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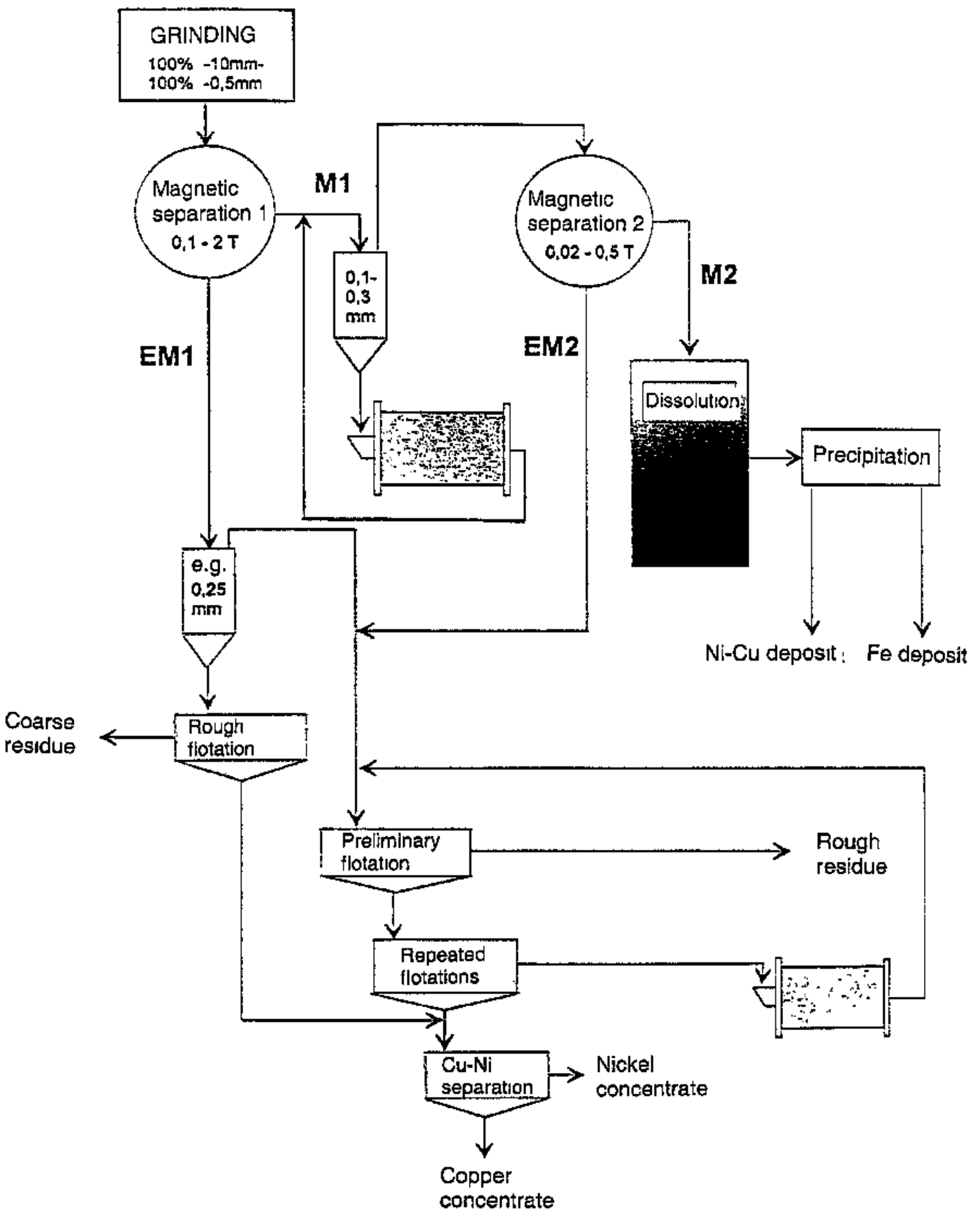
(54) Titre : PROCEDE DE PREPARATION DE CONCENTRE DE NICKEL
(54) Title: METHOD FOR THE PREPARATION OF NICKEL CONCENTRATE

FLOW DIAGRAM OF THE PROCESS OF THE INVENTION



(57) Abrégé/Abstract:

The invention relates to a method for preparing nickel concentrate from pyrrhotitepentlandite ore. The method comprises grinding the ore to the maximum grain size at which the major portion of the sulphide minerals is worked off from the silicate and other refuse minerals and the liberated precious sulphides can be concentrated to high-quality end products directly or by means of additional grinding, and separating the particles containing pyrrhotite magnetically from the ground ore to form a magnetic concentrate (M1). The non-magnetic products (EM1, EM2) obtained as a residue after magnetic separation are delivered to flotation, where the nickel and/or other precious sulphide concentrates are produced.

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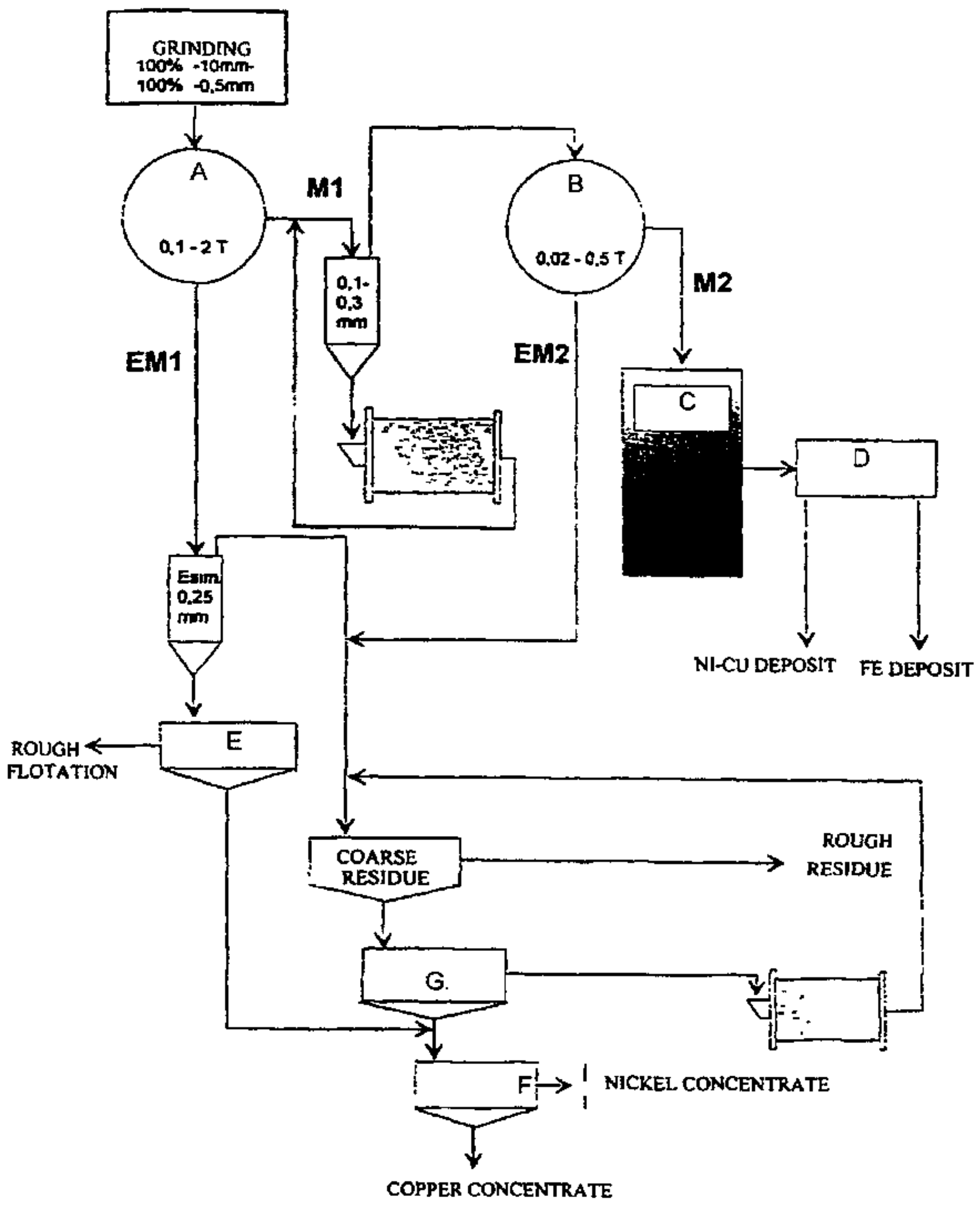
(54) Title: METHOD FOR THE PREPARATION OF NICKEL CONCENTRATE

(57) Abstract: The invention relates to a method for preparing nickel concentrate from pyrrhotitepentlandite ore. The method comprises grinding the ore to the maximum grain size at which the major portion of the sulphide minerals is worked off from the silicate and other refuse minerals and the liberated precious sulphides can be concentrated to high-quality end products directly or by means of additional grinding, and separating the particles containing pyrrhotite magnetically from the ground ore to form a magnetic concentrate (M1). The non-magnetic products (EM1, EM2) obtained as a residue after magnetic separation are delivered to flotation, where the nickel and/or other precious sulphide concentrates are produced.



A... MAGNETIC SEPARATION 1 0,1-2T
B... MAGNETIC SEPARATION 2 0,02-0,5 T
C... DISSOLUTION
D... PRECIPITATION
E... PRELIMINARY FLOTATION
G... REPEATED FLOTATION
F... CU-NI SEPARATION

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Method for the preparation of nickel concentrate

Introduction

The invention relates to mineral concentrating technology and concerns a method for preparing nickel concentrates (= a process for concentrating nickel ores).

The major part of the global nickel production is derived from pyrrhotite-pentlandite-copper pyrite ore of magmatic origin, in which the quantitatively predominating minerals are silicates and pyrrhotite. The amounts of precious sulphides, pentlandite and pyrrhotite, are smaller, accounting only for a few per cent. The following methods have been conventionally implemented in the concentration of these minerals:

- crushing and grinding the material to flotation fineness, the different precious mineral particles being principally discrete grains (with a high degree of liberation at this early stage). This is followed by flotation of the conjugate sulphide concentrate (Ni content in the range of 4 to 6%). Before the nickel flotation, the Cu concentrate has occasionally been separated from the ores that are richest in copper.

- Increasing the Ni content of the concentrate (to a level in the range of 6 to 10%) and decreasing the amount by pressing pyrrhotite into the residue during flotation. The yield losses have prevented quantitative pyrrhotite pressing, because the difference found between pentlandite and pyrrhotite has not been sufficient for selective separation to be successful.

- Performing magnetic separation of pyrrhotite from the products in various process steps (feed material, rough concentrate, concentrate), usually from a material ground to flotation fineness. However, this has only yielded a partial solution, because pyrrhotite with this fineness will not be removed in its totality by magnetic means. The different pyrrhotite phases, monoclinic/ferromagnetic and hexagonal/-paramagnetic, are separated at this degree of fineness. On the other hand, in very fine grain size groups, magnetic separation is not sufficiently selective, and fine precious minerals will be present in the magnetic fraction. The losses of nickel yield have been so great that even magnetic separation has not become a commonly implemented method of producing high-quality nickel concentrates.

General description of the invention

The method for preparing nickel concentrates as defined in claim 1 has now been found. The other claims define a number of aspects of useful application of the invention.

The method may comprise the following steps:

1. Selective gradual comminution of the minerals in different process steps

2. Pyrrhotite separation by magnetic means

3. Rough flotation and removal of coarse fraction

4. Flotation

5. Dissolution of pyrrhotite concentrate and precipitation of precious metals

Compared with conventional nickel concentration, the method provides a preparation concentrate with higher nickel content and higher yield of precious minerals at lower investment and operating costs. The enhanced quality of the concentrate will also have an appreciable economic and ecologic impact on the further refining chain of the concentrates.

Detailed description of the invention

The new method is based on the utilisation of natural selective desintegration of the minerals to be prepared by using old, approved means of concentration, classification, magnetic separation and flotation aiming at high-quality nickel (and copper) concentrates with optimal yields of precious metals. The grinding of the invention and the choice and new combination of concentrating methods are based on the observed occurrence of precious minerals in the ore to be utilised:

- In this particular type of ore, sulphides occur in agglomerations in the interspaces between silicates having usually a notably greater crystal size than these. Pentlandite always occurs and copper pyrite nearly always occurs within or at the side of pyrrhotite.

- The major part of pentlandite (70 to 80%) occurs as idiomorphic crystals (∅ 0.3-20 mm), which are internally splintered (∅ 0.01-0.3 mm). An originally intact pentlandite crystal most frequently is splintered into dozens of fragments in its original position in pyrrhotite. A small portion of pentlandite (5-10%) form small-crystal (under ∅ 0.1 mm) grain sequences on the interfaces between the pyrrhotite crystals and a small portion (5%) occur as filtering flames (under ∅ 0.02 mm) in the pyrrhotite.

- In most ores, pyrrhotite is a mixture of the monoclinic (ferromagnetic) and hexagonal (paramagnetic) phases. The mineral contains an average of 0.3 to 0.4% of nickel (so-called grid nickel) as an iron substitute.

Based on the mineralogical matters presented above and supported by the preliminary indicative test results given below, the following process is proposed for the production of high-quality nickel concentrates:

1. Selective mineral comminution

Fine crushing or coarse grinding

The purpose of comminution is to liberate sulphides from silicates and to grind the precious minerals pentlandite and pyrrhotite to flotation fineness at as early a stage as possible in order to minimise over-grinding. Liberating sulphides from silicates does not require the silicates to be ground under their crystal size. In the tests conducted with exemplifying ore, a degree of fineness of 100% - 4 mm in this comminuting step was enough. The choice of communitor may consist of the most efficient, economical device which performs optimal grinding following the grain limits (coarse, harder silicate/softer sulphide) such that the soft sulphide fraction is crushed (the pentlandite splinters are liberated from pyrrhotite and pyrrhotite is liberated from silicates as far as possible), but the silicate crystals are not necessarily reduced to a notable degree. A significant portion of pentlandite and pyrrhotite is liberated to flotation fineness already in this comminuting step.

Grinding of a highly magnetic product

The magnetic product of high-magnetic separation should contain all the grains containing pyrrhotite even in small amounts. In that case, all the unliberated pentlandite and the major portion of unliberated copper pyrite would end up in this product. The fine and coarse material of the magnetic product is separated into different groups. Depending on the type of ore, the classification limit is in the range ∅ 0.1 to 0.3 mm. The coarse fraction is led to further grinding in order to crush mixed grains and to liberate precious minerals.

Grinding of repeatedly prepared residue in the flotation circuit

Considering the coarseness of the material fed into the flotation circuit, coarse mixed grains which are unfit for use in the final concentrate may naturally cumulate in the repeatedly prepared residue, and these grains can be easily disintegrated with light additional grinding and the product can be returned to a suitable point of the flotation circuit.

2. Magnetic separation

High-magnetic separation

Primary magnetic separation is carried out from fine metal/coarse powder, all the grains containing pyrrhotite being separated to the magnetic product. The separator should have adequate field intensity for each individual case. Lower field intensity will be enough for monoclinic pyrrhotite, whereas hexagonal pyrrhotite requires considerably stronger field intensity in order to separate into the magnetic product. From the magnetic product of high-magnetic separation, a fine, pure pyrrhotite and a coarse pyrrhotite with mixed grains are separated by classification, the latter being further ground.

Low-magnetic separation

The separation is performed with a separator, which separates only pure pyrrhotite into the magnetic product. This yields a product with a nickel content of the usual order of 0.8 to 1.0% (containing the grid nickel in the pyrrhotite and a small amount of pentlandite particles, which are mainly small xenoliths within the pyrrhotite). The nickel yield in the magnetic product is accordingly of the order of 10-15%. The magnetic separation residue (= a non-magnetic product containing silicates, pentlandite and pyrrhotite) has a small volume and contains precious sulphides in a significant amount. It is conducted to the flotation circuit along with the primary separation residue.

3. Rough flotation and removal of the coarse silicate fraction

The high-magnetic separation residue (the major portion of the feed material) is classified using as a classification limit the maximum grain size in which precious sulphides are still quantitatively flotated (e.g. ∅ 0.25 mm) in a conventional flotation +process). From the fraction below classification, precious sulphides are flotated with rough-flotation techniques and the concentrate is fed into the suitable process step of the actual flotation circuit according to the product quality. The flotation residue is a coarse silicate material, which is either taken to a dump or reclaimed.

4. Flotation

In flotation, precious sulphides are separated from silicates as a separate concentrate (Ni-Cu conjugate concentrate) or separate concentrates (Ni and Cu concentrates) in normal sulphide flotation conditions. The flotation aims at separating pentlandite and copper pyrite from silicates and any other sulphides present, such as hexagonal pyrrhotite and pyrite. The repeated preparation of rough concentrate focuses on silicate removal using conventional techniques. The coarse end of repeatedly prepared residues can be returned to intermediate grinding and from there to flotation, where the mixed grains would produce a cumulative circulating load on the repetitive circuit and would also entail losses of yield if no intermediate grinding were performed.

5. Dissolution

Pyrrhotite removal causes yield losses of approx. 10 to 20% regarding nickel. The pyrrhotite fraction usually has a nickel content of 0.8 to 1.5%, preferably 0.8 to 1.0%. If desired, the nickel contained in this product can be further recovered by dissolution, for instance atmospheric oxygen dissolution, oxygenating pressure dissolution or bacterial dissolution methods. Precious minerals are precipitated from the solution with a suitable method, resulting in a deposit (or deposits in the case of selective precipitation), which can be further refined jointly with the concentrates, for instance.

Benefits gained by the method

Estimated on the results of preliminary batch tests on laboratory scale, the method yields a 20% nickel content in the concentrate, the Ni yield being at the level of 70 to 75%. In addition, the nickel contained in the pyrrhotite can be utilised by dissolution, so that the overall yield loss will be of the order of 10 to 15% or less. The concentrate amounts are notably smaller than those produced with conventional methods.

The process control is simpler than at conventional nickel concentrating plants, because the mass flows in the flotation circuit are notably smaller owing to the pyrrhotite and silicate removal. The products to be further ground have a small mass and relatively homogenous quality, thus allowing better process control.

This method also allows the elimination of the typical problem of many nickel ores, which is caused by fine grinding in the presence of silicates, given that the method of the invention removes a major portion of the silicates from the process in a notably coarser form than in conventional processes. The conventional simultaneous fine grinding of the total material produces over ground (colloidal) mineral material (sludge) which has a negative effect on the flotation and the quality of the products, and also calls for a more complex flotation circuit and increased chemical consumption (i.e. higher production costs).

The investment and operating costs of concentrating plants will diminish with the method of the invention i.a. for the following reasons:

- Minimised comminuting apparatus and use of grinding energy,

- Smaller-sized apparatus and reduced use of the flotation circuit compared to conventional flotation circuits

- A decrease in the concentrate amount, resulting in reduced processing costs (filtering, drying etc.).

The higher concentrate quality and smaller concentrate amount has a significant bearing on the further refining chain:

- Lower freight charges

- Lower smelting plant costs

- Smaller amount of sulphur to be recovered from flue gases

The enclosed figure is an exemplifying flow diagram of a process in accordance with the invention.

Exemplifying tests conducted with the method of the invention

An ore sample was crushed with a jaw crusher and was further comminuted with a roll crusher during gradual screening to a grain size of -1.4 mm. No screen analysis was performed. The screen analysis of the products of the further process allows the conclusion that approx. 60% of the material was under 0.25 mm (approx. 90% of pentlandite and approx. 85% of copper pyrite). The - 0.032 mm fraction accounted for 20% of the total amount of crushed product.

The pyrrhotite was removed with a drum separator equipped with high-magnetic neorem magnets (magnet field intensity of approx. 0.1 T in the separation duct and of 0.3 T on the drum surface) in wet separation. The removal of pyrrhotite was almost totally successful. The calculatory loss to the non-magnetic product was 2.9%.

Using classification (0.125 mm mesh) of the magnetic product of high-magnetic separation, the coarse grains containing pyrrhotite were separated for further grinding from the fine pyrrhotite, which was already free from precious minerals (with the exception of pentlandite filtering products). The purpose of further grinding of the coarse product was to liberate the pentlandite and copper pyrite grains accompanying the pyrrhotite particles. After this magnetic repeated preparation was performed with the ground product using a SALA low-magnetic wet separator.

The fine pyrrhotite, which was almost free of precious minerals, was removed as a separate product (pyrrhotite concentrate). The apparatus was a SALA low-magnetic wet drum separator. The test was conducted by subjecting the magnetic product to an additional iterative separation (purification) with the same separator.

The pyrrhotite concentrate from the preceding test had

- A pyrrhotite content of 99%

- A nickel content of 0.88%

- A pyrrhotite yield of 91%

- A nickel yield of 14%

- A copper yield of 7%

- A yield of granular pentlandite of 3%

Flotation was performed with the combined non-magnetic products of the magnetic separation. During the flotation, pentlandite and pyrrhotite were concentrated while the silicates were left in the residue (partly even in quite a coarse form). The flotation residue also comprised the pyrrhotite (of which a small amount was hexagonal and had been subjected to magnetic separation) and pyrite.

Preliminary flotation, in which the pH conditions were controlled with sulphuric acid (H_2SO_4) to a value of 6.5 of the natural ore value (9.0). The acid consumption was 0.57 kg/t of material feed. 300 g/t of NaIBX (sodium isobutyl xanthate) and 60 g/t of frothing agent (Dow froth 250) were added to the preliminary flotation. The result was:

- A concentrate with a nickel content of 8.1% and a copper content of 2.4%

- A sulphide content of 41%

- Yields: 78.2% of nickel, 82.9% of copper, 88% of pentlandite

- Nickel losses in the residue: +0.25 mm 4.1%, 0.032-0.25 mm 1.5%-0. 032 mm 2.4%

The rough concentrate was subjected to two iterative preparations, with additions of 100 g/t of CMC and 50 g/t of NaIBX to the first one. In this iterative flotation, the pH range was 8.3 to 8.2. 170 g/t of soda (Na_2CO_3) was added to the second iterative flotation, whereby the pH value of the sludge rose to 10.0. In addition, 50 g/t of CMC and 75 g/t of NaIBX were batched in this step. In both the iterative preparations, the preparation periods were 5 minutes for CMC and 2 minutes for NaIBX. Thus a concentrate that had been prepared twice was obtained, whose main properties were:

- A concentrate with a nickel content of 14.5% and a copper content of 4.3%

- A sulphide content of 74%

- Yields: 72.9% of nickel, 77.5% of copper, 82.1% of pentlandite

The enclosed table shows the test analyses, mineral contents and yields.

An ulterior test achieved a Ni content of 20.3% of the concentrate with 1% Ni ore and one single iterative flotation. This test differed from the first test mainly in that the flotation feed material was rich in Ni, with a double Ni content compared to the preceding one (1.48% of Ni/0.65% of Ni). The content ratio of the original ore samples was accordingly 1.0% of Ni/0.67% of Ni. A further difference was the pH value of the rough flotation, i.e. in the range from 9.0 to 8.4 in the latter test; the pH regulating agent was $Ca(OH)_2$ and the sulphide collector used in the preliminary flotation was potassium butyl xanthate KBX (100 g/t), instead of NaIBX as in the preceding test. The collecting chemicals are practically the same, so that the different results were chiefly brought about by the slightly richer ore and the more advantageous flotating conditions (the other sulphides were not flotated in the concentrate due to the higher pH value and the smaller collecting chemical batching). Thus the better result mentioned above was achieved.

Analyses, mineral contents, yields

Product	Screen class	Weight	Analyses			Mineral contents					Yields					Yields in the circuit		
	mm	%	Ni	Cu	S	NIP*	CUK	FEK	SK	OTHERS	Ni	NIP	CUK	FEK	OTHERS	NIP	CUK	FEK
Feed material	Bulk	100.0	0.635	0.177	5.12	1.64	0.51	10.8	0.1	87.0	100.0	100.0	100.0	100.0	100.0			
MR	Bulk	9.99	0.878	0.119	39.90	0.54	0.34	99.0		0.1	13.8	3.3	6.7	91.3	0.0			
ES	Bulk	90.02	0.608	0.184	1.26	1.77	0.53	1.0	0.2	96.7	86.2	96.8	93.3	8.0	100.1			
ER	Bulk	6.11	8.125	2.377	15.44	23.68	6.86	10.3	2.1	59.1	78.2	88.0	82.0	5.8				
Rough flot. residue	0.500-1.000	18.81	0.070	0.050	0.38	0.20	0.14	0.5	0.1	99.0	2.1	2.2	5.3	0.9	21.4			
	0.250-0.500	18.35	0.070	0.030	0.22	0.20	0.09	0.2	0.0	99.4	2.0	2.2	3.1	0.4	21.0			
	0.125-0.250	10.98	0.030	0.010	0.10	0.09	0.03	0.1	0.0	99.7	0.5	0.6	0.6	0.1	12.6	90.9	87.9	72.6
	0.063-0.125	11.13	0.030	0.009	0.09	0.09	0.03	0.1	0.0	99.8	0.5	0.6	0.6	0.1	12.8			
	0.032-0.063	9.67	0.030	0.010	0.09	0.09	0.03	0.1	0.0	99.8	0.5	0.5	0.5	0.1	11.1			
	-0.032	14.97	0.101	0.014	0.34	0.29	0.04	0.5	0.1	99.1	2.4	2.6	1.2	0.6	17.1			
	Bulk	83.91	0.060	0.024	0.229	0.17	0.07	0.4	0.0	99.4	8.0	8.7	11.3	2.9	95.8			
KR1	Bulk	3.88	12.45	3.66	23.27	36.30	10.56	15.0	3.0	35.1	76.1	85.7	80.1	5.4	1.6	97.3	97.7	92.3
KJ1	+0.125	0.41	1.300	0.286	3.26	3.75	0.83	3.4	0.7	91.3	0.8	0.9	0.7	0.1	0.4			
KJ1	-0.125	1.82	0.451	0.120	1.50	1.29	0.35	1.9	0.4	96.1	1.3	1.4	1.2	0.3	2.0			
	Bulk	2.23	0.607	0.151	1.82	1.74	0.43	2.2	0.4	95.2	2.1	2.4	1.9	0.4	2.4			
KR2	+0.125	0.90	11.800	3.000	21.40	34.42	8.66	13.8	2.8	40.3	16.7	18.8	15.2	1.2	0.4			
	-0.125	2.30	15.500	4.800	28.60	45.23	13.86	17.4	3.5	20.0	56.2	63.3	62.3	3.7	0.5			
	Bulk	3.20	14.46	4.29	26.58	42.19	12.40	16.4	3.3	25.7	72.9	82.1	77.5	4.9	0.9	95.9	96.8	90.3
KJ2	Bulk	0.68	2.970	0.659	7.69	8.56	1.90	8.3	1.7	79.6	3.2	3.5	2.5	0.5	0.6			

NIP*= granular pentlandite Pyrrhotite Ni=0.7 % contains grid nickel – 0.4 % - and the nickel contained in the estimated pentlandite filtrates

Claims

1. A method for preparing a nickel concentrate from pyrrhotite-pentlandite ore, characterised in that the following steps are performed:

- comminuting the ore to the maximum grain size at which the major portion of the sulphide minerals is liberated from silicate and other refuse minerals and the liberated precious sulphides can be concentrated to high-quality end products directly or by means of additional grinding, the maximum grain size being preferably in the range 100% - 10 mm...100% - 0.5 mm,

- separating by magnetic means from the comminuted ore the particles containing pyrrhotite as a magnetic concentrate (M1), the resulting residue being a non-magnetic product (EM1),

- if desired, additional grinding of the magnetic concentrate (M1) and separating the pyrrhotite from the refuse minerals and precious sulphides by means of additional separation,

- delivering the non-magnetic products (EM1, EM2) obtained as a residue after the magnetic separation to flotation, where nickel and/or other precious sulphide concentrates are produced.

2. A method as defined in claim 1, in which coarse material is removed from the non-magnetic products (EM1, EM2) before flotation.

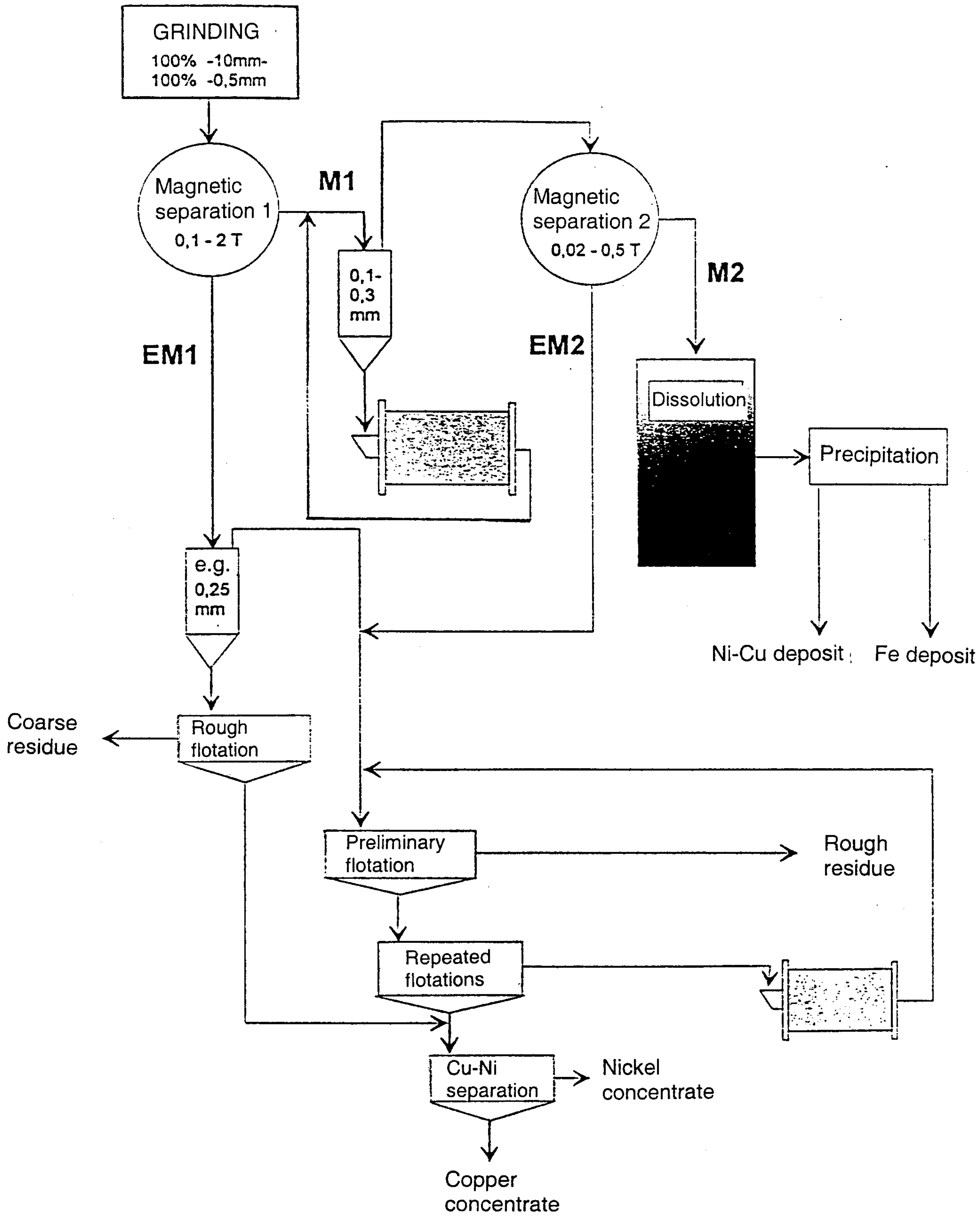
3. A method as defined in claim 1 or 2, in which the magnetic concentrate (M1) is additionally ground, and during the additional grinding fine material such as material under 0.1 to 0.2 mm is removed.

4. A method as defined in any of claims 1 to 3, in which rough concentration is first carried out during the flotation by means of rough flotation techniques.

5. A method as defined in any of claims 1 to 4, in which the flotation is carried out repeatedly and in which the coarse end of the repeatedly prepared refuse is returned to intermediate grinding.

6. A method as defined in any of claims 1 to 5, in which nickel is recovered from the magnetic fine crushed product by means of dissolution.

FLOW DIAGRAM OF THE PROCESS OF THE INVENTION



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