

UNITED STATES PATENT OFFICE.

JAMES D. DARLING, OF PHILADELPHIA, PENNSYLVANIA, ASSIGNOR TO THE HARRISON BROS. & COMPANY, INCORPORATED, OF SAME PLACE.

PROCESS OF MAKING PEROXID OF LEAD FOR GALVANIC BATTERIES.

SPECIFICATION forming part of Letters Patent No. 603,361, dated May 3, 1898.

Application filed December 23, 1896. Serial No. 616,775. (No specimens.)

To all whom it may concern:

Be it known that I, JAMES D. DARLING, of Philadelphia, in the State of Pennsylvania, have invented a certain new and useful Process of Making Elements of Peroxid of Lead for Galvanic Batteries, of which the following is a specification.

My invention relates to the use of peroxid of lead (PbO_2) as an element either for a primary or secondary cell. The desirability of this material itself for the purpose indicated has of course long been recognized, but its use has been attended with certain practical disadvantages. When compressed into a stick or plate or applied mechanically to the grid of a secondary battery, it has been found impossible, even by the use of great pressure, to so compact the peroxid of lead that it will remain permanently coherent. Thus it is found that when an element so made is placed in dilute sulfuric acid, which is the usual electrolyte, disintegration of the surface commences and sometimes continues throughout the mass until practically all of it has fallen away. This weakness of structure is particularly noticeable where the element is intended to be discharged and recharged, as in the case of a storage battery.

The use of a foreign binding material mixed with peroxid of lead is, so far as I am aware, impracticable, since it destroys the homogeneity of the element and even if capable of being made permanent would impair its efficiency.

Extraneously-applied retaining devices have been employed in connection with the mass of compressed peroxid of lead—as, for instance, plaster-of-paris or other adhesive material of a foreign character. So far as I am aware, however, no one has practically succeeded in obtaining an element of peroxid of lead with a surface rendered permanently coherent without the use of foreign binding material or extraneous support.

The object of my invention is to dispense with the use of an extraneously-applied coating and of binding material of a foreign character and to obtain an element of compressed peroxid of lead having a permanently coherent superficial layer without impairing the characteristic properties of the material or

producing any notable effect other than the accomplishment of the desired purpose.

I take a suitable quantity of peroxid of lead and moisten it with water to form a paste or dough, which is then compressed into the form desired, a suitable conductor being embedded therein. The next step of treatment requires that there should be present in the superficial layer of the compressed mass a small percentage of a proper acid, such as sulfuric acid. This condition is attained as follows: If the peroxid has been electrolytically made, a minute percentage of free sulfuric acid is practically always present in the product as an incident of its manufacture. In such case it is usually only necessary to moisten the peroxid with water. If, however, the peroxid has been made by a chemical process as distinguished from an electrolytic one it is usually free from such incidentally-contained sulfuric acid, and in this case the acid should be added to the water with which the material is moistened. Three per cent. of free acid in solution in the water will usually suffice for the purpose. In either condition, therefore, it will be noted that the compressed stick or plate will contain a slight percentage of free acid, and while for the purposes of my invention it is probably only essential that the acid should reside in the outside layer, yet for convenience of manipulation the entire mass may as well be allowed to contain it. I then carefully place the compressed mass in a neutral electrolyte—such as a neutral solution of sulfate of zinc, composed, say, of one ounce of sulfate to three ounces of water—and electrically connect the embedded conductor with a plate of zinc, also immersed in said solution; thus in effect short-circuiting the pair. They are allowed to remain in electrical connection for a short period—say five or ten minutes—after which the stick or plate of lead peroxid is removed and may be dried for shipment or may be placed at once in the battery where it is to be used. It will be found that after this treatment the surface of the mass of peroxid of lead has become, so to speak, “indurated,” without impairment of its efficiency, and that the superficial particles have become coherently bound together, so as to be no longer

readily friable nor liable to disintegrate when placed in the usual electrolyte. Furthermore, it can be recharged after discharge in the usual manner practiced with storage batteries without substantially losing its characteristics.

I believe that the superficial binding effect may be explained upon the theory that when the mass is placed in the neutral solution and electrically connected with a proper element some of the peroxid of lead is slightly deoxidized at the surface, and simultaneously with such deoxidation a minute quantity of sulfate of lead is formed through the attack of the inherently-contained sulfuric acid. This product appears to bind together the particles of unconverted peroxid, and as by the short time of exposure the action only penetrates to a slight depth the decomposition is exceedingly limited in extent. Without, however, wishing to import this theory into my invention I find the term "inherently formed" a convenient one to describe the nature of the binding material, meaning thereby that such material is formed *in situ* and from the peroxid itself, as distinguished from being a foreign body or a coating extraneously applied.

Having thus described my invention, I wish it to be understood that I do not of course claim, broadly, the manufacture of elements from peroxid of lead, since I am aware that

various processes have been described for that purpose. On the other hand, I do not limit myself to the use of the exact reagents above mentioned for producing the inherently-formed binding material, it being only essential that the compressed mass with an acidulated surface should be treated as a cathode in a neutral electrolyte which is of such composition as not to excessively deoxidize the peroxid of lead. Successful conduct of the process depends upon the fact that the electrolyte, proper in other respects, is practically neutral, for I have even found that after treatment of a number of sticks with a solution of sulfate of zinc, neutral at the start, a slight acid reaction may be obtained in the solution and that thereafter its efficiency is greatly impaired.

I claim—

The hereinbefore-described process of making elements of peroxid of lead, which consists in compressing a mass of peroxid of lead containing free acid, and then exposing said mass to electrolytic action as a cathode in a neutral electrolyte until the superficial layer of the mass has been rendered coherent by the production of inherently-formed binding material, substantially as set forth.

JAMES D. DARLING.

Witnesses:

JAMES H. BELL,
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