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Influence of Inoculation on Properties of Aluminium EN AW-Al99,5

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Abstract

The main aim of studies was to determine common influences of two different structure refinement methods on EN AW-Al99,5 electrical conductivity and deformability. The first method is internal factor – inoculation with small amount of (Ti+B) less than obligatory standard PN-EN 573-3 and the second method is external factor – influence of electromagnetic field on crystallization process. These methods of structure refinement are particularly important in 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. 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 Al_3Ti and $CuTi_2$ to heterogeneous nucleation formation. Moreover, with increase in inoculant content decrese in electrical conductivity is observed. Size reduction in primary structure strongly influences on deformability.

Keywords: Mechanical properties, Aluminium, Titanium, Boron, Electromagnetic field

1. Introduction

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 controlling of heat abstraction velocity from cast, change in chemical constitution and liquid metal convection [1].

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÷5].

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 on phase boundary of liquid – nucleus, decrease of angle of contact between nucleus and “washer” and increase of density of “washers” to heterogeneous nucleation [1, 2].

This leads to increase of equiaxed crystals zone, which guarantee of mechanical properties improvement, decrease of constituents segregation and limitation of hot cracks [1, 6].

Active base to heterogeneous nucleation for aluminium are particles which have high melting point i.e. TiC, TiB_2, AlB_2 and Al_3Ti (tab.1) [1÷5, 7].

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 (fig. 1).

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].

Table 1.
Characteristic of crystal lattice of "washres" to heterogeneous nucleation formation in aluminium [2, 4, 5, 8]

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_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

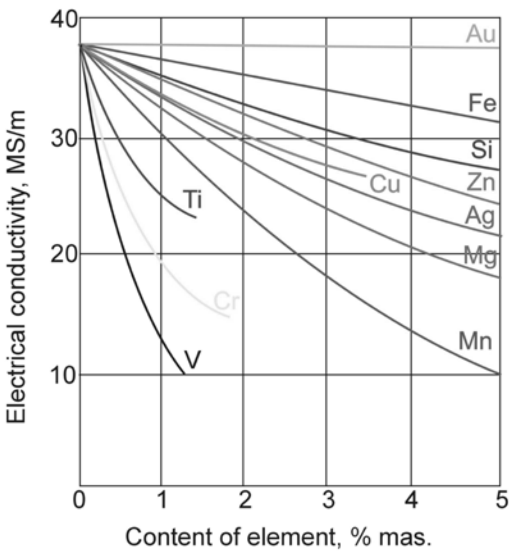


Fig. 1. Influence of different elements on electrical conductivity of EN AW-Al99,99 [9]

Usage of electromagnetic field as result of feeding of coil by current intensity (*J_0*), induce of rotaty current (*J*) in metal bath. Interaction between rotary current and magnetic field (*B*) result in creation of magnetohydrodynamic force (*F*), which generetes liquide metal movement in mould (fig. 2) [10÷12].

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, 13÷15]:

– 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.

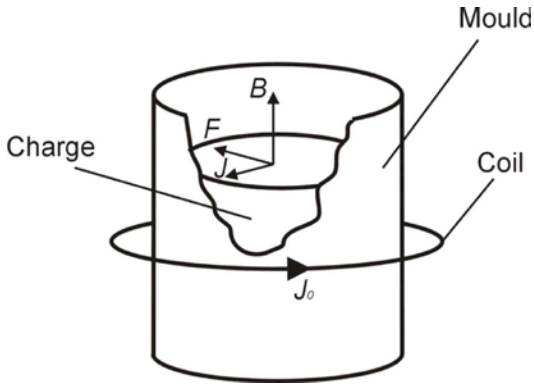


Fig. 2. Generation of liquide metal movement in mould [10]

2. Range of studies

The main aim of studies was to determine common influences of two different structure refinement methods on EN AW-Al99,5 electrical conductivity and deformability. The first method is internal factor – inoculation with small amount of (Ti+B) less than obligatory standard PN-EN 573-3 (concerning about aluminium purity) and the second method is external factor – influence of electromagnetic field on crystallization process.

The investigations were made using cylindrical ingots ϕ45 and 180 mm length from aluminium EN AW-Al99,5 and inoculant sort AlTi5B1. In investigations the influence on crystallization process can be realize by use of impulse reverse elektromagnetic field – IRPM. In IRPM was applied reversing movement with pause between following changes of electromagnetic field direction. Test castings were casted with exactly specified parameters: pulse frequency of electromagnetic field (f), magnetic induction (B) and time of magnetic field action (t), which became optimized on basis of earlier investigations of EN AW-Al99,98 [13÷15] and they were suitably 0,5 [Hz], 50 [mT] i 30[s].

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 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 lenght while the pulse counting time was 1s. 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 $HClO_4$ and 80ml CH_3OH.

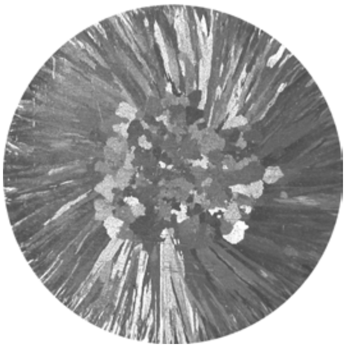
Electrical conductivity was measured with use of Thomsons bridge on sample with dimensions ϕ45 and 15mm length, which were cuting directly from ingots and on sample wuth dimension ϕ10 and 400 mm length, which were casted in shell mould.

Deformability was estimated on basis of sheet press forming test with use of Erichsen method with according to norm PN-EN ISO 20482. Diameter of used stamp was 20 mm. Sheet of thickness 1 mm, were obtained as result of rolling of sample with double roll pass.

3. Results and analysis

Selected results of metallographic research are presented on fig.3÷6. After inoculation with 25ppm Ti + 5ppm B, increase in size reduction of primary structure is observed (fig. 5). It result from "washers" to heterogeneous nucleation formation. Whereas, increase in size reduction in aluminium EN AW-Al99,5 structure, after casting with influence of impulse reverse electromagnetic field (fig. 4) 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.

a)



b)

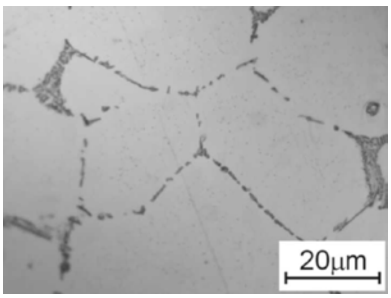


Fig. 3. Macro- (a) and microstructure (b) of alumnium EN AW-Al99,5 without inoculation

a)

b)

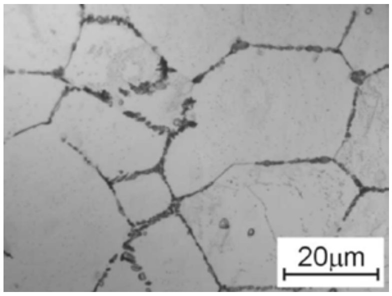
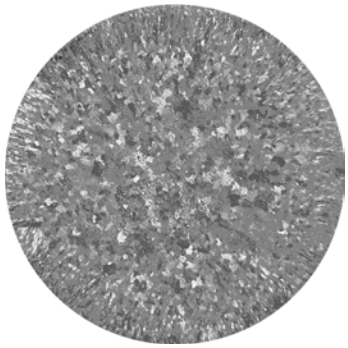


Fig. 4. Macro- (a) and microstructure (b) of alumnium EN AW-Al99,5 after casting with influence of IRPM

a)



b)

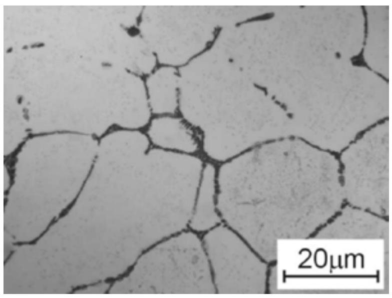
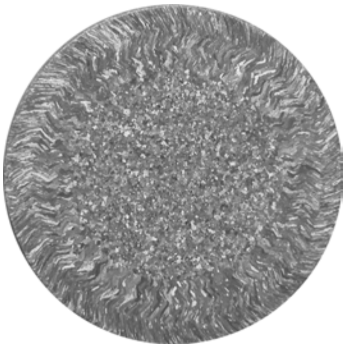


Fig. 5. Macro- (a) and microstructure (b) of alumnium EN AW-Al99,5 after inoculation with 25ppm Ti + 5ppm B

Moreover, common influence of impulse reverse electromagnetic field and inoculation with (Ti+B) (fig. 6) result in larger equiaxed crystals zone content and smaller size of macrograin than in standard sample (fig.3) and comparable in sample which was casted only with inoculation (Ti+B) (fig.5) but it has larger size of macrograin than sample which was casted with influences of electromagnetic field and inoculation.

a)



b)

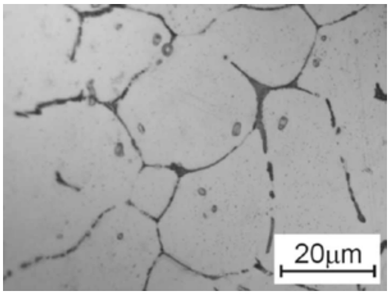


Fig. 6. Macro- (a) and microstructure (b) of alumnium EN AW-Al99,5 which was casted with influence of IRPM and with inoculation with 25ppm Ti + 5ppm B

As result of X-ray analyses it was identified "washer" of type titanium carbide TiC (fig.7). 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 Al_3Ti (fig. 8) and $CuTi_2$ (fig. 9). Probably occurrence of copper results from presence in chemical composition of this element in metallic charge and in inoculant.

In table 2 are presented results of electrical conductivity measurements of aluminium EN AW-Al99,5 which was casted with influences of electromagnetic field. Size reduction as result of use of electromagnetic field does not influence negatively on value of electrical conductivity.

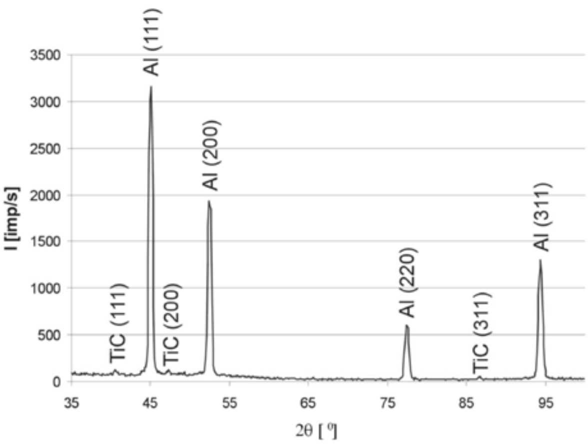
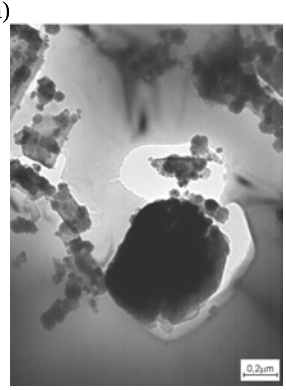


Fig. 7. X-ray diffraction of aluminium EN AW-Al 99,5 after inoculations Ti and B

a)



b)

c)

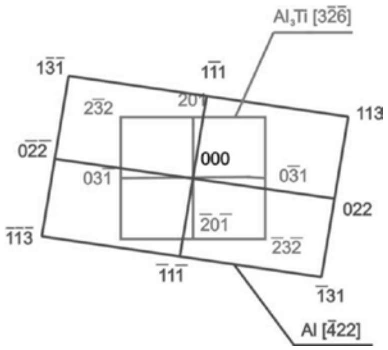


Fig. 8. a): Structure of thin foil from alumnium EN AW-Al99,5 after inoculation with (Ti + B) – 30000x , b): diffraction pattern from the area as in fig. a, c): solution of the diffraction pattern from fig. b

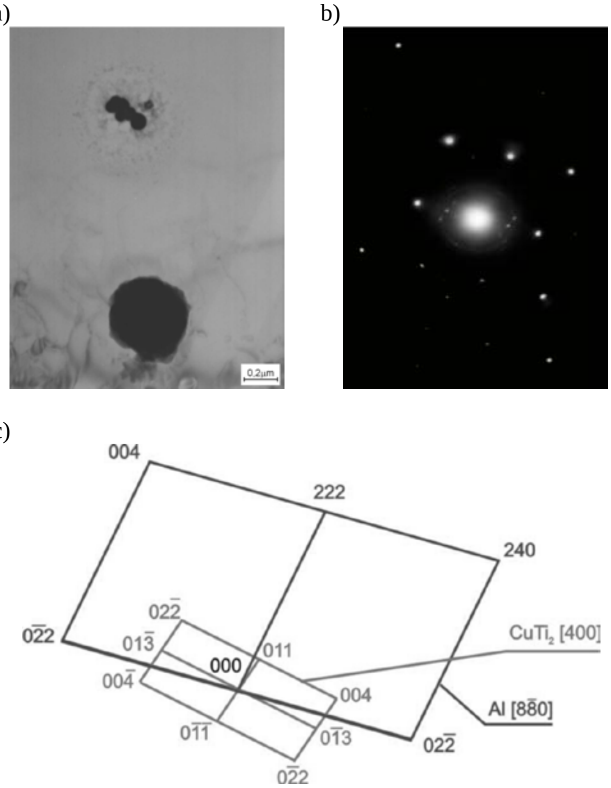


Fig. 9. a): Structure of thin foil from alumnium EN AW-Al99,5 after inoculation with (Ti + B) – 30000x , b): diffraction pattern from the area as in fig. a, c): solution of the diffraction pattern from fig. b

Table 2.
Results of electrical conductivity measurements for sample with area of cross-section 1589,625 mm^2 and length 15 mm

Type of inoculation	R [μΩ]	ρ [μΩ·m]	γ [MS/m]
lack	2,0	0,24	4,16
IRPM	2,0	0,24	4,16
IRPM and (25ppm Ti + 5ppm B)	2,0	0,24	4,16

where:
R – resistance (measured value),
ρ - resistivity,
γ - conductivity.

In table 3 are presented results of electrical conductivity measurements of aluminium EN AW-Al99,5 after inoculkation with (Ti + B). With increase in inoculant content decrese in electrical conductivity is observed (fig. 10). Decrease in electrical conductivity result from influence of Ti, which segregation on grain boundary of Al is observed.

Table 3.
Results of electrical conductivity measurements for sample with area of cross-section 78,5 mm^2 and length 400 mm

Quantity of inoculant		R [μΩ]	ρ [μΩ·m]	γ [MS/m]
Ti [ppm]	B [ppm]			
0	0	14940,13	2,9320	0,3411
25	5	15513,38	3,0445	0,3285
50	10	16244,08	3,1879	0,3137
100	20	16688,92	3,2752	0,3053
150	30	17021,66	3,3405	0,2993
200	40	17340,13	3,4030	0,2938

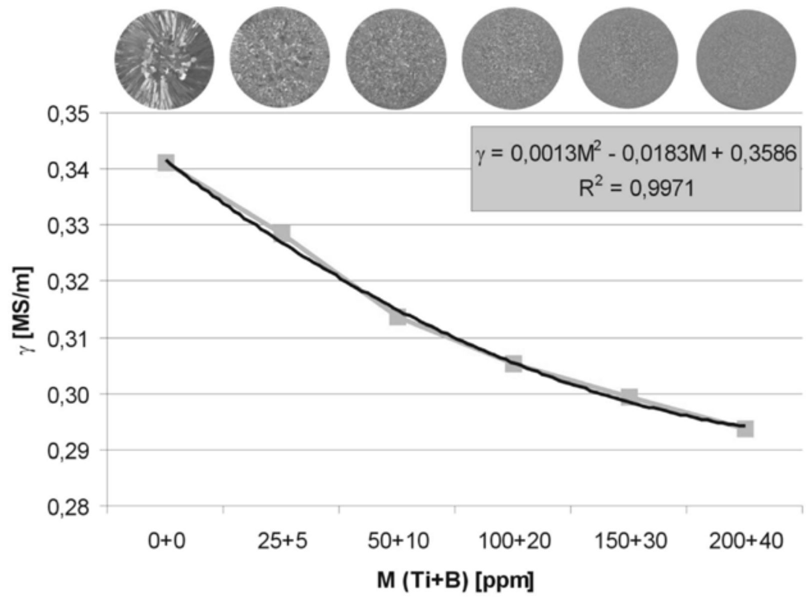


Fig. 10. Electrical conductivity (γ) of aluminum EN AW-Al99,5 in quantity of inoculants M (Ti + B) function

Value of size reduction in ingots primary structure strongly influences on deformability of sheet. After rolling, delamination of external layers occur in ingots, which were casted without inoculation or only with influence of (Ti + B) inoculation (fig. 11a) or only with influence of electromagnetic field. The best quality has sheet from ingot which was casted with common influence of (Ti+B) inoculation and electromagnetic fired (fig. 11b). It is confirmed by press forming test. With increase in size reduction in primary structure decrese in Erichsen number (IE) i.e. depth of entry of stamp into sheet till to appearance of crack (value in mm), is observed (fig. 12).

a) b)

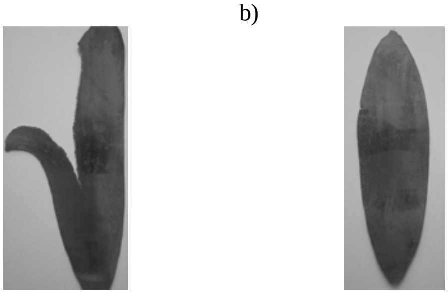


Fig. 11. View after rolling of sample of aluminium EN AW-Al99,5: a) after inoculation with 25ppm Ti + 5ppm B, b) which was casted with influence of IRPM and with inoculation with 25ppm Ti + 5ppm B

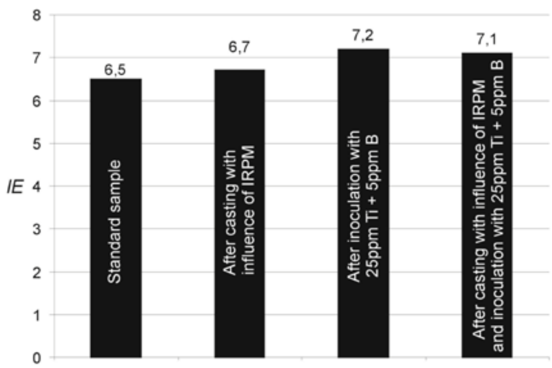


Fig. 12. Results of press forming test

4. Conclusion

Based on conducted studies following conclusions have been formulated:

1. Inoculation with (Ti + B) strongly increase size reduction in pure aluminium structure. But this effective method of increasing size reduction is limited for Al, because inoculants decrease the degree of purity and electrical conductivity.
2. Influence of impulse reverse electromagnetic field on solidification process of pure aluminium, aided size reduction, which creates mainly by introduction of small amount of inoculant sort AlTi5B1 - less than obligatory standard PN-EN 573-3 (concerning about aluminium purity).
3. Compounds TiC, Al_3Ti and $CuTi_2$ are active base for heterogeneous nucleation and strongly influences on size reduction increases in aluminium structure.
4. Size reduction in primary structure i.e. value of equiaxed crystals zone content on cross-section of ingot and size of macrograin in this zone, strongly influences on deformability.

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