

INVESTIGATION OF THE EFFECT OF HOLDING TIME AND MELT STIRRING ON THE GRAIN REFINEMENT OF AN A206 ALLOY

PREISKAVA VPLIVA ČASA ZADRŽEVANJA IN MEŠANJA TALINE NA ZMANJŠANJE VELIKOSTI ZRN ZLITINE A206

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Effects of melt stirring and holding time were studied with an Al-4.5 % of mass fractions of Cu (A206) alloy. The optimum level of a grain refiner was determined in conjunction with both continuously stirred and non-stirred melts during the holding time. Results showed that a mass fraction of a Ti addition of as low as 0.03 % was sufficient to obtain the 82 µm average grain size, while a Ti addition exceeding the mass fraction of 0.03 % showed no significant effect on the grain size of castings. The grain refinement tends to fade with a long holding time in a non-stirred liquid. The results also showed that an effective grain refinement of the A206 alloy can be achieved with a long holding time as long as the liquid alloy is continually stirred.

Keywords: grain refinement, melt stirring, holding time, Al-4.5Cu alloy, A206 alloy

Raziskan je bil vpliv mešanja in časa zadrževanja taline zlitine Al-4,5 % masnega deleža Cu (A206). Določena je bila optimalna količina udrobnjevalca zrn, v povezavi s stalnim mešanjem ali brez mešanja taline med zadrževanjem. Rezultati so pokazali, da je bilo že 0,03 % masnega deleža dodatka Ti, dovolj za doseganje povprečne velikosti zrn 82 µm, medtem ko dodatek Ti večji od 0,03 % masnega deleža, ni pokazal vpliva na velikost zrn ulitkov. Učinkovitost drobnjenja zrn se zmanjša pri dolgih časih zadržanja in brez mešanja taline. Rezultati so pokazali še, da je mogoče doseči učinkovito drobnjenje zrn tudi pri dolgih časih zadrževanja, dokler se talina stalno meša.

Ključne besede: drobnjenje zrn, mešanje taline, čas zadrževanja, zlitina Al-4.5Cu, zlitina A206

1 INTRODUCTION

Al-Cu alloys are one of the most important Al-based alloys because they provide good castability and excellent mechanical properties.¹⁻⁶ Due to their superior mechanical properties, Al-Cu alloys can be used in many areas such as aircraft construction, military field and automobile manufacturing.⁴ Grain refinement of Al-Cu alloys significantly improves the microstructure and mechanical properties.⁷⁻⁹ Along with many advantages, hot tearing, which frequently occurs during solidification due to a long freezing range, is a severe problem in producing cast components with these alloys. Previous works on casting and solidification of Al-Cu alloys consistently indicated that hot tearing can be eliminated with a good grain refinement.^{1-4,7-9} Grain refinement was also found to be effective for reducing the amount of the porosity and size of the pores, and improving the feeding of cast Al-Cu alloys.⁹ Titanium and boron are added in the form of Al-Ti-B master-alloy rods to cast aluminium alloys for the grain refinement. Rod-type additions were found to be more effective for providing, controlling and optimizing TiB₂ particles than the salt form.^{10,11} It was reported in recent years that Al-Ti-C master alloys are

also effective grain refiners.^{4,12} Due to its high refining potential, an Al5Ti1B alloy in the rod form is one of the most commonly used grain refiner and has been widely accepted in controlling the grain size and microstructures of aluminium alloys in industrial applications.¹³

The A206 alloy is a well-known Al-Cu casting alloy and a research on grain refinement with this alloy indicated that a proper grain refinement can be achieved with a 0.15–0.30 % titanium mass fraction of the final cast part.¹⁴ On the contrary, in recent years other researchers^{3,5,7-9} showed that modern grain refiners containing Al-Ti and B are more suitable for an acceptable grain refinement of A206 alloys if the Ti content is lower than 0.15 %.

Despite a number of studies conducted in the past to investigate the grain refinement of Al-Cu alloys,^{3,5-9,12,15-16} studies on the effects of the holding time and melt stirring on the grain-refinement process have not been published. Therefore, in this work, the optimum amount of the grain refiner for the A206 alloy, using an Al5Ti1B rod-shape grain refiner, in combination with the holding time and melt stirring, was studied.

2 EXPERIMENTAL PROCEDURES

A206 alloys with two different chemical compositions were prepared using commercial-purity Al ingots and commercially pure Cu wires in a new SiC crucible. The final chemical compositions of these alloys before the grain-refiner additions are given in **Table 1**. A Spectro-type optical spectrometer was employed to perform a chemical analysis of the alloys throughout this study. An industrial electrical-resistance furnace with a 600 kg capacity and a SiC crucible were used for the melting.

An Al5Ti1B master alloy was introduced into the liquid A206 alloy at 730 °C followed by rotary degassing for 10 min with dry argon. The temperature of the liquid metal in the melting furnace was continuously controlled with a K-type thermocouple connected to the control unit of the furnace to ensure the holding of the liquid at 730 °C.

Grain-refining experiments were carried out using the Alcan standard grain-refining test¹⁰ with two sets of samples. In the first set of experiments, samples were taken from the crucible at 730 °C, before the grain-refiner addition. Samples were also taken after each addition of the Al5Ti1B rod-type master alloy introducing (0.01, 0.02, 0.03, 0.05, 0.1, 0.2, 0.3) % Ti contents into the liquid alloy. After each addition, the liquid alloy was re-degassed for 10 min at 730 °C. This resulted in 10-min intervals between the sample-taking processes.

The second set of experiments was carried out to determine the effects of the holding time coupled with melt stirring on the grain refinement. To implement this, a new heat of the A206 alloy was melted in a new crucible and heated up to 730 °C. The Al5Ti1B master alloy was introduced into the melt to obtain a 0.05 % Ti content within the alloy. After a 10-min degassing treatment, the purging argon was turned off and the molten alloy was continuously stirred using the graphite lance of the rotary degassing unit with a rotating speed of 150 min⁻¹ for 90 min. Samples for the Alcan grain-refining tests were taken during the whole stirring period, in 30-min intervals from the beginning to the end.

Finally, the melt was re-degassed for 10 min and held for another 90 min without any stirring actions. During this second 90-min period, Alcan test samples were also

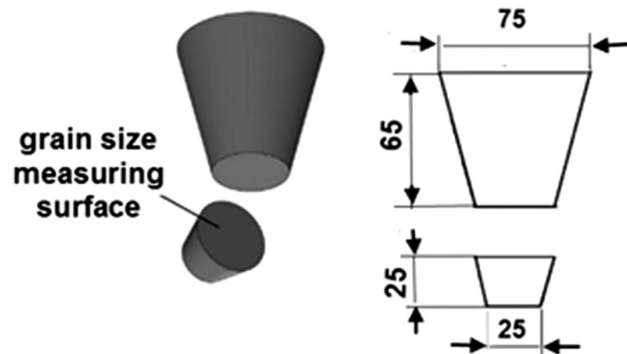


Figure 1: Alcan test dimensions and grain-size measuring surface
Slika 1: Dimenzije Alcan preizkušanca in površina za merjenje velikosti zrn

taken in 30-min intervals, in the same manner as explained above. At the end of this period, the melt was re-stirred for only 1 min and the final sample was taken.

The specimens for the metallographic examinations were cut as shown in **Figure 1**. The surface of each specimen was electro-polished using 5 mL of HClO₄, 15 mL of 2-Butoxyethanol, 60 mL of ethanol and 20 mL of distilled water. The average grain size was determined with the linear intercept method according to the ASTM E112 standard, at different regions of each sample.

3 RESULTS AND DISCUSSION

3.1 Grain-size measurements

The mean values of the measured grain sizes with various Ti contents are shown in **Table 2**, which indicates that the titanium contents in the alloy were determined within a narrow variance. **Table 2** also shows that the grain size of the samples decreased dramatically with the addition of the Al5Ti1B master alloy regardless of the titanium content. The average grain size versus the Ti content is also shown in **Figure 2**. **Figure 2** indicates that the addition of the grain refiner, even with a Ti content as low as 0.01 %, resulted in a remarkable reduction in the grain size of the alloy. **Figure 2** also indicates that the lowest grain size of the alloy is about 80 μm, which was obtained with a 0.03 % Ti content. A higher Ti con-

Table 1: Chemical compositions of A206 alloys before the grain refinement, in mass fractions (w%)

Tabela 1: Kemijska sestava zlitin A206, pred udrobnjenjem zrn, v masnih deležih (w%)

Experiment number	in mass fractions (w%)								
	Si	Fe	Cu	Mn	Mg	Zn	B	Ti	Al
1	0.041	0.105	4.510	0.281	0.214	0.098	0.001	0.000	bal.
2	0.039	0.107	4.610	0.353	0.199	0.084	0.002	0.007	bal.

Table 2: Average grain size of the samples with different Ti contents

Tabela 2: Povprečna velikost zrn pri vzorcih z različno vsebnostjo Ti

Ti (w%)	Target	0	0.01	0.02	0.03	0.05	0.1	0.2	0.3
	Realized	0	0.013	0.019	0.032	0.044	0.098	0.194	0.292
Average grain size (μm)	970	112	92	82	81	79	81	79	

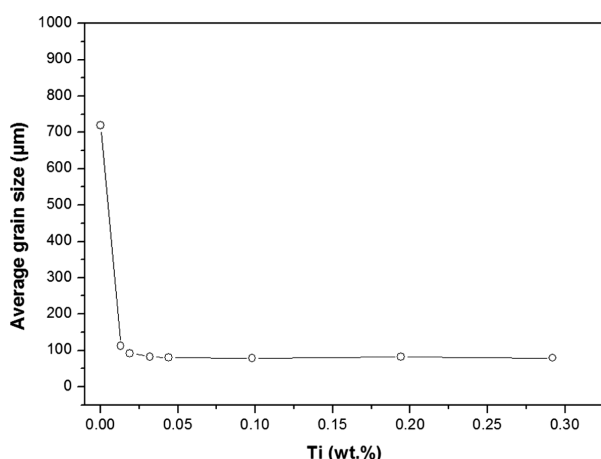


Figure 2: Effect of the Ti content on the grain size of the A206 alloy
Slika 2: Vpliv vsebnosti Ti na velikost zrn zlitine A206

tent within the A206 alloy did not further reduce the grain size of the samples. These results are in good agreement with the previous work carried out by Sigworth and co-workers.^{3,7-8}

The relationship between the titanium recovery and the microstructure is given in **Figure 3**. **Figure 3a** shows the microstructure of the sample obtained with no grain-refiner addition. The microstructure consists of coarse dendrites, heterogeneously distributed in equiaxed grains. **Figure 3b** shows a small addition of Ti, as low as a 0.01 % mass fraction, which caused the grain refinement of the A206 alloy. However, a Ti content of up to 0.05 % was found to be more effective for further reducing the average grain size of the alloy, as seen in **Figure 3c**. The microstructure obtained with a 0.3 % mass fraction of Ti in the melt is shown in **Figure 3d**. It indicates that the increased amount of Ti no longer affected the reduction of the grain size of the A206 alloy used in this study.

The Al5Ti1B master alloy is an effective grain refiner of Al-Cu alloys as it increases the number of heterogeneous nucleation sites for achieving a finer equiaxed grain structure.¹⁰ **Figure 3** clearly shows that a Ti content exceeding the mass fraction of 0.01 % is adequate to turn

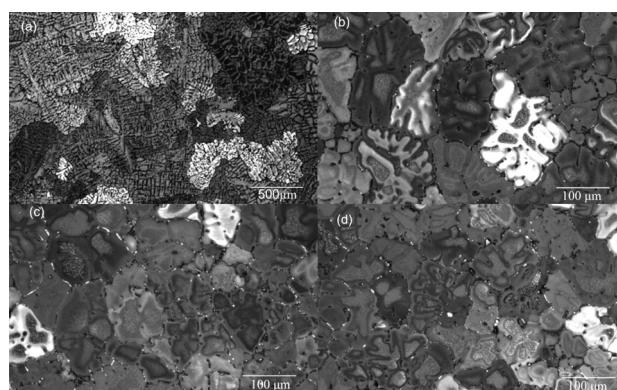


Figure 3: Microstructure of the A206 alloy with different Ti additions: a) 0 % of mass fractions, b) 0.01 % of mass fractions, c) 0.05 % of mass fractions and d) 0.3 % of mass fractions

Slika 3: Mikrostruktura zlitine A206 z različnimi dodatki Ti: a) 0 % masnega deleža, b) 0,01 % masnega deleža, c) 0,05 % masnega deleža in d) 0,3 % masnega deleža

the solidification morphology of the alloy from a fully dendritic to a globular or near-dendritic structure. Thus, the grain refinement of the A206 alloy by adding the Al5Ti1B master alloy can be expected to increase the mechanical properties of A206 castings via reducing the amount of solidification defects such as hot tearing, micro-shrinkage and micro-segregation. These results are in good agreement with the report from H. Kamali et al.⁵ who reported that Ti additions of 0.05–0.3 % showed no significant effect on the grain size, although the minimum Ti mass fractions of 0.05 % was necessary to eliminate the hot-tearing defects.

3.2 Effects of the holding time and melt stirring on the grain size

In the present study, possible effects of melt stirring during the holding period after the master-alloy addition were also investigated. Experiments were carried out with the castings from two different heats of stirred and non-stirred melts. Samples were cast in 30-min intervals throughout the 90-min holding time, during which the amount of Ti was fixed at around 0.05 % of mass fractions.

Table 3: Effect of the holding time on the average grain size

Tabela 3: Vpliv časa zadrževanja na povprečno velikost zrn

Liquid-metal condition	Holding time (min)	Ti recovery (w%)	B recovery (w%)	Ti:B ratio	Average grain size (µm)
Heat 1 Stirred melt	0	0.049	0.009	5.4	84
	30	0.046	0.008	5.8	84
	60	0.047	0.008	5.9	85
	90	0.048	0.009	5.3	84
Heat 2 Non-stirred melt	0	0.046	0.009	5.1	84
	30	0.036	0.005	7.2	88
	60	0.035	0.004	8.8	102
	90	0.031	0.003	10.3	111
	90*	0.048	0.009	5.3	85

(90*) Stirring after 90 min.

A summary of the quantitative results obtained from the test samples throughout these experiments are shown in **Table 3**. Titanium and boron measured in the samples, cast with the stirred melt, show that both elements remained at almost their initial values during the 90-min holding time. Accordingly, the initial Ti:B ratio also remained constant, which was around 5:5. The average grain size of the stirred melt was about 85 μm regardless of the holding time. These results emphasise the fact that the refining efficiency of the Al5Ti1B type master alloy is consisted, at least during the 90-min holding time, even with the Ti recovery as small as 0.05 % of mass fractions of Ti in the A206 alloy. On the other hand, the results obtained for the non-stirred melt reveal that both titanium and boron recovery decreased with the increasing holding time. Interestingly, the diminution in boron was faster compared to titanium, especially in the first 30 min of the holding time, for the non-stirred melt. Therefore, the measured Ti:B ratio shows a continuous increase as the period of the holding time increases.

In **Table 3**, the average measured grain size for the samples of the non-stirred melt also shows a consisted increase with the holding time. This is associated with the diminishing of the Ti and B recovery and the ever increasing Ti:B ratio with the increasing holding time for the non-stirred heat. It is also obvious from **Table 3** that

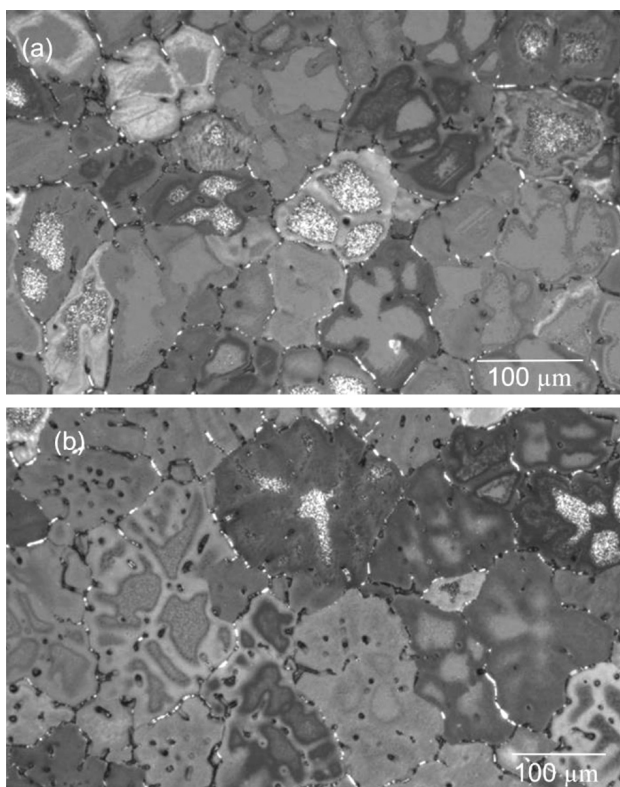


Figure 4: Microstructures of Alcan test samples after a 90 min holding time and an Al5Ti1B master-alloy addition. Samples were cast from: a) continuously stirred melt and b) non-stirred melt.

Slika 4: Mikrostruktura Alcan preizkušancev po 90 min zadrževanju po dodatku predzlitine Al5Ti1B. Vzorci so bili uliti iz: a) kontinuirno mešane taline in b) taline brez mešanja.

an immediate stirring action followed by a 90-min holding time facilitated the Ti and B levels as well as the Ti:B ratio to remain almost at their initial values. This also caused a similar consequence in the measured grain size. These results are in good agreement with a number of previous studies on Al-Cu alloys. Grain-refining studies on non-stirred melts were concluded so that the grain size increased continuously with the increasing holding time;^{11–12,16} however, it began to decrease when the stirring action was resumed.¹⁶

The phenomenon of decreasing Ti and B with the increasing holding time in the non-stirred A206 alloy can be attributed to the settling of TiB₂. This compound has been widely accepted as one of the potential nucleation sites during the solidification of aluminium.^{19–22} Since the density of a solid TiB₂ compound is higher (4.48 g/cm³)¹⁷ than that of the liquid A206 alloy (2.78 g/cm³), it is quite probable that some potential TiB₂ nuclei are disqualified because they sink to the bottom of the crucible as the holding time increases.

The microstructures representing the Alcan test samples obtained from the heats after the 90-min holding time are shown in **Figures 4a** and **4b**. A comparison of the two microstructures provides an explanation of the difference between the stirred and the non-stirred melts. The larger grain size for the non-stirred melt in **Figure 4b** can be associated with a weaker grain-refining action of the master alloy during the 90-min holding. This can also be related to the fading of the potential nuclei probably due to the gravity action of the TiB₂ compound.¹⁸

4 CONCLUSIONS

The grain-refining effects of the Al5Ti1B rod-type master alloy on a commercial A206 alloy with different addition levels were studied. The effects of the holding time under stirring and no-stirring conditions were also studied. From the experimental results, the following conclusions can be drawn:

- 1) Different amounts of titanium recovery ranging from 0.01 to 0.3 were formed in the A206 alloys. Measurements showed that the grain size of the Alcan test samples decreased dramatically with an addition of the Al5Ti1B master alloy regardless of the titanium recovery.
- 2) The smallest average grain size of the A206 alloy was 82 μm , achieved with a 0.03 % of mass fraction of Ti recovery in the melt. Increasing the Ti recovery up to a 0.3 % of mass fractions did not result in a further decrease in the grain size of the A206 alloy.
- 3) Grain-size measurements for two different heats showed that the average initial grain size of the samples increased from 84 μm to 111 μm at the end of the 90-min holding time for the non-stirred liquid. However, a constant grain size was achieved for the stirred melt throughout the holding period.
- 4) The results obtained for the non-stirred melt revealed that both titanium and boron recovery decreased with

the increasing holding time. During the holding period, the diminution in boron was larger compared to titanium, as measured on the cast samples. This was attributed to the formation of a TiB_2 compound in the melt, which is widely accepted as a heterogeneous nucleation site for aluminium. Since the density of TiB_2 is higher than that of the liquid A206 alloy, the compound tends to settle in the non-stirred melt, which may be the reason for the fading of the grain-refining efficiency as the holding time increases.

- 5) The refining efficiency of the Al5Ti1B-type master alloy can be stimulated via re-stirring the melt. The results showed that an immediate stirring action followed by a 90-min holding time reverted Ti and B and also the Ti:B ratio almost to their initial conditions. The Alcan test samples of the re-stirred melt also showed a well-refined grain structure.

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