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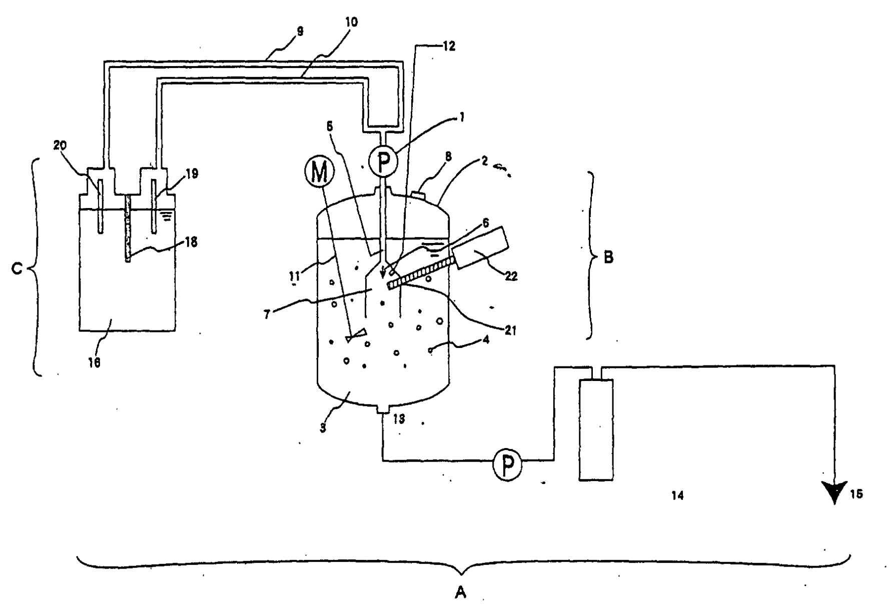
(54) METHOD AND APPARATUS FOR PRODUCING METAL POWDER

(57) Produce metal powder, especially titanium powder, offering high purity and uniform granular shape and size, in an economical manner using an apparatus that comprises a pressure-resistant container comprising a high-pressure water tank, an injector nozzle for mixture gas of oxygen and hydrogen, a material element-metal feeder part, an ignition plug and a combustion chamber.

Fig. 2



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Description

Field of the Invention

[0001] This invention relates to a method and apparatus for producing, in an economical manner, metal powder offering high purity and uniform granular shape and size.

[0002] The invention also relates to a production of titanium powder, among others, as the aforementioned metal powder.

Background of the Invention

[0003] Raw element metals are processed into various forms, such as molded shapes, sheet, bar, thin wire or foil, according to applications. In recent years the use of metal powder as molding material is drawing the attention in the fields of powder metallurgy, thermal spraying and other molding techniques. Particularly, powder metallurgy is regarded as an important technology offering wide applications, including production of metal parts, and therefore demand for powder metal—which is the base material for powder metallurgy—is also growing.

[0004] Production of metal powder traditionally used the classic method of mechanically and directly crushing metal granules into powder form or the method to blow molten metal with gas pressure to form powder. However, all these and other methods had difficulty achieving uniform granular shape and size, economy, and so on.

[0005] Electrolysis is one of relatively new methods for metal powder production. It has been reported that smooth, minute and uniform crystalline structures can be deposited under appropriate conditions, and that performing electrolysis outside the range of these conditions produces brittle metal of sponge or powder form.

[0006] Still, these newer production methods did not produce metal grains of satisfactory shape and size uniformity nor did they resolve other problems such as economy.

[0007] Among other metals, titanium is a relatively new metal compared with iron, copper and aluminum that have been in use since ancient times. Titanium is light and offers excellent strength at high temperature as well as corrosive resistance, and is therefore used widely in industrial applications.

[0008] The sample applications of titanium include jet engine material and structural member for aircraft/spaceship, material for heat-exchangers used in thermal and nuclear power generation, catalyst material used in polymeric chemical products, articles of daily use such as eyeglass frame and golf club head, and material for health equipment, medical equipment and medical/dental material. The applications of titanium are expected to grow further. Titanium, which is already competing with stainless steel, duralumin and other high-performance metals in terms of applications, is likely to surpass its rivals in the future.

[0009] Since titanium metal has poor processability and machinability, producing a mechanical part having complex shape from molten titanium will add to manufacturing man-hours and costs. It is because use of molten titanium as material will require cutting and other machining steps following the plastic working process such as hot forging and rolling.

[0010] Therefore, powder metallurgy is widely used in titanium metal processing, which is the reason for the growing demand for titanium powder, particularly one offering high purity and good uniformity of granular shape and size. Titanium powder produced by the conventional powder production methods designed for general metals is subject to the same problems with other metals; i.e., irregular granular shape and size, poor economy, and so on. As a result, development of a production method that can provide titanium powder offering high purity and uniform granular shape and size is eagerly awaited.

[0011] For example, the hydrogenative dewatering method and rotary electrode method are being put to practical use as improved production methods for titanium metal powder. The hydrogenative dewatering method uses sponge titanium, molten titanium or titanium chips generated from cutting/machining as material. The material titanium is heated in a hydrogen atmosphere to cause it to absorb the hydrogen gas and thus become brittle. This brittle titanium is then crushed and heated again in vacuum so that the hydrogen gas will be released and powder formed. In the rotary electrode method, molten titanium or titanium melted then forged, rolled or otherwise worked is formed into a round bar to be used as material. This material round bar is turned at high speed in an atmosphere of argon, helium or other inactive gas, while its tip is melted by a heat source such as an arc or plasma-arc torch. The drips of molten metal are then scattered via centrifugal force to produce spherical powder grains.

[0012] The grains of titanium powder obtained by the hydrogenative dewatering method have irregular sphericity. Although this powder can be used in die molding, the heating process must be repeated twice. A crushing process using a ball mill or other mechanical means may be incorporated, but oxygen contamination of titanium powder cannot be avoided. In the rotary electrode method, material titanium is melted in an inactive gas and made into powder form. Therefore, grains are spherical and offer good flowability. They are not subject to oxygen contamination, either. However, the solidification property when molded will be reduced. Both methods are a batch system, so the power production cost is high.

[0013] The atomization method was developed as a titanium powder production method addressing the aforementioned problems relating to quality and production cost. In the atomization method, material titanium is melted in a water-cooled copper crucible using a plasma-arc torch or other heat source, in order to cause molten titanium to drip continuously from one end of the crucible. Argon, helium or other inactive gas is then injected onto the molten titanium to atomize it and obtain powder. However, this method could not reduce the production cost significantly from the levels of the conventional methods, because molten titanium or melted and worked titanium had to be used as material.

[0014] In the meantime, a method for producing powder titanium offering improved sphericity and flowability for easier molding, in a manner requiring less cost and avoiding oxygen contamination, is disclosed in Japanese Patent Application Laid-open No. 5-93213. In this method, sponge titanium is isostatically pressed cold into a solid bar. This bar material is then melted in an inactive gas, after which argon, helium or other inactive gas is injected onto the dripping molten titanium to atomize it and obtain powder. However, this improved method did not offer good purity or uniformity of granular shape and size and the production cost was not at a satisfactory level, either.

Summary of the Invention

[0015] As described above, there is an increasing need and demand for metal powder, especially titanium metal powder, with the progress of powder metallurgy and other new molding methods. However, powder production methods that sufficiently answer such demand were not available and the existing methods had problems, particularly in regard to the purity of element metal, uniformity of granular sphericity and size of powder, and production cost

[0016] In view of the above, the purpose of the present invention is to provide element-metal powder material offering excellent uniformity of granular sphericity and consistency of granule size, for use in powder metallurgy and other types of molding.

[0017] The inventors conducted various studies to resolve the problems associated with the production of element metal powder such as titanium powder, including those pertaining to the purity of element metal, uniformity of granular sphericity, consistency of granule size and production cost.

[0018] As a result, the inventors resolved these problems by utilizing the technology relating to a use of titanium material for production of high-function water containing titanium, as specified in a patent application filed earlier by the inventors (Japanese Patent Application No. 2000-136932).

[0019] The aforementioned invention relating to a production of high-function water containing titanium (Japanese Patent Application No. 2000-136932), developed earlier by the inventors, provides a method for producing high-function water in which molten titanium is dissolved, wherein the method is characterized by the burning of a mixture gas of oxygen and hydrogen in high-pressure water and the melting of titanium metal using the combustion gas. By utilizing this technology, powder offering high purity and excellent uniformity of granular sphericity and size was obtained for use in the production of element metal powder, especially titanium metal powder, and the method also reduced the production cost of such powder significantly.

[0020] The present invention differs completely from the conventional production methods for metal powder such as titanium powder, in both concept and structure, in that a mixture gas of oxygen and hydrogen is burned in high-pressure water and the combustion gas is used to melt element titanium metal, thereby allowing metal powder to precipitate in water. It is an improved method for producing metal powder that adopts an approach completely different from those taken by the conventional methods.

[0021] Specifically, the present invention allows for a production of element metal powder in a very efficient manner without using thermal melting, arc discharge or laser irradiation to heat material metal and also without a need to let molten metal drip or inject gas onto it to atomize the metal and produce powder.

[0022] The method proposed by the present invention generates virtually no byproducts or impurities other than the target metal powder. Occurrence of metal oxidation due to heating of material metal is also very small, and since the obtained metal powder has excellent uniformity of granular sphericity and consistency of granule size, the production cost can be reduced significantly. The method also allows for continuous production in addition to batch production, which opens a door to mass-production of metal powder having uniform granule size.

[0023] In the above production process, a mixture gas of oxygen and hydrogen is burned in high-pressure water to achieve a high-temperature state, and this heat is used to melt material element metal, allowing it to instantly disperse in water and form fine grains. Unlike the preceding invention explained earlier (Japanese Patent Application No. 2000-136932), in this process the water pressure and degree of combustion of mixture gas can be adjusted to cause the obtained element metal powder to precipitate in a short period without floating, thereby yielding titanium powder aimed by the present invention.

[0024] To sum up, the basic structure of the present invention is to burn a mixture gas of oxygen and hydrogen in high-pressure water and use the combustion gas to heat material element metal and then convert it into powder form, thereby producing metal powder of uniform granule size. A schematic drawing of the production process is shown in the production flow chart given in Fig. 1.

[0025] The present invention consists of components (1) through (5) below, which basically serve to burn a mixture gas of oxygen and hydrogen in high-pressure water and use the combustion gas to melt material metal and then convert it into powder form:

(1) A method for producing metal powder by burning a mixture gas of oxygen and hydrogen in high-pressure water, using the combustion gas to heat material element metal, and converting it into powder form.

(2) A method for producing metal powder as described in (1) above, wherein the material element metal is titanium (Ti), zirconium (Zr), germanium (Ge), tin (Sn), gold (Au), platinum (Pt) or silver (Ag).

(3) An apparatus for producing metal powder, consisting of a pressure-resistant container that comprises a high-pressure water tank, an injector nozzle for mixture gas of oxygen and hydrogen, a material element-metal feeder part, an ignition plug and a combustion chamber.

(4) An apparatus for producing metal powder as described in (3) above, wherein the apparatus has as an adjunct a water electrolyzer for producing a mixture gas of oxygen and hydrogen.

(5) A method for producing metal powder as described in (3) or (4) above, wherein the material element metal takes a shape of bar, sheet, granule or foil.

Brief Description of the Drawings

[0026]

Fig. 1: Flow chart of metal powder production as proposed by the present invention

Fig. 2: An apparatus for producing metal powder as proposed by the present invention

Description of the Symbols

[0027]

A: Apparatus for producing metal powder
B: Pressure-resistant container for metal powder production
C: Electrolyzer
1: Mixture-gas injection pump
2: High-pressure water tank
3: Purified water
4: Fine metal grains
5: Mixture-gas injector nozzle
6: Mixture gas
7: Combustion chamber
8: Pressure control valve
9: Hydrogen-gas feed pipe
10: Oxygen-gas feed pipe
11: Mixing bar
12: Ignition plug
13: Metal powder outlet
14: Filter system
15: Product
16: Water
18: Partition
19: Electrode
20: Electrode
21: Material element metal
22: Metal feeder part

Best Mode for Carrying Out the Invention

[0028] The following explains the present invention by taking a production of titanium metal powder as an example. Note, however, that the invention is not limited to production of titanium powder.

[0029] According to the present invention, purified water such as distilled water is filled into the high-pressure water tank inside the pressure-resistant container for titanium-metal powder production, and material titanium metal such as a titanium metal bar is fed from the material element-metal feeder part and pressurized at high pressure. A mixture gas consisting of hydrogen and oxygen is injected from the nozzle, ignited and let completely bum in the combustion chamber. Upon achievement of a perfect combustion state leaving an ultrahigh-temperature steam gas, material titanium is instantly melted in the combustion gas and dispersed in water. At this time, very fine titanium grains of micron order are produced and dispersed in powder form. The produced fine titanium powder does not melt or float and precipitates in a short period.

[0030] The present invention can produce titanium powder of high purity at a very high efficiency. To achieve this, it is important to control the amounts of gases to be mixed and burned, reaction pressure and feed rate of material titanium metal.

[0031] With the production apparatus proposed by the present invention, an ideal injection amount of mixture gas is approx. 2 to 4 liters per second when the container can hold one ton of purified water. Applying too high a gas pressure may damage the apparatus structure, while a low pressure may cause the gas to flow upward from the nozzle, causing the heated, molten fine metal grains to be encapsulated in air bubbles and diffused from the water surface. This will reduce the generation efficiency of fine metal grains. The water pressure in the pressure tank should be 1.2 to 1.5 atmospheres.

[0032] The supplied material titanium metal should preferably have the highest possible purity, in order to prevent impurities from mixing into the produced titanium powder.

[0033] A mixture gas of hydrogen and oxygen provides the most efficient and stable means of burning titanium metal in water, where high pressure is required to ensure stable combustion. Physical or chemical explanations as to why element titanium metal melts instantly and becomes super-fine grains in a combustion gas in high-pressure water have not been found yet.

[0034] Material titanium metal may take a shape of bar, sheet, granule or foil, and it may be appropriate to feed titanium metal granules instead of bar if the capacity of the production container is much smaller than one ton.

[0035] In addition to titanium, the material element metals that can be used in the production of metal powder using the production apparatus proposed by the present invention include, but not limited to, zirconium, germanium, tin, gold, platinum and silver.

[0036] The high-pressure water tank used in the apparatus proposed by the present invention is a pressure-resistant tank made of metal, or preferably steel, and ideally other parts such as the combustion chamber should also be made of steel. The gas pump is installed to blow out a mixture gas at high pressure. Material element metal is fed continuously in accordance with the melt amount.

[0037] Material element metal must be fed into a position where the mixture gas bums completely and fully turns into a steam gas of ultrahigh temperature. The combustion chamber is installed to burn the mixture gas to achieve this purpose. This setup allows for production of pure metal powder free from impurities or byproducts. High pressure is also required to completely burn a pure mixture gas.

[0038] An actual embodiment of the present invention is explained according to the drawings. Note, however, that the invention is not limited to this example.

[0039] Fig. 1 shows a flow chart of metal powder production as proposed by the present invention, as described earlier. An apparatus for producing metal powder (A) shown in Fig. 2 consists of a pressure-resistant container (B) that comprises a high-pressure water tank (2), an injector nozzle for mixture gas of oxygen and hydrogen (5), a material element-metal feeder part (22), an ignition plug (12) and a combustion chamber (7).

[0040] The apparatus for producing metal powder (A) consists of a pressure-resistant container for metal powder production (B), and the pressure-resistant container for metal powder production comprises a gas injection pump (1), a high-pressure water tank (2), a combustion chamber (7), a pressure control valve (8), a metal powder outlet (13), purified water (3), material element metal for powder production (21), an ignition plug (12), a material element-metal feeder part (22) and a mixture-gas injector nozzle (5). (4) indicates produced metal powder.

[0041] Purified water (3) such as distilled water is filled into the high-pressure water tank (2) of the pressure-resistant container for metal powder production (B), and material titanium metal (21) such as a titanium metal bar is fed from the material element-metal feeder part (22), after which the container is pressurized at a high pressure. Hydrogen and oxygen are injected from the nozzle (5) as a mixture gas and the mixture gas is ignited by the ignition device (12). The mixture gas is completely burned in the combustion chamber (7) to obtain a perfect combustion state leaving an ultrahigh-temperature steam gas, and the material titanium melts instantly in this combustion gas and disperses in water.

[0042] At this time, very fine titanium grains of micron order (4) are produced and dispersed in powder form. The titanium metal powder does not melt or float and precipitates as powder in a short period. The separated powder is then released from the outlet for titanium metal powder (13) and becomes titanium powder.

[0043] The supply of mixture gas of hydrogen and oxygen must be precisely controlled to achieve a hydrogen-to-oxygen ratio of 2 to 1. While a mixture gas of hydrogen and oxygen is supplied from commercial gas cylinders, adding a water electrolyzer (C) as an adjunct to produce a mixture gas of hydrogen and oxygen via electrolysis of water will generate completely pure gases to facilitate an optimal, efficient supply of mixture gas.

[0044] In the present invention, adding a water electrolyzer (C) as an adjunct, instead of supplying a mixture gas of hydrogen and oxygen from commercial gas cylinders, will generate completely pure gases via electrolysis of water, thereby facilitating a supply of mixture gas in a simple and efficient manner. When adding a water electrolyzer for production of mixture gas of oxygen and hydrogen as an adjunct, the electrolyzer (C) is considered an optional adjunct unit to produce and supply a mixture gas of hydrogen and oxygen via electrolysis of water, which consists of feed pipes for hydrogen and oxygen gases (9, 10), electrodes (19, 20), a partition (18) and water (16). The electrolyzer causes electrolysis of acid or alkali raw water to generate oxygen gas at the anode and hydrogen gas at the cathode, and supplies them as a material mixture gas.

<u>Production Conditions and Results</u>

[0045] Pressurized water: 1 ton Pressure: 1.5 kg/m^2

Internal pressure of production tank: 1.5 atmospheres

Mixture gas: 5 L/sec (2 atmospheres)

Injection period: 1 hour

Feed rate of titanium metal: 25 kg

Production volume of titanium powder: Approx. 25 kg

<u>Evaluation of Produced Titanium Powder</u>

[0046] The element titanium powder contained no byproducts or impurities and exhibited excellent uniformity of granular sphericity and consistency of granule size. The production cost was reduced around a half compared with the conventional technologies.

<u>Industrial Field of Application</u>

[0047] The present invention allows for production of high-purity metal, especially titanium powder, in a very efficient manner. The production method proposed by the present invention achieves pure powder free from byproducts or impurities other than the elemental component, wherein the produced powder offers excellent uniformity of granular sphericity and size and can be produced at significantly less cost. Batch production, continuous production and mass production are also possible.

Claims

1. A method for producing metal powder, comprising burning a mixture gas of oxygen and hydrogen in high-pressure water, using the burning gas to heat material metal, and converting the material metal into powder form.

2. The method for producing metal powder as described in Claim 1, wherein said material metal is titanium, zirconium, germanium, tin, gold, platinum or silver.

3. An apparatus for producing metal powder, comprising a pressure-resistant container that comprises a high-pressure water tank, an injector nozzle for mixture gas of oxygen and hydrogen, a material element-metal feeder part, an ignition plug and a combustion chamber.

4. The apparatus for producing metal powder as described in Claim 3, comprising a water electrolyzer for producing a mixture gas of oxygen and hydrogen.

5. The method for producing metal powder as described in Claim 3 or 4, wherein said material element metal is a shape of bar, sheet, granule or foil.

Fig. 1

Production Flow Chart

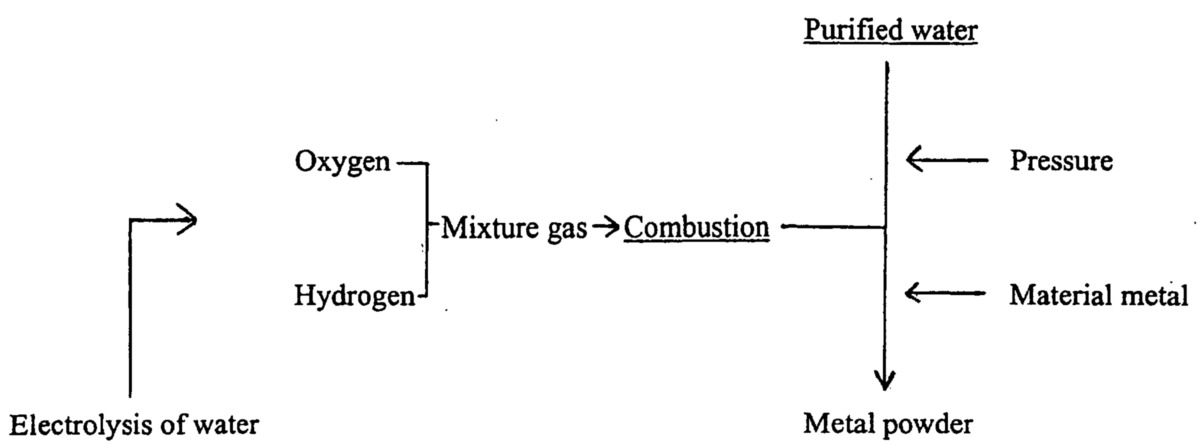
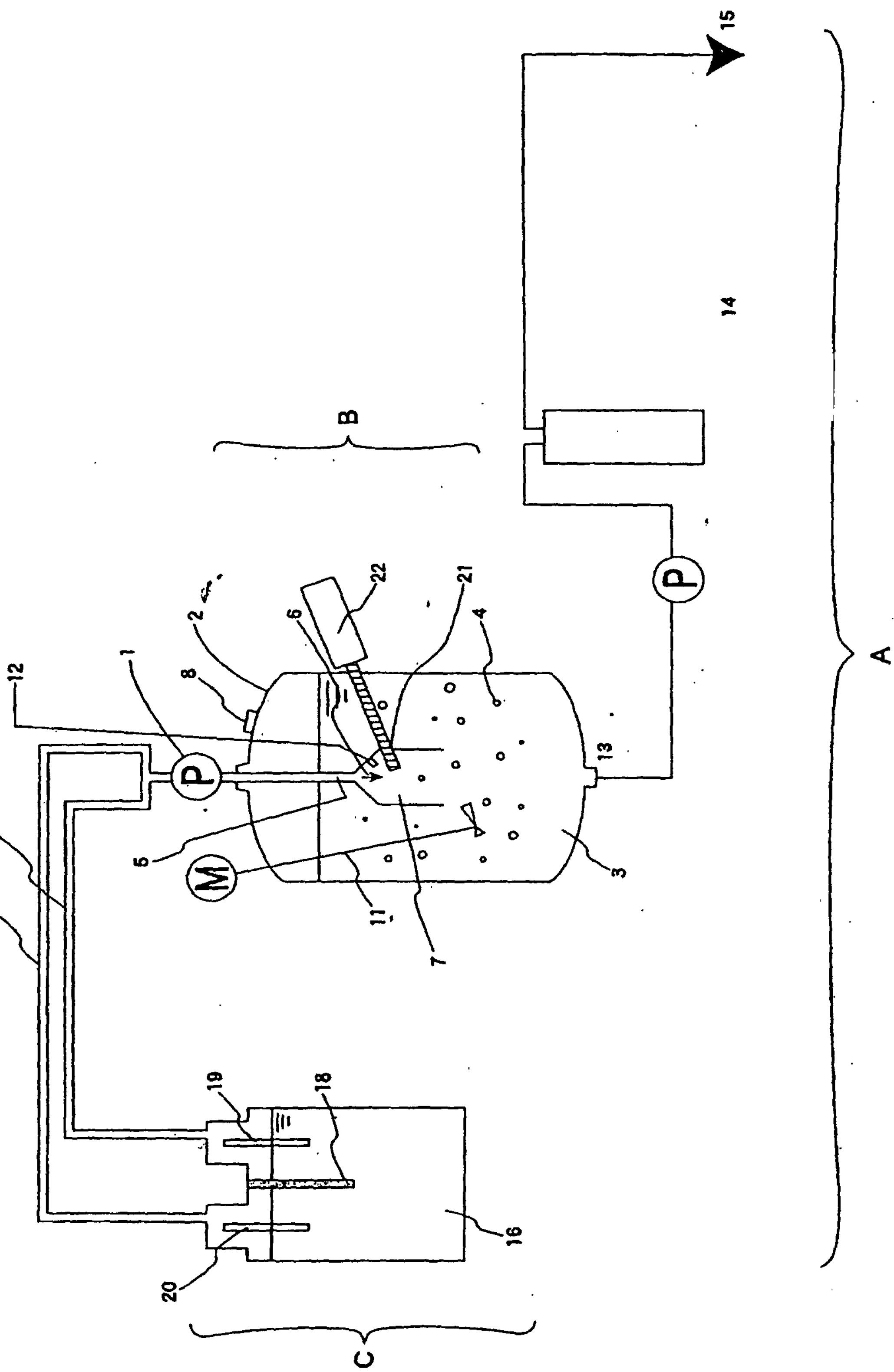


Fig. 2



INTERNATIONAL SEARCH REPORT

International application No.
PCT/JP02/02911

A. CLASSIFICATION OF SUBJECT MATTER
Int.Cl^7 B22F9/02

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)
Int.Cl^7 B22F9/02

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Jitsuyo Shinan Koho	1926-1996	Toroku Jitsuyo Shinan Koho	1994-2002
Kokai Jitsuyo Shinan Koho	1971-2002	Jitsuyo Shinan Toroku Koho	1996-2002

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.
P,X	JP 2001-137866 A (Fairudo Kabushiki Kaisha), 22 May, 2001 (22.05.01), Claims; Par. No. [0011]; Fig. 2 (Family: none)	1-5
X	JP 62-263903 A (Kyuzo KAMATA), 16 November, 1987 (16.11.87), Full text; drawings (Family: none)	1-5

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

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Date of the actual completion of the international search 07 June, 2002 (07.06.02)	Date of mailing of the international search report 18 June, 2002 (18.06.02)
Name and mailing address of the ISA/ Japanese Patent Office	Authorized officer
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