UNITED STATES PATENT OFFICE.

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

PROCESS FOR MAKING CONDUCTING-FILMS.

No. 821,628. Specification of Letters Patent. Patented May 29, 1906.

Application filed May 26, 1905. Serial No. 262,476.

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, county of Essex, and State of New Jersey, have invented a certain new and useful Process for Making Conducting-Films for Storage Batteries, of which the following is a description.

My invention relates to a new process for making conducting-films adapted particularly for use for addition to the nickel hydroxid employed in the make up of positive electrodes for storage batteries of the Edison type. These scales or films are composed either wholly or partly of cobalt or cobalt-nickel alloy, the advantages of which are pointed out and claimed in my application for Letters Patent filed March 30, 1905, Serial No. 252,935.

By the improved process it will be possible to make use of other insoluble or relatively insoluble metals in connection with the cobalt, (such as bismuth, copper, cadmium, and iron,) the resulting compound or alloy presenting very beautiful scales or films which are eminently suited for the desired purpose.

My object is to provide a simple and effective process for the purpose which can be carried out commercially at low cost.

To this end the invention consists in fusing a sulfur compound of the metal or metals, the resulting fused mass being then cooled to result in the crystallization of the metallic sulfur compound in thin scales or films, which are then separated, subsequently roasted to convert the sulfur compound into an oxygen compound of the metal or metals, and the oxygen compound thus secured is finally reduced to the metallic state in a hydrogen atmosphere or in any other suitable way.

As a specific example of the process as applied to the manufacture of scales or films of cobalt or cobalt-nickel alloy I proceed substantially as follows: I first take the oxid of the metal or metals from which the films are to be formed (in the present case cobalt or a mixture of cobalt and nickel in various proportions) and convert the same by the addition of sulfur in any suitable way to form the sesquisulfid of the metal or metals. The sesquisulfid thus secured is now mixed with from six to eight times its weight with sulfid of potash or soda, (commonly called "liver of sulfur,") and the resulting mixture is then fused in a crucible until fully liquid, or instead the metallic oxid, sulfur, and alkaline sulfid may be added together at the same time, whereby the fusion of the mass will result in the formation of the metallic sesquisulfid. The fused mass is now cooled throughout, resulting in the crystallization of the sesquisulfid of the metal or metals, such crystals ranging from .0001 to .0002 in thickness. When the sesquisulfid of two metals, such as cobalt and nickel, are thus fused and cooled, the resulting crystals will be composed of the sesquisulfid of both metals in the proportions in which the oxids thereof were originally used. After the mass has been entirely cooled it is washed in warm water, dissolving out the potassic or sodic sulfid, leaving the scales or films, which are then dried. Generally there will be present in the dried mass a proportion of the sesquisulfid crystals which do not form scales or films, but which exist as very fine particles capable of being effectively separated from the usual scales or films by a screening operation through a screen about two hundred mesh. Such a screening operation is therefore performed to effect this separation, and the fine non-scaly crystals thus separated are returned to the crucible and re-fused with a fresh batch of the mixture of sesquisulfid and the potassic or sodic sulfid. In this case, of course, the proper allowance should be made for the sulfur contents presented by the additional increment of the fine non-scaly particles to the crucible.

Having obtained the scales or films in the form of sesquisulfid of the metal or metals desired, I now roast the same in the air to eliminate the sulfur and reoxidize the scales or films and reconvert the latter into an oxid of the metal or metals, but without in any way affecting the scale-like or crystalline form thereof. The oxid films or scales thus obtained are reduced to the metallic state by heat in a hydrogen atmosphere and are then ready for use.

Since both nickel and cobalt on the crystallization of their sulfids result in the production of scales or films, I am enabled to vary the proportion of the two metals in any suitable manner; but good results are secured when seventy per cent. of cobalt and thirty per

cent. of nickel are used. Furthermore, the tendency of both nickel and cobalt to result in the formation of scales or films in the manner described is so marked that it becomes possible to admix oxids of other insoluble or relatively insoluble metals therein—such as bismuth, copper, cadmium, or iron—resulting in the formation of scales or films composed of nickel or cobalt, or both, with other metals incorporated therewith, whereby the cost of manufacture may be considerably reduced, and in some instances superior effects may be obtained.

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

1. The process of making conducting-films, which includes fusing a sulfur compound of a metal or metals, in cooling the fused mass to result in the crystallization of the metallic sulfur compound in thin scales or films, in separating such scales or films, and in finally converting the latter to the metallic state, substantially as set forth.

2. The process of making conducting-films, which consists in fusing a sulfur compound of a metal or metals, in cooling the fused mass to result in the crystallization of the metallic sulfur compound in thin scales or films, in separating the latter, in roasting the scales or films to oxidize the same, and in finally reducing the scales or films to the metallic state, substantially as set forth.

3. The process of making conducting-films, which consists in fusing a sulfur compound of a metal or metals, in cooling the fused mass to result in the crystallization of the metallic sulfur compound in thin scales or films, in separating the latter, in roasting the scales or films to oxidize the same, and in finally reducing the scales or films in a hydrogen atmosphere to the metallic state, substantially as set forth.

4. The process of making conducting-films, which includes fusing a sulfur compound of the metal or metals, in cooling the fused mass to result in the crystallization of the metallic sulfur compound in thin scales or films, in screening the scales or films to eliminate non-scaly crystals, and in finally reducing the scales or films to the metallic state, substantially as set forth.

5. The process of making conducting-films, which includes fusing a mixture of a sulfur compound of the metal or metals with an alkaline sulfid, in cooling the fused mass to result in the crystallization of the metallic sulfur compound in thin scales or films, in removing the alkaline sulfid therefrom, and in finally reducing the scales or films to the metallic state, substantially as set forth.

6. The process of making conducting-films, which consists in fusing a mixture of a sulfur compound of the metal or metals with an alkaline sulfid, in cooling the fused mass to result in the crystallization of the metallic sulfur compound in thin scales or films, in removing the alkaline sulfid therefrom, in oxidizing the scales or films, and in finally reducing the same to the metallic state, substantially as set forth.

7. The process of making conducting-films, which includes fusing a sulfur compound of cobalt, in cooling the fused mass to result in the crystallization of the sulfur compound in thin scales or films, and in finally reducing the scales or films to the metallic state, substantially as set forth.

8. The process of making conducting-films, which consists in fusing a sulfur compound of cobalt, in cooling the fused mass to result in the crystallization of the metallic sulfur compound in thin scales or films, in oxidizing the scales or films, and in finally reducing the same to the metallic state, substantially as set forth.

9. The process of making conducting-films, which consists in fusing a sulfur compound of cobalt, in cooling the fused mass to result in the crystallization of the metallic sulfur compound in thin scales or films, in roasting the scales or films to eliminate sulfur and oxidize the same, and finally in reducing the oxid to the metallic state, substantially as set forth.

10. The process of making conducting-films, which consists in fusing a sulfur compound of cobalt, in cooling the fused mass to result in the crystallization of the metallic sulfur compound in thin scales or films, in screening the mass to eliminate non-scaly crystals, in roasting the scales or films to eliminate sulfur and oxidize the same, and in finally reducing the oxid to the metallic state, substantially as set forth.

11. The process of making conducting-films, which includes fusing a mixture of a sulfur compound of cobalt and alkaline sulfid, in cooling the fused mass to result in the crystallization of the metallic compound in thin scales or films, in washing the mass to eliminate the alkaline sulfid, and in finally reducing the scales or films, substantially as set forth.

This specification signed and witnessed this 20th day of May, 1905.

THOS. A. EDISON.

Witnesses:
FRANK L. DYER,
MINA C. MACARTHUR.