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[54] **PROCESS FOR THE COMMINUTION OF PARTICULATE ALLUMINACEOUS MATERIALS**

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[57] **ABSTRACT**

Particulate aluminaceous materials employed in the manufacture of aluminum, such as aluminaceous ores or intermediates, are comminuted prior to the extraction of aluminum therefrom, by soaking the material in an alkaline or acidic electrolyte for a period of from about 0.2 to about 24 hours without the need for stirring and other agitation, whereby the structure of the particulate material becomes loosened, and then completing the comminution by subjecting the particulate material to primarily shearing forces until the average particle size of the material is under 1 mm.

7 Claims, No Drawings

PROCESS FOR THE COMMINUTION OF PARTICULATE ALLUMINACEOUS MATERIALS

DETAILED DESCRIPTION

This is a continuation-in-part of our U.S. application for Letters Pat. Ser. No. 143,973, filed 17 May 1971, now abandoned.

This invention relates to a novel method for the comminution of aluminaceous materials employed in the manufacture of aluminum, more particularly the present invention relates to the comminution of such materials with or without precrushing before the commencement of the present process to an intermediate particle size, which materials are aluminaceous ores or intermediates employed in the manufacture of aluminum.

In aluminum manufacturing processes based on the wet extraction of aluminum from aluminaceous raw materials such as from bauxite, the extraction is carried out usually by leaching out finely ground particulate aluminaceous materials. In such aluminum manufacturing processes, such as the Bayer process, ball mills, rod mills or other mills are employed which operate principally on the impact principle. This is, because in the Bayer process the lixiviation step requires that the average particle size of the ore be under 1 mm. and about 80% of the ore have an average particle size of around 0.2 mm. As the aluminum requirements of the world economy increase immense quantities of ore have to be ground to the necessary fineness. The efficiency of the extraction is greatly influenced by the fineness of the particle size, therefore adequate grinding of the ore is of utmost importance.

Bauxite and other aluminaceous materials are usually precrushed to an intermediate particle size and then are subjected to fine grinding until the final desired particle fineness is achieved. Fine grinding is usually carried out in ball or other rotary mills, hammer mills, drypans and the like comminution equipment operating principally on the basis of compression by impact or otherwise. Fine grinding is carried out with the material being in the dry state or a liquid grinding medium is added to the particles immediately before grinding is commenced. Fine grinding, such as in the case of ball mills, requires a sizable installation of a number of large mills operating in parallel. Such an installation of ball mills requires a relatively large capital investment, and a large amount of operating space. The energy requirement of operating a ball mill installation is relatively large, due to the requirement of rotating a relatively large proportion of the charge of grinding balls. Furthermore, the charge and the mill lining wear away at a relatively fast rate, requiring frequent maintainance and considerable downtime. Rotary mills are very noisy and their opertion is objectionable from the point of view of health.

A variety of bauxites can be found in nature, such as those composed mainly of hydrargillite, boehmite or diaspore. The softer bauxites, such as those containing mainly hidrargillite, for example bauxites from Surinam or the lately very frequently used Australian bauxite can be fine ground with ball mills only with a very poor grinding efficiency. When wet grinding is attempted with these softer or semi-hard ores (e.g., of up to 5–6 on the Mohs scale), the grinding efficiency is even further reduced because the bauxite forms a thick paste on the walls of the mill and the grinding balls become embedded in the paste.

It is an object of the present invention to provide a process for the comminution of aluminaceous materials, such as bauxite, without the need for fine grinding in accordance with the prior art, thus eliminating all of the attendant disadvantages.

In accordance with the present invention a process is provided, and the product obtained thereby, for the comminution of particulate aluminaceous starting materials, the process comprising before or after coarse crushing soaking the starting material in an alkaline or acidic electrolyte for a period of from about 0.2 to about 24 hours without the need for stirring, and completing the comminution by subjecting the particulate material to predominantly shearing forces until the average particle size of the material is reduced to under 1 mm.

The present invention is based on our discovery that the structure of aluminaceous materials will become sufficiently loosened by soaking in an alkaline or acidic electrolyte without any need for stirring or other agitation that the material can be reduced in its particle size by the application of predominantly shearing forces without the drawbacks attendant upon compressive grinding such as in the case of ball mills. The starting material is not of a homogenous character, but is comprised of various crystalline and/or amorphous aggregates which are loosened during the soaking in the electrolyte. As the liquid penetrates into the particles the aluminum content goes into solution even if only to a slight extent, but to an extent sufficient to permit the individual aggregates to be shifted apart from each other when subjected to shearing influences. This manifests itself in an apparent reduction in the hardness of the material. Particle size diminution by shearing forces can be accomplished in a manner which requires a relatively small consumption of energy to the optimum particle size for subsequent lixiviation.

The starting materials which can be employed in the process of the present invention include aluminaceous ores as well as intermediates in the manufacture of aluminum, which have to be ground to a fine particle size before further processing. Such intermediates can include the products of various poorer quality bauxites or of clays, which were calcined at about 500°C to about 900°C. Such intermediates can suitably be leached with acids, such as nitric acid. Other byproducts include the products obtained by heat treatment together with limestone or CaO, of poorer grade bauxites, aluminaceous coal shales, coal slags, fly ashes, clays and the like. The aluminum content of the latter type of byproducts was thus converted into calcium aluminates prior to treatment in accordance with the present process. These latter types of byproducts are suitably leached out with dilute alkaline caustic solutions. The aluminaceous ores which can be suitably treated in accordance with the process of the present invention are primarily bauxites which, if they are to be used in the Bayer process, are suitably extracted with more concentrated caustic solutions.

It is to be understood that while some of the further disclosure of the present invention may principally refer to the treatment of bauxite with a caustic solution, the employment of other solutions with other materials is equally contemplated, as set forth hereinabove.

The electrolyte in which the aluminaceous material is soaked is suitably the same as will be used, after the comminution is completed, for the lixivation of the aluminum for the further processing thereof. In the case of bauxites the soaking electrolyte is suitably a more concentrated caustic solution of the kind used for aluminum extraction in the Bayer process. Part of the aluminum will become leached out during the soaking period, therefore the material, after soaking is completed, is suitably further treated in conjunction with the soaking solution to recover ultimately all aluminum values therefrom. The soaking period will vary according to the particle size of the aluminaceous starting material, as well as according to the character of the material itself. For example, in the case of bauxite precrushed to an average particle size of 10–30 mm. a soaking period of 0.5 – 2.5 hours has been found to be generally satisfactory. The soaking period can be accelerated to a certain extent by heating the electrolyte, suitably a while after the soaking has commenced. As the soaking progresses, the structure of the bauxite particles becomes loosened, partly due to a partial lixiviation of the aluminum and partly due to wetting of the interior of the particle due to capillary phenomena. Due to this partial decomposition of the particles their apparent hardness is decreased by up to 3–4 Mohs hardness numbers.

The resistance of the aluminaceous particles soaked in the electrolyte is reduced vis a vis shearing forces as we discovered, therefore, in accordance with the present invention the particles can be comminuted by requiring a small part of the energy, such as one fourth, which is required by comminution in accordance with prior art comminution techniques such as ball milling.

In accordance with one embodiment of the process of the present invention raw bauxite from the mines is first optionally subjected to particle size classification and is then subjected to a very coarse precrushing to obtain particles having a maximum average size for example of 60 mm. A slurry is prepared from the very coarsely precrushed particles and a caustic solution. It is from this point where the beginning of the soaking period takes place. The slurry is then suitably transported with the aid of a slurry pump through a pipeline to the place where its further treatment takes place. While the ore particles do not require any expenditure of energy for mixing or other forms of agitation during soaking, any incidental agitation to which the particles may be subjected, such as during transportation through a pipeline, is not detrimental to the material. In fact, depending on the character of the bauxite particles, friction of the particles against each other and against the walls of the pipeline through which it is transported may contribute to the further comminution of the bauxite. Instead of delivery of the bauxite such as from the mine, together with the caustic soaking solution, the soaking of the particles can also be carried out in one or more tanks while the particles are maintained in a stationary condition.

A variety of devices are known which can be used to exert shearing forces upon the particulate material after the soaking has been completed. Grinding pumps provided with a revolving disc have been found most suitable for the purpose, because in addition to fine grinding with shearing forces, these devices also operate as pumps for the transportation of the finely particulate slurry. Depending on the construction of the grinding means, the size and character of the bauxite particles and the duration of the soaking period, the grinding step can be carried out in a single pass through the grinder or by passing the material several times through the grinder or through a plurality of shearing-type grinders connected in series.

Grinding pumps which have been found suitable for carrying out the process of the present invention are generally based on a disc arranged on a slant for revolution in a housing. Thus the disc describes a tumbling motion in the axial direction while revolving in the housing. Pumping is carried out due to the centrifugal forces acting upon the slurry. Serrations, teeth or other protrusions on the inner surface of the housing and/or the slanting disc result in a comminution of the particles of the slurry transported by the pump. The degree of comminution can be adjusted by varying the gap between the effective rotating diameter of the disc and the inward extremities of the housing. A particularly suitable device is manufactured under the trade designations Gorator and Rekord-Gorator by the firm Hoelschertechnic G.m.b.H. in Germany.

The bauxite particles, whether precrushed or not, are suitably exposed to soaking in transportation pipelines and/or one or more soaking tanks. If desired coarse crushing devices can be provided in or prior to the soaking tanks. If the ore is crushed prior to soaking, the output of the one or more grinding pump stages is suitably recirculated for greater uniformity of particle size. If the ore was not precrushed prior to or during soaking, the output of the grinding pump or pumps is suitably conducted with a slurry pump to a classification device, such as a hydrocyclone or hydrocyclone battery, from which oversize fractions can be recirculated into the fine grinding stage or stages. The liquid content of the slurry can be adjusted by the addition of further amounts of electrolyte at any point of the process. Suitably a screw conveyor is used to transport the coarse slurry from the soaking stage, such as series-and/or parallel-connected soaking tank or tanks, to the fine grinding stage, because such transport devices are suitable for the simultaneous conveyance of the solid and liquid components of the coarser slurry.

The wear on the components of the grinding pump is minimized due to the softening of the bauxite particles during the prior soaking. Thus the caustic slurry also acts as a lubricant. Maintainance of the grinding pump, such as changing of the replaceable components is simpler, faster and less costly than in the case of ball mills.

An experiment was carried out to compare the parameters of the process of the present invention and of the prior art. A bauxite from Iszkaszentgyorgy (hungary) was used, having an average particle size between 15 and 20 mm. The starting slurry contained 240 g/liter solids in the caustic solution. The soaking period was 0.5 hours. The inside diameter of the Rekord Gorator grinding pump was 250 mm., its RPM was 1440 and its transporting capacity was 40 m^3/hour slurry at an about 2 m. head. The amount of ground bauxite was 240 × 40 kg/hour = 9.6 tons/hour. The specific energy requirement of the grinding pump was 16 kWhours/9.6 tons= 1.7 kWhours/ton. When the grinding was carried out in a ball mill having a 40 tons/hour grinding capacity with the same bauxite and having the same particle size dis-

tribution, the specific energy requirement was 6.5 kWhours/ton.

The foregoing comparative experiment demonstrates that the energy requirement in the case of compressive inpact-type grinding was 3.8 times as high as that required for shearing-type grinding in accordance with the present invention. This observation can be explained also by the fact that the case of impact-type compressive grinding large masses of ballast are required (such as balls or other grinding media) to exert the necessary impact forces and also due to the large contacting surfaces. In the case of shearing-type grinding, on the other hand, the forces are concentrated along small, edge-like surfaces and the bauxite also manifests lesser resistance against grinding, because its structure was loosened during the soaking.

In the following examples the best modes contemplated for carrying out the present invention, are illustrated. All parts, percentages and ratios are by weight and temperatures are in °C, unless otherwise specified.

EXAMPLE 1

6.3 metric tons of buaxite from Iszkaszentgyorgy, Hungary, having an alumina content of 50.5% and a modulus (SiO_2 / Al_2O_3) of 8.2 is precrushed to a maximum particle size of about 30 mm, and then is mixed with 7.3 m^3 of a sodium aluminate soaking solution having a temperature of 54° and containing 190.2 g/l Na_2O and 78.4 g/l Al_2O_3. The slurry is allowed to stand in a tank for 30 minutes. The bottom of the tank is concavely conical above its outlet and a pair of crusher rolls is arranged above the outlet and rotating in opposite directions. The rolls crush the presoaked buaxite to an average particle size of about 10–15 mm. The bauxite slurry is fed into a screw conveyor placed horizontally below the tank. Rotation of the crusher rolls is provided by a V-belt of adequate gear ratio exterior of the tank, from the axle of the screw conveyor to synchronize the speed of the crushing rolls with the transport speed of the conveyor screw.

Further amount of soaking liquor are continuously added to the slurry which is horizontally transported by the screw conveyor into a Gorator grinding pump (type 25/10–10). The pump is run at the rate of 1.400 r.p.m. The soaking liquor feed before the grinding pump is adjusted so that the bauxite content of the slurry in the grinding pump is about 300 g/l. After the grinding process is started, the precrushed bauxite and soaking liquor are continuously fed into the soaking tank for continuous operation with an output of about 9 t/hour bauxite and 10.3 m^3/hour leaching liquor. The soaking time of the bauxite in the tank is adjusted to an average of 30 minutes for continuous operation.

According to a wet screen test of a sample of the finely ground slurry the sieve residue is 6.7 percent on a screen having a 315 micron nominal aperture and 9.1 percent on a screen having a 200 micron nominal aperture. Recirculation of the ground bauxite slurry to the grinding pump results in a slurry having screen test residue results of 4.2 percent and 6.6 percent repsectively.

EXAMPLE 2

1.8 metric tons of moist bauxite from Nyirad, Hungary, having an alumina content of 49.5% and a modulus of 7.2 is mixed with 5 m^3 of sodium aluminate soaking liquor containing 205 g/l Na_2O and 83.3 g/l Al_2O_3. The slurry is allowed to stand in a tank for 90 minutes. The bauxite is then ground by a pair of crushing rolls, arranged as in Example 1, to a grain size of about 30 mm and then continuously led into a screw conveyor as in Example 1. The bauxite slurry is continuously horizo-tally fed into a Gorator grinding pump ran at a rate of 1,400 r.p.m. The average particle size of the bauxite in the discharged finely ground slurry is about 2 mm. The pretreated slurry is conducted into a disintegrator-type grinding pump which is similarly structured as a centrifugal pump. The slurry is ground here to a very fine particle size and simultaneously it is transported to the next processing location. According to a wet screen test of a resulting slurry sample, the sieve residue is 2.1 percent on a screen having 315 micron nominal aperture and 3.6 percent on a screen having 200 micron nominal aperture.

EXAMPLE 3

3 metric tons of moist bauxite from Iszkaszentgyorgy, Hungary, having an Al_2O_3 content of 50.5 percent and a modulus of 8.2, is mixed with 4 m^3 of sodium aluminate soaking solution containing 190.2 g/l Na_2O and 78.4 g/l Al_2O_3. The resulting slurry is allowed to stand in a tank for a period of 30 minutes. The soaked bauxite is then ground to a grain size of about 25–30mm by a pair of crusher rolls arranged in the bottom of the tank. The slurry is continuously conveyed into another tank with a screw conveyor located below the first tank. 2 m^3 of soaking liquor of like composition is added to the second tank and the bauxite is kept standing for 30 minutes. During discharge from the second tank the slurry is ground to a grain size of about 10–15 mm by a pair of crusher rolls arranged at the outlet of the second tank. The crushed slurry is conveyed continuously with a screw conveyor located below the second tank into a Gorator grinding pump operating at the speed of 1,400 r.p.m. A quantity of soaking liquor is added continuously to the slurry so that the bauxite content of the slurry in the grinding pum is about 300 g/l.

The ground slurry is discharged from the Gorator pump under a pressure of 3.5 atm. into a hydrocyclone from which the bottom product (about 20 percent of the whole) is recycled to the grinding pump. According to a wet screen test of a sample of the slurry discharged from the dip tube of the hydrocyclone the screen residue is 5.8% on a screen with 315 microns nominal aperture and 7.8 percent on a screen having 200 micron nominal aperture.

EXAMPLE 4

Crude bauxite from Iszkaszentgyorgy, Hungary, having an Al_2O_3 content of 50.88 percent and a modulus of 7.51 is precrushed by bar roll breakers to about 20–50 mm average grain size and is then introduced into a tank provided with a stirrer. A sodium aluminate soaking liquor containing 193.4 g/l Na_2O and 80.1 g/l Al_2O_3 is added to the tank. Continuous feeding into, and discharge from, the tank is adjusted so that the soaking time of the bauxite is about 10 minutes. From below the outlet end of the tank the slurry of bauxite is pushed by a slurry pum through a 30 m long and 150 mm pipeline while additional amounts of soaking solution are added. At a flow rate of 2.8–3 m/sec the slurry contains 4 parts of liquor per part of bauxite. The

spends about 2 minutes in the pipeline. At the end of the pipe the slurry is fed into the receiving throat of a Gorator grinding pump (Type ZZQQH 25/15–15), operating at a speed of 1,000 r.p.m. A slit of about 1.5 mm is provided between the teeth of the rotor of the grinding pump and the teeth of a vertical segment of the housing thereof. The toothed segment located before the pressure end of the pump is densely perforated with 4 mm diameter openings. The slurry having an average particle size in the neighborhood of 1.5 mm as discharged through the pressure end of the Gorator grinding pump, is introduced into another Gorator grinding pum the slit width of which is adjusted to 0.6 mm. The particle size of the slurry solids exiting from the second pump is about 0.6 mm. The slurry is transported from the second grinding pump with a slurry pump through a hydrocyclone battery having a rubber lining. The cyclone bottoms are recirculated to the second Gorator grinding pump. A wet screen test on a sample taken from the dip tube of the hydrocyclone gives a 4.4 percent residue on a screen with a 315 micron nominal aperture and 6.3 percent on a screen with 200 micron nominal aperture.

EXAMPLE 5

Crude bauxite from Iszkaszentgyorgy, Hungary, having an alumina content of 50.88 percent and a modulus of 7.51 is precrushed by bar roll breakers to an average particle size of 20–50 mm. The crushed bauxite is introduced into a screw conveyor-mixer in which it is contacted with a sodium aluminate soaking solution containing 193.4 g/l Na_2O and 80.1 g/l Al_2O_3. The lumps of bauxite are thoroughly mixed with the solution and are allowed to stand 7–8 minutes. Thereafter the slurry is introduced into a Gorator grinding pump (Type ZZQQH 25/15–15) run at the rate of 1,000 r.p.m. and the bauxite/soaking liquor ratio is adjusted to about 1/3.5 by the addition of further amounts of liquor. The slit between the rotating rotor disc of the grinding pump and the teeth of the vertical segment on the housing is about 2 mm. The slurry is discharged from the pressure end of the grinding pump and has an average particle size of about 2 mm. The slurry is introduced into a 300 m long ring duct having a diameter of 150 mm with the assistance of a slurry pump. The slurry circulates for a period of 40 minutes which corresponds to a flow rate of 3 m/sec and a recirculation number of 24, or a transportation distance of 10 kilimeters. A wet screen test on samples taken after a circulation period of 40 minutes results in a residue of 5 percent on a screen with 315 micron nominal aperture size and 6.1 percent on a screen having 200 micron nominal aperture size.

EXAMPLE 6

6 metric tons of bauxite from Nyirad, Hungary, having an Al_2O_3 content of 49.5 percent, a modulus of 7.6 and a maximum average particle size of about 20 mm, are mixed with 4.5 m^3 of alkaline soaking solution containing 6.2 g/l Na_2O, at about 100°, and is allowed to soak for a period of 24 hours. The slurry is thereafter introduced into a Gorator grinding pump (Type 25/15–15) operated at a rate of 1,400 r.p.m., and provided with an elliptical toothed rotor and toothed segments. Together with the slurry and additional amount of alkaline solution containing 258 g/l Na_2O, is added to the grinding pump, so that the solids content of the slurry exiting from the pump is about 300 g/l, and the Na_2O content of the slurry is about 190 g/l. The toothed segment located in advance of the pressure end of the grinding pump is provided with 6 mm diameter perforations. The slurry output from the grinding pump is introduced into a second grinding pump of the same type and operated at the same speed, but the gap between the toothed rotor and the toothed segments is smaller than in the first grinding pump and the segment before the pressure end is provided with perforations having a 3 mm diameter. The slurry exiting from the second grinding pump, after a wet screen test leaves a 2 percent residue on a screen with 315 micron apertures and 3.9 percent on a sieve with 200 micron apertures.

EXAMPLE 7

An intermediate product from the manufacture of aluminum, containing 18.8% SiO_2, 16.7% Al_2O_3, 3.2% Fe_2O_3, 58.4% CaO and 2.9% other components, the alumina content of which is largely in the form of calcium aluminates, and has an average particle size of about 20 mm, is soaked for 24 hours in a solution containing 50 g/l Na_2O. The 2 tons of intermediate are slowly stirred in 4 m^3 of solution at 75°. The slurry is then introduced into a Gorator grinding pump (Type 25/15–15) having a 2 mm gap between the toothed rotor and the toothed segments on the housing. The grinding pump is operated at a rate of 1,400 r.p.m. The slurry exiting from the grinding pump is introduced into a second grinding pump which has only a 1 mm gap. The solids in the slurry obtained from the second grinding pump show a 6 percent residue in a wet screen test on a sieve with 315 micron apertures.

EXAMPLE 8

A calcined intermediate product in the manufacture of aluminum, containing 34.6% Al_2O_3, 45.3% SiO_2, having a maximum average particle size of about 25 mm is soaked for 24 hours during slow stirring in a nitric acid solution. 2.1 tons of material are employed with 4 m^3 of the solution. The slurry containing the softened particles is introduced into a Gorator grinding pump (Type 25/15–15) provided with an acid-resistant steel lining. The gap between the toothed rotor and the segments of the housing is 2 mm and the pump is run at a speed of 1,400 r.p.m. The slurry output of the grinding pump is introduced into a second grinding pump in which the gap is adjusted to 0.8 mm. The slurry from the second grinding pump is classified in a hydrocyclone and a sample obtained from the dip tube when subjected to wet sieve analysis has a residue of 5.2 percent on a sieve with 200 micron apertures.

We claim:

1. A process for comminution of particulate aluminaceous starting materials, which comprises before or after coarse crushing soaking said starting material in an alkaline or acidic electrolyte for a period of from about 0.2 to about 24 hours thereby to loosen the structure of the particles, and completing the comminution by subjecting the particulate material to predominantly shearing forces until the average particle size of the material is reduced to under 1 mm.

2. The process of claim 1, wherein the average particle size of the starting material is from about 10 mm to about 30 mm, and said starting material is either a pre-

crushed ore or an intermediate in the manufacture of aluminum.

3. The process of claim 1, wherein said step of soaking is carried out in a pipeline while the starting material is being transported from one processing location to another.

4. The process of claim 2, wherein the maximum temperature of the soaking electrolyte is about 120°C.

5. The process of claim 1, wherein said step of completing the comminution is carried out with a grinding pump.

6. The process of claim 2, wherein said step of completing the comminution is carried out with a grinding pump.

7. The process of claim 2, wherein the starting material is a bauxite having an average particle size of from about 20 to about 30 mm, and wherein said step of soaking is carried out for a period from about 0.5 to about 2.5 hours in a caustic sodium aluminate solution.

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