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(54) **METHOD FOR OBTAINING ALLOYING ADDITIVES USED FOR PRODUCING ALLOYS**

(57)    The invention relates to metallurgy. The inventive method for obtaining alloying additives used for producing alloys consists in melting a base metal and operating ingredients (additives) and in carrying out the crystallization associated with the volumetric cooling of the melt at different rates in such a way that intermetallic compound particles are broken. The increased stability of additives during the preparation of alloys and the improved quality thereof by using additives of a "substitution-introduction solid solution" type are obtained by carrying out crystallization in the stress field of centrifuges at a gravitation factor ranging from 20 to 240. The crystallization process and the subsequent treatment of the ingot is carried out for a time equal to the relationship $t = m_3/ak_g$, wherein a is the coefficient, $k_8$ is the gravitation factor, $m_3$ is the relative mass of ingredients dissolved in an additive.

**Fig. 3**

EP 2 108 709 A1

**Description**

FIELD OF THE INVENTION

**[0001]**    The invention relates to metallurgy, particularly to a method for producing various alloying additives altering the service properties of alloys, and also to methods for producing hardeners.

DESCRIPTION OF THE PRIOR ART

**[0002]**    Existing methods for producing additives are based on various physical principles that help achieve two key objectives.
**[0003]**    The first embodiment in which additives are used comprises modification (of the first and second types) of the basic melt in order to significantly disintegrate the structure thereof. Accordingly, the various methods for preparing modifying additives (hardeners), depending on the type of the modifier itself, are to fulfill the following purposes:

-    when surfactants are used, they are to block growth of large crystals in the hardeners without altering the chemical composition of the matrix, the surfactants serving as adsorbents; and
-    when refractory materials are used, they are to be distributed uniformly over the hardener volume to comminute the hardener structure thoroughly. It is also required in this embodiment to comminute the new intermetallic compounds by, for example, establishing relatively stable bonds between ultra-dispersed particles of different oxides.

**[0004]**    As hardeners of the first type are melted again during modification of working melts, relatively little overheating destroys intergrain links, in accordance with the laws of thermodynamics, the modifier (adsorbent) is extracted, and then removed from the grain surface, followed by disintegration of the grain. The overheating value is very small (20°C to 40°C), yet enough for ultra-dispersed hardener grains to be unsuitable in the substrate role even in the case of brief significant "overheating" periods. In practice, however, the user of this hardener type mostly has the original alloying element as the modifier, at best distributed uniformly over the periphery of the hardener matrix.
**[0005]**    Repeated melting of second type hardeners that are, as a rule, a mechanical mixture of a basic metal and highly dispersed oxide, carbide, and nitride particles serving as nuclei for its growing grains, is only used in its modifying role after significant overheating (more than 50°C to 70°C) that results in grain disintegration has been achieved. These are the only conditions in which the alloying element can be used most effectively for modification purposes. The alloying element in these hardeners is to be distributed uniformly over the hardener volume.
**[0006]**    The second embodiment using additives consists generally in obtaining a desired composition of any alloys with the aid of these additives. In this case, additives are to have a second component dissolved to the maximum extent in the basic matrix, including oversaturated solutions.
**[0007]**    To prepare such additives, manufacturers try to comminute their structure as a whole and the eutectic part beyond the grains as well. In this case, the additive as a whole contains, for example, 20% of nickel in the aluminum base, whereas in the nickel grain itself it may not exceed 5%. When additives of this type, which are, at best, intrusion solutions having a large relative volume of eutectics, are melted again, it is again required, as was initially, to introduce a second component(s) into the original working melt by attempting to fully dissolve the eutectics and equalize the concentrations of the second component between an additive grain and the working melt by diffusion.
**[0008]**    With solubility still an unresolved problem, the expected result achieved by using such additives is production of any alloys of a desired chemical composition that only have a fraction of the possible service properties.
**[0009]**    Additives are prepared today by the method used for producing hardeners for aluminum alloys comprising comminuting intermetallic particles in the hardener and improving their stability during melt preparation by establishing stable bonds between ultra-dispersed synthetic oxide particles (SU Inventor's Certificate No. 1,650,746, C22 C1/03, published May 23, 1991).
**[0010]**    These methods are relatively simple to implement, and when used they help to comminute the structure of castings. As alloys are prepared, however, they practically duplicate (at best) the structure of the additive because the additive cannot be physically dissolved in the alloy matrix and do not have the possible physical properties.
**[0011]**    A prior art method for preparing hardeners by exposing their melts to an electromagnetic field (RU No. 2,210,611, C22 C1/03, published August 20, 2003) comprises melting a basic metal and working ingredients, stirring, filtering, and solidifying the melt cooled volumetrically at different rates to comminute the intermetallic particles, the uniformity of the additives being improved by reducing the proportion of primary intermetallic compounds as solidification is carried out by applying an outside permanent magnetic field. This method has been accepted as immediate prior art of the claimed invention.
**[0012]**    These operations, however, can only comminute the structure of the additive, without changing the quality of the solid solution because in both cases the difference between the concentration of substances (Fick's law) is the active

mechanism for dissolution of any component in the matrix (diffusion). No mechanism to accelerate diffusion processes exists in either case.

## DESCRIPTION OF THE INVENTION

**[0013]** This invention is intended to solve a technical problem of changing the solidification conditions of additives by using a novel physical phenomenon that accelerates diffusion processes in melts at the stage when a crystalline structure is formed in external fields of force. The technical result achieved by this invention consists in the production of additives that are more resistant to overheating and have an ideal solubility of their matrix and working ingredients in the basic alloy melt, with the result that the service properties of the melt are sharply improved (by between 25% and 30%).

**[0014]** This invention is basically aimed at developing a method for producing additives, including hardeners, for use in making various alloys comprising melting the basic metal and working ingredients; stirring; filtering, and solidifying the mix as it is cooled volumetrically at different rates to comminute the intermetallic particles, wherein, according to the invention, the stability of additives during the preparation of alloys is improved and their quality raised by using additives of the substitution-intrusion solid solution type by solidifying the additive melt in the field of force of centrifuges at a gravitation factor of 20 to 240, solidification and successive treatment of the ingot being carried out for a time period equal to the ratio $t = m_3/ak_g$, wherein $a$ is the process coefficient that is a numerical value found individually for each pair of metals depending on the thermodynamic characteristics of the mold and the rate of thermal processes developing therein; $k_g$ is the gravitation factor; and $m_3$ is the relative mass of ingredients being dissolved in the additive.

**[0015]** The claimed method is based on the use of an essentially novel physical phenomenon that accelerates diffusion processes in melts at the stage when a crystalline structure is formed in external fields of force.

**[0016]** Production of substitution-intrusion solid solutions without separating the eutectics requires additional energy inputs that distort the initial potential profiles and, as a result, creates conditions for accelerating diffusion of the working ingredients into the basic metal.

**[0017]** The external field of force, for example, the gravitational field of centrifuges, is easy to control and is indifferent to material type, so any kinds of additives, including those using nonmetallic materials, can be obtained.

**[0018]** The degree of distortion of potential profiles is identical to the overcooling fields created in melts that, according to the Tamman dependences, may, given their own specific value, promote formation of grains in a crystalline additive structure of any size.

**[0019]** Additives of the claimed type are more resistant to overheating and make their own matrix ideally soluble, together with the working ingredients, in the basic alloy melt, improving sharply (by 25% to 30%) the service properties of the alloy.

## DESCRIPTION OF THE DRAWINGS

**[0020]** The present invention will be more apparent from the detailed description of the following embodiments thereof with reference to the accompanying drawings, in which:

FIG. 1 is a diagrammatic view of the potential profile in the vicinity of any melt atom in the absence of an external field of force;
FIG. 2 is a diagrammatic illustration of diffusion; and
FIG. 3 is a diagrammatic view of a potential profile in the vicinity of any melt atom under the effect of an external field of force.

## PREFERRED EMBODIMENT OF THE INVENTION

**[0021]** According to this invention, the claimed method for preparing additives, including hardeners, used for producing various alloys is carried out in revolving molds of arbitrary design. Ingot molds have arbitrary shapes as well, from those for producing castings to those for making wire. A foundry mold is to maintain the required lifetime of reasonably over-heated melt (several minutes) and tolerate gravitation factors not exceeding 240.

**[0022]** The claimed method is based on the use of an essentially new physical phenomenon initiating diffusion processes in melts at the stage when a crystalline casting is shaped in an external field of force.

**[0023]** Where there is a need to prepare an additive of the substitution solid solution type, it is essential to achieve a required level of dissolution of element A in element B, corresponding to diffusion of A in B, that is, transfer of an atom of element A to a node of the crystalline lattice of element B.

**[0024]** According to Frenkel's theory, the number of atoms n transferred from a melt in a time unit in a volume unit to the crystalline lattice in any initial concentration of impurity A in the melt and the solid is equal to:

$$n = n_s \, P \, f \, \exp \left[ -\frac{\bar{\bar{U}}_A + \triangle A_i}{KT} \right] \tag{1}$$

wherein: $n_s$ is the number of impurity A atoms in the zone in question;
P is the probability of an atom being transferred in the desired direction;
f is the oscillation frequency of the atom about the equilibrium position;
$\overline{U}_A$ is the average activation energy of B atom transfer to a node of the crystalline lattice of the same element from the melt; and
$\triangle A_i$ is additional activation energy required for a similar transfer of an A atom (the energy originates from the difference between the A atom and B atom).

**[0025]** In an ordinary situation, with no external fields of force present, the potential profile in the vicinity of any melt atom is symmetric (FIG. 1), which accounts for the absence of a force causing diffusion through PQ (FIG. 2). A force causing atoms to move from environment 2 to environment 1 only exists in the absence of concentration equality, that is, in the case of self-diffusion.

**[0026]** Presence of an external field of force F at a potential energy E causes distortion of the initial potential profile (FIG. 3). The heights of the potential barriers in both directions of A atom transfer are equal, respectively, to:

$$U_1 = \bar{\bar{U}}_A - F\delta/2 + \triangle A_i$$
$$U_2 = \bar{\bar{U}}_A + F\delta/2 + \triangle A_i \tag{2}$$

wherein: $\delta$ is the crystalline lattice order.

**[0027]** The general asymmetry of the potential profile is equal to:

$$\triangle U = F\delta \tag{3}$$

**[0028]** The presence of $\triangle U$ causes an asymmetric stream of atoms $n_2$ that can be found from the formula:

$$n_2 = s \bar{V} c_i \tag{4}$$

wherein: $\overline{V}$ is the average speed of type A atoms;
S is the area of the solid phase; and
Ci is the concentration of atoms of impurity A.

**[0029]** The following computations are made for evaluation purposes. The atom remains in a settled condition in the *i*-th hole for a time:

$$\tau = \tau_0 \, \exp[\bar{\bar{U}}_A + \triangle A_i / KT] \tag{5}$$

wherein: $\tau_0 = 1/f$ is the oscillation period of the atom.

**[0030]** In this case, the probability of the atom moving in direction X is equal to:

$$P_x^x = P_0^x \, dt/\tau \tag{6}$$

wherein: $P_o^x$ is the probability of it jumping in a random direction (normally accepted to be 1/6), equal to:

$$P_o^x = \frac{1}{6} U_2/U_1 \tag{7}$$

**[0031]** Formula (6) is transformed, in view of (7), to:

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

**[0032]** The probability of an atom of impurity A jumping in direction (+X), in view of formulas (2) and (5), is equal to:

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

in direction (-X), to:

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

**[0033]** The probable movement of an atom of impurity A over the time *dt* can be obtained as a product of δ multiplied by the difference of these probabilities:

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

**[0034]** Hence, the movement speed $\overline{V}$ of atoms of impurity A is equal to:

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

**[0035]** Substitution of this formula in formula (4) gives:

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

**[0036]** Series expansion of the exponents:

$$\exp[\frac{F\delta}{2KT}] = 1 + \frac{F\delta}{2KT} + \cdots \cdots \tag{14}$$

$$\exp\left[-\frac{F\delta}{2KT}\right] = 1 - \frac{F\delta}{2KT} + \cdots\cdots$$

gives:

$$n = \frac{sc_i\delta}{6\tau^0}\left[\frac{F\delta}{2KT}\frac{U_1^2+U_2^2}{U_1U_2} + \frac{U_2^2 \cdot U_1^2}{U_1U_2}\right]\exp\left[-\frac{\overline{U}_A+\triangle A_i}{KT}\right] \qquad (15)$$

[0037]   It may be assumed that construction of a combined crystalline lattice during solidification in the presence of A and B type atoms will be described by the formula:

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

[0038]   In other words, if an impurity atom A has $\triangle A_i$=0 (atom parameters identical to matrix B), the probability of preparing an additive of the substitution solid solution type is equal to 1. In view of (16), equation (15) is transformed to:

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

[0039]   The total stream of diffusion atoms is equal to:

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

[0040]   This formula can be simplified (with $n_D$ reduced 1-6-fold), assuming that $V_1$=$V_2$:

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

[0041]   The difference of the streams $n_D$ depends on total activation energy ($\overline{U}_A + \triangle A_i$), which may be assumed to the equal to the hardening or melting activation energy. Formula (19) describes the total stream of atoms A and B having an activation energy ranging from $\overline{U}_A$ to $\overline{U}_A + \triangle A_i$. It follows, then, that with atoms of types A and B present in the melt, with five respective dimensions and solidification activation energies at the same time, production of a substitution solid solution is a result of solidification carried out in a field of force. Moreover, there is a probability P that the resulting solid solution will have 50% of element A, if $\triangle A_i$ =0, because force F is indifferent to atom type.
[0042]   If the concentration of impurity $C_i$(A) in the melt is above 0.5, a solid phase dominated by type A atoms will develop in this situation at $\triangle A_i$ =0.
[0043]   Certainly, if A atoms are significantly bigger in size than B atoms of the solvent (> 15-20%), a substitution solid solution cannot exist in a sustainable state, and intrusion solid solutions having a minimum volume of intermediate phases are formed.
[0044]   The situation changes significantly if the concentration of A atoms of larger dimensions exceeds 0.5, in which case the melt of A atoms becomes a solvent relative to B atoms. In this case, B atoms move into the nodes of the crystalline lattice of A atoms, and a substitution solid solution is formed.

**[0045]** The efficiency of the additive preparation process in fields of force may be evaluated from the formula:

$$Z = \frac{n_D}{n_i} = \frac{s c_i \delta^2 P_i m g}{K T n_3} K_g - 1 \qquad (20)$$

wherein: m the melt mass in grams;
g is free fall acceleration; and
Kg is the gravitation factor of the centrifuge.
Formula (20) applies to additive production in centrifuges.
**[0046]** A significant role is played by the time $t$ required for completing the diffusion processes:

$$t = n_3 / n_D \qquad (21)$$

wherein: $n_D$ is a preset number of diffusing impurity atoms.
**[0047]** After the right part of formula (21) is multiplied by the atomic weight $m_0$ of A, the formula is transformed to:

$$t = m_3 / m'_D, \qquad (22)$$

wherein: $m_3$ is a preset quantity (kg) of diffusing impurity atoms (A), $m'_D = n_D$, $m_0$ is diffusion rate (kg/sec), and $m_0$ is the atomic weight of A.
**[0048]** Following transformation, formula (19) becomes:

$$m'_g = a_1 K_g - a_2 \qquad (23)$$

wherein:

$$a_1 = \frac{s m_i c_i \delta^2 P_i M g}{6 K T \tau^9} \exp\left[ -\frac{\overline{\overline{U}}_A + \triangle A_i}{K T} \right]$$

$$a_2 = \frac{n_g}{6 \tau^0} \exp\left[ -\frac{\overline{\overline{U}}_A + \triangle A_i}{K T} \right]$$

**[0049]** In view of (23), formula (22) is transformed to:

$$t = m_3 / (a_1 k_g - a_2) \qquad (24)$$

**[0050]** Numerical analysis of formula (20) shows that, beginning with the gravitation factor value of 130 to 140, the efficiency of additive preparation by the claimed method increases by a double-digit factor. This makes for the lowermost value of Kg. The highest value is determined by other causes.
**[0051]** The lifetime of a melt at a selected gravitation factor calculated from formula (24) is normally several minutes. For example, the required lifetime of an Al + 20% Ni melt is about 4 or 5 minutes. A shorter time is not enough for nickel to be dissolved completely in aluminum. A longer time $t$ results in unjustified energy costs, without a significant improvement in additive quality.
**[0052]** The use of the present method for producing an Al + 20% Ni additive is described below for illustration purposes.

**[0053]** This additive is currently produced in centrifuges provided with ingot molds to obtain 3.5 kg ingots, with the gravitation factor possibly ranging from 0 to 500.

**[0054]** The studies comprised metallographic evaluation of Δe eutectic dispersion across the body of the ingots depending on changes in $k_g$ and $t$.

**[0055]** The results of the studies are summarized in Tables 1 and 2.

Table 1

| kg | 60 | 80 | 100 | 140 | 180 | 240 | 280 | 350 |
|---|---|---|---|---|---|---|---|---|
| Δe, micron | 76 | 55 | 41 | 32 | 16 | 9.5 | 19.6 | 19.8 |

Table 2

| t, min | 0,5 | 1 | 2 | 3 | 4 | 5 | 6 | 7 |
|---|---|---|---|---|---|---|---|---|
| Δe, micron | 81 | | 40 | 15 | 10 | 9.4 | 10.8 | 12.0 |

**[0056]** It is clear from Table 1 that the settled conditions for nickel dissolution in aluminum to obtain a minimum eutectic size (9.5 microns) correspond (for the melt lifetime $t = 4$ minutes) to the gravitation factor Kg = 240. It is reasonable to keep the gravitation factor below 260 because doubling the gravitation factor reduces the eutectic size (volume) by 20% only, while the use of such additives is confronted with specific problems.

**[0057]** Table 2 shows that the settled conditions for nickel dissolution in aluminum to obtain a minimum eutectic size (9.5 microns) correspond (for the gravitation factor Kg = 240) to a lifetime t>4 minutes. Further increase in $t$ does not produce significant changes in Kg value.

INDUSTRIAL APPLICABILITY

**[0058]** This invention may be used for preparing any additives of both the metal and nonmetal groups of materials, including salts and any crystallizing and polymerizing materials. The invention is used most effectively for producing alloying additives and hardeners.

**Claims**

1. A method for obtaining alloying additives for producing alloys, comprising melting a basic metal and soluble ingredients, and solidifying the melt during volumetric cooling thereof for comminuting intermetallic particles, wherein, with the purpose of obtaining alloying additives of the "substitution-intrusion solid solution" type, the melt is solidified in the field of force of centrifuges at a gravitation factor ranging within 20 to 240 for a time period equal to the ratio:

$$t = m_3 / a k_g$$

wherein:
a is a coefficient determined as a numerical value separately for each metal-ingredient pair with consideration for the thermodynamic characteristics of the mold and the rate of thermal processes occurring therein;
$k_g$ is the gravitation factor; and
$m_3$ is the relative mass of the ingredients soluble in the alloying additive.
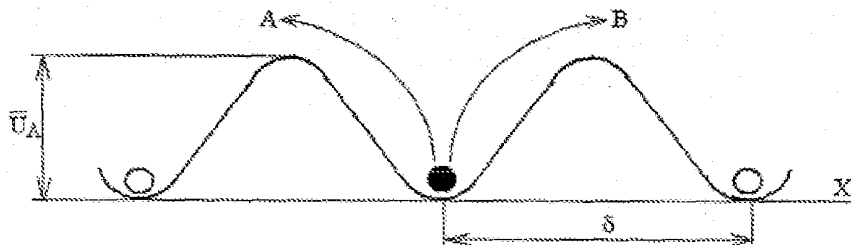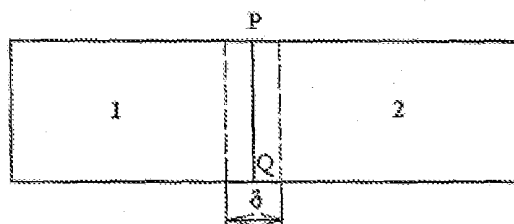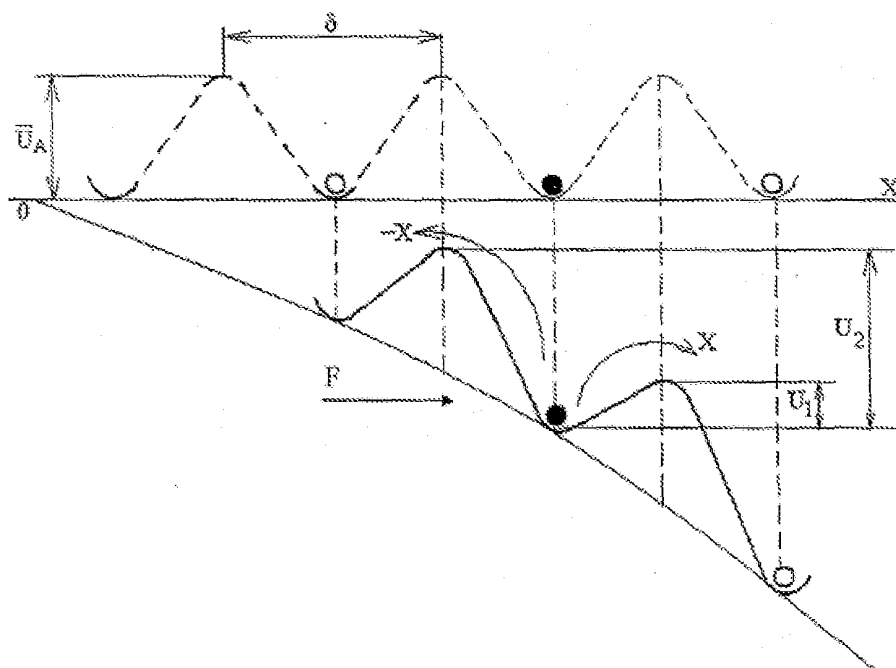
# Fig. 1

# Fig. 2

# Fig. 3

## INTERNATIONAL SEARCH REPORT

| | International application No. |
|---|---|
| | PCT/RU 2006/000680 |

**A. CLASSIFICATION OF SUBJECT MATTER**

*C22C 35/00 (2006.01)*
*C22C 1/03 (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 1/00, 1/03, 1/06, 35/00, B22D 13/00, 27/02

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, USPTO DB, Esp@cenet, PAJ

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

| Category* | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
|---|---|---|
| A | SU 1650746 A1 (OMSKII POLITEKHNICHESKII INSTITUT) 23.05.1991 | 1 |
| A | RU 2210611 C2 (REGIONALNY OBSCHESTVO FOND SODEISTVIYA ZASCHITE INTELLECTUALNOI SOBSTVENNOSTI) 20.08.2003 | 1 |
| A | SU 865948 A (ALTMAN M.B. et al.) 23.09.1981 | 1 |
| A | US 3592636 A (POHLMAN R. et al) 13.07.1971 | 1 |
| A | GB 1244082 A (KAWECKI BERILCO INDUSTRIES INC.) 25.08.1971 | 1 |
| A | WO 1994/002270 A1 (POPOV ANATOLII VLADIMIROVICH et al.) 03.02.1994 | 1 |

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**REFERENCES CITED IN THE DESCRIPTION**

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**Patent documents cited in the description**

- SU 1650746 **[0009]**
- RU 2210611 **[0011]**