The examination of the grain refining effects of titanium in case of AlSi7MgCu0.5 alloy

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Abstract. Due to the rapidly developing technologies and the various expectations of the customers, the continuous development of the products is necessary. In case of automotive industry, the increasingly more demanding expectations and requirements pose great challenge for the casting industry. One of the most significant requirements is the production of lightweight but high strength castings. Thus, aluminium became one of the most widely used base material for the automotive industry. However, to maintain a lasting presence on the market, continuous development and the improvement of the mechanical properties of the castings is necessary. During the experiments, the effect of grain refinement with AlTi5B1 master alloy on the mechanical properties, porosity, secondary dendrite arm spacings (SDAS) and grain size of AlSi7MgCu0.5 alloy was examined. The initial 1000 ppm titanium concentration was increased to 2000 ppm, with the addition of 200 ppm at each step.

1. Introduction

The crystallization of the Al-Si hypoeutectic alloy in the mould starts with the formation and growth of the nuclei of the aluminium solid solution. The nuclei continuously grow until reaching the eutectic temperature. The solidification ends with the crystallisation of the eutectic structure. The mechanical properties are highly affected by the number and the fineness of the grains in the solid solution: the finer the granular structure is, the better the mechanical properties are. Grain size can be decreased by accelerating the nucleation in the crystallisation process. Thus, the structure can be composed of more and smaller crystals. To achieve such structure, the so-called inoculation, grain refinement is applied [1].

Apart from wrought alloys, grain refinement has several benefits in cast alloys like improved mechanical properties that are uniform throughout the casting, distribution of second phase and microporosity on a fine scale, better feeding to eliminate shrinkage porosity, improved ability to achieve a uniform anodized surface, better strength and fatigue life. [2, 3]

The size and distribution of grains and their secondary dendrite arm spacing (SDAS), which is highly influenced by the cooling rate, are parameters to be tightly controlled [4]. The grain size of aluminium castings is usually between 0.1 and 10 mm. Castings have fine structure if the average diameter of the grains are less than 1 mm. Due to overheating, long holding time or slow solidification rate, the structure of aluminium alloys become coarse. Therefore, the mechanical properties are deteriorated. The aim of grain refinement is to eliminate the above-mentioned issues by adding an additive to the molten metal to increase the number of nuclei [5]. Elements which react with aluminium in a peritectic reaction during solidification are the most sufficient grain refiners. Thus, titanium is one of the most commonly used grain refiner. Grain refinement should always occur after degassing and cleaning (slag removal) on 730-750 °C, depending on the composition of the alloy [5].
2. Experimental part

The examined AlSi7MgCu0.5 foundry alloy melt was alloyed with AlTi5B1 master alloy under operating conditions to achieve finer grain structure and better mechanical properties. The master alloy was added to the melt right before degassing the melt with N₂. Portions of 1000 kg melts were alloyed in the holding furnace during the experiment. The measured density index values of the degassed melt were always below 2.0%, according to specification. The casting temperature of the melt was 733±3 °C. The holding time – the time until the casting of the test bars – was ~40 minutes.

The changes in the composition of the melt was continuously monitored by the composition analysis of the chill tests. Based on the results, the titanium master alloy was added to the melt. The sought and achieved Ti concentrations can be found in Table 1, while Table 2 contains information on the average composition of the examined alloy.

| Portion | Sought Ti concentration | Achieved Ti concentration |
|---------|-------------------------|---------------------------|
| Base material | 1200 ppm= 0.12 m/m% | 1227 ppm |
| 1. alloying | 1400 ppm= 0.14 m/m% | 1453 ppm |
| 2. alloying | 1600 ppm= 0.16 m/m% | 1623 ppm |
| 3. alloying | 1800 ppm= 0.18 m/m% | 1879 ppm |
| 4. alloying | 2000 ppm= 0.20 m/m% | 1970 ppm |

Table 2. The average composition of the examined alloy

| Elements | Al | Si | Ti | B | Fe | Cu | Mn | Mg | Zn | Sr |
|----------|----|----|----|---|----|----|----|----|----|----|
| m/m%     | 91.80 | 6.95 | 0.10 | 0.00026 | 0.11 | 0.53 | 0.06 | 0.37 | 0.01 | 0.0238 |

In order to draw conclusions from the cooling curves of the melts about the degree if grain refinement, thermal analyses were carried out. Microstructure – grain number and secondary dendrite arm spacing – and strength tests – tensile tests, Brinell hardness test – were also carried out during the experiments. The tests were carried out at the specific sampling points of the test bars determined by standard.

2.1. Thermal analysis

The development of primary crystalline phase is accelerated by the addition of nucleating agents to homogenous melt. Thus, more grains and finer structure can be achieved. In case of the examined melts, the maximum temperature of the liquidus must increase, as less time is needed for the critical nucleus size, and consequently to start crystallisation, because of the addition of the proper grain refiner.

Based on the cooling curves of the degassed melts prepared under operating conditions, the maximum liquidus temperatures increased with the increased titanium concentration, as it can be observed in Figure 1. Because of data loosing we could not see the whole curve in case of 1400 ppm.
Figure 1. The liquidus temperature intervals of the cooling curves defined during thermal analysis

However, a decline can be observed in the cooling curve for the 2000 ppm titanium concentration, so the microstructure analyses of the test bars were also carried out. The results are discussed in detail in Chapter 2.2.

2.2. Microstructure analysis

The investigated areas of the experimental castings and thermal analysis samples can be seen in Figure 2.

Figure 2. The investigated areas of the experimental castings (1, 2) and the thermal analysis sample (3)

2.2.1. Determination of grain numbers with colour etching

Barker [8] etching was carried out to examine the grain structure with a Struers LectroPol-5 instrument. The samples were examined with an optical microscope (M=25x). The approximate number of particles were determined on the surface of the samples with the help of their different colours. Five microstructure images of each sample have been made and calculations carried out based on them.

The average grain numbers determined at the 1st and 2nd sampling points can be seen in Figure 3.
The grain refining ability of titanium is confirmed by the number of grains, as the grain number increased throughout the experiments. The average grain numbers at the 1\textsuperscript{st} and 2\textsuperscript{nd} sampling points can be seen in Table 3., including the characteristic images of the Barker-etched microstructures as well.

**Table 3.** The number of grains at the 1\textsuperscript{st} and 2\textsuperscript{nd} sampling points of the test bars (magnification 25x)

| Samples          | Ti concentration |
|------------------|------------------|
|                  | 1000ppm | 1200ppm | 1400ppm | 1600ppm | 1800ppm | 2000ppm |
| 1. sampling point|          |          |         |         |         |         |
| Average grain numbers | 25,70  | 25,70   | 33,30   | 30,30   | 38,30   | 52,00   |
| 2. sampling point |          |          |         |         |         |         |
| Average grain numbers | 21,00  | 18,70   | 32,00   | 29,30   | 41,70   | 42,00   |
| Casting temperature (°C) | 730  | 732     | 736     | 734     | 734     | 735     |

As it can be observed in Table 4, the increased titanium concentration resulted in finer grain structure. The average grain number also increased with the titanium addition. Similar tendencies could be observed in case of both sampling points and the test bars for thermal analysis (Figure 4.). Unfortunately because of an industrial problem we could not take samples in case of 1000 ppm casting.
Figure 4. Average grain numbers of the thermal analysis test bars

The average grain numbers at the thermal test bars sampling points can be seen in Table 4, including the characteristic images of the Barker-etched microstructures as well.

Table 4. The number of grains at the sampling points of the thermal test bars (magnification 25x)

| Samples          | Ti concentration |
|------------------|------------------|
|                  | 1200ppm | 1400ppm | 1600ppm | 1800ppm | 2000ppm |
| 2. sampling point|          |         |         |         |         |
| Average grain numbers | 19,70   | 30,30   | 34,00   | 45,70   | 60,30   |
| Casting temperature (°C) | 732     | 736     | 734     | 734     | 735     |

2.2.2. Determination of secondary dendrite arm spacing

The average secondary dendrite arm spacings (SDAS) were determined at the 1st and 2nd sampling points of the test bars. For the measurement of SDAS, first, 10 structural images per cross sections were prepared with the help of an optical microscope at 100x magnification. Three SDAS values were measured in each image and the results were averaged. The measured average secondary dendrite arm spacings are summarised in Figure 5. As it can be seen in Figure 5, the secondary dendrite arm spacings decreased in the experimental castings with the increased amount of grain refiner addition.
2.3. Mechanical properties

Mechanical tests were carried out on test bars prepared from the castings (3 castings /experiment) to examine if further titanium addition improves the mechanical properties. The mechanical tests were carried out after the T5 heat treatment of the castings. 3 castings were examined in case of each experiment, Brinell hardness was measured at 3 points and 3 tensile test specimens were used for tensile tests. The Brinell hardness tests were carried out with a 5-mm ball and 250 kp – 2451,66 N load. The test bars were cylindrical, 5 mm in diameter and 50 mm long.

Figure 6. shows the average Brinell hardness values. The Brinell hardness values were between 70 – 90 HB during alloying. The hardness was slightly improved by the increased titanium concentration, mainly in the middle range.
During tensile tests, the tensile strength, elongation and yield point of the test bars were examined. It can be seen in Figure 7 that the tensile strength values followed a maximum curve as the titanium concentration was increased. Above 1200 ppm titanium concentration, the tensile strength values decreased.

![Figure 7](image)

**Figure 7.** The changes in the average tensile strength in relation to the titanium concentration

The strength of the test bars can be improved by decreasing the secondary dendrite arm spacings, which was also confirmed by the experiments. It can be observed in Figure 8 that higher tensile strength values can be achieved than the average values of castings with 1000 ppm titanium concentration by decreasing the SDAS with alloying.

![Figure 8](image)

**Figure 8.** The changes in the average tensile strength and SDAS during alloying

The values of relative elongation were significantly higher than the required 1.5% *(Figure 9).*
Figure 9. The average relative elongation in relation to titanium concentration

Conclusion
1. The increased titanium addition to the melt lead to higher liquidus maximum temperature, confirming that lower supercooling temperature is necessary for the crystallisation process to start in case of increased titanium concentration.
2. The number of grains increased and the secondary dendrite arm spacings decreased in both the experimental castings and the thermal analysis test bars when the titanium concentration was increased.
3. The strength tests revealed that the required mechanical properties can also be achieved with higher titanium concentrations. Based on the experiments, it can be concluded that the optimal titanium concentration is between 1000 and 1400 ppm. For the determination of a more precise concentration further experiments are needed.

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