(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) EP 0 803 579 A1

(12) EUROPEAN PATENT APPLICATION

published in accordance with Art. 158(3) EPC

(43) Date of publication:
29.10.1997 Bulletin 1997/44

(21) Application number: **94909359.5**

(22) Date of filing: **24.02.1994**

(51) Int. Cl.^{6}: **C21C 7/10**

(86) International application number:
PCT/RU94/00034

(87) International publication number:
WO 95/20057 (27.07.1995 Gazette 1995/32)

(84) Designated Contracting States:
AT BE DE ES FR GB IT LU SE

(30) Priority: **19.01.1994 RU 94000727**

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(54) PROCESS FOR THE VACUUM REFINING OF METAL AND ASSOCIATED DEVICE

(57) 1. A method of vacuum refining of metal, in which the pressure of a mixture of gases above the molten metal surface is reduced to a pressure sufficient for generating partial pressures of the gases above the melt below the partial pressures of the gases in the melt, and the metal is treated by pressure pulsations with an amplitude of 0.02-0.8 MPa in a low-frequency range of 0.03-5 Hz and, and at the same time, in addition, the pressure in the medium-frequency range of 55-195 Hz with an amplitude of 0.005-0.01 MPa and the pressure in the high frequency range of 350-3500 Hz with an amplitude of 0.0001-0.001 MPa are varied. For this purpose, the device for vacuum refining of metal realizing the above method comprises a vacuum-tight container (1) having a gas-exhaust nipple (2), an ejector (3), having a housing (4), a nozzle (5) and a mixing channel (6), a unit (7) to produce low-frequency pulsations of the working gas through the nozzle (5) of the ejector (3), a unit (8) to produce medium-frequency pulsations of the gas flow rate at the inlet of the gas-exhaust nipple (2), and a unit (10) to produce high-frequency pulsations of the flow rate of the gas being evacuated from the container (1), said regulator being made in the form of a regulator of varying, in a particular case, the flow section of the nozzle (5) and consisting of an insert (20).

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Printed by Rank Xerox (UK) Business Services
2.14.18/3.4

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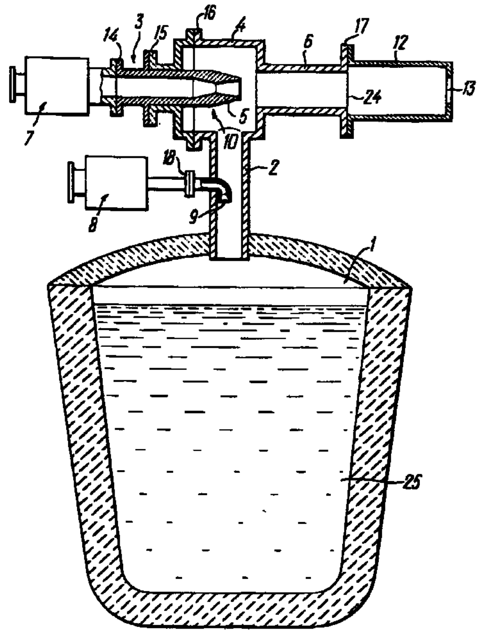

FIG. 1

Description

This invention relates to ferrous metallurgy and, moreparticularly, the invention relates to methods of vacuum refining of metal and to devices for performing these methods.

Prior Art

Known in the art is a method of out-of-hearth refining of molten metal, in which a vacuum is produced above the melt surface, said vacuum being continuously varied from 0.03 to 0.07 MPa. Superimposed on this vacuum is an additional pulsed vacuum of 0.02 to 0.05 MPa with a frequency from 5 to 50 Hz and from 200 to 250 Hz (RU, A, 1,547,323).

The prior art method features inadequate refining of the molten metal, because the effect of pressure pulsation does not penetrate to a required depth, from which the growth of gas bubbles of a necessary size is provided.

Furthermore, this method does not realize the process of rectified diffusion, which also contributes to the growth of the bubbles and has no effect on the "buffer" surface layer of fine gas bubbles in the melt.

Know in the art is a device for out-of-hearth refining of molten metal effecting the above method and consisting of a Known in the vacuum-tight container in which vacuum pulsations are produced by means of a vacuum pump (cf. the same reference).

This device realizing the above mentioned method does not allow one to maintain stable conditions of the treatment with variable gas evolution from the metal and cannot operate with heating by high-temperature gases being evacuated from the container.

Also known in the art is a method of vacuum refining of metal, in which the pressure of a gas mixture above the metal surface is reduced to a valuesufficient for producing partial pressures of the gases above the melt, said pressure being lower than the partial pressure of the gases in the melt, and the metal is acted on by pressure pulsations (RU, A, 1441809).

According to this method, a vacuum is produced above the melt surface, said vacuum pulsating in a high frequency range.

This method does not provide a required degree of metal refining, because the depth of treatment is inadequate.

Furthermore, this method does not realize the rectified diffusion process, while the process of floating up and fragmentation of the bubbles features low intensity and generally does not allow a metal of a required quality to be obtained.

Known in the art container with a gas-exhaust nipple and an ejector having a housing, a nozzle and a mixing channel and mounted on the gas-exhaust nipple (cf. the same reference).

This device realizing the above method also includes a tuyere for blowing the melt with an inert gas.

This device inconvenient, and unreliable in operation and has relatively low efficiency.

Disclosure of the Invention

The basic object of the invention was to provide a method of vacuum refining of metal having such regimes of pressure pulsations that would enable one to obtain a metal of a required quality, and to provide a device for vacuum refining of metal effecting the above method that would be convenient and reliable in operation and have the maximum possible efficiency.

This object is attained by providing a method of vacuum refining of metal, in which the pressure of a mixture of gases above the molten metal is reduced to a pressure sufficient for producing the partial pressures of the gases above the melt lower than the partial pressures of the gases in the melt, and the metal is treated by pressure pulses; according to the invention, the pressure above the melt surface is varied with an amplitude of 0.02-0.08 MPa in a low-frequency range of 0.03-5 Hz and, in addition, the pressure is simultaneously varied in a medium-frequency range of 55-195 Hz with an amplitude of 0.005-0.01 MPa, and in a high-frequency range of 350-3500 Hz with an amplitude of 0.0001-0.001 MPa.

The same method is suitable for treatment of individual portions of metal periodically taken from the total volume of metal.

This object is attained by providing a device for vacuum refining of metal effecting the said method and comprising a vacuum-tight container, having a gas-exhaust nipple, and an ejector, having a housing, a nozzle and a mixing channel and mounted on the gas-exhaust nipple; according to the invention, the device is equipped with a unit for producing low-frequency pulsations of the working gas flow rate in the ejector nozzle, a unit for producing medium-frequency pulsations of gas flow rate at the inlet of the gas-exhaust nipple, and a unit for producing high-frequency pulsation of the flow rate of the gas evacuated from the container made as a controller varying the flow section of the nozzle and/or mixing channel of the ejector according to the following expression:

$$F_a = K_1 \cdot F_c^{\,2}/F$$

where

F_a is the cross-sectional area of the mixing channel;

K_1 is the coefficient frown 0.5 to 0.7;

F_b, F_c are the areas of the critical and output sections of the nozzle, respectively;

$F_b = K_2 \cdot F_c$, where K_2 is the coefficient from 0.5 to 0.02.

The regulator for varying the flow section of the ejector nozzle can be made as a set of nozzles.

The regulator for varying the flow section of the nozzle and/or mixing channel of the ejector can also be made in the form of an insert mounted on the inner surface of the nozzle and/or mixing channel of the ejector.

The insert of the regulator varying the flow section of the ejector mixing channel can be made as a conical member with a taper angle of 1-3° whose narrowing part extends towards the mixing channel outlet.

At the mixing channel outlet the ejector can have a cylindrical channel with a cross-sectional area of $F_d = (2\text{-}5) \cdot F_a$ a and a length of $L_a = 9(7\text{-}12)D_a$, where D_a is the cylindrical channel diameter, and an output throat with an area of $F_c = (1.2\text{-}2.5)\ F_a$.

The advantages of the claimed method consist in that its realization allows one to significantly reduce the content of residual dissolved gases and nonmetallic inclusions in the metal due to an increase of the depth of treatment of the melt in the container, organization of a rectified diffusion process, intensification of the floating function of the bubbles, reduction of the resistance to the evolution of gases from the metal and passing them through a slag layer, all these facts allowing the metal of a preset quality to be obtained. The claimed device effecting the method, according to the invention, makes it possible to obtain all regimes of pressure pulsations corresponding to the physical and chemical characteristics of the melt being treated. In so doing the device utilizes the working gas energy with maximum efficiency due to minimization of losses by selecting optimum geometry along the gas-dynamic duct and using the automatic adjustment phenomenon following the changes in the evolution of the gas from the melt. The claimed device is long-life and reliable in operation, because the basic units have no contact with the high temperature gases evacuated from the vacuum chamber and has maximum possible efficiency.

Brief Description of the Drawings

The invention is further described by way of example with reference to the accompanying drawings, in which:

Figure 1 shows a general diagram of the claimed device for vacuum refining of metal to perform one of the embodiments of the method according to the invention (a partial longitudinal section);
Figure 2 is a general diagram of another embodiment of the device of Figure 1 (a partial longitudinal section);
Figure 3 is still another embodiment of the device shown in Figure 1 (a partial longitudinal section);
Figure 4 is yet another embodiment of the claimed device shown in Figure 1 (a partial longitudinal section);
Figure 5 is an embodiment of the device of Figs. 3 and 4 (a partial longitudinal section);
Figure 6 shows a general diagram of the claimed device effecting another embodiment of the method according to the invention (a partial longitudinal section).

Best Embodiments of the Invention

The claimed method of vacuum refining of metal consists in that supplied into a container with molten metal is a working gas having predetermined parameters, the pressure of gaseous mixture above the melt surface is reduced within 2-5 seconds to a pressure sufficient for producing partial pressures of the gases in the melt, and the metal is treated by pressure pulsations.

The pressure above the melt surface is varied with an amplitude of 0.02-0.08 MPa in a low-frequency range of 0.03-5 Hz and, at the same time, the pressure is varied in a medium-frequency range of 55-195 Hz with an amplitude of 0.005- 0.01 MPa, and in a high-frequency range of 350-3500 Hz with an amplitude of 0.0001-0.001 Hz.

Since the pressure is continuously varied, the resultant value of this pressure is summed up from the components of different frequency and amplitude. The low-frequency component in a range of 0.03-5 Hz with an amplitude of 0.02-0.08 Mpa leads to such a phenomenon that during the pressure decrease the bubbles of CO or inert gas expand and during the pressure increase they compress. During the expansion the diffusion flow is directed into the bubbles while during the compression it is directed to outside, however, the former process is much more intensive and leads to a fast growth of the bubble. The bubble pulsation results in permanent renewal of the melt in its boundary layer; when the bubble reaches the resonance size, it disintegrates into smaller bubbles which start grow as well. The low-frequency pulsations of the pressure above the melt surface generate vacuum and pressure waves in the melt itself. In view of the fact that a frequency range of 0.03-5 Hz is selected, overlapping the band of resonance frequencies of the containers with the melt having a depth of 0.5 to 4 m with a gas content from 0 to 50%, the amplitude of pulsations below 0.02 MPa does not result in significant intensification of the process of rectified diffusion of gas in the molten metal, whereas the amplitude above 0.08 MPa increases the energy consumption and reduces the resistance of the vacuum-tight container.

The pressure pulsation component in a frequency range of 55-195 Hz with an amplitude of 0.005-0.02 MPa generates in (the region of the container walls intensive microflows which break the formed and newly forming bubbles.

Furthermore, this component affects the oscillation and fragmentation of the bubbles resonant to the given frequency range.

The parameters of the pressure pulsation at the medium frequencies are associated with the velocity of the melt microflows near the walls, which correspond to the rate of roughness of the walls, and the rate of cavitation strength of the melt associated with its temperature and composition.

The high frequency pressure pulsations in a range of 350-3500 Hz with an amplitude of pressure 0.0001-0.001 MPa result in oscillation and fragmentation of the bubbles of a different diameter having already floating on the metal surface and forming the so called "buffer layer" preventing the hydrogen diffusion from the upper metal layer due to a low pressure above the metal surface. The hydrogen diffusion from the upper layer, when realizing the method, has a significant effect on the total intensity of refining the entire metal, because a high amplitude of the CO bubbles under the pressure pulsation acting through the whole volume of the melt produces intensive ascending streams delivering the metal from the deep layers to the surface, i.e. the hydrogen-free layers are being replaced continuously.

An increase of the radius of capture of the bubbles during the processes of coagulation and flotation taking place deep in the melt has a significant effect on the high-frequency pulsations. The range of high-frequency pulsations of the bright-line spectra is connected to the resonance characteris-tics of the bubbles reaching the surface and was obtained on the basis of experimental data by realizing the maximum evolution of gas from the melt, flow rate of the active gas and the well known ejection coefficient under the given operating conditions. The gas release from the melt during the evacuation is given in the table below.

Table

Melt No. spectrum band	Volume flow rate of gas release, m^3/s	Frequency Hz
2250	0.031	50-1000
2671	0.094	350-3500
2680	0.949	1000-3000
3112	0.043	50-500
3120	0.167	350-3500
3215	0.070	1000-3000

In another embodiment of realisation of the claimed method individual portion of metal periodically taken from the whole volume of metal are treated.

Described below in detail is the claimed device for vacuum refining of metal effecting the method under consideration.

This device comprises a vacuum-tight container 1 (Fig. 1) having a gas-exhaust nipple 2, an ejector 3 having a housing 4, a nozzle 5 and a mixing channel 6 and mounted on the gas-exhaust nipple 2. The operation of the ejector 3 depends on such characteristic geometric parameters as the cross-sectional area F_a of the mixing channel 6, the critical section area F_b of the nozzle 5 and the output cross-sectional area F_c of the nozzle 5.

At the inlet of the nozzle 5 of the ejector 3 the device is provided with a unit 7 for producing low-frequency pulsations of the working gas flow rate through the nozzle 5, a unit 8 for producing medium-frequency pulsations of the gas flow rate at the inlet of the gas-discharge branch pipe 2 through the nozzle 9, and a unit 10 for producing high-frequency pulsations of the flow rate of the gas being evacuated from the container 1.

The unit 10 is made in the form of a regulator varying the cross sectional area of the nozzle 5 and/or mixing channel 6 of the ejector 3 according to the following expression:

$$F_a = K_1 \cdot F_c^{\,2}/F_b,$$

where
F_a is the cross-sectional area of the mixing channel;
K_1 is the coefficient from 0.5 to 0.7;
F_b, F_c are the areas of the critical and output sections

of the nozzle, respectively;

$F_b = K_2 \cdot F_c$, where K_2 is the coefficient from 0.5 to 0.02.

In the above described embodiment of the claimed device realizing the method, according to the invention, the regulator for varying the flow section of the nozzle 5, 11 (Figure 2) of the ejector 3 is made in the form of a set of nozzles 5 and 11, where in Figure 1 the nozzle 5 has a greater flow section than the nozzle 11 in Figure 2.

The ejector 3 has at the outlet of the mixing channel 6 a cylindrical channel 12 with a cross-sectional area $F_d = (2\text{-}5)F_a$, a length $L_a = (7\text{-}12)D_a$, where D_a is the diameter of the cylindrical channel 12, and with an output throat 13 having an area $F_c = (1.2\text{-}2.5)F_a$.

The unit 7 is secured to the ejector 3 by a fastener 14, the elements of the ejector 3 are secured by means of fasteners 15, 16, 17, and the unit 8 is secured to the nozzle 9 by means of a fastener 18.

In another embodiment of the claimed device effecting the method, according to the invention, the regulator varying the flow section of the nozzle 19 is made in the form of an insert 20 mounted on the internal surface of the nozzle 19, as shown in Figure 3.

In the embodiment of the claimed device shown in Figure 4 the regulator varying the flow section of the mixing channel 6 of the ejector 3 is made in the form of an insert 21 mounted on the internal surface of the mixing channel 6.

In the embodiment of the claimed device shown in Figure 5 regulator The varying the flow section of the nozzle 19 and mixing channel 6 of the ejector 3 is made in the form of two inserts 20 and 22 mounted, respectively, on the internal surface of the nozzle 19 and mixing channel 6. The insert 22 is conical with a taper angle of 1-3°, the narrowing portion 23 of the insert 22 extending towards the outlet 24 of the channel 6.

The embodiment of the claimed device shown in Figure 6 performs one version of effecting the method, according to the invention, in which there are treated individual portions of metal periodically taken from the total volume of metal 26 placed in the container 27. For this purpose, the vacuum-tight container 28 is immersed into the metal 26.

Figure 6 shows a device according to Figure 1 partially immersed into the metal 26. However, it should be noted that any device according to Figures 2-5 can successfully be used for this purpose.

The principle of operation of the claimed device for effecting the method according to the invention is as follows.

Fed into the vacuum-tight container 1 (Figure 1) filled with molten metal 25 is a working gas having predetermined parameters, and ejection evacuation of the gas from the vacuum-tight container 1 is started. The unit 7 providing low-frequency pulsations changes the flow rate of the working gas through the nozzle 5 so that the ejection of the gases from the container 1 and, therefore, the pressure therein. The unit 8 is used for producing the medium-frequency pulsations of the gas flow rate, and a gas jet is periodically blown into the gas-exhaust nipple 2 resulting in pressure pulsations in the container 1.

The flow section of the nozzle 5 is adjusted so that, when the working gas is supplied, the nozzle 5 forms a jet which interacts with the mixing channel 6 and performs the ejection of gases from the container 1. In so doing the flow rate of the gases ejected from the container 1 pulsates with a high frequency. Then the stream of a mixture of the working and ejected (passive) gas flow from the mixing channel 6 into the cylindrical channel 12. When the gas jet interacts with the walls of the channel 12 and with its throat 13 having an area of F_c, a reduced pressure is produced at the output of the mixing channel 6 increasing the productivity and, therefore, efficiency of the claimed device effecting the method according to the invention.

The principle of operation of the device shown in Figures 2-5 is similar to that of the device in Figure 1. The difference consists in that here use is made of the flow section of the nozzle 11 (Figure 2), or nozzle 19 (Figure 3), or mixing channel 6 (Figure 4), or nozzle 19 (Figure 5) and mixing channel 6.

The device of Figure 6 effecting the method, according to the invention, in a portion regime allows the metal 26 to be treated not in bulk but by separate portions of metals 25 within 10-300 seconds. During this period the processes originated by the proposed sequence of operations are over completely, while some nucleus of the bubbles remaining in this portion are discharged into the whole volume, in which intensive gas release and removal of non-metallic inclusions are initiated. Then a new portion of metal is sucked and the process is repeated.

The treatment is terminated upon reaching the required characteristics of the melt. The processing time is determined in the run of experimental melts or by the express-analysis results.

Therefore, the convenient and reliable device claimed for effecting the method according to the invention, having maximum possible efficiency, enable one to produce a metal of a required quality.

Industrial Applicability

The invention can be used in non-ferrous metallurgy when casting metals and alloys.

Claims

1. A method of vacuum refining of metal, in which the pressure of a mixture of gases above the molten metal surface is reduced to a value sufficient for producing partial pressures of the gases above the melt below the partial pressures of the gases in the melt, and the metal is treated by pressure pulsations, **characterized** in that the pressure above the melt surface is varied with an amplitude of 0.02-0.08 MPa in a low-frequency range of 0.03-5 Hz and, in addition and at the same time, the pressure in the medium-frequency range of 55-195 Hz is varied with an amplitude of 0.005-0.01 MPa and in the high-frequency range of 350-3500 Hz with an amplitude of 0.0001-0.001 MPa.

2. A method according to Claim 1, **characterized** in that subjected to the treatment are individual portions of metal periodically taken from the total volume of metal.

3. A device for vacuum refining of metal comprising a vacuum-tight container (1) having a gas-exhaust nipple (2) and an ejector (3) having a housing, a nozzle (5) and a mixing channel (6) , said ejector (3) being mounted on said gas-exhaust nipple (2), **characterized** in that the device has a unit (7) to produce low-frequency pulsation of the flow rate of working gas through the nozzle (5) of the ejector (3), a unit (8) to produce medium-frequency pulsation of the flow rate of gas at the inlet of the gas-exhaust nipple (2), and a unit (10) to produce high-frequency pulsations of the flow rate of gas having evacuated from the container (1) made in the form of a regulator varying the flow section of the nozzle (5) of the ejector (3) in accordance with the following expression:

$$F_a = K_1 \cdot F_c^{\,2}/F_b,$$

where F_a is the cross-sectional area of the mixing channel (6);
K_1 is the coefficient from 0.5 to 0.7;
F_b, F_c are the areas of the critical and output sections of the nozzle (5), respectively;
$F_b = K_2 \cdot F_c$, where K_2 is the coefficient from 0.7 to 0.02.

4. A device according to Claim 3, **characterized** in that the regulator varying the flow section of the nozzle (5,11) of the ejector (3) is made in the form of a set of nozzles (5,11).

5. A device according to Claim 3, **characterized** in that the regulator varying the flow section of the nozzle (19) of the ejector (3) is made in the form of an insert (20) mounted on the internal surface of the nozzle (19) of the ejector (3).

6. A device for vacuum refining of metal comprising a vacuum-tight container (1) having a gas-exhaust nipple (2) and an ejector (3) having a housing (4), a nozzle (5) and a mixing channel (6), said ejector (3) being mounted on said gas-exhaust nipple (2), **characterized** in that the device has a unit (7) to produce low-frequency pulsation of the flow rate of working gas through the nozzle (5) of the ejector (3), a unit (8) to produce medium-frequency pulsation of the flow rate of gas at the inlet of the gas-exhaust nipple (2), and a unit (10) to produce high-frequency pulsation of the flow rate of gas being evacuated from the container (1) made in the form of a regulator varying the flow section of the mixing channel (6) of the ejector (3) in accordance with the following expression:

$$F_a = K_1 \cdot F_c^{\,2}/F_b,$$

where F_a is the cross-sectional area of the mixing channel (6);
K_1 is the coefficient from 0.5 to 0.7;
F_b, F_c are the areas of the critical and output sections of the nozzle (5), respectively;
$F_b = K_2 \cdot F_c$, where K_2 is the coefficient from 0.7 to 0.02.

7. A device according to Claim 6, **characterized** in that the regulator varying the flow section of the mixing channel (6) of the ejector (3) is made in the form of an insert (21) mounted on the internal surface of the mixing channel (6) of the ejector (3).

8. A device for vacuum refining of metal comprising a vacuum-tight container (1) having a gas-exhaust nipple (2) and an ejector (3) having a housing (4), a nozzle (19) and a mixing channel (6), said ejector (3) being mounted on said gas-exhaust nipple (2), **characterized** in that the device has a unit (7) to produce low-frequency pulsation of the flow rate of working gas through the nozzle (19) of the ejector (3), a unit (8) to produce medium-frequency pulsation of the flow rate of gas at the inlet of the gas-exhaust nipple (2), and a unit (10) to produce high-frequency pulsations of the flow rate of gas being evacuated from the container (1) made in the form of a regulator varying the flow section of the nozzle (19) and mixing channel (6) of the ejector (3) in accordance with the following expression:

$$F_a = K_1 \cdot F_c^{\ 2}/F_b,$$

where F_a is the cross-sectional area of the mixing channel (6);
K_1 is the coefficient from 0.5 to 0.7;
F_b, F_c are the areas of the critical and output sections of the nozzle (19), respectively;
$F_b = K_2 \cdot F_c$, where K_2 is the coefficient from 0.7 to 0.02.

9. A device according to Claim 8, **characterized** in that the regulator varying the flow section of the nozzle (19) and mixing channel (6) of the ejector (3) is made in the form of inserts (20,21)) mounted on the internal surface of the nozzle (19) and mixing channel (6) of the ejector (3) respectively.

10. A device according to Claim 7 or Claim 9, **characterized** in that the insert (22) of the regulator varying the flow section of the mixing channel (6) of the ejector (3) is made conical with a taper angle of 1-3° and with a narrowing portion (23) extending towards the outlet (24 of the mixing channel (6).

11. A device according to Claim 3, or Claim 4, or Claim 5, or Claim 6, or Claim 7, or Claim 8, or Claim 9, **characterized** in that the ejector (3) at the outlet of the mixing channel (6) has a cylindrical channel (12) with a cross-sectional area of $F_d = (2\text{-}5)F_a$, a length of $L_a = (7\text{-}12)D_a$, where D_a is the diameter of the cylindrical channel (12) with a cross-sectional area $F_d = (2\text{-}5)F_a$, a length $L_a = (7\text{-}12)D_a$, where D_a is the diameter of the cylindrical channel (12), and with an output throat (13) with an area $F_c = (1.2\text{-}2.5)F_a$.

11. A device according to Claim 10, **characterized** in that the ejector (3) at the outlet of the mixing channel (6) has a cylindrical channel (12) with a cross-sectional area of $F_d = (2\text{-}5)F_a$, a length of $L = (7\text{-}12)D_a$, where $D_a$is the diameter of the cylindrical channel (12), and with an output throat (13) having an area $F_c = (1.2\text{-}2.5)F_a$.

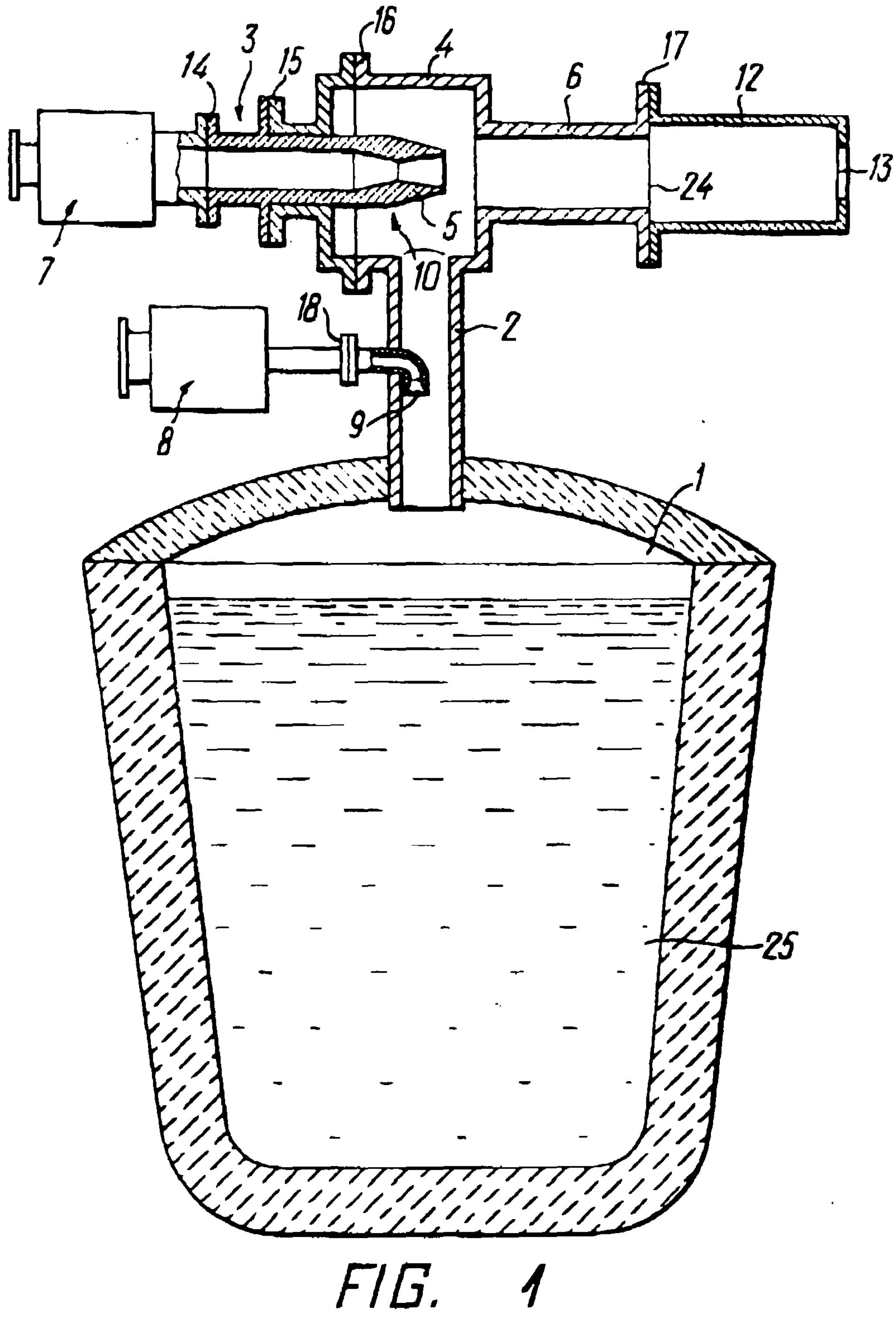

FIG. 1

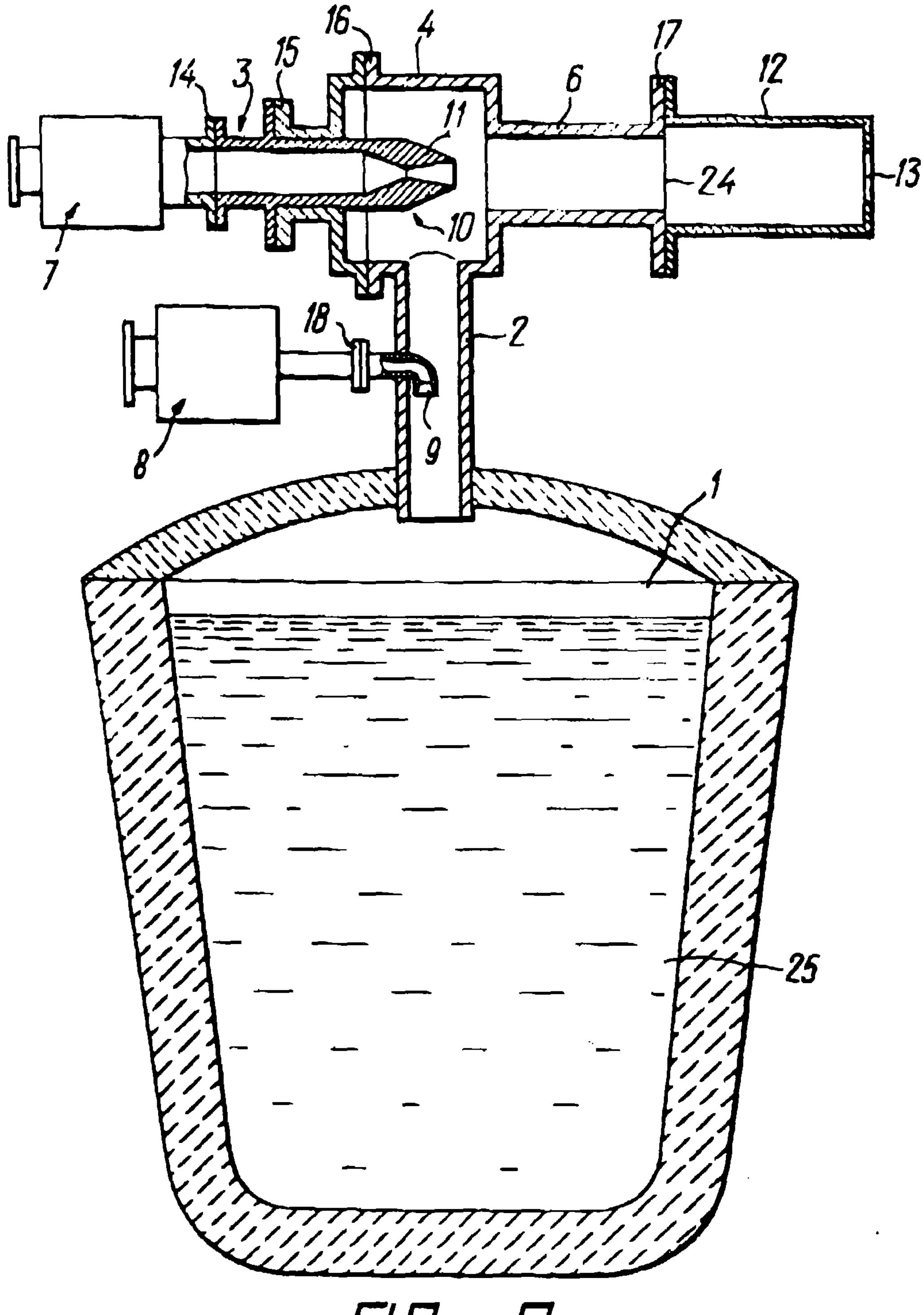

FIG. 2

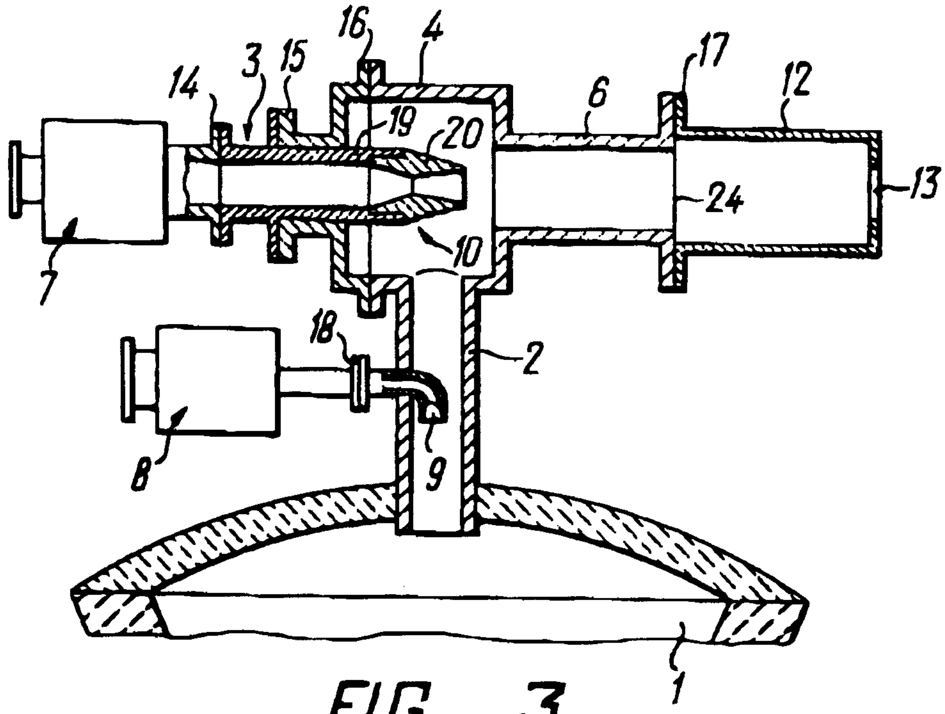
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FIG. 3

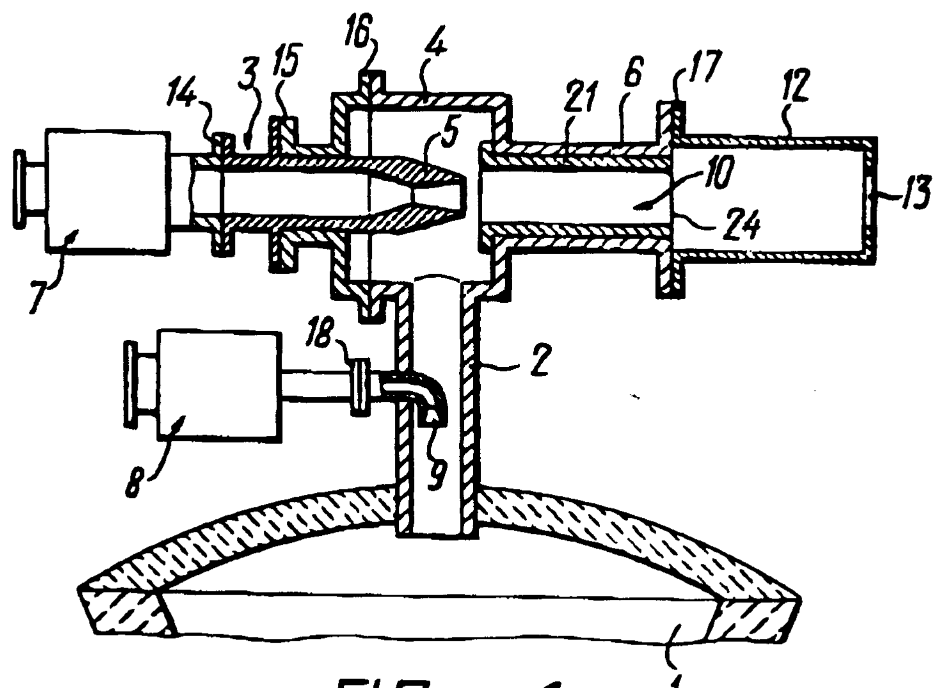
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FIG. 4

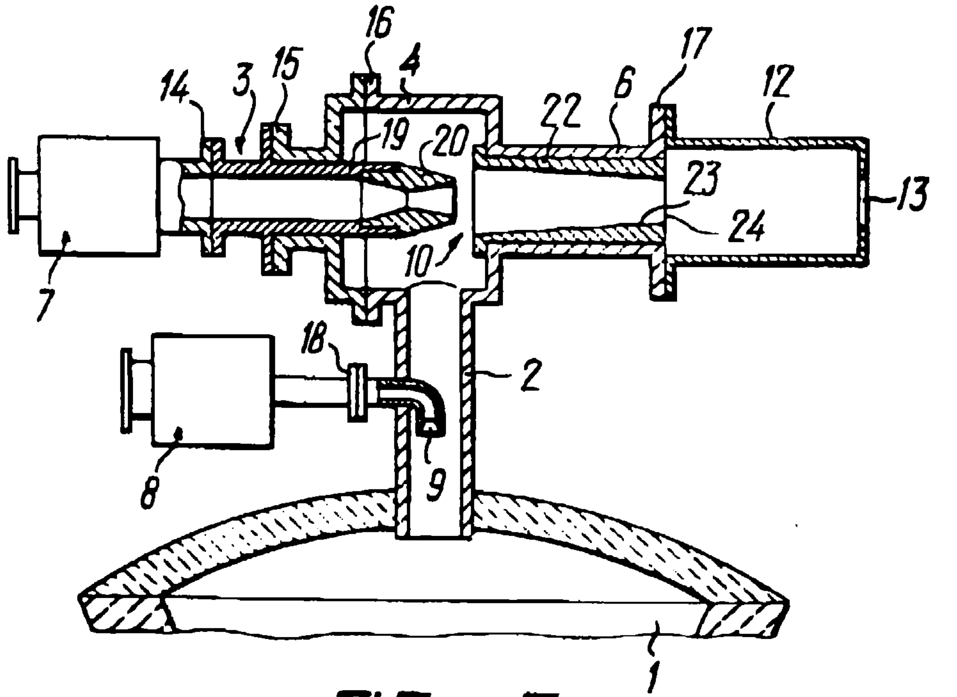

FIG. 5

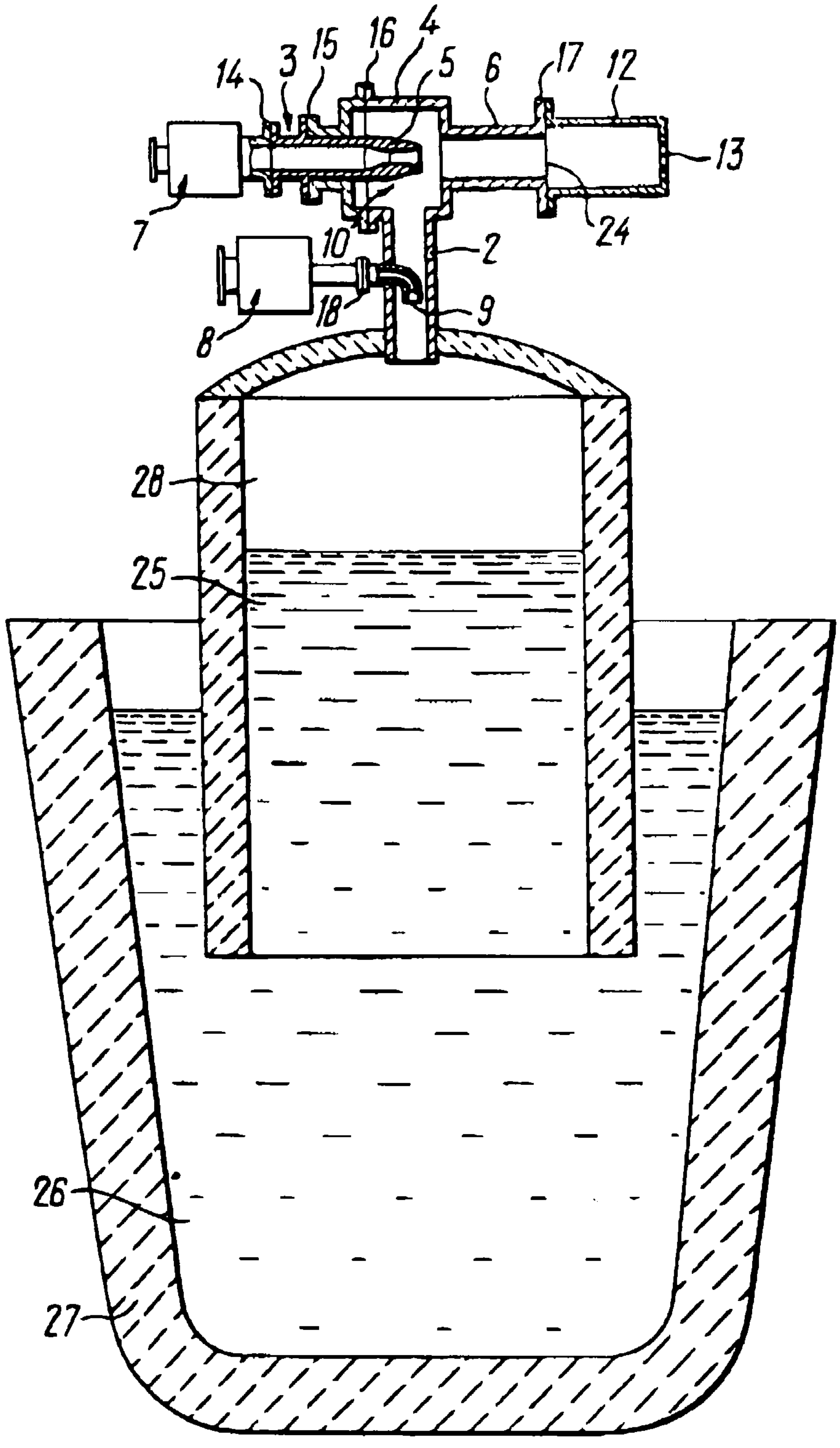

FIG. 6

INTERNATIONAL SEARCH REPORT

International application No.
PCT/RU 94/00034

A. CLASSIFICATION OF SUBJECT MATTER

IPC5: C 21 C 7/10

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)

IPC5: C 21 C 7/10, C 22 B 9/04, B 22 D 27/15

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)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US, A, 3798025 (ALLEGHENY LUDLUM INDUSTRIES, INC.), 19 March 1974 (19.03.74), the abstract, Fig.1	1-3
A	US, A, 4057421 (SUMITOMO METAL INDUSTRIES LIMITED), 8 November 1977 (08.11.77)	1-2
A	DE, B1, 1236541 (FRIEDRICH KOCKS), 16 March 1967 (16.03.67), the claims	1-3
A	DE, B1, 1458935 (STANDARD-MESSO DUISBURG, GESELLSCHAFT FUR CHEMIETECHNIK MBH & CO), 25 May 1972 (25.05.72), the claims, Fig.1	1-3
A	EP, A2, 0151872 (J. MULCAHY ENTERPRISES INCORPORATED), 21 August 1985 (21.08.85), the abstract	1-3

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

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"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
20 September 1994 (20.09.94)	22 September 1994 (22.09.94)
Name and mailing address of the ISA/RU Facsimile No.	Authorized officer Telephone No.

Form PCT/ISA/210 (second sheet) (July 1992)