

## EFFECT OF AlTi3B1 AND AlTi5B1 INOCULANT ADDITION ON THE AlCu4Mg1 MICROSTRUCTURE OF GRAVITY CAST CASTINGS

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### Abstract

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. The inoculation of hypoeutectic aluminum alloys is carried out with Ti or a combination of Ti and B. These elements are added to the melt with seed salts (eg.  $K_2TiF_6$ ,  $KBF_4$ ), seed tablets or in the form of Al-Ti or Al-Ti-B master alloys. Aluminum-titanium-boron master alloys (in the form of Coil / Stick / Waffle / Piglet / Bar) AlTi5B1, AlTi3B1, AlTi5B0.2 are used for refining grain of pure aluminum or aluminum alloys during melting. The paper focuses on the inoculation of AlCu4Mg1 alloy with two types of AlTi3B1 and AlTi5B1 master alloys. To map the effect of these two inoculants, we added different concentrations of these master alloys to the alloy. Subsequently, we monitored the effect on grain size, intermetallic phase fraction and chemical heterogeneity. The alloy after inoculation with stated master alloys was also heat-treated and the effect of heat treatment was evaluated. Microstructural analysis was performed using an optical and scanning electron microscope (SEM).

**Keywords:** AlCu4Mg1, inoculation, heat treatment, microstructure, segregation

### 1. INTRODUCTION

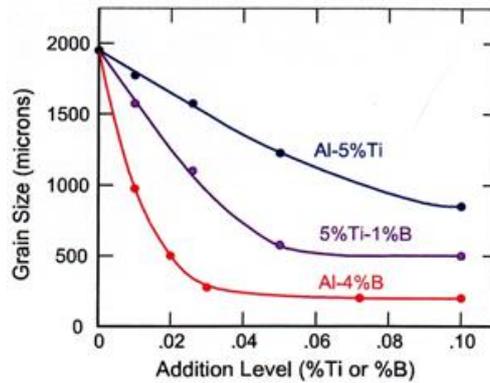
Inoculation of aluminum alloys is essentially the treatment of the liquid metal by the addition of a small amount of a suitably selected substance. This substance affects the crystallization process. Inoculation preferentially affects the number of nuclei and the effect of this process is, therefore, refinement of the final structure of the material/alloy. Al alloys are inoculated to refine the metal matrix, i.e. the solid solution of  $\alpha$  with additives of some transition metals [1,2].

We know of several theories that explain the causes of  $\alpha$ -phase refinement:

- 1) Theory based on peritectic transformation in the aluminum-transition metal system.
- 2) Theory based on the formation of intermetallic aluminum-transition metal compounds (or transition metal carbide).
- 3) Theory based on the electron structure of transition metals (the refining effect of a metal is greater the fewer electrons are in the d-path of its atoms) [2].

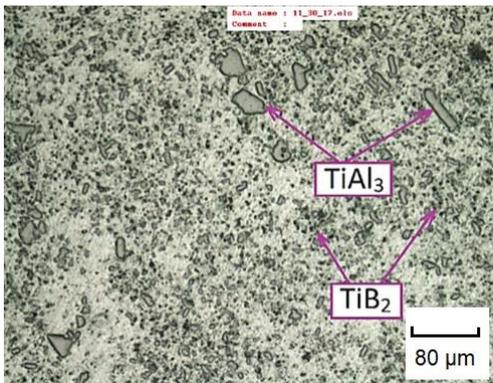
The refinement of the solid solution  $\alpha$  is realized by the addition of Ti and B. These elements are added to the melt either individually or in combination. They are added to the melt in the form of intermetallic compounds, which are contained in the appropriate master alloys (AlTi5B1, AlTi3B1). Titanium is added to the alloy in the form of an AlTi-type master alloy (e.g. AlTi6), which contains the intermetallic compound  $TiAl_3$ . Boron is added to the melt in the form of an AlB type master alloy (e.g. AlB4) and forms the intermetallic compound  $AlB_2$ . The combination of titanium and boron is added to the melt in the form of an AlTiB type master alloy (e.g. AlTi5B1, AlTi3B1, and AlTi5B0.2). This type of master alloy contains active elements Ti and B in the form of  $TiB_2$  and

TiAl<sub>3</sub> intermetallic compounds. The intermetallic compounds AlB<sub>2</sub> and TiAl<sub>3</sub> are soluble in the solid solution  $\alpha$ , while the intermetallic compound TiB<sub>2</sub> is insoluble in the solid solution. This fact leads to the characteristic manifestations of the refinement of a solid solution  $\alpha$  with one of the elements Ti and B separately [4]. **Figure 1** shows the effect of individual master alloys on SDAS (secondary dendrite arm spacing) for AlSi7Mg0.3 alloy [2,5].

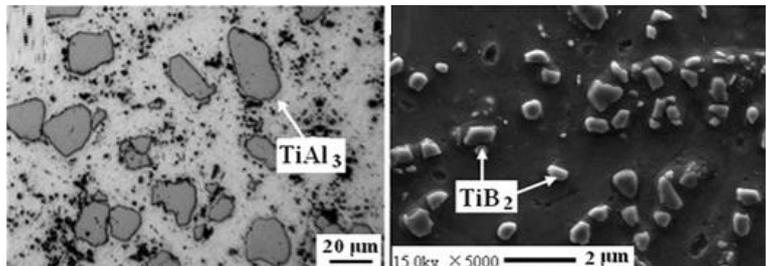


**Figure1** Influence of individual master alloys on SDAS [6]

Inoculation with titanium and boron in combination has a weaker softening effect than boron itself, but a higher softening effect than titanium itself. This combination is commonly used in practice. The grain refinement of the solid solution  $\alpha$  is carried out by the effect of the following phases TiAl<sub>3</sub> and TiB<sub>2</sub>, or (Al, Ti)B<sub>2</sub>, which are introduced into the melt by AlTiB-type master alloys. **Figure 2** shows structure of used AlTi3B1 inoculation wire. **Figure 3** shows structure of AlTi5B1 SEM – TiAl<sub>3</sub> and TiB<sub>2</sub> phases. The structure is very important for a given inoculation wire, as already mentioned in the publication [5]. It very much depends on the size of intermetallic particles, when the size of about 20  $\mu\text{m}$  seems to be the most advantageous. The optimal endurance time for inoculation with AlTiB type alloys is 5-10 minutes [2]. But it can be even more. The author [6] tested different times and achieved satisfactory results even at 30 minutes.



**Figure 2** Structure of the used AlTi5B1 inoculation wire



**Figure 3** Structure of AlTi5B1 SEM - TiAl<sub>3</sub> and TiB<sub>2</sub> phases

Many articles have been published on Ti-B master alloy inoculation [5-8]. However there is no comprehensive description of this issue in any of the specific AlCu4Mg1 alloys using two types of AlTiB inoculants. This paper is focused on the evaluation of grain size, chemical heterogeneity after the addition of two types of inoculants in combination with subsequent heat treatment, their comparison and finding the most suitable combination of processing of this particular alloy from the point of view of microstructure. Analysis and evaluation of the structure of the castings was performed using optical microscopy and SEM to approximate and understand the result of grain refinement when adding inoculum to the alloy.

## 2. MATERIAL AND EXPERIMENTAL SAMPLES

AlCu4Mg1 alloy (EN AW-2024, dural) is used in the hardened state and therefore belongs to the 2000 series Al-Cu alloys. These are the most widely used forming alloys used in the automotive and aerospace industries for components that operate at normal temperatures. It is a light alloy with high strength but low corrosion resistance. It is one of the most important aluminum alloys. The alloy mainly contains the binary eutectic  $\alpha + \text{CuAl}_2$  and a small amount of the ternary eutectic  $\alpha + \text{CuAl}_2 + \text{S}$  ( $\text{Cu}_2\text{Mg}_2\text{Al}_5$ ). In addition to these basic phase components, other phases may occur in the alloy, namely:  $\text{Mg}_2\text{Si}$ ,  $\text{FeAl}_3$ ,  $\text{AlFeMnSi}$ ,  $\text{AlCuFeMn}$ , etc. [1,2].

The aluminum alloy was melted (1000 g) in an induction furnace at 720 °C. The furnace temperature was captured using a digital thermometer with an accuracy of  $\pm 2^\circ\text{C}$ . 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 or AlTi5B1 wire was added to the alloy in concentrations 0; 0.1; 0.5; 1; 3; 5 (wt%). The samples were taken from the melt 30 minutes after the addition and solidified in small graphite crucibles. The heat treatment parameters were set: heating of castings 500°C, holding at a temperature of 360 min and cooling to water, the subsequent ageing took place naturally. 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. Below (**Table 1**) is a chemical composition of AlCu4Mg1 alloy (EN AW-2024).

**Table 1** Chemical composition of AlCu4Mg1 alloy with AlTi3B1 addition (in wt%)

Element	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
0 %	0.048	0.062	5.049	0.476	1.216	<0.002	0.255	0.043	92.73	residue
AlTi3B1 – 0.1 %	0.549	0.065	2.441	0.276	1.890	<0.002	0.258	0.131	94.20	residue
AlTi3B1 – 0.5 %	0.533	0.066	4.589	0.673	1.171	<0.002	0.243	0.154	92.39	residue
AlTi3B1 – 1 %	0.490	0.162	4.873	0.430	1.507	<0.002	0.235	0.163	96,87	residue
AlTi3B1 – 3 %	0.380	0.070	4.870	0.528	1.534	<0.002	0.208	0.238	92.03	residue
AlTi3B1 – 5 %	0.465	0.087	4,951	0.607	1.195	<0.002	0.197	0.351	92.01	residue
AlTi5B1 – 0.1 %	0.482	0.486	5.177	0.507	0.916	0.069	0.045	0.102	92.08	residue
AlTi5B1 – 0.5 %	0.368	0.449	5.224	0.485	0.926	0.070	0.046	0.125	92.17	residue
AlTi5B1 – 1 %	0.341	0.431	4.470	0.479	1.147	0.079	0.031	0.185	92.71	residue
AlTi5B1 – 3 %	0.359	0.429	4.717	0.466	1.039	0.074	0.034	0.288	92.46	residue
AlTi5B1 – 5 %	0.334	0.448	4.637	0.470	1.144	0.077	0.035	0.342	92.32	residue

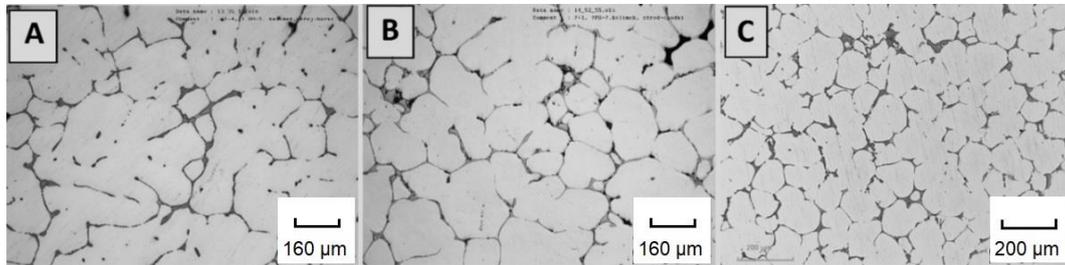
## 3. RESULTS AND DISCUSSION

### 3.1. Optical microscopy

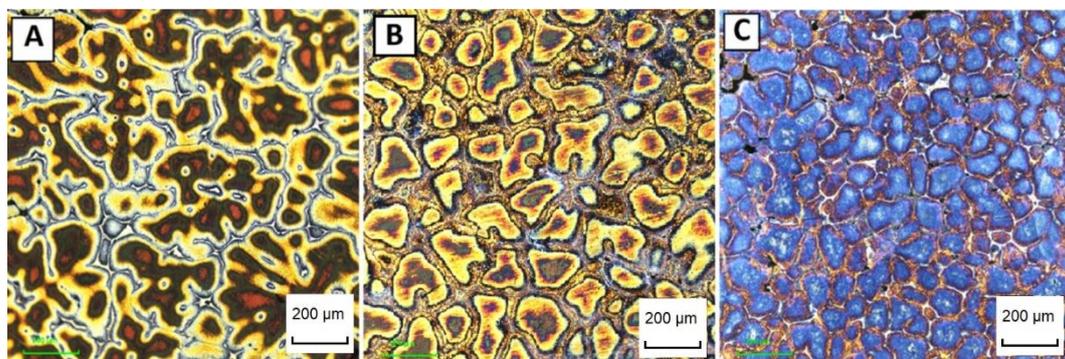
Metallographic experimental samples were prepared from castings by the classical procedure of preparation of metallographic samples (cutting, grinding and polishing). Thus prepared samples were observed and scanned by confocal laser microscope. For the purposes of this paper, only representatives from all experimental samples are listed. The grain structure of the AlCu4Mg1 alloy before and after inoculation is illustrated in **Figure 4**. As part of the microscopic analysis, a structure was observed after colour etching, which will allow the evaluation of chemical heterogeneity after inoculation and heat treatment, **Figure 5**.

**Figure 5** documents experimental samples of B - after inoculation with AlTi3B1 0.5 wt%, C - after inoculation with AlTi5B1 0.5 wt% - colour etched - as an example of chemical heterogeneity (crystal segregation within individual cells) of experimental alloys. Each colour shade in cross section of the dendritic cell represents an inhomogeneity of chemical composition. The images show the heterogeneity of the chemical composition of

individual dendritic cells and the interdendritic space in colour, where in **Figure 5 C** (AlTi5B1 inoculant) the chemical heterogeneity is more uniform than in **Figure 5 B** (AlTi3B1 inoculant). Boron usually occurs near the intermetallic phases, dispersed in solid solution  $\alpha$ .



**Figure 4** Structure of AlCu4Mg1 alloy in cast state, A-without inoculant, B-after inoculation with AlTi3B1 0.5 wt%, C-after inoculation with AlTi5B1 0.5 wt%



**Figure 5** Structure of AlCu4Mg1 alloy in cast state, A-without inoculant, B-after inoculation with AlTi3B1 0.5 wt%, C-after inoculation with AlTi5B1 0.5 wt%- colour etched

### 3.2. Grain size measurement

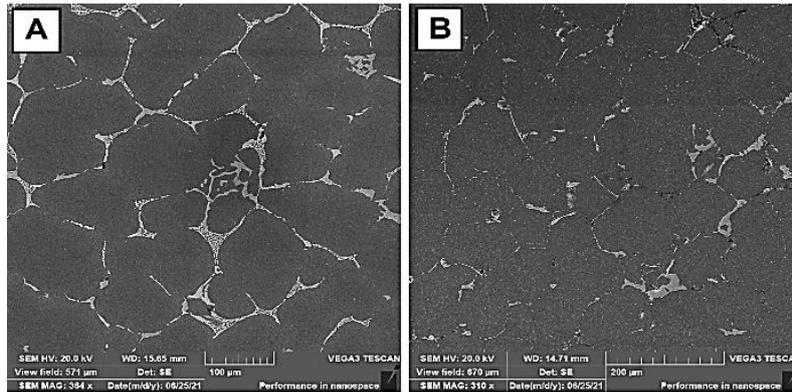
Grain size measurements were performed on metallographic sections by image analysis of Grain Intercept, resp. measurement Grain size number  $G$  according to ASTM. The method is used to measure and document the grain size according to the standard ASTM E 112 Grain Size Measuring Methods. The ASTM E112-13 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. The results of the analysis are shown in **Table 2**.

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

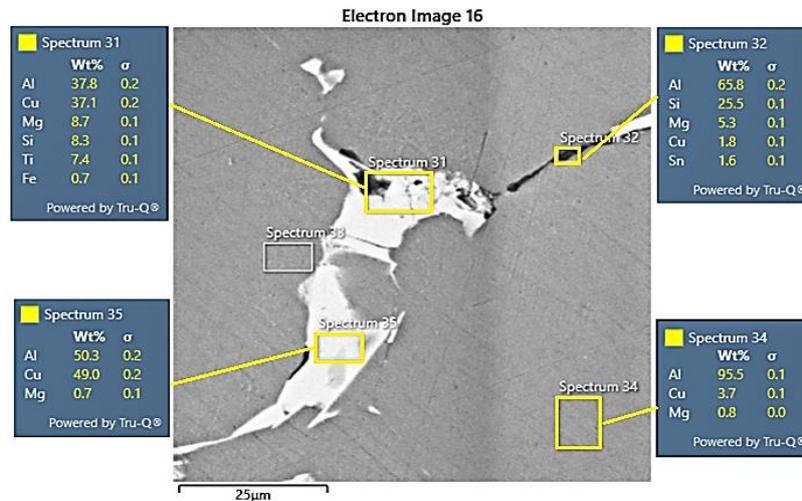
<b>Sample</b>	<b>1</b>	<b>2</b>	<b>21</b>	<b>22</b>	<b>23</b>	<b>24</b>	<b>25</b>	<b>26</b>	<b>27</b>	<b>28</b>	<b>29</b>	<b>30</b>	<b>31</b>	<b>32</b>
AlTi5B1(wt%)	0	0	0.1	0.1	0.3	0.3	0.5	0.5	1	1	3	3	5	5
Fraction (%)	7.01	3.71	8.03	5.40	7.13	4.27	7.76	4.04	7.20	6.10	7.75	4.26	7.53	4.50
Heat treatment	-	✓	-	✓	-	✓	-	✓	-	✓	-	✓	-	✓
Grain size no.	1.76	-	2.97	-	3.06	-	2.93	-	3.02	-	3.79	-	2.93	-
<b>Sample</b>	<b>1</b>	<b>2</b>	<b>3</b>	<b>4</b>	<b>5</b>	<b>6</b>	<b>7</b>	<b>8</b>	<b>9</b>	<b>10</b>	<b>11</b>	<b>12</b>	<b>13</b>	<b>14</b>
AlTi3B1(wt%)	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	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	-	✓	-	✓	-	✓	-	✓	-	✓	-	✓	-	✓
Grain size no.	1.76	-	2.26	-	3.32	-	2.93	-	2.48	-	2.93	-	3.41	-

### 3.3. Evaluation using SEM and EDS (Electron Dispersive Spectroscopy) analysis

To evaluate the effect of heat treatment on chemical heterogeneity, the AlCu4Mg1 alloy was subjected to EDS analysis (point - **Figure 7**), which showed the distribution of aluminum as the base metal and individual alloying elements in a selected area of the AlCu4Mg1 alloy sample before and after heat treatment (**Figure 6**). Sample 26 is presented in this paper (AlCu4Mg1, 0.5 wt% of AlTi5B1, heat-treated). The results of the sample 6 (AlCu4Mg1, 0.3 wt% of AlTi3B1, heat-treated) were published in the article [4].

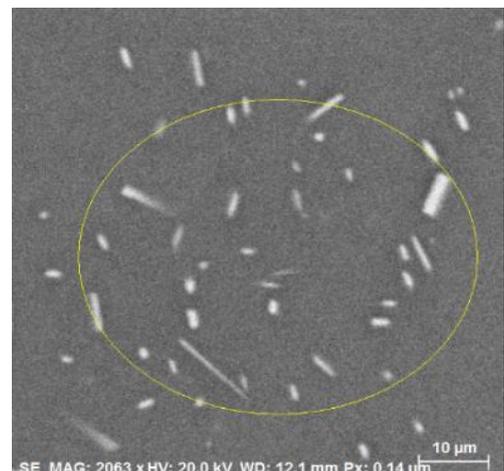


**Figure 6** Structure of AlCu4Mg1 alloy in cast state, AlTi5B1 0.5%, A-without HT (25), B-after HT (26)



**Figure 7** EDS of the alloy AlCu4Mg1, 0.5 wt% of AlTi5B1, heat-treated sample 26

Increased copper concentration is in interdendritic spaces. The Ti, B particles are dispersed in  $\alpha$  solid solution and Ti is also a part of intermetallic phases. The results for the AlTi5B1 inoculation are very similar to those for the AlTi3B1 vaccine, as published in [4]. The evidence of the boron presence shows **Figure 8**. The result of the EDS analysis is shown in the **Table 3**.



**Figure 8** Presence of boron in alloy AlCu4Mg1

**Table 3** Content of elements from EDS analysis in alloy AlCu4Mg1 (see **Figure 8**)

Spectrum:

Element	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (3 Sigma) [wt.%]
Aluminium	K-series	85.66	88.25	80.32	12.36
Copper	K-series	3.04	3.14	1.21	0.47
Boron	K-series	7.57	7.80	17.71	12.58
Titanium	K-series	0.08	0.08	0.04	0.11
Magnesium	K-series	0.50	0.52	0.52	0.18
Silicon	K-series	0.22	0.23	0.20	0.13
Total:		97.07	100.00	100.00	

#### 4. CONCLUSION

The experimental work described in this paper is focused on the microstructure evaluation and identification of chemical heterogeneity in the alloy EN AW-2024 (AlCu4Mg1). The structure of the alloy was influenced using AlTi5B1 and AlTi3B1 inoculant in different wt% amount and heat treatment (homogenization annealing). To investigate the heterogeneity of the structure, the samples were colour etched before and after heat treatment. Both inoculants affected the structure of the alloy (the grain size  $G$ ). Grain size is one of the important factors that affect the properties of the alloy. Grain refinement has certain advantages, such as a homogeneous distribution of the second phases, better supply eliminating shrinkage porosity, better strength and better fatigue life. The smaller grain size thus also affects the mechanical properties, as is generally known. Subsequent heat treatment affects the proportion of fraction, i.e. intermetallic phases in the microstructure. We compared the results for both inoculants in terms of grain size  $G$ , then we notice the difference between the inoculants, but it is not as significant as we expected. For AlTi5B1, sample 29 with 3% inoculant content has the best result (**Table 2**). Higher concentrations are not effective, at lower concentrations the results are comparable. In the case of the AlTi3B1 inoculant, we achieve the best result at a 5% concentration of the inoculant. If we consider all the results, then the AlTi5B1 inoculant appears to be more suitable for this particular type of AlCu4Mg1 alloy. We also used the colour metallography for the evaluation of the microstructure. It is effective method which allows to observe and document intermetallic phases and heterogeneity of chemical composition or segregation of crystals in dendritic cells. Individual colours tones after a cross-section of a dendritic cell documenting inhomogeneity of chemical composition. After heat treatment, it is possible to observe the dissolution of one or more phases in the  $\alpha$  solid solution. Chemical heterogeneity was also partially removed as a result of the heat treatment.

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