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(54) METHOD FOR PRODUCING ADDITIONS AND HARDENERS FOR PRODUCING ALLOYS

(57) The invention relates to metallurgy, in particular to methods for producing alloying additions used in the production of different alloys. The method consists in melting and creating a process-required superheating above the liquidus temperature of a base metal and production ingredients, agitating the melt, filtering the melt and crystallising the superheated melt in the centrifuge force fields at different gravitation factors ranging from 20 to 500 so as to produce casts in the form of substitutional-interstitial solid solutions. The processing of the cast by a centrifuge force field is carried out until the temperature of the cooling cast achieves the temperature of the crystallisation termination process in regular conditions. The invention makes it possible to modify the crystallisation conditions of additions in external force fields by speeding up processes in melts at the stage of the crystalline structure formation.

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Description

Pertinent art

[0001] The invention relates to metallurgical production, in particular to methods for producing different additions to alloys, modifying their operating properties, and including methods for producing alloys.

Antecedent state of the art

[0002] Additions and hardeners are intermediate alloys, usually consisting of a base component of production alloy with one or several alloying components, which quantity essentially exceeds those in the base melt. They should meet the following requirements: to have the melting temperature, close to melting temperature of the base metal; to ensure chemical composition homogeneity; to comprise the most possible quantity of the alloying element; to have sufficient brittleness for grinding convenience. The hardeners are preferred for using at introduction into the melt of the following:

- high-melting additions, for which dissolution a substantial melt superheating is required;
- volatile components, that are per se in gaseous state at the melt temperature;
- chemically active additions, that are per se in the air and might interact with oxygen and nitrogen;
- expensive additions, if they cannot be produced in their pure form, and when the production of master alloys is already developed, they are available and relatively cheap;
- super-fine additions (0,02 - 0,01%), that are almost impossible for reliable introduction at per se;
- alloying elements, with a density above or below the density of the base metal; in first case they are sinking, while in second case they are floating over the melt surface; in any case it is difficult to monitor the solution of such additions.

[0003] The weight of hardeners at charge fluctuates from 30 to 70%, depending on the complexity of the alloy composition. Chemical composition, defects and mechanical properties of the alloys are largely depend on the quality of the applied hardeners.

[0004] The main alloys quality criteria is a correspondence with a chemical composition, absence of oxide and other nonmetallic inclusions, uniform distribution of the hardener's components across its total volume.

[0005] Existing methods for producing additions are based on use of different physical principles, allowing solution of two major problems.

[0006] The first option of use of additions is based on modification (of first and second type) of base melt with the aim of significant structure grinding. In this regard, during the preparation of additions-modifiers (hardeners), depending on the type of the modifier itself, different preparation methods should ensure the following:

- to block growth of large crystals in hardeners without changing the chemical composition of the matrix by use of the surface-active substances in the form of the adsorbent;
- to ensure their uniform distribution in hardener, which would lead to good grinding of its structure, by use of the refractory materials. In this option it is also necessary to grind intermetallics, which appear, for example, as a result of formation of relatively stable relations between superdispersed particles of various oxides.

[0007] As per laws of thermodynamics, at re-melting of hardeners of the first type during modification of operating melts at a relatively low superheat, takes place the following: the destruction of grain interconnections, the extraction of modifier-adsorbent and further, its withdrawal from the grain surface, the disintegration of the grain. The magnitude of this superheat is very small (20-40 °C), so that even short-term superheating leads to the inability to use the ultradisperse ligature grains as a base. Practically, the user of hardener of this type is in most cases uses as a modifier the base alloying element, which at best is uniformly distributed across the periphery of the hardener matrix.

[0008] Re-melting of hardeners of the second type that are as a rule represented by a mechanical mixture of base metal and superfine refractory oxide, carbide, nitride particles, which are centers of its grains formation, performs the necessary task of modification only after essential superheating (over 50-70 °C), which lead to the grain disintegration. Only these conditions ensure the most effective modification by the alloying element. Such hardeners should be homogeneous in terms of distribution of the alloying element.

[0009] The second option of use of additions is generally consists in bringing with their help to a desired chemical composition of any alloy. In this case, additions should have a second component, which is maximum-dissolved in the base matrix, including cases of supersaturated solutions.

[0010] During the preparation of such additions, the manufacturers are trying to grind their structure in general and the eutectic non-grain part as well. At that the supplement in general contains, for example, 15% of magnesium in aluminum base, whereas the grain itself could contain no more than 7% of magnesium. At re-melting of additions of this

type, which represent at best interstitial solutions with a relatively large volume of eutectics, the original problem arises when it is necessary to introduce a second (next) component(s) into the base operating melt by means of complete dissolution of eutectics and equalization of this second component between the addition's grain and operating melt by diffusion processes.

[0011] The natural outcome of use of such additions, due to unresolved problems of solubility is the obtainment of any alloys with given chemical composition, having only part of the possible service properties.

[0012] Currently, a method for producing hardeners for aluminum alloys is used for producing additions. This method is based on the refinement of intermetallic particles in hardener and enhancing their stability during melt preparation due to the formation of stable links between synthetic ultradisperse oxide particles (SU No 1650746, C22C1/03, publ. 23.05.1991).

[0013] These methods are fairly simple for implementation, their use leads to the castings structure refinement, however, during the preparation of alloys due to the physical impossibility of dissolving of the addition in its matrix, the alloys are virtually repeat (at best) the addition structure and do not have the possible service properties.

[0014] It is known a method for producing hardeners by means of electromagnetic field exposure on their melt (RU No 2210611, C22C1/03, publ. 20.08.2003), which consists in melting the base metal and production ingredients, agitating, filtering and crystallizing the melt with its volumetric cooling at different speeds for grinding intermetallic particles. At that in order to increase the homogeneity of additions by reducing the proportion of primary intermetallics, the crystallization is conducted at applied external constant magnetic field.

[0015] However, even such actions lead only to refinement of addition structure without qualitative change of solid solution. This is due to the fact that in described cases the operating mechanism, in terms of dissolution of any component in matrix (diffusion), is a difference of substance concentration (Fick law); namely there is no mechanism of speeding up of diffusion processes.

[0016] It is known a method for producing alloying additions for the producing alloys, which consists in melting the base metal and soluble ingredients and crystallizing the melt at its volumetric cooling in order to grind intermetallic particles. The method is different in that for producing alloying additions in the form of substitutional-interstitial solid solution, the melt is crystallized in the centrifuge force field at gravitation coefficient ranging from 20 to 240 during time, that equal to the ratio:

$$t=m_3/aK_g\ ,$$

where a - coefficient, defined separately as a value for each pair of metal-Ingredient, based on the thermodynamic characteristics of the crystallizer and velocity of heat processes in the last; Kg - gravitation coefficient; m_3 - relative weight of soluble components in the alloying addition (RU No 2296175, C22C1/02, publ. 27.03.2007).

[0017] However, additional studies have shown that the declared by formula of invention parameters do not allow to obtain the stated results.

[0018] Ignoring the process-required superheating of melt before its pouring into the mold, that is necessary for speeding up diffusion and formation of grain size during crystallization at the temperature above the liquidus, leads to the breakdown of processes and its transition to the category of ordinary centrifugal casting.

[0019] Under-dissolution of components and destruction of the expected ingot structure occurs.

[0020] Moreover, ignoring the necessity of continuation of ingot treatment by the centrifuge force field until the cooling ingot would have the temperature of termination of all processes in general conditions, finally eliminates the results of treatment, since after premature withdrawal of the force field, the usual processes begin.

[0021] The data in Tables 1 and 2 is not systemic in isolation from the fact of processes flowing above the liquidus.

[0022] The proposed formula for determining the processing-required time (:$t=m_3/aKg$, where a - coefficient, Kg - gravitation coefficient, m_3 - the relative weight of soluble ingredients in the addition) is not theoretically consistent, because of indifference of the gravitation forces impact upon the melt weight and is not practically logical, because of the substitution with coefficient "a" of actual dependences "t" on the chemical composition of the melt, cooling rate, module of the temperature above the liquidus and gravitation coefficient.

[0023] In terms of theoretical assumptions, the transformation of formula (21), multiplication of the right side by the atomic weight "Mo" are not correct due to the indifference of the processes in the centrifuge force fields from the weight of material processed.

[0024] Avowal the use of the same gravitation coefficient at all treatment stages is acceptable for certain groups of materials. But for most alloys, it is optimal to change the value of gravitation coefficient Kg with respect to temperature module and phase state of the treated alloy. Especially, this dependence distinctly appears in the range of temperatures of the melt processed from the initial superheating temperature to liquidus temperature.

[0025] This known decision was made as the prototype for the claimed subject, taking into account the disadvantages

identified.

Disclosure of invention

[0026] The present invention is aimed at solving the technical problem of changing the conditions of crystallization of additions against the use of new physical phenomena of speeding up diffusion in melts at the stage of crystalline structure formation in the external force fields. The achieved technical result consists in producing additions that are more heat resistant and provide the ideal solubility of its matrix with the production ingredients in the base alloy melt, which leads to a drastic (25-30%) increase of its service properties.

[0027] The stated technical result is achieved by method for producing additions, including hardeners, for producing different alloys. This method consists in melting and creating a process-required superheating above the liquidus of the base metal and production ingredients, agitating the melt, filtering the melt and crystallizing the superheated melt in the centrifuge force fields at different gravitation coefficients (Kg) ranging from 20 to 500 so as to produce casts in the form of substitutional-interstitial solid solutions. At that the value of technological melt superheating above the liquidus at the moment of its pouring into the lined ingot of the crystallizer together with its thermodynamic characteristics, determined by the volumetric cooling of superheated melt at a speed ranging from 0,5 to 10 ° /sec, are providing the lifetime of the liquid phase of the melt sufficient for ensuring two major processes:

a) melt treatment by the centrifuge force field at different gravitation coefficients (Kg) ranging from 20 to 500. At that the treatment time at the given gravitation coefficient depends on the chemical composition of the melt, temperature module and speed of homogeneous volumetric cooling of the melt in the ingot. This is identified experimentally and doesn't depend on the weight of the treated melt. For majority of ligatures this time doesn't exceed 3 minutes.
b) crystallization in the centrifuge force field at gravitation coefficients ranging from 20 to 500 until the cooling in the ingot melt will reach the temperature of the onset of crystallization in general conditions.

[0028] The movement rate of the crystallization front from the ingot outwall to the centre is determined by the chosen regime of crystallization.

[0029] At constant Kg the front is moving with the rate of fall of temperature of the cooling melt during time, necessary for the front coming through the cast radius.

[0030] The process of cast treatment by a centrifuge force field continues till the cooling cast will reach the temperature of termination of all crystallization processes in general conditions.

Description of drawings

[0031] For better understanding of the invention, the following specific examples of its implementation with references to the supplemented drawings are presented below. At that:

Fig. 1 - scheme of the potential relief in the surroundings of any atom of the melt without the external force field influence;
Fig. 2 - scheme of diffusion;
Fig. 3 - scheme of the potential relief in the surroundings of any atom of the melt with the external force field influence.

The best option of the invention implementation

[0032] The present application describes the method for producing additions and hardeners for producing alloys. This method consists in melting of the base metal and soluble ingredients and crystallization of the melt at its cooling. At that for producing alloying additions of the substitution-interstitial solid solution type, the melt is crystallized in the force field of centrifuge. The crystallization of a slightly superheated composition with the given chemical content is conducted in the force field at the corresponding with the given melt gravitational coefficient between 20 and 500, in a lined (heated) rotor ingot of the centrifuge in three stages:

at first stage the solution is treated at the homogenous volumetric cooling of the melt, poured into the ingot at a speed 0,5-10°/sec, with its superheating above the liquidus to the time of existence of liquid phase of the melt, sufficient for dissolution of the ingredients in the melt during the time lag;
$t=m_3/aKg$, where a - coefficient, Kg - gravitation coefficient, m_3 - relative weight of soluble components in the addition,
at the second stage the cooling solution is treated in a centrifuge force field at the selected gravitation coefficient so as the crystallization takes place in a centrifuge force field until the melt, cooling in the ingot of the crystallizer, would reach the temperature of the onset of crystallization processes in general conditions,

at the third stage a cooling cast is treated in a centrifuge force field at the selected gravitation coefficient until the cast, cooling in the ingot of the crystallizer, would reach the temperature of the termination of crystallization processes in general conditions.

[0033] Production of the substitutional-interstitial solid solutions without eutectic discharge requires additional energy, which will lead to a distortion of the initial potential reliefs and consequently to a creation of the conditions for speeding up diffusion of operating ingredients into the base metal.

[0034] External force field, such as the centrifuge gravitation field, is easily manageable and indifferent to the type of material that allows for producing all types of additions, including with the use of nonmetallic materials.

[0035] Degree of distortion of the potential reliefs is identical to the creation of corresponding supercooling in the melts, which, according to the Tammann dependencies, at their specific values can lead to the formation of any size crystal structure grains of additions.

[0036] Additions of the proposed type are more resistant to heat and provide the ideal solubility of its matrix with the production ingredients in the base alloy melt, which leads to a drastic (25-30%) increase of its service properties.

[0037] Method for producing additions, including hardeners, for producing different alloys consists in melting and creating a process-required superheating above the liquidus of the base metal and production ingredients, agitating the melt, filtering the melt and crystallizing the superheated melt in the centrifuge force fields at different gravitation coefficients (Kg) ranging from 20 to 500 so as to produce casts in the form of substitutional-interstitial solid solutions.

[0038] At that the value of technological melt superheating above the liquidus at the moment of its pouring into the lined ingot of the crystallizer together with its thermodynamic characteristics, determined by the volumetric cooling of superheated melt at a speed ranging from 0,5 to 5 °/sec, are providing the lifetime of the liquid phase of the melt sufficient for ensure two major processes:

a) melt treatment by the centrifuge force field at different gravitation coefficients (Kg) ranging from 20 to 500. At that the treatment time at the given gravitation coefficient depends on the chemical composition of the melt, temperature module and speed of homogeneous volumetric cooling of the melt in the ingot. This is identified experimentally and doesn't depend on the weight of the treated melt. For majority of ligatures this time doesn't exceed 4 minutes.
b) crystallization in the centrifuge force field at gravitation coefficients ranging from 20 to 500 until the cooling in the ingot melt will reach the temperature of the onset of crystallization in general conditions.

[0039] The movement rate of the crystallization front from the ingot outwall to the centre is determined by the chosen regime of crystallization.

[0040] At constant Kg the front is moving with the rate of fall of temperature of the cooling melt during time necessary for the front coming through the cast radius.

[0041] The process of cast treatment by a centrifuge force field continues till the cooling cast will reach the temperature of termination of all crystallization processes in general conditions.

[0042] The technical result consists in producing additions, which are more resistant to superheating and providing ideal dissolution of the whole matrix with the operational ingredients in the melt of base alloy. This leads to enhancement of the operating properties by 20 - 30%.

[0043] The declared method is based on use of principally new physic phenomena on formation of diffusion processes in melts at the stage of cast crystallization in the external force field.

[0044] If it is necessary to produce addition in the form of substitutional solid solution, a required degree of dissolution of the element A in the element B should be provided, which corresponds to the diffusion of A in B, i.e. jump of the element A atom to the crosspoint of the crystal lattice of the element B.

[0045] According to the Frenkel Y. theory, the amount of atoms n, transferred per time unit and per volume unit from the melt to the crystal lattice at any initial concentrations of the admixture A in the melt and solid body equals to the following:

$$n = n_s P f \exp\left[-\frac{\bar{U}_A + \Delta A_i}{KT}\right] \qquad (1)$$

where n_s - amount of atoms of the admixture A in the considered area;
P - probability of atom jump in the required direction;
f - atom vibration frequency around equilibrium position;
$\bar{U}_A$ - average energy of activation of the atom B jump to the crosspoint of crystal lattice of the same element from the melt;
ΔA_i - additional energy of activation, required for the analogous jump of atom A (this is due to the distinction between atoms A and B).

[0046] In natural conditions at the absence of the external force fields, the potential relief in the surroundings of any atom of the melt is symmetric (fig.1), which leads to the absence of the leading force of diffusion through PQ (fig.2). The cause of atoms transition from the environment 2 to 1 in this case is only possible at deficiency of concentration equality, i.e. in case of self-diffusion.

[0047] Availability of the external force field F with the potential energy E would lead to the distortion of the initial potential relief (fig.3). Heights of the potential barriers in both directions of the possible atom A jump are correspondingly equal to the following:

$$U_1 = \bar{U}_A - F\delta/2 + \Delta A_i$$
$$U_2 = \bar{U}_A + F\delta/2 + \Delta A_i \qquad (2)$$

where δ - sequence of the crystal lattice.

[0048] General asymmetry of the potential relief equals to:

$$\Delta U = F\delta \qquad (3)$$

[0049] Availability of ΔU leads to the occurrence of the asymmetric atomic flow n_2, which can be determined from the expression:

$$n_2 = s\bar{V}c_i \qquad (4)$$

where $\bar{V}$ - average speed of the A-type atoms;
S - solid phase square;
Ci - atomic concentration of the admixture A.

[0050] For estimation purposes the following calculations will be done. Time of atom being in the settle position in the i-th hole equals to:

$$\tau = \tau_0 \exp[\bar{U}_A + \Delta A_i / KT] \qquad (5)$$

where τ_0=1/f - period of atom fluctuation.

[0051] In this case the probability of atom moving in the direction X equals to:

$$P_x^x = P_0^x dt / \tau \qquad (6)$$

where P_0^x _ probability of jump in random direction (usually it is accepted as 1/6th) equals to:

$$P_0^x = \frac{1}{6} U_2 / U_1 \qquad (7)$$

[0052] Expression (6) with a glance of (7) will take on form:

$$P^x = \frac{dt}{6\tau^x} U_2 / U_1 \qquad (8)$$

[0053] The probability of admixture A atom jump in the direction (+X) with a glance of expressions (2) and (5) equals to:

$$\frac{dt}{6\tau^x} U_2 / U_1 = U_2 / U_1 \frac{dt}{6\tau^0} \exp[-U_2 / KT] \qquad (9)$$

in the direction (-X), equals to:

$$\frac{dt}{6\tau^{-x}} U_1 / U_2 = U_1 / U_2 \frac{dt}{6\tau^0} \exp[-U_2 / KT] \qquad (10)$$

[0054] The probability of admixture A atom moving over time dt can be derived as the product of $\delta$and difference of probability data:

$$\delta \frac{dt}{6\tau^0} (U_2 / U_1 \exp[-U_1 / KT] - U_1 / U_2 \exp[-U_2 / KT]) \qquad (11)$$

[0055] Hereof the velocity $\overline{V}$ of travel of admixture A atoms equals to:

$$\overline{V} = \frac{\delta}{6\tau^0} \exp[-\frac{\overline{U}_A + \Delta A_i}{KT}] (\frac{U_2}{U_1} \exp[\frac{F\delta}{2KT}] - \frac{U_1}{U_2} \exp[-\frac{F\delta}{2KT}]) \qquad (12)$$

[0056] Substituting the last expression in (4), we obtain:

$$n = \frac{sc_i\delta}{6\tau^0} \exp[-\frac{\overline{U}_A + \Delta A_i}{KT}] (\frac{U_2}{U_1} \exp[\frac{F\delta}{2KT}] - \frac{U_1}{U_2} \exp[-\frac{F\delta}{2KT}]) \qquad (13)$$

[0057] Using a series expansion of exponents:

$$\exp[\frac{F\delta}{2KT} = 1 + \frac{F\delta}{2KT} + \ldots\ldots \qquad (14)$$
$$\exp[-\frac{F\delta}{2KT} = 1 - \frac{F\delta}{2KT} + \ldots\ldots$$

we obtain:

$$n = \frac{sc_i\delta}{6\tau^0} [\frac{F\delta}{2KT} \frac{U_1^2 + U_2^2}{U_1 U_2} + \frac{U_2^2 - U_1^2}{U_1 U_2}] \exp[-\frac{\overline{U}_A + \Delta A_i}{KT}] \qquad (15)$$

[0058] It can be assumed that during crystallization at presence of atoms A and B, the construction of combined crystalline lattice will be derived as follows:

$$P_i = \frac{\bar{U}_A - \Delta A_i}{\bar{U}_A} = 1 - \frac{|\bar{U}_A|}{\bar{U}_A} \qquad (16)$$

[0059] That is, if atom A of admixture has $\Delta A_i=0$, (identical to the atomic parameters of matrix B), the probability of construction of addition in the form of substitutional solid solution equals to 1.

[0060] Taking into account (16), the expression (15) will take on form:

$$n = \frac{sc_i\delta}{6} P_i \left[\frac{F\delta}{2KT}\frac{U_1^2+U_2^2}{U_1U_2} + \frac{U_2^2-U_1^2}{U_1U_2}\right] \exp\left[-\frac{\bar{U}_A + \Delta A_i}{KT}\right] \qquad (17)$$

[0061] Total atomic flow of diffusion equals to:

$$n = \frac{1}{6\tau^0}\left(sc_iP_i\delta\left[\frac{F\delta}{2KT}\frac{U_1^2+U_2^2}{U_1U_2} + \frac{U_2^2-U_1^2}{U_1U_2}\right] - n_s\right) \exp\left[-\frac{\bar{U}_A + \Delta A_i}{KT}\right] \qquad (18)$$

[0062] This expression can be simplified (by reducing n_D in 1-6 times), if it is allowed for $V_1=V_2$:

$$n_D = \frac{1}{6\tau^0}\left[\frac{sc_iF\delta}{KT}P_i - n_s\right] \exp\left[-\frac{\bar{U}_A + \Delta A_i}{KT}\right] \qquad (19)$$

[0063] Flow differential n_D depends on the total activation energy $(\bar{U}_A + \Delta A_i)$, which can be taken as equal to the activation energy of solidification or melting. The expression (19) describes a total flow of atoms A and B, having activation energies from $\bar{U}_A$ to $\bar{U}_A + \Delta A_i$. This implyies that if atoms of A and B type, with the corresponding sizes and activation energies of solidification and melting simultaneously, exist in the melt - the consequence of crystallization in the force field is the creation of substitutional solid solution. At that, if $\Delta A_i=0$, with the probability P there will be obtained a solid solution with 50% of element A, since action of force F is indifferent to the type of atom.

[0064] If the concentration of admixture C_i(A)d in the melt exceeds 0,5 at $\Delta A_i=0$, then in this case a solid phase with the dominant A-type atoms will be obtained.

[0065] Undoubtedly, if atoms A essentially exceeds in size atoms B of dissolvent (>15-20%), then the stable existence of the substitutional solid solution is impossible, and interstitial solid solutions with the minimal amount of intermediate phases are formed.

[0066] The situation changes radically, if the concentration of large atoms A is more that 0,5 - at that the melt of atoms A becomes a dissolvent with respect to atoms B. In this case atoms B move to the crosspoints of the crystalline lattice, made of atoms A, and the substitutional solid solution is formed.

[0067] For evaluation of efficiency of the addition preparation in the force field, the following expression is derived:

$$Z = \frac{n_D}{n_i} = \frac{sc_i\delta^2P_img}{KTn_3}K_g - 1 \qquad (20)$$

where m - melt weight, gr;
g - free fall acceleration;
Kg - centrifuge gravitation coefficient.

[0068] The expression (20) is given for the situations of additions production in the centrfuges.

[0069] Time t, necessary for completion of diffusion, is of interest:

$$t = n_з / n_д \qquad (21)$$

where n_d - given amount of diffuse atoms of admixture.

[0070] Numerical analysis of the expression (20) shows, that for the hardener Al15Mg, the efficiency of production of additions by means of the proposed method increases tenfold at the gravitation coefficient beginning from 100-110. For the hardener A120Ni the optimal gravitation coefficient is 140-150.

[0071] Lifetime requirement of the melt Al15Mg at chosen gravitation coefficient 100-110 is about 1,5-2 minutes. Reduction of this time leads to incomplete dissolution of magnesium in aluminium. For the hardener A120Ni this time is 3,5-4 minutes.

[0072] Method for producing additions, including hardeners, for producing different alloys as per present method:

a) differs in that treatment and crystallization of the overheated melt with the given chemical composition are conducted in the lined (heated) ingot of the centrifuge rotor in the force field at the gravitation coefficients ranging from 20 to 500,

b) differs in that the lining (heating) of the centrifuge rotor ingot allows for homogenous volumetric cooling of the poured melt at a speed 0,5 - 5°/sec,

c) differs in that before melt pouring into the ingot, a process-required superheating above the liquidus is created. At that the value of technological melt superheating above the liquidus together with its thermodynamic characteristics of the ingot, are providing the lifetime of the liquid phase of the melt sufficient for ensuring all treatment and crystallization processes in the centrifuge force field until the cooling in the crystallizer ingot melt would reach the temperature of the onset of crystallization in general conditions.

d) differs in that treatment and crystallization of the superheated melt, poured into the ingot, is carried out in three stages:

1 stage - melt treatment by the centrifuge force field at different gravitation coefficients (Kg) ranging from 20 to 500. At that the treatment time at the given gravitation coefficient depends on the chemical composition of the melt, temperature module and speed of homogeneous volumetric cooling of the melt in the ingot. This is identified experimentally and doesn't depend on the weight of the treated melt. For majority of ligatures this time doesn't exceed 4 minutes.

2 stage - crystallization in the centrifuge force field at gravitation coefficients ranging from 20 to 500 until the cooling in the centrifuge ingot melt will reach the temperature of the onset of crystallization in general conditions,

3 stage - cast treatment by a centrifuge force field at the gravitation ranging from 20 to 500, until the cooling cast would reach the temperature of the termination of crystallization processes in general conditions.

e) differs in that the desired centrifuge gravitation coefficient can be the same (average) at all three stages, or can at every stage there can be set an optimal factor, ranging from 20 to 500.

[0073] The laboratory machine, implementing the proposed invention, corresponds to the centrifuge with the vertical axis, at which a spinning rotor with the ingot is fixed. The rotor is driven by an electrical engine with a variable speed. The preset speed of the centrifuge rotor is stabilized by a special electronic stabilization system of the given rotations. The desired thermodynamic characteristics of the crystallizer ingot, providing the cooling rate of not higher than 5 °/sec, are supported by construction of the ingot lining and preliminary (before the melt poured into the ingot) heating of the inner side of the ingot by the flame of gas burner up to 200-250°C. Rotary bed is made of 5 mm thick structural steel and consists of lower fixed part and upper removable cover. The interior of the fixed part and cover contain the 25 mm thick lining, formed from a mixture of chamotte crumb - main filler, refractory clay - cluster, and graphite - an agent that resists the cracking of the lining, in proportion of 7/3/1, that gives the ingot the required thermodynamic characteristics, as well as 5mm of chemically neutral graphite protect the lining from heat stroke at melt poured into the ingot. Thermodynamic characteristics of the rotor ingot of laboratory centrifuge crystallizer provide the average cooling rate of 2-3 degrees per second at pre-heating of the lining above 200 °C.

[0074] Preliminary prepared melt of 15 % magnesium Mg95 in aluminium A99 was superheated up to 950 °C. in the induction furnace and poured into the pre-heated by the flame of gas burner rotor ingot of the centrifuge crystallizer, taking into account the filling of the ingot with a 25% radius. Rotor speed was set between 1000-1600 rpm, which with the radius of 130 mm allowed for the desired gravitation coefficient. Because of the imperfections of the laboratory crystallizer control system, the gravitation coefficient was set the same for the entire treatment and crystallization. The crystallizer was stopped and the cast ring was taken out after 5 minutes of the rotor spinning with the melt - the experimentally identified time, sufficient for reducing the temperature of the hardened cast below 400 °C.

[0075] Metallographic tests of the obtained eutectic dispersion of casts section has showed a high efficiency of the proposed method, in particular a cast of the hardener Al15Mg, produced as per proposed invention technology, at its main properties exceeds a traditionally produced cast in dozen times. The similar results are shown at the experimental production of the hardeners Al10Mn, Al10Si, Al3Ti, Al20Ni by means of the proposed technology.

Chem.comp.	T (°C)	Kg	t (sec)	e (mkm)
Al15Mg	820	210	130	7 - 7,4
Al10Mn	900	200	177	5,1-5,4
Al10Si	1000	210	226	12 - 12,7
Al20Ni	1050	280	243	6,4 - 6,9
Al3Ti	910	180	197	5,2 -5,4

where: T - temperature of the superheated melt at its pouring into the crystallizer ingot, Kg - average gravitation coefficient, t - total process time, e - eutectic dispersion in the cast.

Industrial application

[0076] The present invention might be applied at the production of any additions of both metallic and non-metallic groups of materials, including salt and any crystallizing and polymerizing materials. The most effective application of the invention is for producing additions and hardeners.

Claims

1. Method for producing additions and hardeners for producing alloys, which consists in melting of the base metal and soluble ingredients and crystallizing at the volumetric cooling of the melt. At that for producing alloying hardeners in the form of substitutional-interstitial solid solution, the melt is crystallized in the centrifuge force field, **characterized in that**
   crystallization of the slightly overheated melt with the given chemical composition is carried out at the corresponding to the given melt gravitation coefficient ranging from 20 to 500, in the lined (heated) ingot of the centrifuge rotor in three stages:

   at first stage the solution is treated at the homogenous volumetric cooling of the poured into the ingot melt at a speed 0,5 - 5°/sec, with its superheating above the liquidus to the time of existence of liquid phase of the melt, sufficient for dissolution of the ingredients in the melt during the time lag;
   $t=m_3/aKg$, where a - coefficient, Kg - gravitation coefficient, m_3 - relative weight of soluble components in the addition,
   at the second stage the cooling solution is treated in a centrifuge force field at the selected gravitation coefficient so as the crystallization takes place in a centrifuge force field until the melt, cooling in the ingot of the crystallizer, would reach the temperature of the onset of crystallization processes in general conditions,
   at the third stage a cooling cast is treated in a centrifuge force field at the selected gravitation coefficient until the cast, cooling in the ingot of the crystallizer, would reach the temperature of the termination of crystallization processes in general conditions.

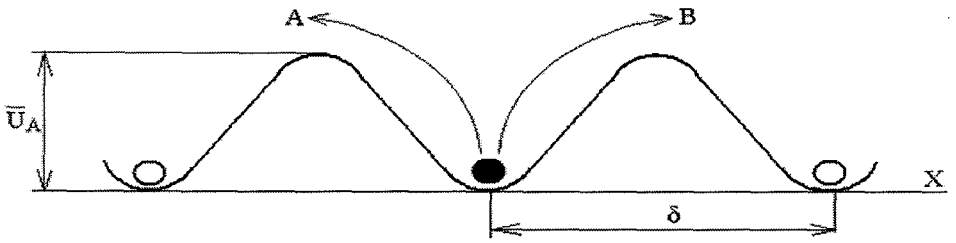

Fig. 1

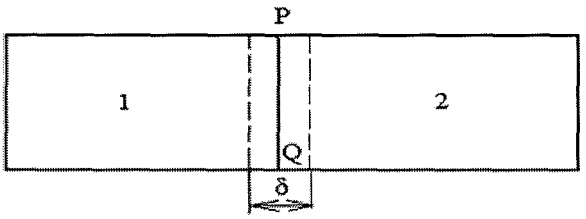

Fig. 2

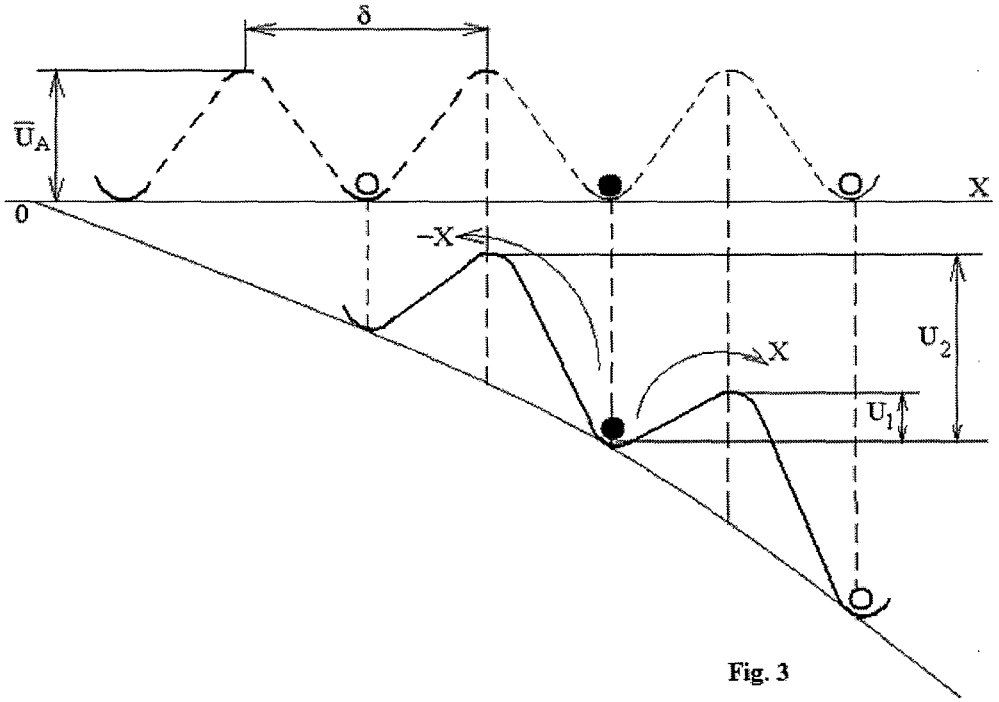

Fig. 3

INTERNATIONAL SEARCH REPORT

International application No.
PCT/RU 2009/000123

A. CLASSIFICATION OF SUBJECT MATTER

C22C 35/00 *(2006.01)*
C22C 1/02 *(2006.01)*

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C22C 35/00, 1/00, 1/02, 1/03

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

RUPAT, Esp@cenet, et Patserch USPTO, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	RU 2296175 C1 (ANISIMOV OLEG VLADIMIROVICH) 27.03.2007	1
A	WO 2003/080881 A1 (COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH) 02.10.2003	1
A	CA 2424595 A1 (ALCOA INC.) 18.04.2002	1

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:

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"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
03 July 2009 (03.07.2009)	20 August 2009 (20.08.2009)
Name and mailing address of the ISA/	Authorized officer
Facsimile No.	Telephone No.

Form PCT/ISA/210 (second sheet) (July 1998)

REFERENCES CITED IN THE DESCRIPTION

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Patent documents cited in the description

- SU 1650746 **[0012]**
- RU 2210611 **[0014]**
- RU 2296175 **[0016]**