

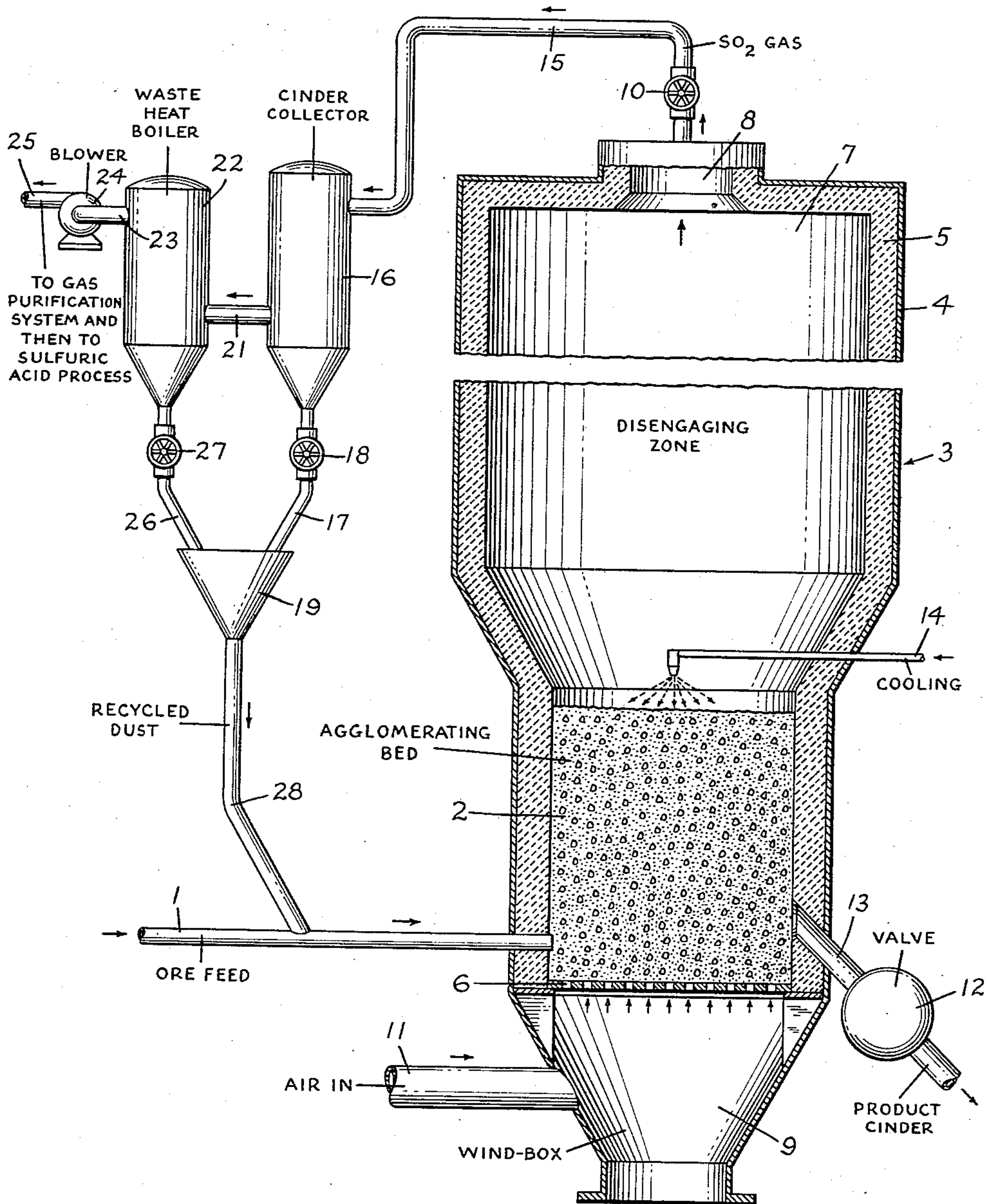
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FLUID BURNING OF FINELY DIVIDED SULFIDE ORE CONCENTRATES

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FLUID BURNING OF FINELY DIVIDED SULFIDE ORE CONCENTRATES

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This invention relates to treatment of sulfur-bearing ore and more particularly refers to a new and improved process for roasting metal sulfides to produce sulfur dioxide and metal oxide.

An object of the present invention is to provide an efficient economical process for roasting sulfide ore to effect simultaneous oxidation and effective agglomeration of the ore.

Another object of the present invention is to provide an efficient method for roasting finely divided sulfide ore and simultaneously agglomerating the ore to produce a cinder product considerably coarser in size than the original ore feed.

A further object of the present invention is to provide an improved method for converting finely divided sulfide ore into sulfur dioxide and concomitantly effect the agglomeration of the finely divided particles in a single zone fluid burner. A still further object is to provide a method for regulating the size of the agglomerates when roasting sulfide ore to produce sulfur dioxide and metal oxide.

Other objects of the present invention will be apparent from the following description and accompanying drawing.

The roasting or burning of sulfide ores to obtain sulfur dioxide gas and desulfurizing of said sulfides in a fluid bed is a fairly well known art. In this type of ore burning, the ore particles are suspended in an upwardly moving stream of gas so that the entire dense mass behaves much like a boiling liquid. The fluid bed has an apparent density much lighter than that of the solid particles. The bed consistency is uniform as to particle size distribution, chemical composition and temperature. There is a minimum air or gas velocity required to maintain bed fluidity which is a function of the true density of the sulfide ore, the average size distribution of the ore or desulfurized oxide particles present in the bed, but is considerably below the calculated free falling velocity of the largest particles present.

In the operation of fluid burners, upward gas velocities will range from about one to six feet per second. At these velocities all particles smaller than about 48 to 65 mesh are swept out of the fluid bed and must be later removed from the gas stream. Little difficulty is encountered when roasting coarse ground ores where the bulk of the ore particles are in the 4 to 40 mesh range. However, many sulfide ore concentrates are by-products from other metal recovery operations and have been ground so that substantially all the particles are 200 mesh or finer. Considerable difficulty is encountered in burning these fine sulfide concentrates in a fluid bed. In order to maintain burning in the fluid bed, essential for stable operation, upward air velocity must be greatly reduced, limiting burner capacity. Practically all the cinder particles are carried out of the bed along with the gas stream and must be subsequently removed. Cinder recovered is excessively fine and requires further processing before it can be utilized in metallurgical operations. The present invention

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is particularly concerned with the burning of these fine sulfide ore concentrates.

In the usual operation of fluid ore burners, it has been necessary to hold the temperature level of the fluid bed below the softening, or sintering point of the bed particles. Cinder bed, when burning sulfide concentrates, consists of iron oxides, gangue and undecomposed sulfides. While pure iron oxides have melting points above 2600° F., they begin to soften and become sticky at temperatures as low as 2200° F. The fluxing action of gangue plus undecomposed sulfides further lower the softening point so that softening of the particles and subsequent agglomeration will be experienced at temperatures as low as 1900–1950° F. Since oxidation of pyrites (FeS_2) and pyrrhotite ($\text{Fe}_{1-x}\text{S}_x$) to iron oxide and SO_2 is an exothermic reaction, it is necessary that heat be removed from the fluid bed to prevent the temperature level reaching a point where fusion is encountered. Heat removal may be accomplished by various known means, such as, by water or steam heat transfer wall tubes or bayonettes; by direct injection of water, waste acids or sulfates into the fluid bed; by the use of recycled cooled inert gas into the fluid bed; and by the use of cool recycled cinder. If temperature level of the bed falls below the point where good desulfurization takes place, sulfur content of the bed will increase and fusion again encountered since iron sulfides will melt at temperatures as low as 1200° F.

When softening of the cinder particles in the fluid bed is encountered, either from overheating or incomplete desulfurization, agglomeration of the individual bed particles takes place. As the agglomerates gradually grow, they reach a point where the upward gas velocity through the bed is insufficient to maintain them in the fluid state and they segregate to the bottom of the bed. Unstable fluidization with poor heat transfer ensues and the entire bed soon fuses to a solid mass.

From the foregoing it will be evident that in order to obtain successful roasting and effect simultaneous agglomeration of finely divided sulfide ore in the fluidized bed the following conditions must exist: (a) agglomeration of the finely divided sulfide ore to produce a cinder product considerably coarser in size than the original ore feed; (b) controlled degree of agglomeration, i. e. regulating agglomerates of a sufficiently large size adapted for use in metallurgical operations and preventing formation of oversized agglomerates which will not fluidize and will destroy the operation of the fluidized roasting bed; (c) agglomeration at a high rate—formation of agglomerates at a low rate would reduce the capacity of the unit and make the operation commercially impractical; (d) desulfurization at a high rate of reaction; (e) material reduction in the proportion of fines carried by sulfur dioxide gases leaving the fluid burner; and (f) substantially complete desulfurization of cinder, i. e. producing a cinder agglomerate below 1.0% sulfur.

In accordance with the present invention finely divided metal sulfide ore may be converted to substantially complete desulfurized (below 1.0% sulfur) cinder of controlled size, predominantly in the 4 to 40 mesh range, at a high rate of oxidation and agglomeration in a fluid bed without loss of fluidity of the bed by cyclically varying the bed temperature, causing the temperature of the fluid bed to rise to a point of incipient sinter, i. e., a point where slight softening and agglomeration of the individual cinder particles take place, and then cooling the bed to a point below the cinder softening and agglomeration temperature, i. e. below incipient sintering, and continuously repeating said cyclic variation in bed temperature from a point of incipient sinter to a point below incipient sinter.

The accompanying drawing is a diagrammatic flow sheet illustrating one method of practicing the present invention.

Referring to the drawing, the charge to the system is a sulfide ore, particularly an iron sulfide ore, containing an appreciable percent, at least 25%, preferably more than 50%, of finely divided iron sulfide particles finer than 40 mesh in size. Although the process is particularly directed to the burning of iron sulfide flotation concentrates such as pyrite and pyrrhotite, it can also be applied to roasting iron sulfide ores consisting of a mixture of particles larger than about 40 mesh up to about $\frac{1}{4}$ inch size and particles finer than 40 mesh in an amount of at least about 25 percent of the finer particles and also to roasting other metallic sulfides. Some of the ores contain appreciable amounts of moisture. However, it is unnecessary to go to the expense of subjecting the feed ore to preliminary drying prior to introducing the ore feed through line 1 into the fluidized bed 2 contained in agglomerating chamber 3. Agglomerating chamber 3 may be any suitable reaction chamber for carrying out fluidized bed operations constructed of a metallic outer shell 4 and inner heat resistant refractory lining 5, a perforated bed support 6 sometimes termed "perforated plate," "screen," "hearth," and "grate," an area of free space above the fluidized bed called disengaging zone 7 to permit heavier particles of cinder and ore to separate from the rising gases and fall down into the bed, an outlet 8 near the top of chamber 3 for the release of SO_2 gas generated by combustion of the sulfide ore, a wind-box 9 beneath perforated support 6, an air inlet 11 for the introduction of air into wind-box 9 and up through perforated support 6, and an outlet for the discharge of cinder product regulated by valve 12 in discharge conduit 13. The size of agglomerating chamber 3 will vary depending particularly upon the capacity desired—roughly a minimum of one net ton per day of ore may be treated per square foot of grate 6 area. The height of fluidized bed 2 may be varied over a fairly wide range, e. g. from about 2–6 feet.

The agglomerating bed 2 consists principally of cinder ranging from 4 to 40 mesh and is maintained in a fluidized condition by air or other oxygen-containing gas entering through line 11 and passing up through perforations in grate 6 upwardly through agglomerating bed 2. The velocity of the air up through bed 2 is maintained sufficiently low to prevent any substantial elutriation of particles greater in size than about 40 mesh from the bed. Ordinarily, a gas velocity through the bed of about 3–6 feet per second will maintain the fluidized bed in good condition without elutriating the heavier particles of cinder. The operation may be initiated by first introducing a charge of cinder to chamber 3, passing air at a low rate into the chamber through line 11 and introducing fuel such as gas or oil, not shown in the drawing, into chamber 3, which fuel will burn and heat the unit up to about the ignition temperature of iron sulfide, say about $1100\text{--}1400^\circ\text{F}$. whereupon ore feed is introduced through line 1 into cinder bed 2, the introduction of external fuel stopped, and the air feed increased with increasing ore feed until the desired bed temperature attained and the ore feed and air feed adjusted to the desired regulated rate. The cinder charge may also be heated to ignition temperature by means of air, oil or gas-air burner impinging on the fluid bed.

Roasting of the ore in bed 2 must be conducted as a cyclic operation, i. e. a thermal cycle wherein the roasting temperature is caused to fall and rise repetitively by introduction of a cooling medium into the fluidized burner at regulated intervals of time. Temperature rise or restoration takes place in the intervals when introduction of cooling medium into the fluidized burner is interrupted. The more or less regularly fluctuating temperature of the fluidized burner results in sintering of finely divided particles together but the sintering process is controlled by

the regularly interrupted introduction of cooling medium so that clinker formation is avoided but agglomeration of fines is accomplished. Cinder thus produced is more acceptable to metallurgical users and the value of the cinder is enhanced. The maximum temperature level to which the fluidized bed should be permitted to rise is that of incipient sintering which is the point where slight softening and agglomeration of the individual cinder particles take place. The incipient sintering point will, of course, vary somewhat with the character of the particular ore feed. Cinder from pyrrhotites and pyrites usually have an incipient sintering point within the range of about $1880\text{--}2100^\circ\text{F}$., more generally within the range of about $1900\text{--}2050^\circ\text{F}$. Merely as illustrative a pyrites flotation concentrate had an incipient sintering point of approximately 2050°F . whereas a dry ground pyrrhotite concentrate with a lower fusion temperature due to its higher silica content, had an incipient sintering point of about 1900°F . The lower temperature level of the thermal cycle is desirably about $25^\circ\text{--}100^\circ\text{F}$., preferably about $50\text{--}70^\circ\text{F}$. lower than the incipient sintering point. As illustrative, good results are obtained in processing pyrrhotite concentrate in the thermal cyclic operation in which the upper temperature is 1900°C . and the lower temperature 1850°C .; and with pyrites flotation concentrate in which the range is $2030\text{--}1950^\circ\text{F}$. The length of each thermal cycle may be varied to control the extent of agglomeration. However, the period during which agglomeration is encountered must not be extended so that segregation in the fluidized bed take place in order that stable bed conditions are continuously maintained. We have obtained good operation with the heating cycle of about 5–15 minutes, preferably 8–12 minutes during which period of time the bed is being elevated from the low temperature level to the high temperature level called heating period which is approximately 2–6 times, preferably 3–5 times the cooling period, the period of time required to drop the temperature from the high level to the low level. Cooling of the bed to accomplish lowering of the temperature during the cycle may be provided by the direct water injection through line 14. Other fluids or methods such as the use of waste acids or sulfates may be utilized to effect the cyclic drop in temperature of bed 2.

Another convenient method of cyclic cooling is by means of recycled cooled cinder periodically injected directly with ore feed or introduced into the fluid bed through separate entry ports. Cyclic introduction of cooled recycled gas may also be employed.

In view of the high exothermic heat of reaction generated by the oxidation of iron sulfide additional cooling may be continuously provided if necessary to aid in maintaining the thermal cycle.

For comparative purposes the following will illustrate the differences between operating in a fluidized bed with finely divided ore in conventional manner as compared with thermo-cyclic operation as described herein. Iron pyrrhotite concentrate containing 3.4% coarser than 65 mesh and 73.8% finer than 200 mesh fractions can be burned in a fluidized burner of 18 square feet cross-sectional area at the rate of 2000 pounds per hour in a 60 inch fluid bed with 1150 cubic feet per minute of fluidizing air and the bed maintained at a temperature of 1850°F . by continuous direct injection of 1.5 gallons of water per minute to the bed. Under these conditions practically 100% of the cinder product is carried out of the fluid bed along with the SO_2 stream. The bulk of the cinder can be removed from the gas stream in subsequent dust collectors and has substantially the same screen analysis as the ore feed. Finest particles passing through the collectors must be removed by other methods before the SO_2 gas can be utilized for conversion to H_2SO_4 or other uses.

In contrast by removing all cooling, in this instance water, the fluid bed temperature increases from 1850°

F. to 1900° F. over a ten minute period. Cooling water is then admitted to the bed at the rate of 3 to 4 gallons per minute and the bed temperature brought back to 1850° F. in about 2½ minutes. Cooling water is then removed and the cycle repeated. Under these conditions, agglomeration of the cinder particles in the bed is achieved and, equally important, fluid bed conditions are maintained. Over 50% of the agglomerated cinder is discharged from the fluid bed as 4 to 48 mesh aggregates. The balance of the cinder is recovered in dust collectors and can be recycled back into the burner along with fresh ore feed, if desired.

Some variation in the height of agglomerating bed 2 is not critical to the operation and therefore the discharge of the cinder through line 13 and valve 12 may be intermittent or continuous. The cinder product has a low sulfur content, less than 1.0%, usually less than 0.5% and is in the form of aggregates ranging generally from about 4 to 45 mesh.

SO₂ gas and suspended cinder released from the top of agglomerating chamber 3 through line 15 and valve 10 pass through cinder collector 16 wherein most of the cinder settles out and discharges from the bottom thereof through line 17 and valve 18 into hopper 19. Gases containing some remaining cinder are passed through conduit 21 into and through heat boiler 22 wherein further settling of the cinder takes place discharging through line 26 and valve 27 into hopper 19. Gases leaving waste heat boiler 22 discharge through line 23 and are sent by blower 24 through conduit 25 to conventional gas purification and then contact sulfuric acid process for the production of sulfuric acid. If desired a portion of the cooled gases discharging through line 25 may be returned to chamber 3. Fine cinder collecting in hopper 19 may be recycled via lines 28 and 1 to fluidized bed 2 for agglomeration into coarser particles.

The following example illustrates one method of carrying out the invention.

Ore feed analyzing 33% sulfur and 49% iron, and having the following screen analysis: —20 mesh +28 mesh, 0.1%, —28 mesh +35 mesh, 0.2%, —35 mesh +48 mesh, 0.7%, —48 mesh +65 mesh, 1.8%, —65 mesh +100 mesh, 3.9%, —100 mesh +200 mesh, 17.9%, —200 mesh, 75.4%, was continuously fed into a fluidized burner substantially as illustrated in the drawing having a lower internal diameter of 4 feet, 4 inches and an upper diameter of 6 feet, 4 inches and an overall height of about 30 feet, at the rate of about 24.0 net tons per day. Primary air was fed up through the grate into a fluidized bed of cinder at the rate of 900 cubic feet per minute. Additional air was introduced with ore injection at the rate of 200 cubic feet per minute making the total air fed to the fluidized bed at the rate of 1100 cubic feet per minute. The fluidized bed depth was maintained between 57–59" H₂O. The heating cycle was 1850° to 1900° F.—10 minutes average. The cooling cycle was 1900° to 1850° F.—2½ minutes average. Cooling water was not added to the bed during the heating cycle. Cooling water in an amount of 3–4 gallons per minute was added to the bed during the cooling cycle. The gas stream released from the burner contained about 12% SO₂.

The cinder discharge from the fluidized bed was 14.7 tons per day and contained 0.19% sulfur. The cinder had the following screen analysis: +4 mesh, 1.6%, —4 mesh +6 mesh, 3.0%, —6 mesh +10 mesh, 10.9%, —14 mesh +20 mesh, 12.4%, —20 mesh +28 mesh, 34.2%, —28 mesh +35 mesh, 21.5%, —35 mesh +48 mesh, 7.0%, —48 mesh +65 mesh, 1.8%, —65 mesh +100 mesh, 0.2%. Cinder separated from the dust collector was at the rate of six tons per day and contained 0.62% sulfur. This cinder had the following screen analysis: —10 mesh +14 mesh, .1%, —14 mesh +20 mesh, .3%, —20 mesh +28 mesh, .4%, —28 mesh +35 mesh, .3%, —35 mesh +48 mesh, .3%, —48 mesh +65 mesh, .9%,

—65 mesh +100 mesh, 2.9%, —100 mesh, +200 mesh, 19.0%, —200 mesh, 75.8%.

From the foregoing example it will be noted that 99% of the ore feed was of a particle size finer than 48 mesh. However 90% of the cinder discharged from the fluidized bed was of a particle size larger than 48 mesh. Also, it should be noted that almost 70% of the cinder was discharged from the cinder bed and did not carry over with the gases thereby greatly simplifying the task of separating finely divided particles of cinder from the SO₂ gas which is necessary before conversion into sulfuric acid.

Another example of carrying out the invention is by the use of cooled recycled cinder dust for cyclic temperature control. In this instance equipment arrangement shown in the diagrammatic flow sheet was modified in that the dust-laden gas passed through the waste heat boiler 22 ahead of the collector 16 so that the dust was cooled to 800°–900° F. The dust collector cinder discharged into a storage bin from which it was cyclically withdrawn at a controlled rate and conveyed into the fluid bed by means of an air stream. The recycled dust inlet was about 90° distant but in the same plane as the ore feed. Pyrites ore feed analyzing 48% sulfur and 48% iron and having the following screen analysis: —10 mesh +100 mesh, 0.7%, —100 mesh +200 mesh, 12.4% and —200 mesh 86.9%, was continuously fed into the fluidized burner described in the previous example at the rate of about 22.9 net tons per day. Primary air was fed up through the grate into the fluidized bed of cinder at the rate of 850 cubic feet per minute. Additional air was introduced with ore injection at the rate of 200 cubic feet per minute and continuously through the recycled dust return line at the rate of 200 cubic feet per minute making total air fed to the fluidized bed at the rate of 1250 cubic feet per minute. The fluidized bed depth was maintained at about 55" H₂O. The heating cycle was 1975° to 2025° F.—7½ minute average. The cooling cycle during which recycled dust at about 800° F. was fed to the bed at the rate of about 0.07 net ton per minute or 0.175 net ton per 2½ minute cycle was 2025° F. to 1975° F. The gas stream released from the burner contained about 14% SO₂ and 1% O₂. The cinder discharged from the fluid bed was 15.4 tons per day, the theoretical amount of cinder equivalent to the ore feed, and contained 0.18% S. The cinder had the following screen analysis: +4 mesh none, —4 mesh +10 mesh, 1.8%, —10 mesh +14 mesh, 7.3%, —14 mesh +20 mesh, 26.5%, —20 mesh +28 mesh, 24.3%, +28 mesh +35 mesh, 15.7%, —35 mesh +48 mesh 11.0%, —48 mesh +65 mesh, 7.0%, —65 mesh +100 mesh, 5.3%, —100 mesh 1.1%. Fine cinder recycled through the system for cyclic cooling of the bed contained 0.56% S and had the following screen analysis: +65 mesh 2.0%, —65 mesh +100 mesh 4.0%, —100 mesh +200 mesh 22.0%, —200 mesh 72.0%. In this case substantially the entire amount of ore feed was agglomerated as well as burned in the fluid bed and discharged from the bed. Dust fed through the system to effect cyclic cooling was collected and substantially all recycled through the system.

Although certain preferred embodiments of the invention have been disclosed for purpose of illustration it will be evident that various changes and modifications may be made therein without departing from the scope and spirit of the invention.

We claim:

1. A process for roasting finely divided iron sulfide to produce sulfur dioxide and simultaneously agglomerate the iron sulfide to produce a cinder product considerably coarser in size than the iron sulfide feed which comprises maintaining a fluidized bed of particles of low sulfur cinder, feeding finely divided iron sulfide containing predominantly finely divided iron sulfide particles below a 200 mesh size into said fluidized bed at a point below the surface of said bed, roasting the iron sulfide by passing a

stream of oxygen-containing gas through the fluidized bed at a velocity sufficiently high to maintain the solids in the bed in the fluidized state, and cyclically varying the bed temperature by causing the temperature of the fluidized bed to rise to a point of incipient sintering, i. e. a point where slight softening and agglomeration of the cinder particles take place, and then cooling the bed to a point below the cinder softening and agglomeration temperature, i. e. below incipient sintering, and continuously repeating said cyclic variation in bed temperature from a point of incipient sinter to a point below incipient sinter.

2. A process for roasting finely divided iron sulfide to produce sulfur dioxide and iron oxide cinder containing less than 1% sulfur, and simultaneously agglomerating the iron sulfide to produce a cinder product considerably coarser in size than the iron sulfide feed which comprises maintaining a fluidized bed of agglomerated particles of low sulfur cinder, feeding the iron sulfide containing predominantly finely divided iron sulfide particles below a 200 mesh size into said fluidized bed at a point below the surface of said bed, roasting the iron sulfide by passing a stream of oxygen-containing gas through the fluidized bed at a velocity sufficiently high to maintain the solids in the bed in the fluidized state, and cyclically varying the bed temperature by causing the temperature of the fluidized bed to rise to a point of incipient sintering, i. e. a point where slight softening and agglomeration of the cinder particles take place, within the range of about 1880–2100° F., and then cooling the bed about 25–100° F. below the cinder softening and agglomeration temperature, i. e. below incipient sintering and continuously repeating said cyclic variations in bed temperature from a point of incipient sinter to point below incipient sinter.

3. A process as claimed in claim 2 wherein cooled iron oxide dust is introduced into the fluid bed to effect cooling during each cycle.

4. A process for roasting finely divided iron sulfide to produce sulfur dioxide and iron oxide cinder containing less than 1% sulfur, and simultaneously agglomerating the iron sulfide to produce a cinder product considerably coarser in size than the iron sulfide feed which comprises maintaining a fluidized bed of agglomerated particles of low sulfur cinder, feeding the iron sulfide containing predominantly finely divided iron sulfide particles below a 200 mesh size into said fluidized bed at a point below the surface of said bed, roasting the iron sulfide by passing a stream of oxygen-containing gas through the fluidized bed at a velocity sufficiently high to maintain the solids in the bed in the fluidized state, and cyclically varying the bed temperature by causing the temperature of the fluidized bed to rise to a point of incipient sintering, i. e. a point where slight softening and agglomeration of the cinder particles take place, within the range of about 1900–2050° F., and then cooling the bed about 50–70° F. below the cinder softening and agglomeration temperature, i. e.

below incipient sintering and continuously repeating said cyclic variations in bed temperature from a point of incipient sinter to a point below incipient sinter, discharging low sulfur cinder from said fluidized bed, releasing sulfur dioxide gas containing suspended finely divided particles of cinder from the top of said fluidized bed, separating the finely divided particles of cinder from the sulfur dioxide gas, and recycling separated finely divided particles of cinder to the fluidized bed.

5. A process for roasting finely divided iron sulfide ore to produce sulfur dioxide and iron oxide cinder containing less than 1% sulfur and simultaneously agglomerating the iron sulfide to produce a cinder product considerably coarser in size than the iron feed which comprises maintaining a fluidized bed of agglomerate particles of low sulfur iron sulfide cinder consisting principally of cinder ranging from about +4 mesh to -40 mesh, feeding iron sulfide ore containing more than 50% of finely divided iron sulfide particles below a 200 mesh size into said fluidized bed at a point below the surface of said bed, roasting the iron sulfide by passing a stream of air through the fluidized bed at a velocity sufficiently high to maintain the solids in the bed in the fluidized state, and cyclically varying the bed temperature by causing the temperature of the fluidized bed to rise to a point of incipient sintering, i. e. a point where slight softening and agglomeration of the cinder particles take place, within the range of about 1900–2050° F., and then cooling the bed about 50–70° F. below the cinder softening and agglomeration temperature, i. e. below the incipient sintering by the introduction of a liquid in contact with the fluidized bed, terminating the introduction of liquid thereby causing the fluidized bed to again rise to a point of incipient sintering, again introducing cooling liquid to cool the fluidized bed to a point below incipient sintering and continuously repeating said cyclic variations in bed temperature from a point of incipient sinter to a point below incipient sinter.

6. A process as claimed in claim 5 wherein the cooling liquid employed is water and wherein the heating period of each cycle is about 5–15 minutes and wherein said heating period is approximately 2–6 times the cooling period of each cycle.

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