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(56) Related Art

W.KUNDA et al., "THE REDUCTION OF COBALT FROM ITS AQUEOUS AMMINE AMMONIUM SULPHATE SYSTEM USING HYDROGEN UNDER PRESSURE", Hydrometallurgy, (1979), vol. 4, no. 4, pages 347 - 375
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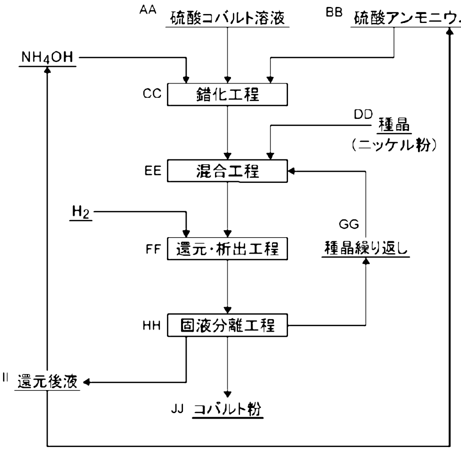
[続葉有]

(54) Title: METHOD FOR PRODUCING SEED CRYSTAL OF COBALT POWDER

(54) 発明の名称: コバルト粉の種結晶の製造方法



AA	Cobalt sulfate solution
BB	Ammonium sulfate
CC	Complexing step
DD	Seed crystal (nickel powder)
EE	Mixing step
FF	Reduction/deposition step
GG	Seed crystal/repeated
HH	Solid-liquid separation step
II	Liquid after reduction
JJ	Cobalt powder

(57) Abstract: Provided is a production method for efficiently obtaining a cobalt powder by means of a process which increases the reduction reaction efficiency during the production of the cobalt powder from a solution that contains a sulfuric acid cobalt ammine complex. A method for producing a seed crystal of a cobalt powder, which is characterized by sequentially performing: a complexing step for obtaining a solution containing a sulfuric acid cobalt ammine complex by adding ammonia and/or a solution of an ammonia compound into a cobalt sulfate solution; a mixing step for obtaining mixed slurry by adding a solid material into the solution containing a sulfuric acid cobalt ammine complex obtained in the complexing step; a reduction/deposition step for obtaining a cobalt powder slurry containing a cobalt deposit, in which the cobalt component is deposited, as a cobalt powder, on the solid material surface, by introducing the mixed slurry obtained in the mixing step into a reactor and reducing cobalt contained in the mixed slurry by blowing a hydrogen gas into the reactor; and a solid-liquid separation step for obtaining the cobalt deposit and the liquid after the reduction by subjecting the cobalt powder slurry obtained in the reduction/deposition step to solid-liquid separation.

(57) 要約: 硫酸コバルトアンミン錯体を含有する溶液からコバルト粉を製造する上で還元反応効率を上げる方法により効率良くコバルト粉を得る製造方法を提供。 硫酸コバルト溶液に、アンモニア、アンモニア化合物の溶液、或いはアンモニア及びアンモニア化合物の溶液の両者を添加して硫酸コバルトアンミン錯体を含有する溶液を得る錯化工程と、その錯化工程で得た硫酸コバルトアンミン錯体を含有する溶液に、固形物を加えて混合スラリーとする混合工程と、その混合工程で得た混合スラリーを反応槽内に装入し、反応槽に水素ガスを吹き込んで混合スラリーに含まれるコバルトを還元してコバルト成分がコバルト粉として固形物表面に析出したコバルト析出物を含有するコバルト粉スラリーを得る還元・析出工程と、その還元・析出工程で得たコバルト粉スラリーを固液分離してコバルト析出物と還元後液を得る固液分離工程を順に経ることを特徴とするコバルト粉の種結晶の製造方法。

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Description

Title of Invention:
METHOD FOR PRODUCING SEED CRYSTAL OF COBALT POWDER

Technical Field

[0001]

The present invention relates to a method for producing cobalt powder from a solution containing a cobalt ammine sulfate complex, and particularly relates to a method for obtaining seed crystals to use for crystal growth.

Background Art

[0002]

Various methods for obtaining cobalt, which is used as an electronic material or heat resistant alloys, have been known. In recent years, a demand for cobalt salts, which become raw materials such as battery materials, has been increased.

The cobalt salts are generally produced by dissolving a cobalt metal in an acid. However, sheet-shaped and massive forms such as conventionally common electrolytic cobalt are easily handled, but are extremely slowly dissolved in an acid. Meanwhile, the form of fine powder is easily dissolved in an acid, but has the handling disadvantage of being easily scattered and the like. Ones called briquets and obtained by consolidating or sintering grains or powder are preferred to make good use of the advantages of the both.

[0003]

As a method for obtaining such a small size of cobalt grains or powder, an atomizing method for dispersing melted cobalt in gas or water to obtain fine powder and a dry method such as CVD for obtaining cobalt powder by volatilizing cobalt and reducing it in a gas phase as shown in Patent Literature 1 are known.

[0004]

As a method for producing cobalt powder by a wet process, there are a method for generating it using a reducing agent as shown in Patent Literature 2 and a spray thermal decomposition method for obtaining cobalt powder by a thermal decomposition reaction by spraying a cobalt solution in a reduction atmosphere at a high temperature as shown in Patent Literature 3, and the like.

However, since these methods require expensive reagents and a large amount of energy, they are not economical as methods for industrially obtaining a large amount of material for the battery materials or the like.

[0005]

There has remained a problem that in a method for growing using cobalt powder as seed crystals as shown in patent Literature 4, seed crystals and grown cobalt powder are remelted, resulting in a decrease in the recovery since the reaction is performed in an acidic solution having a pH of 4 or less.

[0006]

Additionally, there has remained a problem that, in a method for adding a sodium hydroxide solution to a cobalt sulfate solution for a raw material and performing hydrogen reduction, the hydroxide of cobalt is generated and thereby allowing a reduction reaction not to proceed when the pH cannot be maintained at 4 and increases, and consequently the efficiency in the reduction reaction decreases.

[0007]

Meanwhile, a method for feeding hydrogen gas to a cobalt ammine sulfate complex solution having cobalt in the form of an ammonia complex to reduce cobalt ions in the complex solution and obtain cobalt powder as shown in Non Patent Literature 1 is industrially low-priced and useful.

However, also in this method, there is a problem that many crystal nuclei are generated heterogeneously, resulting in the prevention of the growth in the same way as the above-mentioned prior arts since particles are intended to be generated from a solution and grow by a wet reaction. That is, it is essential to control the number of the generated nuclei of the crystal in a proper range, resulting in efficient growth.

Then, as mentioned above, a method for feeding a reducing agent to a slurry in which a small amount of fine crystals called seed crystals coexist, and growing objects on the surfaces of the seed crystals to obtain powder having a predetermined particle size is commonly used.

[0008]

The seed crystals added in the above are often used by conducting treatment such as grinding a portion of a product repeatedly. However, there has remained a problem that the processing also took time and effort and the yield decreased as the processing is repeated more times and therefore this lead to an increase in cost. There has also remained the problem of seed crystals having the optimal particle size and properties being capable of necessarily being obtained stably only by grinding simply, or the like.

That is, a method for stably obtaining seed crystals to use for crystal growth has been required.

Citation List

Patent Literature

[0009]

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

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

Patent Literature 3: Japanese Patent No. 4286220

Patent Literature 4: Japanese Patent Laid-Open No. S57-54207

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), pp 40-52.

Summary of Invention

Technical Problem

[0011]

In such a situation, the present invention provides a production method for obtaining cobalt powder efficiently by a way for increasing reduction reaction efficiency to produce the cobalt powder from a solution containing a cobalt ammine sulfate complex.

Solution to Problem

[0012]

A first aspect of the present invention to solve such a problem is a method for producing seed crystals of cobalt powder, sequentially including: a complexing step of adding ammonia, an ammonium compound solution, or both of ammonia and an ammonium compound solution to a cobalt sulfate solution to obtain a solution containing a cobalt ammine sulfate complex; a mixing step of adding nickel powder as a solid that is insoluble or slightly soluble in the solution containing the cobalt ammine sulfate complex, to the solution containing the cobalt ammine sulfate complex obtained in the complexing step to form a mixture slurry; a reduction and precipitation step of charging a reaction vessel with the mixture slurry obtained in the mixing step, and blowing hydrogen gas into the reaction vessel to reduce cobalt contained in the mixture slurry and obtain a cobalt powder slurry containing cobalt precipitate with a cobalt component precipitated on the surface of the solid as cobalt powder; and a solid-liquid separation step having a solid-liquid separation treatment for separating the obtained cobalt powder slurry into the cobalt precipitate and a solution after reduction, and a solid separation treatment for separating the obtained cobalt precipitate into the added nickel powder of the solid and the cobalt powder that is precipitated on the surface of the nickel powder of the solid, to form the solution after reduction, the nickel powder of the solid, and the cobalt powder.

[0013]

A second aspect of the present invention is the method for producing seed crystals of cobalt powder, wherein the nickel powder of the solid in the first aspect has an average particle size of 0.1 µm or more and 5 µm or less.

[0014]

A third aspect of the present invention is the method for producing seed crystals of cobalt powder, wherein the cobalt concentration in the solution containing the cobalt ammine sulfate complex in the first and second aspects is 75 g/L or less.

Advantageous Effect of Invention

[0015]

According to the present invention, seed crystals having a size suitable to be add to a cobalt ammine sulfate complex solution as seed crystals to form cobalt powder can be obtained efficiently when a cobalt ammine sulfate complex solution is reduced with hydrogen gas to produce cobalt powder.

Brief Description of Drawing

[0016]

[Figure 1] Figure 1 is a production flow chart of the method for producing cobalt powder according to the present invention.

Description of Embodiment

[0017]

The present invention is a method for efficiently producing seed crystals to be add when hydrogen gas is blown into a cobalt ammine sulfate complex solution to produce cobalt powder.

[0018]

Hereinafter, the method for producing cobalt powder according to the present invention will described with reference to the production flow chart shown in Figure 1. In the present invention, cobalt powder is obtained by subjecting a cobalt sulfate solution serving as an original solution to a complexing step, a mixing step, a reduction and precipitation step and a solid-liquid separation step.

The reduction rate mentioned in the present invention is defined as a rate obtained by dividing the weight (g) of the obtained cobalt powder by the cobalt amount (g/L) contained in the fed cobalt sulfate solution (L).

[0019]

[Complexing Step]

A cobalt sulfate solution that can be used in the present invention is not particularly limited, but a cobalt leachate obtained by leaching/dissolving a cobalt-containing material such as an industrial intermediate comprising one or a mixture of two or more selected from mixed sulfide containing cobalt, crude cobalt sulfate, cobalt oxide, cobalt hydroxide, cobalt carbonate, cobalt powder, and the like with sulfuric acid or ammonia can be used. Usually, various impurities are also industrially contained in the cobalt leachate. The leachate is generally used by removing impurity elements in the leachate by subjecting the leachate to a purification step such as solvent extraction, ion exchange, or neutralization.

[0020]

Next, aqueous ammonia and ammonium sulfate are added to the cobalt leachate to obtain a cobalt ammine sulfate complex solution.

The concentration of ammonium sulfate in the solution is preferably in the range of 10 to 500 g/L. If the concentration is more than 500 g/L, it is higher than the solubility, and crystals may be precipitated. This is not preferable since an operational trouble occurs. If the concentration is less than 10 g/L, since ammonium sulfate is newly produced by the reaction, it is industrially difficult to keep the concentration at a level of less than 10 g/L.

The cobalt concentration in the cobalt ammine sulfate complex solution is 75 g/L or less. It is because in the reaction of a solid after the addition in later steps, if the cobalt concentration in the cobalt ammine sulfate complex solution is too high, the reduction rate will be decreased due to the shortage of reaction sites.

[0021]

[Mixing Step]

In this step, to the cobalt ammine sulfate complex solution prepared as described above is added a solid that is used as a matrix for precipitation.

The solid to be added is not particularly limited as long as it is insoluble or sparingly soluble with a low solubility in a cobalt ammine sulfate complex solution, an aqueous ammonium sulfate solution, or an alkali solution.

[0022]

Specifically, it is preferable to use nickel powder.

When cobalt powder is used for a solid, it is the same as cobalt precipitate, and therefore these do not need to be separated. Cobalt powder is thus the most suitable to be used as seed crystals, but it is difficult to industrially obtain fine cobalt powder at a low price stably.

[0023]

Although there is an advantage of iron powder being available at a low price easily, there is a disadvantage of iron powder being easily dissolved in an acidic solution and hardly serving as crystal nuclei. Because dissolved iron ions becomes a new cause of contamination, and the like, iron powder is not suitable.

In a method for using a slightly soluble or insoluble solid and precipitating cobalt thereon like the present invention, the influence of remelting is so avoidable as to be negligible and it is not necessary to repeat some of product, differently from a method for precipitating cobalt using seed crystals that is generally used conventionally and forming the cobalt including the seed crystals into a product. Therefore, there is a characteristic that cobalt complex ions contained in an ammine complex solution can be reduced nearly completely in a viewpoint of the balance between the amounts of materials in the process.

[0024]

It is preferable that the solid have a smooth surface so that the precipitated cobalt powder can be separated effectively. As mentioned above, the amount of the solid added equivalent to the amount of cobalt existing in the solution or more is required. Specifically, when nickel powder is used for a solid, 75 g/L or more need to be added.

[0025]

[Reduction and Precipitation Step]

Next, the reaction vessel of a pressure vessel is charged with the slurry formed by adding nickel powder in the mixing step, and cobalt complex ions in the slurry is reduced by blowing a reducing agent such as hydrogen gas into the reaction vessel to precipitate cobalt on the surface of the solid.

[0026]

Here, the temperature of the mixture slurry, namely, the reaction temperature, is preferably in the range of 150 to 200°C. If the reaction temperature is less than 150°C, reduction efficiency is decreased, and even if the reaction temperature is higher than 200°C, the reaction is not affected, but the loss of thermal energy increases.

[0027]

The pressure of the gas phase part, which is a space other than the solution in the reaction vessel, is preferably maintained in the range of 1.0 to 4.0 MPa by feeding hydrogen gas. If the pressure is less than 1.0 MPa, reaction efficiency is reduced since the amount of gas migrating from the gas phase part into the solution is little. Meanwhile, even if the pressure is higher than 4.0 MPa, there is no influence such as the reaction being enhanced, but the loss of hydrogen gas just increases, resulting in no advantage.

Hydrogen gas may be blown into the gas phase part in the reaction vessel or blown directly into the slurry.

[0028]

[Solid-Liquid Separation Step]

The solid having cobalt precipitate on the surface obtained in the reduction and precipitation step is taken out of the pressure vessel with the solution after reduction in the pressure vessel and subjected to solid-liquid separation from the solution after reduction.

This solid-liquid separation may be any of, for example, a method using a nutsche and a vacuum flask, a method using a centrifuge, a method using a filter press.

[0029]

Then, when a substance other than cobalt is used for the solid, operation of separating the cobalt precipitate on the surface from the solid may be performed.

Specific separation method can be properly performed by applying a shock to the solid and the cobalt precipitate, or the like.

[0030]

When the size of the cobalt precipitate still including the solid or the cobalt precipitate after separation from the solid is still smaller than that used for seed crystals, the size of the cobalt precipitate can be increased by repeating the mixing step again.

The solid collected here can be reused for the mixing step repeatedly.

The solution after reduction can be used repeatedly as a complexing agent in the complexing step without any treatment or by the recycling thereof into ammonia by treatment such as heating and distillation.

Examples

[0031]

Examples for generating seed crystals for obtaining cobalt powder of the present invention will be described below.

The average particle size was measured using a commercial laser particle size analyzer (Microtrac).

Example 1

[0032]

A solution containing a cobalt ammine sulfate complex was prepared by adding 191 ml of 25% aqueous ammonia to cobalt sulfate equivalent to 75 g of cobalt and 330 g of ammonium sulfate, dissolving them and adjusting the total volume of the solution to 1000 ml.

To this solution was added 75 g of nickel powder having an average particle size of 1 μm and used as a matrix for precipitation to obtain a mixture slurry.

[0033]

Next, an inner cylinder of an autoclave having a volume of 3 L was charged with the mixture slurry, the mixture slurry was then heated to 185°C with stirring, hydrogen gas was blown into the mixture slurry while keeping the temperature, and hydrogen gas was further fed so as to maintain the pressure in the inner cylinder of the autoclave at 3.5 MPa. After a lapse of 60 minutes from the start of the feeding of hydrogen gas, the feeding of hydrogen gas was stopped, and the inner cylinder was cooled.

[0034]

After cooling to room temperature, the mixture slurry in the inner cylinder was filtered, and the insoluble solid having cobalt precipitate formed on the surface was removed. Then, solid-liquid separation was conducted by suction filtration using a vacuum flask and a nutsche.

The reduction reaction rate of cobalt at this time was 99%.

[0035]

(Comparative Example 1)

A solution containing a cobalt ammine sulfate complex was prepared by obtaining a solution containing 75 g of cobalt, 330 g of ammonium sulfate and 191 ml of 25% aqueous ammonia, adding 3.73 g of polyacrylic acid having a concentration of 40 wt% as a dispersant instead of a solid of seed crystals of the present invention thereto, and adjusting the total volume of the solution to 1000 ml.

[0036]

The same inner cylinder of the autoclave as Example 1 was charged with the prepared solution, which was then heated to 185°C with stirring, hydrogen gas was blown into the mixture slurry while keeping the temperature, and hydrogen gas was further fed so as to maintain the pressure in the inner cylinder of the autoclave at 3.5 MPa.

After a lapse of 60 minutes from the start of the feeding of hydrogen gas, feeding hydrogen gas was stopped, and the inner cylinder was cooled.

[0037]

After cooling to room temperature, the solution in the inner cylinder was filtered, and the insoluble solid having cobalt precipitate formed on the surface was removed. Then, solid-liquid separation was conducted by suction filtration using the vacuum flask and the nutsche. The cobalt reduction reaction rate at this time was 72%, and as high efficiency as that of Example of the present invention was not obtained.

[0038]

(Comparative Example 2)

A solution containing a cobalt ammine sulfate complex was prepared by adding a solution containing 330 g of ammonium sulfate to a cobalt sulfate solution containing 75 g of cobalt, adding 191 ml of 25% aqueous ammonia to the resulting solution, and adjusting the total volume of the solution to 1000 ml.

To this solution was added 75 g of commercial iron powder having an average particle size of 1 μm as a solid soluble in the solution to prepare a mixture slurry.

[0039]

The inner cylinder of the autoclave used in Example 1 was charged with the prepared mixture slurry, the mixture slurry was then heated to 185°C with stirring, hydrogen gas was blown into the mixture slurry while keeping the temperature, and hydrogen gas was further fed so as to maintain the pressure in the inner cylinder of the autoclave at 3.5 MPa. After a lapse of 60 minutes from the start of the feeding of hydrogen gas, the feeding of hydrogen gas was stopped, and the inner cylinder was cooled.

[0040]

After cooling to room temperature, the mixture slurry in the inner cylinder was filtered, and the iron powder having cobalt precipitate formed on the surface was removed. Then, solid-liquid separation was conducted by suction filtration using the vacuum flask and the nutsche.

[0041]

It was found that the cobalt reduction reaction rate as to this Comparative Example 2 was 76%, which was higher than that in the case where a conventional dispersant was used, but as high effect as that of Example of the present invention was not obtained even if easily soluble solid was added.

[0042]

Throughout this specification and the claims which follow, unless the context requires otherwise, the word "comprise", and variations such as "comprises" or "comprising", will be understood to imply the inclusion of a stated integer or step or group of integers or steps but not the exclusion of any other integer or step or group of integers or steps.

[0043]

The reference in this specification to any prior publication (or information derived from it), or to any matter which is known, is not, and should not be taken as, an acknowledgement or admission or any form of suggestion that that prior publication (or information derived from it) or known matter forms part of the common general knowledge in the field of endeavour to which this specification relates.

The claims defining the present invention are as follows:

[Claim 1]

A method of producing seed crystals of cobalt powder, sequentially comprising:

a complexing step of adding ammonia, an ammonium compound solution, or both of ammonia and an ammonium compound solution to a cobalt sulfate solution to obtain a solution containing a cobalt ammine sulfate complex;

a mixing step of adding nickel powder as a solid that is insoluble or slightly soluble in the solution containing the cobalt ammine sulfate complex, to the solution containing the cobalt ammine sulfate complex obtained in the complexing step to form a mixture slurry;

a reduction and precipitation step of charging a reaction vessel with the mixture slurry obtained in the mixing step, and blowing hydrogen gas into the reaction vessel to reduce cobalt contained in the mixture slurry and obtain a cobalt powder slurry containing cobalt precipitate with a cobalt component precipitated on a surface of the solid as cobalt powder; and

a solid-liquid separation step having

a solid-liquid separation treatment for separating the cobalt powder slurry obtained into the cobalt precipitate and a solution after reduction, and

a solid separation treatment for separating the cobalt precipitate obtained into the nickel powder of the solid added and the cobalt powder that is precipitated on the surface of the nickel powder of the solid, to form the solution after reduction, the nickel powder of the solid, and the cobalt powder.

[Claim 2]

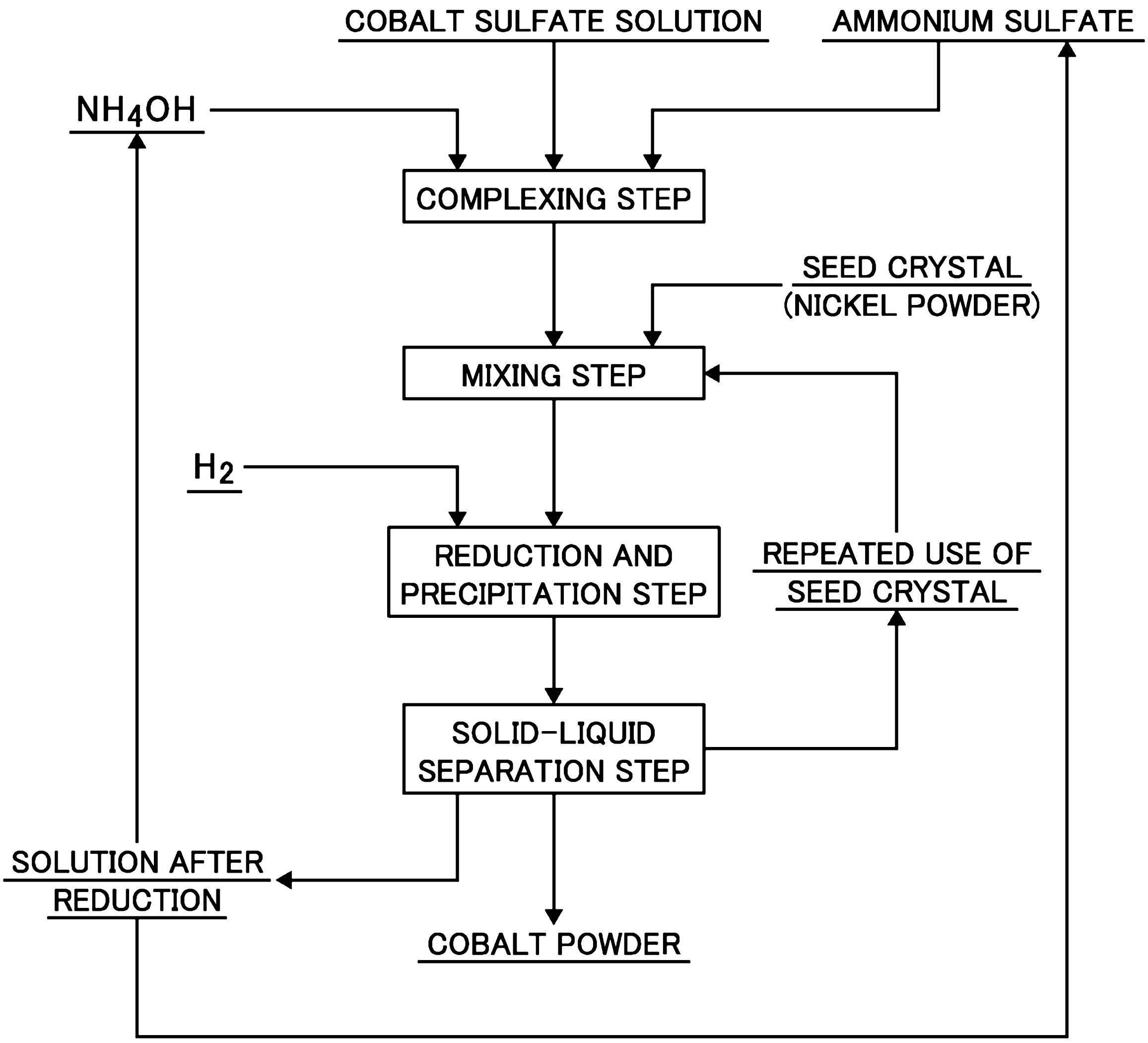
The method of producing seed crystals of cobalt powder according to claim 1,

wherein the nickel powder of the solid has an average particle size of 0.1 to 5 μm.

[Claim 3]

The method of producing seed crystals of cobalt powder according to claim 1 or 2, wherein a cobalt concentration in the solution containing the cobalt ammine sulfate complex is 75 g/L or less.

Fig.1


COBALT SULFATE SOLUTION
AMMONIUM SULFATE
NH4OH
COMPLEXING STEP
SEED CRYSTAL
(NICKEL POWDER)
MIXING STEP
H2
REDUCTION AND
PRECIPITATION STEP
REPEATED USE OF
SEED CRYSTAL
SOLID-LIQUID
SEPARATION STEP
SOLUTION AFTER
REDUCTION
COBALT POWDER