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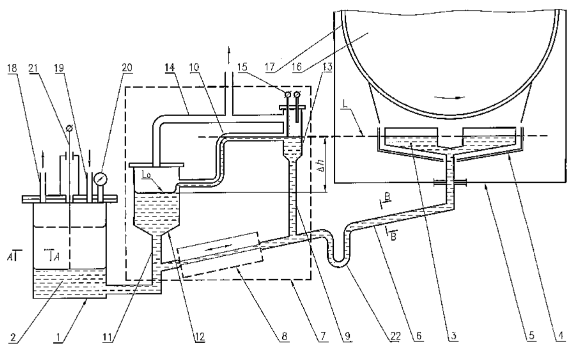
(54) **Title:** METHOD AND APPARATUS FOR VACUUM DEPOSITION BY VAPORIZING METALS AND METAL ALLOYS



(57) **Abstract:** The invention relates to a method and device for vacuum deposition by thermal evaporation of metals and alloys. The offered device (figure 1) contains a melting crucible (1) with molten material (liquid metal) (2), one or several crucibles (3) of evaporation device (4) in a vacuum chamber (5), a heated liquid-metal pipe (6), connecting the said melting crucible to the said evaporation crucibles through a magnetohydrodynamic (MHD) circuit (7) of static melt pressure. The circuit (7) is provided with an MHD pump (8) and incorporates the liquid-metal pipe (6) sectors, which are adjacent to the MHD pump, heated liquid-metal pipes (9, 10) and (11), a heated reservoir (13), connected to the liquid-metal pipe (6) sector before the MHD pipe through the liquid-metal pipe (11) and to an expander (12), installed in the said pipe (9), through the liquid-metal pipe (10). Spaces above the melt in the expander and reservoir are interlinked with a pipe (14), connected to a vacuum pumping system (not shown). Two electrical sensors (15) of the melt level L are installed in the expander. The melt level L in the expander and in the evaporator is delta h high relative the melt level L0 in the MHD circuit reservoir, i.e. the MHD pump should provide pressure delta h. The present technical solution allows increasing the stability of metals and alloys evaporation in long-run processes and thus increasing the productivity. The solution may be used for the deposition of various functional coatings in electronics, metallurgy, mechanical engineering. It is possible to evaporate zinc, magnesium, cadmium, lithium, zinc - magnesium alloy with this method.

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METHOD AND APPARATUS FOR VACUUM DEPOSITION BY VAPORIZING METALS AND METAL ALLOYS

FIELD OF THE INVENTION

The invention relates to vacuum deposition technique, mainly for coating roll substrates by thermal evaporation of metals and alloys in the commercial equipment of continuous or semicontinuous operation.

BACKGROUND OF THE INVENTION

Methods and devices for vaporizing materials for the deposition of corrosion-resistant coatings in metallurgy, active layers in manufacture of chemical current sources, various functional coatings in electronics and other technical fields are intensively investigated. Mainly metals and alloys, such as zinc, magnesium, cadmium, indium, zinc-magnesium, are used for these purposes.

Continuous evaporation of a great amount of these metals, measured dozens and sometimes hundred kilograms, is necessary in industrial processes. It is difficult to sustain such amounts of evaporated materials at the temperature of evaporation inside the vacuum chamber (as a rule, it is the temperature not lower than 500 °C). Therefore there is the problem of continuous feeding the evaporated material into the chamber without vacuum interruption during a lengthy period.

Well known methods of feeding the materials as rods, wire, granules or powder, are of little use for solving such task. The matter is that when the substance arrives directly into the evaporation device as powder or granules, fast heating of the substance to the evaporation temperature takes place, accompanied with release of gases, adsorbed and dissolved on the surface and inside the powder particles and granules. It has an adverse effect on quality of the coatings, especially of such active metals as lithium. Feeding the substance as wire or rods is also accompanied by release of gases, though to a lesser degree. Apart from that, the deposition process should inevitably be interrupted for replenishment of the reserve of wire or rods.

Methods of feeding the substance in a molten state into the evaporation device are substantially devoid of the above-mentioned drawbacks. Advantages of the evaporator replenishment with liquid metal are most fully implemented in vaporizing low-melting-point metals, such as lithium, indium, zinc, cadmium and partly magnesium.

A Russian patent application of G. Goncharov 93026154 of 27.12.1996 discloses an apparatus for liquid metal feeding into the evaporation device. The said apparatus contains a metal melting furnace, located outside the vacuum chamber, an evaporation device, located inside the vacuum chamber, and a pipeline, connecting the said furnace and evaporation device. The molten metal in the furnace is under atmospheric pressure. Feeding the molten metal into the evaporator is accomplished by the pressure difference between the vacuum chamber and the environment. The metal level in the evaporator is determined by the balance between, first, sum of atmospheric pressure and pressure of the metal column in the melting furnace, and second, pressure of the metal column in the feeding pipeline and the evaporator.

During operation of the evaporation device without substance replenishment in the melting crucible the melt level is decreasing both in the furnace crucible and the evaporation device. In the device under consideration it causes decrease of the evaporator productivity, because a part of the generated vapour is condensed on the evaporation crucible walls and does not arrive directly onto the substrate. Consequently, as metal is consumed and the metal level in the evaporator changes the evaporation rate will come down and the coating thickness on different areas along the substrate, e.g. film or foil roll, will vary.

Though the inventor mentions a stabilizer of the melt level in the evaporation device the capabilities of such stabilizer seem to be rather limited.

It is necessary to emphasize that replenishment systems on the basis of atmospheric pressure action are strongly limited by the density of used melts. Thus, for feeding zinc, indium and cadmium the melt level difference in the melting crucible and the evaporation device should be not less than 2.0....2.5 meters, for magnesium it should be 6 meters, while for lithium it will be 19 meters (!). Apart from that, contact of any metal with atmosphere is undesirable because of its oxidation and accumulation of slag, but for lithium it is totally inadmissible owing to its instant ignition.

Another solution of the problem of feeding liquid metals is based on elimination of the contact of the molten metal with atmospheric air in the furnace crucible by its sealing and pumping out. It allows minimizing overall dimensions of the feeding system and improving purity of the melt in the melting crucible. For example, such a device is described in the paper of E. Yadin "Deposition of Coatings or Free Foils of Sublimating Metals", SVC 40th annual Technical Conference Proceedings, 1997, p. 69. In this device the molten magnesium is fed from the melting furnace into the evaporation device by cutting the pump-down of the space above the melt in the melting furnace and controlled admission of inert gas into the said space. Experience of this device operation by the applicant of the present patent application has shown that wetting of the walls of the liquid metal pipeline and the evaporation element changes at relatively small temperature variation of the said pipeline and element. Therefore it is necessary to adjust and sustain the inert gas pressure in the melting crucible with high accuracy to exclude overfilling of the evaporation device. It requires manufacture and use of rather complicated regulation systems, what is a drawback.

Other solutions are based on providing the melt level in the evaporator by sustaining the melt level in the furnace crucible with mechanical aids when the evaporator and the said crucible are connected with each other. Thus, a method of molten metal feeding by elevating the melting furnace, controlled with a sensor of metal level in the evaporator, is described in a Japanese laid-open application of Sekiguchi Yasuaki 62-267470 of 20.11.1987. As a rule, the furnace should have a big reserve of metal, sufficient for implementation of the long-run evaporation cycles, e.g., during one or several working shifts. In its turn heavy weight of the furnace causes grand problems for the furnace lifting drive, as well as for adjusting and sustaining the melt level in the evaporator.

Technical solution on the basis of sustaining the melt level in the evaporator with mechanical aids, published in a Japanese laid-open application of Fukui Yasushi et al 09-053173 of 25.02.1997 "Method of Stably Feeding Evaporating Material", was considered as the prior art.

The prior art device contains the melting crucible, liquid metal pipeline, evaporator, installed in the vacuum chamber, instrumentation of measurement of the melt level in the evaporator, a body, immersed into the melting crucible melt, facilities of the evaporator melt level control, facilities of controlling the depth of the said body immersion. As the deposited substance is consumed, the said body is immersing into the melt by the signals of the said measurement instrumentation, thus the melt level in the melting crucible and, correspondingly, in the evaporator, connected to it with the liquid metal pipeline, remains stable.

The prior art technical solution has substantial drawbacks.

It is quite obvious that performance capabilities of such system are limited by the volume of the immersed body.

After full immersion of the body into the melt it is necessary to stop the process, elevate the body into the initial position, cool the melting crucible with the molten metal residues, admit air and load the crucible with a new portion of metal.

Installation of the melt level sensors in the evaporator is another drawback of the prior art. Monitoring the melt level directly in the evaporator, whose working temperature is above the metal melting temperature and whose environment is filled with metal vapours, requires special protection of the sensors, using the materials, resistant to such conditions.

One more drawback of the prior art is preconditioned by the procedure, when both melting and feeding of metal are made from the same vessel, namely the melting crucible. It means that some impurity substances, such as oxides, nitrides and other compounds, may be accumulated because of multiple loading the melting crucible with metal to be evaporated. The said impurity substances together with the melt can come in the evaporator and further onto the substrate, what depresses the coating quality.

SUMMARY OF THE INVENTION

The object of the present invention is to avoid the above said drawbacks and provide vacuum deposition at constant productivity due to constancy of the melt level in the evaporator irrespective of the evaporated substance amount. For achievement of the said object a magnetohydrodynamic (MHD) circuit, including at least one reservoir, a system of pipelines and an MHD pump, is arranged between the melting crucible and evaporator.

Separation of the operations of melting and partial refining of the evaporated metal together with sustaining constancy of the melt level in the evaporator crucibles allow combining one constant pressure circuit with several melting crucibles, which may be put out of operation periodically for entire cleaning from slag and accumulated impurities. With all this going on the evaporation system operation is not interrupted.

BRIEF DESCRIPTION OF DRAWINGS

Basic elements and some embodiments of the offered invention are shown graphically on figures 1 – 2, while some elements of the technical solution are given more in detail on figures 3-4. On figure 1 the preferred embodiment of the invention is shown.

Figure 2 presents the alternative simplified embodiment of the technical solution when deposition cycles are relatively short and there is no necessity of periodical replenishment of the system with molten metal.

On figures 3-4 the systems of heating and cooling the liquid metal pipelines and corresponding vessels are shown.

DETAILED DESCRIPTION OF THE INVENTION

The suggested device contains a melting crucible 1 with molten material (liquid metal) 2 to be evaporated, one or several crucibles 3 of an evaporation device 4 in a vacuum chamber 5, a heated liquid-metal pipe 6, connecting the said melting crucible to the said evaporation crucibles through an MHD circuit 7 of static melt pressure.

The circuit 7 is provided with an MHD pump 8 and incorporates the liquid-metal pipeline 6 sectors, which are adjacent to the MHD pump, liquid-metal pipelines 9, 10 and 11, a heated reservoir 12, connected with the liquid-metal pipe 11 to the liquid-metal pipe 6 sector before the MHD pump and with the liquid-metal pipe 10 to an expansion tank 13, installed in the pipeline 9. Spaces above the melts in the reservoir 12 and expansion tank 13 are connected through a pipe 14 to the vacuum pumping system (not shown). Two electrical sensors 15 of a melt level L are installed in the expansion tank. The melt level L in the expansion tank and in the evaporator is Δh high relative the melt level L_0 in the MHD circuit reservoir, i.e. Δh is the operating pressure of the MHD pump.

In the preferred embodiment a substrate holder 16 is in the form of a cooled rotatable drum, while a substrate 17, to be coated, is roll material, e.g. polymeric film or metal foil, though the present invention is applicable also for other types of the substrate with another design embodiment of its fixation and/or transportation during the deposition process.

The melting crucible 1 is connected to the vacuum pumping system (not shown) through a branch pipe 18 and to an inert gas (e.g. argon) feeding system (not shown) through a branch pipe 19 and provided with a gage 20 to measure pressure in the space above the melt, as well as with a sensor 21 to measure the melt level.

The liquid metal pipe 6 may be equipped with a U – form elbow 22 and controlled system of emergency cooling (not shown) as an additional safety means.

When deposition cycles are relatively short, there is no necessity of periodical replenishment of the static pressure circuit 7 during the cycle by loading the evaporated metal into the melting crucible, as described above. In this case the simplified embodiment may be used, as shown on figure 2. In the said embodiment the heated reservoir 12 of the static pressure circuit 7 is excluded and the melting crucible 1 with its sensors is installed directly in the said circuit 7 instead of the reservoir 12. In this case the expansion tank 13 is connected directly to the melting crucible 1 though the liquid metal pipeline 10, while the pipe 14 connects the spaces above the melt in the melting crucible and expansion tank to the vacuum pumping system.

The melting crucible 1, reservoir 12 (when it is used) and liquid metal pipelines 9, 10 and 11 are heated electrically by common methods. Apart from that, the said assemblies are provided with cooling channels, preferably with air coiled pipes. The said channels may be also fluidic, yet manufacture of such channels is more complicated and sometimes absolutely unacceptable for reasons of safety (e.g. in lithium evaporation). Using the cooling channels provides the possibility to increase the productivity due to reducing run-to-run operations. For simplification the heating and cooling systems are not shown on figures 1 and 2.

A cross section AA (figure 1 and 2) of the system of electrical heating and air cooling the melting crucible 1 and the reservoir 12 is presented on figure 3. The system includes walls 23 of the melting crucible 1 or reservoir 12, a resistive heater 24, electrically insulated from the said walls, thermal insulation 25 and air cooling pipes 26.

A cross section BB (figure 1 and 2) of the system of electrical heating and air cooling the liquid metal pipelines 9, 10 and 11 is shown on figure 4. The system includes liquid metal 2, walls of the liquid metal pipelines 27, a heater 28, thermal insulation 29, air cooling pipes 30, elements 31 of bonding the air cooling pipes to the liquid metal pipelines (e.g. welds).

The device operates in the following way.

The deposition cycle starts after filling-up the MHD circuit 7 and its reservoir 12 with metal 2 from the melting crucible 1. Meanwhile it is possible to cool the melting crucible 1, open it and load next portion of metal without interrupting the deposition process. Of course, it is necessary to cool the pipeline section between the melting crucible and MHD circuit below the metal melting point beforehand.

The melt level in the evaporator 4 is monitored by the melt level in the MHD circuit 7, where the melt temperature is above the metal melting point only by 30...50 °C and practically there are no metal vapours, in that way operation reliability of the melt level sensors and the system in general is provided.

The reservoir 12 of the MHD circuit 7 is filled with liquid metal 2 by any known method. For example, filling-up the circuit by displacement of the melt from the melting crucible 1 due to pressure difference, generated by admittance of inert gas (e.g. argon) through the branch pipe 19 into the space above the melt (figure 1). When the MHD pump starts, filling the liquid metal pipeline 6 and expansion tank 13 begins. If pressure is insufficient, the pipeline may be filled only at the part of its height. In this case there is no melt circulation. When the MHD pressure increases, the melt starts to fill the significant part of the expansion tank 13 gradually. When the melt runs up to the level L, its flow starts along the liquid metal pipeline 10 into the reservoir 12. Thus the melt circulation in the static pressure circuit 7 is set in. The melt flow speed in the expansion tank 13, connected to the liquid metal pipeline 6, decreases drastically, approaching to the speed, characteristic for laminar flow.

The sensors 15 of the melt column height in the static pressure circuit provide the signal of pressure, developed by the MHD pump, so that the expansion tank 13 and the whole circuit 7 were not overfilled in case of excessive pressure. The said sensors presence directly in the circuit 7 together with common aids allows providing constancy of the pressure, developed by the MHD pump, and, consequently, the melt level in the circuit. If now to heat the liquid metal pipeline 6 sector, connecting the circuit 7 to the crucible 3 of the evaporator 4 to the corresponding temperature, the melt starts to fill the crucibles, at that the melt levels both in the circuit and evaporator will be equal, because operating pressure of the MHD pump in the liquid metal pipelines 6 and 9 is the same.

It is known from the experience of the present invention applicant that pressure F in the MHD pump channel, which is required for sustaining the necessary melt level, is determined by the expression:

$$F \geq \rho \bullet g \Delta h,$$

where ρ is melt density,
g is gravitational acceleration,
Δh is operating pressure of the MHD pump.

Owing to decrease of the liquid metal level in the system due to evaporation (figures 1 and 2) the melt level L_0 tends to constant lowering. Yet the sensors 15 allow compensating the said lowering by signalling to the MHD pump to increase pressure. This procedure may be automated by known means.

A distinctive feature of the MHD pump, which may be inverted to reversal pressure practically in a moment, is an additional advantage of the offered technical solution. This feature becomes useful in evaporation with alkaline metals, whose melt contact with air is dangerous.

Therefore in emergency situations, caused by pressure increase in the deposition chamber, it is possible to empty the evaporator crucibles quickly by corresponding commutation of the MHD pump 8.

In operation with alkaline metals seal failure of the melt circulation and feeding system is also dangerous. It is fraught with spreading of large amount of the melt into the vacuum chamber. Therefore it is offered to fit out the pipeline 6 of the melt feeding into the evaporator 4 with the U-shaped elbow 22 with the emergency cooling system (not shown) for minimization of the above said after-effects. This member allows clogging the pipeline quickly with the stopper of solidified melt at the command of the system.

Example. The device, shown graphically on figure 1, was embodied in the design of the vacuum machine for lithium coating polymeric film by the method of lithium thermal evaporation. The evaporator of four steel crucibles has been installed in the vacuum chamber of the machine. The lithium melting crucible has been installed outside the vacuum chamber and connected to the evaporator with the liquid metal pipeline. The circuit of liquid metal static pressure, consisting of the MHD pump, reservoir and system of pipes, has been arranged on the above said liquid metal pipeline. In its upper part the circuit had the expansion tank, where two ground-insulated thin rods were inserted and connected to the power and control unit of the MHD pump. The rods could travel vertically at the range of 10-15 mm.

The circuit was manufactured in such a way that the middle of the horizontal liquid metal pipeline, coming out from the expansion tank, was at the level, equal to the desired level of filling-up the evaporator crucibles with lithium melt. In its lower part the circuit is connected to the lithium melting crucible with the liquid metal pipeline.

All members of the evaporator feeding system had sectioned heaters of indirect electrical heating and wall temperature sensors. The melting crucible was located in the room, adjacent to the vacuum machine, where relative air humidity was sustained not higher than 2%. The lithium ingots, weighing 840 g each, were loaded into the melting crucible at 2% relative air humidity. Initial volume of lithium was about 6.3 litres.

Pumping out the melting crucible started after its sealing. The vacuum chamber and static pressure circuit were pumped out simultaneously with the melting crucible pumping. When pressure in the melting crucible space achieved 2 Pa, its heating started and continued to temperature 250 °C. Monitoring the temperature of the melting crucible wall allowed determining the starting moment of lithium melting, while the extent of the crucible filling with the melt was determined by the level sensor signal.

Pumping out the melting crucible continued after melting accomplishment till removal of gases, dissolved in lithium. Simultaneously the circuit was heated to 250 °C and the power unit of the MHD pump was live. After three hours the pumping out line of the melting crucible was cut and argon admittance through a fine adjustment inlet valve started. Starting moment of lithium arrival into the circuit was registered by the signal of the melting crucible level sensor. Lowering the level sensor to rated depth allowed determining the moment, when filling the circuit with lithium was accomplished. The MHD pump pressure was gradually increased till the moment of actuation of the sensor, installed in the circuit expansion tank. Then heating of the melting crucible and liquid metal pipeline, connecting it to the static pressure circuit, was switched off, heating of the pipeline, feeding lithium into the evaporator, was switched on and continued to 250 °C. When the temperature setpoint was achieved, filling-up the evaporator crucibles with the melt was observed through the viewing device on the vacuum chamber.

After heating the lithium-filled crucibles to temperature 580 °C the cycle was started for lithium deposition onto PET film 25 micron thick, pre-coated with "Inconel 400" underlayer 40 nm thick. The process continued 5 hours without interruption; 300 m of the coated product was manufactured. It was observed periodically that lithium level in the evaporator crucibles remained invariable.

After accomplishment of lithium deposition heating of the crucibles was switched off and lithium was cooled to 300 °C. Then the MHD pump was reversed and lithium was drained back into the circuit during one minute. Further the liquid metal pipeline, connecting the circuit to the evaporator, was switched off and compressed air was supplied to cool its walls and crucibles. After achievement of the temperature level 50-60 °C dry air was admitted into the vacuum chamber, the roll of coated product was unloaded, a new roll was loaded and a new cycle was initiated.

The U-shaped elbow on the pipeline of lithium feeding into the evaporator was constantly filled with metal and after cooling the liquid metal pipeline before air admittance into the chamber the elbow functioned as a valve, preventing air penetration into the hot circuit,

Similarly five deposition cycles were carried out with one lithium load and 1500 m of the coated product for chemical current sources manufactured. Lithium thickness measurement along all coated rolls showed that its spreading did not exceed 5%, what is typical for any vacuum deposition processes. With all this going, monotone decrease of thickness due to lowering of the melt level in the crucibles without feeding was not observed.

WE CLAIM

1. A method for vacuum deposition by vaporizing metals and alloys, including melting the deposited substance in a melting crucible, installed outside a vacuum deposition chamber and connected with liquid metal pipelines to the evaporator, installed in the said vacuum chamber, feeding the said substance melt into the said evaporator, monitoring and sustaining constancy of the melt level in the evaporator, said method characterized by MHD-pump feeding the said melt into the evaporator, when the melt level in the evaporator is stabilized with an MHD circuit of static pressure and constancy of the melt level in the evaporator are monitored and sustained by signals of a sensor, installed in the said circuit.

2. An apparatus for vacuum deposition by vaporizing metals and alloys, including a vacuum chamber with pumping means, an evaporator inside the said vacuum chamber, the said evaporator comprising one or several evaporation crucibles, a melting crucible outside the vacuum chamber, the said melting crucible connected with the evaporator by a liquid metal pipeline, a means of measuring and adjusting the melt level in the evaporator, said apparatus characterized by the MHD circuit of the melt static pressure.

3. The apparatus of claim 2, wherein the MHD circuit of the melt static pressure consists of an inductive MHD pump, a system of liquid metal pipelines and a reservoir with a reserve of metal to be evaporated, the said reservoir is installed between the melting crucible and the evaporator.

4. The apparatus of claim 2, wherein the MHD circuit of the melt static pressure consists of the said melting crucible, the inductive MHD pump and the system of liquid metal pipelines.

5. The apparatus of claim 2, wherein the said MHD pump is reversal.

6. The apparatus of claim 2, wherein the walls of the melting crucible, reservoir and liquid metal pipelines are provided with air cooling channels.

7. The apparatus of claim 2, wherein a sector of the liquid metal pipeline between the evaporator and the MHD circuit of the melt static pressure contains a U-form elbow with a controlled system of emergency cooling.

AMENDED CLAIMS

[received by the International Bureau on 30 September 2005 (30.09.2005); originally filed claims 1-4 and 6-7 are amended; claim 5 has been cancelled]

1. A method for vacuum deposition by vaporizing metals and alloys, including melting the metals and metal alloys in a melting crucible, installed outside a vacuum deposition chamber and connected with liquid metal pipelines to an evaporator, installed in the said vacuum chamber, feeding the said metal into the said evaporator, monitoring and sustaining constancy of the liquid metal level in the evaporator, said method characterized by providing the liquid metal flow by magnetohydrodynamic pumping along the liquid metal circuit, stabilization of the liquid metal level in the said circuit, feeding the said liquid metal into the evaporator from the said circuit and providing constancy of the liquid metal level in the evaporator by the stabilized liquid metal level in the said circuit.

2. An apparatus for vacuum deposition by vaporizing metals and metal alloys, including a vacuum chamber with pumping means, an evaporator inside the said vacuum chamber, the said evaporator comprising one or several evaporation crucibles, a melting crucible outside the vacuum chamber, the said melting crucible connected with the evaporator by a liquid metal pipeline, a means of stabilization of the melt level in the evaporator, said apparatus characterized by a liquid metal circuit, containing an inductive magnetohydrodynamic pump, a closed system of heated pipelines and an expansion chamber, a level sensor in the said expansion chamber, the said circuit is connected to the said evaporator with a heated pipeline.

3. The apparatus of claim 2, wherein the said liquid metal circuit contains a reservoir with a reserve of metal to be evaporated and the said melting crucible is installed outside the said liquid metal circuit.

4. The apparatus of claim 2, wherein the said melting crucible is installed in the said liquid metal circuit.

6. The apparatus of claim 2, wherein the walls of the components, containing liquid metal, are provided with air cooling channels.

7. The apparatus of claim 2, wherein the liquid metal pipeline between the evaporator and the liquid metal circuit contains a U-form elbow with a controlled system of emergency cooling.

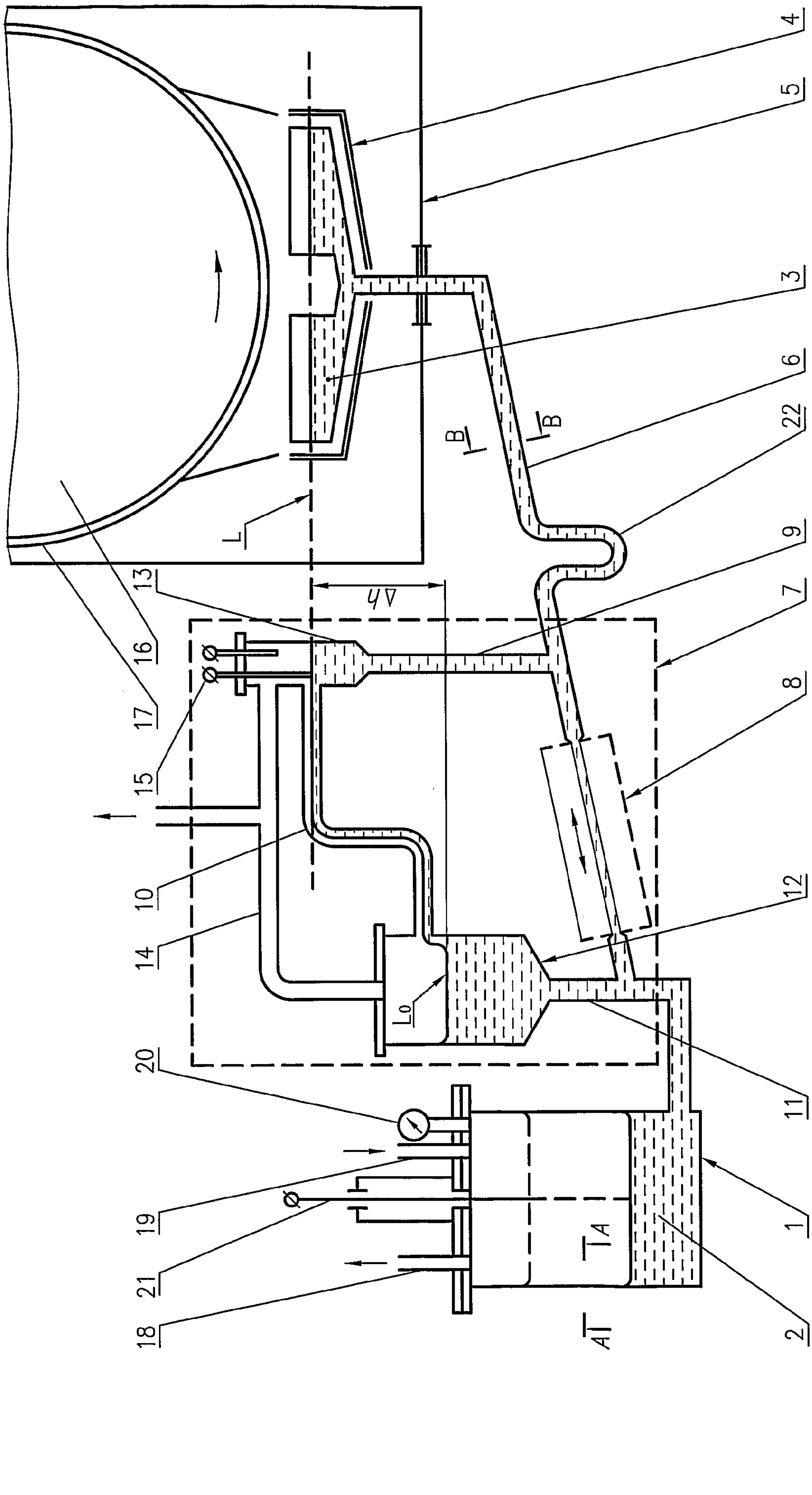

Fig.1

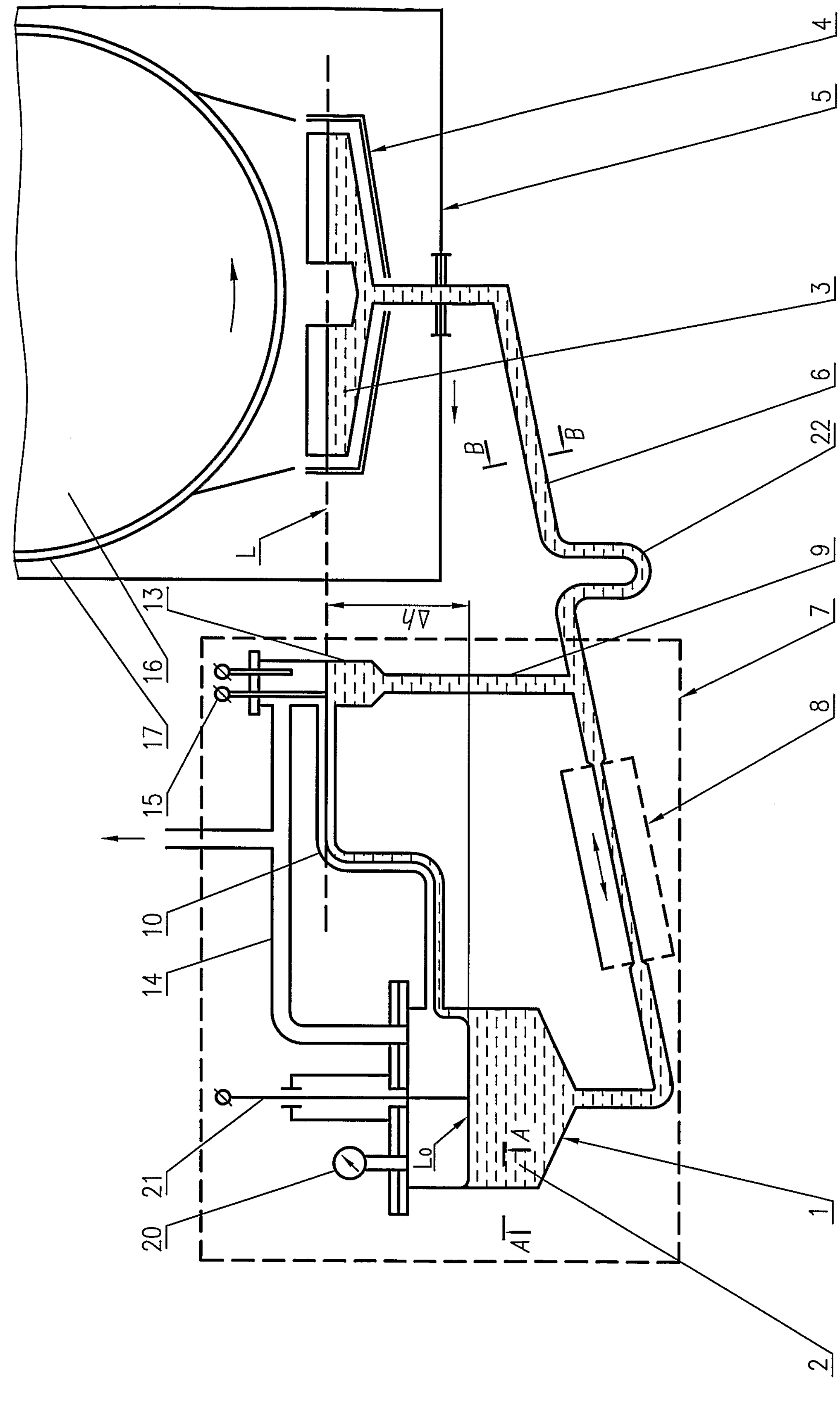

Fig.2

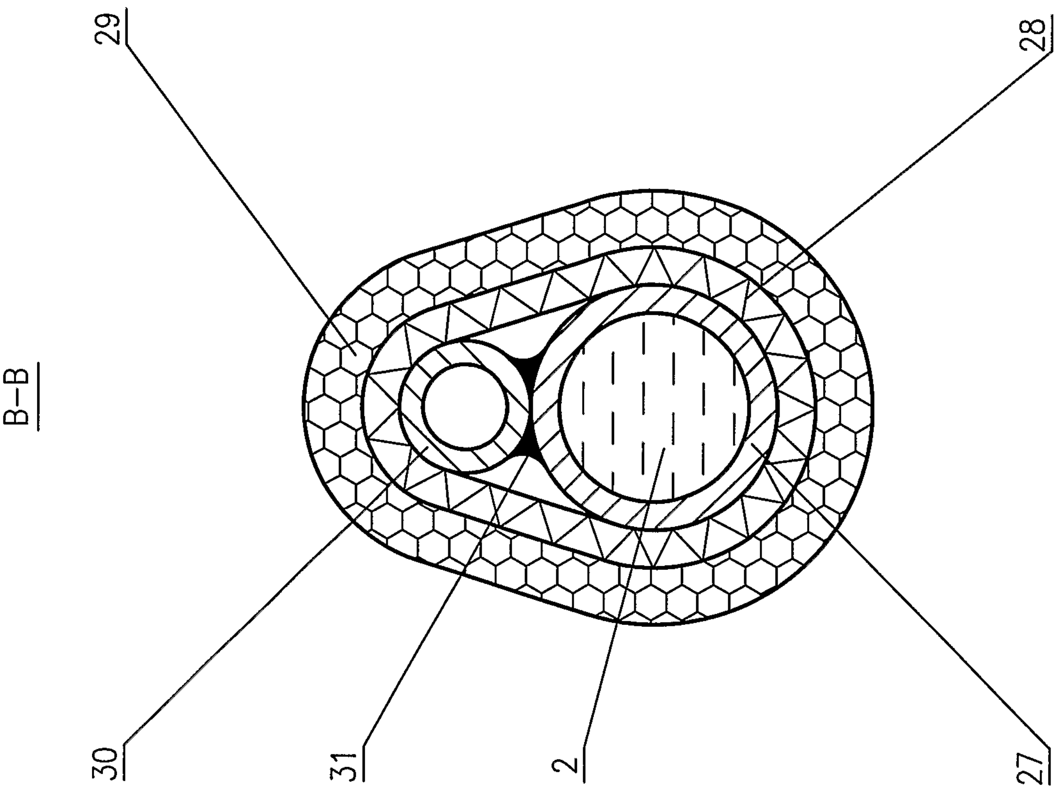

Fig.4

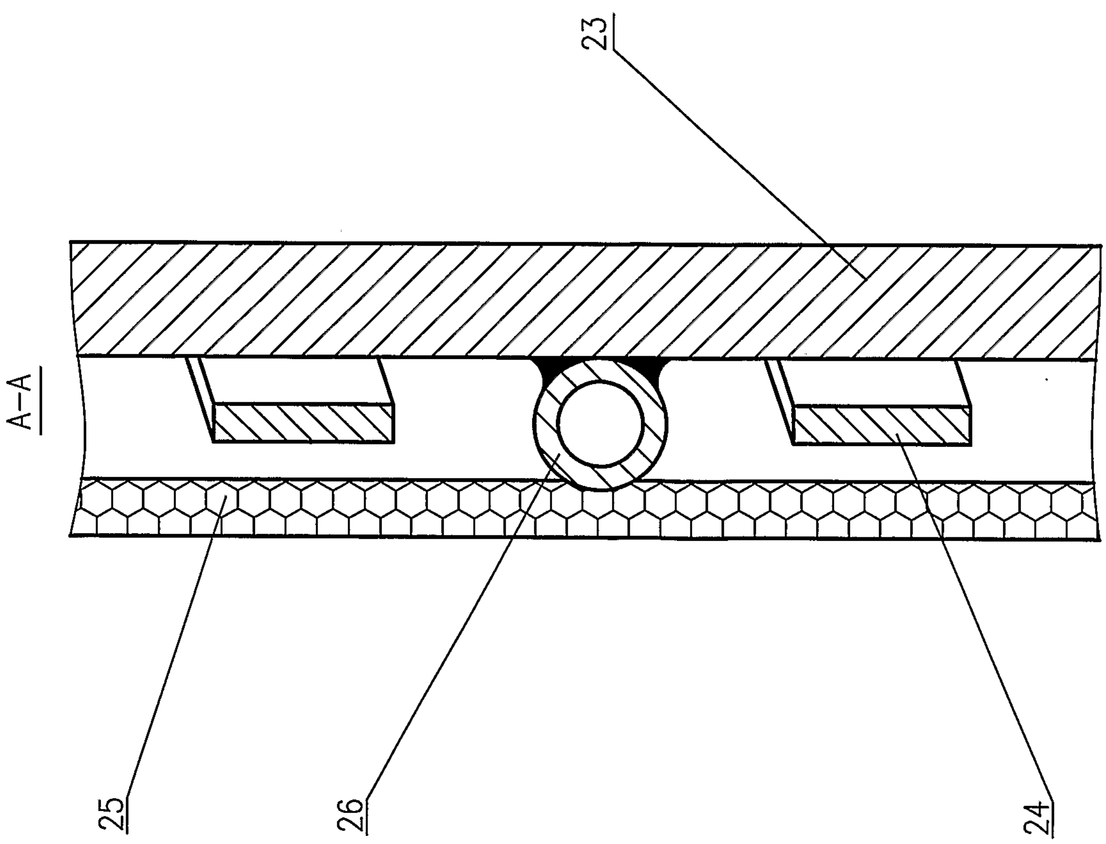

Fig.3

INTERNATIONAL SEARCH REPORT

International Application No
PCT/LV2005/000005

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C23C14/56 C23C14/54 C23C14/52 C23C14/24 H02K44/00

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)
IPC 7 C23C H02K

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 practical, search terms used)

EPO-Internal, INSPEC, PAJ, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	PATENT ABSTRACTS OF JAPAN vol. 012, no. 155 (C-494), 12 May 1988 (1988-05-12) -& JP 62 267470 A (MITSUBISHI HEAVY IND LTD; others: 01), 20 November 1987 (1987-11-20) cited in the application	1,2
A	abstract	3-7
A	PATENT ABSTRACTS OF JAPAN vol. 1997, no. 06, 30 June 1997 (1997-06-30) -& JP 09 053173 A (NISSHIN STEEL CO LTD), 25 February 1997 (1997-02-25) cited in the application abstract	1-7

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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

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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.

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Date of the actual completion of the international search	Date of mailing of the international search report
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European Patent Office, P.B. 5818 Patentlaan 2 NL – 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Teppo, K-M

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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