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## PROCESS OF PEROXIDIZING STORAGE-BATTERY PLATES.

SPECIFICATION forming part of Letters Patent No. 712,995, dated November 4, 1902.

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To all whom it may concern:

Be it known that I, RUFUS N. CHAMBER-LAIN, a citizen of the United States, residing in Depew, in the county of Erie and State of New York, have invented certain new and useful Improvements in Peroxidizing Storage-Battery Plates, of which the following is a specification.

This invention relates to improvements in

10 peroxidizing storage-battery plates.

It has been proposed to use in peroxidizing lead plates a solution containing sulfuric acid, to which is added a certain proportion of alkaline nitrates, resulting in a bath containing sulfuric acid, with a small percentage of nitric acid. This process has the disadvantage of leaving a certain amount of soluble alkaline sulfates in the bath, which may lead to injurious secondary reactions. It has also been proposed to add a small proportion of nitric acid direct to the bath; but in this case the disadvantage exists that the nitric acid not being set free in a nascent state in the bath is not as energetic as it would be if it were nascent.

The object of my invention is to present the nitric acid to the plate in a more effective manner, and to this end I add to the sulfuricacid solution a suitable proportion of nitrate 30 of a metal whose sulfate is substantially insoluble in the bath produced—for example, nitrate of lead. As a result, the sulfate is immediately removed from the bath, and the reaction proceeds in the most efficient man-35 ner without secondary reactions. The nitric acid is liberated whether wholly by the direct chemical action or partly by such action and partly by electrolytic effect in a nascent state in the bath itself, and its peroxidizing 40 effect is correspondingly increased. I may and prefer to use for the supply of nitrate of lead the by-product of a previous stage of the plate-treating process—namely, the nitrate-of-lead solution resulting from the pick-45 ling operation. In case the nitrate-of-lead solution so produced is not strong enough to use directly it may be made sufficiently strong either by concentration or by addition of lead or of solid lead nitrate. The precipitate also

from the peroxidizing bath—namely, lead sul- 50 fate—may be recovered and utilized, for example, by reduction or fusion into metallic lead for grids or otherwise.

The process is preferably carried on as follows: The plates are first placed in a pick- 55 ling-bath composed of dilute nitric acid and are therein pickled by the action of said acid, assisted by an electric current, the nitric acid being replenished from time to time, so as to maintain an effective solution, while the ni- 60 trate of lead is gradually accumulated in the bath, and eventually the solution becomes so charged therewith as to be serviceable in the following operation, an additional amount of lead or lead nitrate being supplied to such 65 solution, if necessary. The plates are then peroxidized in a bath prepared as follows: To a solution of sulfuric acid containing 36.7 parts of H<sub>2</sub>SO<sub>4</sub> to one hundred parts of water are added sixteen parts of nitrate of lead, or 70 if the nitrate of lead is added in dissolved state, as in the form of solution from the pickling-bath, then the amount of water in the original sulfuric-acid solution is correspondingly decreased, so that the propor- 75 tions of water, sulfuric acid, and nitrate of lead remain substantially as above stated. The chemically cleaned or pickled plates are placed in this bath, preferably before the addition of the nitrate of lead, so that on 80 such addition nitric acid is liberated in a nascent state and a powerful oxidizing bath is produced. At the same time the plate is connected with suitable electrodes and electric energizing connections, the plate to 85 be peroxidized being arranged as the anode. Upon passage of the electric current the nitric acid, which is set free by the chemical and electrolytic actions, is caused by the electric current to attack the surface of the anode, go forming nitrate of lead, which is immediately converted by the sulfuric acid present into sulfate of lead, and as this effect takes place in direct contact with the lead plate the sulfate of lead adheres to the lead plate or grid. 95 This sulfate of lead is gradually converted into peroxid of lead by the oxygen which is liberated at the anode through the electrolysis

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of the water and the decomposition of the nitric acid, the oxidizing effect being increased by the nascent character of the nitric acid. As many plates may be treated in the bath as may be found desirable, nitrate of lead being added from time to time to maintain the efficiency of the bath, and the precipitate—namely, sulfate of lead—which gradually accumulates in the bath may be removed from time to time and utilized by reduction to metallic lead or otherwise. After the plates have thus been provided with a sufficient coating of peroxid they are removed

from the above-described bath, washed, and placed in the ordinary forming solution and are there electrolyzed sufficiently to effectually clear them of any slight amount of sulfate which remains therein, although the same effect may be produced in the peroxidizing bath itself by allowing the period.

izing bath itself by allowing the action to proceed in this bath without renewal of the nitric acid until such acid is practically used up and then continuing the electrolysis to peroxidize all the sulfate present on the plates.

It will be seen that the by-product of the pickling operation—namely, nitrate of lead—is utilized in the peroxidizing operation, while the by-product of the latter operation—namely, lead sulfate—is also utilized.

What I claim as new, and desire to secure by Letters Patent, is—

1. The process of treating storage-battery

plates which consists in subjecting the plates as anodes to electrolysis in a bath consisting of a solution of sulfuric acid to which nitrate 35 of lead is added.

2. The process of treating lead plates for storage batteries which consists in pickling the lead plates in a dilute nitric-acid solution and thereafter subjecting the lead plates as 40 anodes to electrolysis in a bath consisting of a solution of sulfuric acid to which the nitrate of lead resulting from the pickling operation is added.

3. The process of treating lead plates for 45 storage batteries which consists in subjecting the plates as anodes to electrolysis in a bath consisting of a solution of sulfuric acid to which nitrate of lead is added and removing from such solution the resulting precipitate 50 of sulfate of lead.

4. The process of peroxidizing lead plates for storage batteries which consists in subjecting the plates as anodes to electrolysis in a bath consisting of a solution of sulfuric acid 55 to which is added the nitrate of a metal whose sulfate is substantially insoluble in the bath, thereby setting free nascent nitric acid and at the same time precipitating the resulting sulfate.

RUFUS N. CHAMBERLAIN.

Witnesses:

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