

UNITED STATES PATENT OFFICE.

HENRY LEITNER, OF MAYBURY, WOKING, ENGLAND.

METHOD OF PRELIMINARY TREATMENT FOR ACCUMULATOR-PLATES.

No. 867,517.

Specification of Letters Patent.

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To all whom it may concern:

Be it known that I, HENRY LEITNER, of Maybury, Woking, in the county of Surrey, England, have invented a certain new and useful Improved Method of Preliminary Treatment for Accumulator-Plates; and I do hereby declare the following to be a full, clear, and exact description of the invention, such as will enable others skilled in the art to which it appertains to make and use the same.

My invention refers to an improved process or method for bestowing a preliminary treatment on accumulator plates by the use of which the formation of the plates so treated as electrodes on the Plante principle, is greatly facilitated and improved.

A leading feature of my invention consists of the practical utilization of the discovery which I have made that the rapid forming effect obtainable by the use of very dilute or attenuated solutions can be greatly increased by the use of high current densities, provided that the temperature resulting from the use of high current density is prevented from rising so high as to exercise a prejudicial effect upon the plates while subjected to the process.

In carrying out my invention in practice I employ plates of cast, stamped, perforated or laminated lead of any shape or form suitable for a Plante electrode, and subject the same as anodes to the action of an electric current in an electrolyte, consisting of a highly attenuated or dilute solution of a suitable sulfate or sulfates, to which mere traces of substances capable of forming soluble lead compounds, preferably chlorids, are added.

A suitable electrolyte consists of a solution of from $\frac{1}{2}$ to 2 per cent. of a sulfate of one of the alkali metals, to which solution is added not more than 2 parts per 1000 of one of the chlorids of one of the alkali metals. Corresponding mixtures of the sulfates and chlorids of these metals may be employed, but the effect of action depends upon the proportions not being practically exceeded. But I have also found that very dilute acids may be employed with equal and even better effect. An acid electrolyte which gives admirable results consists of water acidulated with from $\frac{1}{2}$ to $3\frac{1}{2}$ per cent. of pure sulfuric acid of sp. gr. of 1.820 to which $\frac{1}{2}$ to $3\frac{1}{2}$ parts per 1000 of pure hydrochloric acid containing 28 per cent. HCL have been added.

It is preferable in carrying out this process that the distance between anodes and cathodes should be considerable, and that the current density employed should be as great as possible, namely preferably such as to raise the temperature of the electrolyte to from 75° to 85° F. but not to exceed the latter temperature.

In order to facilitate and increase the rapidity of the process, I may artificially cool the bath of electrolyte in which it is being carried out by any convenient

means, such as a stream of running cold water arranged to traverse a tank in which the cells or vessels in which the process is being carried out are arranged or by surrounding them with a freezing mixture, or dissolving salts around them, the solution of which is accompanied by a fall of temperature. By this means I am enabled to shorten the process by using higher current densities without causing the above mentioned limits of temperature, which would be prejudicial to the plates if exceeded, to be surpassed. The best results are obtained by so adjusting the temperature and current density that within a period ranging from 18 to 46 hours the whole surface of the plates treated as anodes becomes converted into a granular highly oxidized layer or pellicle penetrating everywhere to the same depth (which can be predetermined) and not displaying the slightest trace of corrosion or irregular action. The plates which have been thus treated as anodes are then subjected to certain further manipulations and processes. Firstly, they are immersed in running water or water that is frequently changed in which they remain for some length of time in order to cleanse and consolidate the oxidized layer. Thereafter the plates are treated as cathodes in any convenient innocuous electrolyte, such as very dilute sulfuric acid. In this way all the highly oxidized lead particles are converted into granular yet finely divided particles of electrolytic lead firmly adherent to the surface of the electrode and its laminations. As a variation of my process I may perform this reduction in the same solution in which the plates were in the first instance formed. The electrolytic action taking place in this process to some considerable extent reconstitutes the electrolyte composition, enabling a second set of positive electrodes to be partly formed simultaneously with the said reduction thereby economizing current. As a further variation I may instead of the used electrolyte employ an electrolyte consisting of water to which mere traces of substances sufficient to render it conductive to the electric current are added. The plates which are being reduced in that case contribute to the electrolyte by reducing the substances necessary for the formation of the positive plates.

After reduction the reduced electrodes are allowed to remain in water for a moderate length of time and are subsequently exposed to the action of dry, warm air, which has the effect of gradually oxidizing the finely divided electrolytic lead layer into a lower oxid of lead and the whole mass becomes gradually very firm and tenacious.

The plates or electrodes after having undergone the processes above described may be built up into sections and formed either into negatives or positives but are more specially adapted for use as the latter. They may be formed at once in sulfuric acid of the ordinary

strength employed in accumulators and will not be injured if formed in acid of even somewhat greater strength, while three or four discharges are sufficient to give the plates a full and high capacity.

- 5 The formula of sulfuric acid being H_2SO_4 I have herein considered it as a sulfate, the formula being analogous to that of sodium and potassium sulfates.

I claim:—

1. The process of preparing electrodes for secondary batteries, which consists in immersing the plate in a weak electrolyte, comprising a sulfate solution containing not more than three and one-half per cent. of the sulfate, to which is added not more than three and one-half parts per 1000 of a chlorid, and subjecting the plate as an anode to an electric current of as high density as possible, without raising the temperature of the electrolyte above $85^\circ F.$, artificially cooling the electrolyte to permit the use of a higher current density than could otherwise be employed, then washing the plate to cleanse it and consolidate the oxidized layer, then treating the plate as a cathode in an innocuous electrolyte, treating the plate with water and then exposing it to the action of warm air, substantially as described.
2. The process of preparing electrodes for secondary batteries which consists in immersing the plate in a weak electrolyte, comprising a sulfate solution containing not more than three and one-half per cent. of the sulfate, to

which is added not more than three and one-half parts per 1000 of a chlorid, and subjecting the plate as an anode to an electric current of as high density as possible without raising the temperature of the electrolyte above $85^\circ F.$, artificially cooling the electrolyte to permit the use of a higher current density than could otherwise be employed, substantially as described.

3. The process of preparing electrodes for secondary batteries which consists in immersing the plate in a weak electrolyte comprising a sulfate solution containing not more than three and one-half per cent. of the sulfate, to which is added not more than three and one-half parts per 1000 of a chlorid, and subjecting the plate as an anode to an electric current of as high density as possible, without raising the temperature of the electrolyte above $85^\circ F.$, substantially as described.

4. In a process of preparing electrodes for secondary batteries, immersing the plate in a weak electrolyte comprising not more than three and one-half per cent. of sulfuric acid, to which is added not more than three and one-half parts per 1000 of hydrochloric acid, subjecting the plate as an anode to an electric current of high density and maintaining the electrolyte at a temperature of not more than $85^\circ F.$, substantially as described.

In witness whereof I have hereunto set my hand in the presence of two witnesses

HENRY LEITNER.

Witnesses:

RICHARD NORMAN LUCAS,
R. SMITH.