

# Grain refinement of 7075Al alloy microstructures by inoculation with Al-Ti-B master alloy

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**Abstract.** This paper aims to bring some clarification on grain refinement and modification of high strength alloys used in aerospace technique. In this work it was taken into account 7075 Al alloy, and the melt treatment was carried out by placing in the form of master alloy wire ternary AlTiB the casting trough at 730°C. The morphology of the resulting microstructures was characterized by optical microscopy. Micrographs unfinished and finished with pre-alloy containing ternary Al5Ti1B evidence fine crystals, crystal containing no columnar structure and highlights the size of the dendrites, and intermetallic phases occurring at grain boundaries in Al-Zn-Mg -Cu alloy. It has been found that these intermetallic compounds are MgZn<sub>2</sub> type. AlTiB master alloys finishing ensures a fine eutectic structure, which determines the properties of hardware and improving the mechanical properties of aluminum alloys used in aeronautical engineering.

## 1. Introduction

One of the most common aluminum alloys used for structural applications is alloy 7075 T6 from Al-Zn-Mg alloys series due to its attractive properties, such as low density, high resistance ductility, toughness and fatigue resistance [1-4]. This alloy is extensively used in aircraft structural parts and other structural applications highly demanded from mechanical point of view and corrosion.

The structure of the grain is an important feature and easily observable in casting high-strength aluminum alloys. Refining is one of the prevalent techniques in strength and hardness control of processing by hot plastic deformation. Refining and finishing grains by adding ternary master alloys such as Al-Ti-B, mentioned as inoculants is the best way due to its simplicity [5], [6].

The specialized literature indicates a number of potential finishers grain for aluminum alloys, but some of them have a limited industrial application [7], [8].

Spittle [9] and others have studied a number of finishers and they have shown that at Ti /B ratios close to the stoichiometric ratio, a lower finishing is achieved than when using an excess of Ti or B. The industrial practice suggests the addition of 0,1% excess Ti to obtain good performance for stoichiometric finishers [10], [11].

Easton and John have tested the effect of increasing titanium content in the maintenance of the content of TiB<sub>2</sub>, finding a negligible change of the grain size [12].

In recent years, the finisher products market has increased by the special use of TiB Alloy and Strobloy. Using Alloy TiB for hypoeutectic alloys used in foundries allowed carrying out studies



referring to porosity and lack sedimentation of the borides and they are available as coiled wire, rod shaped cut and tablets.

Grain finishers, added to liquid alloys of aluminum may provide nucleation and favor the production of fine grain and uniform material [13]. Research on new generations of master alloys finishers / modifiers are still young and require a great deal of experimentation to establish the optimal pre-alloy complex and for studying the kinetics and the mechanism of the amendment process and finishing, practically running concurrently. There is also needed clarification on the role of alloying elements and impurities on the effect of titanium and boron introduced to melt together in the form of pre-alloy complex. In this context, the present work aims to bring some clarification on finishing/changing of medium and high strength alloys used in aeronautical engineering.

## 2. Experimental procedure

### 2.1. Materials

In this work it was considered aluminum alloy 7075 and the melt treatment was carried out by placing in the form of ternary alloy wire AlTiB directly to the pouring spout at 730 °C. Microstructure morphology results were characterized by optical microscopy. In this work it was considered 7075 aluminum alloy according to the American Association of Al classification, the samples being taken from UAC Europe SRL (Dumbravita). This alloy is commonly used in the aeronautical industry for the high strength structural components (fuselage, wings). The alloy composition is shown in Table 1 [14].

**Table 1.** The chemical composition of the 7075 alloy (% wt.)

Element	Al	Mg	Si	Mn	Cu	Zn	Fe	Cr	Ti	Others
% wt.	difference	2.1-2.9	0.40	0.30	1.2-2	5.1-6.1	0.50	0.18	0.20	0.5

The treatment of Al 7075 melt alloy was achieved by introducing AlTi5B1 ternary master alloy wire on the pouring spout at 730 °C. The parameters of the grain finishing process in Al-Zn-Mg alloys system are shown in Table 2.

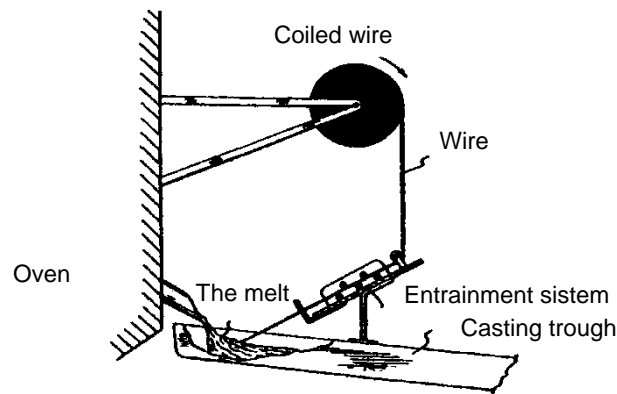
**Table 2.** Process parameters for finishing Al 7075 alloy with pre-alloy AlTiB [13]

Phase	Process parameters	
	Temperature, °C	Time, min.
Alloying with pre-alloy AlTiB	730	10
The degassing with argon (55 l/min.)	730	12
Ternary pre-alloy AlTi <sub>5</sub> B1 (2,8 kg/t)	5% Ti	1% B

By treating the metal melt with AlTi5B1 pre-alloy occurs:

- Improvement of the mechanical properties and refractoriness;
- Improvement of the casting properties (high fluidity).
- Shrinkage porosity reduction and increased density;
- Easier achievement of the uniformity response to heat treatment;
- Improved extrudability and processability by chip cutting, surface finishing due to the fine structure.

In principle, the master alloy is (Figure 1) as a wire passing through a feeder or drive system, a guide tube for casting gutter where it is injected.



**Figure 1.** The scheme of finishing with titanium as Ti-B master alloys of Al-Zn-Mg alloys

In Figure 2 it is sequentially shown the practice of supply and injecting process on the pouring spout at the UAC Europe SRL Company.



**Figure 2.** Supply system of the Al-Ti-B master alloy (Source: UAC Europe SRL)

AlTi<sub>5</sub>B1 finisher as wire is more efficient than the tablets as they have higher dissolution speed in the melt, leading to an effective grain finish at solidification, within 30 s after the addition. It is available as coiled wire of 9.5 mm (3/8 in) diameter in standard coils of 180 kg per pallet packaged in three coils protected from moisture. For the addition of AlTi<sub>5</sub>B1 pre-alloy finisher, the titanium content recommended and the added amount are listed in Table 3.

**Table 3.** The content of titanium and the added amount recommended [12]

Zn	% mass	5	5.2	5.4	5.6	6
Ti	% mass	0,05	0,04	0,03	0,02	0,01
AlTi <sub>5</sub> B1	g/100 kg	1000	800	600	400	200

If cast at high speed cooling (with thin sections or pressure casting), a smaller amount of finisher is added. In some cases, it is only necessary to prevent the formation of large grains while in other cases it is necessary to form a fine structure to optimize the properties of molded products. In some alloys there are residual titanium content from recycled materials. If the percentage of titanium exceeds 0.1%, benefits could be easily achieved by adding pre-alloy TiBAl.

It is necessary to avoid excessive addition to prevent the formation of agglomerates borides and / or aluminides which may lead to their sedimentation in long maintenance issues and problems in chip cutting processing. For titanium addition to the content above 0.1% is recommended inoculation of Al-Ti master alloys and products 100% ALTABTM.

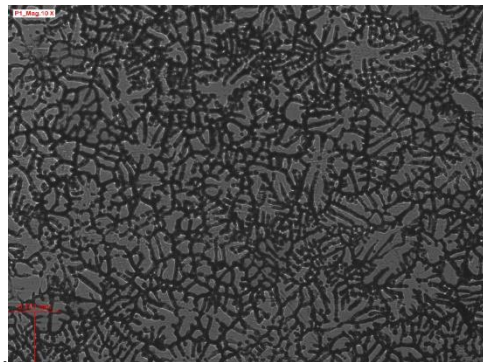
## 2.2. Optical metallography

The castings samples for chemical and microscopic analysis, shown in Figure 3 were made and taken by the metallographic process: the selection of samples before and after finishing with Al-Ti-B, grinding, polishing and etching. Microscopy samples were cleaned with grit sandpaper (400-1200) and polished with paste (10, 7, 5, 3.5, 2.5) followed by etching with a Keller freshly prepared solution (1.0 ml HF, 1.5 ml HCl, 2.5 ml HNO<sub>3</sub> and 95.0 ml H<sub>2</sub>O).



**Figure 3.** The collected samples for chemical and microscopic analysis

The morphology of the microstructures before and after finishing for two magnification shown in Figures 4-5 were characterized by optical microscopy.

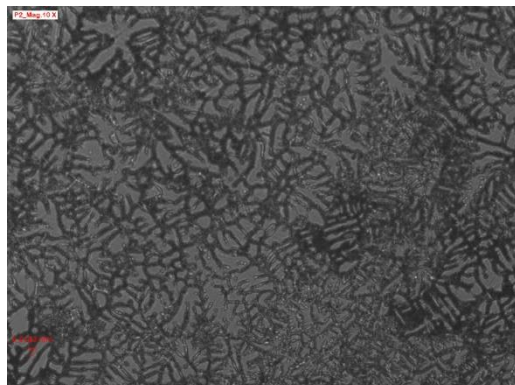


**(a)**

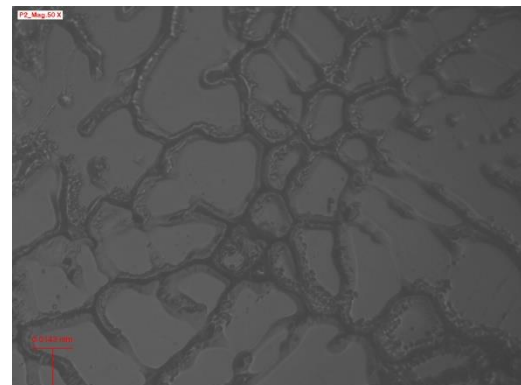


**(b)**

**Figure 4.** Optical micrographs: a) before treatment, AlTi<sub>5</sub>B1 finisher; b) after treatment (100x)



**(a)**



**(b)**

**Figure 5.** Optical micrographs: a) before treatment, AlTi<sub>5</sub>B1 finisher; b) after treatment (500x)

The Micrographs of the samples obtained by optical microscopy (Leica DM2500 microscope) unfinished and finished with AlTi<sub>5</sub>B1 ternary pre-alloy contain fine crystals, the structure containing no columnar crystals and highlights the size of the dendrites and intermetallic phases occurring at grain boundaries in the alloy Al-Zn-Mg-Cu. It has been found that these intermetallic compounds are MgZn<sub>2</sub> type. These compounds may form during solidification below the solidus as cast aluminum alloy 7xxx series due to the metals redistributing (Mg, Zn) in solidification [15].

In the case of cast molding, at high cooling speed (with thin sections or die casting), a small amount of the finisher is added. In some cases, it is only necessary to prevent the formation of large grains while in other cases it is necessary to form a fine structure to optimize the properties of molded products.

In the practice of aluminum alloys grain finishing there are a series of issues such as: decreasing degree of the finishing time due to the particles agglomeration and sedimentation, the "poisoning" phenomenon (eg. due to the zirconium presence, dissolved in the melt) and the performance of the operation [16]. The borides sedimentation appears in unstirred melts due to the high density of TiB<sub>2</sub> (4.5 g / cm<sup>3</sup>) compared to the density of the liquid aluminum alloy (~ 2.3 g / cm<sup>3</sup>).

The borides agglomeration reduces the performance of the grain finishing by agglomerates fast decanting. Partial reactivation can be achieved by rapid stirring of the melt, which, however, it is sometimes is difficult to achieve. Also, the borides agglomeration can block the ceramic filters.

The "poisoning" of the grain finisher is a very serious problem because it can be produce very quick and generally it is irreversible. "Poisoning" most often caused by Zr is considered as a result of the Ti substitution from TiAl<sub>3</sub> or TiB<sub>2</sub> structure with the formation phase (Zr,Ti)Al<sub>3</sub> or (Zr,Ti)B<sub>2</sub> having unfavorable network parameters for nucleating.

### 3. Conclusions

- ✓ Finishing is a treatment method of the melts before casting, which ensures a fine eutectic structure, which leads to an improvement of molding properties and the physical-mechanical properties of aluminum alloys used in aeronautical engineering.
- ✓ In recent years there has been a tendency of modifying and finishing the high strength aluminum alloys, in a single step, by treating the melt with complex master alloys containing particles of grain finishers (TiAl<sub>3</sub> and TiB<sub>2</sub>).
- ✓ In this paper, a type of pre-alloy ternary system Al-Ti-B has been characterized, with reduced Ti and B, as well as treating Al-Zn-Mg liquid alloys (alloy high strength Al 7075) in bringing together the grain finishing and modifying operations to improve the mechanical characteristics and the cost of the molded products.
- ✓ The treatment in a liquid state of 7075 alloy with AlTi<sub>5</sub>B1pre-alloy and its characterization with optical microscopy, using Leica DM2500 optical microscope, revealed the unfinished samples microstructure and the one finished with pre-alloy ternary AlTi<sub>5</sub>B1, that contains fine crystals, the structure containing no columnar grain and highlighting the size of the dendrites and intermetallic phases that occur in grain boundaries in the of Al-Zn-Mg-Cu alloy. It has been found that these intermetallic compounds are MgZn<sub>2</sub> type. These compounds may form during solidification below the solidus as cast aluminum alloy 7xxx series, due to metals (Mg, Zn) redistributing in solidification.

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