



(51) International Patent Classification:

C22B 1/06 (2006.01) *C22B 23/02* (2006.01)
C22B 1/10 (2006.01)

(21) International Application Number:

PCT/FI2012/050163

(22) International Filing Date:

20 February 2012 (20.02.2012)

(25) Filing Language:

English

(26) Publication Language:

English

(30) Priority Data:

20110054 21 February 2011 (21.02.2011) FI

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(81) Designated States (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BR, BW, BY, BZ,

CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

(84) Designated States (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, GH, GM, KE, LR, LS, MW, MZ, NA, RW, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Declarations under Rule 4.17:

- as to applicant's entitlement to apply for and be granted a patent (Rule 4.17(ii))
- of inventorship (Rule 4.17(iv))

Published:

- with international search report (Art. 21(3))

(54) Title: METHOD FOR ROASTING NICKEL SULPHIDE

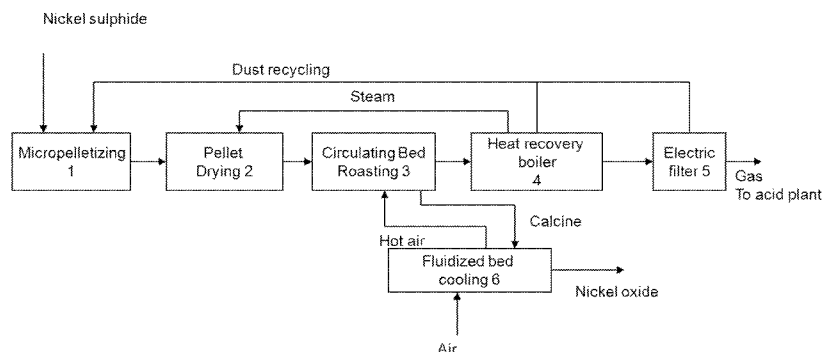


Figure 1

(57) Abstract: The invention relates to a method for roasting nickel sulphide produced by precipitation into nickel oxide in a fluidized bed furnace. The precipitated, very fine-grained nickel sulphide precipitate is micropelletized and the micropelletized precipitate is routed to fluidized bed treatment. Fluidized bed treatment may take place in a single fluidized bed furnace, in which there is a circulating fluidized bed, or in two fluidized bed furnaces in which there is a bubbling fluidized bed.

METHOD FOR ROASTING NICKEL SULPHIDE

FIELD OF THE INVENTION

The invention relates to a method for roasting nickel sulphide that is produced by precipitation into nickel oxide in a fluidized bed furnace. The precipitated, very fine-grained nickel sulphide precipitate is micropelletized and the micropelletized precipitate is routed to fluidized bed treatment. Fluidized bed treatment may take place in a single fluidized bed furnace in which there is a circulating fluidized bed, or in two fluidized bed furnaces having a bubbling fluidized bed.

10 BACKGROUND OF THE INVENTION

Metal sulphides have been oxidized on industrial scale in various kinds of roasting furnaces for decades. In sulphation roasting, sulphides are oxidized into sulphates. The formation of sulphates occurs at a lower temperature than that of oxides. In dead roasting the entire amount of sulphides is oxidized into oxide in oxidizing conditions at such a high temperature that the sulphates are no longer stable. Roasting can be done in different kinds of furnaces, such as a multi-hearth furnace, a rotary drum furnace or a fluidized bed furnace. The fluidized bed furnace is the one most commonly used, because it boasts efficient energy and heat transfer as well as temperature control.

20 Roasting technology has also been used in the oxidation of nickel sulphide, whereby the sulphide is roasted into either sulphate or oxide. The starting material in that case is either a nickel sulphide concentrate formed in flotation or a nickel matte generated in pyrometallurgical treatment. When the feed material is a concentrate, the nickel content is around 1–10%, but in nickel matte the content is generally around 40–70%. Both concentrate and matte usually also contain some amount of other metals, for example copper, iron, cobalt and arsenic. Nickel sulphide roasting is generally performed in a fluidized bed, in what is termed a bubbling bed.

30 Use of fluidized bed technology is sometimes restricted by excessive formation of molten phases and the resultant sintering of the bed. The oxidation of nickel sulphide into oxide occurs, in principle, at a temperature as low as about 700°C, but because of kinetics and for other reasons a temperature of about 900°C is used. According to the literature, molten phases appear in the Ni-S system at temperatures as low as 635°C, for example. The for-

mation of nickel sulphate is significantly reduced when the temperature rises above 700°C.

The roasting of sulphide concentrate containing nickel, such as pyrrhotite, is described in patent publications GB 769,267 and US 3,957,484. In the method according to the GB publication, the concentrate is roasted into sulphate and in the method described in the US publication, it is roasted into oxide. In sulphation roasting, sodium sulphate, for instance, can be used as an additive and a clay-containing material as a binder. In the methods, a fine concentrate (80% below 45 µm) is fed from the ceiling of a fluidized bed furnace as slurry, whereupon the slurry dries as it falls downward and agglomerates in the upper section of the furnace and the actual oxidation takes place in the lower section of the furnace. The fluidized bed consists of either calcine particles formed during roasting or calcine particles that were fed there in advance. The iron in the pyrrhotite is oxidized into hematite and the sulphides into sulphur dioxide. The temperature used was in the region of 1000–1100°C.

US patent publication 3,094,409 describes nickel sulphide roasting into nickel oxide in a fluidized bed. The sulphide to be treated is either a matte generated in pyrometallurgical treatment or flotation concentrate, which has a high nickel content of around 64–72%. If the material to be treated is a matte, the molten matte is granulated to the desired particle size for roasting. Flotation concentrate is fine-grained, the particle size mentioned is 95% minus 200 mesh, i.e. the majority is below 0.074 mm. Flotation concentrate is agglomerated to a size of 0.25–1.3 mm (65 mesh–0.5 in), whereupon for example water, nickel sulphate, sulphuric acid, lignosol, fuel oil or bentonite can be used as additive. According to the examples, the roasting temperature is around 1050–1100°C. Dust formed in roasting is either returned to the roaster or agglomeration.

GB patent publication 1,502,986 describes the roasting of a nickel-bearing iron sulphide in a fluidized bed furnace for the fabrication of ferronickel. The material to be roasted contains 5–35% nickel and it is agglomerated to a particle size of 6.7–1.2 mm. Roasting was carried out at a temperature of 800–1150°C. The higher the roasting temperature, the smaller the amount of sulphur remaining in the calcine. A large pellet size allows the use of a higher temperature, but requires a large gas flow for fluidization to succeed. At a high temperature part of the material melts and becomes dense, and slows diffusion. The oxide calcine that is formed is pre-reduced and fed into an electric

furnace to fabricate ferronickel. Since the calcine is fed into the smelter, sand can be used as the bed material.

A roasting treatment for sulphidic iron material is described in published SE patent application 346 703, in which the material to be treated contains one of the following substances as an impurity: arsenic, antimony, tin or bismuth. In addition to iron, the sulphide material may also contain cobalt, nickel, cadmium, copper or zinc, and it is observed that they may form ferrites during roasting. Roasting is performed in two fluidized bed reactors. In the first fluidized bed the temperature is controlled to be in the range of at least 800°C and the conditions are regulated to be reducing. In these conditions the impurities evaporate and the formation of ferrites is prevented. Roasting is brought to completion in the second fluidized bed reactor at a temperature of 700–800°C, giving rise to a sulphur-free magnetic material.

PURPOSE OF THE INVENTION

Roasting treatment of nickel sulphide concentrate or nickel matte has been disclosed in the prior art. However, the particle size of a concentrate and nickel matte are far larger than that of nickel sulphide formed by hydrometallurgical precipitation, and for that reason fluidized bed roasting of precipitated nickel sulphide as such creates major difficulties. The aim has been to solve the problems related to the fluidized bed roasting of fine-grained material within the framework of this invention.

SUMMARY OF THE INVENTION

The invention relates to a method for oxidizing nickel sulphide into oxide in a fluidized bed furnace, whereby a very fine-grained nickel sulphide precipitate formed by precipitation is treated in the following stages:

- a) the nickel sulphide precipitate is micropelletized by means of the sulphate dust separated from the fluidized bed furnace exhaust gases and
- b) the micropelletized precipitate is treated in one or two fluidized bed furnace(s) at a temperature of 650–1000°C in oxidizing conditions to form nickel oxide and sulphur dioxide-containing gas.

More particularly the invention relates to a method for oxidizing nickel sulphide into oxide in a fluidized bed furnace, whereby a very fine nickel sulphide precipitate formed by precipitation is treated in the following stages:

a) a micropelletizing stage wherein the nickel sulphide precipitate is micropelletized by means of sulphate dust separated from the fluidized bed furnace exhaust gases and

b) a roasting stage wherein the micropelletized precipitate is treated
5 in one or more fluidized bed furnace(s) at a temperature of 650–1000°C in oxidizing conditions to form nickel oxide and a gas containing sulphur dioxide.

According to one embodiment of the present invention in the roasting stage, the micropelletized precipitate is treated in two or more fluidized bed furnaces.

10 According to another embodiment of the present invention the micropelletized precipitate is treated in 1 to 6 fluidized bed furnaces. According to a further embodiment of the present invention the micropelletized precipitate is treated in 2 to 6 fluidized bed furnaces, for example in 2, 3, 4, 5 or 6 furnaces.

According to one embodiment of the invention, the roasting of the
15 micropelletized precipitate takes place as circulating fluidized bed roasting in a single fluidized bed furnace and a cyclone connected to it, whereby the temperature of the whole cycle is adjusted to 750–1000°C. In this case, the flow of micropellets to be circulated is at least 100–300% of the amount of fresh micropelletized nickel sulphide precipitate fed into the furnace.

20 According to another embodiment of the invention, the micropelletized nickel sulphide precipitate is treated in at least two fluidized bed furnaces, in which roasting occurs in a bubbling bed, whereby oxidation of the surface of the particles is carried out in the first fluidized bed furnace when the temperature is regulated to the region of 650–750°C and the temperature of
25 the second fluidized bed furnace is regulated to the region of 750–1000°C in order to oxidize the nickel sulphide completely into nickel oxide.

It is typical of the method according to the invention that the particle size of the precipitated nickel sulphide precipitate is approximately 5–20 µm.

According to one preferred embodiment of the invention, the particle
30 size of the micropelletized nickel sulphide precipitate is approximately 0.2–1 mm.

In the method according to one embodiment of the invention, a fuel is used for the control of the fluidized bed furnace temperature if the thermal content produced by the oxidation process is not sufficient.

35 According to one embodiment of the invention, the nickel oxide formed in the fluidized bed furnace is cooled by fluidized bed cooling.

According to one preferred embodiment of the invention, the heat recovered from the exhaust gases of the fluidized bed furnace is used for drying the micropellets that are formed.

It is typical of the method according to the invention that the sulphur dioxide gas that is formed is routed after scrubbing to sulphuric acid fabrication.

LIST OF DRAWINGS

Figure 1 is a flow chart of one method according to the invention, and

Figure 2 is a flow chart of another method according to the invention.

DETAILED DESCRIPTION OF THE INVENTION

When a nickel sulphide ore or concentrate is leached and the nickel-containing solution that is generated is subjected to sulphide precipitation, a very fine-grained nickel sulphide precipitate is produced with a particle size of approximately 5–20 μm . The nickel sulphide content of the nickel sulphide precipitate is approximately 60–70% and it contains, in addition, only a few percent of other metals in sulphide form, such as iron, zinc and cobalt. Since the precipitate in question is formed hydrometallurgically, no gangue is included. Due to its high nickel sulphide content, the melting point of the precipitated material is significantly lower, at approximately 650°C, than a kind of nickel sulphide concentrate in which the majority is pyrrhotite, which has a melting point of approximately 1200°C. Since the nickel sulphide precipitate to be treated is very fine and its nickel sulphide content is quite high, it means that the precipitate has a very large specific surface area. Additionally, in the oxidizing treatment of nickel sulphide-containing material, there is the problem that the oxidation and melting zones of nickel sulphide are very close to each other and there is a danger in fluidized bed oxidation that the sulphide will melt and form large agglomerates, which prevent fluidization of the bed.

As it is generally known, a fluidized bed may be either a Bubbling Fluidized Bed (FB) or a Circulating Fluidized Bed (CFB). One fluidized bed more rarely used is, for example, the Annular Fluidized Bed (AFB).

The method according to the invention is described in more detail by means of the attached figures. In the solution according to Figure 1, the roasting of nickel sulphide takes place on the CFB principle. The particle size

of the fine nickel sulphide precipitate formed by precipitation is approximately 5–20 μm and it is subjected first to micropelletizing 1 in an equipment suitable for the purpose. Sulphated dust generated in the cooling of fluidized bed furnace exhaust gases is used as binder in micropelletizing. Micropelletizing reduces the specific surface area of the sulphidic material that melts at low temperatures, thus minimizing the formation and sintering of harmful large agglomerates. Micropelletizing also minimizes oxidation time, because diffusion takes place right to the core faster than with large pellets. The particle size of the pellets fed into the circulating bed is determined on the basis of the material, the selected process, the temperature used in the process and the required residence time. Preferably the size of the micropellets is controlled to be in the region of 0.2–1 mm.

The dust recovered from the exhaust gases in heat recovery boiler 4 and electric filter 5 is oxidized up to sulphate, because it has travelled onwards with the sulphur-dioxide-containing gas at a temperature at which sulphur dioxide and oxygen form sulphur trioxide and sulphates with the metals. The sulphated dust binds fine sulphide, and the desired particle size of the micropellets can be specified with the amount of dust fed into pelletizing. If the amount of dust is not sufficient, additional nickel sulphate in addition to the dust can be routed to pelletizing in order to bind the sulphide precipitate. Besides the dust, water is fed into the sulphide precipitate to achieve pelletizing. To strengthen the pellets before the fluidized bed furnace, they are subjected to drying. Pellet drying 2 preferably takes place by using steam formed in heat recovery boiler 4.

Circulating fluidized bed roasting 3 of the dried, micropelletized nickel sulphide material takes place in a single fluidized bed furnace. In the flow chart CFB roasting 3 includes both a fluidized bed furnace and a cyclone connected to it. The pellets in the circulating bed are not in as close contact with each other and do thus not adhere to each other as easily as in a bubbling bed. The temperature in the fluidized bed furnace operating on the CFB principle and the connected cyclone, i.e. in the entire circuit, is controlled to remain higher than 700°C all the time, thereby preventing the sulfation of the nickel sulphide. However, in order to prevent the pellets from sintering and forming molten surfaces, the process is run at a maximum of 900°C. At this temperature range nickel oxide is stable.

The flow of the product removed from the fluidized bed furnace and the flow of the material to be recirculated are controlled by the output of the cyclone underflow. The flow of pellets to be recirculated is approximately 100–300% of the amount of fresh micropellets fed into the furnace. The size of the circulating pellet flow is determined by the properties of the feed, such as oxidation, as well as according to how much oxidized inert material is required to prevent sintering of the fresh feed. The tendency to sinter is, in turn, affected by the composition of the nickel sulphide feed and the particle size of the micropellets. Naturally the circulation is minimized, because in this way the energy requirement is minimized. If the oxidation reactions occurring in the fluidized bed furnace are not sufficient to produce the required temperature, additional fuel can be added to the furnace, which may be gaseous, liquid or solid, such as elemental sulphur.

The gas exiting the fluidized bed furnace is routed to the cyclone and from there onwards to heat recovery boiler 4, in which the gas is cooled and some of the dust contained in the gas is recovered. As stated above, the steam generated in the boiler is routed to the micropellet-drying stage. The gas from the heat recovery boiler is routed on to electric filter 5, in which the final removal of dust from the gas takes place. The scrubbed gas flow is routed onwards to sulphuric acid production.

The hot nickel oxide material removed from the fluidized bed furnace is cooled indirectly by means of air or some other oxide-containing gas. Cooling may take place for example as fluidized bed cooling 6. As for the hot oxide-containing gas produced in the cooling stage, it is fed into the fluidized bed furnace as process/fluidizing gas.

The solution according to Figure 2 is similar to the solution presented in Figure 1 in all respects except for the fact that roasting is performed in two fluidized bed furnaces 7 and 8 operating on the bubbling bed principle. In accordance with the invention, the temperature of the first fluidized bed furnace 7 is regulated to be in the range of 650–750°C, whereby generation of molten phases is minimized. Since roasting is performed at such a low temperature, oxidation up to oxide does not occur completely in an economic period of time, so some sulphur remains in the product of the first stage. The amount of sulphur remaining in the calcine is controlled by means of the roasting time and oxygen coefficient. In the first stage the nickel sulphide is oxidized only to the extent that the heat generated in the reactions is sufficient to raise

the temperature of the furnace to the level mentioned and, at the same time, an intermediate is produced with an oxidized surface. The material is simultaneously coarsened.

The intermediate calcined in the first fluidized bed furnace is routed
5 hot to second fluidized bed furnace 8. In this second stage, the temperature of the fluidized bed furnace is regulated to the range of 750–1000°C, because the risk of sintering is significantly decreased due to the coarsening of the material, the lowering of the sulphide level and the oxide layer formed on the particle in the first stage. In the second stage the sulphides are oxidized completely as
10 the oxygen is diffused easily through the porous surface of the pellets formed in the first fluidized bed furnace as far as to the core. If the heat formed when the sulphides are oxidized is not sufficient to raise the temperature adequately, the above-mentioned fuels can be used as an aid. The gas flow of both the first and the second fluidized bed furnaces is routed to heat recovery boiler 4
15 and from there onwards via electric filter 5 to acid production. The dust carried in the gas is routed to the micropelletizing stage as binder. Some or all of the dust from the heat recovery boiler can also be returned to the second fluidized bed furnace. The further processing of the generated nickel oxide material with related cooling is carried out in the same way as described in the context of
20 Figure 1.

EXAMPLES

Example 1

The example relates to CFB roasting.

Precipitated nickel sulphide: Ni 60%, Fe 6%, Zn 3% and S 29%.

25 Micropellets prepared from precipitated nickel sulphide are roasted in a circulating fluidized bed. 2900 Nm³ of air is fed into the reactor per 1000 kg of fresh micropellets. 2700 kg of micropellets are recirculated back to the bed. The temperature is maintained at 850°C for the entire circuit. A calcine is obtained from the cyclone that has a sulphur content of 0.2–0.5%.

30 Example 2

The example relates to two-stage roasting.

Precipitated nickel sulphide: Ni 60%, Fe 6%, Zn 3% and S 29%.

Micropellets prepared from precipitated nickel sulphide are roasted in the first stage in a bubbling fluidized bed. 310 Nm³ of air is fed into the bed

per 1000 kg of micropellets. In this case the temperature of the bed rises to 650°C while part of the sulphides are oxidized. The sulphur level of the material thus drops to a value of approximately 22%. The hot product of the first stage is routed to the second stage, into which 2500 Nm³ of air is fed. The
5 temperature of the second stage is maintained at 800°C by cooling or heating. Dust from the boiler is also returned to this second bubbling bed. A calcine is obtained from the second stage that has a sulphur content of 0.3%.

CLAIMS

1. A method for oxidizing nickel sulphide into oxide in fluidized bed furnace(s), wherein a very fine nickel sulphide precipitate formed by precipitation is treated in the following stages:

5 a) a micropelletizing stage wherein the nickel sulphide precipitate is micropelletized by means of sulphate dust separated from the fluidized bed furnace exhaust gases and

 b) a roasting stage wherein the micropelletized precipitate is treated in one or more fluidized bed furnace(s) at a temperature of 650–1000°C in oxidizing conditions to form nickel oxide and a gas containing sulphur dioxide.

10 2. The method according to claim 1, wherein the roasting of the micropelletized precipitate occurs as circulating fluidized bed roasting in a single fluidized bed furnace and a cyclone connected to it, whereby the temperature of the entire circuit is adjusted to 750–1000°C.

15 3. The method according to any one of the preceding claims, wherein the flow of micropellets to be circulated is at least 100–300% of the amount of fresh micropelletized nickel sulphide precipitate fed into the furnace.

 4. The method according to claim 1 wherein in the roasting stage the micropelletized precipitate is treated in two or more fluidized bed furnaces.

20 5. The method according to claim 1 or 4, wherein the micropelletized nickel sulphide precipitate is treated in at least two fluidized bed furnaces, in which roasting takes place in a bubbling fluidized bed, so that oxidation of the particle surface is performed in the first fluidized bed furnace when the temperature is adjusted to the range of 650–750°C and the temperature of the second fluidized bed furnace is adjusted to the range of 750–1000°C in order to oxidize the nickel sulphide completely into nickel oxide.

 6. The method according to any one of the preceding claims, wherein the particle size of the precipitated nickel sulphide precipitate is approximately 5–20 µm.

30 7. The method according to any one of the preceding claims, wherein the particle size of the micropelletized nickel sulphide precipitate is approximately 0.2–1 mm.

 8. The method according to any one of the preceding claims, wherein a fuel is used to control the temperature of the fluidized bed furnace.

9. The method according to any one of the preceding claims, wherein the nickel oxide formed in the fluidized bed furnace is cooled by fluidized bed cooling.

10. The method according to any one of the preceding claims,
5 wherein the heat recovered from the fluidized bed furnace exhaust gases is used for drying the micropellets that are formed.

11. The method according to any one of the preceding claims, wherein the sulphur dioxide gas that is formed is routed after purification to sulphuric acid production.

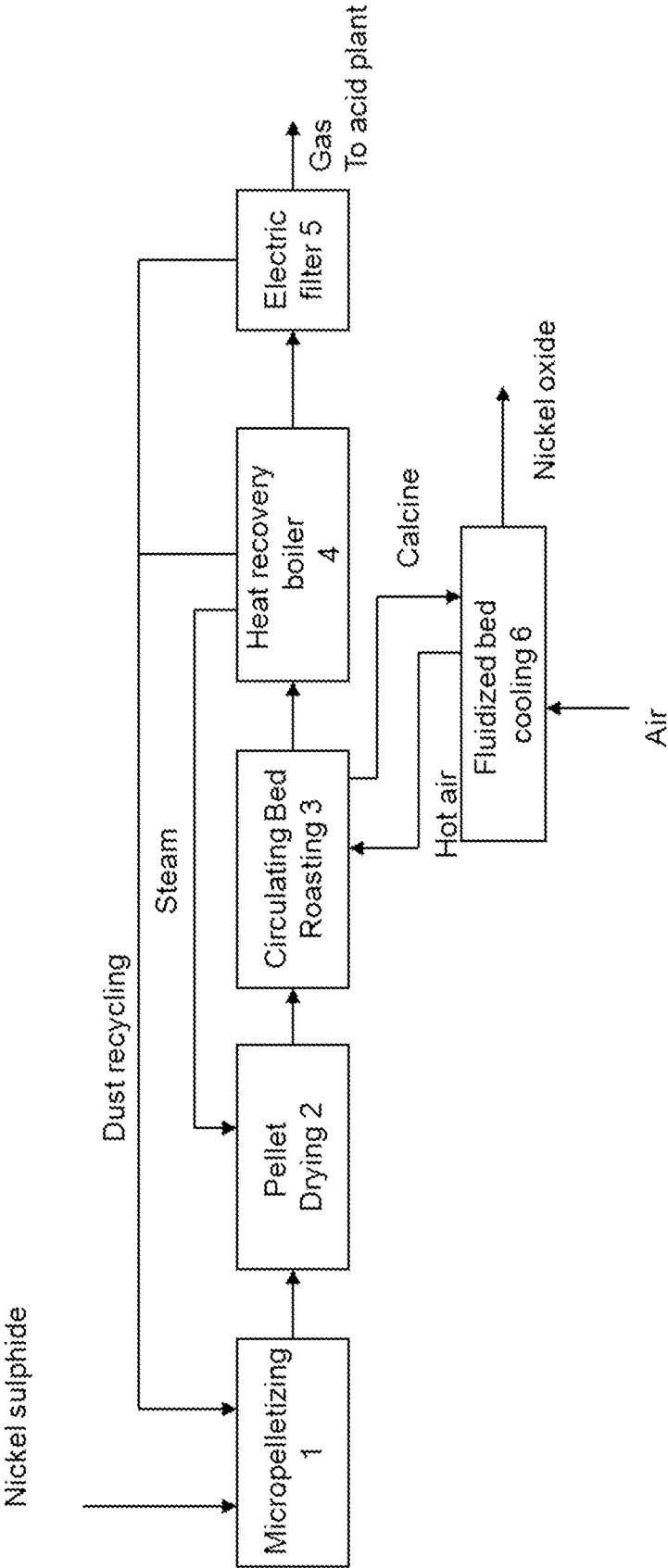


Figure 1

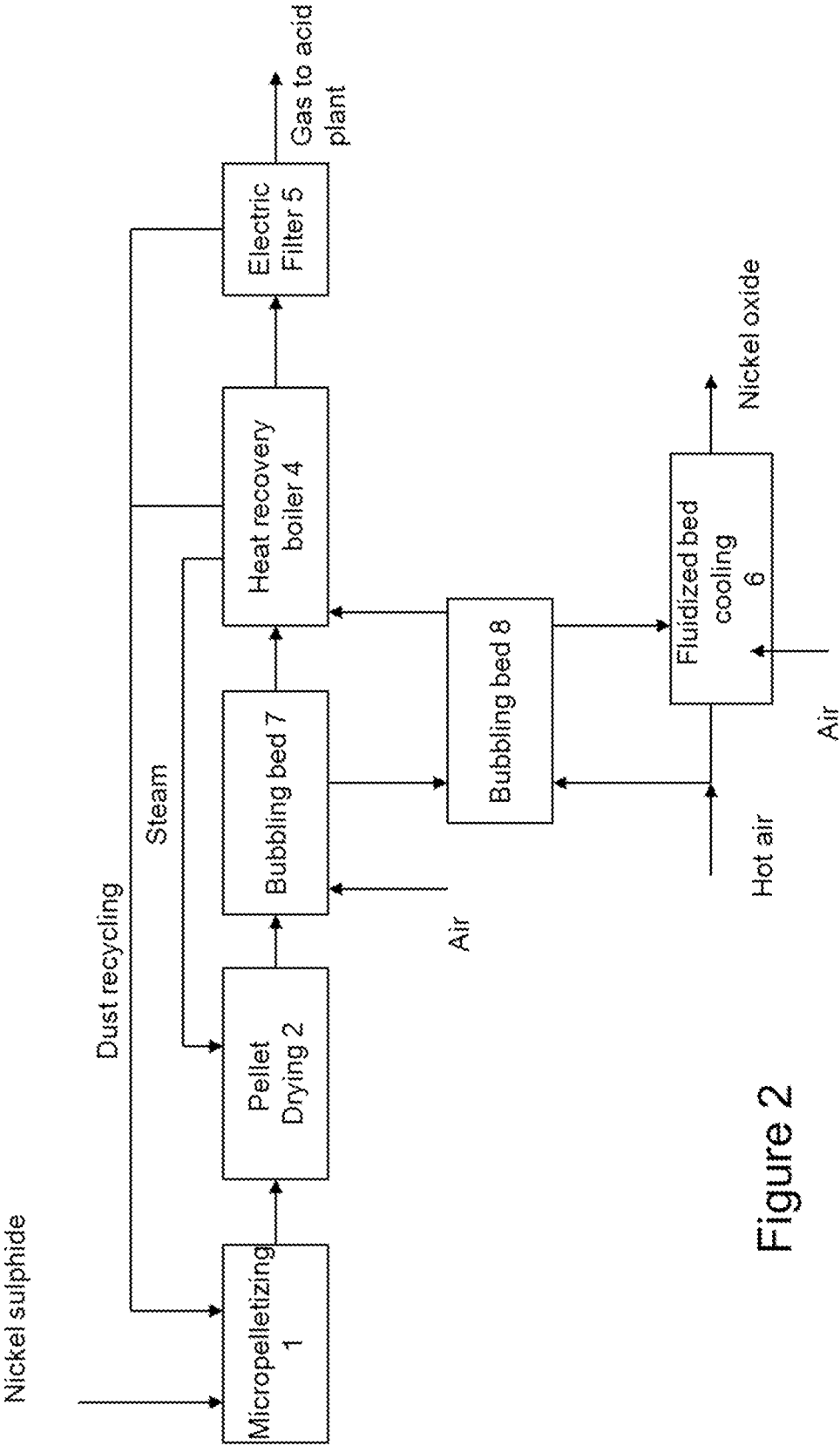


Figure 2

INTERNATIONAL SEARCH REPORT

International application No
PCT/FI2012/050163

A. CLASSIFICATION OF SUBJECT MATTER
INV. C22B1/06 C22B1/10 C22B23/02
ADD.

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)
C22B

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

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 3 094 409 A (SECONDO RENZONI LOUIS ET AL) 18 June 1963 (1963-06-18) column 1, line 11 - line 19 column 2, line 37 - line 59 column 3 column 4, line 43 - line 58 column 5, line 5 - line 13 column 6, line 33 - line 42; figure 1; example 1	1-3,7-9
X	GB 1 502 986 A (INCO LTD [CA]) 8 March 1978 (1978-03-08) page 1 - page 2	1-3,8,9



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents :

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Date of the actual completion of the international search

29 May 2012

Date of mailing of the international search report

06/06/2012

Name and mailing address of the ISA/

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/FI2012/050163

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
US 3094409	A	18-06-1963	NONE	

GB 1502986	A	08-03-1978	AU 7801275 A	12-08-1976
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