UNITED STATES PATENT OFFICE.

THOMAS A. EDISON, OF LLEWELLYN PARK, ORANGE, NEW JERSEY, ASSIGNOR TO EDISON STORAGE BATTERY COMPANY, OF WEST ORANGE, NEW JERSEY, A CORPORATION OF NEW JERSEY.

PROCESS OF MAKING METALLIC FILMS OR FLAKES.

936,525. Specification of Letters Patent. Patented Oct. 12, 1909.

No Drawing. Application filed January 18, 1907. Serial No. 353,002.

To all whom it may concern:

Be it known that I, THOMAS ALVA EDISON, a citizen of the United States, residing at Llewellyn Park, Orange, in the county of Essex and State of New Jersey, have invented a certain new and useful Process of Making Metallic Films or Flakes, of which the following is a description.

My invention relates to an improved process of making metallic films or flakes, and particularly flakes of metallic cobalt or metallic nickel or cobalt-nickel alloy, for use in the make-up of the positive electrodes of storage batteries of the Edison type. As I have previously indicated, flakes of metallic cobalt or cobalt-nickel alloy are peculiarly fitted for this use, owing to the high character of the contact which will be afforded for the particles of active material, such as nickel hydroxid, as well as the permanency of the flakes under the effect of electrolysis. In Letters Patent No. 821,626 dated May 29, 1906, I describe a process for this purpose in which the flakes are formed by depositing electrolytically upon a cathode layers or films of a soluble metal alternating with successive films of the desired metal (cobalt or nickel or cobalt-nickel) after which the soluble metal is dissolved, so as to result in the separation and segregation of the cobalt or nickel or cobalt-nickel films. In the patent referred to, I describe the use of zinc as a suitable soluble metal, and I describe the breaking up or sizing of the cobalt or nickel or cobalt-nickel flakes by means of a screening operation.

My present invention relates to an improvement on the process described in said patent by which flakes of cobalt or nickel or cobalt-nickel can be secured which will be very much smoother, of more uniform thickness and more coherent than when zinc is used, and which flakes will also be more uniform in size than when sub-divided by a screening operation.

To this end the invention consists, broadly speaking, first in making use of copper as the soluble metal on which the films of cobalt or nickel, or cobalt-nickel are deposited, and second, in cutting up the composite strips or sheets to the required size after separation of the same from the cathode.

The invention also contemplates details of procedure, all as will be more fully hereinafter described and claimed.

In carrying my invention into effect, I proceed substantially as follows: The cathode consists of a plate or cylinder of polished copper, preferably nickel-plated, and which may be conveniently rotated during the plating and subsequent operations, as I describe in my application for Letters Patent filed October 12, 1905, Serial No. 282,380. The cathode is first rubbed with graphite so as to polish the surface thereof and permit the effective separation of the deposited composite sheet. I first introduce the cathode in a copper plating bath employing any suitable copper salt, such as the sulfate thereof with metallic copper anodes and deposit a thin layer or film of copper on the cathode, as will be understood. Such a copper layer will be extremely smooth and coherent and in this respect very superior to zinc. The cathode is now washed and then immersed in a cobalt or nickel or cobalt-nickel bath, the solution used being preferably an ammonium sulfate solution of the metal or metals to be plated, and anodes of cobalt or of nickel or of cobalt and nickel being employed. In the latter case, the anodes and the depositing current will be so regulated as to secure the desired relative deposit of the two metals. When the cobalt or nickel, or cobalt-nickel film has been thus deposited on the copper film, the cathode is again washed, returned to the copper bath and a second layer of copper is deposited on the cobalt or nickel or cobalt nickel film. After washing, a second film of cobalt or nickel or cobalt-nickel is deposited on the second copper film, and these operations are continued until a sufficient number of layers of copper and cobalt or nickel or cobalt-nickel are secured. The composite sheet thus obetained on the cathode is easily stripped from the same by cutting the sheet longitudinally, so as to permit the sheet to be peeled off. To facilitate this cutting of the composite sheet, the cathode is formed with one or more longitudinal grooves which act as effective guides for the cutter. After the sheet has been thus separated from the cathode, it is preferably cut up into strips about three inches wide, and these strips are subdivided by means of a suitable cutting machine of any desired

type, into squares or other forms, the dimensions of which in length and breadth determine the ultimate size of the flakes to be produced. Ordinarily, each flake will be about $\frac{1}{16}$ of an inch square. At this stage of the method, I will obtain a great number of very small squares or bodies each formed of successive and alternating layers of copper and cobalt or nickel or cobalt-nickel, as will be understood. It now becomes necessary to dissolve the copper without affecting the cobalt or nickel or cobalt-nickel flakes, thereby eliminating the copper and separating the flakes desired. This is preferably effected by soaking the sub-divided composite bodies in a very strong solution of cyanid of potassium, and agitated at times during the treatment. The effect of the cyanid is to dissolve the metallic copper, without appreciably affecting the cobalt or nickel or cobalt-nickel, thus freeing the flakes of cobalt or nickel or cobalt-nickel and effectively separating the same. These flakes may now be used directly in the make-up of the battery electrodes or they may be first annealed in hydrogen before such use. In applying the cobalt or nickel or cobalt-nickel flakes to the active particles, I prefer to make use of the process described in my patent dated December 25, 1906, No. 839,371, wherein the active particles are first coated with a sticky material, such as molasses or glucose, after which the conducting flakes are added, and will be caused to adhere to the surface of the active particles to thereby coat the same in the most effective manner.

Having now described my invention, what I claim as new and desire to secure by Letters Patent is as follows:—

1. The process of making films or flakes of cobalt or nickel or cobalt-nickel, which consists in electrolytically depositing on a suitable cathode a layer or film of copper, then in depositing on the copper film a layer or film of cobalt or nickel or cobalt-nickel, and in finally dissolving the deposited copper to free the film of cobalt or nickel or cobalt-nickel, substantially as set forth.

2. The process of making films or flakes of cobalt or nickel or cobalt-nickel, which consists in electrolytically depositing upon a suitable cathode a layer or film of copper, then in depositing on the copper a layer or film of cobalt or nickel or cobalt nickel, then in separating the composite sheet from the cathode and in finally dissolving the deposited copper film to free the film of cobalt or nickel or cobalt nickel, substantially as set forth.

3. The process of making films or flakes of cobalt or nickel or cobalt-nickel, which consists in depositing upon a suitable cathode a layer or film of copper, then in depositing upon the copper film a layer or film of cobalt or nickel or cobalt-nickel, then in separating the composite sheet from the cathode, then in cutting up the composite sheet into bodies of the ultimate shape and size and in finally dissolving the copper to free the deposited cobalt or nickel or cobalt-nickel, substantially as set forth.

4. The process of making films or flakes of cobalt or nickel or cobalt-nickel, which consists in depositing upon a suitable cathode a layer or film of a soluble metal, then in depositing on the soluble film a layer or film of cobalt or nickel or cobalt-nickel, then in separating the composite sheet from the cathode, then in cutting up the composite sheet into bodies of the ultimate shape and size and in finally dissolving the soluble metal to free the flakes of cobalt or nickel or cobalt-nickel, substantially as set forth.

5. The process of making films or flakes of cobalt or nickel or cobalt-nickel, which consists in depositing upon a suitable cathode successive and alternating layers of copper and cobalt or nickel or cobalt-nickel, and in finally dissolving the copper to free the films of cobalt or nickel or cobalt-nickel, substantially as set forth.

6. The process of making films or flakes of cobalt or nickel or cobalt-nickel, which consists in applying graphite to a suitable cathode, then in depositing a layer or film of copper thereon, then in depositing on the copper film a layer or film of cobalt or nickel or cobalt-nickel, and in finally dissolving the copper to free the film of cobalt or nickel or cobalt-nickel, substantially as set forth.

7. The process of making films or flakes of cobalt or nickel or cobalt-nickel, which consists in depositing upon a suitable cathode a layer or film of copper, then in depositing a layer or film of cobalt or nickel or cobalt-nickel on the copper film, and in finally subjecting the composite sheet so formed to the action of a cyanid of an alkali to dissolve the copper and free the deposited cobalt or nickel or cobalt-nickel, substantially as set forth.

This specification signed and witnessed this 17th day of January 1907.

THOS. A. EDISON.

Witnesses:
FRANK L. DYER,
ANNA R. KLEHM.