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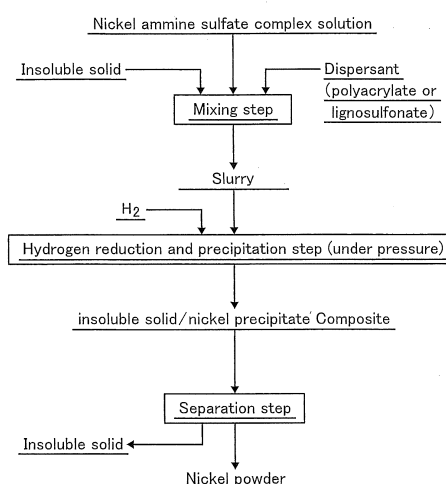
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(54) **NICKEL POWDER PRODUCTION METHOD**

(57) Provided is a method for producing nickel powder, wherein fine nickel powder serving as seed crystals needed for production of nickel powder is produced from a solution containing a nickel ammine sulfate complex according to the amount needed for the production of the nickel powder. The method for producing nickel powder is characterized in that: the solution containing the nickel ammine sulfate complex, an insoluble solid, and a dispersant are continuously fed into a reaction vessel, followed by stirring to prepare a solution containing a nickel complex ion; hydrogen gas is blown into the prepared solution to reduce the nickel complex ion in the solution containing the nickel complex ion, thereby forming a precipitate of nickel particles on the surface of the insoluble solid; and thereafter the post-reduction solution is extracted from the reaction vessel.

(135 words)

Fig.1



Description

Technical Field

5 **[0001]** The present invention relates to a method for producing fine nickel powder available as seed crystals from a solution containing a nickel ammine sulfate complex, and the invention can be applied, in particular, in a treatment for controlling the number of nickel powder generated to requirement.

Background Art

10 **[0002]** Methods for producing fine nickel powder have been known including atomization methods of dispersing molten nickel in gas or water to obtain fine powder, and dry processes, such as CVD processes, of volatilizing nickel, and reducing the nickel in a gaseous phase to obtain nickel powder as described in Patent Literature 1.

15 **[0003]** Methods for producing nickel powder by a wet process have been known including a method of producing nickel powder using a reducing agent as described in Patent Literature 2, and an atomization thermal decomposition method of atomizing a nickel solution in a reduction atmosphere at a high temperature to obtain nickel powder through a thermal decomposition reaction as described in Patent Literature 3. However, these methods are not economical because these methods require expensive reagents and a large amount of energy.

20 **[0004]** In contrast, a method as described in Non Patent Literature 1 is industrially inexpensive and useful, in which hydrogen gas is fed to a nickel ammine sulfate complex solution to reduce nickel ions in the complex solution, obtaining nickel powder. However, this method tends to produce coarse particles of the nickel powder, and has difficulties in producing a fine powder which can be used as seed crystals.

25 **[0005]** When particles are produced from an aqueous solution and are grown, a small amount of fine crystals called seed crystals is added, and a reducing agent is fed thereto to grow the seed crystals into a powder having a predetermined particle size.

[0006] The seed crystals used in this method are often obtained through pulverization of a product, which requires labor, reduces the yield, and thus increases cost. In addition, the pulverization does not necessarily produce seed crystals having an optimal particle size or optimal properties.

30 **[0007]** Furthermore, an appropriate amount of seed crystals is always required to stably advance the operation to produce nickel powder. On the other hand, preparation of excess seed crystals increases stock in progress and the labor for management thereof, thus reducing the production efficiency.

[0008] As described above, a method for stably obtaining an amount of seed crystals needed for the actual operation has been demanded.

35 Citation List

Patent Literature

40 **[0009]**

Patent Literature 1: Japanese Patent Application Laid-Open No. 2005-505695

Patent Literature 2: Japanese Patent Application Laid-Open No. 2010-242143

Patent Literature 3: Japanese Patent No. 4286220

45 Non Patent Literature

[0010] Non Patent Literature 1: "The Manufacture and properties of metal powder produced by the gaseous reduction of aqueous solutions", Powder metallurgy, No. 1/2 (1958), 40-52.

50 Summary of Invention

Technical Problem

55 **[0011]** In such circumstances, the present invention provides a method for producing nickel powder, wherein fine nickel powder serving as seed crystals required for production of nickel powder is produced from a solution containing a nickel ammine sulfate complex according to the amount needed for the production of the nickel powder.

Solution to Problem

[0012] A first aspect of the present invention to solve the above problem is a method for producing nickel powder, wherein: a solution containing a nickel ammine sulfate complex, an insoluble solid, and a dispersant are continuously fed into a reaction vessel, followed by stirring to prepare a solution containing a nickel complex ion; hydrogen gas is blown into the prepared solution to reduce the nickel complex ion in the solution containing the nickel complex ion, thereby forming a composite including a precipitate of nickel particles on a surface of the insoluble solid, to thereby prepare a reduced slurry containing the composite; and thereafter the reduced slurry is extracted from the reaction vessel.

[0013] A second aspect of the present invention is a method for producing nickel powder, including, in sequence: a mixing step of adding an insoluble solid as seed crystals and a polyacrylate or a lignosulfonate as a dispersant to a solution containing a nickel ammine sulfate complex to form a slurry; and a reduction and precipitation step of charging the slurry into a reaction vessel, and blowing hydrogen gas into the slurry in the reaction vessel to reduce a nickel complex ion in the slurry, thereby forming a precipitate of nickel particles on the surface of the insoluble solid, wherein the precipitate is made into nickel powder.

[0014] A third aspect of the present invention is a method for producing nickel powder, including, in sequence: a mixing step of adding an insoluble solid as seed crystals and a polyacrylate or a lignosulfonate as a dispersant to a solution containing a nickel ammine sulfate complex to form a slurry; and a reduction and precipitation step of charging the slurry into a reaction vessel, blowing hydrogen gas into the slurry charged to reduce a nickel complex ion in the slurry, thereby forming a nickel precipitate on the surface of the insoluble solid, wherein the precipitate is made into nickel powder, and an amount of dispersant to be added in the mixing step is controlled to control the number of nickel powder obtained through the formation of the nickel precipitate in the reduction and precipitation step.

[0015] A fourth aspect of the present invention is a method for producing nickel powder, wherein when the polyacrylate is used as the dispersant added in the mixing step in the second and third aspects, the polyacrylate is added in an amount of more than 1.0% by weight and 10.0% by weight or less based on the weight of the insoluble solid added to the slurry.

[0016] A fifth aspect of the present invention is a method for producing nickel powder, wherein the polyacrylate is added in an amount of 2.0 to 6.0% by weight based on the weight of the insoluble solid as seed crystals in the fourth aspect.

[0017] A sixth aspect of the present invention is a method for producing nickel powder, wherein the polyacrylate in the fourth and fifth aspects is sodium polyacrylate (PAA).

[0018] A seventh aspect of the present invention is a method for producing nickel powder, wherein when the lignosulfonic acid is used as the dispersant added in the mixing step in the second and third aspects, the lignosulfonic acid is added in an amount of 2.0% by weight or more and 20.0% by weight or less based on the weight of the insoluble solid added to the slurry.

Advantageous Effects of Invention

[0019] The present invention can provide a method for economically and efficiently producing fine nickel powder, which is optimal as the seed crystals used in production of nickel powder, from a nickel ammine sulfate complex solution through reduction and precipitation using hydrogen gas according to the amount needed, and which exhibits a remarkable industrial effect.

Brief Description of Drawings

[0020]

[Figure 1] This is a production flowchart in a method for producing nickel powder according to the present invention, in which a dispersant and an insoluble solid are added.

[Figure 2] This is a graph illustrating a change in the concentration of nickel in the solution after the reaction in Examples 1 to 4 where sodium polyacrylate is used.

[Figure 3] This is a graph illustrating a change in the concentration of nickel in the slurry of Comparative Example 2 (dispersant is not added) plotted against the reaction time, where the change is caused by the concentration of the dispersant during the reduction with hydrogen.

[Figure 4] This is a graph illustrating the relation between the number of nickel powder and the amount of sodium polyacrylate added according to Example 5.

[Figure 5] This is a graph illustrating the relation between the number of nickel powder and the amount of sodium lignosulfonate added according to Example 6.

[Figure 6] This is a graph illustrating the comparison in particle size distribution between the insoluble solid (nickel powder) as seed crystals and the produced nickel powder according to Example 7.

Description of Embodiments

[0021] The present invention is a method for producing nickel powder by adding a dispersant and an insoluble solid as seed crystals to a nickel ammine sulfate complex solution, and blowing hydrogen gas thereinto, wherein a target amount of fine nickel powder is produced through control of the amount of dispersant to be added.

[0022] Hereinafter, the method for producing nickel powder according to the present invention will be described with reference to the production flowchart in Figure 1.

[Nickel ammine sulfate complex solution]

[0023] 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 depending on the components 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.

[Mixing step]

[0024] In this step, first, a dispersant is added to the nickel ammine sulfate complex solution.

[0025] Any polyacrylate or lignosulfonate dispersant can be used in this step without particular limitation. Among those industrially available at low cost, suitable polyacrylates are calcium polyacrylate, sodium polyacrylate and potassium polyacrylate, and suitable lignosulfonates are calcium lignosulfonate, sodium lignosulfonate, and potassium lignosulfonate.

[0026] The concentration of ammonium sulfate in the solution is preferably in the range of 10 to 500 g/L in conjunction with the production method illustrated in Figure 1. A concentration of 500 g/L or more is beyond the solubility, precipitating crystals. A concentration of less than 10 g/L is difficult to achieve because ammonium sulfate is newly generated by the reaction.

<Addition of insoluble solid>

[0027] In the production method according to the present invention illustrated in Figure 1, in the next step, an insoluble solid, which is at least insoluble in the nickel ammine sulfate complex solution having an adjusted concentration of the dispersant and serves as a matrix for a precipitate, is added to the complex solution.

[0028] Any insoluble solid having low solubility to the nickel ammine sulfate complex solution, an ammonium sulfate aqueous solution, or an alkali solution can be added here without particular limitation. For example, nickel powder, iron powder, alumina powder, zirconia powder, or silica powder can be used.

[0029] Unlike the conventional methods generally used in which powder is precipitated using seed crystals to make it into a product containing the seed crystals, in the present invention, after the necessary precipitation of particles onto the surface of the insoluble solid is completed, the insoluble solid is separated from the yielded and grown precipitate, and only the powder of the separated precipitate is extracted as a product. Such a method in the present invention is intended to avoid the influences of impurities in the seed crystals itself over the product.

[0030] Any amount of insoluble solid can be added without particular limitation. The amount thereof is selected according to the type of the solid such that the insoluble solid can be mixed with the nickel ammine sulfate complex solution through stirring. As one example, an amount of about 50 to 100 g/L may be added.

[0031] The insoluble solid can also have any shape and size without particular limitation. Suitable are those having strength to stand impact or friction and having a smooth surface so as to effectively separate the nickel precipitate, because the nickel precipitates on the surface of the insoluble solids may be separated through application of collision or vibration as described later.

[0032] Considering the effective separation of the insoluble solid from the nickel precipitate on the surface thereof, the insoluble solid suitably used in the actual operation has a rounded shape such as a spherical or elliptical shape and has a diameter of about 0.05 to 3 mm. Preferably, the insoluble solid according to the present invention is used after adhering substances to the surface of the insoluble solid are removed through the application of collision or impact to the insoluble solid prior to the precipitation of nickel.

[0033] Furthermore, the insoluble solid after the separation of the nickel precipitate can be subjected to a pre-treatment such as washing, when necessary, and can be repeatedly used.

<Addition of dispersant>

[0034] The present invention is characterized in that the insoluble solid is used as seed crystals, and the dispersant is added. The dispersant can sufficiently disperse the added insoluble solid in a complex solution to generate a fine nickel precipitate on the surface of the insoluble solid. The dispersant is added desirably in an appropriate amount in the range of 1.0 to 20.0% by weight of the weight of the insoluble solid added to the complex solution. In particular, a polyacrylate and a lignosulfonate are preferred.

1. Case where a polyacrylate is used as dispersant

[0035] In the case where the insoluble solid is used as seed crystals and a polyacrylate is used as a dispersant (production method illustrated in the production flowchart in Figure 1), the amount of the dispersant to be added is more than 1.0% by weight and 10.0% by weight or less based on the weight of the insoluble solid added to the slurry, and desirably 2.0% by weight or more and 6.0% by weight or less.

[0036] At an addition amount of 1.0% by weight or less, the nickel powder does not precipitate. An amount of 2.0% by weight or more is preferred because the insoluble solid can be sufficiently dispersed to control the number of generated nickel powder proportionately with the amount of the dispersant added.

[0037] In contrast, the seed crystals tend to be increased even if the upper limit of the amount of the dispersant is beyond 6.0% by weight. An excess large number of seed crystals generated causes handling difficulties and aggregation between the dispersants. Considering the effect commensurate with the amount of the dispersant added, such an upper limit is not preferred. Accordingly, the upper limit is 10.0% by weight or less, more preferably 6.0% by weight or less.

2. Case where a lignosulfonate is used as dispersant

[0038] In the case where a lignosulfonate is used as a dispersant (production method illustrated in the production flowchart in Figure 1), the amount of the dispersant to be added is 2.0% by weight or more and 20.0% by weight or less based on the weight of the insoluble solid added to the slurry. An amount of 2.0% by weight or less cannot yield nickel powder, so that the amount of the dispersant is required to be more than 2.0% by weight. In particular, an amount of more than 5.0% by weight is preferred because the number of nickel powder generated can be controlled proportionately with the amount of the dispersant added.

[Reduction and precipitation step]

[0039] Next, the nickel complex ion in the slurry is reduced with hydrogen to form a composite including the insoluble solid and a nickel precipitate on the surface thereof. This "reduction and precipitation step" can be performed in a batch manner and a continuous manner.

[0040] First, in the "reduction and precipitation step" where the reduction and the precipitation are performed in batch, a slurry formed of a dispersant and an insoluble solid added is charged into a reaction vessel in a container resistant to high pressure and high temperature. Hydrogen gas is blown into the slurry contained in the reaction vessel of the container resistant to high pressure and high temperature to reduce the nickel complex ion in the slurry, thereby forming a reduced slurry containing a composite including the insoluble solid contained in the slurry and nickel generated as a precipitate on the surface thereof.

[0041] At this time, the reaction temperature is preferably in the range of 150 to 200°C. A reaction temperature of less than 150°C reduces the reduction efficiency. A reaction temperature of 200°C or more has no influences over the reaction; rather, such a temperature is not suitable because it increases loss of thermal energy.

[0042] Furthermore, the pressure during the reaction is preferably 1.0 to 4.0 MPa. A pressure of less than 1.0 MPa reduces the reaction efficiency. A pressure of more than 4.0 MPa has no influences over the reaction; rather, it increases loss of hydrogen gas.

[0043] Next, the slurry formed of the dispersant and the insoluble solid added is continuously fed into a reaction vessel of a container resistant to high pressure and high temperature, and hydrogen gas is continuously blown into the slurry flowing in the reaction vessel to reduce nickel complex ion in the slurry. Thereby, a reduced slurry containing a composite including the insoluble solid and a nickel precipitate formed on the surface thereof is obtained. After the reduction reaction to generate the nickel precipitate, the resulting reduced slurry is continuously extracted and recovered from the reaction vessel, and is fed to the subsequent step.

[0044] In other words, by performing the reduction reaction by such a continuous step, the time required to replace the slurry or set the conditions for the reduction treatment can be reduced, and an improvement in production efficiency can be expected. Control of the flow rate of the slurry enables the adjustment of the amount of production, resulting in a reduction in size of the reaction vessel and economical advantages such as a reduction in capital investment and

repairs of facilities.

[0045] In such a reduction and precipitation step, the reaction temperature is preferably in the range of 150 to 200°C. A reaction temperature of less than 150°C reduces the reduction efficiency. A reaction temperature of 200°C or more has no influences over the reaction; rather, it is not suitable because it increases loss of the thermal energy.

[0046] Furthermore, a pressure of 1.0 to 4.0 MPa is preferably applied to a gaseous phase portion in the reaction vessel during the reaction. A pressure of less than 1.0 MPa reduces the reaction efficiency. A pressure of more than 4.0 MPa has no influences over the reaction; rather, it increases loss of hydrogen gas.

[0047] Because of the effect of the dispersant in the reduction and precipitation treatment according to the present invention, the insoluble solid can be sufficiently dispersed in the slurry. In such a state, a nickel precipitate in the form of a finely powdery precipitate can be formed on the surface of the insoluble solid. Nickel can be extracted and recovered from the nickel ammine sulfate complex solution. Furthermore, the amount of nickel powder generated through precipitation can also be adjusted through the adjustment of the amount of the dispersant to be added.

[Separation step]

[0048] This step is performed when the insoluble solid is used. In this case, the nickel precipitate generated in the reduction and precipitation step adheres to the surface of the insoluble solid, and cannot be used in such a state. For this reason, the nickel precipitate formed on the surface of the insoluble solid is separated from the insoluble solid, and is recovered.

[0049] Examples of specific separation methods include a method of placing an insoluble solid and a nickel precipitate into water to avoid oxidation due to heat generated, colliding the insoluble solids each other under rotating to separate the nickel precipitate from the surface of the insoluble solid, and sieving the nickel precipitate to obtain nickel powder; a method of rotating a nickel precipitate on a wet sieve, and simultaneously sieving the separated nickel precipitate to obtain nickel powder; or a method of separating a solution through ultrasonic vibration, and sieving the resulting nickel precipitate to obtain nickel powder. Any sieve having an opening smaller than the insoluble solid can be used in the sieving step.

[0050] The nickel powder thus produced can be used in application to nickel pastes as an inner constitutional substance of stacked ceramic capacitors, for example. Besides, high purity nickel metal can be produced through repetition of the reduction with hydrogen using the recovered nickel powder as seed crystals to grow particles.

Examples

[0051] Hereinafter, the present invention will be described by way of Examples.

Example 1

[Mixing step]

[0052] 336 g of nickel sulfate hexahydrate, which amount is equivalent to 75 g of nickel content, 330 g of ammonium sulfate, and 191 ml of 25% aqueous ammonia were added to prepare a nickel ammine sulfate complex solution. According to the production flow in Figure 1, first, 75 g of nickel powder having an average particle size (D50) of 85 μm as an insoluble solid serving as a precipitation matrix for seed crystals, and sodium polyacrylate having a molecular weight of 4000 as a dispersant in an amount of 1.5 g was added, which was equivalent to 2% by weight of the weight of the insoluble solid serving as seed crystals. Pure water was added so that the volume of the solution was adjusted to 1000 ml. Thereby, a slurry was formed.

[Reduction and precipitation step]

[0053] The resulting slurry was then placed into an internal cylindrical can of an autoclave. The slurry was heated to 185°C with stirring. While the temperature was maintained, hydrogen gas was blown and fed into the slurry from a hydrogen tank so that the pressure in the autoclave became 3.5 MPa.

[0054] A sample of reduced slurry was extracted from the sample outlet of the autoclave every two minutes after the start of feed of hydrogen gas, and was subjected to solid liquid separation. The concentration of nickel in the filtrate was analyzed.

[0055] As the reaction progresses, nickel is precipitated as powder, and in turn, the concentration of nickel in the filtrate is reduced by the amount of the nickel precipitate. As shown in Figure 2, from the calculation of a change in the concentration of nickel, 80% or more of nickel could be reduced and recovered in 30 minutes.

[0056] After the lapse of 30 minutes from the start of feeding hydrogen gas, the feed of hydrogen gas was stopped,

and the internal cylindrical can was cooled. After the cooling, the slurry in the internal cylindrical can was filtered to recover 42.7 g of precipitated nickel powder.

[0057] The recovered nickel powder was observed. It was confirmed that fine nickel powder usable as seed crystals was generated.

Example 2

[0058] Nickel powder was produced, and was recovered by the same method and conditions as in Example 1 except that 4.5 g of sodium polyacrylate, which amount is equivalent to 6% by weight of the weight of the seed crystals, was added.

[0059] The results are shown in Figure 2. As shown in Figure 2, 80% or more of nickel could be reduced and recovered in 30 minutes as in Example 1.

Example 3

[0060] Nickel powder was produced, and was recovered by the same method and conditions as those in Example 1 except that 7.5 g of sodium polyacrylate, which amount is equivalent to 10% by weight of the weight of the seed crystals, was added.

[0061] The results are shown in Figure 2. As shown in Figure 2, 80% or more of nickel could be reduced and recovered in 30 minutes as in Example 1.

Example 4

[0062] Nickel powder was produced, and was recovered by the same method and conditions as in Example 1 except that 0.75 g of sodium polyacrylate, which amount is equivalent to 1% by weight of the weight of the seed crystals, was added.

[0063] The results are shown in Figure 2. As shown in Figure 2, from the calculation of a change in the concentration of nickel, about 50% of nickel could be reduced and recovered in 30 minutes.

(Comparative Example 1)

[0064] Nickel powder was prepared with the same solution composition and reduction conditions as in Example 1 except that the dispersant and the insoluble solid were not added.

[0065] The concentration of nickel in the sampled solution was reduced from 75 g/L to about 45 g/L. Any nickel powder could not be recovered from the solution after the blowing of hydrogen gas was completed. It was confirmed that scaling of plate-like nickel was generated at the side walls inside the internal cylindrical can and the stirrer.

(Comparative Example 2)

[0066] Nickel powder was produced by the same method as in Example 1 except that the dispersant was not added and 75 g of nickel powder as an insoluble solid was added.

[0067] The results are shown in Figure 3. As shown in Figure 3, from the calculation of a change in the concentration of nickel, only about 20% of nickel could be recovered in 30 minutes.

Example 5

[0068] 191 ml of 25% aqueous ammonia was added to a solution containing 336 g of nickel sulfate hexahydrate, which amount is equivalent to 75 g of nickel, and 330 g of ammonium sulfate to prepare a nickel ammine sulfate complex solution. Furthermore, according to the production flow illustrated in Figure 1, a solution of sodium polyacrylate having a molecular weight of 4000 and a concentration of 40% was added in an amount of 0.38 g, 1.88 g, 3.75 g, 7.5 g, and 11.3 g to the resulting nickel ammine sulfate complex solution, respectively, and the total volume of the solutions was adjusted to 1000 ml. Thereby, five solutions were prepared.

[0069] 75 g of nickel powder having an average particle size (D50) of 85 μm as an insoluble solid serving as the precipitation matrix was added to each of the resulting solutions to prepare a desired slurry. The net amounts of the sodium polyacrylate added here were equivalent to 0.2% by weight, 1.0% by weight, 2.0% by weight, 4.0% by weight, and 6.0% by weight, respectively, based on the amount of the insoluble solid.

[0070] The resulting mixture slurries were placed into an internal cylindrical can of an autoclave. Each slurry was heated to and held at 185°C with stirring. In this state, hydrogen gas was blown and fed into the slurry so that the pressure in the autoclave became 3.5 MPa. After the lapse of 60 minutes from the start of feeding hydrogen gas, the feed of

hydrogen gas was stopped, and the internal cylindrical can was cooled.

[Separation step]

[0071] After the cooling, the slurry in the internal cylindrical can was filtered to recover a composite of the insoluble solid and a nickel precipitate. The insoluble solid as the matrix and the nickel precipitate on the surface of the insoluble solid were separated under vibration using a wet sieve having an opening of 75 μm to recover nickel powder.

[0072] The particle size of the recovered nickel powder under the sieve was measured with a particle size distribution analyzer (made by Microtrac, Inc., trade name 9320-X100 type) to determine the particle size distribution.

[0073] Assuming that the recovered nickel powder was a true sphere, using the determined average particle size: D and the nickel density: $\rho=8.9 \text{ g/cm}^3$, the number of recovered nickel powder was calculated from Expression (1):

[Expression 1]

$$\text{The Number of nickel powder} = (\text{Mass of recovered nickel powder}) / [8.9 \times 4\pi \times (D/2)^3 / 3] \dots (1)$$

[0074] The relation between the number of nickel powder calculated from Expression (1) and the amount of sodium polyacrylate added is shown in Figure 4.

[0075] From Figure 4, it is found that there is a correlation between the amount of sodium polyacrylate added and the number of nickel powder, and that the amount of nickel powder generated can be adjusted by the amount of sodium polyacrylate added. In particular, it is found that an amount of added sodium polyacrylate of 1.0% by weight or less cannot yield nickel powder, and an amount of more than 1.0% by weight can control the number of generated nickel powder proportionately with the amount of sodium polyacrylate added.

Example 6

[0076] Nickel powder was produced by the same method as in Example 1 except that sodium lignosulfonate was used as a dispersant in amounts of 1.5 g, 3.0 g, 4.5 g, 7.5 g, 11.3 g, and 15.0 g, respectively. The amounts of insoluble solid of sodium lignosulfonate added are equivalent to 2.0% by weight, 4.0% by weight, 6.0% by weight, 10.0% by weight, 15.0% by weight, and 20.0% by weight, respectively.

[0077] For the resulting nickel powder, the number of nickel powder was calculated as in Example 5 through calculation using Expression (1).

[0078] The relation between the number of nickel powder calculated from Expression (1) and the amount of sodium lignosulfonate added is shown in Figure 5.

Example 7

[0079] Water and 3 g/l of sodium polyacrylate as a dispersant, which amount is equivalent to 2% by weight of the weight of an insoluble solid, were added to a nickel ammine sulfate complex solution containing 83 g/L of nickel ions, 120 g/L of ammonium sulfate, and 182 g/L of 25% aqueous ammonia and nickel powder having an average particle size (D50) of 90 μm as the insoluble solid to prepare a seed crystal slurry containing nickel powder having a concentration of 165 g/L.

[0080] The nickel ammine sulfate complex solution and the seed crystal slurry prepared above were then continuously fed to an autoclave using a pump. While the autoclave was held at 185°C with stirring, hydrogen gas was blown and fed into the slurry from a hydrogen tank so that the pressure in the cylindrical can of the autoclave became 3.5 MPa. At this time, after hydrogen gas was blown into the slurry, the slurry in the autoclave was stagnated for one hour. To keep a constant amount of the solution in the autoclave, the feed and discharge amounts of the nickel ammine sulfate complex solution and the seed crystal slurry were adjusted, and the slurry after the reaction was continuously extracted and recovered from the autoclave.

[0081] The number of nickel powder was calculated through the calculation using Expression (1) from the weight of the obtained nickel powder.

[0082] As a result, Table 1 shows that the number of particles increased, and the particle size distribution in Figure 6 shows that fine nickel powder was produced.

[Table 1]

	Seed crystals*1	Produced nickel powder
The number of nickel powder [$\times 10^6$ particles/L]	410	18000
*1: 90 μ m nickel powder		

Claims

1. A method of producing nickel powder, wherein: a solution containing a nickel ammine sulfate complex, an insoluble solid, and a dispersant are continuously fed into a reaction vessel, followed by stirring to prepare a solution containing a nickel complex ion; hydrogen gas is blown into the solution containing the nickel complex ion to reduce the nickel complex ion in the solution containing the nickel complex ion, thereby forming a composite including a precipitate of nickel particles on a surface of the insoluble solid, to thereby prepare a reduced slurry containing the composite; and thereafter the reduced slurry is extracted from the reaction vessel.
2. A method of producing nickel powder, comprising, in sequence:
 - a mixing step of adding an insoluble solid as seed crystals and a polyacrylate or a lignosulfonate as a dispersant to a solution containing a nickel ammine sulfate complex to form a slurry; and
 - a reduction and precipitation step of charging the slurry into a reaction vessel, and blowing hydrogen gas into the slurry to reduce a nickel complex ion in the slurry, thereby forming a precipitate of nickel particles on a surface of the insoluble solid, wherein
 the precipitate is made into nickel powder.
3. A method of producing nickel powder, comprising, in sequence:
 - a mixing step of adding an insoluble solid as seed crystals and a polyacrylate or a lignosulfonate as a dispersant to a solution containing a nickel ammine sulfate complex to form a slurry; and
 - a reduction and precipitation step of charging the slurry into a reaction vessel, blowing hydrogen gas into the slurry to reduce a nickel complex ion in the slurry, thereby forming a nickel precipitate on a surface of the insoluble solid, wherein
 - the precipitate is made into nickel powder, and
 - an amount of dispersant to be added in the mixing step is controlled to control the number of nickel powder obtained through the formation of the nickel precipitate in the reduction and precipitation step.
4. The method of producing nickel powder according to claim 2 or 3, wherein when the polyacrylate is used as the dispersant added in the mixing step, the polyacrylate is added in an amount of more than 1.0% by weight and 10.0% by weight or less based on a weight of the insoluble solid added to the slurry.
5. The method of producing nickel powder according to claim 4, wherein the polyacrylate is added in an amount of 2.0 to 6.0% by weight based on the weight of the insoluble solid as seed crystals.
6. The method of producing nickel powder according to claim 4 or 5, wherein the polyacrylate is sodium polyacrylate (PAA).
7. The method of producing nickel powder according to claim 2 or 3, wherein when the lignosulfonic acid is used as the dispersant added in the mixing step, the lignosulfonic acid is added in an amount of 2.0% by weight or more and 20.0% by weight or less based on the weight of the insoluble solid added to the slurry.

Fig.1

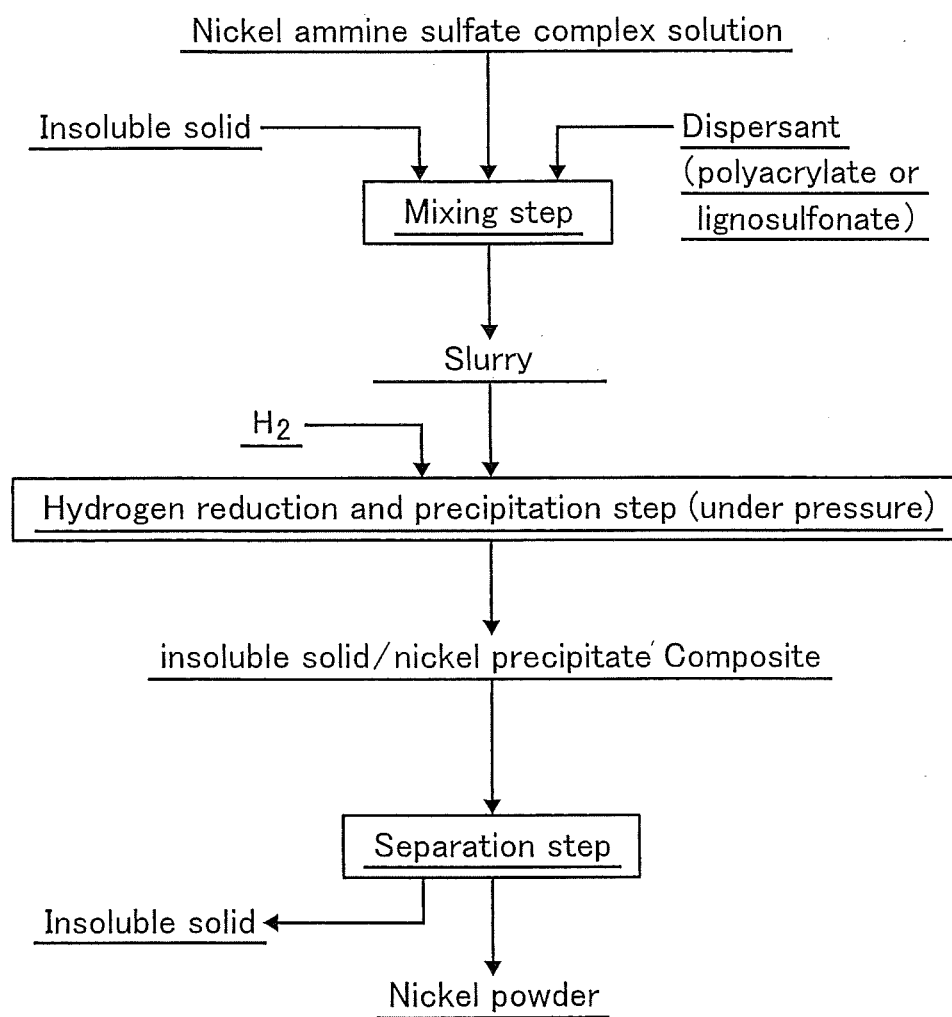


Fig.2

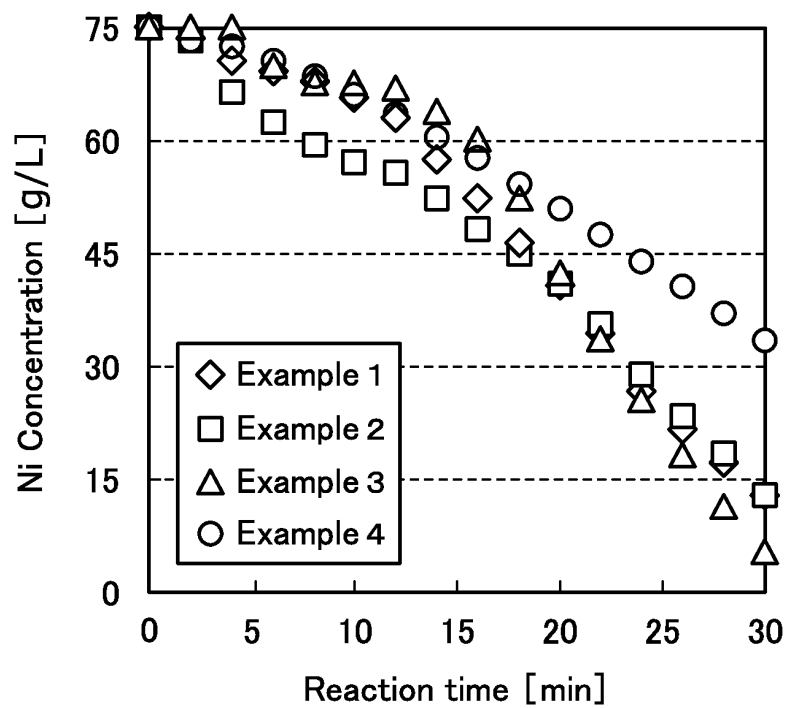


Fig.3

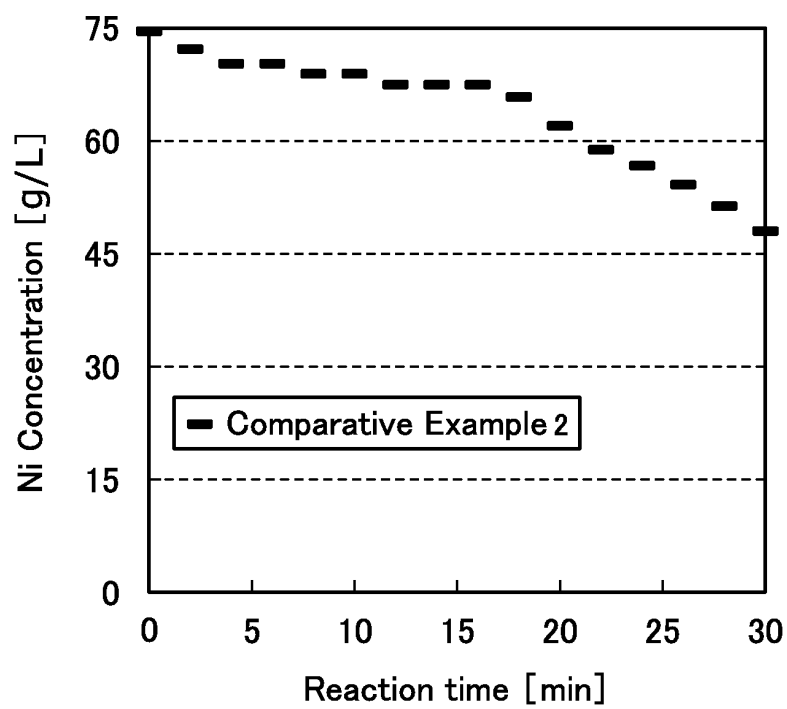


Fig.4

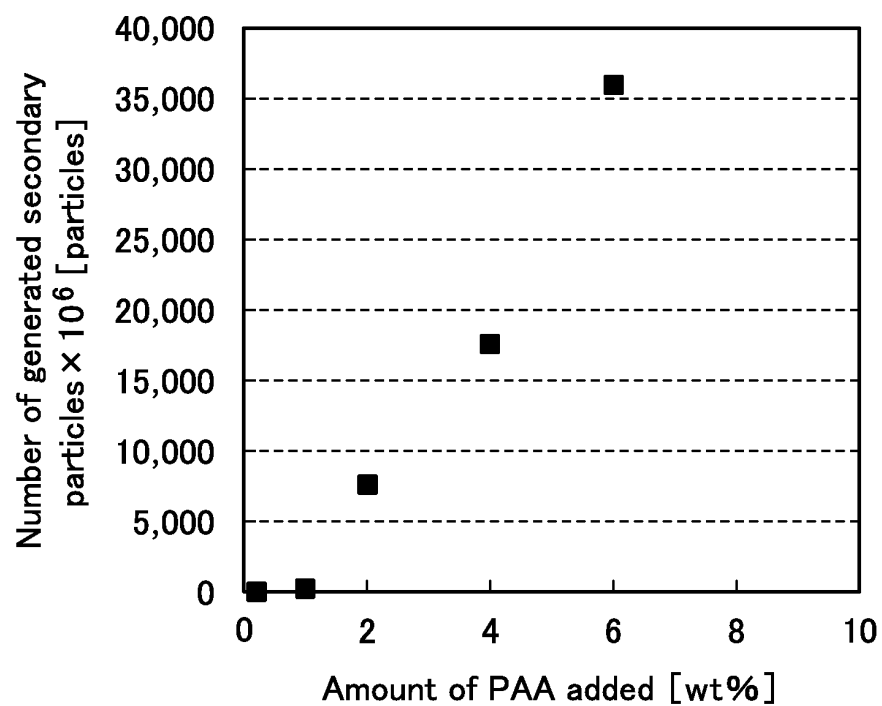


Fig.5

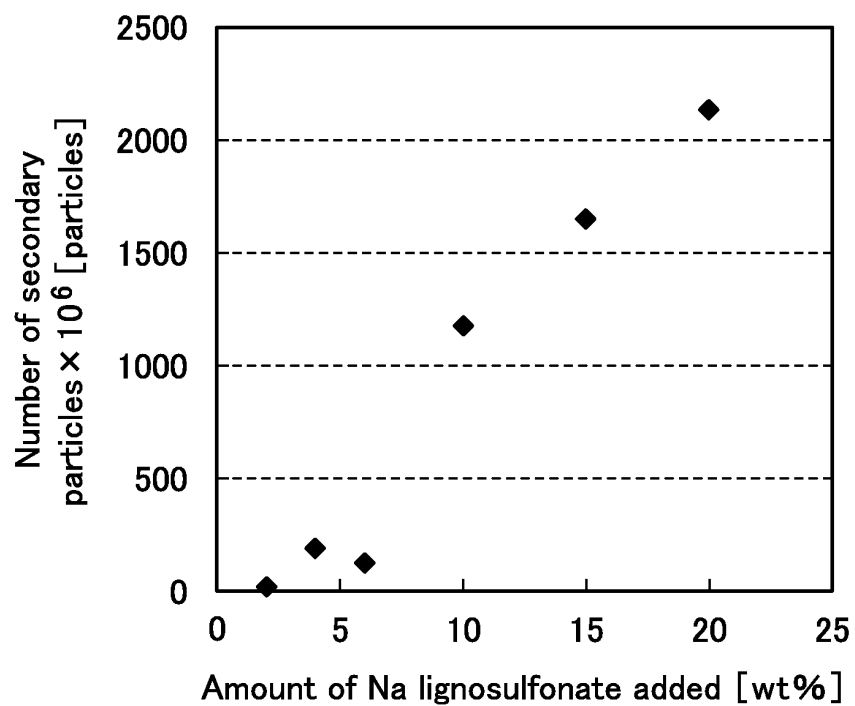
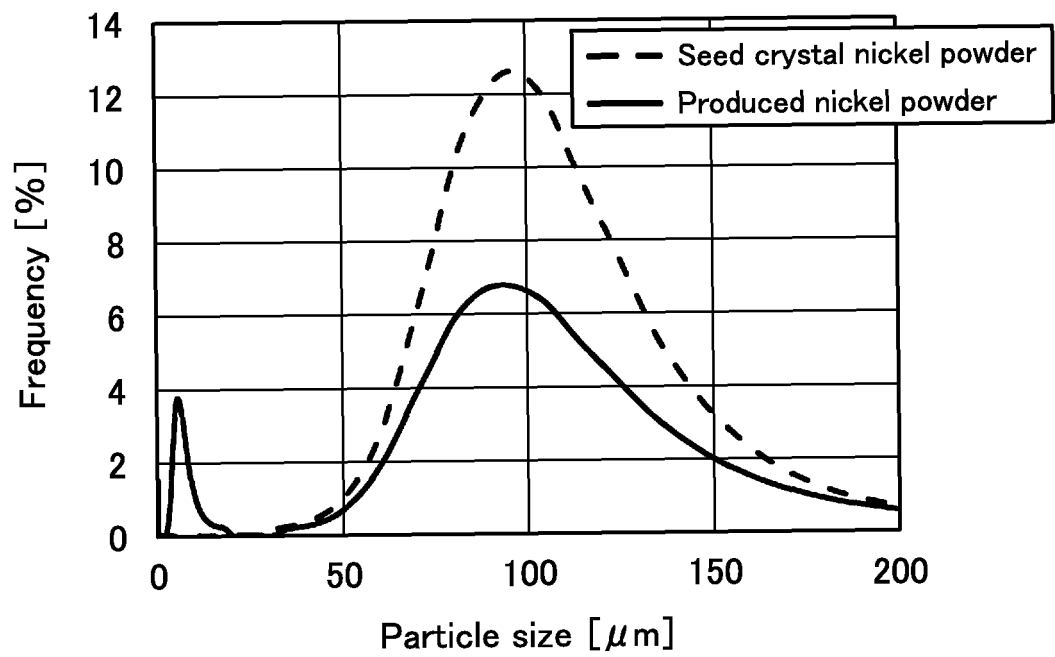


Fig.6



INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2017/006623

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.
X	JP 5796696 B1 (Sumitomo Metal Mining Co., Ltd.), 21 October 2015 (21.10.2015), claims 2 to 3, 5 to 8 & WO 2016/117138 A1	1-7
X	WO 2015/122534 A1 (National University Corporation Kochi University), 20 August 2015 (20.08.2015), claim 1; paragraph [0031] & JP 2015-166488 A & US 2016/0354844 A1 & EP 3108986 A1	1-2, 7

☐ 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
06 April 2017 (06.04.17)Date of mailing of the international search report
16 May 2017 (16.05.17)Name and mailing address of the ISA/
Japan Patent Office
3-4-3, Kasumigaseki, Chiyoda-ku,
Tokyo 100-8915, Japan

Authorized officer

Telephone No.

REFERENCES CITED IN THE DESCRIPTION

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Non-patent literature cited in the description

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