Journal of Achievements in Materials and Manufacturing Engineering
VOLUME 50 ISSUE 2 February 2012

The influence of inoculants sort on pure Al structure

T. Wróbel*, J. Szajnar
Foundry Department, Silesian University of Technology,
ul. Towarowa 7, 44-100 Gliwice, Poland
* Corresponding e-mail address: tomasz.wrobel@polsl.pl

Received 12.12.2011; published in revised form 01.02.2012

Manufacturing and processing

ABSTRACT

Purpose: In paper the problem concerning inoculation of pure aluminum primary structure, which is realized by introducing into metal bath of specified substances, called inoculants is presented.
Design/methodology/approach: In paper the influence of different inoculants introduced to metal bath in form of master alloy sort AlTi5, AlB3, AlTi5B1, AlTi5B3 and AlTi3C0.15 on refinement of primary structure of pure aluminum is presented. The degree of structure refinement was represented by equiaxed crystals zone content on transverse section of ingot and average area of macro-grain in this zone.
Findings: The results of studies and their analysis show that the most effective refinement of pure aluminum primary structure is obtained with use of master alloy sort AlTi5B1.
Research limitations/implications: In further research, authors of this paper are going to apply this inoculation of pure aluminum in continuous casting technology.
Practical implications: The work presents refinement of structure method which is particularly important in continuous and semi – continuous casting where products are used for plastic forming.
Originality/value: The value of this paper resides in review of refinement effectiveness of different inoculants.
Keywords: Casting; Inoculation; Aluminium; Ingot

Reference to this paper should be given in the following way:
T. Wróbel, J. Szajnar, The influence of inoculants sort on pure Al structure, Journal of Achievements in Materials and Manufacturing Engineering 50/2 (2012) 92-98.

1. Introduction

The phenomenon of crystallization following after pouring molten metal into the mould, determines the shape of the primary casting (ingot) structure, which significantly affects on its usable properties. The crystallization of metal in the mould may result in three major structural zones [1-4]:

- zone of chilled crystals (grains) formed by equiaxed grains with random crystallographic orientation, which are in the contact area between the metal and the mould,
- zone of columnar crystals (grains) formed by elongated crystals, which are parallel to heat flow and are a result of directional solidification, which proceeds when thermal gradient on solidification front has a positive value,
- zone of equiaxed crystals (grains) formed by equiaxed grains with random crystallographic orientation in the central part of the casting. The equiaxed crystals have larger size than chilled crystals and are result of volumetric solidification, which proceeds when thermal gradient has a negative value in liquid phase.

Depending on the chemical composition, the intensity of convection of solidifying metal, the cooling rate i.e. geometry of casting, mould material and pouring temperature (Fig. 1), in the casting may be three (Fig. 2a), two (Fig. 2b) or only one (Figs. 2c and d) structural zone [1-3].

The primary structure of pure metals independently from the crystal lattice type creates practically only columnar crystals [1,5]. This type of structure gives low mechanical properties of castings and mainly is unfavourable for the plastic forming of continuous and semi-continuous ingots, because causing forces extrusion rate reduction and during the ingot rolling delamination of external layers can occur [6-11].

This structure can be eliminated by controlling the heat removal rate from the casting, realizing inoculation, which consists in the introduction of additives to liquid metal and/or influence of external factors for example infra- and ultrasonic vibrations or electromagnetic field [1,5-29].

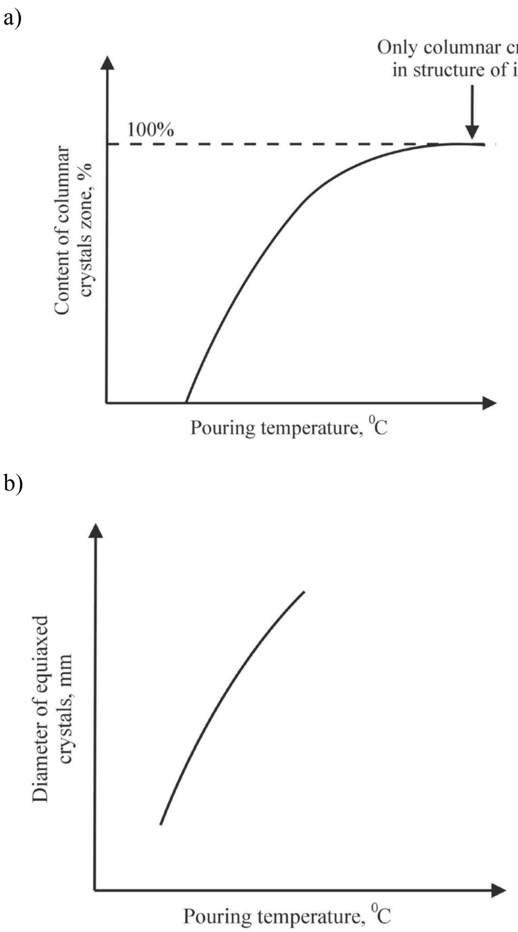


Fig. 1. The influence of pouring temperature on primary structure of ingot i.e. on content of columnar crystals (a) and diameter of equiaxed crystals (b) [1]

As mentioned earlier, the effective method of columnar crystals zone elimination is inoculation, which consists in introducing into metal bath of specified substances, called inoculants. Inoculants increase grains density as result of creation of new particles in consequence of braking of grains growth velocity, decrease of surface tension on interphase boundary of liquid - nucleus, decrease of angle of contact between the nucleus and the base and increase of density of bases to heterogeneous nucleation [1].

The effectiveness of this type of inoculation depends significantly on crystallographic match between the base and the nucleus of inoculated metal. This crystallographic match is described by type of crystal lattice or additionally by index [1]:

$$\xi = \left(1 - \frac{x_b - x_n}{x_n}\right) \cdot 100\% \qquad (1)$$

where:
ξ - match index,
x_b, x_n - parameter of crystal lattice in specified direction, suitable for base and nucleus.

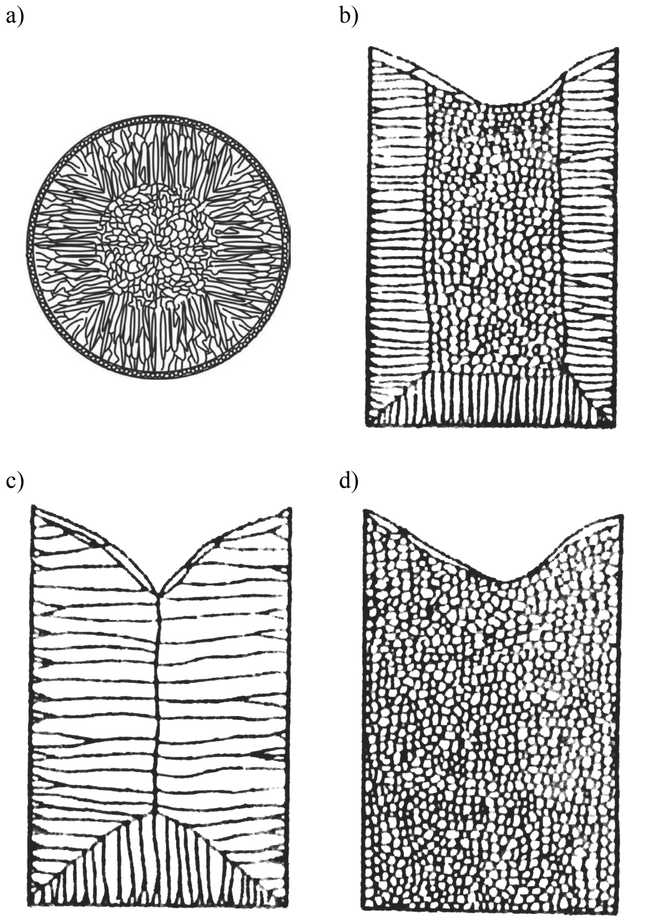


Fig. 2. Types of ingot primary structure: a - chilled, columnar and equiaxed crystals on transverse section, b - columnar and equiaxed crystals, c - only columnar crystals, d - only equiaxed crystals on longitudinal section [3]

When the value of index (ξ) is closer to 100%, it the more effective is the base to heterogeneous nucleation of inoculated metal.

Therefore active bases to heterogeneous nucleation for aluminum are particles which have high melting point i.e. TiC, TiN, TiB, TiB_2, AlB_2 and Al_3Ti (Tab.1) [1, 5-29].

Moreover the effectiveness of inoculants influence can be assessed on the basis of the hypothesis presented in the paper

[12]. This hypothesis was developed at the assumption that the fundamental physical factors affecting on the crystallization process are the amount of give up heat in the crystallization process on the interphase boundary of liquid - solid and the rate of give up heat of crystallization. After analyzing the results of own researches, the author proposed to determine the index (α), which characterizes the type of inoculant.

$$\alpha = \frac{(\Delta E_k / \nu)_s}{(\Delta E_k / \nu)_p} \cdot W \quad (2)$$

where:
ΔE_k - heat of crystallization of 1 mol of inoculant or inoculated metal, J/mol,
ν - characteristic frequency of atomic vibration calculated by the Lindemman formula, 1/s,
s - symbol of inoculant, p - symbol of inoculated metal.
W - parameter dependent on the atomic mass of inoculant and inoculated metal.

On the basis of equation (2) the additives can be divided into three groups:
At $\alpha > 1$ - additives which inhibit crystals growth by the deformation of the crystallization front, thus are effective inoculants.
At $\alpha = 1$ - additives which do not affect on structure refinement.
At $\alpha < 1$ - additives which accelerate crystals growth, favoring consolidation of the primary structure of the metal, thus are deinoculants.

In case of inoculation of Al the index $\alpha = 2.35$ for inoculant in form of Ti and 1.76 for inoculant in form of B.

Table 1.
Characteristic of bases to heterogeneous nucleation of aluminum [30]

Phase	Melting point (circa),°C	Type of crystal lattice	Parameters of crystal lattice, nm
Al	660	Cubical A1	a = 0.404
TiC	3200	Cubical B1	a = 0.431
TiN	3255	Cubical B1	a = 0.424
TiB	3000	Cubical B1	a = 0.421
TiB_2	2900	Hexagonal C32	a = 0.302 c = 0.321
AlB_2	2700	Hexagonal C32	a = 0.300 c = 0.325
Al_3Ti	1400	Tetragonal $D0_{22}$	a = 0.383 c =0.857

2. Range of studies

The aim of studies was assessment of effectiveness of inoculants influence on refinement of pure aluminum primary structure. To studies were used aluminum with a purity of 99.5% and inoculants introduced to metal bath in form of master alloy sort AlTi5, AlB3, AlTi5B1, AlTi5B3 and AlTi3C0.15 (Table 2). Inoculants were introduced to metal bath with small amount i.e. inoculant content did not exceeded values specified in obligatory standards. In studies the authors took the assumption, that amount of all introduced additives $M \leq 30$ ppm.

Table 2.
Chemical composition of master alloy

Elements content in % mas.									
Ti	B	C	Si	Fe	V	Cu	Zn	Cr	Al
AlTi5									
5.45	-	-	0.24	0.31	-	0.04	0.04	0.01	rest
AlB3									
-	3.30	-	0.10	0.14	-	-	-	-	rest
AlTi5B1									
4.70	0.94	-	0.09	0.11	0.09	-	-	-	rest
AlTi3C0.15									
3.10	-	0.15	0.02	0.14	0.15	-	-	-	rest

During the studies cylindrical EN AW-Al99.5 ingots with dimensions of 25 mm diameter and 220 mm length were poured into graphite mould with wall thickness 10 mm. Metal was melted in inductive furnace and temperature was measured with use of NiCr-NiAl thermocouple (pouring temperature was set to 740°C).

The degree of structure refinement was represented by equiaxed crystals zone content (SKR) on transverse section of ingot and average area of macro-grain in this zone (PKR). In aim of realization of refinement measurements in aluminum structure were made macroscopic metallographic studies on transverse section of ingots, which were cut at 100 mm from the bottom. Analized surface was etched with use of solution of: 50 g Cu, 400 ml HCl, 300 ml HNO_3 and 300 ml H_2O.

3. Results of studies

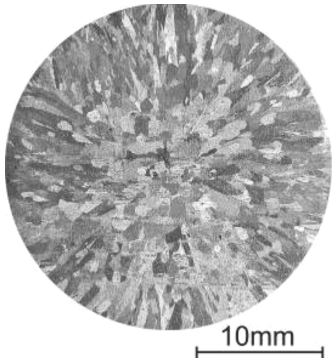
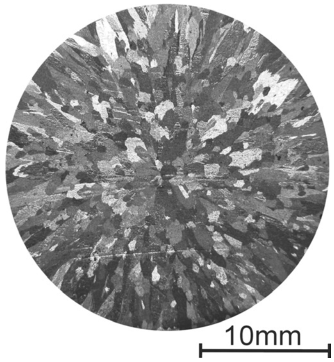
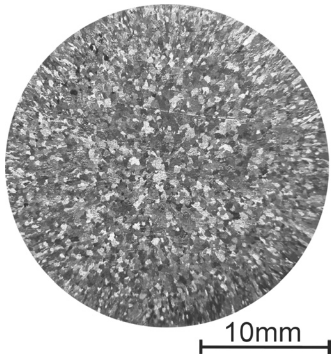
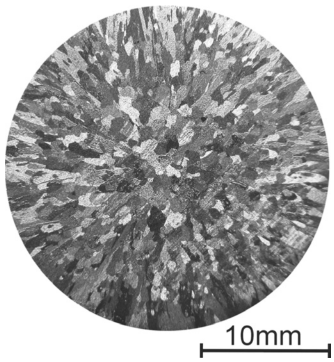
In Table 3 and Figure 3 is presented results of macroscopic metallographic studies of aluminum with a purity of 99.5% ingots. In initial state structure of aluminum is two-zonal, which contains mainly columnar crystals and small amount of equiaxed crystals in central area of ingot. Increase in refinement of structure results from increase in equiaxed crystals zone content on transverse section of ingot and decrease in average area of equiaxed crystal. This refinement was achieved by use of inoculation, which consists in introducing to metal bath of additions in form of titanium and/or boron and/or carbon.

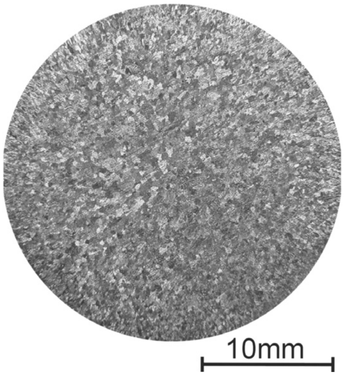
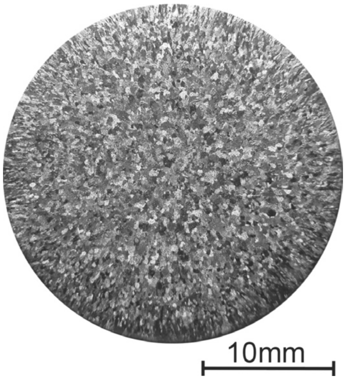
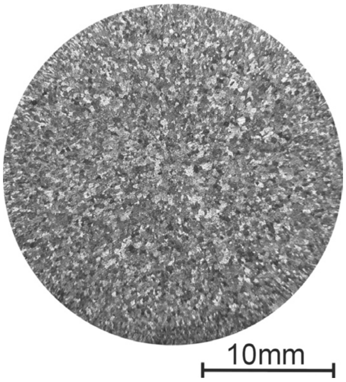
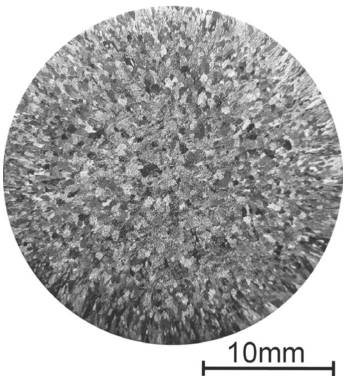
On the basis of obtained results was affirmed that the largest degree of refinement of pure Al, both in respect of the value of SKR and PKR was achieved in result of use of master alloy sort AlTi5B1. Therefore was affirmed that application of Ti:B ratio equals 5:1 assures the greatest degree of structure refinement in comparison to others Ti:B ratio for example 5:3, what is show also in paper [13].

Moreover was demonstrated relatively advantageous influence of B, which was introduced in form of master alloy sort AlB3 on refinement of pure Al structure. This type of inoculation is more effective than inoculation only with use of Ti.

The presence of C in place of B, for example in master alloy sort AlTi3C0.15 weakens the effect of inoculation. This statement is confirmed by the refinement results obtained in ingots inoculated with common use of master alloy sort AlTi3C0.15 and AlB3 or AlTi3C0.15 and AlTi5B1.

Table 3.
Range and results of studies

No.	Inoculant content, ppm			Sort of master alloy	Macrostructure of ingot
	Ti	B	C		
-1-	-2-	-3-	-4-	-5-	-6-
1	-	-	-	-	
2	25.0	-	-	AlTi5	
3	-	25.0	-	AlB3	
4	25.0	-	~1.0	AlTi3C0.15	

-1-	-2-	-3-	-4-	-5-	-6-
5	25.0	5.0	-	AlTi5B1	
6	25.0	5.0	-	AlTi5 + AlB3	
7	25.0	4.0	~1.0	AlTi3C0.15 + AlB3	
8	25.0	3.0	~0.5	AlTi3C0.15 + AlTi5B1	

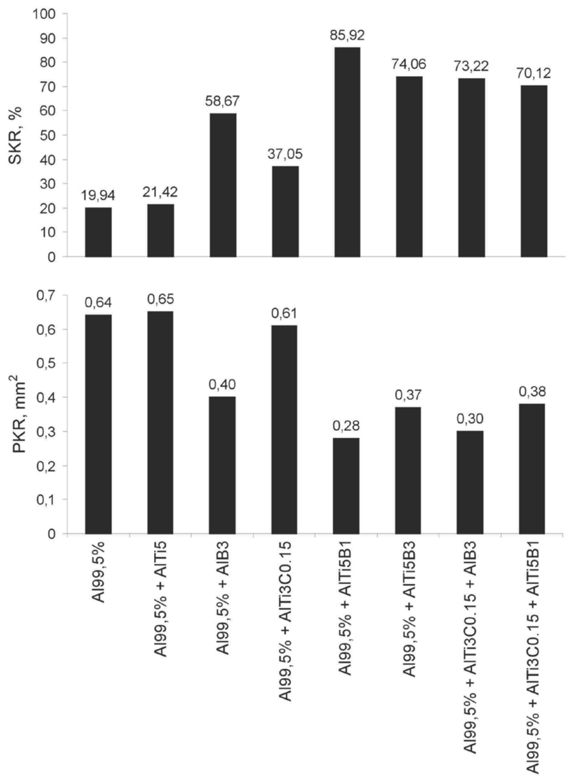


Fig. 3. The influence of master alloy sort on degree of refinement of aluminum with a purity of 99.5% structure

4. Conclusions

Based on conducted studies following conclusions have been formulated:

1. Even a very small amount of inoculating additives introduced to metal bath in the form of Ti, B and C guarantee a significant increase in structure refinement of pure Al ingots.
2. The largest degree of refinement of pure Al was achieved in result of use of master alloy sort AlTi5B1.
3. The master alloys contain Ti and C assure worse effect of refinement of pure Al structure than master alloys contain Ti and B.

Acknowledgements

Project financed from means of National Science Centre.

References

[1] E. Fraś, Crystallization of metals, WNT, Warsaw, 2003 (in Polish).
[2] B. Chalmers, The structure of ingots, Journal of the Australian Institute of Metals 8 (1963) 255-262.
[3] A. Ohno, The solidification of metals, Chijin Shokan Co. Ltd, Tokyo, 1976.
[4] J. Adamczyk, Development of the microalloyed constructional steels, Journal of Achievements in Materials and Manufacturing Engineering 14 (2006) 9-20.
[5] T. Wróbel, Review of inoculation methods of pure aluminium primary structure, Archives of Materials Science and Engineering 50/2 (2011) 110-119.
[6] J. Szajnar, T. Wróbel, Exogeneous inoculation of pure Al with use of electromagnetic field, Journal of Achievements in Materials and Manufacturing Engineering 43/1 (2010) 448-454.
[7] J. Szajnar, T. Wróbel, Inoculation of pure aluminum with an electromagnetic field, Journal of Manufacturing Processes 10/2 (2008) 74-81.
[8] J. Szajnar, T. Wróbel, Inoculation of primary structure of pure aluminium, Journal of Achievements in Materials and Manufacturing Engineering 20/1 (2007) 283-286.
[9] J. Szajnar, T. Wróbel, Methods of inoculation of pure aluminium structure, Journal of Achievements in Materials and Manufacturing Engineering 27/1 (2008) 95-98.
[10] J. Szajnar, T. Wróbel, Influence of magnetic field and inoculation on size reduction in pure aluminium structure, International Journal of Materials and Product Technology 33/3 (2008) 322-334.
[11] T. Wróbel, J. Szajanr, Exogeneous inoculation of pure aluminum, Archives of Foundry Engineering S3 (2011) 241-248 (in Polish).
[12] S. Jura, Modeling research of inoculation process in metals, Publishers of Silesian University of Technology, Gliwice, 1968 (in Polish).
[13] H. Li, T. Sritharan, Y. Lam, N. Leng, Effects of processing parameters on the performance of Al grain refinement master alloy Al-Ti and Al-B in small ingots, Journal of Materials Processing Technology 66/1 (1997) 253-257.
[14] J. Fjellstedt, A. Jarfors, T. El-Benawy, Experimental investigation and thermodynamic assessment of the Al-rich side of the Al-B system, Materials and Design 22/6 (2001) 443-449.
[15] M. Guzowski, G. Sigworth, D. Sentner, The role of boron in the grain refinement of aluminum with titanium, Metallurgical and Materials Transactions 18 (1987) 603-619.
[16] S. Pietrowski, Complex silumins, Journal of Achievements in Materials and Manufacturing Engineering 24/1 (2007) 101-105.
[17] E. Tillová, M. Chalupová, L. Hurtalová, M. Bonek, L.A. Dobrzański, Structural analysis of heat treated automotive cast alloy, Journal of Achievements in Materials and Manufacturing Engineering 47/1 (2011) 19-25.
[18] K. Labisz, L.A. Dobrzański, R. Maniara, A. Olsen, Microstructure evaluation of the Al-Ti alloy with magnesium addition, Journal of Achievements in Materials and Manufacturing Engineering 47/1 (2011) 75-82.
[19] A. Whitehead, Grain refiners (modifiers) of the Al-Ti-C type - their advantages and application, Foundry Review 5 (2000) 179-182.
[20] T. Campanella, C. Charbon, M. Rappaz, Grain refinement induced by electromagnetic stirring: a dendrite fragmentation criterion, Metallurgical and Materials Transactions A 35/10 (2004) 3201-3210.
[21] R. Doherty, H. Lee, E. Feest, Microstructure of stir-cast metals, Materials Science and Engineering 65 (1984) 181-189.

[22] B. Hu, H. Li, Grain refinement of DIN226S alloy at lower titanium and boron addition levels, Journal of Materials Processing Technology 74 (1998) 56-60.
[23] K. Kashyap, T. Chandrashekar, Effects and mechanism of grain refinement in aluminium alloys, Bulletin of Materials Science 24/4 (2001) 345-353
[24] Z. Zamkotowicz, T. Stuczński, B. Augustyn, M. Lech-Grega, W. Wężyk, Sedimentation of intermetallic compounds in liquid aluminium alloys of type AlSiCu(Ti), Proceedings of the Scientific Conference Founding of Nonferrous Metals - Science and Technology, 2003, 77-82 (in Polish).
[25] P. Li, E. Kandalova, V. Nikitin, Grain refining performance of Al-Ti master alloy with different microstructures, Materials Letters 59/6 (2005) 723-727.
[26] B. Murty, S. Kori, M. Chakraborty, Grain refinement of aluminium and its alloys by heterogeneous nucleation and alloying, International Materials Reviews 47/1 (2002) 3-29.
[27] I. Naglič, A. Smolej, M. Doberšek, Remelting of aluminium with the addition of AlTi5B1 and AlTi3C0.15 grain refiners, Metalurgija 47/2 (2008) 115-118.
[28] T. Sritharan, H. Li, Optimizing the composition of master alloys for grain refining aluminium, Scripta Materialia 35/9 (1996) 1053-1056.
[29] C. Vives, R. Ricou, Experimental study of continuous electromagnetic casting of aluminum alloy, Metallurgical and Materials Transactions B 16/2 (1985) 377-384.
[30] J. Donnay, H. Ondik, Crystal date - Determinative Tables, NSRDS – JCPDS, 1973.