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RECOVERY OF NICKEL AND COBALT FROM MIXTURES OF THE SAME WITH IRON SULFIDE, AS IMPURITY

Raphael F. Matson, New Orleans, La., assignor to Freeport Sulphur Company, New York, N.Y., a corporation of Delaware

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This invention relates to the recovery of the nickel and cobalt content, and incidentally also any other valuable non-ferrous metal content, from mixtures of sulfides of such metals with iron sulfides, or with both iron sulfides and aluminum oxide or aluminum basic sulfate. More particularly, it relates to the treatment of crude nickel-cobalt sulfide concentrates obtained in a separation step following acid leaching of ferruginous, lateritic, nickel ores, for the purification of the valuable metal content.

The crude nickel-cobalt concentrates are obtainable, for example, from the types of ore and by the leaching processes set forth in the copending application of Courtney S. Simons et al., Serial No. 605,240, filed August 10, 1956. There, nickeliferous lateritic limonitic ores are described as being leached with sulfuric acid whereby a leach liquor is produced containing the sulfates of nickel, cobalt, copper and zinc with smaller amounts of iron, aluminum, manganese, chromium and magnesium, which leach liquor is subjected to sulfidation to precipitate the nickel, cobalt, copper and zinc in the form of sulfides, leaving most of the iron but not all of it and the other metals in the residual solution, the precipitation process utilized being described in more detail in U.S. Patents No. 2,722,480 and No. 2,726,953.

It has heretofore been suggested that the nickel and cobalt content of these precipitates or concentrates be purified for the removal of iron by selectively redissolving the nickel-cobalt content. This separation is accomplished by introducing air or oxygen into an acidified aqueous slurry of the concentrate under pressure. See U.S. Patents No. 2,588,265 and No. 2,718,455. In these prior processes, the oxidation is said to convert the nickel and cobalt into soluble sulfates and at least a portion if not all of the iron into an insoluble basic iron sulfate which is thereupon filtered out.

The separation of the iron content from the nickel-cobalt content by these prior procedures is not complete. The precipitation of the iron carried with it a prohibitive proportion of the nickel and cobalt values which necessitates additional treatment for the recovery of these values from the iron residue. This treatment or recovery operation is difficult to acomplish because of the presence of the iron in oxidized form with the nickel and cobalt sulfides to be dissolved. Furthermore the sulfate solution obtained in these prior oxidation processes retains a prohibitive amount of iron dissolved therein, and this iron must be removed in an additional purification step.

In these prior oxidation processes, attempts are apparently made to dissolve all of the nickel and cobalt values from the concentrates through the action of the oxygen. Now it has been found that when this oxidation is carried to completion or to a point of maximum dissolution of nickel and cobalt, ferric oxide and basic iron sulfate begin to precipitate before all of the nickel and cobalt values have been converted from the sulfide form to the sulfate form, and the presence of this ferric oxide and basic iron sulfate precipitate prevents solubilization of a part of the nickel and cobalt sulfide values. Recovery of these values is extremely difficult if this precipitated residue is combined with iron precipitated and removed from the sulfate solution in a subsequent purification step.

The primary object of the invention is to simplify and reduce the number of steps required for purifying iron-containing nickel-cobalt sulfide concentrates.

A more immediate object of the invention is to provide a process for sulfating and dissolving the nickel and cobalt values from the above-described mixed sulfide concentrates without oxidizing the iron and precipitating the same in the form of the oxide or the basic sulfate, in which process all of the sulfide content of the concentrate ultimately goes into solution in the form of sulfates.

Another object is to separate the cobalt and nickel values from the iron contained in said concentrates by a process which avoids the necessity of two treatments heretofore applied (one to the impure nickel-cobalt solution and the other to the precipitated iron residue).

Broadly defined, the instant invention may be considered to involve an improvement in the treatment of mixtures of non-ferrous metal sulfides with ferrous sulfide impurity for the purpose of recovering the non-ferrous metals in purer form wherein oxygen is introduced into an acidic aqueous suspension of the sulfide mixture while the suspension is maintained at an elevated temperature under pressure above the vapor pressure of the solution until substantial but incomplete sulfation and dissolution of the metal content including the iron content has occurred, this sulfation operation being stopped before any of the iron content has precipitated, after which the product liquor containing the dissolved metal sulfates derived from most of the nickel, cobalt and iron is separated from the undissolved metal sulfide content, and treated for the precipitation of the iron in accordance with known procedures.

The prevention of the precipitation of the iron is accomplished by maintaining a predetermined, adequate amount of sulfide in the aqueous suspension of the metal sulfides during the entire oxidation treatment. The presence of the free sulfides apparently prevents oxidizing conditions from arising in the suspension which are sufficiently active to cause oxidation of the iron to a form which precipitates either as the basic iron sulfate or the oxide form, under the temperature and other conditions maintained for dissolving the sulfides. The maintenance of adequate sulfides in the aqueous suspension is suitably accomplished by timely discontinuance of the oxidizing treatment or withdrawing the product liquor containing the sulfates from the oxidizing reactor before too much of the sulfide has been dissolved and adding fresh sulfide slurry to the reactor in amounts equivalent to the water and sulfates withdrawn.

In a preferred embodiment of the invention, the process is operated continuously by filtering the oxidized slurry withdrawn from the reactor to separate the undissolved sulfide solids and recycling these acid-soluble solids to the reactor to which fresh sulfide concentrate is added with additional dilute sulfuric acid, the oxygen or oxygen-containing gas, such as air, being continuously introduced. When the oxidation process for dissolving the sulfide concentrate is carried out in this manner, none of the iron is oxidized or precipitated and all of the sulfided metal including the iron contained in the concentrate treated ultimately goes into solution in the sulfate form.

To maintain the required oxidizing conditions in the reactor the minimum recycle rate of undissolved sulfide solids is about 10% of the fresh input sulfide rate. Any lower percentage involves danger of iron oxidation and