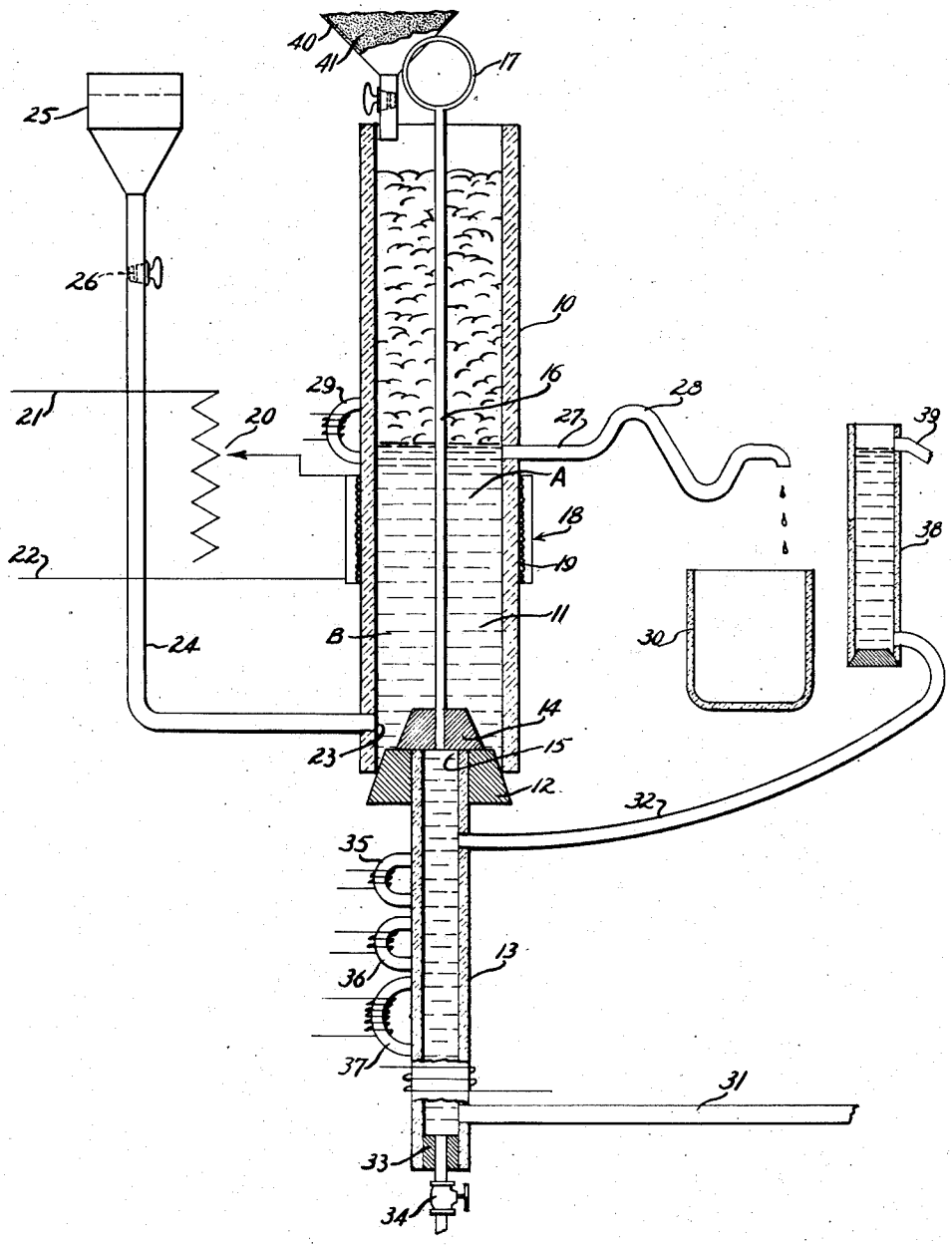
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CONTINUOUS PROCESS FOR LEACHING AN IRON-ALUMINUM ALLOY

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CONTINUOUS PROCESS FOR LEACHING AN IRON-ALUMINUM ALLOY

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This invention relates to a continuous leaching process, and more particularly to a continuous process for the leaching of iron-aluminum alloys to produce a finely divided residue having permanent magnet properties and consisting largely of well dispersed, finely divided Fe_3O_4 in a matrix of oxyferrite.

In order to develop the highest possible magnetic properties in the material prepared in leaching iron-aluminum alloys, I have found it desirable to limit the time of contact between the particles of the iron-aluminum alloy and the leachant, which is preferably an aqueous solution of a caustic alkali, such as sodium hydroxide. The control of contact time to a minimum can best be accomplished in a continuous process in which the finely divided particles of the iron-aluminum alloy are charged into the top of a vertical column of the leachant and maintaining the top portion of the column at or near its boiling point, while keeping the lower part of the column relatively cool. The alloy particles react vigorously upon coming into contact with the hot leachant, but when the reaction has gone to completion, the particles fall by gravity through the hot portion of the column into the relatively cool caustic alkali solution. Since there is an evolution of hydrogen from the reaction between the alloy particles and the hot caustic alkali solution, there is a tendency of the alloy particles to be buoyed up until the completion of the reaction. Consequently, each alloy particle, independently of its size within certain limits, remains in the hot leachant until the completion of the reaction and then automatically falls by gravity into the relatively cold portion of the column. In this way, the time of contact with the hot leachant is limited to that required for complete reaction. The leaching reaction is complete when the leached particles enter the cold caustic solution zone. Due to the lack of gas evolution in this cold zone, the leached particles pass through the zone without substantial hinderance.

From the relatively cool lower portion of the column, the leached alloy particles are either continuously or periodically transferred into a countercurrent washing system. The washing removes substantially all of the water soluble products of reaction which in the case of iron-aluminum alloys leached with caustic soda would be principally sodium aluminate. By the use of the countercurrent washing system, the leached particles are in contact with the washing solutions for the shortest length of time consistent with substantially complete removal of by-products formed

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during the leaching operation. In this way oxidation and loss of magnetic properties of the leached product is avoided. From the washing system, the washed, leached particles are collected for further processing in the manufacture of permanent magnet material from such particles.

It is therefore an important object of this invention to provide a continuous leaching process wherein the time of leaching is automatically regulated in accordance with the time required for completion of the reaction that occurs between the particles to be leached and the leachant.

It is a further important object of this invention to provide a process for the continuous leaching of iron-aluminum alloys by means of aqueous caustic alkali solutions, wherein the alloy in finely divided particle form is first introduced into the upper, heated portion of a column of the leachant for reaction there and for gravity discharge through a relatively cool lower portion of the leachant, whereby the particles remain in contact with the hot leachant until the reaction is substantially completed and then automatically drop by gravity into the cool lower portion of the column, from which the leached particles may be discharged into a countercurrent washing system.

Other and further important objects of this invention will be apparent from the disclosures in the specification and the accompanying drawings.

On the drawings, the figure is a vertical sectional view, partly in elevation, of apparatus suitable for carrying out the process of my invention.

The reference numeral 10 indicates an elongated cylindrical vessel arranged vertically for confining a column of leachant, indicated at 11. The vessel 10 may be open at both ends and provided at its lower end with an apertured closure member 12, into which is inserted the upper end of a second, relatively smaller diameter cylindrical vessel 13. A gravity acting valve 14 serves to close the upper end 15 of the lower cylindrical vessel 13 and thus also close the lower end of the upper cylindrical vessel 10. A stem 16 extends from the valve 14 to a point above the upper end of the upper vessel 10, where a loop 17 is provided for manual manipulation of said valve 14.

The upper vessel 10 is provided intermediate its height with means, indicated generally at 18, for heating the leachant in the portion of the column indicated at A. Said means 18 may suitably comprise an electric heater, the coils 19 of which are wound around the vessel 10 and are connected through a rheostat 20 with power lines 21 and 22 leading from a suitable source of cur-

rent, such as A. C. at 110 volts. When the heater 18 is in operation, it serves to bring the leachant in the portion A of the column to a temperature at or near its boiling point. The portion of the leachant in the lower portion B of said column, however, is to be maintained at a relatively cool temperature, such as around room temperature.

In order to maintain the lower part B of the column at a relatively low temperature, cold leachant is introduced into the bottom of said vessel 10, as at 23, through a tube 24 connected to a reservoir 25 and provided with a petcock 26 for the regulation of flow of leachant into said lower portion B of the column. The rate of flow is so regulated as to maintain the height of the column of leachant in the vessel 10 substantially constant. A tube 27 inserted into the side wall of the vessel 10 just above the heating unit 18 serves to draw off spent leachant at a rate proportional to the rate of consumption of the active ingredients in the leachant. Said tube 27 is provided with an upwardly offset portion 28 to retard the passage of particles of the alloy along with the spent leachant. As a further means of preventing excessive loss of alloy particles through the side drain 27, an electromagnet 29 is positioned on the opposite wall of said vessel 10 to attract the magnetic particles away from the opening into the side tube 27. The spent leachant passing through the side tube 27 discharges into a vessel 30.

The lower cylindrical vessel 13 serves for the washing of the leached particles and for this purpose is provided with a water intake 31 near the bottom thereof and a water outlet 32 near the top thereof.

The lower end of the cylindrical vessel 13 is provided with a plug 33 and a valved outlet therethrough 34, through which the leached and washed particles may be withdrawn from the system. When the valve 14 is lifted from the upper end 15 of the vessel 13 leached particles are discharged into said lower vessel 13 for countercurrent washing by means of the upward flowing stream of water introduced through the inlet 31. In order to assist the washing operation, electromagnets, indicated at 35, 36 and 37, may be positioned along the wall of the vessel 13 to effect magnetic agitation of the particles as they fall downwardly through said vessel 13. In this way much of the water soluble products of reaction is removed from the leached particles as they fall through the upwardly flowing stream of water in the vessel 13.

The head of water maintained in the vessel 13 is controlled by means of a vertically adjustable tube 38 connected to the outlet tube 32 and provided with an overflow 39 near its upper end. By adjusting the vertical height of the tube 38, the pressure head on the column of water in the vessel 13 may be controlled so as to prevent substantial upward flow of the washing water past the valve 14 when said valve is lifted. The rate of flow of water upwardly through the vessel 13 is also so controlled as to insure the desired washing effect on the particles as they proceed downwardly toward the discharge outlet 34.

The process will now be described in connection with the leaching of an iron-aluminum alloy. It will be understood, however, that the principles of my invention are equally applicable to other problems of leaching.

A caustic alkali solution, which may suitably be an aqueous solution of caustic soda of 30% concentration by weight, is introduced into the vessel 10 to the level of the side tube 27. The heater 18 is then energized to heat up the solution in the portion A of the column to a temperature near the boiling point of the solution, which in this case will be around 110° C. When this temperature is reached, the valve 14 being closed, of course, throughout this operation, finely divided particles of the iron-aluminum alloy are introduced from a valve controlled funnel 40 containing a supply 41 of said particles into the upper open end of the vessel 10 at a suitable rate. This rate, in general, should be proportional to the rate of introduction of the fresh caustic soda solution through the inlet 24. The optimum proportion has been found to be about one part of alloy particles to 20 parts of caustic soda solution of around 30% concentration, by weight.

The particle size of the alloy should be finer than 40 mesh and preferably such as to pass through a 100 mesh screen. The caustic soda solution should be between 10 and 40% concentration and preferably 30% by weight.

The iron-aluminum alloy powder introduced at the top of the chamber reacts vigorously when it reaches the hot caustic soda solution. Due to the rapid evolution of hydrogen as a result of such reaction, a great deal of frothing occurs and this froth, or foam, substantially fills the vessel above the liquid level therein, which is controlled by the height of the side tube 27. The froth is comparatively stationary and so light as not to increase appreciably the head above the side tube connection to the vessel 10. The rate of introduction of cold caustic soda solution into the vessel 10 is such that the overflow out through the side tube 27 is at a sufficiently low enough linear velocity to avoid carrying particles up past the upwardly, offset portion 28.

Because of the rapid formation of gas bubbles during the active leaching of the particles, and the tendency of the bubbles to cling to the reacting particles until the particles rise to the top surface of the leaching solution, the rapidly leaching particles remain in the reaction zone A of the vessel until leaching is substantially completed. As the leaching reaction continues, the more fully reacted particles tend to drop downwardly from the upper surface of the leachant, trailing bubbles of gas as they sink, but usually developing renewed activity and rising again to the surface several times before the reaction slows to such a degree that very few bubbles are formed. When the leaching reaction is nearly completed, the rate of gas formation is no longer sufficient to provide the bubbles required for buoying up the particles, and the particles then drop downwardly through the reaction chamber A and through the lower chamber B.

The caustic soda solution in the lower portion B of the column is maintained at a relatively cool temperature, say, not above 60° C. and preferably around room temperature. If this cannot be accomplished by the rate of inflow of cold caustic soda from the reservoir 25 through the tube 24, the lower portion of the vessel 10 may be provided with a water jacket for cooling purposes. As the leached particles drop from the reaction zone A into the lower chamber B, the leaching reaction is abruptly ended, but at this point the reaction is substantially complete due to the buoyant effect of the gas bubbles in zone A. Thus, the time of leaching is automatically controlled by the reaction itself, the leached particles dropping out of the reaction zone as soon as the reaction is complete.

As the leached particles pass downwardly through the cold NaOH solution, the sodium aluminate produced by the leaching reaction is largely dissolved away from the particles by the caustic soda solution. Since the particles are widely separated from each other as they pass through this washing zone, they are each washed thoroughly in a copious supply of caustic soda washing solution, yet are in contact with that solution only a very limited time, of the order of 5 to 30 seconds.

By means of the apparatus disclosed, the temperature of the solution in the lower portion B of the column may be kept below 60° C. while the temperature in the upper part A is maintained at its boiling point. In this way a minimum amount of mixing occurs between the solutions in the two portions of the column.

As leached particles reach the bottom of the vessel **10**, the valve **14** is lifted to discharge these particles into the lower vessel **13**. While the valve **14** is lifted, the pressure adjusting tube **38** is lowered sufficiently to reduce any tendency of the water to flow upwardly through the upper end **15** of the vessel **13** and to permit the leached particles to proceed downwardly into the lower vessel **13**. In the column **13**, the particles are subjected to countercurrent washing with water to remove adhering soluble products of the reaction, such as sodium aluminate, and also such caustic soda as enters the upper end **15** of the vessel **13** along with the leached particles. The electromagnets **35**, **36** and **37**, may be operated if desired to magnetically agitate the particles falling through the vessel **13**.

By the continuous leaching process as herein described, the alloy particles are each leached under identical conditions. This maintenance of constant conditions results in the production of a product having consistent properties not obtainable by former batch operations.

The fully washed particles are removed either continuously or periodically from the bottom of the vessel **13** through the valve controlled outlet **34**. These particles may then be further processed as disclosed in the pending Howell application Serial No. 731,721, filed February 28, 1947, assigned to a common assignee.

As there described, the residue from the leaching and washing steps consists largely of Fe_3O_4 in a matrix of oxyferrite. Such residue has very desirable magnetic properties that are possessed to a degree not found in the ordinary magnetic oxides of iron.

While my process has been described in connection with the leaching of iron-aluminum alloys, it is equally applicable to the leaching of aluminum alloys of other heavy metals, such as nickel and cobalt, and to the leaching of other metallic alloys generally.

It will, of course, be understood that various details of construction may be varied through a wide range without departing from the principles of this invention and it is, therefore, not the purpose to limit the patent granted hereon otherwise than necessitated by the scope of the appended claims.

I claim as my invention:

1. A continuous process for the leaching of an iron-aluminum alloy, which comprises introducing finely comminuted particles of said alloy into an aqueous caustic alkali solution, maintaining the solution in the upper part of said column at near its boiling temperature during introduction of said alloy, introducing a cold fresh aqueous caustic alkali solution near the bottom of said column, withdrawing spent caustic alkali solution near the top of said column, controlling the inflow of fresh caustic alkali solution at such a rate as to maintain the lower part of said column relatively cool with respect to the upper part of said column but not at a sufficiently rapid rate to cause particles present in said upper part to pass out with the spent caustic alkali solution and intermittently releasing the leached particles from the bottom of the caustic alkali solution column into the top of an upwardly flowing column of wash water.

2. A continuous process for the leaching of an iron-aluminum alloy, which comprises introducing finely comminuted particles of said alloy into a column of an aqueous caustic alkali solution equivalent to about 30% sodium hydroxide concentration, maintaining the solution in the upper part of said column at near its boiling temperature during introduction of said alloy, introducing a cold fresh aqueous caustic alkali solution of similar concentration near the bottom of said column and withdrawing spent aqueous caustic alkali solution near the top of said column, the weight of the fresh solution introduced being about twenty times the weight of the alloy introduced in the same period of time, controlling the inflow of fresh caustic alkali solution at such a rate as to maintain the lower part of said column relatively cool with respect to the upper part of said column but not at a sufficiently rapid rate to cause particles present in said upper part to pass out with the spent caustic alkali solution, and intermittently releasing the leached particles from the bottom of the caustic alkali solution column into the top of an upwardly flowing column of wash water.

3. A continuous process for leaching of an alloy of aluminum in a ferromagnetic metal, which comprises charging the alloy in particle form into the top of a column of an aqueous caustic alkali solution, maintaining the solution near the top of the column sufficiently hot to leach particles charged thereinto, the hot caustic alkali solution reacting with the aluminum of the alloy to evolve hydrogen and the buoyant effect of the hydrogen gas so evolved serving to maintain the particles in the top of the column during the leaching of the particles, introducing a cold fresh aqueous caustic alkali solution near the bottom of said column and withdrawing spent aqueous solution near the top of said column, controlling the inflow of such fresh caustic alkali solution at such a rate as to maintain the lower part of said column relatively cool with respect to the upper part of said column but not at a sufficiently rapid rate to cause particles present in said upper part to pass out with the spent caustic alkali solution, and intermittently releasing the leached particles from the bottom of the caustic alkali solution column into the top of an upwardly flowing column of wash water.

4. A continuous process for the leaching of an alloy of aluminum and a ferromagnetic metal, which comprises subjecting the alloy in particle form to the action of a hot aqueous alkali solution with resultant evolution of hydrogen gas, the liberation of such gas during the leaching operation maintaining said particles in contact with the hot alkali solution until leaching of the particles is substantially complete, introducing a cold fresh caustic alkali solution beneath the hot aqueous caustic alkali solution and withdrawing spent aqueous caustic alkali solution near the top

of the hot caustic alkali solution, controlling the inflow of fresh caustic alkali solution at such a rate as to maintain the lower part relatively cool with respect to the upper part of the resulting body of solution but not at a sufficiently rapid rate to cause particles present in said upper part to pass out with the spent sodium hydroxide solution, and intermittently releasing the leached particles from the bottom of the solution body into the top of an upwardly flowing body of wash water.

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