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# Methods of inoculation of pure aluminium structure

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# Manufacturing and processing

#### **ABSTRACT**

**Purpose:** The main aim of investigations was the reduction of grain size and unification of structure for pure Al casting by introduction of small amount of inoculant (less than obligatory standart PN-EN 573-3, which concerning about aluminium purity), with electromagnetic field and variable casting parameters.

**Design/methodology/approach:** To investigations it was used light microscopy and TEM. Surfaces of samples which were prepared for macro- and microstructure analysis were etched with use of solution of: 50g Cu, 400ml HCl, 300ml HNO<sub>3</sub> and 300ml H<sub>2</sub>O. Thin foils for TEM investigations were electropolished with use of 20 ml HClO<sub>4</sub> and 80ml CH<sub>3</sub>OH.

**Findings:** The results of investigations and their analysis show possibility of effective inoculation of pure aluminium structure by use of some factors such as: different materials of the mould, influencing of stirring electromagnetic field into metal during solidification, inoculation by introducing AlTi5B1 inoculant into liquid aluminium and changing the pouring temperature.

**Research limitations/implications:** I further research, authors of this paper are going to application of introduced method of inoculation in industrial tests.

**Practical implications:** The work presents refinement of structure method which are particularly important in continuous and semi – continuous casting where products are used for plastic forming. Large columnar crystals zone result in forces extrusion rate reduction and during the ingot rolling delamination of external layers can occur. Thus, in some cases ingot skinning is needed, which rises the production costs.

Originality/value: Contributes to research on size reduction in pure aluminium structure.

Keywords: Casting; Inoculation; Aluminium; Electromagnetic field

# **<u>1. Introduction</u>**

Columnar crystals which are parallel to heat flow, creates primary structure of pure metals independently from type of crystal lattice. This unfavourable structure for plastic forming of ingots can be eliminated by suitable use of technological factors, which are connected with making of mould and preparation of liquid metal (Fig.1) [1,2].

Effective method of columnar crystals zone elimination is inoculation with introduction into metal bath of specified substances, called inoculants, increase grains density as result of creation of new particles in consequence of braking of grains growth velocity, decrease of surface tension on phase boundary of liquid – nucleus, decrease of angle of contact between nucleus and "washer" and increase of density of "washers" to heterogeneous nucleation. This leads to increase of equiaxed crystals zone, which guarantee of mechanical properties improvement, decrease of constituents segregation and limitation of hot cracks. Active base to heterogeneous nucleation for aluminium are particles which have high melting point i.e. TiC, TiB, TiB<sub>2</sub>, AlB<sub>2</sub> and Al<sub>3</sub>Ti (Tab.1) [1-10].

However, this method of inoculation of primary structure is limited for pure metals, because inoculants decrease the degree of purity specified in EN-PN standards. Moreover, inoculant influences negatively on physical properties i.e. electrical conductivity of pure aluminium [11,12].



Fig. 1. Influence of technological parameters on structure of casting [1]

Table 1.									
Characteristic	of	crystal	lattice	of	"washres"	to	heterogeneous		
nucleation formation in aluminium [1,3,13]									

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
TiB	3000	Cubical B1	a = 0.421
TiB <sub>2</sub>	2900	Hexagonal C32	a = 0.302 c = 0.321
AlB <sub>2</sub>	2700	Hexagonal C32	a = 0.300 c = 0.325
Al <sub>3</sub> Ti	1400	Tetragonal D0 <sub>22</sub>	a = 0.383 c =0.857

But introduction of small amount of inoculant can be strengthened by use of size reduction other method i.e. use of ultrasonic vibration or electromagnetic field to force liquid metal movement in mould [1,2,11,12,14,15].

Forced liquid metal movement influences in diversified way on changes in structure of casting i.e. by changes of thermal and concentration conditions on crystallization front, which decrease or completely stops the velocity of columnar crystals growth and by [1,2,11,12]:

• tear off of crystals from mould wall, which are transferred into metal bath, where they can convert in equiaxed crystals,

- parting of dendrite by coagulation and melting as result of influences of temperture fluctuation and breaking as result of energy of liquid metal movement,
- crystals transport from free surface to inside the liquid metal,
- crystals from over-cooled outside layer of bath are transported into liquid metal.

# 2. Range of studies

In the paper there is a review of methods modifying disadvantageous primary structure of aluminium using different factors which can change crystallization conditions. In the practical part of the paper the research included presentation of some particular factors modifying primary structure of aluminium EN AW-Al99.5 such as: different materials of the mould, influencing of stirring electromagnetic field into metal during solidification, inoculation by introducing AlTi5B1 inoculant into liquid aluminium and changing the pouring temperature in order to form advantageous primary structure of aluminium.

Full experimental plan with results of equiaxed crystals zone content (SKR) on cross-section of cylindrical castings \$45\$ and 180 mm length of aluminium EN AW-AI 99.5 and average area of equiaxed crystal (PKR) measurements are shown in Table 2.

Metallografic examinations of the material structure were made on Nikon light microscope with magnification from 50x to 100x. Surfaces of samples which were prepared for macrostructure analysis were etched with use of solution of: 50g Cu, 400ml HCl, 300ml HNO<sub>3</sub> and 300ml H<sub>2</sub>O.

			Parameters of size reduction			
Sample number	Inoculant AlTi5B1	Electromagnetic field <sup>*</sup>	Material of mould <sup>**</sup>	Pouring temperature, °C	PKR, mm <sup>2</sup>	SKR, %
1	-			700	6.213	81.01
2	25Ti + 5B ppm	-	Shell mould $g = 7mm$	/00 =	2.731	100.00
3	-	-		740 -	19.146	92.61
4	25Ti + 5B ppm	-			3.097	100.00
5	-	-		700	17.645	8.63
6	25Ti + 5B ppm	-			0.919	66.20
7	-	$\mathbf{B}=50\mathrm{mT},$			1.551	34.68
8	25Ti + 5B ppm	f = 0.5Hz, t = 30s	Graphite		0.333	58.93
9	-	-	g = 7mm	740 -	26.320	24.34
10	25Ti + 5B ppm	-			3.830	51.44
11	-	$\mathbf{B}=50\mathrm{mT},$			0.680	32.05
12	25Ti + 5B ppm	f = 0.5Hz, t = 30s			0.570	45.24
13	-	-		_	0.811	83.51
14	25Ti + 5B ppm	-		700	0.471	88.05
15	-	$\mathbf{B}=50\mathrm{mT},$	$C \rightarrow 1$		0.400	100.00
16	25Ti + 5B ppm	f = 0.5Hz, t = 30s	V10CrNi18 8		0.262	100.00
17	-	-	g = 3mm	740 —	3.885	65.63
18	25Ti + 5B ppm	-			0.189	87.83
19	-	$\mathbf{B}=50\mathrm{mT},$			0.226	69.83
20	25Ti + 5B ppm	f = 0.5Hz, $t = 30$ s			0.209	100.00

#### Table 2. Range and results of investigations

\*- impulse reverse electromagnetic field with parameters: magnetic induction – B, pulse frequency of electromagnetic field – f, time of electromagnetic field action – t.

\*\*- g – wall thickness.

Ivestigations of diffraction and thin foils were made on the JEM JEOL 2000 FX transmision electron microscope at the accelerating voltage of 200kV. Thin foils for TEM investigations were electropolished with use of 20 ml HClO<sub>4</sub> and 80ml CH<sub>3</sub>OH.

#### **3. Results and analysis**

Selected results of metallographic research are presented on Figs.2-4. After inoculation with 25ppm Ti + 5ppm B, increase in size reduction of structure is observed (Fig. 2). It result from "washers" to heterogeneous nucleation formation. As result of investigations of thin foils in TEM, it was identified "washers" of type Al<sub>3</sub>Ti (Fig. 5). Moreover on basis of literature datas [1, 3, 6-8] is possible to say, that important contribution in size reduction of Al structure have also "washers" of type TiB, TiB<sub>2</sub>, AlB<sub>2</sub> and TiC.

Whereas, increase in size reduction in aluminium EN AW-Al99.5 structure, after casting with influence of impulse reverse electromagnetic field (Fig. 3) result from high velocities that are attained in liquid metal and which lead to columnar crystals tearing occurring on crystallization front and additional crystal nucleuses formation. Effect of mechanical erosion of crystallization front in form of breaking columnar crystals is shown on Figure 4. Effect of mechanical erosion of crystallization front is strengthened by melting of dendrite as result of influences of temperture fluctuation, which result from influence of electromagnetic field.



Fig. 2. Macrostructure of alumnium EN AW-Al99.5: a - sample 3, b - sample 4



Fig. 3. Macrostructure of aluminium EN AW-Al99.5: a – sample 17, b – sample 20

Moreover with increase in degree of undercooling and decrese in pouring temperature increase in size reduction is observed (Tab.2). For example comparison between Al structure after casting to cast steel mould (large cooling - Fig. 3) and after casting to shell mould (low cooling - Fig. 2).



Fig. 4. Microstructure of alumnium EN AW-Al99.5 - 100x: a - area 1 from Fig. 3b, b - area 2 from Fig. 3b



Fig. 5. a) Diffraction and structure of thin foil from EN AW-Al99.5 after inoculation with (Ti+B) - 30000x, b) solution of the diffraction pattern from Fig. 5a

# 4.Conclusions

Based on conducted studies following conclusions have been formulated:

- 1. Introduction of small amount of inoculant sort AlTi5B1 less than obligatory standard PN-EN 573-3 (concerning about aluminium purity), strongly increase size reduction in pure aluminium structure.
- 2. Influence of electromagnetic field increas size reduction in Al structure as result of mechanical and thermal erosion of crystallization front.
- 3. With increase in degree of undercooling and decrese in pouring temperature, increase in size reduction in Al structure is observed.

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