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(54) **METHOD AND DEVICE FOR PURGING A PRODUCTION SPACE FOR METAL POWDER PRODUCTION**

(57) The present invention refers to a method and a device for purging a production space for metal powder production wherein a liquid inert cryogen is fed to the

production space in order to reduce oxygen and/or moisture levels in the production space.

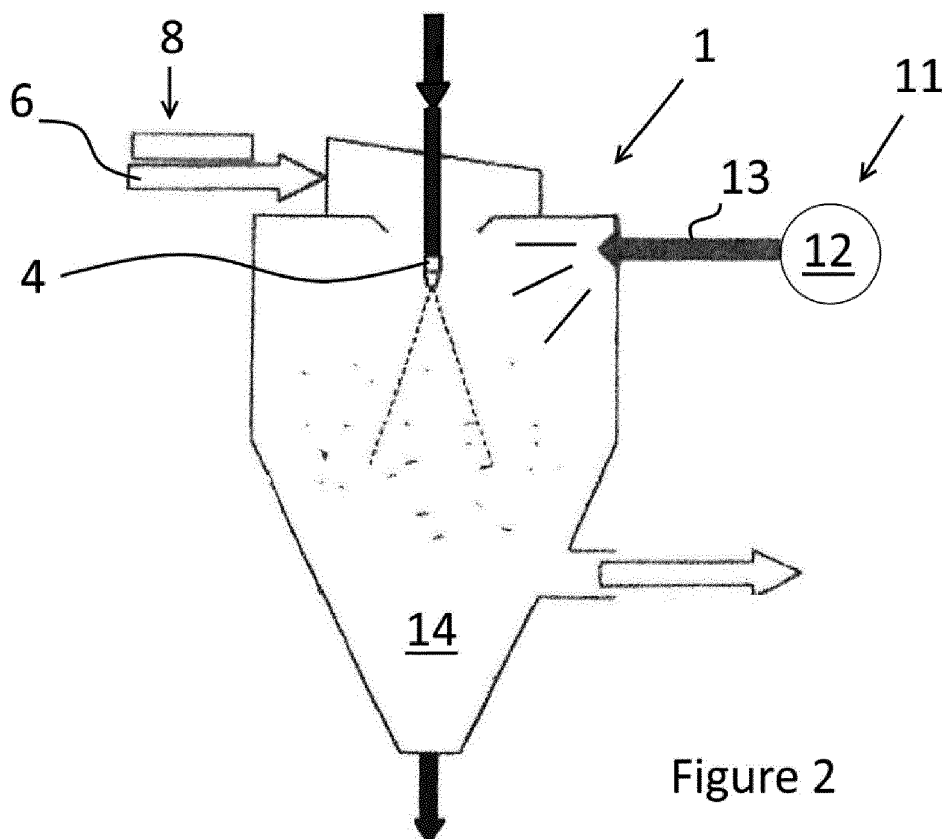


Figure 2

Description

[0001] The present invention relates to a method and to a device for purging a production space for metal powder production.

[0002] There are numerous processes for producing metal powder. These include the mechanical comminution of solid metal, the precipitation from salt solutions, the thermal decomposition of a chemical compound, the reduction of a chemical compound, usually the oxide in the solid phase, the electrolytic deposition and the atomization of liquid metal. The latter three methods are most commonly used in practice for the production of metal powder.

[0003] During atomization, molten metal is broken up into small droplets and rapidly solidified before the molten droplets come into contact with each other or with a solid surface. The principle of this process is based on the division of a thin liquid metal jet through a high velocity gas or liquid stream. Air, nitrogen and argon are the most commonly used gases, as a liquid, especially water is used.

[0004] Other methods of melt distribution are increasingly used, i.e. centrifugal atomization, in which molten droplets are spun off a rotating source.

[0005] While water atomization is used in particular for the production of powders from iron, steel, copper and copper alloys, the atomization of aluminum and zinc takes place predominantly, that of copper partially under air.

[0006] For the compressed gas atomization, first a melt of the metal to be atomized or the alloy to be atomized is built up and overheated accordingly. This superheated melt usually runs over a second smaller crucible or a pouring funnel and forms there a melt jet, which falls vertically through a nozzle construction. The melt jet is atomized by a gas (carrier gas) and the resulting droplets solidify in a spray chamber. In the atomization chamber and/or in downstream gas cleaning instructions (cyclones, filters), the metal powder is separated from the carrier gas.

[0007] In the industrial steel powder extraction by water atomization, preference is given to using low-carbon steel melt produced in the LD process. Another way to extract steel powder is to use sorted scrap and melt it in an electric arc furnace.

[0008] High-purity powders made of special steel, super alloys and other high-alloy or oxidation-sensitive materials can be advantageously produced by atomizing with inert gas. This process usually yields spherical powders which are more suitable for conventional mechanical molding of molded parts, for isostatic pressing and powder injection molding processing.

[0009] On an industrial scale, the ASEA-STORA process is frequently used for atomizing high-speed steel melts. By using purified inert gas, such as N₂ and Ar, and working in a closed facility, powders can be produced with approximately 100 ppm oxygen. To increase the

cooling rate of the metal droplets, the atomization chamber is cooled from the outside and a water-cooled bottom is used to collect the powders.

[0010] Another method involves atomization with gases in a NANOVAL Laval nozzle. For the production of pure spherical metal powders from reactive metals such as titanium or zirconium, methods are advantageous which do not allow contact of the molten metal with ceramic crucible material, since this could lead to oxidation of the melt and possibly destruction of the crucible. Therefore, the reactive metal is melted inductively or by means of plasma in a cooled copper crucible. Between the copper crucible and the melt, a thin solidified layer of the metal to be atomized forms, which effectively prevents a reaction of the melt with the crucible material.

[0011] Another possibility of ceramic-free metal atomization, which is particularly suitable for reactive materials and is used i.e. in the production of titanium powder, represents the EIGA process. In this method, the metal to be atomized or the alloy to be atomized is fed as an electrode in rod form perpendicular to an annular induction coil and melted superficially here. In order to ensure a uniform melting, the rod undergoes a rotary movement during the process. The melt thus produced finally drips in free fall through an annular nozzle, is atomized and solidified here. Subsequently, the powder is deposited in a atomization holder.

[0012] Also to make pure spherical titanium and titanium alloy powders plasma atomization is used. An approximately 3 mm diameter wire made from the alloy to be atomized is fed to an array of three plasma torches, where it is melted and atomized in one step. The purity of the starting material, the absence of any crucible material and the melting under inert atmosphere gives a final product of the highest purity.

[0013] A number of melts under vacuum, which must be assigned to atomization in principle, is possible with the help of noble gases or hydrogen. The gas-enriched melt under pressure is forced in a thin stream into an evacuated chamber. The expansion of the dissolved gas in the melt divides these into fine droplets.

[0014] The rapid reduction of residual oxygen from vessels that will be handling the production of metal powders either in the primary spray chamber of vessels up and down stream of that vessel. Residual oxygen often is held in parts of a vessel that has little flow and relies on the diffusion properties of the gases involved to reduce the oxygen concentration within the vessel.

[0015] In the process of fine metal power production, it is important to reduce both the oxygen and humidity that exists within the spray chamber before the start of the atomization process.

[0016] In a conventional system this is undertaken by the use of purge gas into the chamber. This will bring the oxygen level down and the humidity will be reduced but at a very slow rate. Even with such a purge system residual H₂O remains in the spray chamber.

[0017] It is an object of the present invention to provide

an improved method and an improved device for purging a production space for metal powder production.

[0018] It is a further object of the present invention to provide a method and a device for purging production space for metal powder production wherein oxygen and/or moisture levels in the production space can be reduced efficiently.

[0019] Furthermore it is an object of the present invention to provide a method and a device for purging a production space for metal powder production which reduces the purging time.

[0020] One or more of these problems are solved by a method according to independent claim 1 and by an apparatus according to independent claim 10. Advantageous embodiments are defined in the sub-claims.

[0021] According to the present invention a method is provided for purging a production space for metal powder production wherein a liquid inert cryogen is fed to the production space in order to reduce oxygen and/or moisture levels in the production space.

[0022] Within the scope of the present invention a production space is a space or a chamber in which metal powder is generated, for example a spray chamber of a device for producing metal powder.

[0023] The inventors of the present invention have recognized that the current purging methods for the production of metal powders are insufficient in that oxygen and moisture (H₂O) remain in the spray chamber.

[0024] A cryogenic fog can be produced when the liquid inert cryogen is entering the production space, in which the metal powder is atomized.

[0025] The cryogenic fog is preferably produced by a high-pressure liquid spray system directly into a spray chamber at the start of a purge cycle, wherein the high-pressure liquid spray system provides a pressure wave of inert gas through the chamber initially displacing the unwanted gases within the spray chamber into an exhaust system.

[0026] The use of the cryogenic fog produced by the high-pressure liquid spray system directly into the spray chamber at the start of the purge cycle provides the pressure wave of inert gas through the spray chamber. Thereby most of the unwanted gases within the spray chamber are displaced into an exhaust system of sufficient size and back pressure to prevent the over pressurization of the chamber. The exhaust system has a non return device, for example a non-return valve, to prevent the back flow of oxygen or H₂O into the chamber.

[0027] The cryogenic fog can rapidly condense any water vapor followed by droplets freezing within the chamber, wherein this increase in mass of the frozen droplet will cause frozen particles to be swept out on the purge cycle produced by the vaporizing cryogen.

[0028] The high-pressure liquid spray system can create a pulsating flow of cryogenic gas via an injector creating a series of pressure waves within the chamber.

[0029] The fog within the chamber can coalesce fine particles within the chamber, wherein these fine particles

are removed prior to the start of a production process.

[0030] The ability to remove moisture very quickly from a spray chamber will reduce cycle times for the operator and will improve product quality by reducing residual humidity and a reduced purge and/or cycle time.

[0031] The use of the fog technology within the chamber will act to coalesce any very fine particles within the chamber which have to be removed prior to the start of the process.

[0032] Normal practice of reducing the oxygen concentration within a chamber or vessel is to purge the vessel with an appropriate inert gas. The time taken for this purge to reduce the residual oxygen concentration depends on the flow rate of the inert gas, a vessel size and a vessel internal shape.

[0033] According to the present invention it is proposed to inject inert gas into the vessel as a cryogenic liquid. This is further improved by creating a pulsating flow of cryogenic gas via an injector creating a series of pressure wave within the vessel.

[0034] These pressure swings of inert gas will improve the rate of oxygen depletion.

[0035] Preferably an injection nozzle is provided that has a variable frequency of cryogenic inert gas injection. Initial injection frequency will be of a long duration to rapidly reduce the oxygen concentration by a sweep purge. As the oxygen concentration reduces the frequency of the cryogenic injection will be of a shorter duration producing the desired pressure wave to spread within the vessel. The pressure waves of inert gas will promote a pressure purge principle to promote the removal of oxygen within any "dead" volumes within the vessel.

[0036] The pressure swing of the pulse will promote the displacement of "bound" oxygen on the material surface within the purge space (production space; spray chamber).

[0037] The utilization of this method for varying the purging method and pulsation frequency reduces the cycle time of the operation.

[0038] The method of operation will speed up the removal of the residual oxygen (or other gases required to be removed). Gases resulting from the metal melting process may be present from the previous operation cycles such as NO, NO₂ and CO₂.

[0039] The liquid inert cryogen can be liquid Nitrogen, liquid Argon or liquid Helium or corresponding mixtures thereof.

[0040] The Method for producing metal powder comprises the further following steps: providing a melt, forming a melt jet or liquid sheet, atomizing the melt jet or liquid sheet by means of an atomizing fluid, forming metal powder particles from the melt jet.

[0041] An atomizing fluid can be an inert gas such as Argon, Helium, Neon, Krypton, Xenon or Radon or an active gas such as O₂, CO₂, H₂, and N₂, or mixtures thereof. Above all, water can be provided as the liquid atomizing fluid.

[0042] In this regard, reference is made to the method

of atomization with gas, water or centrifugal force mentioned in the introduction to the description.

[0043] Furthermore, a device for metal powder manufacturing according to the present invention is provided. This device comprises for producing a metal powder, comprising a device for providing a melt, a nozzle device for atomizing the melt by means of an atomizing fluid, a spray chamber for forming metal powder particles from the atomizing melt by means of an atomizing fluid, characterized in that the device for feeding liquid inert cryogen comprises storage container/vessel for a liquid inert cryogen and a feeding device for feeding the liquid inert cryogen to the spray chamber.

[0044] The same advantages mentioned in connection with the method for purging a production space according to the present invention apply mutatis mutandis to the device for producing metal powder according to the present invention.

[0045] The device for feeding liquid inert cryogen can comprise a high-pressure liquid spray system.

[0046] An oxygen sensor (electronic device) for measuring the proportion of oxygen (O₂) in the spray chamber can be provided in the production space, wherein the oxygen sensor is connected to a control unit and the control unit is connected to the device for feeding liquid and creating the pressure pulse frequencies of inert cryogen for controlling the device for feeding liquid inert cryogen according to the value measured by the oxygen sensor. The depletion of O₂ measured by the sensor can be used to change the frequency of the pulses of inert cryogen. A high concentration of O₂ corresponds to long pulse and a low concentration results in a fast pulse to promote pressure purging at low O₂ concentrations.

[0047] The invention is explained below with the aid of an embodiment shown in the drawings. The drawing show in:

Figure 1 a schematic, side-sectional view of a device according to the present invention for producing metal powder, and

Figure 2 another embodiment of a device according to the present invention for producing metal powder in a schematic, side-sectional view.

[0048] In the following, a device 1 according to the present invention for producing metal powder is described (Figures 1 and 2).

[0049] This device 1 comprises a melting crucible 2 for providing a molten metal.

[0050] Furthermore, the device 1 comprises a pouring funnel 3, which can be filled with melt by means of the melted crucible 2. The pouring funnel 3 is provided with a ceramic coating.

[0051] An outlet channel of the pouring funnel 3 opens into a nozzle device 4.

[0052] The nozzle device 4 comprises centrally a passage opening 5, through which a melt jet formed by the outlet channel of the pouring funnel 3 can pass.

[0053] The passage opening 5 is surrounded by an annular atomizing fluid chamber 6 for receiving and distributing an atomizing fluid.

[0054] The atomizing fluid chamber 6 opens into an annular gap 7 arranged concentrically with the passage opening 5. The annular gap 7 forms an atomizing nozzle for generating melt droplets from the melt jet.

[0055] In addition, a device for feeding atomizing fluid 8 is provided, by means of which the atomizing fluid can be provided to the atomizing fluid chamber 6.

[0056] The atomizing fluid supply device 8 has a storage tank 9 for the atomizing fluid, wherein the storage tank 9 is connected via a conduit 10 with the atomizing fluid chamber 6.

[0057] Furthermore, a device for feeding a liquid inert cryogen to a spray chamber 14 is provided. The device for feeding a liquid inert cryogen includes a storage container 12 for a liquid inert cryogen.

[0058] The storage container 12 is connected to the spray chamber 14 via a conduit 13.

[0059] In the storage container 12 liquid Argon or liquid Helium or liquid Nitrogen is stored.

[0060] An oxygen sensor (electronic device) for measuring the proportion of oxygen (O₂) in the spray chamber can be provided in the production space, wherein the oxygen sensor is connected to a control unit and the control unit is connected to the device for controlling the frequency of feed liquid inert cryogen for controlling the device for feeding liquid inert cryogen according to the value measured by the oxygen sensor. Preferably the oxygen sensor sets the frequency of the pulses of cryogen into the purge space. Shorter pulses are provided as the O₂ level lowers.

[0061] In the following, a method according to the present invention for purging a production chamber 14 will be described.

[0062] Unless otherwise stated, all technical features described in connection with the embodiments of the apparatus are applicable in connection with method steps for the method according to the present invention.

[0063] A purge cycle is started in the spray chamber by feeding a liquid cryogen to the spray chamber via a high-pressure liquid spray system. Thereby a cryogenic fog is produced.

[0064] The liquid inert cryogen can be liquid Nitrogen, liquid Argon or liquid Helium or other suitable liquid gas.

[0065] The high-pressure liquid spray system provides a pressure wave of inert gas through the chamber initially displacing the unwanted gases within the spray chamber into an exhaust system.

[0066] The cryogenic fog rapidly condenses any water vapor followed by the droplets freezing within the chamber. The increase in mass of the frozen droplet will cause frozen particles to be swept out on the purge cycle produced by the vaporizing cryogen.

[0067] The high-pressure liquid spray system preferably creates a pulsating flow of cryogenic gas via an injector creating a series of pressure waves within the chamber.

[0068] The fog within the chamber can coalesce fine particles within the chamber, wherein these fine particles are removed prior to the start of a production process.

[0069] The fog within the chamber will act to coalesce any very fine particles within the chamber which have to be removed prior to the start of the process.

[0070] The proportion of oxygen (O₂) in the spray chamber can preferably be measured via an oxygen sensor (for example: Linde ADDvance™ O₂ precision) which is disposed in the production space, wherein the oxygen sensor is connected to a control unit and the control unit is connected to the device for controlling the frequency of feed liquid inert cryogen for controlling the device for feeding liquid inert cryogen according to the value measured by the oxygen sensor. That means the oxygen sensor is connected to the control unit and the control unit is connected to the device for feeding liquid and creating the pressure pulse frequencies of inert cryogen for controlling the device for feeding liquid inert cryogen according to the value measured by the oxygen sensor.

[0071] The depletion of O₂ measured by the sensor can be used to change the frequency of the pulses of inert cryogen. A high concentration of O₂ corresponds to long pulse and a low concentration results in a fast pulse to promote pressure purging at low O₂ concentrations.

[0072] Therefore the oxygen sensor preferably sets the frequency of the pulses of cryogen into the purge space. Shorter pulses are provided as the O₂ level lowers. Longer pulses are provided as the O₂ level rises.

[0073] After this, a melt of a metal to be atomized or an alloy to be atomized is first built up and superheated in the melting crucible 2.

[0074] Subsequently, the superheated melt is introduced into the pouring funnel 3 and forms in its outlet channel a melt jet, which passes vertically through the through hole 5 of the nozzle device 4.

[0075] This melt jet is atomized via the atomizing nozzle 7 of the nozzle device 4 in the atomizing/spray chamber 14 by means of the atomizing fluid.

[0076] A atomizing fluid can be an inert gas such as Argon, Helium, Neon, Krypton, Xenon or Radon or an active gas such as O₂, CO₂, H₂, and N₂, or mixtures thereof. Above all, water can be provided as the liquid atomizing fluid.

[0077] The resulting droplets solidify in the atomization chamber 14 in motion or in the movement respectively.

[0078] Furthermore, it can be provided to separate the metal powder from the atomizing fluid either in the atomization chamber 14 and / or in downstream gas purification systems (cyclones, filters).

List of Reference Numbers

[0079]

- | | |
|----|---|
| 1 | device |
| 2 | melting crucible |
| 3 | pouring funnel |
| 4 | nozzle device |
| 5 | through hole |
| 6 | atomizing fluid chamber |
| 7 | atomizing fluid supply |
| 8 | powder applying device |
| 9 | atomizing fluid reservoir |
| 10 | conduit |
| 11 | device for feeding a liquid inert cryogen |
| 12 | storage container |
| 13 | conduit |
| 14 | spray/atomizing chamber |

Claims

1. Method for purging a production space for producing metal powders, **characterized in that**, that a liquid inert cryogen is fed to the production space in order to reduce oxygen and/or moisture levels in the production space.
2. Method according to claim 1, **characterized in that**, that a cryogenic fog is produced when entering the production space in which the metal powder is atomized, and wherein this cryogenic fog displaces most unwanted gases within the spray chamber.
3. Method according to claim 1 or 2, **characterized in that**, that the cryogenic fog is produced by a high-pressure liquid spray system directly into a spray chamber at the start of the purge cycle, wherein the high-pressure liquid spray system provides a pressure wave of inert gas through the chamber initially displacing the unwanted gases within the spray chamber into an exhaust system.
4. Method according to one of the claims 1 to 3, **characterized in that**, that the liquid inert cryogen is liquid Nitrogen, liquid Argon or liquid Helium or corresponding mixtures thereof.
5. Method according to one of the claims 1 to 4, **characterized in that**, that the cryogenic fog will rapidly condense any water vapor followed by droplets freezing within the chamber, wherein this increase in mass of the frozen droplet will cause frozen particles to be swept out on the purge cycle produced by the vaporizing cryogen.
6. Method according to claim 5, **characterized in that**, that the high-pressure liquid spray system creates a pulsating flow of cryogenic gas via an injector creating a series of pressure waves of varying frequency within the chamber.

7. Method according to claims 1 to 6, **characterized in that**, that the fog within the chamber coalesces fine particles within the chamber, wherein these fine particles are removed prior to the start of a production process. 5

8. Method according to claims 1 to 7, **characterized in that**, that a depletion of O₂ measured by an oxygen sensor is used to change the frequency of pulses of inert cryogen wherein a high concentration of O₂ results in long pulses and a low concentration results in a fast pulse to promote pressure purging at low O₂ concentrations. 10

9. Method for producing metal powder according to a method for purging a production space for producing metal powders according to claims 1 to 8, comprising the further following steps: 15
 providing a melt, forming a melt jet, atomizing the melt jet or liquid metal sheet by means of an atomizing fluid, forming metal powder particles from the melt jet or liquid metal sheet. 20

10. Device for producing a metal powder, comprising a device for providing a melt, 25
 a nozzle device for atomizing the melt by means of an atomizing fluid,
 a spray chamber for forming metal powder particles from the atomizing melt by means of an atomizing fluid, **characterized in that** the device for feeding liquid inert cryogen comprises storage container/vessel for a liquid inert cryogen and a feeding device for feeding the liquid inert cryogen to the spray chamber. 30
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11. Device according to claim 10, **characterized in that**, the device for feeding liquid inert cryogen comprises a high-pressure liquid spray system. 40

12. Device according to claims 10 or 11, **characterized in that**, that an oxygen sensor is provided a production space and is embodied to set the frequency of pulses of cryogen into the purge space. 45

13. Device according to claim 12, **characterized in that**, that the oxygen sensor (electronic device) for measuring the proportion of oxygen (O₂) in the spray chamber is connected to a control unit and the control unit is connected to the device for feeding liquid inert cryogen for controlling the device for feeding liquid inert cryogen according to the value measured by the oxygen sensor. 50
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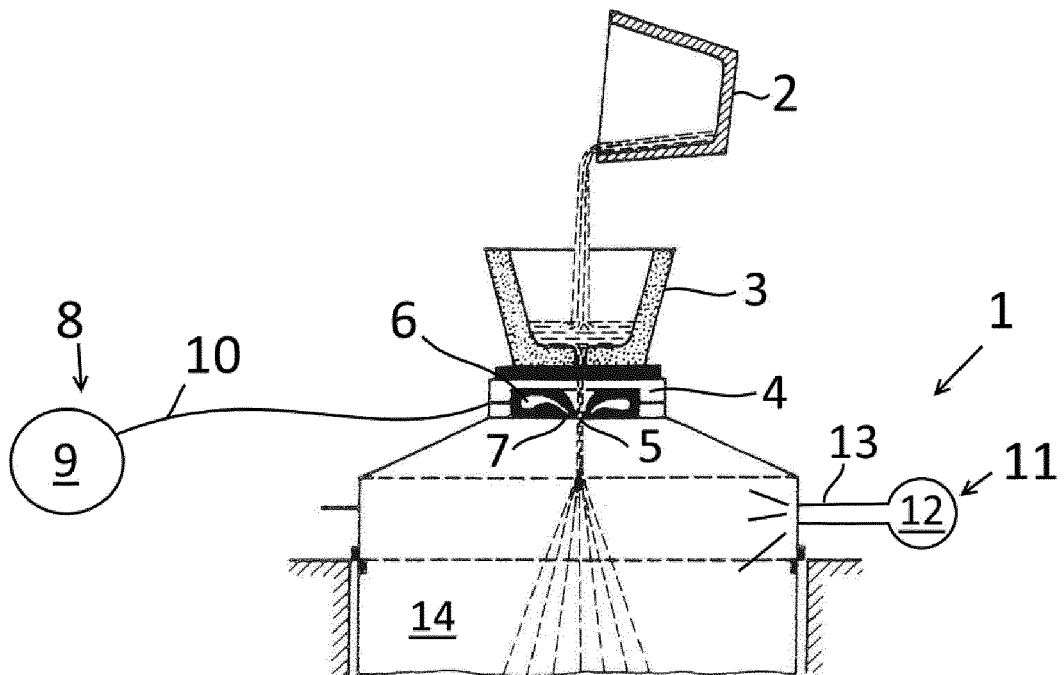


Figure 1

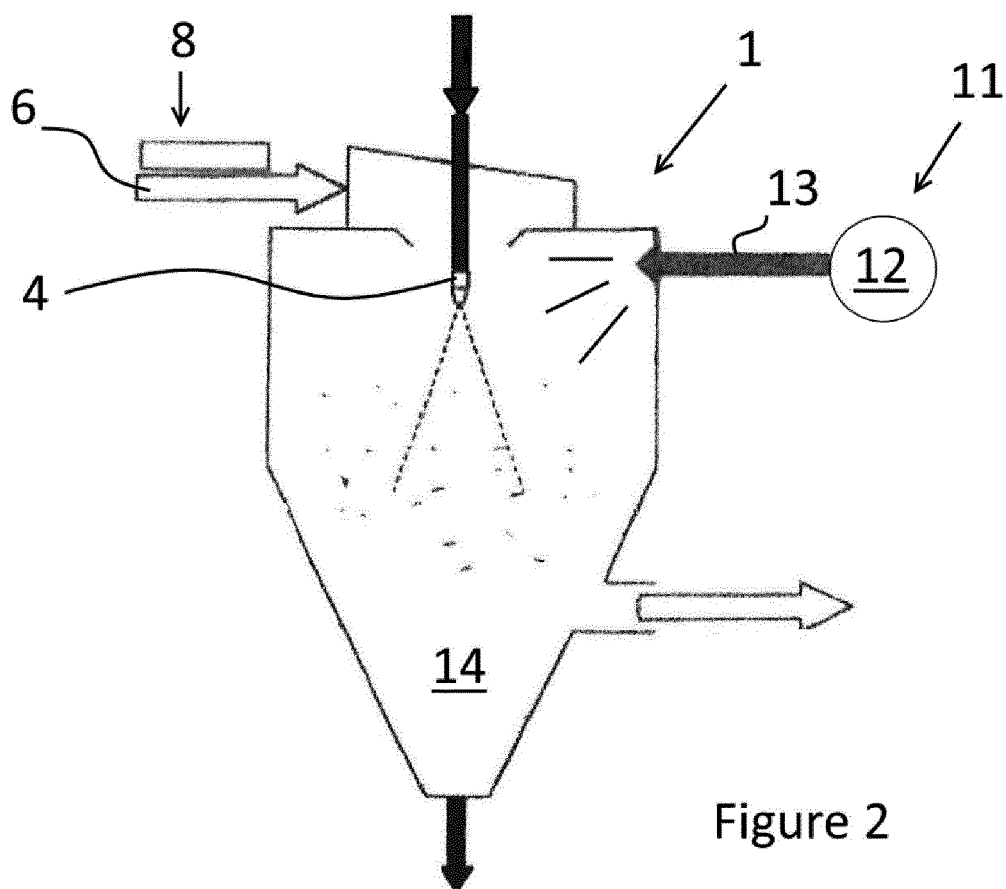


Figure 2



EUROPEAN SEARCH REPORT

Application Number
EP 19 02 0434

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A	* column 3, lines 16-25, 43-48 * * column 4, lines 13-18 * * figure 1 *	8,13	

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A	* figure 1 *	6,8,13	

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Y	* claims 1, 3, 5 *	12	
A	* column 1, lines 35-43 * * column 1, line 51 - column 2, line 2 * * column 2, lines 25-46 * * figure 1 *	6,8,13	

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	* claims 1, 2, 5, 9, 10 * * paragraph [0021] * * figure 1 *		

The present search report has been drawn up for all claims			
Place of search The Hague		Date of completion of the search 20 November 2019	Examiner Traon, Nicolas
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	

EPO FORM 1503 03.82 (P04C01)

**ANNEX TO THE EUROPEAN SEARCH REPORT
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5 This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report.
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