METHOD FOR PRODUCING HIGH-PURITY NICKEL FROM NICKEL MATTE

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ABSTRACT

A method for producing high purity nickel from nickel matte comprising successively the following steps: comminuting and roasting said matte at a temperature near 900°C to obtain nickel oxide; dissolving this impure nickel oxide in concentrated hydrochloric acid; oxidating to the ferric state the iron which is contained in the impure solution resulting from the preceding step removing the impurities from this solution by means of ion exchange resin and eluting said impurities to recover cobalt; and subjecting said purified solution to an electrolysis to recover high-purity nickel.

10 Claims, 1 Drawing Figure
METHOD FOR PRODUCING HIGH-PURITY NICKEL FROM NICKEL MATTE

The present invention relates to the production of high-purity nickel from nickel matte, whereby the expression "high-purity nickel" designates nickel with an amount of impurities lower than 0.05 wt.-percent. Nickel matte contains about 73 to 77 percent nickel and 20 to 23 percent sulfur and is generally obtained by subjecting nickel oxide ore to which a mixture of gypsum and coke is added to a sulfidizing smelting operation in a low-shaft furnace. Iron, a great amount of which is present in the obtained matte, is then burnt by blowing air into the molten matte and turned to slag by silica added during this operation which is carried out in convertors of the Bessemer type.

A matte which has been refined in this way presents for instance the following composition:

- Ni : 75.0 wt-%
- Co : 1.5 %
- Fe : 2.0 %
- S : 21.5 %

Various methods are known for treating such nickel mattes, but these known methods generally represent only one step in the long sequence of operations which leads to high-purity metal, i.e., a product containing more than 99.95 percent of nickel. Each of these steps presents a great number of possible treatments, which leads to a nearly infinite number of possible methods of production, so that it is improbable that even those skilled in the art can determine which method is industrially the most advantageous.

Therefore, one of the objects of the present invention is to provide a complete method for producing high-purity nickel from nickel matte, which involves lower cost and lower capital investment than do known methods.

Another object of the invention is to place an optimum technological solution at the disposal of those skilled in the art.

A further object of the invention is to provide such a method which can easily be employed on an industrial scale.

Yet a further object of the invention is to provide for the recovery of cobalt contained in the initial product.

In accordance with the present invention, the method comprises successively the following steps:

- comminuting and roasting the matte at a temperature near 900°C to obtain nickel oxide;
- dissolving this impure nickel oxide in concentrated hydrochloric acid;
- oxidating to the ferric state the iron which is contained in the impure solution resulting from the preceding step;
- removing the impurities from this solution by means of ion-exchange resin and eluting said impurities to recover cobalt and, subjecting said purified solution to an electrolysis to recover high-purity nickel.

In a first embodiment of the present invention, the elution of the impurities which are retained by said resin during the purification step is carried out in a single step and preferably the resin "AMBERLITE IRA 400" is used. "AMBERLITE IRA 400" is a registered trade-mark of the Company "ROHM & HAAS".

In another embodiment, the elution of the impurities is carried out in two consecutive steps whereby the first one washes away zinc, copper and iron and the second one allows the recovery of cobalt. The aforementioned resin IRA 400 is preferably used in this embodiment.

In order to lower the operating expense, it is useful to recover the chlorine released during the electrolysis of the purified solution, to bring it to react with hydrogen by any known method and to use the hydrochloric acid thus obtained for dissolving fresh amounts of the product resulting from the roasting of the initial matte.

When thus broadly stated, the process may appear simple, but the process is essentially a combination of steps, several of which are themselves novel, and in each step a considerable degree of care must be exercised.

An embodiment of the present invention will now be described, by way of example, with reference to the accompanying drawing which is a diagrammatical flow-sheet representing the various steps of the method according to the invention.

The composition of the initial matte, shown as 1 on the drawing, has been given hereinbefore. This matte is first comminuted, either by granulating molten matte or by crushing solidified matte.

In the first case, molten matte flows into a high-pressure water stream. 85 to 90 percent of the particles thus produced have a particle dimension lower than 800 microns.

In the second case, solid matte is first coarsely crushed in a jaw-crusher to a size of 80 to 100 mm, then finely crushed in a percussion-crusher till about 40 percent of the particles have the desired granulometry. The use of a sieve and of a ball-mill completes the comminution. The matte presents now the following granulometry:

- 5% tailings at 500 microns
- 50% tailings at 250 microns
- 90% tailings at 40 microns

The comminuted matte is then roasted at 2 at a temperature near 900°C.

The impure oxide 3 resulting from the roasting step 2 has the following composition:

- Ni : 75.8 wt-%
- Co : 1.52 %
- Fe : 2.02 %
- S : 2.0 %

and it is crushed till its particle size becomes lower than 125 microns.

It is then dissolved at 4 by hydrochloric acid under the following conditions: oxide and acid are continuously introduced into one or several reactors in series made of glass-coated steel and provided with means for agitating the mixture and for condensing the vapors. As the reaction is exothermal, it is easy to maintain the temperature near 106°C with, if necessary, providing a small amount of heat from outside.

The acid concentration is near 8 moles per liter and the amount of the hydrochloric solution introduced at 4 is about 1.1 times the amount which is stoichiometrically necessary for dissolving the whole amount of nickel and impurities.
The volume of the reactor or reactors is such that the time needed for dissolving the oxide is not longer than 4 hours. In these conditions, the dissolution yield is higher than 99 percent for nickel, iron and cobalt.

After filtering at 5 and removing the dissolution residue 9a, a solution is obtained which presents the following composition:

- Ni : 200 g/l
- Co : 4 g/l
- Fe : 5.3 g/l
- Cl⁻ : 270 g/l

Residual acidity : 0.4 N.

A fraction of the iron which is present in this solution is in the ferrous state and it is oxidized at 6 to the ferric state by bubbling air into the solution and adding hydrogen peroxide to the solution, whereby this addition is controlled by means of potentiometric measurements: this method avoids introducing hydrogen peroxide in excess.

This solution 7 is then subjected to a purification by percolation in two columns 8 and 9 in series which contain ion-exchange resin. A resin of the anionic type is used and, more particularly, a resin with quaternary ammonium active radicals, like the resin which is sold by the Company "ROHM & HAAS," under the trademark "AMBERLITE IRA 400."

During the percolation, the elements which are in the form of complex ions : Me⁺⁺ (Cl⁻)₄⁻ -->³⁰⁻, are retained by the active radicals of the resin.

This method makes use of the fact that the concentration of chloride ions in the solution is not high enough for the formation of a nickel complex. On the contrary, all the metallic impurities like zinc, iron, copper and cobalt are complexed and thus removed from the solution during the percolation.

In a first embodiment, which has been shown schematically on the left-hand side of the drawing, two resins columns 8a and 9a in series are used and the purification is stopped as soon as the presence of cobalt is detected in the solution flowing from the second column 9a.

The first column 8a is then rinsed by means of a concentrated solution of hydrochloric acid containing about 8 moles of acid per liter. The first fraction of the rinsing solution represents the impregnation solution of the resin and is added to the solution to purify 7. The second fraction is substantially only concentrated hydrochloric acid which contains all the metals of the initial solution at a low concentration. This second fraction is advantageously recycled at 4 for dissolving fresh amounts of oxide 3.

On the drawing, the path of the nickel solution has been shown by full lines, whereas rinsing and elution operations are shown by dotted lines.

The impurities retained by the first column 8a, are then subjected to elution by means of a diluted solution of hydrochloric acid at a concentration of 0.1 mole per liter. The eluate M contains the totality of the impurities zinc, copper, iron and cobalt. It is then possible to begin with a new purification cycle and to percolate fresh amounts of the solution 7 to purify, but now first on the second column 9a and then on the first column 8a. This operation has been shown by double lines on the drawing.

In another embodiment which has been shown on the right-hand side of the drawing, the flow of the solution on the first resin column 8b retains the following impurities iron, cobalt, copper and zinc. As cobalt is the least strongly retained element, it is displaced by the other metals and flows away into the nickel solution. The purification is interrupted when copper flows also away from the resin column 8b.

The column 8b is then rinsed by hydrochloric acid 3 times normal and regenerated by a decinormal solution of the same acid. The elution solutions are flushed away and the solution containing nickel and cobalt is passed on the second resin column 9b which retains only cobalt.

This column 9b is rinsed by hydrochloric acid eight times normal and eluted, which gives a pure cobalt solution, the total recovery yield of cobalt being about 70 to 80 percent.

Finally, an electrolysis 10 is carried out in both cases by means of insoluble anodes, for example graphite or of titanium anodes. With a current density of 600 A/m² and a voltage near 4.5 volts, this electrolysis step produces at 11 nickel with a purity higher than 99.95 percent.

The chlorine released during the electrolysis step 10 is brought to react at 12 with the same volume of hydrogen for the production of hydrochloric acid which is used at 4 for the dissolution of fresh amounts of oxide 3.

It is noteworthy to mention that this method can easily be made continuous by using two identical installations each being alternately used for purification and for elution.

What we claim is:

1. A method for producing high-purity nickel from impure nickel matte containing cobalt and other impurities comprising successively the following steps: comminuting and roasting said impure matte at a temperature of about 900°C to obtain impure nickel oxide; dissolving this impure nickel oxide in concentrated hydrochloric acid; oxidizing to the ferric state the iron which is contained in the impure solution resulting from the preceding step; removing the impurities from this solution by means of a ion-exchange resin to produce a purified nickel chloride solution; selectively eluting said impurities to recover a cobalt chloride solution; and subjecting said purified nickel chloride solution to an electrolysis to recover high-purity nickel.

2. A method as claimed in claim 1, wherein said impurities are eluted in a single step operation by means of an approximately decinormal hydrochloric acid.

3. A method as claimed in claim 2, wherein the first part of the solution resulting from said rinsing is added to said impure solution and the second part of the solution resulting from said rinsing is used for dissolving fresh amounts of said impure nickel oxide.

4. A method as claimed in claim 3, wherein said impurities are removed by means of two ion-exchange devices in series, said impure solution being introduced alternately into the first and the second ion-exchange device.

5. A method as claimed in claim 4, wherein the removing of said impurities is interrupted as soon as cobalt appears in said purified solution.
6. A method as claimed in claim 1, wherein the removing of said impurities is carried out by means of two ion-exchange devices in series, the elution of said impurities being carried out by approximately decinormal hydrochloric acid, the first one of said ion-exchange device being rinsed by hydrochloric acid about three times normal and the second one of said ion-exchange devices being rinsed by hydrochloric acid about eight times normal.

7. A method as claimed in claim 6, wherein the removing of said impurities is interrupted as soon as copper appears in the solution flowing from the first one of said ion-exchange devices.

8. A method as claimed in claim 7, wherein cobalt is recovered from the elution solution flowing from the second one of said ion-exchange devices.

9. A method as claimed in claim 1, wherein chlorine released during said electrolysis is recovered and brought to react with hydrogen, the hydrochloric acid thus produced being used for dissolving fresh amounts of said impure nickel oxide.

10. A method as claimed in claim 1, wherein the active radicals of said resin are quaternary ammonium radicals.

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