

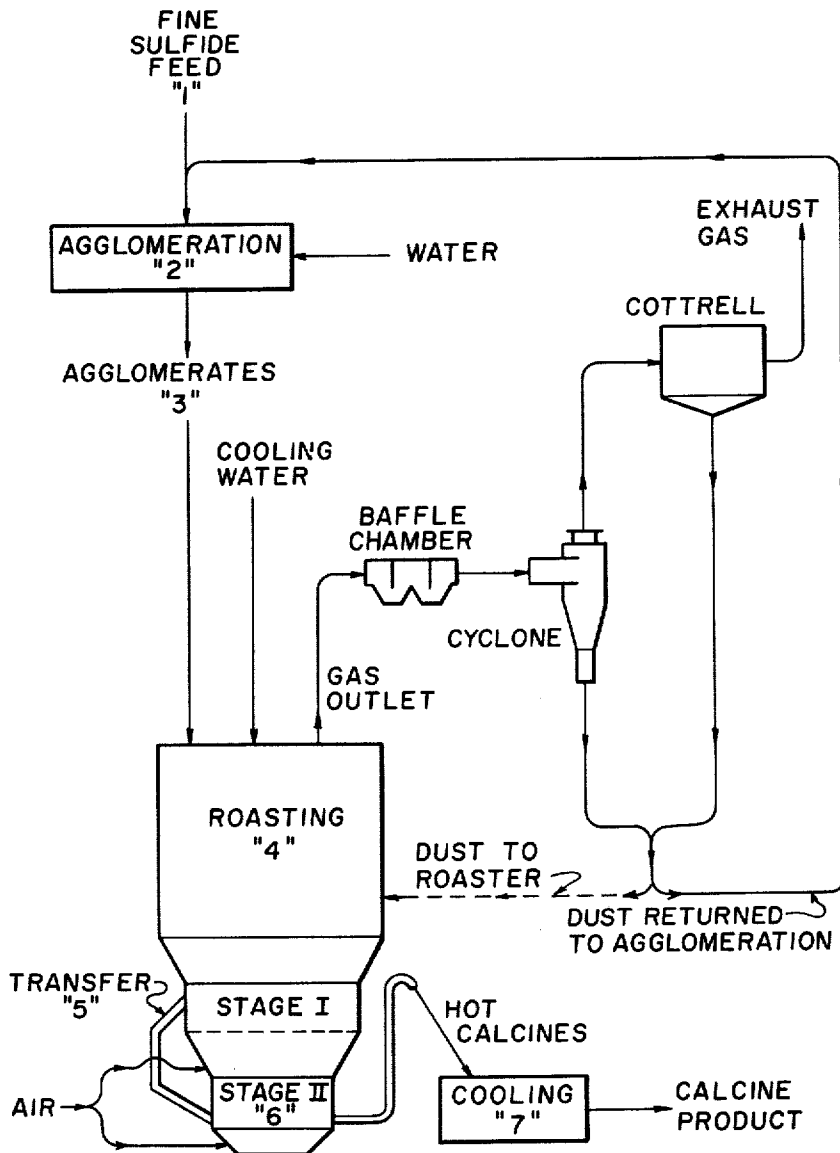
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METHOD FOR ROASTING SULFIDES

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LOUIS SECONDO RENZONI
WALTER CURLOOK
IAN WILLIAM LAING
INVENTORS

BY *Q. Dr. Keller*

ATTORNEY

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METHOD FOR ROASTING SULFIDES

Louis Secondo Renzoni, Copper Cliff, Ontario, Walter Curlook, Port Colborne, Ontario, and Ian William Laing, Lively, Ontario, Canada, assignors to The International Nickel Company, Inc., New York, N.Y., a corporation of Delaware

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The present invention relates to an improved method for roasting metal sulfides and more particularly the present invention provides an improved method for roasting sulfide materials to a low sulfur calcine at temperatures in excess of the softening point of the initial sulfide material while substantially avoiding fusion between sulfide or calcine particles while producing a granular, dust-free oxide product.

It is well known that metal sulfides such as mattes and other sulfide materials containing nickel and varying amounts of copper, iron and cobalt have relatively low melting points. It is also known that roasting of such sulfides at roasting temperatures below the softening temperature range thereof can be achieved only at very slow rates and it is frequently extremely difficult to reduce the sulfur in the calcine to less than about 2%. Because of the low melting points of nickel sulfides it is much more difficult to roast nickel-containing metal sulfides to a residual sulfur content of less than about 2% than it is to roast metal sulfides of higher melting points to a similar low sulfur content. To achieve the desired low sulfur content in the calcine at attractive rates it becomes necessary to carry out the roasting operation at a temperature above that at which the aforementioned sulfide materials begin to soften. Metal sulfides may be roasted by a number of established practices, e.g., hearth roasting, sintering, flash roasting and fluid bed techniques. However, in commercial operations of the prior art great care must be exercised to avoid fusion of the charge during roasting. To this end hearth roasting is conducted under conditions which permit the temperature to rise slowly from well below the softening point of the sulfide to a high final temperature to permit significant oxidation prior to passing through the critical temperature zone at which fusion of the charge might occur.

Sinter machines have been widely used for carrying out the roasting and agglomeration of the aforementioned metal sulfides. However, when sinter machines are used for this purpose it is a necessary prerequisite that the sinter charge carry a minimum quantity of crushed sinter equivalent to several times the quantity of sulfide feed to avoid serious bed fusion. Sintering has other disadvantages such as the excessively high temperatures involved, high capital and maintenance costs and, in addition, sintering produces a relatively dense product which is insufficiently active for certain applications. Flash roasting requires a finely-divided feed material and results in a finely-divided calcine. Other disadvantages of flash roasting stem from the difficulty in maintaining adequate control and in the large roaster volume required per unit of throughput.

Known advantages of fluid bed roasting are high capacity, ease of accurate control of operating conditions and mechanical simplicity. However, fluid bed roasting of finely-divided sulfides normally requires the use of a large diameter roaster to provide a space velocity sufficiently low to avoid excessive carryover of fines from the bed. In addition, at low fluidizing velocity it has been found that fusion or agglomeration of the material in the fluid bed is prevalent, particularly in the case of low melting sulfides such as nickel sulfides. Apart from operat-

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ing disadvantages such as defluidization and blockage of air inlets, the oxidation of partly fused agglomerates is incomplete resulting in an increase in calcine sulfur content beyond desired levels.

Although attempts were made to overcome these and other difficulties, none, as far as we are aware, was entirely successful when carried into practice commercially on an industrial scale.

It has now been discovered that metal sulfides having relatively low melting points, such as mattes and other sulfide materials containing varying amounts of nickel, copper, cobalt and iron, can be successfully roasted by means of the fluid bed technique at high throughput rates to a granular product of desired low sulfur content while producing gases rich in sulfur dioxide.

It is an object of the present invention to provide a novel method for the substantially complete roasting of metal sulfides at temperatures above their melting points.

Another object of the invention is to provide a method for the fluid bed roasting of low temperature melting metal sulfides which achieves substantially complete removal of sulfur at high throughput rates.

The invention also contemplates providing a novel method for the fluid bed roasting of finely-divided, low melting sulfide materials to produce a coarser dust-free, granular calcine of low sulfur content.

It is a further object of the invention to provide a novel process for the granulation of molten mattes containing varying amounts of nickel, copper, cobalt and iron and subsequently fluid bed roasting the solidified, granulated mattes at temperatures above their softening points to produce a granular, dust-free, low sulfur calcine.

The invention further contemplates providing a unique method of producing from nickel sulfide materials a granular, substantially dust-free, low sulfur calcine of exceptional reducibility at relatively low temperatures.

Generally speaking, the present invention contemplates roasting granules, which may be formed by agglomeration or granulation, of metal sulfides having relatively low melting points such as mattes and other sulfide materials containing varying amounts of nickel, copper, cobalt and iron, but no other metals which form low melting sulfides except in trace amounts, in a fluid bed reactor using an oxidizing medium which may be air, oxygen-enriched air or pure oxygen at fluidizing rates sufficiently high to elutriate fines from the bed while maintaining the coarser agglomerates or granules in a fluidized condition.

The essential concept involved in the present invention which requires that the sulfide material, characterized by relatively low softening temperature, fed to the roaster be in the form of granules, i.e., as pelletized finely-divided sulfides or as granulated sulfides, enables the successful roasting thereof in the fluid bed reactor; whereas feeding of such sulfides directly to the roaster in the finely divided form resulted in melting of the sulfide particles at the high roasting temperatures employed, i.e., temperatures above the softening temperature thereof, with concomitant mechanical difficulties, incomplete removal of sulfur, and commercial impracticability for the process.

The roasting operation is conducted at a temperature sufficiently above the softening point of the initial sulfide material to insure rapid and substantially complete oxidation to less than about 2% total residual sulfur and, if desired, to less than about 0.2% total residual sulfur. The hot calcine may be transferred to a second stage in the fluid bed reactor for activation or cooling purposes. The hot calcine product discharged from the roaster may be cooled by known means.

Fine material entrained in the roaster gases, particularly when treating agglomerated, fine material, is only partially roasted and is in a semi-molten condition. It

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is therefore essential that such gases and the particles entrained therein be cooled below the softening point of the solids prior to or during gas-solids separation. The collected fines may be agglomerated and returned to the roaster or diverted to separate treatment. Alternatively, where the presence of dust in the calcine product is not objectionable, a portion of the collected fines may be returned directly to the roaster for retreatment. The substantially dust-free exhaust gases may be treated for recovery of their sulfur content.

In carrying the invention into practice, sufficiently coarse granular material, such as granulated matte produced by breaking up and solidifying the molten matte into small granules, may be fed directly to the fluid bed roaster. Mattes treated according to this invention may contain iron, which may be a substantial proportion of the matte, and in addition may contain one or more elements from the group consisting of nickel, cobalt and copper. The balance of these mattes is usually substantially all sulfur, i.e., usually between about 15% and about 30% sulfur. Generally, besides containing iron, these sulfide mattes will contain more than about 5% of nickel and/or cobalt and/or copper. However, fine sulfides such as finely ground mattes of the aforementioned contents or those obtained as flotation concentrates, e.g., nickel sulfide concentrates from the flotation of ores or relatively pure nickel sulfide concentrates containing about 64% to about 72% nickel plus cobalt, up to about 8% copper, up to about 2% iron and the balance essentially all sulfur, i.e., about 23% to about 25% sulfur, which may contain over 90% by weight of material passing through a 200 mesh Tyler screen, must be formed into aggregates by either melting and granulation or by other agglomeration means before being fed to the fluidized roasting operation.

Agglomeration of such fine concentrates must be conducted with great care to provide a suitable feed material for the roasting operation. Improperly formed agglomerates tend to disintegrate prior to or during the roasting operation to produce excessive amounts of dust. In addition, improperly formed agglomerates may impair the strength of the final calcine granules and may interfere with the proper removal of sulfur therefrom during the roasting operation.

Referring in detail to the flow diagram, fine sulfide feed "1" is formed into agglomerates with a particle diameter of between not less than about 65 Tyler mesh and up to about 0.5 inch. The agglomeration operation "2" can be performed by any of the well known operations, such as balling on discs or in drums, extrusion and pressing. An important factor in the formation of suitable agglomerates is the adjustment of the admixed liquid. This liquid may be water which may be partly replaced by fuel oil if additional heat is required for roasting. Surprisingly, such addition of fuel oil does not impair pellet quality, does not interfere with roasting operation and avoids difficulties which may arise from separate addition of oil to the reactor. It has been further shown to be advantageous to use binding agents such as nickel sulfate, sulfuric acid and lignosol. Additional agents such as bentonite clay improve the strength of the agglomerates but may be undesirable from the standpoint of product contamination. The optimum moisture content for agglomeration will vary with the particle size distribution of the starting material and size of agglomerates desired, generally being higher in the case of finer materials, but in any case should be controlled within narrow limits, advantageously within one half of one percent. For example, nickel sulfide flotation concentrate with a particle size of 95% minus 200 mesh requires a water content of between about 7½% and about 8½%.

The aforementioned binding agents such as nickel sulfate, sulfuric acid and lignosol may be added in amounts of up to about 2% of the fresh sulfide feed to increase the strength of the green agglomerates. Additions of these

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binding agents in higher quantities generally does not warrant the additional expense.

It has been further discovered that drying of green agglomerates prior to their introduction into the roaster materially improves their resistance to attrition during the roasting operation, e.g., the crushing strength of ⅛ inch diameter green nickel sulfide pellets increased from less than one pound to over twenty pounds by drying at 1000° F. for fifteen minutes. The minimum temperature at which the benefit of drying becomes noticeable depends upon the composition of the sulfide material and the type of binder. For example, drying of nickel sulfide material containing 1% nickel sulfate, by weight, at 800° F., 900° F., and 1000° F. for fifteen minutes in each case increased the crushing strength of ⅛ inch pellets from less than one pound to 3.5, 11 and 22 pounds, respectively.

Agglomerates "3," shown on the flow diagram, are introduced into the roasting operation "4" in the fluid bed roaster through a suitable gas sealing feeder. In treating granulated mattes, the granules, which are of a substantially uniform, coarse particle size, are introduced directly into the roasting operation "4," by-passing agglomeration step "2."

A suitable oxidizing medium, such as air, oxygen-enriched air, oxygen or other oxygen-bearing gas is blown into the reactor at a fluidizing rate which is sufficiently high to elutriate fines from the bed while maintaining the coarser agglomerates or granules in a fluidized condition. The gas velocities which must be used to maintain fluidization vary with the size of the agglomerates or granulated material fed to the bed. It has been found that gas velocities of between about 2 feet per second to about 7 feet per second are usually used to fluidize the granules. However, velocities of up to about 13 feet per second have been used with material formed into coarse granules. Advantageously, the velocity should be above about 3 feet per second. The fluid bed roasting is conducted at a temperature above the softening point of the sulfide material and below the softening point of the calcine product to obtain a roasted material with a total residual sulfur content of less than about 2%. The roasting temperature is advantageously 200° F. above the softening point of the sulfide material. Sulfide materials containing nickel, cobalt and copper and minor amounts of iron commence softening at between about 1000° F. and about 1100° F. while sulfides containing major amounts of iron will generally soften at between about 1500° F. and 1600° F. Higher roasting temperatures within this range tend to increase rate of sulfur elimination and to decrease the total residual sulfur in the calcine product to less than about 0.2%, if so desired. It has been found that in roasting sulfide materials with high nickel sulfide contents, i.e., containing 64% to 72% nickel and cobalt, up to about 8% copper, up to about 2% iron and the balance sulfur, which melt at between about 1400° F. and 1450° F., a roasting temperature of at least 1650° F. should be used. For example, when roasting pelletized nickel sulfides containing 70% nickel plus cobalt, 25% sulfur, 2.5% copper and 0.6% iron at a rate of 2.5 tons per day of sulfide per square foot of roaster hearth the sulfur contents in the calcine product, as shown in the following table, were produced.

Roaster bed temperature, ° F.:	Percent total sulfur in calcine product
1650	2.0
1830	1.0
1920	0.5
2050	0.15

It has been found that for this nickel sulfide material when roasting at temperatures above about 2100° F. the residual sulfur content of the calcine is essentially determined by the short circuiting of partially roasted mate-

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rial to the calcine outlet. The degree of such short circuiting can be reduced by known means such as by the division of the roaster bed into compartments connected in series.

The calcines obtained by this novel process are a uniform and granular product which is substantially dust free. In the case of calcines formed from agglomerated sulfide fines it is found that the calcine product is not larger in particle size than the sulfide agglomerates fed into the fluid bed reactor. Fusion between granules is substantially avoided during the roasting operation with production of a porous product which has exceptional reducibility at relatively low temperatures.

It has been found that by a cyclic operation of the roaster residual sulfur in the product calcine can be reduced to less than about 0.05% if so desired. For example, pelletized nickel sulfide containing 70% nickel plus cobalt, 25% sulfur, 2.5% copper and 0.6% iron, fed to a fluid bed reactor at a rate of 3.5 pounds per minute per square foot of roaster hearth, was roasted at 2000° F. using an oxidizing gas made up from 90% air and 10% oxygen for forty minutes with no product being discharged during this period. Thereafter, feed was interrupted for twenty minutes while roasting continued for twenty minutes at 2000° F. using the aforementioned oxidizing gas and maintaining roaster temperature at 2000° F. by means of the addition of fuel oil to the bed. Thereafter, sufficient calcines were withdrawn in five minutes to restore the bed to its level at the start of the cycle. The final product contained 0.04% total sulfur.

In a variation of the present process calcines from roasting operation "4" may be transferred through a conduit as shown by "5" on the flow diagram to Stage II, shown at "6" for activating or cooling purposes. This Stage II follows Stage I in series arrangement and may be located below roasting Stage I as shown on the flow diagram or it may be part of a separate roaster unit.

It has been established that the reducibility of nickel oxide-containing materials can be significantly improved by a controlled oxidizing treatment at temperatures somewhat below that at which the original calcine was produced. The activation is of special importance for operations which involve gaseous reduction at relatively low temperatures required to produce an active metal for certain operations, e.g., extraction by carbonyl and cementation of copper from solution. To illustrate the effect of this low-temperature, controlled activation, a nickel oxide calcine was prepared by the fluid bed roasting of pelletized nickel sulfide containing 70% nickel plus cobalt, 25% sulfur, 2.5% copper and 0.6% iron at 2000° F. to a residual total sulfur content of 0.4%. Activation of the calcine was accomplished by transferring the calcine to a two-hour second stage treatment operation conducted using air as the activating agent and supplying heat to maintain a temperature of about 1350° F. Reduction to metal was obtained using hydrogen at 700° F. The time required for substantially complete reduction to metal of the activated calcine was 77% of that required for the same degree of reduction of the unactivated calcine. Although air was used in this test, pure oxygen or oxygenated air may be used to advantage in the activation treatment. For nickel sulfide materials it has been observed that the effectiveness of the activation treatment, after initial oxidation, greatly decreases at temperatures above about 1450° F. and below about 1250° F.

Fluid bed roasting followed by a low temperature activation, as described hereinbefore, is particularly applicable to mattes and other sulfide materials which have high nickel contents and which contain minor amounts of iron and minor amounts of cobalt and/or copper with the balance substantially all sulfur. Such mattes and other sulfide materials may have a nickel content of at least about 60%.

Where activation of the calcine product is not neces-

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sary, the second stage shown by "6" on the flow diagram may be used to advantage to cool the calcines from Stage I and at the same time recuperate part of the sensible heat from the calcines by using the hot calcines to preheat the roaster air. The second stage, as shown at "6" on the flow diagram, may consist of a fluid bed in which gas-solids contact is carried out or it may consist of any suitable means for effecting gas-solids contact, e.g., a rotary kiln. For cooling and activation purposes the oxygen-carrying gas is split between Stages I and II to obtain the desired temperature in Stage II. The oxygen-carrying gas before being split may be preheated in cooling step "7" or by heat exchange with the roaster exhaust gases and/or dust. If desired (as for purposes of heat economy) the air fed through Stage II may thereafter be fed to Stage I.

Cooling step "7" of the calcine product may be accomplished by any known cooling means, e.g., a fluidized bed, cooling screws and rotary coolers.

Gases issuing from roasting operation "4" contain varying amounts of incompletely roasted, partly molten solids, particularly when treating agglomerated fine sulfides, which must be cooled to below their softening range prior to or during gas-solids separation. Such cooling may be effected by direct or indirect heat exchange with cooling media such as water or air. Thus, cooling water may be introduced into the roaster, advantageously near the gas offtake as shown on the flow diagram. As a further safeguard against carrying partly molten solids into dust collecting equipment a baffle chamber can be installed ahead of the dust collecting or gas handling equipment as shown on the flow diagram.

Dust separated from the roaster exhaust gases in known dust collecting equipment such as cyclones, cottrells, scrubbers or baghouses, can be returned to agglomeration step "2" as shown on the flow diagram or can be diverted to separate treatment, especially where granulated sulfides are being roasted. Alternatively, a portion of the dust may be returned directly to roasting operation "4" for retreatment if desired. The substantially clean exhaust gases may be treated for recovery of their sulfur content by known means.

As described hereinbefore, the present invention is concerned with the treatment of granulated or agglomerated metal sulfides such as mattes and other sulfide materials containing iron and one or more elements from the group consisting of nickel, cobalt and copper. Furnace mattes containing as little as 5% nickel and cobalt and up to 55% iron can be successfully treated by this novel process. In treating such low-grade furnace mattes it may be desirable to merely partially roast the granulated material. Roasting of the granules to the desired sulfur content can be easily attained by this process with production of a uniform and granular product.

For the purpose of giving those skilled in the art a better understanding of the invention, the following illustrative examples are given:

Example I

Nickel sulfide filter cake, 95% minus 200 mesh, containing 70% nickel plus cobalt, 25% sulfur, 2.5% copper and 0.6% iron was pelletized with water and 1.5% nickel sulfate on a disc 36 inches in diameter at a rate of 1000 pounds per hour of dry material. The pellets averaged 8% water and had a particle size of up to 1/4 inch and not smaller than 1/46 inch diameter. These pellets were fed directly into a single stage fluid bed roaster at a rate of about 2.5 tons per day per square foot of roaster grate area. The roasting was carried out at 1980° F. using oxygenated air containing 29% oxygen by volume at a rate sufficient to give a space velocity of 7 feet per second at the grate. Sufficient fuel oil was burned above the bed to maintain the temperature of the roaster at 1980° F. The roaster gases were cooled to 1100° F. prior to gas-solids separation in two cyclones in

series. The dust collected from the cyclones averaged 25% by weight of the dry sulfide feed and was returned to the balling disc to be pelletized with fresh sulfide feed. A uniform, granular oxide product, substantially all minus 10 mesh and plus 65 mesh in size, containing 0.42% total sulfur was continuously withdrawn from the roaster.

Example II

The operation was similar to Example I except that pelletizing was carried out adding water and 11 imperial gallons of fuel oil per ton of sulfide feed and eliminating the use of nickel sulfate. Also, oxygen enrichment and the addition of fuel oil above the bed was discontinued. A calcine product similar to that in Example I contained 0.61% total sulfur.

Example III

This test was similar to Example I except that no oil was added to the bed but the oxygen-enrichment of the air was increased to 40% oxygen. A granular product, similar to that of Example I, with a total sulfur content of 0.3% was continuously withdrawn from the roaster.

Example IV

Molten nickel sulfide matte containing 70% nickel plus cobalt, 25% sulfur, 2.5% copper and 0.6% iron was granulated in a water stream to a product 80% minus 10 mesh in size of which 5% was minus 100 mesh in size. It was drained to about 3% moisture and fed continuously to a fluid bed reactor at a feed rate of about 2¼ tons per day per square foot of grate area. Roasting at 2000° F. was accomplished by fluidizing with air preheated to 400° F. at a space velocity of 13 feet per second above the roaster grate. A granular calcine product containing 0.9% total sulfur was continuously discharged.

Example V

This operation was similar to that in Example IV except that about 1½ imperial gallons per hour of oil per square foot of grate area was injected into the bed while increasing the amount of air to maintain 4.5% oxygen in the roaster exhaust gases. A granular calcine product containing 0.6% total sulfur was continuously withdrawn from the roaster.

Example VI

The operation was similar to Example IV except the roasting air was oxygenated to give an oxygen content of 30% by volume. Granular calcine product containing 0.08% total sulfur was continuously discharged from the roaster.

Example VII

Molten Bessemer matte containing 45% nickel, 31% copper, 21% sulfur and minor amounts of iron and cobalt was granulated in a stream of water. The matte granules were dewatered and fed continuously to a fluid bed reactor where they were fluidized and roasted with air at 1900° F. A uniform, granular, calcine product containing 0.5% sulfur was continuously discharged from the reactor.

It is to be observed that the present invention provides a novel process by which sulfide materials which have been formed into granules by agglomerating operations can be roasted by fluid bed roasting techniques at temperatures above their softening points while substantially avoiding fusion between granules in the fluid bed to form a uniform, granular and substantially dust-free calcine product with a particle size not larger than the sulfide granules fed to the fluid bed reactor and which has exceptional reducibility at relatively low temperatures.

The present invention also provides a novel process by which sulfide materials such as molten mattes are first broken up with agents such as water, air or steam, or by other methods such as by mechanical means, into granules which are subsequently roasted in a fluid bed

at temperatures above the softening points of the matte granules and converted into reactive oxide granules of coarse, uniform particle size substantially free of fines.

Although the present invention has been described in conjunction with preferred embodiments, it is to be understood that modifications and variations may be resorted to without departing from the spirit and scope of the invention, as those skilled in the art will readily understand. Such modifications and variations are considered to be within the purview and scope of the invention and appended claims.

We claim:

1. A process for the treatment of finely divided nickel sulfide material, containing more than about 15% sulfur and obtained from mattes and matte flotation products, for formation of a substantially completely granular, roasted, nickel oxide product therefrom, said nickel sulfide material containing minor amounts of iron and minor amounts of at least one element from the group consisting of copper and cobalt and the balance substantially all sulfur, which comprises agglomerating said sulfide material with water to form granules with a particle size of between about 65 mesh and about 0.5 inch, feeding said granules to a fluid bed reaction zone in which said sulfide material is oxidized at a temperature at least about 20° F. above the softening point of the sulfide material and below the melting point of the resulting oxide product in an oxygen-containing gas supplied to said reaction zone at a fluidizing rate sufficiently high to elutriate fines from the bed while maintaining the coarser material in a fluidized condition to insure rapid and substantially complete oxidation to less than about 2% sulfur and to produce a coarse, substantially completely granular, oxide product with a particle size not larger than that of the sulfide granules fed to said reaction zone, separating fine calcine dust from the reaction exhaust gases and returning said separated dust to the agglomerating step.

2. A process as defined in claim 1 in which part of the water in the sulfide agglomerates is replaced by fuel oil in the formation of the pellets.

3. A process as defined in claim 1 in which part of the water in the sulfide agglomerates is replaced by at least one material from the group consisting of nickel sulfate, sulfuric acid and lignosol.

4. A process for the treatment of nickel sulfide material containing more than about 15% sulfur and obtained from mattes and matte flotation products for formation of a substantially completely granular, roasted nickel oxide product therefrom, said nickel sulfide material containing minor amounts of iron and minor amounts of at least one element from the group consisting of copper and cobalt, which comprises granulating said matte material from the molten state to form sulfide granules with a particle size of between about 65 mesh and about 0.5 inch, feeding said sulfide granules to a fluid bed reaction zone in which said sulfide granules are oxidized at a temperature at least about 200° F. above their softening point and below the melting point of the resulting oxide product in an oxygen-containing gas supplied to said reaction zone at a fluidizing rate sufficiently high to elutriate fines from the bed while maintaining the coarser material in a fluidized condition to insure rapid and substantially complete oxidation to less than about 2% sulfur and to produce a coarse, substantially completely granular, oxide product with a particle size not larger than that of the sulfide granules fed to said reaction zone.

5. A process for the treatment of a nickel sulfide material containing more than about 15% sulfur and obtained from mattes and matte flotation products for formation of a substantially completely granular, roasted, nickel oxide product therefrom which comprises forming granules of said sulfide material having a particle size of between about 65 mesh and about 0.5 inch, feeding

said sulfide granules to a fluid bed reaction zone in which said sulfide granules are oxidized at a temperature at least about 200° F. above the softening point of the sulfide material and below the melting point of the resulting oxide product in an oxygen-containing gas supplied to said reaction zone at a fluidizing rate sufficiently high to elutriate fines from the bed while maintaining the coarser material in a fluidized condition to insure rapid and substantially complete oxidation to less than about 2% sulfur and to produce a coarse, substantially completely granular, oxide product with a particle size not larger than that of the sulfide granules fed to said reaction zone.

6. A process as defined in claim 5 in which the oxide product has a total sulfur content of less than about 0.2%.

7. A process as defined in claim 5 in which the feed to the bed is interrupted at intervals so that part of the roasting is performed while no feed material is being introduced into the bed.

8. A process as defined in claim 5 in which the oxide product is subjected to a further oxidation at between

about 1250° F. and about 1450° F. to activate said oxide.

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