to reducing the segregation of alloying elements during the setting time of high-alloyed Al alloys [2]. Grain size is one of the most important factors that affect the properties of an alloy. Grain refinement has certain advantages, such as a homogeneous distribution of secondary phases, better feeding to eliminate shrinkage porosity, better strength and better fatigue life [1, 3]. It also reduces the possibility of hot cracking and dispersed and refined porosity distribution. Hall-Patch material strengthening is one of the important methods of grain refinement [3,4].

By adding grain refining agents (inoculants) we achieve favourable changes in the microstructure (refining of α-Al solid solution) and mechanical properties of the material. There are several possibilities, but in our paper, we will focus on titanium and boron in combination. The refinement of the solid solution α with titanium and boron is carried out by the action of the $TiAl_3$ and TiB_2 phases, respectively $(Al,Ti)B_2$, which are added to the melt in the form of AlTiB type master alloys. AlTiB is the most commonly used ternary master alloy for grain refinement in aluminum and its alloys. Boron is completely bound in the insoluble phase TiB_2, AlB_2 or $(Al,Ti)B_2$, which is usually very finely precipitated in the master alloy. Both TiB_2 and AlB_2 phases have a hexagonal structure [5,3]. As stated [3,6] during the examination of Al-Ti-B master alloy was observed that $TiAl_3$ is in the centre of the solid Al grain whereas TiB_2 particles are on boundaries.

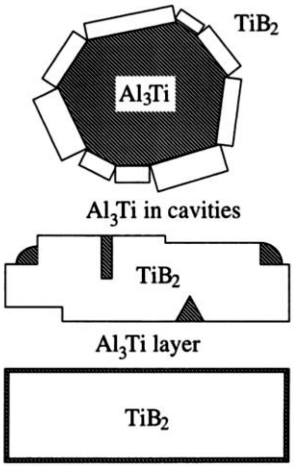

Figure 1 Schema of $TiAl_3$ and TiB_2 activity outside the melt composition [6]

If TiB_2 having powerful characteristics and surface free energy then it should have found TiB_2 in the centre, instead of it we found $TiAl_3$ in the centre as shown in **Figure 1**, which suggests TiB_2 is poor nucleant [3]. The rest of the titanium content is excluded in the form of polyhedral particles of the intermetallic phase of $TiAl_3$ which is soluble in the melt. The TiB_2 phase particles cannot act as active nuclei due to the difference of the crystal lattice with aluminum.

2. EXPERIMENT

We focused on the AlCu4Mg1 alloy in the experiment. The requirement to test this particular alloy using Al-Ti-B type inoculants was made by an unnamed company to determine the practical use. The experiment should answer the question which inoculant (AlTi3B1 [7] x AlTi5B1) is more suitable and in which concentration is more effective. The effect of AlTi3B1 was published in [7]. In this paper we focus on AlTi5B1 in various concentrations from 0.1 to 5 wt%. We prepared 1000 g of AlCu4Mg1 melt (for each concentration of inoculant), which was inoculated with AlTi5B1 after 30 minutes for the experiment. The melt cooled in small graphite crucibles. The aluminum 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. Part of the castings was heat treated at parameters: heating the castings 500 °C, withstanding at the temperature of 360 min and cooling to water, subsequent ageing was unaffected. 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 Olympus LEXT OLS5000. The image phase analysis was used for grain size measurement and intermetallic phase fraction. To evaluate the mechanical properties, the samples were subjected to Brinell hardness measurements. All samples were compared with each other and based on the results, the relevant conclusions were summarized. The chemical composition of the prepared alloys was determined with spectrometric analysis on device Tasman Q4 (OES). We can state that all samples had a suitable chemical composition in the range according to the standard for this material. Chemical composition of experimental alloys AlCu4Mg1 alloy is given below (EN AW-2024) in the **Table 1**.

Table 1 Chemical composition of the alloy AlCu4Mg1 with AlTi5B1

Element (wt%)	**Si**	**Fe**	**Cu**	**Mn**	**Mg**	**Cr**	**Zn**	**Ti**	**Al**	**Other**
EN AW 2024	max. 0.50	max. 0.50	3.8 – 4.9	0.3 – 0.9	1.2 – 1.8	max. 0.1	max. 0.25	max. 0.15	residue	max. 0.15
1	0.048	0.062	5.049	0.476	1.216	<0.002	0.255	0.043	92.73	residue
2	0.479	0.077	4.874	0.511	1.037	<0.002	0.261	0.117	92.53	residue
21	0.482	0.486	5.177	0.507	0.916	0.069	0.045	0.102	92.08	residue
22	0.362	0.486	4.675	0.503	1.035	0.079	0.035	0.108	92.56	residue
23	0.402	0.457	5.169	0.492	1.086	0.070	0.045	0.113	92.02	residue
24	0.355	0.490	4.512	0.506	1.281	0.079	0.032	0.110	92.47	residue
25	0.368	0.449	5.224	0.485	0.926	0.070	0.046	0.125	92.17	residue
26	0.343	0.441	4.576	0.486	1.186	0.078	0.031	0.122	92.57	residue
27	0.341	0.431	4.470	0.479	1.147	0.079	0.031	0.185	92.71	residue
28	0.329	0.523	4.128	0.516	0.977	0.080	0.029	0.187	93.09	residue
29	0.359	0.429	4.717	0.466	1.039	0.074	0.034	0.288	92.46	residue
30	0.349	0.449	4.547	0.475	1.037	0.078	0.032	0.253	92.60	residue
31	0.334	0.448	4.637	0.470	1.044	0.077	0.035	0.342	92.32	residue
32	0.337	0.407	4.313	0.450	1.054	0.075	0.031	0.349	92.87	residue

3. RESULTS AND DISCUSSION

3.1. Optical microscopy analysis and image analysis

Figures 2 and **3** document the basic structure of an alloy without inoculation, before and after heat treatment. The binary eutectic $\alpha+CuAl_2$ mainly occurs in the alloy AlCu4Mg1 (type Al-Cu-Mg) and a small amount of ternary eutectic $\alpha+CuAl_2+S$ ($Cu_2Mg_2Al_5$). In addition to these basic phase components, there may be other phases, namely: $FeAl_3$, AlFeMnSi, AlCuFeMn etc. [1]. **Figure 4** documents the structures of selected experimental samples after inoculation, without and with heat treatment (HT). There are no significant differences in the structures. To obtain specific data and quantify the effect of the inoculant (or its amount) on this particular alloy, image analysis (grain size measurement **Figure 5** and intermetallic phase fraction in **Figure 6** was performed.

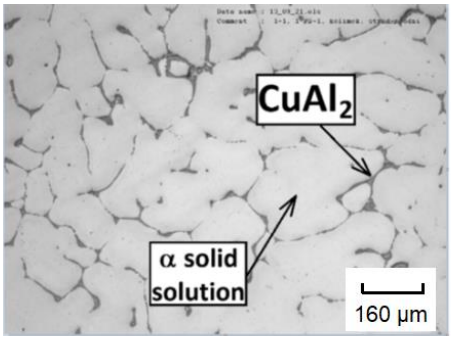

Figure 2 Structure of the AlCu4Mg1 alloy in the cast state, one of the most common phases of the duralumin type is $CuAl_2$ (creates cross-linking along grain boundaries), without heat treatment; sample no. 1; 0 wt% AlTi5B1

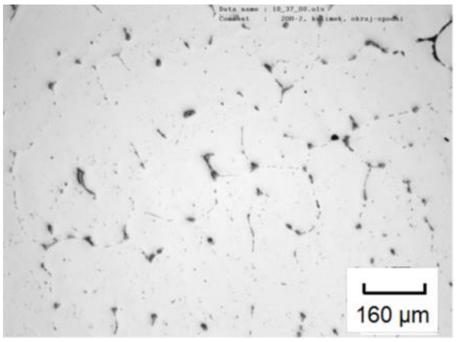

Figure 3 Structure of the AlCu4Mg1 alloy, after heat treatment, dissolution of phases; sample no. 2; 0 wt% AlTi5B1

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 using 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 2**.

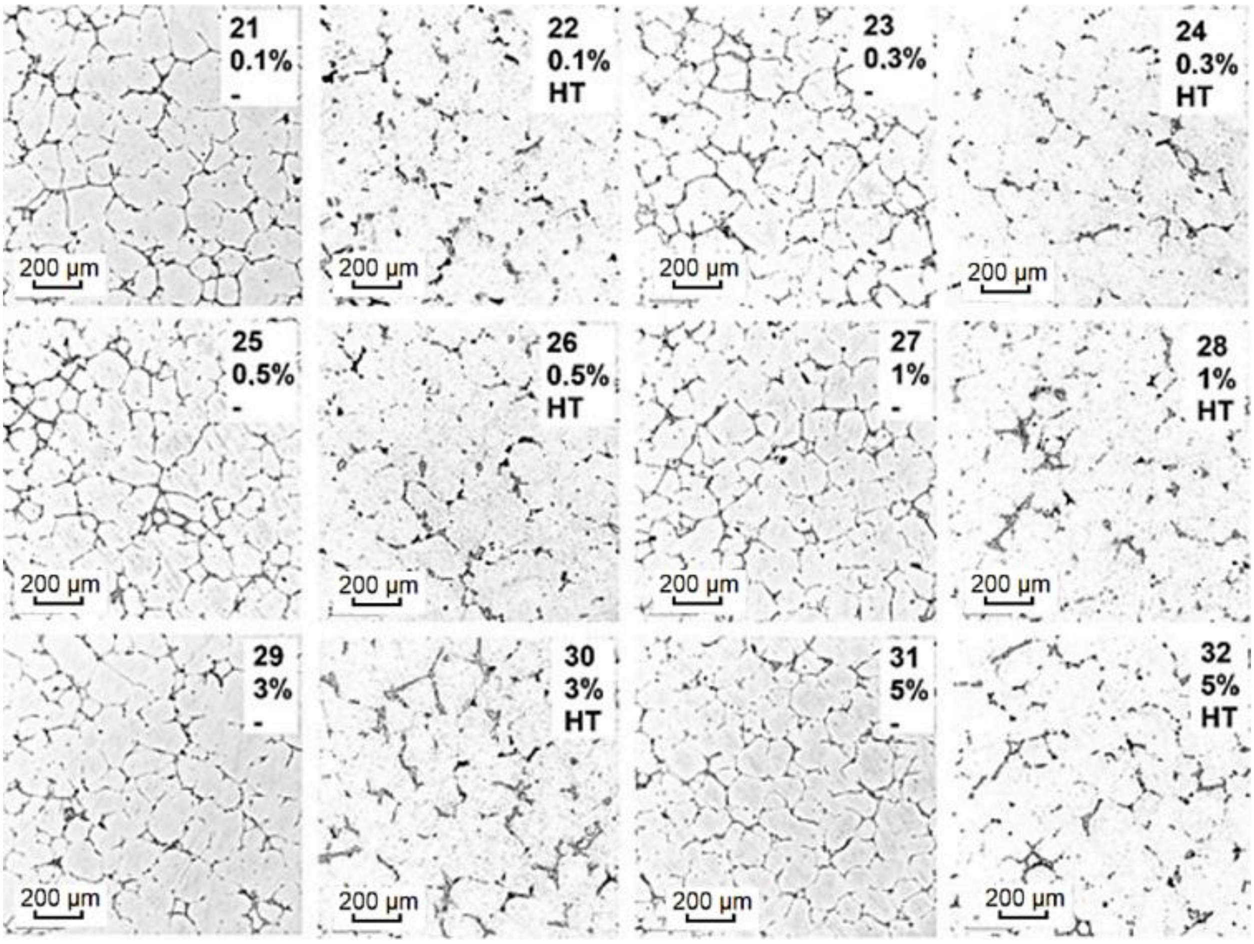

Figure 4 Structures of the experimental AlCu4Mg1 alloy

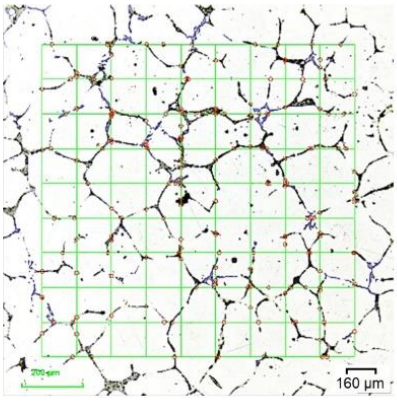

Figure 5 Grain size measurement (*G*)

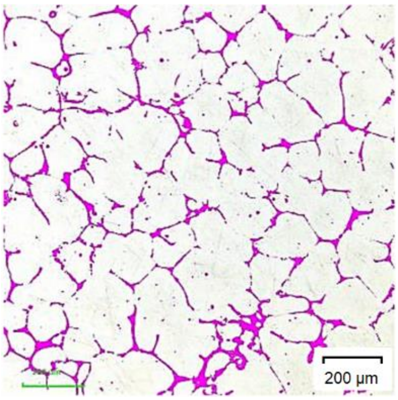

Figure 6 Intermetallic phase fraction (%)

Table 2 The image analysis results – fraction %, Grain size number *G*

Sample	**1**	**2**	**21**	**22**	**23**	**24**	**25**	**26**	**27**	**28**	**29**	**30**	**31**	**32**
% AlTi5B1	0	0	0.1	0.1	0.3	0.3	0.5	0.5	1	1	3	3	5	5
Fraction %	7.01	3.71	8.03	5.40	7.13	4.27	7.76	4.04	7.20	6.10	7.75	4.26	7.53	4.5
Heat treatment	-	✓	-	✓	-	✓	-	✓	-	✓	-	✓	-	✓
Grain size no.	1.76	-	2.97	-	3.06	-	2.93	-	3.02	-	3.79	-	2.93	-

As expected, the phase analysis showed a favourable effect of the heat treatment. The proportion of the fraction after heat treatment is converted into a solid solution in all samples of the smaller soluble phase. 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. The grain size is larger for all samples after the addition of the inoculant. We expected this result. The effect on grain size itself is interesting, when we expected differences in individual concentrations. The best result was obtained with sample 29 (3 wt% AlTi5B1, without HT, see **Figure 7**). There is almost no difference in the other samples. It is therefore not desirable to add more than 3 wt% of inoculant.

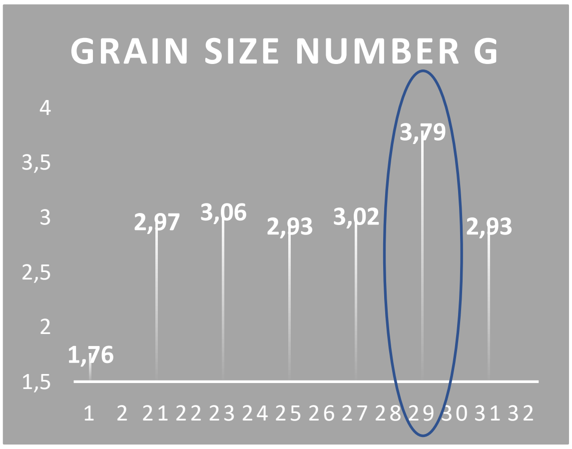

Figure 7 Graphical representation of the Grain size number *G* analysis

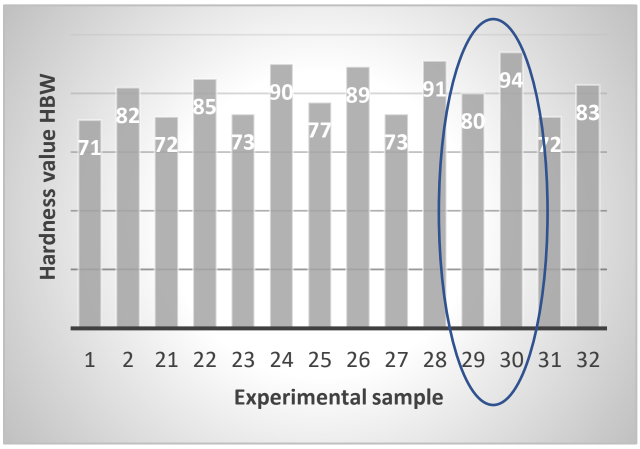

Figure 8 Graphical representation of the hardness measurement result HBW

3.2. Mechanical properties

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 Härteprüfer 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 8** (samples after heat treatment are marked in black). **Table 3** shows the Brinell hardness measurements and the calculated (orientation) value R_m (MPa). The table shows the arithmetic mean of 20 measurements for all experimental samples ($\overline{x}$), standard deviation (σ) and variance of values (σ^2).

Table 3 Hardness measurement HBW

HBW/Sample	1	2	21	22	23	24	25	26	27	28	29	30	31	32
Heat treatment	-	✓	-	✓	-	✓	-	✓	-	✓	-	✓	-	✓
$\bar{x}$	71	82	72	85	73	90	77	89	73	91	80	94	72	83
σ	1.2	7.5	5.6	3.2	9.4	4.8	4.1	8.4	5.0	6.1	7.8	2.5	3.8	5.1
σ^2	1.5	57	31.4	10	88.8	22.6	16.5	71.1	24.6	37.2	60.7	6.4	14.8	26.4
R_m (MPa) (calc.)	184.6	213.7	186.4	220.5	190.6	233.0	199.9	230.1	190.3	236.6	207.7	245.4	188.0	215.8
% AlTi5B1	0	0	0.1	0.1	0.3	0.3	0.5	0.5	1	1	3	3	5	5

The results of the measurements show a favourable effect of the inoculant and heat treatment on the hardness of the AlCu4Mg1 alloy. However, the effect of grain refinement between individual concentrations is not very significant. We achieved the best results at a concentration of 3 wt% AlTi5B1. In essence, there was an increase in hardness after inoculation as a result of grain refinement, as demonstrated by microscopic analysis and grain size evaluation. We achieved the increase in hardness by heat treatment. Defections in variance values and standard deviation are caused by structural inhomogeneity. When evaluating mechanical properties, we must also take into account that the samples contained porosity in the structure due to laboratory casting technology (gravity casting in graphite crucibles. Defects such as pores, inclusions, oxides or equilibrium intermetallic phases generally act to reduce mechanical properties.

4. CONCLUSION

In the present work, the effect of AlTi5B1 grain refiner on microstructure and mechanical properties of AlCu4Mg1 allow was studied. The following conclusions can be drawn based of the experimental results: **a)** the addition of AlTi5B1 master alloy reduced the grain size of AlCu4Mg1 alloy. However, the results were not very significant for individual experimental samples. We achieved the best results with a 3 wt% concentration of AlTi5B1 content in the alloy. Larger quantities have no benefit and at lower quantities the grain size values are almost at the same level; **b)** the heat treatment was evaluated by means of phase analysis and as expected, the intermetallic phases dissolved into a solid solution and the hardness of the alloy increased; **c)** the proportion of fraction was lower in the samples after heat treatment; **d)** due to the favourable results of the image analysis (reduction of grain size, dissolution of intermetallic phases after heat treatment) we can state the favourable effect of the inoculant on the microstructure and mechanical properties of the alloy; **e)** mechanical properties of AlCu4Mg1 alloy were improved by the addition of AlTi5B1 master alloy; **f)** at a content 3 wt% of AlTi3B1 the hardness increased from 71 HBW to 80 HBW and from 82 HBW to 94 HBW after heat treatment; **g)** the results are significantly affected by the sampling site for metallographic sample; **h)** 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 3 wt% AlTi5B1 for specific AlCu4Mg1 alloy processing conditions.

ACKNOWLEDGEMENTS

This article was created due to the project nr. 48101 16 2009 - 4801 under the specific university research on FSI JEPU.

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