PATENT SPECIFICATION

(11) 1 564 005

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(21) Application No. 31297/76 (22) Filed 27 July 1976
(31) Convention Application No. 2533755
(32) Filed 29 July 1975 in
(33) Federal Republic of Germany (DE)
(44) Complete Specification published 2 April 1980
(51) INT CL³ C22B 3/00 B01F 11/00//C01G 9/06
(52) Index at acceptance
C7D 16 17
B1C 10 18E1 18E2A 29 5
B1F 4F 4G 4L
B1X 17
C1A D45 G39 G39D45 P22

(19) THE PATENT OFFICE LONDON

(54) A METHOD AND A DEVICE FOR CARRYING OUT WET METALLURGICAL PROCESSES

(71) We, KLOCKNER-HUMBOLDT-DEUTZ AKTIENGESELLSCHAFT of Deutz-Mulheimer-Strasse 111, 5 Koln 80, Federal Republic of Germany, a German Body Corporate, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The invention relates to a method for carrying out wet metallurgical processes with at least a gaseous reactant in an enclosed container.

Furthermore, the invention relates to a device for carrying out the method.

Wet metallurgical processes play an important part in the treatment of ores during extraction of metals. The method steps which take place are mixing together making suspensions, milling, reaction together as well interchange between materials and heat exchange of solid, liquid and gaseous phases. Among other things, precipitation of metals such as copper or nickel by means of hydrogen is known, particularly in accordance with the equation:

$$Cu^{2}+H_2 \rightarrow Cu^{\circ}\downarrow+2H^+$$

Precipitation of a compound (metallic salt) takes place for example in accordance with the equation:

$$H_2S+Cu^+ \rightarrow Cu_2S\downarrow+2H^+$$

furthermore, oxidation processes, which may take place for example in accordance with the following equations:

$$2H^++Fe^{2+}+\tfrac{1}{2}O_2 \rightarrow Fe^{3+}+H_2O$$

$$ZnS+H_2SO_4+\tfrac{1}{2}O_2 \rightarrow ZnSO_4+S\downarrow+H_2O$$

are of importance.

And finally, frequently metallurgical processes contain solution processes for which the following formula may be an example:

$$ZnO+H_2SO_4 \rightarrow ZnSO_4+H_2O$$

In some of these processes, pressures of 10 to 100 bar are used as well as normal to high and very high temperatures. The problem when carrying out these methods lies in bringing the reactants in varying physical conditions, e.g. liquid/gaseous or solid/liquid/gaseous, into intimate contact. This is very important because for example as during oxidation, the speed of diffusion of the gas (oxygen) in the fluid is the factor determining the speed of the reaction process. The same is true also for diffusion of hydrogen gas during precipitation processes or for mixed or reducing gases during reduction processes.

For this reason, the use of pulsating agitation known *per se* is proposed in German Auslegeschrift No. 1 038 762 for the purpose of leaching sulphide ores. However, this requires an electromagnetic drive system and an agitation element made of corrosion-resistant material. The total cost is not inconsiderable and moreover the device is relatively sensitive to the conditions of a decidedly rough operation, while the efficiency on the other hand still leaves something to be desired.

Another principle made use of in technology, for example during leaching under pressure, was developed by the Sherrit-Gordon Company. In this, the oxygen is distributed by propellers in the electrolyte in large, temperature controllable stirring apparatus autoclaves. However, this process is dependent, and thus limited, by the speed of agitation of the propellers. In another method of smelting complex sulphide ores in a wet process it was proposed in German Patent

Specification No. 888 929 to circulate a flow of oxygen through a suspension of ore.

Furthermore, it is known to use a vessel filled with steel balls—called an attritor—for leaching copper pyrites concentrates, wherein the balls are moved through a central agitator. The gas introduced into the electrolyte via the agitator is to be broken up into a fine diffusion. Disadvantages of this are a high power requirement with a correspondingly high wear for a relatively low throughput and these disadvantages have previously hindered its use on a large scale. Furthermore, the so-called Pachuca tank for leaching by agitation is known in which revolution of the gas/liquid takes place according to the principle of the air lift pump, while, with a vane wheel stirring apparatus, an agitator is arranged in a central conduit, which brings about mixing of the gas with the liquid.

Limits are set qualitatively or quantitatively on all of these known devices as the result of their mode of operation or they will fail under high pressures and/or temperatures.

The difficulties in some electrochemical reactions are different—such as in production of copper from copper sulphate by means of an exchange of electrons with iron—wherein, during reaction, covering layers are formed on the reactant which cause passivation whereby the diffusion processes are hindered or, in extreme cases, are brought to a standstill.

In order to remedy this difficulty, reaction milling is known, for example in a ball mill. This has the advantage that on the one hand considerably shorter milling times are required in order to reach a certain grade of milling as a result of leaching of the solid, on the other hand, during leaching the leaching times are considerably reduced because in each case fresh and reactable areas come into contact with the solvent as a result of milling.

This technique relates to solvent processes in which a gaseous reactant is not required. In contrast to this, however, the invention relates to those metallurgical processes in which the gas phase is reacted with liquid or liquid and solid reactants.

Thus, for the invention, the rate of solution or suspension of a gas in a liquid is the factor determining the speed for the complete reaction.

In order to make gases form a solution in liquids and to allow reaction with the finely distributed matter or the matter dissolved in the liquid, then, in a preferential manner, the following considerations must be taken into consideration:

1. The gas must be present in the liquid in a very finely divided form as a quasi emulsion in order to present as large a surface as possible to this liquid.

2. An increase in the pressure causes an increase in the solubility and a reduction of the volume of gas in the liquid.

3. An increase in the temperature causes, on the one hand, a reduction in the solubility of the gas in the liquid and, on the other hand, causes an increase in the readiness of the dissolved or finely divided solid matter to react with the gas.

All previously mentioned known systems for intimately mixing gases with a liquid, i.e. injection on the one hand or stirring in on the other hand, only achieve their aim partially:—

Injecting gases is limited by a low mixing effect, moreover the size of the gas bubbles is dependent on the cross-section of the injection nozzles used and on the pressure at which the gas is injected. Even in the stirring system, the mixing effect of the gas with the liquid remains limited by the formation of a concave depression during stirring so that, as is known, an increase in the stirring speed does not produce any improvement in the distribution of gas from a certain limit onwards.

The invention seeks to improve on known methods for carrying out wet metallurgical processes both as regards quality and throughput and to enable this to be carried out economically.

According to a first aspect of the invention there is provided a method of carrying out wet metallurgical processes comprising passing the reactants including both a gas and a liquid into an enclosed container with a horizontal axis and oscillating the container energetically in a plane at right angles to the horizontal axis at a frequency greater than 15 Hertz and at an amplitude greater than 8 mm.

An advantageous refinement of the method may be characterized by an almost circular oscillation with the axis of the container substantially describing the surface of a horizontal cylinder.

However, an almost ellipsoidal oscillation may be used, with the axis of the container substantially describing the surface of a horizontal body having an elliptical cross-section.

The oscillation of the container may however also take place as an almost linear oscillation, with the movement of the axis of the container taking place substantially in a vertical plane.

A particularly favourable intensive effect may be given by using freely movable mixing bodies in the container. These serve to provide a strengthened transmission of the oscillation pulses to the liquid and at their edges and surfaces produce a plurality of local increases in speed and turbulences in the liquid.

In a further useful refinement of the invention, the mixing bodies may, at the same time, be milling bodies for crushing the solid.

Attempts at carrying out a crushing process while at the same time the gaseous and liquid phases of the reactive mixture are mixed intimately together and emulsified, show a result which must be regarded as a surprising effect of this particular combination of methods which goes far beyond the summed effect of the two individual measures. The mixing or milling bodies may comprise, for example, ceramics, if they are not intended to take part in the reaction materially, but they can also be used to exercise a certain effect, for example a catalytic effect, at the same time, if, as is proposed in German Auslegeschrift 1 203 746, iron, for example in the form of a ferrous or ferric compound, is mixed with a solution for the purpose of achieving a more intensive reaction.

In other reaction mechanisms, the continuous crushing process prevents possible coating of the surface of the solid with passivating layers, whether the layer is formed by a layer of gas as in zinc leaching or a sulphur coating as when leaching sulphidic metal ores. Furthermore, the fact that short-term high accelerations in different directions of the solid bodies—container wall/milling body/surfaces of the solid—moved rapidly relative to each other lead to compression and decompression at the interface of the gaseous/liquid reactants still plays a part, whereby the transport of material and thus the reaction is very greatly intensified.

In a further useful refinement of the method of the invention, the wet metallurgical process, beginning with low pressure and/or temperature, may be conducted at high pressure and/or high temperature.

Moreover, it is possible and/or advisable according to the type of process, for the wet metallurgical process to be carried out in batches.

With other reactions, on the other hand, it may be an advantage for the reactants to be passed continuously through the container.

A particularly useful realization of the process, which is qualitatively and quantitatively the optimum, may be produced with the method according to the invention in which such a process is carried out in stages alternately in an oscillating container and a stationary reaction vessel connected to the output side of the oscillating container. In such a stationary vessel, the reactive suspensions produced in the oscillating container and made up of reactive gases and/or solid materials may continue to react further with the liquid. This stationary reaction vessel may either comprise a simple pipe (pressure pipe), an agitator container, or an autoclave, or even a simple container. The essential factor is that a device for preventing separation of gas from the suspension is present and this remains effective at least until the reaction comes to a standstill whether as a result of using up the reactive gas or by using up the reactive material distributed finely or dissolved in the liquid. Thus, by connecting several oscillating containers one after the other in stages each followed by a stationary reaction vessel, continuous reaction may take place with concurrent flow or counter-flow of the individual reactants. The advantage lies in the fact that, as opposed to leaching processes according to conventional methods, the times for total reaction are considerably reduced because, by the repeated crushing and emulsifying processes, fresh reactable solid surfaces are brought into contact with the reactive gas/fluid emulsion or solution in the individual stages.

According to a second aspect of the invention, there is provided a device for carrying out wet metallurgical processes comprising an enclosed container with a horizontal axis for receiving the reactants and means for energetically oscillating the container in a plane at right angles to the horizontal axis at a frequency greater than 15 Hertz and at an amplitude greater than 8 mm. The container may be set in oscillation by means of an imbalance or eccentric drive.

Such a container may have either a round or polygonal cross-section or may even have an ellipsoidal cross-section.

In an advantageous manner the oscillating container may also be a ball mill known *per se*.

In many cases, it may prove necessary for the container to have a device for passing a heat carrying medium into contact with the container wall.

In many cases it is useful, in order to avoid damage due to corrosion, for the container to be manufactured with corrosion-resistant material or at least to be coated therewith.

And finally, the device for carrying out the method in stages may be characterized by use of at least one ball mill followed by at least one stationary reaction vessel.

In metallurgy and in chemical technology a large number of applications arise from the described method.

Above all, O_2, H_2, SO_2, Cl_2, NO_2, CO should be mentioned as reactive gases.

Mixtures of gases may also be used, and, in particular, air.

With hydro and wet metallurgical extraction of metals, aqueous solvents are used as liquids, for example liquid ion exchange media such as acids of a mineral, inorganic or organic nature, moreover inorganic salt solutions or organic solvents or basic lyes may be used.

Raw, dressed or metallurgically pretreated ores come into consideration as solid materials, for example materials to be leached, as well as metallurgical intermediate products for example slag, stone, speiss, flue dust, sublimate or a product of precipitation. Sulphidic mineral materials are named as an example of this, wherein this treatment can be undertaken without previous roasting and thus without converting sulphur into gaseous form or into metal sulphate, in that the metals are brought directly to a solution by means of leaching or electrolysis, while the sulphur is obtained as far as possible as an element. This type of use of the process is not only economically favourable but, because the gaseous sulphur compound and the dust are missing, it is also particularly favourable to the environment.

The invention will now be described in greater detail, by way of example, with reference to the drawings, in which:—

Fig. 1 shows a flow chart for precipitation of metal from a salt solution by means of hydrogen gas;

Fig. 2 shows in flow sheet form precipitation of copper by means of hydrogen gas;

Fig. 3 shows the principle of pressure leaching of zinc blende in an electrolyte with the aid of oxygen gas;

Fig. 4 shows the same reaction mechanism in flow sheet form in two stages, in the ball mill with the reaction vessel connected thereafter; and

Fig. 5 shows the time/concentration diagram of leaching in six stages—with a ball mill and stationary reaction vessel.

The flow chart of Figure 1 shows the supply and formation of a solution 1 comprising copper sulphate, sulphuric acid and water. This solution is fed to the pressure pump 2 which conveys it, for example at a pressure of 10 atmospheres, into an oscillatory container 3. In addition, hydrogen gas is blown under pressure into the oscillatory container 3, whereby the process of precipitation, in accordance with the formula stated in the Figure, is set in motion. The oscillatory container 3 is set in very energetic oscillation by means of an out-of-balance drive, not shown, for example at 16 2/3 Hertz at an amplitude of 10 mm. As a result, the liquid is so intensively atomized in the gas chamber that an emulsion-like mixture of both phases results. The intimate mixing of the gaseous reactant hydrogen with the solution causes a reaction course of a high degree of efficiency and exceptionally high speed whereby a continuous mode of operation is made possible at an economical cost. From the oscillatory container 3, the reaction product reaches the pressure reducer 4 which has the object of bringing about the lowering of pressure from the reaction pressure to normal pressure. From here, the fluid mixture may drain away into the decanter 5 which concentrates the precipitation product to form a slurry while the precipitating solvent is passed off for dressing and re-use. The extracted copper slurry is further dehydrated in known manner by means of filtration and is subjected, also in known manner and therefore in a manner not shown, to a further metallurgical refining process.

Figure 2 shows the same process in the form of a flow sheet for the purpose of improved clarity. In this, numeral 21 designates the feed station with the mixing vessel, into which the solution according to the formula is passed and adjusted. The pressure pump 22 conveys the solution into the oscillatory container 23 and thus increases the pressure to the required level for the reaction. The oscillatory container 23 in the example shown has two chambers 27, 27′ which are arranged horizontally and are in connection with one another. These chambers are mounted at 28, 28′ so as to be capable of oscillation. A rotating imbalance 29 sets the oscillating system into circular oscillating movement. Two heat exchangers are shown symbolically at 26, 26′ and these make it possible to supply or remove heat from the oscillatory container and its contents. From the oscillatory container 23 in which the reaction is completed in a manner already mentioned, the reaction product reaches the pressure reducer 24 and thereafter the decanter 25 wherein the copper slurry is separated from the solvent.

Figure 3 shows the principle of pressure leaching of zinc blende as a flow sheet. In the mash station 31, the electrolyte, coming from electrolysis, is made into a suspension with the concentrate and additions of Fe_2O_3 to form a slurry. The concentrate consists of zinc sulphide and other metal sulphides such as copper sulphide or iron sulphides (indicated in Figure 3 as "Me S"). The slurry is directly preheated by introduction of steam or even indirectly by means of a heat exchanger. From the mash station 31 the slurry thus prepared reaches the pumping station 32. This takes over setting of the required pressure and transports the material into the oscillatory container 33, in the present case a ball mill.

The ball mill 33 serves in the present case as a mixing machine for mixing suspension of the electrolyte and the concentrate with oxygen. In the mill, however, the or concentrate is ground to the final size required, while at the same time the reaction starts. Thus, as a result of the reaction heat, the suspension is brought to the maximum reaction temperature, which is above the melting point of sulphur, whilst still in the mill. When maintaining a constant pressure, the oxygen, which has been divided up finely in the electrolyte-concentrate mixture, is constantly redissolved and consumed by the reaction and brought against the reaction material. At the same time, the oscillations at a high energy density cause a short-term sequence of compression and decompression processes where, as a result of rapid local fluid and material movements, similar to pumping processes, the reacted material product is transported away from the point of reaction. This effect also makes it possible that, when leaching above the melting point of sulphur, particles of sulphur which are produced pass into the solution in suspension so that agglomeration with unleached zinc blende particles is prevented. In the present case, the mixing and milling bodies may comprise iron because their friction or corrosion products are oxidized to form ferrous and/or ferric compounds which, as is known, are suitable as catalysts for zinc blende leaching. The container of the ball mill itself, because of its simple formation as a tube, may be protected from corrosion by packing with plastics or manufacturing from acid-resistant steels for example high-alloy steels such as chrome-nickel-steel. Even the problems of sealing are easily handled as there are no rotating parts and thus no glands.

The partially reacted intermediate product reaches the stationary reaction vessel 34 from the ball mill 33. This may, in known manner, be provided with a stirring apparatus, which counteracts separation of the reactants. The reaction temperature may also be maintained or changed in the reaction vessel 34 in a simple manner by means of heat exchangers. In this manner, the reaction may be controlled until it is slowed down considerably or brought to a standstill after the reaction materials have been consumed or as a result of agglomeration of the particles of sulphur.

In addition, the slurry in the reaction vessel 34 may be cooled to a temperature below the melting point of sulphur in order to pass through a second treatment phase in the second oscillatory container 33′ which may also take the form of a ball mill. However, the slurry may also reach the ball mill 33′ directly and uncooled in order to permit the processes described to be carried out. It is essential, in any case, when several stages are arranged one behind the other, that the concentrates should be crushed again to ensure that the fluid sulphur envelope of the concentrate is separated off and the sulphur passes in suspension into the solution. Obviously, it is always of great importance in the complete reaction for a suspension to be formed between the oxygen gas and the slurry which is activated sufficiently far to achieve the next degree of reaction in the following stationary reaction vessel.

Pressure leaching of sulphidic materials in several or many stages as proposed by the invention produces an extraordinarily economical complete process having high throughputs wherein, in one stage, the consumed quantity of electrolyte and oxygen gas may be complemented to such an extent that the concentrate is almost completely leached.

The leaching in stages shown in Figure 3 has been taken as a random example, however it corresponds in principle to practice. Leaching is carried out, for example, in 3 stages providing 15 minutes complete leaching time respectively. Thus 4½ minutes are cut from leaching and treatment in the oscillatory containers 33, 33′, 33″ and 10½ minutes are cut from the period in the reaction vessels 34, 34′, 34″. It was found that during leaching in the ball mill, high reaction speeds occur in which, if the solubility of the zinc is shown as a curve in dependence on time, an almost linear increase is obtained as is shown by C in Figure 5. In subsequent reaction in the stationary reaction vessel, the reaction speed is severely reduced or even brought to a standstill by the molten sulphur as a result of moistening of unreacted blende particles as indicated by D in Figure 5.

In Figure 5, the curve A shows the previous course in conventional agitator autoclaves while carrying out reduction below the melting point of sulphur, while curve B makes clear the leaching already described, but in 6 stages in a ball/mill reaction vessel arrangement.

From the last stage or the reaction vessel 34″ in accordance with Figure 3 the slurry reaches the pressure reducer 35 and from there reaches the decanter 36. The latter extracts zinc sulphate in the overflow while the fed elementary sulphur is concentrated from the suspension in the residue and, together with impure metals (for example lead sulphate, cadmium sulphate etc. indicated in Figure 3 as "$MeSO_4$") the gangue is removed as a sediment.

In Figure 4 in repetition of the same method principle, the flow chart is again shown only for a two-stage plant for the purpose of a better overall view. Here, 41 designates the mash vessel with the agitator 42. The pump station is designated 43 and a first ball mill 44, and the reaction vessel 45 with the agitator 46. The second ball mill 44′ passes the slurry to a second reaction vessel 45′ which is of the same construction as the vessel 45. From there, the slurry reaches the pressure reducer 46 and finally the decanter 47.

WHAT WE CLAIM IS:—

1. A method of carrying out wet metallurgical processes comprising passing the reactants including both a gas and a liquid into an enclosed container with a horizontal axis and oscillating the container energetically in a plane at right angles to the horizontal axis at a frequency greater than 15 Hertz and at an amplitude greater than 8 mm.

2. A method according to claim 1, wherein an almost circular oscillation is produced with the axis of the container substantially describing the surface of a horizontal cylinder.

3. A method according to claim 1, wherein an almost ellipsoidal oscillation is produced with the axis of the container substantially describing the surface of a horizontal body having an elliptical cross-section.

4. A method according to claim 1, wherein an almost linear oscillation is produced with the movement of the container axis taking place substantially in a vertical plane.

5. A method according to any one of claims 1 to 4, wherein freely movable mixing bodies are used in the container.

6. A method according to claim 5, wherein the mixing bodies are at the same time milling bodies for crushing a solid.

7. A method according to any one of claims 1 to 6, wherein the wet metallurgical process, which begins at low pressure and/or temperature, is carried out at high pressure and/or high temperature.

8. A method according to any one of claims 1 to 7, wherein the wet metallurgical process is carried out in batches.

9. A method according to any one of claims 1 to 7, wherein the reactants are continuously passed through the container.

10. A method according to any one of claims 1 to 9, wherein the metallurgical process is carried out in stages, alternately in oscillating containers and in stationary containers.

11. A device for carrying out wet metallurgical processes comprising an enclosed container with a horizontal axis for receiving the reactants and means for energetically oscillating the container in a plane at right angles to the horizontal axis at a frequency greater than 15 Hertz and at an amplitude greater than 8 mm.

12. A device as claimed in claim 11, wherein an imbalance or eccentric drive is provided for oscillating the container.

13. A device according to claim 12, wherein the container has a round or polygonal cross-section.

14. A device according to claim 12, wherein the container has an ellipsoidal cross-section.

15. A device according to any one of claims 11 to 14, wherein the oscillating container is a ball mill.

16. A device according to any one of claims 11 to 15, wherein the container has an arrangement for passing a heat carrying medium into contact with the container wall.

17. A device according to any one of claims 11 to 16, wherein the container is manufactured with corrosion-resistant material or is at least coated with corrosion-resistant material.

18. A device according to claim 15, wherein a stationary reaction vessel is connected after at least one ball mill.

19. A method of carrying out wet metallurgical processes according to claim 1 and substantially as described herein with reference to the drawings.

20. A device for carrying out metallurgical processes according to claim 11 and substantially as described herein with reference to the drawings.

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Printed for Her Majesty's Stationery Office, by the Courier Press, Leamington Spa, 1980
Published by The Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.

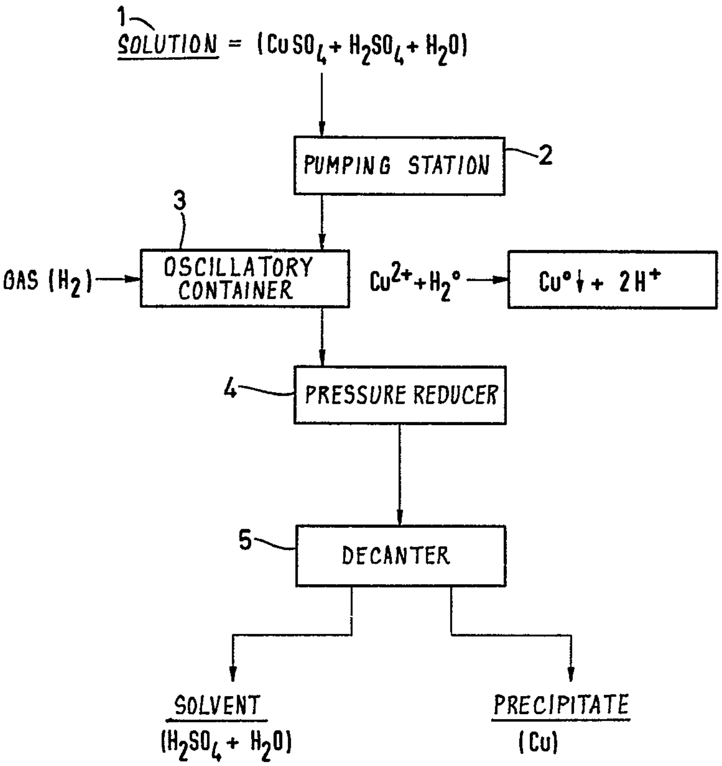

1
SOLUTION = $(CuSO_4 + H_2SO_4 + H_2O)$
PUMPING STATION
2
3
GAS (H_2)
OSCILLATORY CONTAINER
$Cu^{2+} + H_2^{\circ}$
$Cu^{\circ}\downarrow + 2H^{+}$
4
PRESSURE REDUCER
5
DECANTER
SOLVENT
$(H_2SO_4 + H_2O)$
PRECIPITATE
(Cu)

FIG. 1

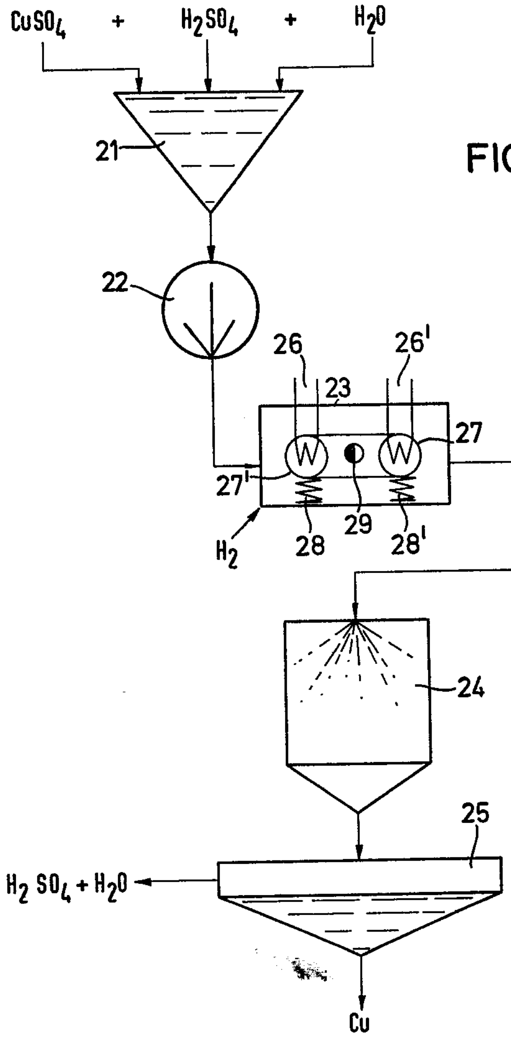


FIG. 2

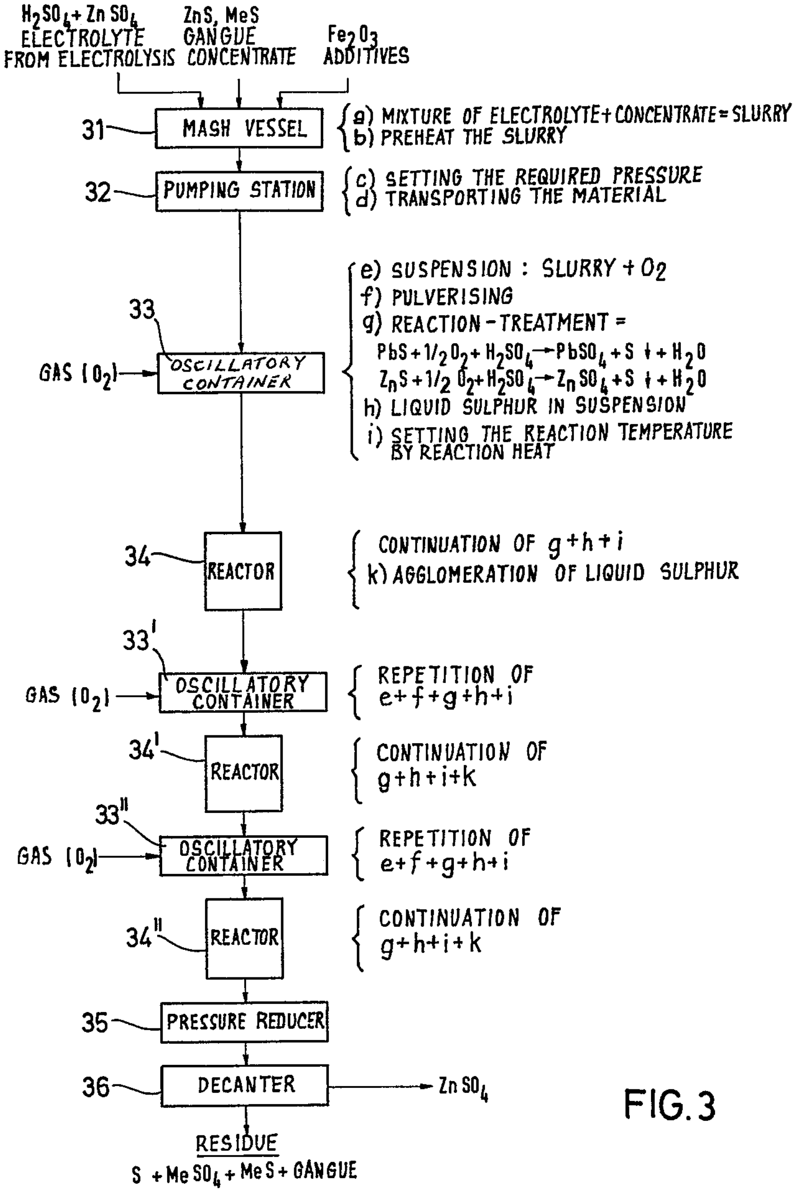

$H_2SO_4 + ZnSO_4$ ELECTROLYTE FROM ELECTROLYSIS
ZnS, MeS GANGUE CONCENTRATE
Fe_2O_3 ADDITIVES
31
MASH VESSEL
a) MIXTURE OF ELECTROLYTE+CONCENTRATE=SLURRY
b) PREHEAT THE SLURRY
32
PUMPING STATION
c) SETTING THE REQUIRED PRESSURE
d) TRANSPORTING THE MATERIAL
33
GAS (O_2)
OSCILLATORY CONTAINER
e) SUSPENSION : SLURRY + O_2
f) PULVERISING
g) REACTION-TREATMENT =
$PbS + 1/2\,O_2 + H_2SO_4 \rightarrow PbSO_4 + S \downarrow + H_2O$
$ZnS + 1/2\,O_2 + H_2SO_4 \rightarrow ZnSO_4 + S \downarrow + H_2O$
h) LIQUID SULPHUR IN SUSPENSION
i) SETTING THE REACTION TEMPERATURE BY REACTION HEAT
34
REACTOR
CONTINUATION OF g+h+i
k) AGGLOMERATION OF LIQUID SULPHUR
33'
GAS (O_2)
OSCILLATORY CONTAINER
REPETITION OF e+f+g+h+i
34'
REACTOR
CONTINUATION OF g+h+i+k
33''
GAS (O_2)
OSCILLATORY CONTAINER
REPETITION OF e+f+g+h+i
34''
REACTOR
CONTINUATION OF g+h+i+k
35
PRESSURE REDUCER
36
DECANTER
$ZnSO_4$
RESIDUE
$S + MeSO_4 + MeS$ + GANGUE

FIG. 3

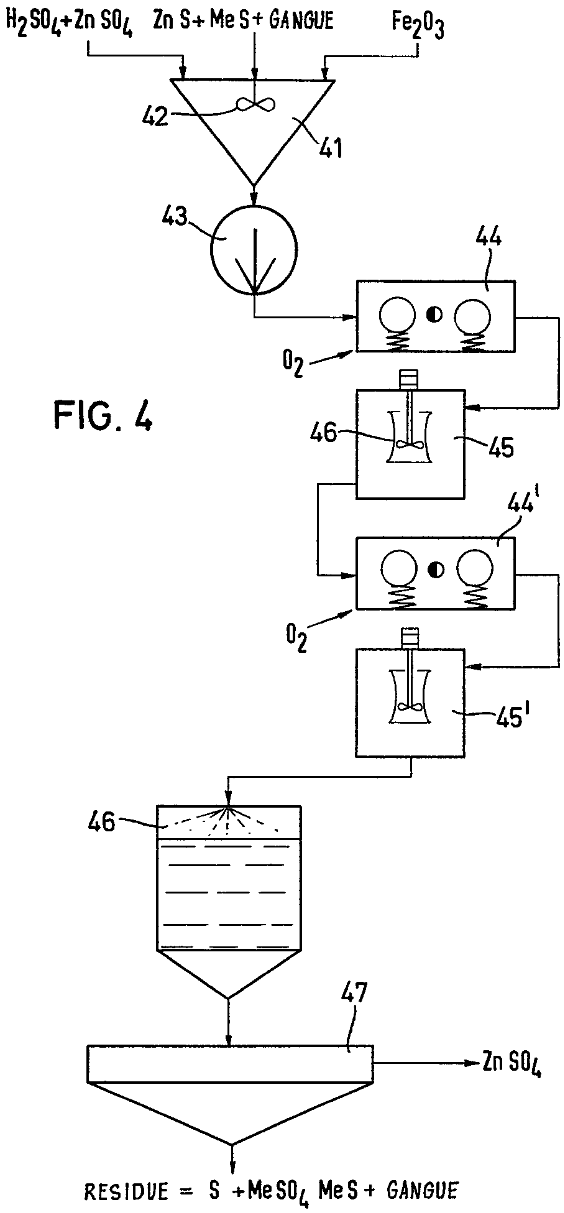
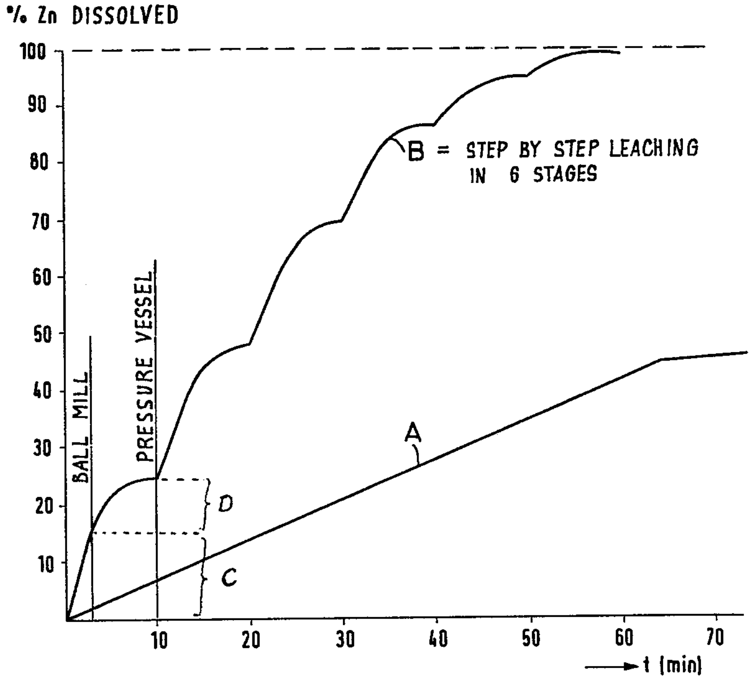

H_2SO_4+Zn SO_4
Zn S+ Me S+ GANGUE
Fe_2O_3
42
41
43
44
O_2
46
45
44'
O_2
45'
46
47
Zn SO_4
RESIDUE = S +MeSO_4 Me S + GANGUE

FIG. 4

FIG. 5


% Zn DISSOLVED
100
90
80
70
60
50
40
30
20
10
BALL MILL
PRESSURE VESSEL
B = STEP BY STEP LEACHING IN 6 STAGES
A
D
C
10
20
30
40
50
60
70
t (min)