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Inoculation of aluminium with titanium and boron addition

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Properties

ABSTRACT

Purpose: The main aim of studies was to determine mechanism of inocualtion of pure alumnium EN AW-Al99,5 structure with addition of titanium and boron, which are introduced to liquid metal in small ammount - less than obligatory standard PN-EN 573-3 (concerning about aluminium purity).
Design/methodology/approach: To investigations it was used light microscopy, X-ray Phase Analysis and TEM. Surfaces of samples which were prepared for macrostructure analysis were etched with use of solution of: 50g Cu, 400ml HCl, 300ml HNO_3 and 300ml H_2O. Surfaces of samples which were were prepared for microstructure analysis were etched with use of solution of: 0,5ml HF, 99,5ml H_2O. Thin foils for TEM investigations were electropolished with use of 20 ml $HClO_4$ and 80ml CH_3OH.
Findings: The results of investigations and their analysis show, that increase of size reduction in aluminium EN AW-Al99,5 after inoculation with (Ti+B) result from "washers" of type Ti_3Al and $CuTi_2$ to heterogeneous nucleation formation.
Research limitations/implications: I further research, authors of this paper are going to identifiy the "washers" to heterogeneous nucleation, in aluminium structure after inoculation with zirconium and vanadium addition.
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: Working properties of materials and products; Aluminium; Titanium; Boron

1. Introduction

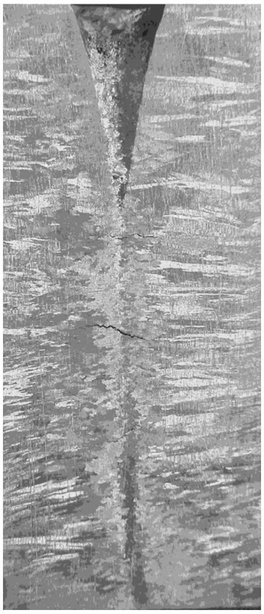
Columnar crystals which are parallel to heat flow, creates primary structure of pure metals independently from type of crystal lattice (Fig. 1). This unfavourable structure for plastic forming of ingots can be eliminated by controlling of heat abstraction velocity from cast, change in chemical constitution and liquid metal convection [1-14].

Effective method of columnar crystals zone elimination is using in practice of theory of heterogeneous nucleation lead to steering of crystallization process by formation of proper base to crystal nucleuses. Producing in crystallizing liquid very small particles of proper compound so-called "washers", which performs conditions of active base for heterogeneous nucleation – show analogy in respect of crystallography with modified metal, that is isomorphus inclusion it increases quantity of crystallizing small crystals in volume unit [1, 8-13].

Thus, 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 $\sigma_{z/l}$ on phase boundary of liquid – nucleus, decrease of angle of contact θ between nucleus and "washer" and increase of density of "washers" to heterogeneous nucleation (Fig. 2) [1, 8, 9].

This leads to increase of equiaxed crystals zone, which guarantee of mechanical properties improvement, decrease of constituents segregation and limitation of hot cracks [1].

a)



b)

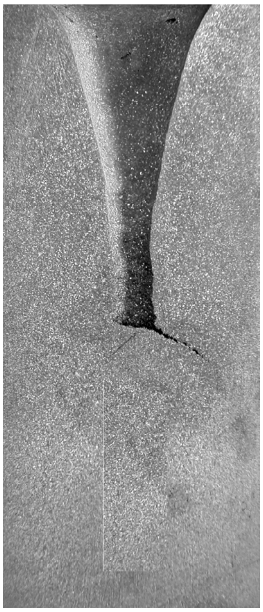


Fig. 1. Structure of aluminium ingot: a) without inoculation, b) after inoculation

Active base to heterogeneous nucleation for aluminium are TiC, TiB_2, AlB_2 and Ti_3Al (Tab. 1) [1, 8-10].

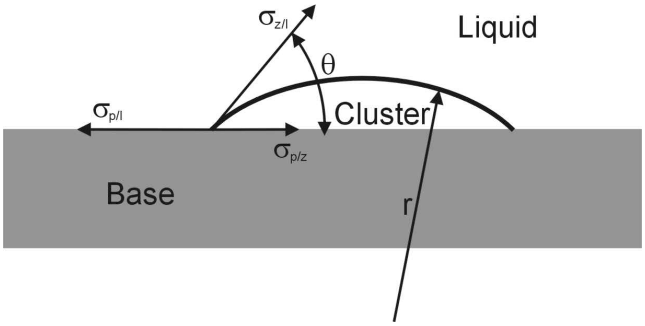


Fig. 2. Theoretical model of heterogeneous nucleation [1, 9]

Table 1.
Characteristic of crystal lattice of "washers" to heterogeneous nucleation formation in aluminium [1, 8-10, 15]

Phase	Type of crystal lattice	Parameters of crystal lattice
Al	Cubical	a = 0,404 [nm]
TiC	Cubical	a = 0,431 [nm]
TiB_2	Hexagonal	a = 0,302 [nm] c = 0,321 [nm]
AlB_2	Hexagonal	a = 0,300 [nm] c = 0,325 [nm]
Ti_3Al	Tetragonal	a = 0,401 [nm] c =0,406 [nm]

2. Range of studies

The main aim of studies was to determine mechanism of inocualtion of pure alumnium EN AW-Al99,5 structure with addition of titanium and boron, which are introduced to liquid metal in small ammount - less than obligatory standard PN-EN 573-3 (concerning about aluminium purity).

The investigations were made using cylindrical castings ϕ45 from aluminium EN AW-Al99,5 (Tab. 2) and inoculant sort AlTi5B1 (Tab. 3).

Metallografic examinations of the material structure were made on Nikon light microscope with magnification from 100x to 600x. Surfaces of samples which were prepared for macrostructure analysis were etched with use of solution of: 50g Cu, 400ml HCl, 300ml HNO_3 and 300ml H_2O. Surfaces of samples which were prepared for microstructure analysis were etched with use of solution of: 0,5ml HF, 99,5ml H_2O. X-ray examinations of investigated Al was made using DRON 2.0 diffractometr with Co anode. X-ray tube was supplied with the current I = 10mA under voltage of U = 25kV. Diffraction examinations were performed within the range of angles 2θ from 35° to 100°. The measurement step was 0,1° in length whilst the pulse counting time was 1s. Investigations of diffraction and thin foils were made on the JEM JEOL 2000 FX transmission electron microscope at the accelerating voltage of 200kV. Thin foils for TEM investigations were electropolished with use of 20 ml $HClO_4$ and 80ml CH_3OH.

Table 2.
Chemical composition of the investigated aluminium EN AW-Al99,5

Mass contents in %									
Si	Mg	Cu	Fe	Mn	Zn	Ti	Ni	Pb	Al
0,110	0,009	0,020	0,298	0,003	0,019	0,003	0,004	0,002	rest

Table 3.
Chemical composition of inoculant sort AlTi5B1

Mass contents in %											
Si	Mg	Cu	Fe	Mn	Zn	Ni	V	Cr	Ti	B	Al
0,08	0,05	0,20	0,20	0,05	0,05	0,04	0,02	0,01	5,50	1,00	rest

3. Results and analysis

Selected results of metallographic research are presented on Fig.3. and 4. After inoculation with 25ppm Ti + 5ppm B, increase in size reduction of structure is observed. It result from "washers" to heterogeneous nucleation formation.

a) b)

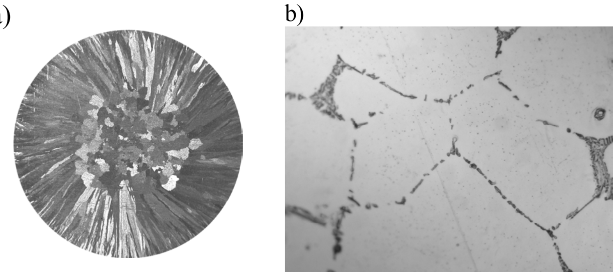


Fig. 3. Macro- (a) and microstructure – 600x (b) of alumnium EN AW-Al99,5 without inoculation

a) b)

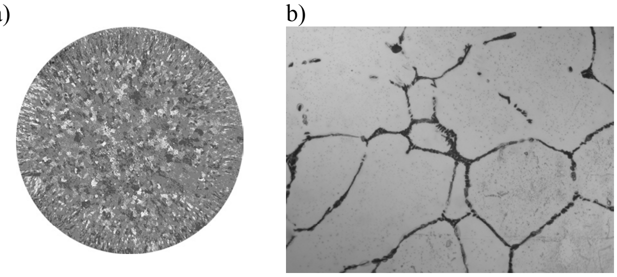


Fig. 4. Macro- (a) and microstructure – 600x (b) of alumnium EN AW-Al99,5 after inoculation with 25ppm Ti + 5ppm B

As result of X-ray analyses it was identified "washer" of type titanium carbide TiC (Fig. 5). Probably occurrence of carbon results from diffusion of this element from graphitoidal metlting pot in which was melted aluminium. Titanium carbide effectively increases refinement in structure because shows analogy in respect of crystallography with aluminium, that is isomorphus inclusion.

As result of investigations of thin foils in TEM, it was identified "washers" of type Ti_3Al and $CuTi_2$ (Fig. 6 and 7). Probably occurrence of copper results from presence in chemical composition of this element in metallic charge and in inoculant (Tab. 1 and 2).

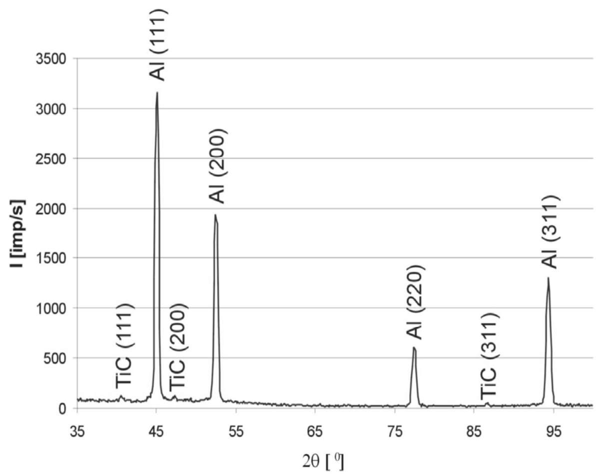


Fig. 5. X-ray diffraction of aluminium EN AW-Al 99,5 after inoculations Ti and B

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. Compounds TiC, Ti_3Al and $CuTi_2$ are active base for heterogeneous nucleation and strongly influences on size reduction increases in aluminium structure.

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a) b) c)

Fig. 6. a) Structure of thin foil from alumnium EN AW-Al99,5 after inoculation with (Ti + B) – 30000x , b) diffraction pattern from the area as in fig., a, c) solution of the diffraction pattern from fig. b

a) b) c)

Fig. 7. a) Structure of thin foil from alumnium EN AW-Al99,5 after inoculation with (Ti + B) – 20000x , b) diffraction pattern from the area as in fig., a, c) solution of the diffraction pattern from fig. b

References

[1] E. Fraś, Crystallization of metals, WNT, Warsaw, 2003 (in Polish).

[2] M. Robert, E. Zoqui, F. Tanabe, T. Motegi, Producing thixotropic semi-solid A356 alloy: microstructure formation x forming behaviour, Journal of Achievements in Materials and Manufacturing Engineering 20 (2007) 19-26

[3] J. Szajnar, T. Wróbel, Inoculation of primary structure of pure aluminium, Journal of Achievements in Materials and Manufacturing Engineering 20 (2007) 283-286.

[4] J. Szajnar, T. Wróbel, Influence of magnetic field and inoculation on columnar structure transformation, Journal of Achievements in Materials and Manufacturing Engineering, 17 (2006) 209-212.

[5] S. Asai, Recent development and prospect of electromagnetic processing of materials, Science and Technology of Advanced Materials 1 (2000) 191-200.

[6] S. Argyropoulos, A. Mikrovas, D. Doutre, Dimensionless correlations for forced convection in liquid metals: Part I. Single-phase flow, Metallurgical and Materials Transactions 4/32B (2001) 239-246.

[7] J. Szajnar, M. Stawarz, T. Wróbel, Inoculation of pure aluminium structure with Ti+B addition in impulse magnetic field, Journal of Achievements in Materials and Manufacturing Engineering 14 (2006) 64-69.

[8] S. Jura, Modeling research of modyfication process in metals, Scientific book of Silesian University of Technology, Mechanic 32, Gliwice, 1968 (in Polish).

[9] Z. Poniewierski, Crystallization, structure and properties of silumines, WNT, Warsaw, 1989 (in Polish).

[10] J. Szymszal, E. Krzemień, T. Zając, Modyfication of metals and alloys, Silesian University Publishers, Gliwice, 1984 (in Polish).

[11] T. Hang, T. Sritharan, P. Seow, Grain refinement of DIN226 alloy at high titanium and boron inoculation levels, Scripta Materiallia 35/7 (1996) 869-872.

[12] B. Hu, H. Li, Grain refinement of DIN226S alloy at lower titanium and boron addition levels, Journal of Materials Processing Technology 78 (1998) 56-60.

[13] H. Banghong, L. Hang, Comparison of effects of masters alloy containig titanium and/or boron on the grain size and dendrite arm spacing of DIN226S aluminium alloy, Journal of Materials Science Letters 16 (1997) 1750-1752.

[14] M. Kciuk, Structure, mechanical properties and corrosion resistance of AlMg5 alloy, Journal of Achievements in Materials and Manufacturing Engineering 17 (2006) 185-188.

[15] P. Hirsch, A. Howie, R. Nicholson., D. Pashley, Electron microscopy of thin crystals, Butterworths, Londyn, 1965.