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Review of inoculation methods of pure aluminium primary structure

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ABSTRACT

Purpose: In paper the problem concerning inoculation of pure aluminium primary structure, which is realized mainly by intensification of liquid metal movement in mould by use of rotating electromagnetic field is presented.

Design/methodology/approach: In paper the influence of electromagnetic field of type rotating, rotating with reverse and rotating with variable frequency, which generating forced movement during the crystallization of liquid aluminium in mould is presented. Effect of structure refinement obtained by influence of electromagnetic field was compared with refinement obtained by use of traditional inoculation, which consists of introducing of additions in form of titanium, boron and carbon to the metal bath. In paper the results of author own researches, supplemented by literature data are mainly presented.

Findings: The results of studies and their analysis show possibility of effective refinement of pure aluminium primary structure, only with use of rotating electromagnetic field.

Research limitations/implications: In further research, author of this paper is going to apply of introduced method of aluminium casting with use of electromagnetic field in continuous casting stand.

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 new effective method of inoculation of pure Al, which was realized only by use of electromagnetic field.

Keywords: Casting; Inoculation; Aluminium; Ingot

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MATERIALS MANUFACTURING AND PROCESSING

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, 2]:

- 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 cooling rate, chemical composition and the intensity of convection of solidifying metal, in the casting may be three, two or only one structural zone.

Due to the small width of chilled crystals zone, the usable properties of casting depend mainly on the width and length of the columnar crystals, the size of equiaxed crystals and content of theirs zone on section of ingot, as well as on interdendritic or interphase distance in grains such as eutectic or monotectic. For example, you can refer here to a well-known the Hall-Peth law describing the influence of grain size on yield strength (Fig. 1) [3]:

$$\sigma_y = \sigma_0 + k \bullet d^{-1/2} \quad (1)$$

where:
σ_y – yield strength, MPa,
σ_o – approximate yield strength of monocrystal, for Al amount to 11.1 MPa,
k – material constant characterizing the resistance of grain boundaries for the movement of dislocations in the initial stage of plastic deformation (strength of grain boundaries), for Al amount to 0.05 $MN \cdot m^{-3/2}$,
d – grain size, mm.

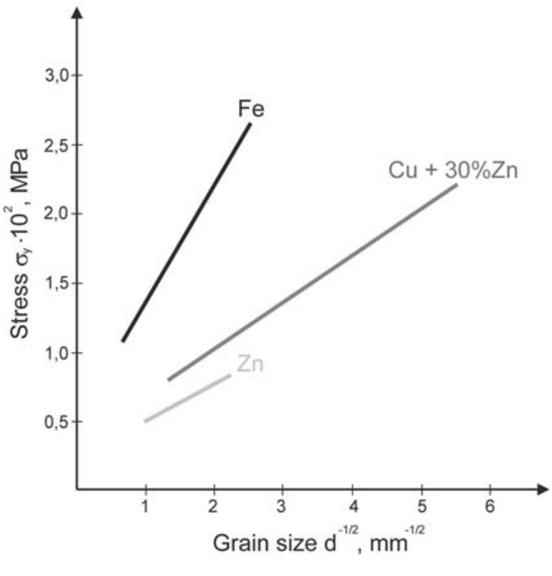

Fig. 1. The influence of grain size on yield strength of Fe, Zn and α brass [3]

The primary structure of pure metals independently from the crystal lattice type creates practically only columnar crystals [1]. According to presented data, 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 [4, 5]. 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.

2. Inoculation of pure Al structure by the introduction of additives

In aim to obtain an equiaxed and fine-grained structure, which gives high mechanical properties of castings, can use inoculation, which occurs in introducing into metal bath of specified substances, called inoculants [1]. 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, 6]. 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]:

$$\xi = \left(1 - \frac{x_b - x_n}{x_n}\right) \bullet 100\% \quad (2)$$

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

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 (Table 1) [1, 4-16].

Table 1.
Characteristic of bases to heterogeneous nucleation of aluminium [17]

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

Moreover the effectiveness of inoculants influence can be assessed on the basis of the hypothesis presented in the paper [6]. 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 analysing the results of own researches, the author proposed to determine the index (α), which characterizes the type of inoculant.

$$\alpha = \frac{(\Delta E_k / \nu)_s}{(\Delta E_k / \nu)_p} \bullet W \quad (3)$$

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 (3) 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, favouring 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.

In case of aluminium casting inoculants are introduced in form of master alloy AlTi5B1. This inoculant has Ti:B ratio equals 5:1. This Ti:B atomic ratio, which corresponds to the mass content of about 0.125% Ti to about 0.005% B, assures the greatest degree of structure refinement (Fig. 2). For this titanium and boron ratio bases of type TiB_2 and Al_3Ti are created [4, 5, 7-11, 15, 16]. The type and amount of bases to heterogeneous nucleation of aluminium depend on Ti:B ratio. For example given in paper [10] the possibility of application of master alloy AlTi1.7B1.4, which has Ti:B ratio equals 1.2:1 is presented. This ratio allows to increase in amount of fine phases TiB_2 and AlB_2 along with the Al_3Ti phase decrease.

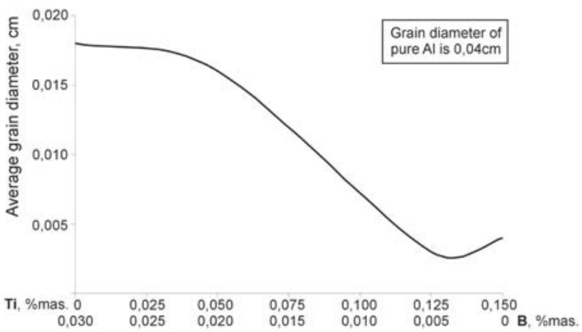

Fig. 2. Influence of Ti and B contents on the average size of Al ingots [16]

Moreover minimum quantities of carbon and nitrogen, which come from metallurgical process of aluminium, create with inoculant the bases in form of titanium carbide TiC and titanium nitride TiN (Fig. 3) [4, 5, 12].

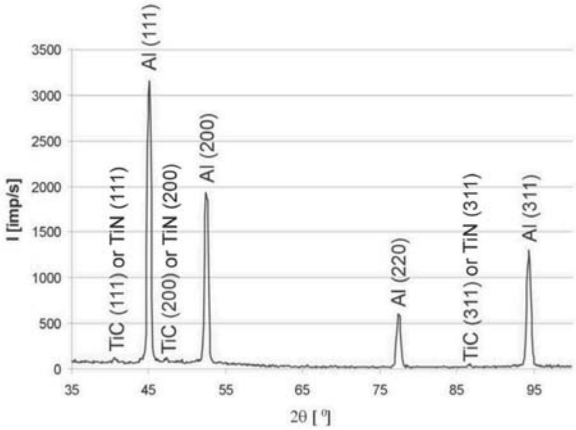

Fig. 3. Result of X-ray diffraction of Al with a purity of 99,5% after inoculation with Ti

a)

b)

c)

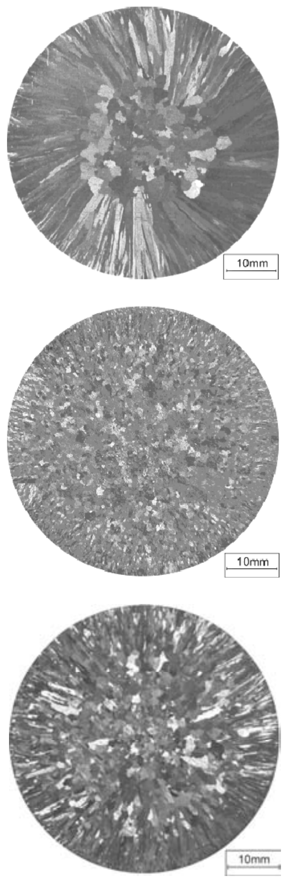

Fig. 4. Macrostructure of ingot of Al with a purity of 99.7%: a – in as-cast condition, b – after inoculation with (Ti+B), c – after inoculation with (Ti+C)

Additionally, because there is a possibility of creation the bases to heterogeneous nucleation of aluminium in form of TiC phase without presence of bases in form of borides, in the practice of casting the inoculation with master alloy AlTi3C0.15 is used [15]. However, on the basis of results of own researches was affirmed that assuming of introducing to Al with a purity of 99.7% the same quantity of Ti i.e. 25 ppm, the result of structure refinement caused by master alloy AlTi3C0.15 is weaker than caused by master alloy AlTi5B1 (Fig. 4).

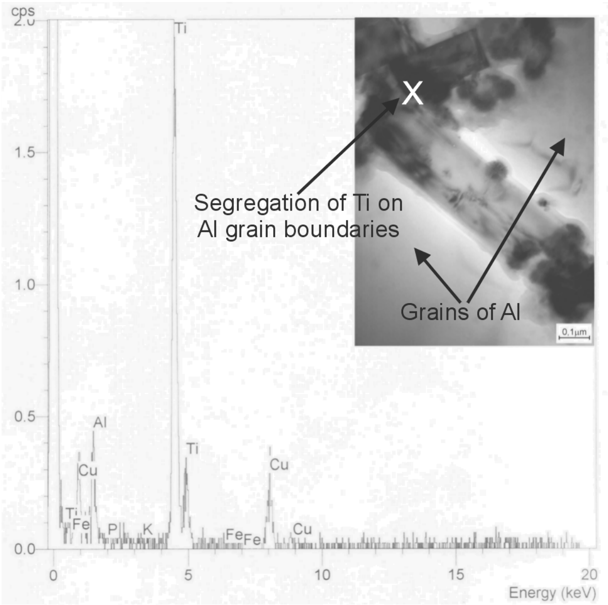

Fig. 5. Segregation of Ti on grain boundaries of Al

However, this undoubtedly effective method of inoculation of primary structure of ingot is limited for pure metals, because inoculants decrease the degree of purity specified in European Standards, and Ti with B introduced as modifying additives are then classified as impurities. Moreover, inoculants, mainly Ti which segregates on grain boundary of Al (Fig. 5) influence negatively on physical properties i.e. electrical conductivity of pure aluminium (Fig. 6).

Moreover the presence of the bases to heterogeneous nucleation in form of hard deformable phases for example titanium borides in structure in aluminium, generate possibility of point cracks formation and in result of this delamination of sheet (foil) during rolling [18].

Therefore important is the other method of inoculation, which consists of influence of electromagnetic field or mechanical vibrations on liquid metal in time of its solidification in mould [4, 5, 19-21].

3. Inoculation of pure Al structure by the electromagnetic field

First research works on the application of stirring of liquid metal at the time of its solidification in order to improve the castings quality were carried out by Russ Electroofen in 1939 and concerned the casting of non-ferrous metals and their alloys. In order to obtain the movement of the liquid metal in the crystallizer in the researches carried out at this period of time and also in the future, a physical factor in the form of a electromagnetic field defined as a system of two fields i.e. an electric and magnetic field was introduced. The mutual relationship between these fields are described by the Maxwell equations. Generated by the induction coil powered by electric current intensity (I_0) electromagnetic field affects the solidifying metal induces a local electromotive force (E_m), whose value depends on the local velocity of the liquid metal (V) and magnetic induction (B) [4]:

$$E_m = \overline{V} \times \overline{B} \quad (4)$$

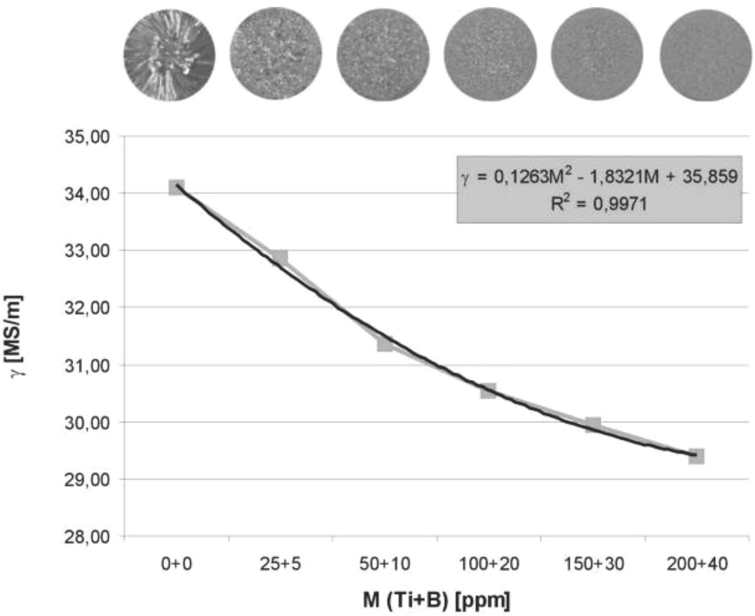

Fig. 6. The influence of quantity of inoculants in form of Ti and B on electrical conductivity γ of Al with a purity of 99.7%

This is a consequence of the intersection of the magnetic field lines with the current guide in form of liquid metal. It also leads to inducing an eddy current of intensity (I) in liquid metal [4]:

$$\bar{I} = \sigma(\bar{V} \times \bar{B}) \tag{5}$$

where:
σ – electrical conductivity proper to the liquid metal.

The influence of the induced current on the magnetic field results in establishing of the Lorenz (magnetohydrodynamic) force (F) [4]:

$$\bar{F} = \bar{I} \times \bar{B} \tag{6}$$

that puts liquid metal in motion e.g. rotary motion in the direction consistent with the direction of rotation of the magnetic field. Strength (F) has a maximum value when the vector (V) and (B) are perpendicular (Fig. 7).

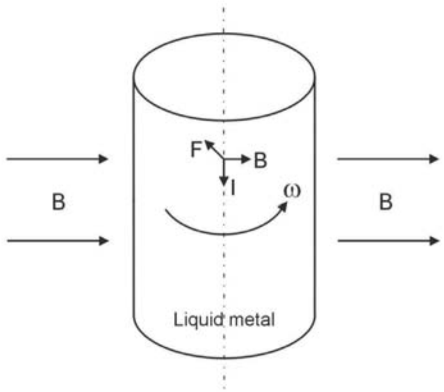

Fig. 7. Scheme of electromagnetic field influence on the liquid metal

In addition, as presented in the papers [5, 21] the rotating velocity of the liquid metal (V) is inversely proportional to the density of the metal (ρ), because with some approximation we can say that:

$$\bar{V} \approx \frac{\bar{F}}{\rho} \text{or} \frac{\bar{B}}{\rho} \tag{7}$$

Forced liquid metal movement influences by 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, 4, 5, 21]:

- tear off of crystals from mould wall, which are transferred into metal bath, where they can convert in equiaxed crystals,
- fragmentation of dendrites by coagulation and melting as a result of influences of temperature fluctuation and breaking as a result of energy of liquid metal movement,
- crystals transport from the free surface to inside the liquid metal,
- crystals from over-cooled outside layer of the bath are transported into liquid metal.

One of the hypotheses regarding the mechanism of dendrites fragmentation caused by the energy of the movement of liquid metal is presented in work [19]. It is based on the assumption of high plasticity of growing dendrites in the liquid metal, which in an initial stage are a single crystal with specified crystallographic orientation (Fig. 8a). The result of liquid metal movement is deformation (bending) of plastic dendrite (Fig. 8b), which causes creation of crystallographic misorientation angle Θ (Fig. 8c). Created high-angle grain boundary (Θ > 20°) has the energy γ_{GZ} much greater than double interfacial energy of solid phase - liquid phase $\gamma_{S\text{-}L}$. In result of unbalancing and satisfying the dependence $\gamma_{GZ} > 2\,\gamma_{S\text{-}L}$ the grain boundary is replaced by a thin layer of liquid metal. This leads to dendrite shear by liquid metal along the former grain boundary (Fig. 8d). Dendrite fragments of suitable size after moving into the metal bath can transform into equiaxed crystals.

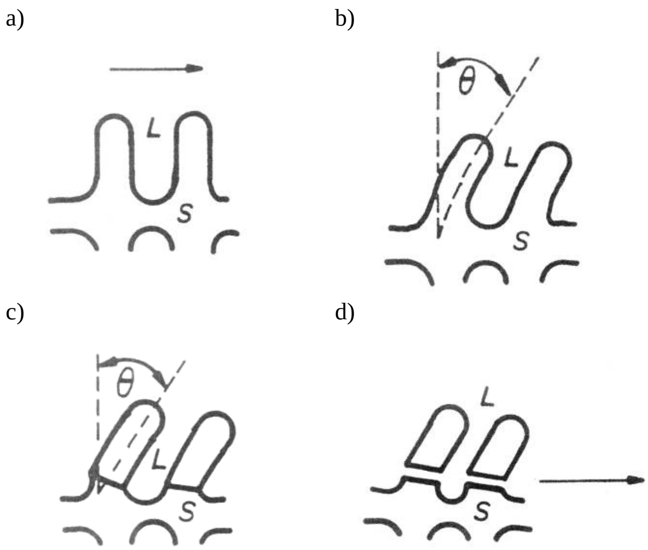

Fig. 8. Schematic model of the grain boundary fragmentation mechanism: a – an undeformed dendrite, b – after bending, c – the reorganization of the lattice bending to give grain boundaries, d – for $\gamma_{GZ} > 2\,\gamma_{S\text{-}L}$ the grain boundaries have been "wetted" by the liquid phase [19]

The influence of electromagnetic field on liquid metal in aim of structure refinement (Fig. 9), axial and zonal porosity elimination and obtaining larger homogeneity of structure, was applied in permanent mould casting and mainly in technologies of continuous and semi-continuous casting [3-5, 19-21].

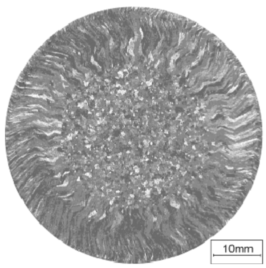

Fig. 9. Macrostructure of ingot of Al with a purity of 99.7% after cast with influence of rotating electromagnetic field with reversion (macrostructure of this Al in as-cast condition is presented in Fig. 4a)

In case of continuous ingots of square and circular transverse section, rotating electromagnetic field induction coils are used. Rotating electromagnetic field forces rotational movement of liquid metal in perpendicular planes to ingot axis (Fig. 10a). Whereas, mainly for flat ingots, longitudinal electromagnetic field induction coils are used, which forced oscillatory movement of liquid metal in parallel planes to ingot axis (Fig. 10b).

a) b)

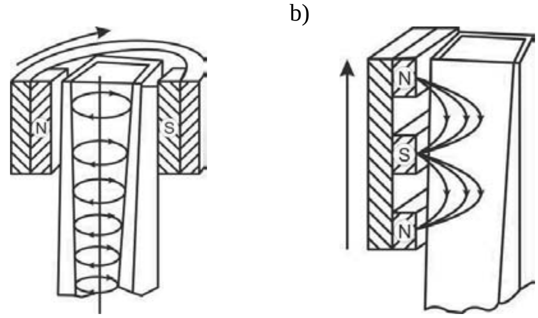

Fig. 10. Scheme of an electromagnetic stirrer (induction coil) forced rotational (a) and oscillatory movement of liquid metal [3]

However in papers [4, 5] was shown that influence of forced movement of liquid metal by use of electromagnetic field to change the structure of pure metals, which solidify with flat crystallization front is insufficient. The effective influence of this forced convection requires a suitable, minimal concentration of additives i.e. alloy additions, inoculants or impurities in casting. Suitable increase of additives concentration causes at specified thermal conditions of solidification, occurs in change of morphology of crystallization front.

However it should be noted that, based on the latest results of author researches was affirmed that in some cases it is possible to obtain a sufficient refinement degree of pure aluminium structure in result of inoculation carried out only with the use of an electromagnetic field. Because it shows a possibility of increasing the force, which creates movement of liquid metal and in result of this the velocity of its rotation in mould, not only by increasing the value of magnetic induction according to the dependences (6) and (7), but also by increasing the frequency of the current supplied to the induction coil (Fig. 11). The effect of refinement of structure of Al with a purity of 99.5% caused by the rotating electromagnetic field produced by the induction coil supplied by current with frequency different from the network i.e. 50 Hz is presented in Table 2. On the basis of macroscopic metallographic researches, which lead to the calculation of the equiaxed crystals zone content on transverse section of ingot (SKR) and average area of macro-grain in this zone (PKR) was affirmed, that application of frequency of supply current f ≤ 50Hz does not guarantee favourable transformation of pure aluminium structure (Fig. 12). Whereas induction coil supplied with frequency of current larger than power network, mainly 100 Hz generates rotating electromagnetic field, which guarantees favourable refinement of structure, also in comparison to this obtained after inoculation with small, acceptable by European Standards amount of Ti and B i.e. 25 and 5 ppm (Table 2 and Figs. 13-34).

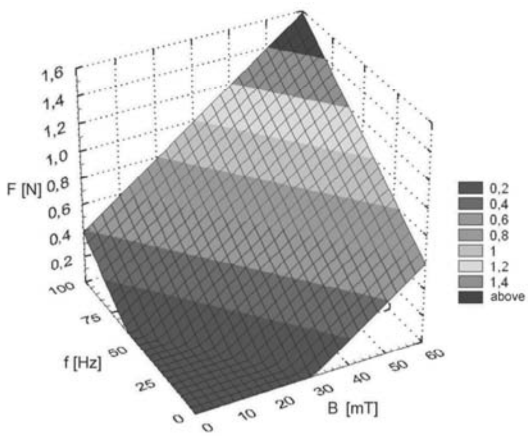

Fig. 11. The influence of magnetic induction (B) and frequency (f) of the current supplied to the induction coil on force value (F), which creates movement of liquid metal

a)

b)

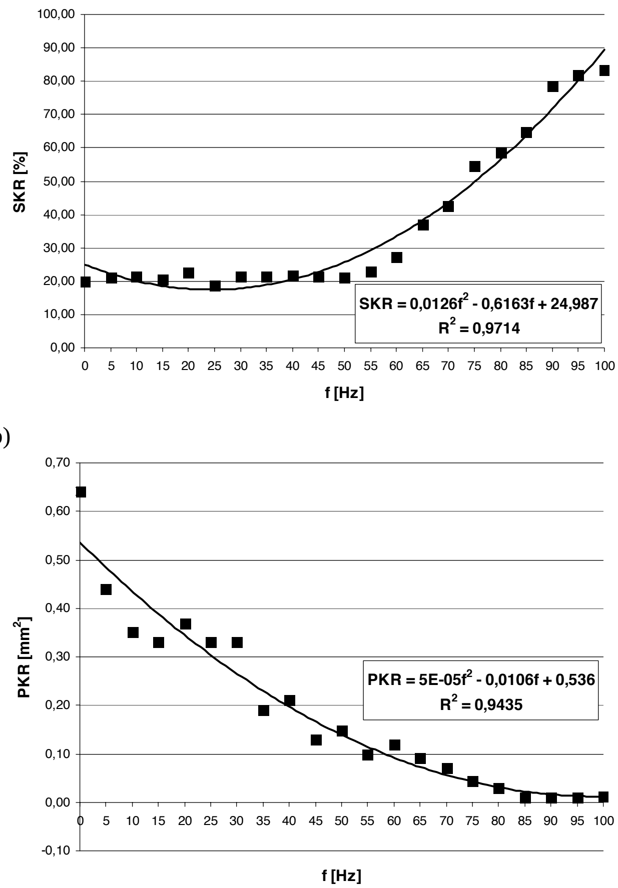

Fig. 12. The influence of current frequency (f) supplied to the induction coil on: a – equiaxed crystals zone content (SKR) on transverse section of pure Al ingot, b – average area of equiaxed crystal (PKR) of pure Al ingot

Table 2.
The influence of rotating electromagnetic field on refinement parameters of structure of Al with a purity of 99.5%

No.	Cast parameters			Refinement parameters*	
	B, mT	f, Hz	(Ti+B), ppm	SKR, %	PKR, mm^2
1	-	-	-	19.94	0.64
2			25+5	80.30	0.42
3	60	5	-	21.01	0.44
4		10		21.36	0.35
5		15		20.66	0.33
6		20		22.63	0.37
7		25		18.90	0.33
8		30		21.42	0.33
9		35		21.44	0.19
10		40		21.68	0.21
11		45		21.46	0.13
12		50		21.21	0.15
13		55		22.87	0.10
14		60		27.22	0.12
15		65		37.05	0.09
16		70		42.53	0.07
17		75		54.63	0.04
18		80		58.56	0.03
19		85		64.70	0.01
20		90		78.67	0.01
21		95		81.78	0.01
22		100		83.36	0.01

* – measurements based on the analysis of macrostructures presented in Figs. 13-34

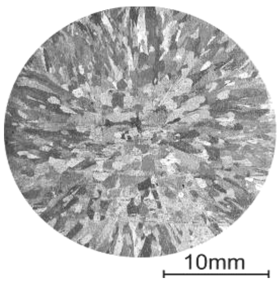

Fig. 13. Macrostructure of Al with a purity of 99.5% in as-cast condition – sample no. 1

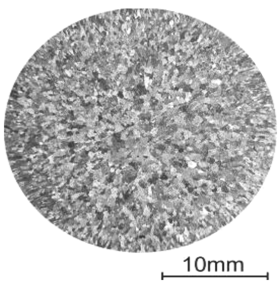

Fig. 14. Macrostructure of Al with a purity of 99.5% after inoculation with (Ti+B) – sample no. 2

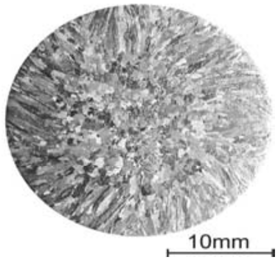

Fig. 15. Macrostructure of Al with a purity of 99.5% after cast with influence of rotating electromagnetic field, f = 5 Hz – sample no. 3

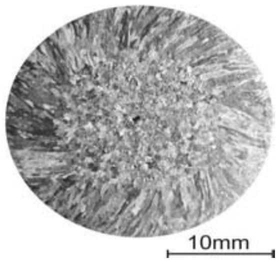

Fig. 16. Macrostructure of Al with a purity of 99.5% after cast with influence of rotating electromagnetic field, f = 10 Hz – sample no. 4

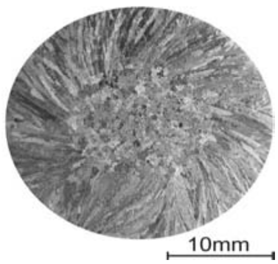

Fig. 17. Macrostructure of Al with a purity of 99.5% after cast with influence of rotating electromagnetic field, f = 15 Hz – sample no. 5

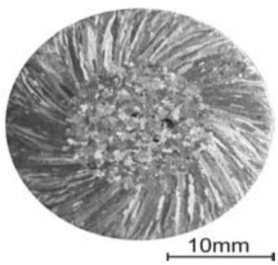

Fig. 18. Macrostructure of Al with a purity of 99.5% after cast with influence of rotating electromagnetic field, f = 20 Hz – sample no. 6

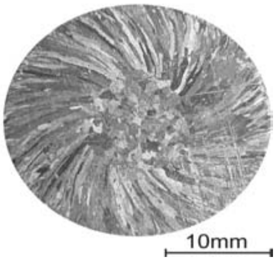

Fig. 19. Macrostructure of Al with a purity of 99.5% after cast with influence of rotating electromagnetic field, f = 25 Hz – sample no. 7

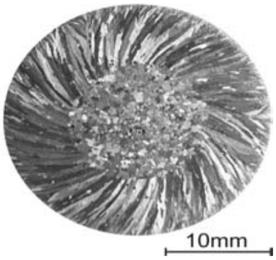

Fig. 20. Macrostructure of Al with a purity of 99.5% after cast with influence of rotating electromagnetic field, f = 30 Hz – sample no. 8

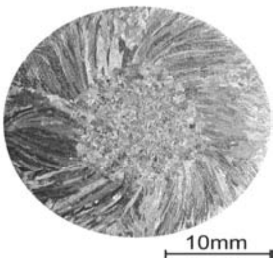

Fig. 21. Macrostructure of Al with a purity of 99.5% after cast with influence of rotating electromagnetic field, f = 35 Hz – sample no. 9

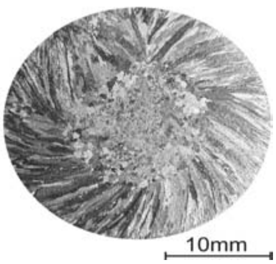

Fig. 22. Macrostructure of Al with a purity of 99.5% after cast with influence of rotating electromagnetic field, f = 40 Hz – sample no. 10

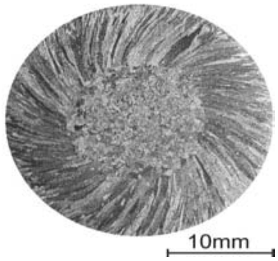

Fig. 23. Macrostructure of Al with a purity of 99.5% after cast with influence of rotating electromagnetic field, f = 45 Hz – sample no. 11

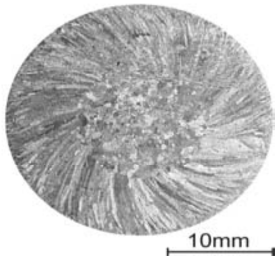

Fig. 24. Macrostructure of Al with a purity of 99.5% after cast with influence of rotating electromagnetic field, f = 50 Hz – sample no. 12

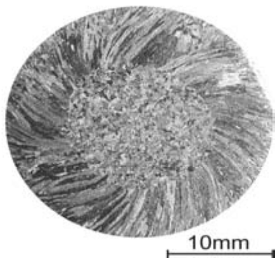

Fig. 25. Macrostructure of Al with a purity of 99.5% after cast with influence of rotating electromagnetic field, f = 55 Hz – sample no. 13

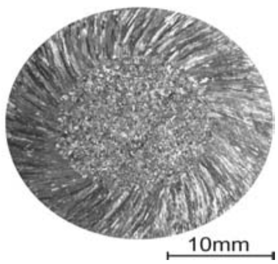

Fig. 26. Macrostructure of Al with a purity of 99.5% after cast with influence of rotating electromagnetic field, f = 60 Hz – sample no. 14

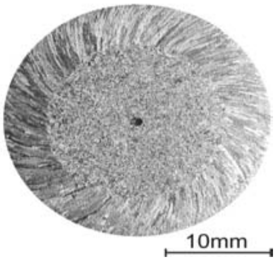

Fig. 27. Macrostructure of Al with a purity of 99.5% after cast with influence of rotating electromagnetic field, f = 65 Hz – sample no. 15

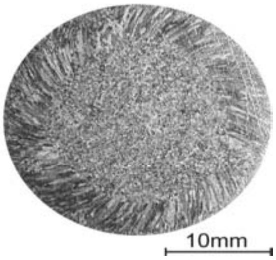

Fig. 28. Macrostructure of Al with a purity of 99.5% after cast with influence of rotating electromagnetic field, f = 70 Hz – sample no. 16

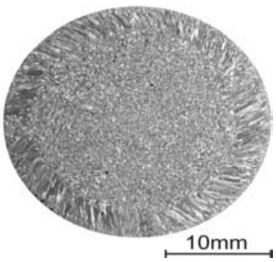

Fig. 29. Macrostructure of Al with a purity of 99.5% after cast with influence of rotating electromagnetic field, f = 75 Hz – sample no. 17

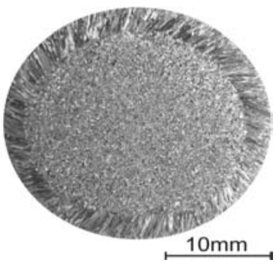

Fig. 30. Macrostructure of Al with a purity of 99.5% after cast with influence of rotating electromagnetic field, f = 80 Hz – sample no. 18

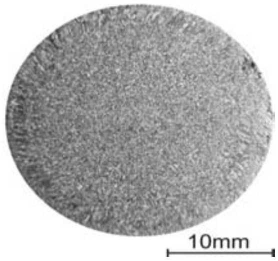

Fig. 31. Macrostructure of Al with a purity of 99.5% after cast with influence of rotating electromagnetic field, f = 85 Hz – sample no. 19

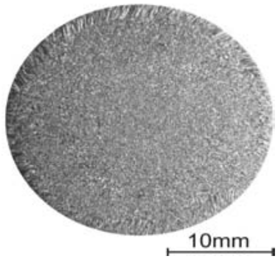

Fig. 32. Macrostructure of Al with a purity of 99.5% after cast with influence of rotating electromagnetic field, f = 90 Hz – sample no. 20

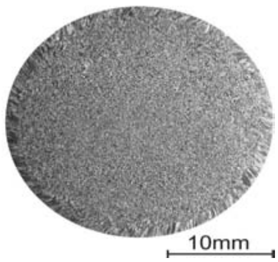

Fig. 33. Macrostructure of Al with a purity of 99.5% after cast with influence of rotating electromagnetic field, f = 95 Hz – sample no. 21

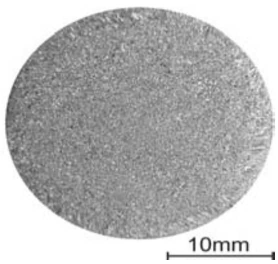

Fig. 34. Macrostructure of Al with a purity of 99.5% after cast with influence of rotating electromagnetic field, f = 100 Hz – sample no. 22

4. Summary

On the basis of conducted analysis of the literature and results of author researches it was affirmed, that the rotating electromagnetic field generated by induction coil supplied by current with frequency larger than power network, influences liquid metal in time of its solidification in mould, guarantees refinement of structure of pure Al without necessity of application of inoculants such a Ti and B. This method of inoculation is important, because Ti and B decrease the degree of purity and electrical conductivity of pure aluminium. Moreover Ti and B are reason of point cracks formation during rolling of ingots.

Presented method of inoculation by use of electromagnetic field is possible to apply in conditions of continuous casting because it allows producing of ingots from aluminium of approx. 99.5% purity with structure without columnar crystals, which are unfavourable from point of view of usable properties.

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