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(54) **NICKEL POWDER PRODUCTION METHOD**

(57) Provided is a production method for maintaining the quality while keeping a high operating rate of the reaction by continuously feeding a solution, seed crystals, and hydrogen gas into a reactor to produce nickel powder, and continuously discharging the resulting powder. The method for producing nickel powder comprises feeding a nickel ammine sulfate complex solution and seed crystals into a reactor, and feeding hydrogen gas into the reactor to subject a nickel complex ion in the nickel am-

mine sulfate complex solution to a reduction treatment and to thereby produce nickel powder, wherein, in the reduction treatment, while the nickel ammine sulfate complex solution is being continuously fed into the reactor, a temperature inside the reactor is controlled within the range of 150 to 185°C and the feed rate of hydrogen gas is controlled to maintain an inner pressure of the reactor in the range of 2.5 to 3.5 MPa. (149 words)

EP 3 424 627 A1

Description

Technical Field

[0001] The present invention relates to a method for obtaining nickel powder from a nickel ammine sulfate complex solution, and specifically relates to a method for continuously adding a solution and hydrogen gas etc., to a high pressure container, and continuously discharging and recovering nickel powder.

Background Art

[0002] As a method for industrially producing nickel powder using a hydrometallurgical process, a method for producing nickel powder disclosed in Patent Literature 1 is known, in which a raw material containing nickel is dissolved in a solution of sulfuric acid, followed by liquid-purification step of removing impurities contained in the dissolution, and thereafter ammonia is added to the resulting nickel sulfate solution to form a nickel ammine complex; and the nickel ammine sulfate complex solution is then placed into a container at high temperature and high pressure, and hydrogen gas is fed to reduce nickel in the nickel ammine sulfate complex solution.

[0003] Because the reaction is performed at high temperature and high pressure in such a production method as described above, batch methods for production are often used from the viewpoint of ease of handling and cost of the apparatus. However, in such batch methods for production, a series of operation to open the reactor, place the solution, tightly seal the reactor, heat the reactor, control the temperature and the pressure, blow hydrogen gas into the reactor to perform reduction, cool the reactor, and extract the reaction product should be performed at each stage. For this reason, the batch methods are not efficient because the methods require large amounts of labor and time, reducing the operating rate. Furthermore, influences of heating and/or cooling before and after the reaction cannot be neglected, causing uneven precipitates called scaling or a variation in particle size during the reaction in some cases. In particular, uneven nickel powder produced due to mixing of coarse nickel powder is more likely to cause wear or clog of the facility during handling, reducing the operating rate. The influences of uneven nickel powder as well as the labor to remove it result in difficulties in maintaining the operating rate of the reaction and the quality of products at constant levels.

[0004] Nickel powder obtained by the batch method has a problem about the quality of impurities compared to the electrolytic nickel in the form of a plate (sheet) obtained by standard electrometallurgy. Specifically, the sulfur grade should be 0.01% by weight or less to obtain the certification of high purity grade in an international nickel market London Metal Exchange (LME). The nickel powder obtained by the batch method may have higher sulfur grade than that in the high purity nickel of the LME

grade specified in the LME, and are difficult to use in applications where the electrolytic nickel is completely replaced.

5 Citation List

Patent Literature

[0005] Patent Literature 1: Japanese Patent Application Laid-Open No. 2015-140480

Summary of Invention

Technical Problem

[0006] The present invention provides a method for continuously feeding a solution, seed crystals, and hydrogen gas into a reactor kept at high temperature and high pressure to produce nickel powder, and continuously discharging and recovering the produced powder, whereby a fine nickel powder with high purity can be sufficiently grown, a variation in particle size can be reduced to maintain the quality of the nickel powder, and a high operating rate of the reaction can be maintained.

Solution to Problem

[0007] To solve the problems above, a first aspect of the present invention is a method of producing nickel powder, including feeding a nickel ammine sulfate complex solution and seed crystals into a reactor, and feeding hydrogen gas into the reactor to subject a nickel complex ion in the nickel ammine sulfate complex solution to a reduction treatment and to thereby produce nickel powder, wherein, in the reduction treatment, while the nickel ammine sulfate complex solution is being continuously fed into the reactor, a temperature inside the reactor is controlled within the range of 150°C or more and 185°C or less and the feed rate of hydrogen gas is controlled to maintain an inner pressure of the reactor in the range of 2.5 to 3.5 MPa.

[0008] A second aspect of the present invention is a method of producing nickel powder, including feeding hydrogen gas into a reactor, and feeding a nickel ammine sulfate complex solution and seed crystals into the reactor to subject a nickel complex ion in the nickel ammine sulfate complex solution to a reduction treatment and to thereby produce nickel powder, wherein, in the reduction treatment, the nickel complex ion in the nickel ammine sulfate complex solution is reduced in such a manner that a slurry containing ammonium sulfate and nickel powder are stored in the reactor to form a liquid phase portion and a gaseous phase portion in the reactor and an inner pressure of the gaseous phase portion is controlled through the feeding of the hydrogen gas into the reactor, a slurry containing seed crystals and the nickel ammine sulfate complex solution are continuously fed into the liquid phase portion, a temperature inside the

reactor is controlled in the range of 150°C or more and 185°C or less, and the feed rate of the hydrogen gas is controlled to maintain an inner pressure of the reactor in the range of 2.5 to 3.5 MPa.

[0009] A third aspect of the present invention is a method for producing nickel powder, wherein polyacrylic acid is added to the nickel ammine sulfate complex solution according to the first or second aspect such that a concentration of the polyacrylic acid is 0.5 to 1.0 g/liter.

[0010] A fourth aspect of the present invention is a method for producing nickel powder, wherein nickel powder having an average particle size in the range of 0.1 to 100 μm is used as the seed crystals according to the first and second aspects.

[0011] A fifth aspect of the present invention is a method for producing nickel powder, wherein nickel powder having an average particle size in the range of 0.1 to 10 μm is used as the seed crystals according to the first to third aspects.

[0012] A sixth aspect of the present invention is a method for producing nickel powder, wherein the amount of the seed crystals to be added according to the first to fifth aspects is in the range of 1 to 100% by weight based on the weight of nickel contained in the nickel ammine sulfate complex solution.

[0013] A seventh aspect of the present invention is a method for producing nickel powder, wherein the nickel ammine sulfate complex solution subjected to the reduction treatment according to the first to sixth aspects contains polyacrylic acid in an amount in the range of 0.5 to 5% by weight based on the weight of the seed crystals in the nickel ammine sulfate complex solution.

[0014] A eighth aspect of the present invention is a method for producing nickel powder, wherein in the reduction treatment according to the first to seventh aspects, the nickel ammine sulfate complex solution containing the seed crystals is continuously fed into the reactor such that the reaction time of the reduction treatment in the reactor takes 5 minutes or more and 120 minutes or less.

Advantageous Effects of Invention

[0015] According to the present invention, a nickel precipitate can be formed on seed crystals and a grown nickel powder can be formed thereon through the repeated reduction treatment with the precipitation of nickel. In addition, nickel powder having a little variation in size can be continuously obtained.

[0016] Also, because of the effect of the dispersant, the nickel powder having lower sulfur grade can be extracted and recovered from the solution in the form of fine powdery precipitate. Furthermore, a coarse nickel powder having a spherical shape and a smooth surface can also be obtained depending on the combination of the particle size of the nickel powder and the concentration of the dispersant.

[0017] The nickel powder produced in the present in-

vention can be used in applications of nickel pastes as an inner constitutional substance of stacked ceramic capacitors. This production method can grow particles through repetition of the reduction treatment with hydrogen to obtain a high purity nickel metal of high quality while maintaining a high operating rate of the reaction. This method attains an industrially remarkable effect.

Brief Description of Drawings

[0018]

[Figure 1] This illustrates an optical microscope photograph (×50) of the nickel powder according to Example 1 of the present invention.

[Figure 2] This illustrates an optical microscope photograph (×100) of the nickel powder according to Example 2 of the present invention.

[Figure 3] This illustrates an SEM photograph (×1000) of the nickel powder according to Example 3 of the present invention.

[Figure 4] This illustrates an SEM photograph (×500) of the nickel powder according to Example 4 of the present invention.

[Figure 5] This illustrates an optical microscope photograph 5A (×50) and 5B its enlarged photograph (×100) of the nickel powder according to Example 4 of the present invention.

Description of Embodiments

[0019] The present invention is a method for producing nickel powder including: producing nickel powder through a reduction treatment with hydrogen gas blown into a reactor as a pressurized container while adding seed crystals to a nickel ammine sulfate complex solution and continuously feeding the seed crystals; and continuously discharging the nickel powder from the pressurized container. Moreover, a high purity, uniform fine nickel powder having lower sulfur grade can be obtained by using a dispersant.

[0020] Hereinafter, the method for producing nickel powder according to the present invention will be described.

[0021] A nickel ammine sulfate complex solution that can be used in the present invention is not particularly limited, but it is suitable to use a nickel ammine sulfate complex solution obtained by dissolving a nickel-containing material, such as an industrial intermediate including one or a mixture of two or more selected from nickel and cobalt mixed sulfide, crude nickel sulfate, nickel oxide, nickel hydroxide, nickel carbonate, and nickel powder, with sulfuric acid or ammonia to prepare a nickel-containing leachate (solution containing nickel), subjecting the nickel-containing leachate to a liquid-purification step such as solvent extraction, ion exchange, or neutralization to remove impurity elements in the solution, and adding ammonia to the resulting solution.

[0022] In the present invention, seed crystals are added to the nickel ammine sulfate complex solution to form a slurry, which is subjected to the reduction treatment.

[0023] The seed crystals added here are powder having an average particle size of preferably 0.1 μm or more and 100 μm or less, more preferably 0.1 μm or more and 10 μm or less.

[0024] Nickel powder is suitably used as a substance which does not become impurities in the final nickel precipitate to contaminate the precipitate. The nickel powder used as the seed crystals can be prepared through addition of a reducing agent such as hydrazine to the nickel ammine sulfate complex solution, for example.

[0025] The weight of the seed crystals to be added is preferably 1% by weight or more and 100% by weight or less based on the weight of the nickel in the nickel ammine sulfate complex solution. A content of less than 1% by weight cannot sufficiently achieve the effect of reducing uneven precipitation. A content of more than 100% by weight has no influences over the effect; rather, it results in excess addition of the seed crystals.

[0026] A dispersant may also be then added to disperse the seed crystals in the slurry.

[0027] Any polyacrylate dispersant can be used without particular limitation. Suitable is sodium polyacrylate because it is industrially available at low cost.

[0028] If the dispersant is added, the amount thereof to be added is suitably in the range of 0.5 to 5% by weight based on the weight of the seed crystals. A content of less than 0.5% does not achieve any dispersing effect. A content of more than 5% has no influences over the dispersing effect; rather, such an addition is excess addition of the dispersant.

[0029] Alternatively, the polyacrylic acid may be added such that the concentration thereof is 0.5 to 1.0 g/liter based on the amount of the nickel ammine sulfate complex solution. The seed crystals added at this time are preferably seed crystals having an average particle size of 0.1 μm or more and 10 μm or less.

[0030] In the present invention, for example, "to" in the description of 0.5 to 5% by weight indicates 0.5% by weight or more and 5% by weight or less.

[0031] In the next step, the slurry prepared by adding the seed crystals or the seed crystals and the dispersant in the nickel ammine sulfate complex solution is continuously placed into a reaction vessel of a container resistant to high pressure and high temperature where a slurry containing ammonium sulfate and nickel powder is stored and the inner pressure is controlled with hydrogen gas. Thereby, a liquid phase portion occupied by the slurry and a gaseous phase portion are formed within the reaction vessel. Alternatively, the slurry containing the seed crystals or the slurry containing the seed crystals and the dispersant, and the nickel ammine sulfate complex solution are continuously charged into a reaction vessel of a container resistant to high pressure and high temperature where a slurry containing ammonium sulfate and nickel powder is stored and the inner pressure is controlled

led with hydrogen gas. Thereby, a slurry is formed, and a liquid phase portion occupied by the slurry and a gaseous phase portion having an inner pressure controlled with hydrogen gas is formed within the reaction vessel.

[0032] Subsequently, in the slurry continuously charged into the reaction vessel, the nickel complex ion contained in the nickel ammine sulfate complex solution is reduced with hydrogen gas to precipitate nickel on the seed crystals added and grow the nickel precipitate into nickel powder. The nickel powder slurry, i.e., the slurry containing the grown nickel powder is simultaneously formed, and is continuously discharged.

[0033] The reaction temperature at this time is preferably in the range of 150°C or more and 185°C or less. A reaction temperature of less than 150°C reduces the reduction efficiency. A reaction temperature of more than 185°C has no influences over the reaction; rather, it is not suitable because it increases loss of thermal energy.

[0034] Furthermore, the gaseous phase portion of the reaction vessel preferably is under a pressure maintained in the range of 2.5 to 3.5 MPa during the reaction. A pressure of less than 2.5 MPa reduces the reaction efficiency. A pressure of more than 3.5 MPa has no influences over the reaction; rather, it increases loss of hydrogen gas.

[0035] A reduction treatment accompanied by the precipitation of nickel under such conditions can form a nickel precipitate on seed crystals and thus a grown nickel powder, continuously yielding nickel powder having a little variation in size.

[0036] Moreover, because of the effect of the dispersant, nickel having lower sulfur grade can be extracted and recovered from the solution in the form of a fine powdery precipitate. In addition, a coarse nickel powder having a spherical shape and a smooth surface can also be yielded depending on the combination of the particle size of the nickel powder and the concentration of the dispersant.

[0037] The nickel powder produced as described above can be used in applications of nickel pastes as an inner constitutional substance of stacked ceramic capacitors. Besides, particles can be grown through repetition of the reduction with hydrogen to produce fine nickel metal with high purity and uniformity which has a particle size of 20 μm or less and is suitable for handling.

Examples

[0038] Hereinafter, the present invention will be described by way of Examples.

Example 1

[0039] A pressurized container (autoclave) having an inner volume of 190 liter was used as a reaction vessel. A solution slurry (90 liter) containing ammonium sulfate (269 g/L) and nickel powder (100 g/L) was placed into the reaction vessel. The reaction vessel was covered with

a lid to maintain the temperature at 185°C. Hydrogen gas was then blown into the reaction vessel to control the pressure to 3.5 MPa.

[0040] In the next step, the starting solution containing 150 g/liter of ammonium sulfate and a nickel ammine sulfate complex solution (concentration of nickel: 110 g/L) was added to the pressurized container at a flow rate of 1 liter per minute, and further a nickel seed crystal slurry (concentration of slurry: 300 g/L) was added at a flow rate of 0.25 liter per minute to advance a reduction treatment.

[0041] The nickel powder used here as the seed crystals forming the nickel seed crystal slurry had an average particle size of 1 μm . Hydrogen gas was blown into the reaction vessel such that the inner pressure of the pressurized container was maintained at 3.5 MPa.

[0042] The following operation was continued for four hours: while the amount of the solution stored in the pressurized container was being controlled in the range of 90 liter $\pm$ 5 liter, the nickel powder slurry containing the nickel powder produced in the reduction treatment was continuously extracted from the pressurized container. The reaction time in the reduction treatment in the reactor was 75 minutes from the charge of the starting solution and the seed crystal slurry to the extraction of the nickel powder slurry.

[0043] As shown in Table 1-1, the extracted nickel powder slurry contained 0.28 g/L of nickel, and the reduction rate (reaction rate), namely, the proportion of hydrogen gas used in the precipitation reaction of the nickel powder was 99.6%.

[0044] As shown in Table 1-2, particles having a particle size of 100 μm to 300 μm were 99% or more of the particle diameter distribution, indicating that a sufficiently grown nickel powder was obtained.

[0045] In the entire particle diameter distribution, the proportion of particles having a particle size of more than 300 μm was less than 0.1%, the proportion of particles having a particle size of more than 150 μm and 300 μm or less was 91%, the proportion of particles having a particle diameter of more than 100 μm and 150 μm or less was 8.3%, the proportion of particles having a particle diameter of more than 75 μm and 100 μm or less and the proportion of particles having a particle diameter of more than 45 μm and 75 μm or less both were less than 0.1%, and the proportion of particles having a particle diameter of 45 μm or less was 0.7%.

[0046] As shown in Figure 1, although the particles having uneven shapes and aggregation are observed, it was confirmed that nickel powder having a little variation in particle size distribution can be continuously produced. The sulfur grade was 0.062%.

[Table 1-1]

Reaction time	4 [Hours]
Concentration of Ni in Ni powder slurry	0.28 [g/L]
Reduction rate	99.6 [%]

(continued)

S grade	0.062 [%]
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[Table 1-2]

Reaction time	4 [Hours]
Particle size [μm]	Particle size distribution [%]
300	<0.1
300~+150	91
150~+100	8.3
100~+75	<0.1
75~+45	<0.1
~45	0.7

Example 2

[0047] The same reactor as in Example 1 was used. A solution slurry (90 liter) containing ammonium sulfate (205 g/L), polyacrylic acid (concentration: 1 g/L), and nickel powder (concentration: 105 g/L) was placed into the reactor. The reaction vessel was covered with a lid to maintain the inner temperature at 185°C.

[0048] Hydrogen gas was then blown into the gaseous phase portion in the reactor to control the inner pressure of the container to 3.5 MPa.

[0049] In the next step, a starting solution containing a nickel ammine sulfate complex solution (concentration of nickel: 83 g/L) and ammonium sulfate at a concentration of 120 g/L was fed into the reactor at a flow rate of 1 liter per minute, and simultaneously the nickel seed crystal slurry (concentration of slurry: 150 g/L) was continuously fed into the reactor at a flow rate of 0.5 liter per minute to advance the reduction treatment.

[0050] Nickel powder having an average particle size of 1 μm was used as the nickel powder forming the nickel seed crystal slurry. Hydrogen gas was blown such that the inner pressure of the reactor was maintained at 3.5 MPa.

[0051] While controlling the amount of solution stored in the reactor to be in the range of 90 liter $\pm$ 5 liter, the slurry subjected to the reduction treatment was continuously extracted. This operation was continued for 16 hours. The extracted slurry subjected to the reduction treatment was subjected to solid liquid separation using a Nutsche funnel into nickel powder and filtrate. The resulting nickel powder was washed, and was vacuum dried. The reaction time in the reduction treatment in the reactor was 60 minutes from the charge of the starting solution and the seed crystal slurry to the extraction of the nickel powder slurry.

[0052] The reduction rate (reaction rate), namely, the proportion of hydrogen gas used in the precipitation reaction of the nickel powder was 98.9%.

[0053] The resulting nickel powder had a finer average

particle size D50 of 5.2 μm but had a less variation in size than those of Example 1 (see Figure 2). Furthermore, the sulfur grade was 0.003%, which indicates that a high purity nickel powder having a low sulfur grade lower than the sulfur quality (0.01%) specified as the LME grade was obtained.

[Table 2]

Reaction time	16 [Hours]
Reduction rate	98.9 [%]
Particle size (D50)	5.2 [μm]
S grade	0.003 [%]

Example 3

[0054] A solution (90 liter) containing ammonium sulfate (205 g/L), nickel powder (105 g/L), and polyacrylic acid (1 g/L) was placed into a reactor having the same structure as in Example 1 and having a volume of 90 liter to maintain the temperature at 185°C. Hydrogen gas was blown into the reaction vessel to control the pressure at 3.5 MPa.

[0055] In the next step, a starting solution containing a nickel ammine sulfate complex solution (concentration of nickel: 83 g/L) and ammonium sulfate at a concentration of 120 g/L was added to this pressurized container at a rate of 1 liter/min, and simultaneously a nickel seed crystal slurry (slurry content: 150 g/L) was added at a rate of 0.5 liter/min. Moreover, polyacrylic acid at a concentration of 1 g/L was added to the nickel ammine sulfate complex solution in the starting solution, which was fed to the reactor. Hydrogen gas was blown into the pressurized container such that its pressure became 3.5 MPa. The extracted nickel powder forming the nickel powder slurry had an average particle size of 5.9 μm .

[0056] While the amount of the solution in the pressurized container was being managed in the range of 90 liter $\pm$ 5 liter, the nickel powder slurry was continuously extracted. This operation was continued for 12 hours. The reaction time in the reduction treatment in the reactor was 60 minutes from the charge of the starting solution and the seed crystal slurry to the extraction of the nickel powder slurry.

[0057] At this time, the reduction rate or the reaction rate was 96.8%.

[0058] The sulfur grade was 0.003%, which was lower than the sulfur grade (0.01%) specified as the LME grade.

[0059] The nickel powder had a particle size D50 of 6.4 μm , which indicates that a very fine powder could be stably obtained as shown in Figure 3.

[Table 3]

Reaction time	12 [Hours]
Reduction rate	96.8 [%]
Particle size (D50)	6.4 [μm]

(continued)

S grade	0.003 [%]
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Example 4

[0060] A starting solution (90 liter) containing ammonium sulfate (200 g/L), nickel powder (11 g/L), and polyacrylic acid (0.1 g/L) was placed into the 90 liter reactor the same as that in Example 1 to maintain the temperature at 185°C. Hydrogen gas was blown thereinto to control the pressure at 3.5 MPa.

[0061] A starting solution having a composition containing a nickel ammine sulfate complex solution (concentration of nickel: 83 g/L) and 360 g/L of ammonium sulfate was added to the reactor at a flow rate of 1 liter/min, and a nickel seed crystal slurry (concentration: 33 g/L) was added at a rate of 0.5 liter/min. Hydrogen gas was blown into the pressurized container such that its pressure was maintained at 3.5 MPa, to advance the reduction treatment.

[0062] While the amount of the solution stored in the reactor was being managed in the range of 90 liter $\pm$ 5 liter, the nickel powder slurry subjected to the reduction treatment was continuously extracted from the reactor. This operation was continued for 6 hours. The nickel powder forming the 33 g/L nickel seed crystal slurry had an average particle size of 53 μm . The reaction time in the reduction treatment in the reactor was 60 minutes from the charge of the starting solution and the seed crystal slurry to the extraction of the nickel powder slurry.

[0063] The reduction rate or the reaction rate was 89.0%.

[0064] The recovered nickel powder has a sulfur grade of 0.01%, which satisfied the sulfur grade (0.01%) specified as the LME grade.

[0065] The nickel powder had a particle size D50 of 78.0 μm , which indicates that a sufficiently grown nickel powder was obtained. As shown in Figures 4 and 5, the nickel powder was obtained in the form of particles having very smooth surfaces and having a true spherical shape.

[Table 4]

Reaction time	6 [Hours]
Reduction rate	89.0 [%]
Particle size (D50)	78.0 [μm]
S grade	0.01 [%]

Example 5

[0066] A pressurized container (autoclave) having an inner volume of 190 liter and having inner walls lined with titanium was used as a reactor (reaction vessel). A solution slurry (90 liter) containing 205 g/liter of ammonium sulfate, 1 g/liter of polyacrylic acid, and 105 g/liter of nickel powder was placed into this reactor. The reactor was

covered with a lid to maintain the temperature at 185°C.

[0067] Hydrogen gas was then blown into the gaseous phase portion of the reactor to control the inner pressure of the container to 3.5 MPa. In the next step, a nickel ammine sulfate complex solution (concentration of nickel: 83 g/liter) and a solution containing 120 g/liter of ammonium sulfate were fed into this reactor at a flow rate of 1 liter per minute, and simultaneously 150 g/liter of nickel powder slurry was continuously fed into the reactor at a flow rate of 0.5 liter per minute.

[0068] Nickel powder having an average particle size of 1 μm was used for forming the nickel powder slurry. Hydrogen gas was blown into the reactor such that the inner pressure was maintained at 3.5 MPa.

[0069] In the next step, while the amount of the solution in the reactor was being controlled in the range of 90 liter $\pm$ 5 liter, the nickel powder slurry was continuously extracted. This operation was continued for 16 hours. The extracted nickel powder slurry was subjected to solid liquid separation using a Nutsche funnel into nickel powder and a filtrate. The resulting nickel powder was washed, and was vacuum dried.

[0070] The reduction rate (reaction rate), namely, the proportion of hydrogen gas used in the precipitation reaction of the nickel powder was 98.9%.

[0071] The resulting nickel powder had an average particle size D50 of 5.2 μm . A fine nickel powder could be stably obtained.

(Comparative Example 1)

[0072] A solution having the same composition as in Example 1 was continuously fed, at the same flow rate, into the same reactor as in Example 1 without containing polyacrylic acid, and was reduced with hydrogen gas under the same condition as that in Example 1 to obtain a nickel powder slurry. The nickel powder slurry was subjected to solid liquid separation to obtain nickel powder. The reduction rate or the reaction rate was 99.6%.

[0073] In the particle size distribution of the resulting nickel powder, the proportion of particles having a particle size of 100 μm to 300 μm was 99% or more. In the entire particle size distribution, the proportion of particles having a particle size of more than 300 μm was less than 0.1%, the proportion of particles having a particle size of more than 150 μm and 300 μm or less was 91%, the proportion of particles having a particle size of more than 100 μm and 150 μm or less was 8.3%, the proportion of particles having a particle size of more than 75 μm and 100 μm or less and the proportion of particles having a particle size of more than 45 μm and 75 μm or less both were less than 0.1%, and the proportion of particles having a particle size of 45 μm or less was 0.7%. The resulting nickel powder was not fine as the nickel powder according to the present invention.

[0074] As described above, it was confirmed that a fine nickel powder can be continuously and efficiently obtained by use of the method according to the present

invention.

Claims

1. A method of producing nickel powder, comprising feeding a nickel ammine sulfate complex solution and seed crystals into a reactor, and feeding hydrogen gas into the reactor to subject a nickel complex ion in the nickel ammine sulfate complex solution to a reduction treatment and to thereby produce nickel powder, wherein in the reduction treatment, while the nickel ammine sulfate complex solution is being continuously fed into the reactor, a temperature inside the reactor is controlled within a range of 150 to 185°C and a feed rate of hydrogen gas is controlled to maintain an inner pressure of the reactor in a range of 2.5 to 3.5 MPa.
2. A method of producing nickel powder, comprising feeding hydrogen gas into a reactor, and feeding a nickel ammine sulfate complex solution and seed crystals into the reactor to subject a nickel complex ion in the nickel ammine sulfate complex solution to a reduction treatment and to thereby produce nickel powder, wherein in the reduction treatment, the nickel complex ion in the nickel ammine sulfate complex solution is reduced in such a manner that a slurry containing ammonium sulfate and nickel powder are stored in the reactor to form a liquid phase portion and a gaseous phase portion in the reactor and an inner pressure of the gaseous phase portion is controlled through the feeding of the hydrogen gas into the reactor, a slurry containing seed crystals and the nickel ammine sulfate complex solution are continuously fed into the liquid phase portion, a temperature inside the reactor is controlled in a range of 150 to 185°C, and a feed rate of the hydrogen gas is controlled to maintain an inner pressure of the reactor in a range of 2.5 to 3.5 MPa.
3. The method of producing nickel powder according to claim 1 or 2, wherein polyacrylic acid is added to the nickel ammine sulfate complex solution such that a concentration of the polyacrylic acid is 0.5 to 1.0 g/liter.
4. The method of producing nickel powder according to claim 1 or 2, wherein nickel powder having an average particle size in a range of 0.1 to 100 μm is used as the seed crystals.
5. The method of producing nickel powder according to any one of claims 1 to 3, wherein nickel powder

having an average particle size in a range of 0.1 to 10 μm is used as the seed crystals.

6. The method of producing nickel powder according to any one of claims 1 to 5, wherein an amount of the seed crystals to be added is in a range of 1 to 100% by weight based on a weight of nickel contained in the nickel ammine sulfate complex solution. 5
7. The method of producing nickel powder according to any one of claims 1 to 6, wherein the nickel ammine sulfate complex solution subjected to the reduction treatment contains polyacrylic acid in an amount in a range of 0.5 to 5% by weight based on a weight of the seed crystals in the nickel ammine sulfate complex solution. 10 15
8. The method of producing nickel powder according to any one of claims 1 to 7, wherein in the reduction treatment, the nickel ammine sulfate complex solution containing the seed crystals is continuously fed into the reactor such that a reaction time of the reduction treatment in the reactor takes 5 to 120 minutes. 20

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Fig.1

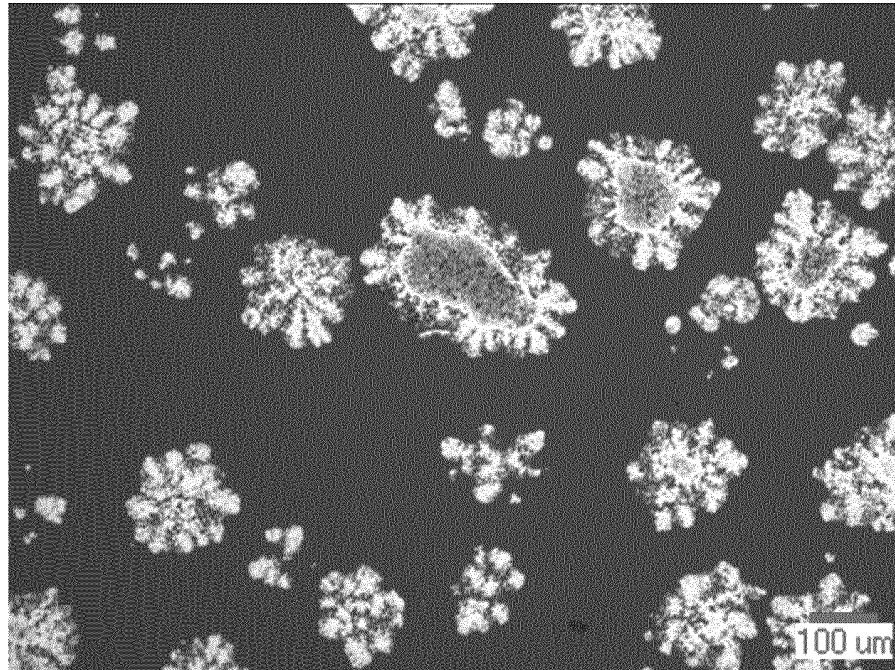


Fig.2

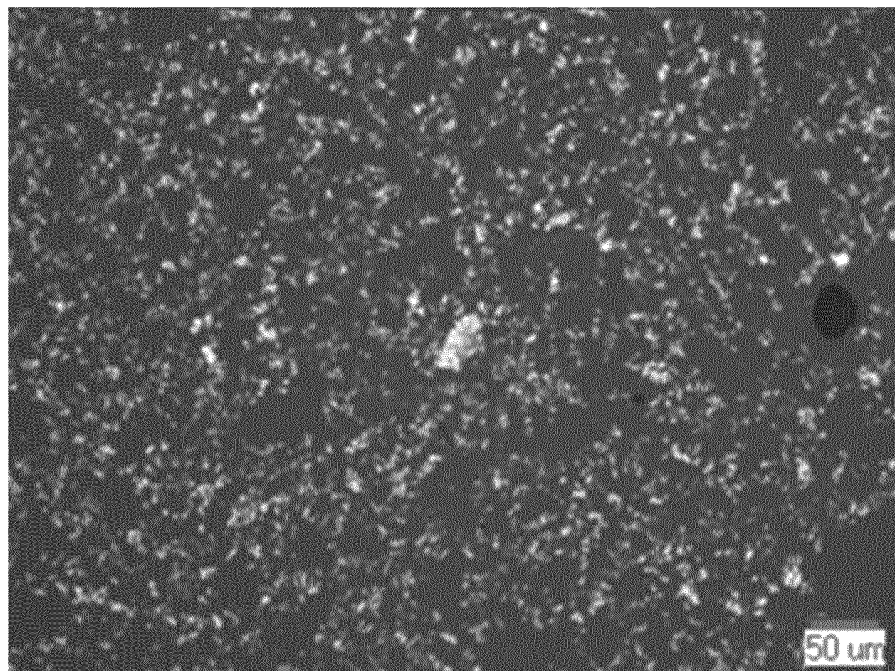


Fig.3

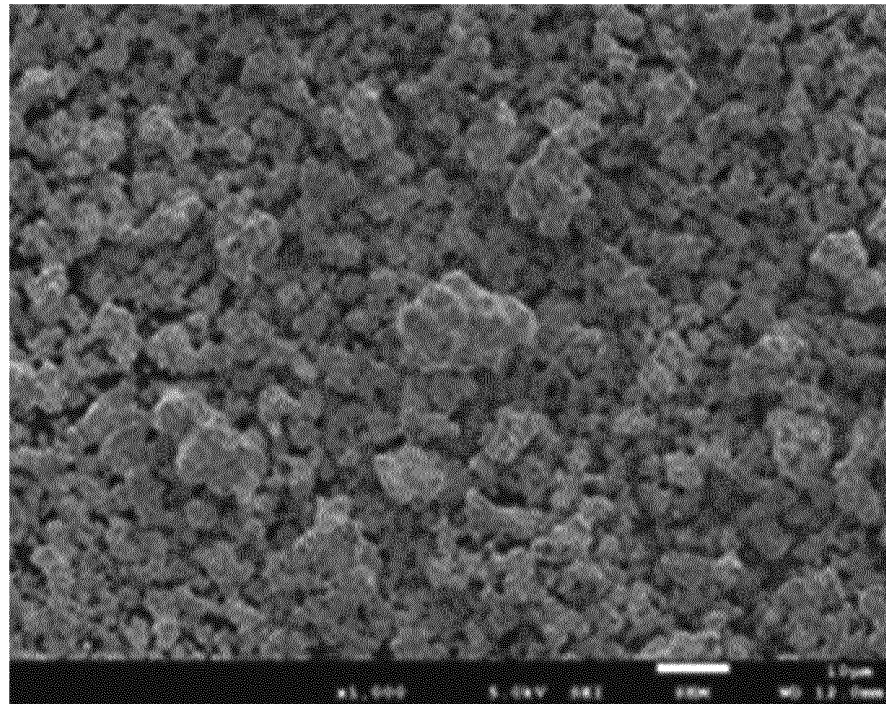


Fig.4

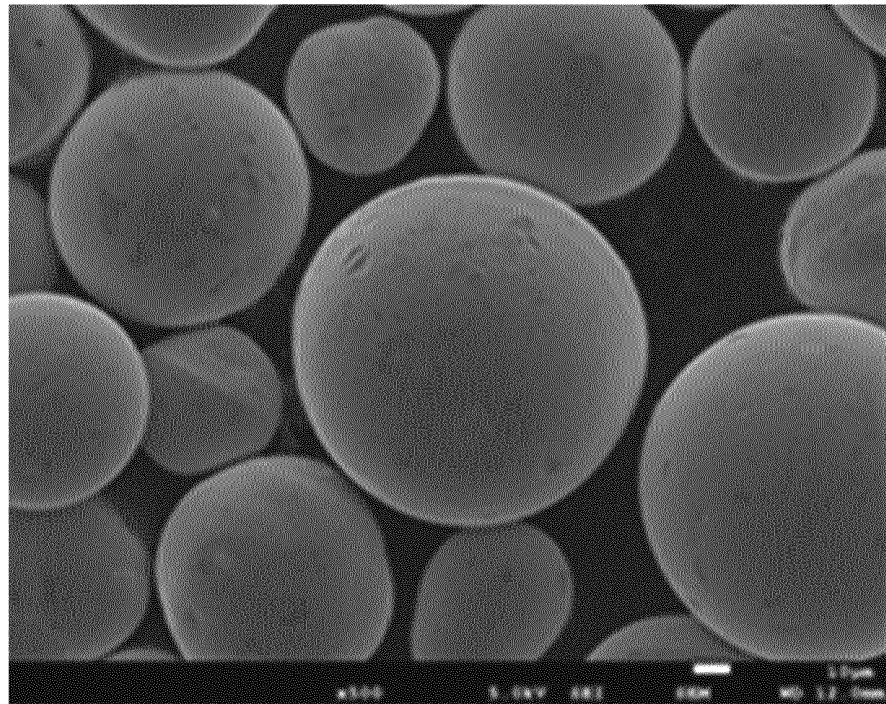


Fig.5A

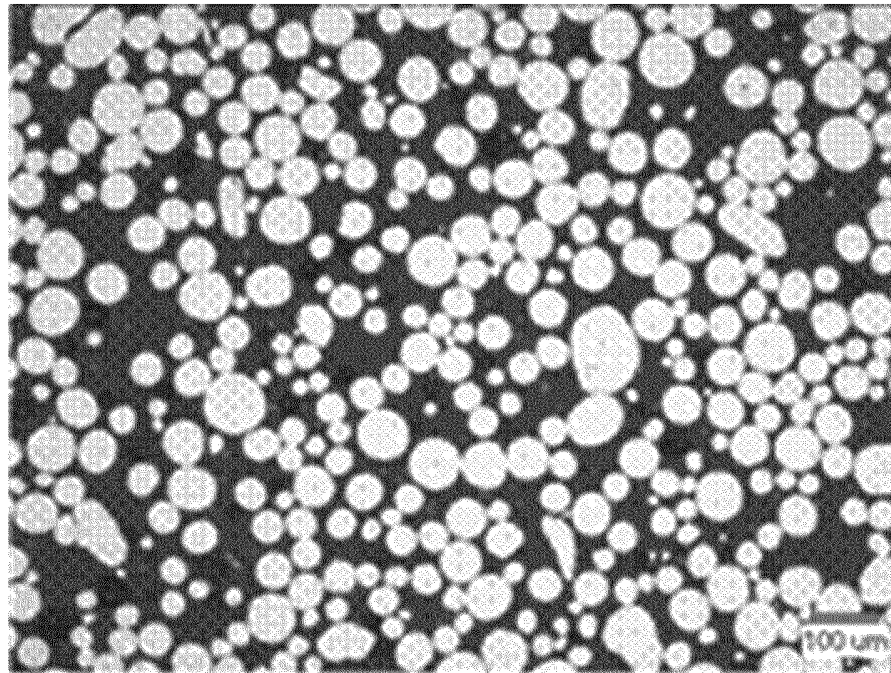
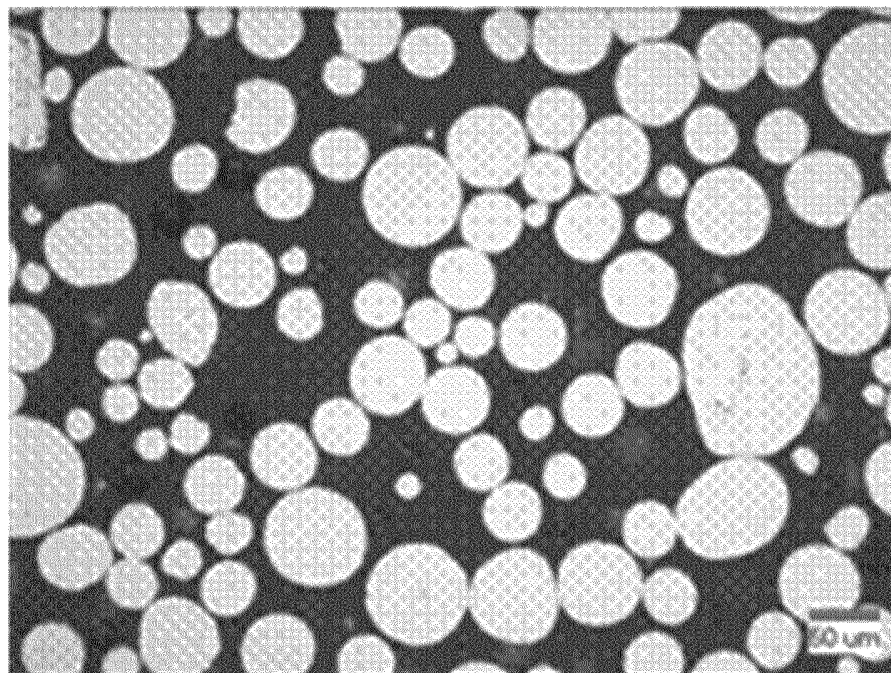


Fig.5B



INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2017/008562

A. CLASSIFICATION OF SUBJECT MATTER

B22F9/26(2006.01)i, B22F1/00(2006.01)i

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)

B22F9/26, B22F1/00

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

Jitsuyo Shinan Koho 1922-1996 Jitsuyo Shinan Toroku Koho 1996-2017

Kokai Jitsuyo Shinan Koho 1971-2017 Toroku Jitsuyo Shinan Koho 1994-2017

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.
Y	WO 2015/146989 A1 (National University Corporation Kochi University), 01 October 2015 (01.10.2015), claims; paragraphs [0018], [0019], [0021], [0027] & JP 2016-033255 A claims; paragraphs [0018], [0019], [0021], [0027] & EP 3124142 A1 claims; paragraphs [0021], [0022], [0024] to [0026], [0032] to [0033] & US 2017/0095862 A1 claims; paragraphs [0028], [0029], [0031] to [0033], [0039] to [0040] & CA 2943649 A1 claims; paragraphs [0018], [0019], [0021], [0027]	1-8

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

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"&" document member of the same patent family

Date of the actual completion of the international search
10 May 2017 (10.05.17)Date of mailing of the international search report
23 May 2017 (23.05.17)Name and mailing address of the ISA/
Japan Patent Office
3-4-3, Kasumigaseki, Chiyoda-ku,
Tokyo 100-8915, Japan

Authorized officer

Telephone No.

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INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2017/008562

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	& AU 2015234992 A1 & CN 106457405 A JP 01-130724 A (Kobe Steel, Ltd.), 23 May 1989 (23.05.1989), claims; page 5, upper right column, lines 15 to 19 (Family: none)	1-8

Form PCT/ISA/210 (continuation of second sheet) (January 2015)

REFERENCES CITED IN THE DESCRIPTION

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Patent documents cited in the description

- JP 2015140480 A [0005]