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(54) **METHOD OF OPERATING A FABRICATION PLANT AND FABRICATION PLANT**

(57) The present invention relates to a method of operating a fabrication plant (1000), the fabrication plant (1000) comprising a fabrication section (200) and a gas provision section (100), the fabrication section (200) being adapted to be operated using nitrogen provided by the gas provision section (100) and using helium, the gas provision section being adapted to perform a cryogenic air separation process, and a first gas mixture comprising nitrogen, one or more noble gases and at least one first gas component being withdrawn from the fabrication

plant (1000) as an offgas. According to the present invention, at least a part of the first gas mixture and/or at least a part of a second gas mixture formed from the first gas mixture and comprising nitrogen, the one or more noble gases and at least one second gas component formed from the at least one first gas component is supplied to the cryogenic air separation process in the gas provision section (100). A corresponding fabrication plant (1000) is also part of the present invention.

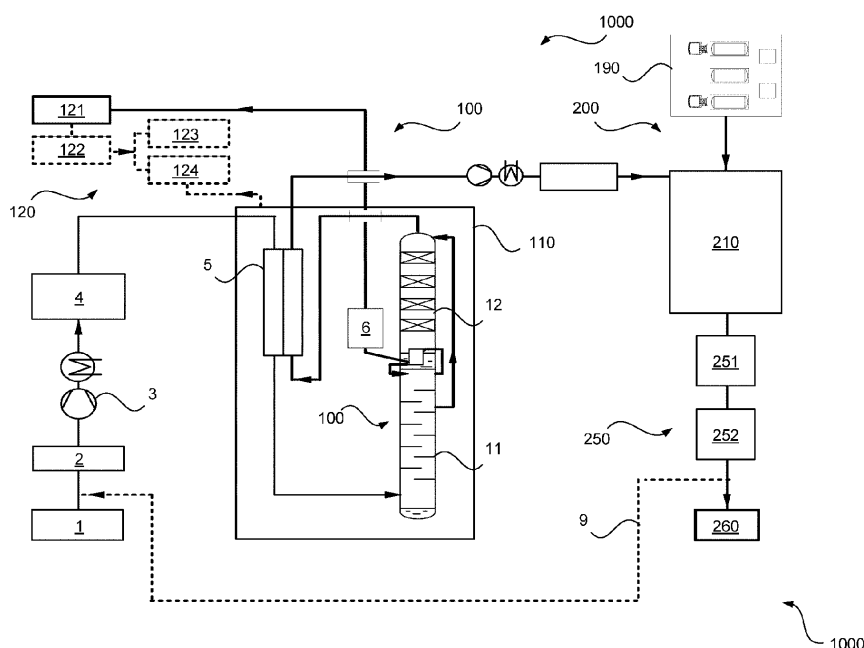


Fig. 1

Description

[0001] The present invention relates to a method of operating a fabrication plant and to a fabrication plant according to the preambles of the independent claims.

Background of the invention

[0002] Large semiconductor fabrication plants (also referred to as "fabs") are operated with considerable amounts of nitrogen and noble gases such as helium, krypton, xenon and argon. Particularly the costs of the noble gases, particularly of helium, are substantial and a recovery of helium is therefore desired.

[0003] Offgases of semiconductor manufacturing plants may be contaminated, however, depending on the specific manufacturing process, with components such as oxygen, fluorine, chlorine, ammonia, carbon monoxide, carbon dioxide, nitric oxide, nitrous oxide, silane, disilane, trimethylsilane and halogenated compounds like boron trichloride, dichlorosilane, trichlorosilane, hexachlorosilane, monomethylsilane, chlorine trifluoride, difluoromethane, hexafluoro-1,3-butadiene, hexafluoroethane, hydrogen bromide, tungsten hexafluoride, hydrogen chloride, hydrogen fluoride, methyl fluoride, fluoromethane, nitrogen trifluoride and octofluorocyclobutane.

[0004] Despite the high costs of noble gases and helium in particular, such offgases are conventionally not regenerated or recycled because of the contaminants being present. Instead, the offgases are typically incinerated at high temperatures to destroy the contaminants, thereafter sent through a scrubber, and subsequently released as substantially cleaned offgases into the atmosphere.

[0005] While the present invention is described with a focus on semiconductor fabrication, it may likewise be used in other processes and fabrication plants where process steps or units are operated with at least nitrogen and one or more noble gas such as helium, and wherein, from the process steps or units, gas mixtures comprising at least a part of the nitrogen and the noble gas(es) and other components, also referred to as contaminants herein, are withdrawn.

[0006] The present invention has the object to operate such processes and fabrication plants in a more effective manner, reducing losses of valuable gases.

Disclosure of the invention

[0007] According to the present invention, a method of operating a fabrication plant and a fabrication plant comprising the features of the independent claims are provided. Preferred embodiments of the present invention are the subject of the dependent claims and the description that follows.

[0008] According to the present invention, the fabrication plant comprises a fabrication section and a gas pro-

vision section, the fabrication section being adapted to be operated using nitrogen provided by the gas provision section and using one or more noble gases, the gas provision section being adapted to perform a cryogenic air separation process, and a first gas mixture comprising nitrogen, the one or more noble gases and at least one first gas component being withdrawn from the fabrication plant as an offgas. As mentioned, the present invention is not limited to particular types of fabrication plants but is particularly useful in connection with semiconductor fabrication plants.

[0009] According to the present invention, the one or more noble gases may be selected from the group of helium, neon, argon, krypton and xenon, even if the focus of the description will be placed on helium hereinbelow for reasons of conciseness. If, therefore, an aspect of the present invention is described in connection with helium, the description is equally valid for other noble gases as well.

[0010] An essential aspect of the present invention is that if such noble gases are supplied to a plant of the kind mentioned, particularly for inertisation and purging purposes, such noble gases will not be consumed yet will leave the plant as noble gases and can therefore be recovered from an offgas by a (cryogenic) air separation process.

[0011] The gas provision section is in particular adapted to provide the nitrogen as ultrapure gaseous nitrogen in pressurized form, but may also be adapted to provide other fluids in liquid or gaseous form to the fabrication section, such as, but not limited to, xenon, krypton, argon and oxygen. Different gases or gas mixtures may be used in different steps of a fabrication process implemented in the fabrication section. The "first" gas mixture may be withdrawn from the fabrication section at any position.

[0012] According to the present invention, at least a part of the first gas mixture and/or at least a part of a second gas mixture formed from the first gas mixture and comprising nitrogen, the one or more noble gases and at least one second gas component formed from the at least one first gas component is supplied to the cryogenic air separation process in the gas provision section. As will be understood, the present invention is not limited to supply only one gas mixture which is withdrawn from the fabrication process to the gas provision section, and the feed position into the gas provision section is not particularly limited but rather depends on the composition of a the gas mixture(s).

[0013] In other words, the present invention proposes feeding a mixture of nitrogen and one or more noble gases, air, carbon dioxide and possibly other impurities to an air separation process, particularly to the suction side of the main air compressor of an air separation unit which supplies a corresponding fabrication section with gaseous nitrogen and optionally other air gases.

[0014] Said mixture may particularly be taken from a scrubber installed in or downstream of the fabrication section and may particularly contain 100 to 5000 ppm of

one or more noble gases such as helium, krypton, xenon, neon and argon. As scrubbers typically dilute the offgas heavily, any optimisation/reduction in dilution improves the efficiency of the proposed solution.

[0015] The production of air products in liquid or gaseous state by cryogenic separation of air in air separation plants (air separation units) is well known and described, for example, in H.-W. Haring (eds.), *Industrial Gases Processing*, Wiley-VCH, 2006, especially section 2.2.5, "Cryogenic Rectification".

[0016] The rectification columns of the column systems just mentioned are operated in different pressure ranges. Known double column systems comprise a so-called pressure column (also called high pressure column, medium pressure column or lower column) and a so-called low pressure column (upper column). The high pressure column is typically operated in a pressure range of 4 to 7 bar, especially at about 5.3 bar, whereas the low pressure column is operated in a pressure range of typically 1 to 2 bar, especially at about 1.4 bar. In certain cases, higher pressures can also be used in both rectification columns. Air separation units supplying the fabs mentioned at the outset, for example, are often operated with a pressure of 7 to 13 bar in the pressure column and 3 to 5 bar in the low pressure column. The pressures given here and below are absolute pressures at the top of the respective columns.

[0017] Air separation plants of the classical type have column systems which can be designed, for example, as two-column systems, especially double-column systems, but also as three- or multi-column systems. In addition to rectification columns for the recovery of nitrogen and/or oxygen in liquid and/or gaseous state, i.e. rectification columns for separation of nitrogen and oxygen, rectification columns can be provided for the recovery of other air components, in particular of noble gases.

[0018] When helium or neon is supplied to the cryogenic air separation process as a part of the first and/or second gas mixture, it rises with neon in the pressure column and can be recovered the usual way on the top of the pressure column or as a non-condensing gas in its head condenser as so-called crude helium-neon. The present invention is not limited to a specific form of recovery. Reference is made to expert literature such as Haring (see above), particularly section 3.4, "Recovery of Neon". The other noble gases (krypton, xenon, argon), which may also be part of the first and/or second gas mixture, typically move with the oxygen molecules and can be recovered the usual way as well. Reference is made to section 2.2.5 in Haring cited above and additionally to section 3.3, "Recovery of Krypton and Xenon".

[0019] In a particularly preferred embodiment, at least a part of the first gas mixture is treated in an offgas treatment unit to form the second gas mixture. Details as to the offgas treatment unit are further given below.

[0020] In a particularly preferred embodiment, the second gas mixture or its part which is supplied to the cryogenic air separation process is in such a case supplied

to the cryogenic air separation process together with feed air upstream of a main air compressor. This allows for the second gas mixture to be treated in all steps to which also a feed air stream is subjected in the cryogenic air separation process, particularly in a so-called prepurification unit.

[0021] The at least one second gas component may particularly include, besides the noble gas(es) mentioned, at least one of nitrogen, carbon dioxide, carbon monoxide, hydrogen chloride, hydrogen fluoride and water, such as typically obtained in combusting and thereafter scrubbing offgases of fabrication plants mentioned.

[0022] The at least one first gas component may, as a typical offgas include at least one of oxygen, fluorine, chlorine, ammonia, carbon monoxide, carbon dioxide, nitric oxide, nitrous oxide, silane, disilane trimethylsilane and halogenated compounds like boron trichloride, dichlorosilane, trichlorosilane, hexachlorodisilane, monomethylsilane, chlorine trifluoride, difluoromethane, hexafluoro-1,3-butadiene, hexafluoroethane, hydrogen bromide, tungsten hexafluoride, hydrogen chloride, hydrogen fluoride, methyl fluoride, fluoromethane, nitrogen trifluoride and octafluorocyclobutane.

[0023] The offgas treatment unit may be adapted to perform at least an incineration step and a scrubbing step, wherein the first gas mixture is at least in part supplied to the incineration step and wherein at least a part of the second gas mixture is withdrawn from the scrubbing step. The combustion step and the scrubbing step may be realized in any manner known in the art. The second gas mixture is particularly obtained as a head gas of a scrubber column.

[0024] The combination of high-temperature incineration and scrubbing using a counter-flow principle (packed tower etc.) allows to achieve removal efficiencies of 99% and better. The exhaust gas of a scrubber, i.e. the "second gas mixture" referred to above, typically contains humid air (with a reduced oxygen content), increased levels of carbon dioxide and approximately 1 to 10 ppm of hydrogen chloride and hydrogen fluoride.

[0025] According to the present invention, different scrubber vents may be combined to form a header, possibly with a buffer vessel. A blower and corresponding piping from the scrubber vent to the air intake of the air separation process may be used. Likewise, a control loop with bypass to the atmosphere in case of upset conditions (e.g. if concentration of impurities too high) may be implemented.

[0026] As mentioned, the gas provision unit used according to the present invention may be adapted to provide a component mixture predominantly comprising helium and neon and comprising at least one of nitrogen and hydrogen ("helium-neon concentrate").

[0027] Reference is made to the explanations already given above. The gas provision unit may particularly comprise a so-called helium-neon column as generally known, which may e.g. be supplied with a non-condensing part of a top gas of the pressure column or a different,

nitrogen-rich stream from the top of the pressure column.

[0028] The gas provision unit may, in such an embodiment, particularly be adapted to further process the component mixture predominantly comprising helium and neon and comprising at least one of nitrogen and hydrogen, particularly to further enrich such a gas mixture, for example as disclosed in EP 2 322 888 A1.

[0029] Helium-neon concentrates obtained in air separation units are usually not further processed on site, but transported to a special plant for obtaining a practically pure helium-neon mixture or pure helium and/or neon.

[0030] As discussed in EP 2 322 888 A1, a helium-neon concentrate is derived from the head fraction of a helium-neon column by partially condensing the head fraction in a head condenser and withdrawing the helium-neon concentrate as the remaining gaseous fraction from the head condenser of the helium-neon column. Liquid nitrogen from the upper section of the pressure column is used as the liquid refrigerant stream which evaporates in indirect heat exchange with the condensing overhead fraction in the overhead condenser of the helium-neon column. An only slightly superatmospheric pressure is used in the evaporation chamber of the top condenser of the helium-neon column so that the nitrogen evaporated in the evaporation chamber can subsequently be fed into the low-pressure column. Alternatively, the evaporated nitrogen can leave the plant via the main heat exchanger.

[0031] The pressure in the liquefaction space of the helium-neon column overhead condenser is equal to the operating pressure of the helium-neon column, which in turn is approximately equal to the operating pressure of the pressure column. Because of these pressures, which are defined by the other operating conditions of the process, a maximum neon content of about 50 mol% and a maximum helium content of about 18 mol% can be achieved in the helium-neon column overhead condenser. Concentration can generally be improved by reducing the operating temperature of the helium-neon column overhead condenser. However, this would require operating the evaporation chamber at a subatmospheric pressure and using a vacuum pump to exhaust the vapor formed there.

[0032] Therefore, as disclosed in EP 2 322 888 A1, and in an embodiment of the present invention, a precursor stream may be derived from the head fraction of the helium-neon column, the precursor stream may be compressed to a first elevated pressure which is higher than the operating pressure of the helium-neon column, and the precursor stream may be partially condensed under the first elevated pressure and subsequently subjected to a phase separation. At least a part of a portion of the precursor stream remaining gaseous during the partial condensation may be withdrawn as a product stream from the phase separation and recovered as helium-neon concentrate. Alternatively, a vacuum pump to lower the pressure of liquid nitrogen in a reboiler of the helium-

neon column can be installed.

[0033] According to a particularly preferred embodiment of the present invention, the gas provision unit may be adapted to provide argon or a component mixture predominantly comprising argon and comprising nitrogen, as generally known. The gas provision unit may also be adapted to provide krypton and/or xenon or a component mixture predominantly comprising krypton and xenon, as generally known.

[0034] In a particularly preferred embodiment of the present invention, the gas provision unit comprises a pre-purification unit adapted to remove the at least one first gas component at least in part from the first gas mixture or a part thereof supplied to the cryogenic air separation process and/or to remove the at least one second gas component at least in part from the second gas mixture or a part thereof supplied to the cryogenic air separation process.

[0035] As the composition of the waste noble gas(es) (i.e. the first or second gas mixture) is not necessarily known exactly, additional adsorbents and/or catalysts are advantageously installed in the prepurification unit, such as standard molecular sieve material to remove carbon dioxide, a hopcalite catalyst to remove carbon monoxide, activated alumina to remove residual hydrogen chloride (which may also be removed, due to its high solubility in water, via a direct contact air cooler stage), or activated carbon to remove residual hydrogen fluoride and volatile organic compounds.

[0036] In case that nitrogen products are specified with hydrogen purities in the ppb range, hydrogen is preferably removed in the prepurification unit catalytically. In this case, the crude helium-neon, if produced, is a mixture of nitrogen and helium, which saves the step of hydrogen combustion and subsequent water removal in subsequent purification processes. A hydrogen removal in the prepurification unit has no impact on the recovery of krypton, xenon and/or argon.

[0037] If an offgas containing helium and/or other inert gases can be sourced with sufficiently clean composition even before incineration or scrubbing, using this gas as (a partial) feedstock to air separation is even more advantageous as the concentration of helium and other inert gases is expected to be higher (less dilution from scrubbing).

[0038] The present invention may also comprise onsite helium-neon purification. Particularly in case of supply schemes for so-called giga-fabs (e.g. 300 kNm³/h to 400 kNm³/h gaseous nitrogen), the purification of helium (and potentially) other inert gases can be done on site (e.g. in combination with EP 2 322 888 A1).

[0039] The main air compressor of an air separation unit may be arranged in proximity to the scrubber(s) used. Depending on distance between the outlet of the scrubber(s) and the air intake of an air separation unit and the volumetric flow of offgas from scrubbers, installing the main air compressor in close proximity to scrubbers and piping the compressed air to the prepurification system

of the air separation unit can be particularly advantageous.

[0040] A fabrication plant comprising a fabrication section and a gas provision section is also part of the present invention, the fabrication section being adapted to be operated using nitrogen provided by the gas provision section and using one or more noble gases, the gas provision section being adapted to perform a cryogenic air separation process, wherein means are provided which are adapted to withdraw a first gas mixture comprising nitrogen, the one or more noble gases and at least one first gas component from the fabrication section as an offgas. According to the present invention, means are provided which are adapted to supply at least a part of the first gas mixture and/or at least a part of a second gas mixture formed from the first gas mixture and comprising nitrogen, the one or more noble gases and at least one second gas component formed from the at least one first gas component to the cryogenic air separation process in the gas provision section.

[0041] As to further details and advantages of an inventive fabrication plant, reference is made to the explanations above in connection with the method according to the present invention and its embodiments. Particularly, such a fabrication plant provided according to the present invention may comprise means adapted to perform a method according to any of the embodiments of the present invention.

[0042] The present invention is further described with reference to the appended drawings illustrating embodiments of the present invention.

Short description of the figures

[0043]

Figure 1 schematically illustrates a fabrication plant according to a preferred embodiment of the present invention.

Figure 2 schematically illustrates a fabrication plant according to a preferred embodiment of the present invention.

[0044] In the figures, elements of similar or identical construction and/or function are depicted with like reference numerals. A repeated explanation is omitted for reasons of conciseness. Explanations relating to process units or components likewise relate to corresponding method steps and vice versa.

[0045] As mentioned before, the present invention may be used to recover a number of noble gases, of which helium serves an example hereinbelow.

Embodiments of the invention

[0046] Figure 1 schematically illustrates a fabrication plant 1000 according to a preferred embodiment of the

present invention.

[0047] The fabrication plant 1000 comprises a fabrication section 200 which is e.g. adapted to perform one or more steps 210 of semiconductor manufacturing, and a gas provision section 100. The fabrication section 200 is adapted being adapted to be operated using nitrogen 20 provided by the gas provision section 100 and being compressed in a compressor 21. The fabrication section 200 is further adapted to be operated using helium from a helium provision system 190 which e.g. comprises pressure tanks.

[0048] The gas provision section is adapted to perform a cryogenic air separation process wherein atmospheric air 1 is passed through a filter 2, compressed using a main air compressor 3, prepurified in a prepurification unit 4, cooled in a main heat exchanger 5 and thereafter rectified in a rectification column system 10 comprising a double column with a pressure column 11 and a low pressure column 12 in a cold section 110. A helium-neon system 6 is also part of the cold section 110.

[0049] Air separation units of the type shown in a highly generalized manner are described elsewhere, for example in Haring (see above), in particular in section 2.2.5, "Cryogenic Rectification". For detailed explanations of the design and operation, reference is made to the relevant technical literature. An air separation unit for use in the present invention can be designed in a wide variety of ways.

[0050] A first gas mixture comprising nitrogen, helium and at least one first gas component is withdrawn from the fabrication section 200 as an offgas and, in the embodiment shown, treated in a offgas treatment sequence 250 comprising a combustion unit 251 and a scrubbing unit, forming a second gas mixture.

[0051] The second gas mixture is, as a gas stream 9, supplied to the gas provision section 100 and, in more detail, compressed together with feed air 1 supplied to the gas provision section. A loaded scrubbing liquid may be supplied to a further treatment unit 260 in a manner generally known per se. If the composition and cleanliness of the second gas mixture is such that it can be mixed with feed air 1 without undergoing combustion and/or scrubbing, gas stream 9 can be withdrawn directly from semiconductor manufacturing 210.

[0052] Crude helium-neon 121, optionally containing nitrogen and hydrogen, may optionally be purified on-site in a purification unit 122 and either compressed in a compression unit 123 or liquefied in a liquefaction section 124, particularly using liquid nitrogen withdrawn from the cold part 110.

[0053] Figure 2 schematically illustrates a fabrication plant 2000 according to a preferred embodiment of the present invention which additionally comprises an argon section 7, e.g. including a crude and a pure argon column, and a krypton-xenon section 8.

Claims

1. A method of operating a fabrication plant (1000, 2000), the fabrication plant (1000, 2000) comprising a fabrication section (200) and a gas provision section (100), the fabrication section (200) being adapted to be operated using nitrogen provided by the gas provision section (100) and using one or more noble gases, the gas provision section being adapted to perform a cryogenic air separation process, and a first gas mixture comprising nitrogen, helium, the one or more noble gases and at least one first gas component being withdrawn from the fabrication section (200) as an offgas, **characterized in that** at least a part of the first gas mixture and/or at least a part of a second gas mixture formed from the first gas mixture and comprising nitrogen, the one or more noble gases and at least one second gas component formed from the at least one first gas component is supplied to the cryogenic air separation process in the gas provision section (100). 5
2. The method according to claim 1, wherein the one or more noble gases is or are selected from the group of helium, neon, argon, krypton and xenon. 10
3. The method according to claim 1 or 2, wherein at least a part of the first gas mixture is treated in an offgas treatment unit (220) to form the second gas mixture. 15
4. The method according to claim 3, wherein the second gas mixture or its part which is supplied to the cryogenic air separation process is supplied to the cryogenic air separation process together with feed air upstream of a main air compressor. 20
5. The method according to claim 3 or 4, wherein the at least one second gas component includes at least one of carbon dioxide, carbon monoxide, hydrogen chloride, hydrogen fluoride and water. 25
6. The method according to any one of claims 3 to 5, wherein the at least one first gas component includes at least one of oxygen, fluorine, chlorine, ammonia, carbon monoxide, carbon dioxide, nitric oxide, nitrous oxide, silane, disilane trimethylsilane and halogenated compounds like boron trichloride, dichlorosilane, trichlorosilane, hexachlorodisilane, monomethylsilane, chlorine trifluoride, difluoromethane, hexafluoro-1,3-butadiene, hexafluoroethane, hydrogen bromide, tungsten hexafluoride, hydrogen chloride, hydrogen fluoride, methyl fluoride, fluoromethane, nitrogen trifluoride and octofluorocyclobutane. 30
7. The method according to any one of claims 3 to 6, wherein the offgas treatment unit (220) is adapted to perform at least an incineration step (221) and a scrubbing step (222), wherein the first gas mixture is at least in part supplied to the incineration step (221) and wherein at least a part of the second gas mixture is withdrawn from the scrubbing step (222). 35
8. The method according to any one of the preceding claims, wherein the gas provision unit (100) is adapted to provide a component mixture predominantly comprising helium and neon and comprising at least one of nitrogen and hydrogen. 40
9. The method according to claim 8, wherein the gas provision unit (100) is adapted to further process the component mixture predominantly comprising helium and neon and comprising at least one of nitrogen and hydrogen. 45
10. The method according to any one of the preceding claims, wherein the gas provision unit (100) is adapted to provide argon or a component mixture predominantly comprising argon and comprising nitrogen. 50
11. The method according to any one of the preceding claims, wherein the gas provision unit (100) is adapted to provide krypton and/or xenon or a component mixture comprising krypton and xenon in a concentration of at least 50 ppm. 55
12. The method according to any one of the preceding claims, wherein the gas provision unit (100) comprises a prepurification unit (2) adapted to remove the at least one first gas component at least in part from the first gas mixture or a part thereof supplied to the cryogenic air separation process and/or to remove the at least one second gas component at least in part from the second gas mixture or a part thereof supplied to the cryogenic air separation process.
13. A fabrication plant (1000, 2000) comprising a fabrication section (200) and a gas provision section (100), the fabrication section (200) being adapted to be operated using nitrogen provided by the gas provision section (100) and using one or more noble gases, the gas provision section being adapted to perform a cryogenic air separation process, wherein means are provided which are adapted to withdraw a first gas mixture comprising nitrogen, the one or more noble gases and at least one first gas component from the fabrication section (200) as an offgas, **characterized in that** means are provided which are adapted to supply at least a part of the first gas mixture and/or at least a part of a second gas mixture formed from the first gas mixture and comprising nitrogen, the one or more noble gases and at least one second gas component formed from the at least one first gas component to the cryogenic air separation process in the gas provision section (100) .

14. The fabrication plant (1000) according to claim 13, comprising means adapted to perform a method according to any one of claims 1 to 12.

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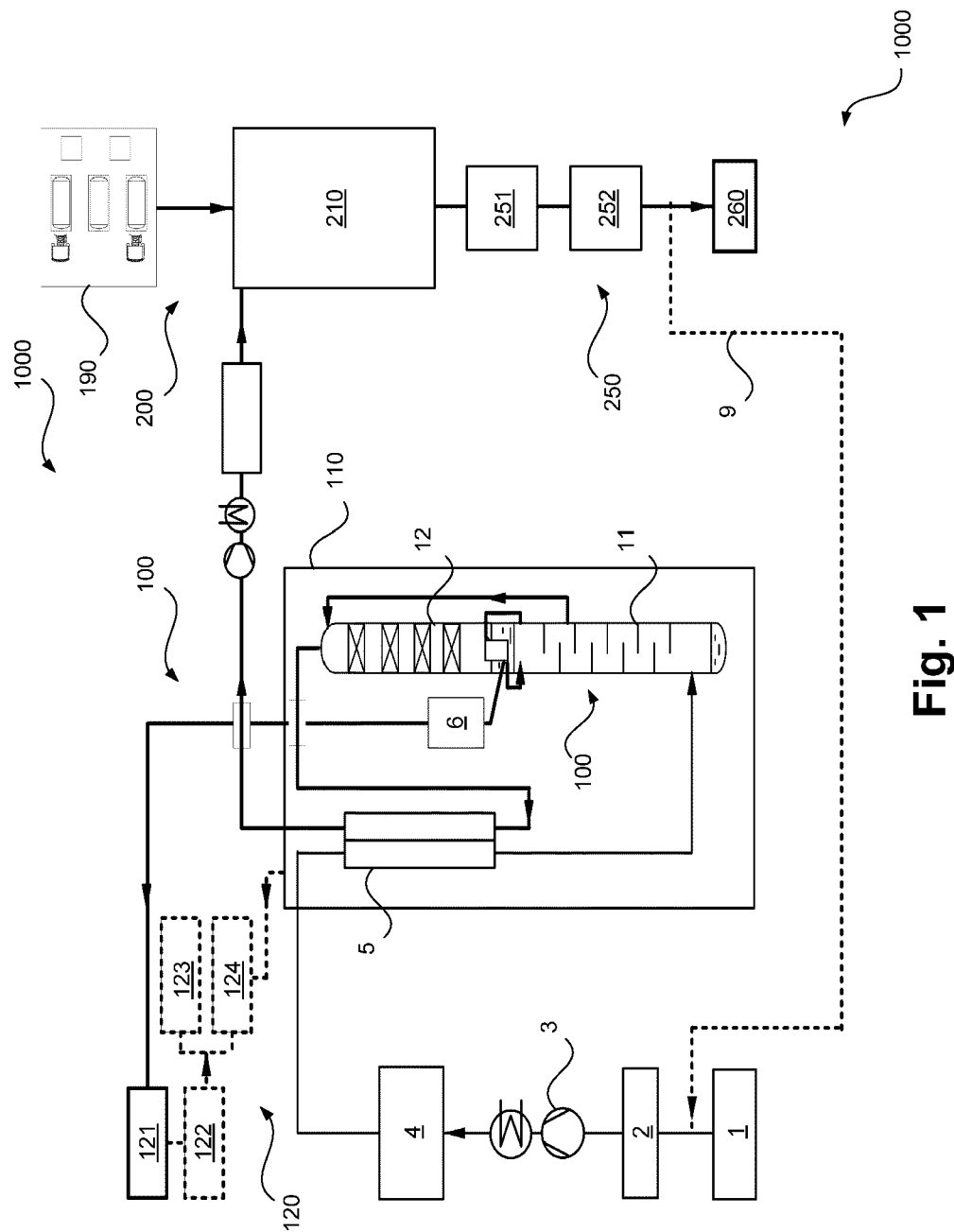


Fig. 1

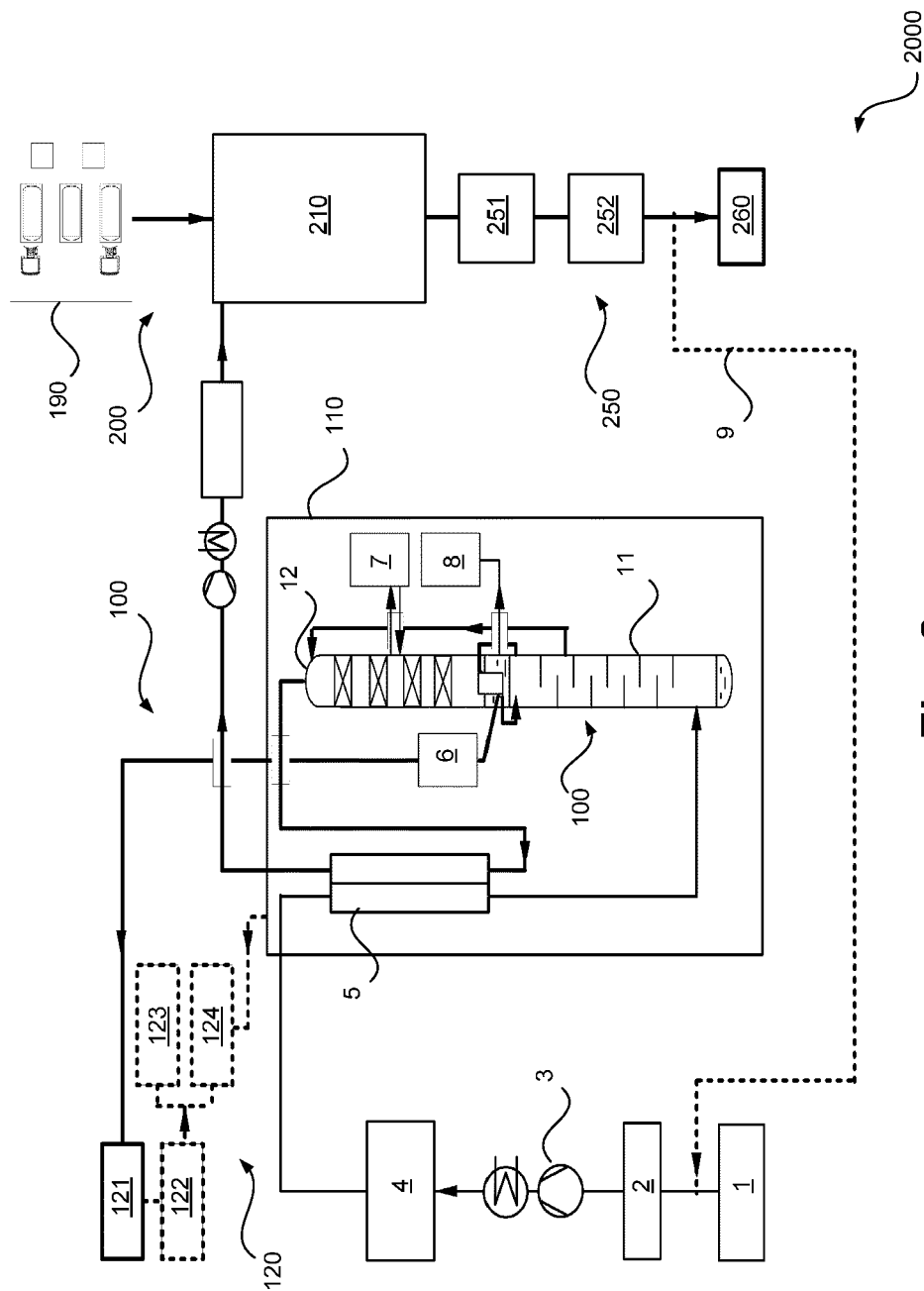


Fig. 2



EUROPEAN SEARCH REPORT

 Application Number
 EP 21 02 0223

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Place of search Munich		Date of completion of the search 5 October 2021	Examiner Göritz, Dirk
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document			

**ANNEX TO THE EUROPEAN SEARCH REPORT
ON EUROPEAN PATENT APPLICATION NO.**

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This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report.
The members are as contained in the European Patent Office EDP file on
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