T. A. EDISON.

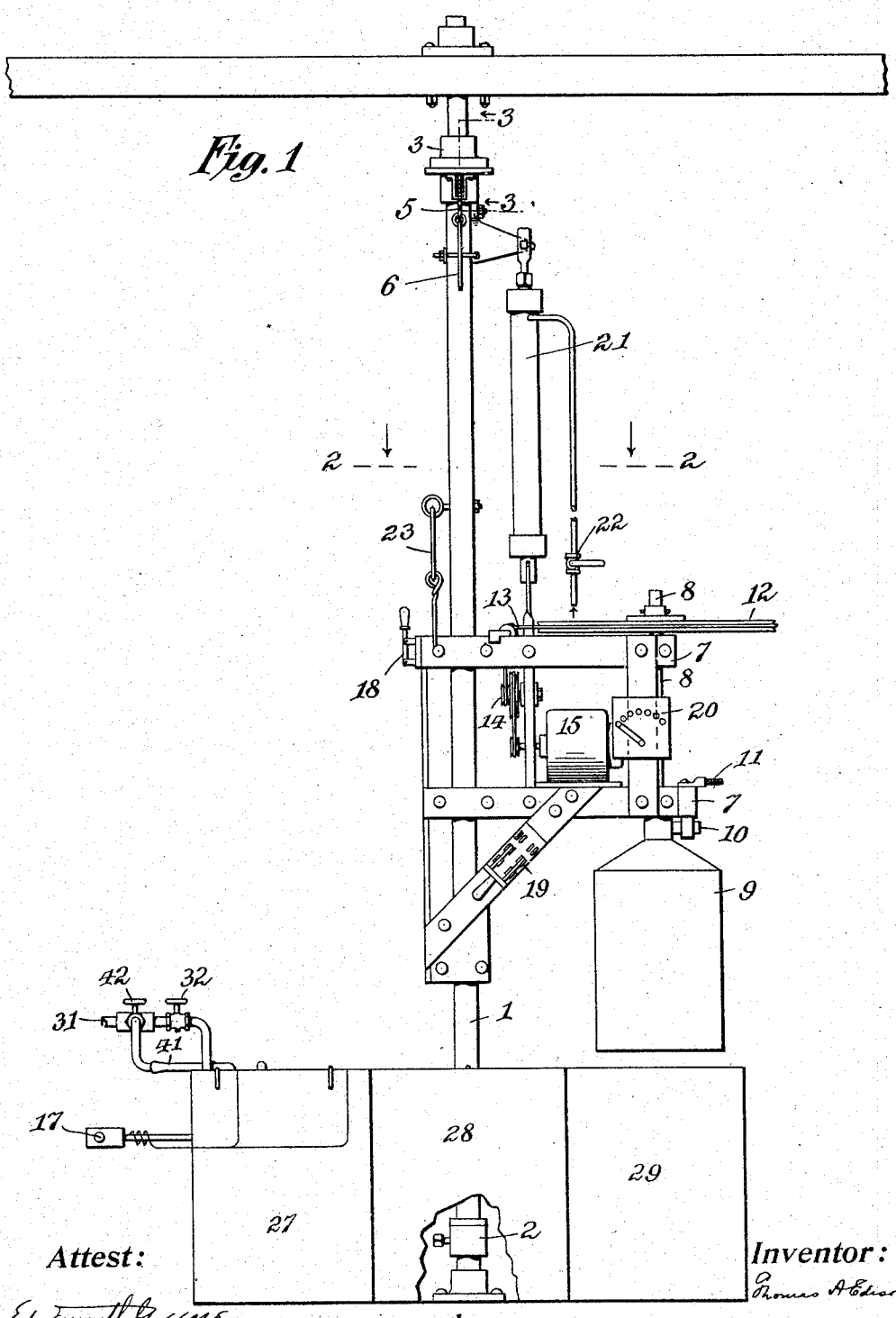
APPARATUS FOR MAKING METALLIC FILMS OR FLAKES.

APPLICATION FILED OCT. 12, 1905.

950,227. Patented Feb. 22, 1910.

3 SHEETS—SHEET 1.

Fig. 1



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3 SHEETS—SHEET 2.

Fig. 4.

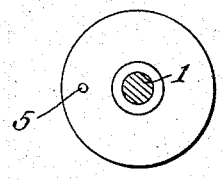


Fig. 3.

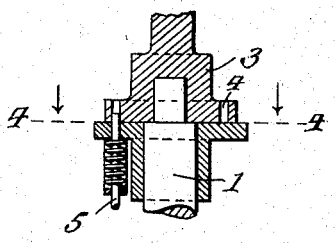
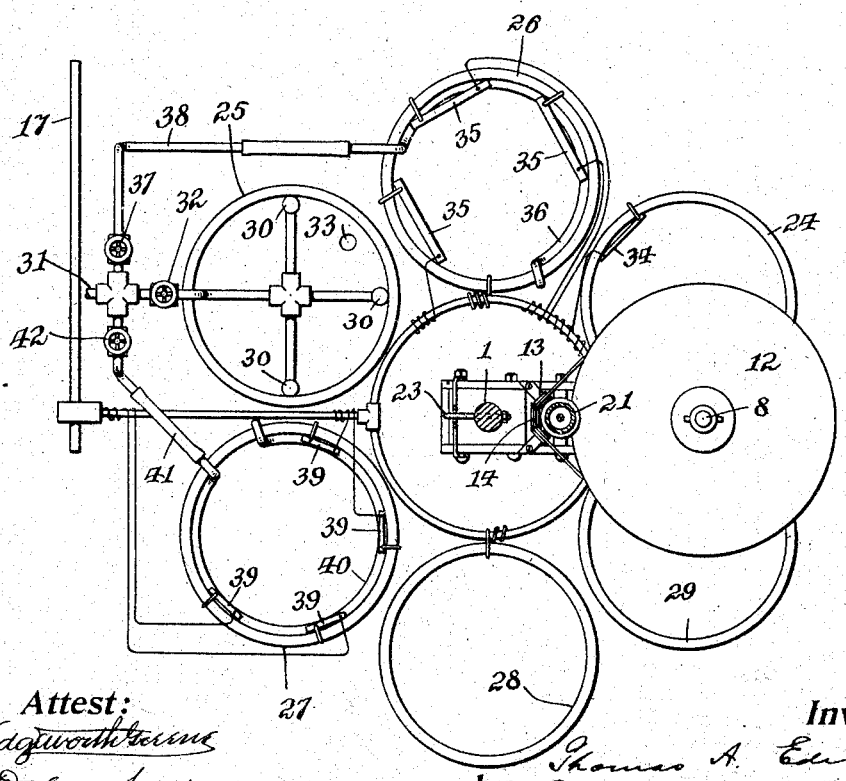


Fig. 2.



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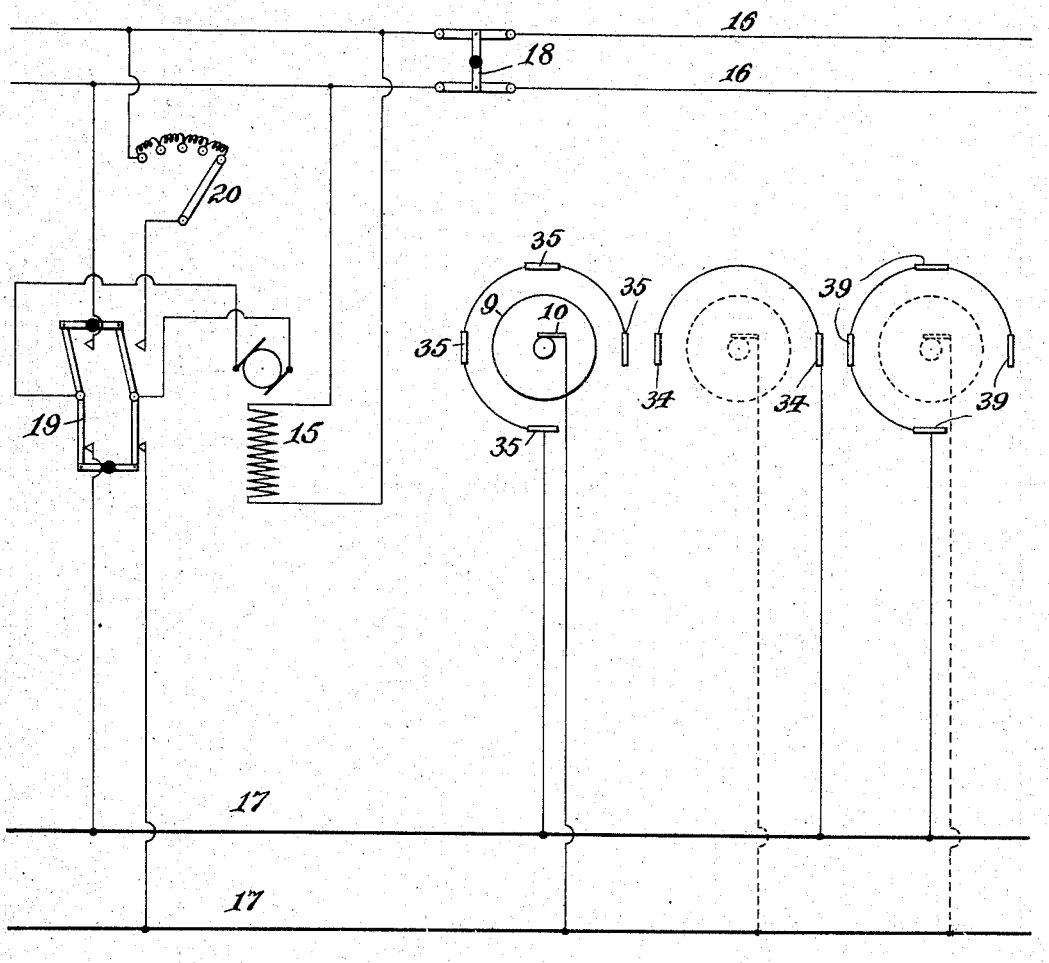
3 SHEETS—SHEET 3.

Fig. 5.



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UNITED STATES PATENT OFFICE

THOMAS A. EDISON, OF LLEWELLYN PARK, ORANGE, NEW JERSEY.

APPARATUS FOR MAKING METALLIC FILMS OR FLAKES.

950,227. Specification of Letters Patent. Patented Feb. 22, 1910.

Application filed October 12, 1905. Serial No. 282,380.

To all whom it may concern:

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

In Patent No. 821,626, granted May 29, 1906, I describe an improved process of making metallic films or flakes, consisting generally in plating an exceedingly thin film of zinc on a copper or nickel cathode, in then plating thereon a film of the desired metal, such as cobalt or nickel, or cobalt-nickel alloy, and in finally, immersing the cathode in a suitable acid, such as hydrochloric acid for the purpose of attacking the zinc, thereby detaching the desired metallic film, and by reason of the evolution of hydrogen gas, facilitating the sub-division of the film into small flakes presenting curved or curled shapes, the latter phenomena being especially characteristic of cobalt.

My present invention relates to an improved apparatus for carrying such process into effect which apparatus presents details of construction by which additional refinements in the process can be utilized.

Generally speaking, the apparatus comprises a series of tanks or receptacles in which the several steps of the process are adapted to be performed, a cathode movable into and out of the several receptacles and means for preferably rotating the cathode whereby the plating and washing operations to be described will be facilitated, all as I will more fully hereinafter describe and claim.

The object of my invention is to present an improved apparatus by which metallic films or flakes can be expeditiously made on a practical scale in large quantities and at low cost. In the accompanying drawings, forming part of this specification, I illustrate one form of apparatus which I have practically used for this purpose, wherein a single cathode and a single tank or receptacle for each operation is employed, but it will be of course obvious that these elements may be duplicated indefinitely, suitable provision being made for simultaneously moving all the cathodes and introducing them within or removing them from the several tanks or receptacles.

In the drawings: Figure 1, is a side elevation of the apparatus, a portion near the bottom being broken away; Fig. 2, a section on the line 2—2 of Fig. 1, looking downward; Fig. 3, a detail of the lock at the upper end of the supporting shaft; Fig. 4, a section on the line 4—4 of Fig. 3; and, Fig. 5, a diagrammatic view of the several circuits.

In all of the above views, corresponding parts are represented by the same numerals of reference.

A supporting shaft 1 is mounted at its bottom on a step-bearing 2 and at the upper end in a bearing 3. The bearing 3 is formed with a series of openings 4, with one of which a latch 5 operated by a wire 6 is adapted to engage, whereby the supporting shaft may be firmly held in any desired position of its adjustment. The shaft 1 carries a frame or bracket 7—7 in which is mounted a shaft 8 in suitable bearings, and at the lower end of said shaft is connected a cathode 9, made preferably in the form of a hollow drum of highly polished copper or nickel. A plating current is supplied to this cathode by a brush 10, and sufficient length of the conductor 11 supplying the plating current is afforded to permit the cathode to be swung around above, and to enter any one of the several tanks or receptacles to be presently described.

The cathode 9 may be rotated in any suitable way, as for instance, from a belt wheel 12 from which a belt 13 extends to a counter-shaft 14, driven from an electric motor 15. Provision is preferably made for operating the electric motor either rapidly or slowly, according to the operation which may be performed on the cathode, and for this purpose, the connections shown in Fig. 5 may be followed. The circuit 16—16 may be the ordinary lighting circuit of 110 volts, and the circuit 17—17 is a suitable plating circuit of about 10 volts. The cutout switch 18 (shown diagrammatically in Fig. 5 and actually in Fig. 1) is used to break the circuit 16—16 when desired. The field of the motor 15 as shown in Fig. 5 is connected permanently across the circuit 16—16. The armature of this motor, however, is arranged to be connected either with the circuit 16—16

or the plating circuit 17—17 by means of a two-pole switch 19, as shown. Preferably, in the high tension armature circuit, a rheostat 20 is employed. Obviously, by connecting the armature circuit to the motor armature with one or the other of the circuits 16—16 or 17—17, the armature speed can be made high or low, while by using the rheostat 20, the high speed can be varied, as desired.

In order to elevate and lower the frame 7—7 to remove the cathode 9 from and into any one of the tanks or receptacles, I make use of a hoist 21, operated by compressed air and of any approved construction. A three-way valve 22 controls the admission of air to or from this hoist in the usual way. When the cathode is to be maintained in an elevated position for sometime, to permit drying, it is locked in this position by a hook 23 to prevent waste of air in the hoist. In the preferred embodiment of the invention, the apparatus utilizes one or more of six different tanks or receptacles 24, 25, 26, 27, 28 and 29, which will be described in detail.

The tank 25 is a wash tank having spray pipes 30 supplied from a pipe 31 and controlled from a valve 32 and with an outlet 33. The spray pipes 30 are so slitted as to direct fine streams of water on the outside of the cathode and longitudinally thereof, so that as the cathode revolves, its exterior will be washed.

The tank 24 is used for the purpose of cleaning the cathode to remove any dirt, grease or oily films therefrom, or any superficial surface oxidation. This tank is therefore provided with two or more anodes 34, connected to the plating circuit 17—17, as shown and formed preferably of nickel or carbon, so that any oxygen developed thereon will not affect the same. The solution used in this receptacle is preferably a solution from which nothing can be plated out on to the cathode, preferably a twenty per cent. solution of potassium hydrate. Within this receptacle, the current serves merely to decompose the solution, liberating oxygen at the anode and large quantities of hydrogen on the surface of the cathode 9. This generation of hydrogen gas on the cathode tends to forcibly detach any dirt or oily impurities or films thereon, as well as to reduce any superficial oxidation, whereby the surface of the cathode may be made absolutely bright and clean, and hence in the best condition for effective plating.

The tank 26 is provided with a plurality of anodes 35 of metallic zinc, which anodes are connected to the plating circuits 17, as shown. The solution within the tank 26 is preferably a solution of zinc sulfate ($ZnSO_4$). Provision is made for preventing overheating of the solution in the zinc plating tank 26 by immersing a cooling coil 36 (made of lead) therein, through which water circulates from the supply pipe 31, being controlled by a valve 37. The pipe 38 connecting the valve 37 with the cooling coil 36 is formed preferably of rubber hose to prevent short circuiting through the same.

The tank 27 is used for the plating of the desired film on the cathode after the latter has been zinc plated, and I shall hereafter refer to it as the cobalt tank, although as explained, any other desired metal or alloy may be plated on the cathode, such as nickel or an alloy of nickel and cobalt. This tank is provided with a series of anodes 39, made of metallic cobalt or metallic nickel, or anodes of both metals may be used, or of any other desired metal or metals or alloys thereof. The anodes 39 are connected to the plating circuit 17, and in case anodes of more than one metal are employed, the circuit is so controlled, or the anodes are so proportioned as to secure the desired relative deposit of the several metals, as will be understood. The solution used in the cobalt tank 27 when cobalt films are to be obtained is preferably a solution of cobalt ammonium sulfate ($Co(NH_4)_2(SO_4)_2$), and when nickel films are to be obtained, a corresponding nickel ammonium sulfate solution ($Ni(NH_4)_2(SO_4)_2$) is preferably used, and when anodes of both nickel and cobalt are employed, the ammonium sulfate solutions of both metals may be used together. For other metals, the solution will, of course, vary, as will be understood. Provision is made for heating or cooling the solution of the cobalt tank by a lead coil 40 connected to the water supply pipe 31 by a rubber hose 41, the circulation being controlled by a valve 42. In practice good results have been secured by maintaining the temperature of the solution in the cobalt tank at about 120 degrees F. but this temperature may be obviously varied within considerable limits. Therefore, as the temperature of the solution in operation tends to become too high, it may be kept at the proper point by varying the amount of circulating water passed through the coil. On the other hand, in starting up the plating operation it may be sometimes necessary to heat the solution to the desired point, and this may be done by disconnecting the hose 41, connecting the same to a steam pipe, as will be obvious.

The tank 28 contains water for washing the cathode after the cobalt plating operation, in order to prevent loss of cobalt sulfate, which latter can be recovered from time to time by evaporation to crystallize out the cobalt salt. Preferably, however, the more or less dilute cobalt solution which accumulates in the water tank 28 is transferred from time to time as may be needed to replace such of the solution in the cobalt

tank 27 as may have been lost by evaporation or otherwise, clear water, of course, being introduced into the water tank 28 to take the place of the liquid removed therefrom.

The tank 29, hereafter referred to as the acid tank, contains a suitable solution of a suitable acid for attacking the zinc, without however, affecting the cobalt or other permanent film. Preferably, dilute hydrochloric acid is used for this purpose, which may be removed and replaced by fresh acid. when necessary.

In carrying out my process with apparatus having the general characteristics above described, and assuming the desired films are to be formed of metallic cobalt, I preferably proceed as follows: The cathode 9 is first introduced in the tank 24, the current is closed across the circuit 17, so as to generate large quantities of hydrogen gas on the surface of the cathode, resulting in a very perfect cleaning operation being performed therein, as I describe in my Patent No. 821,622, granted May 29, 1906. During this operation, the switch 19 connects the motor armature with the high tension circuit 16, so that the motor will rotate at high speed, as I find that in this way a more perfect cleaning is effected. From the cleaning tank 24, the cathode is removed to the wash tank 25, in which a washing operation is performed, also preferably at high speed. After the cathode has been thus cleaned and washed, it is introduced within the tank 26 and a very thin film of zinc plated thereon. This operation is also preferably performed while the cathode is rotating at high speed as I am enabled in this way to secure a very smooth deposit of zinc at a high rate and with a high current density. After this deposit has taken place, the cathode is returned again to the wash tank 25 and any adhering zinc sulfate is washed off, the cathode still being operated at the higher speed of the motor 15. From the wash tank 25 the cathode is now removed to the cobalt tank, and the switch 19 operated to close the motor armature across the plating circuit 17, so that the cathode will turn at a low speed. In this tank, a film of cobalt of the desired thickness, preferably less than one-thousandth of an inch, is plated upon the zinc, the low speed employed permitting the films to become more or less porous to allow for passage of the acid through the same, so as to readily reach and attack the zinc film. When it is desired to secure only a single film of cobalt on the cathode, the latter after being dipped in the water tank 28 to wash washed off, after which the cathode is introduced directly into the acid tank 29, whereby the acid will penetrate through the pores of the cobalt film and through the fissures presented thereby, so as to attack the zinc and result in the generation of hydrogen gas, which effects a detachment of the cobalt film in the form of relatively small flakes. Preferably, however, a large number of cobalt and zinc films are plated on the cathode, and to this end the cathode after leaving the water tank 28, is introduced again into the zinc tank 26, and a zinc film plated on the cobalt film. From the zinc tank, the cathode is returned again to the wash tank 25 and any adhering zinc sulfate washed off, after which the cathode is introduced into the cobalt tank 27, and a second layer of metallic cobalt plated upon the film of zinc. These operations are repeated until as many alternating films of zinc and cobalt, as may be desired, have been plated upon the cathode. After all the films have been thus plated on the cathode, the latter is immersed in the acid tank and a ready separation of the films and a breaking up of the cobalt films are effected. In thus detaching themselves from the cathode the entire layers of films frequently break off in large scales or flakes, whereby the individual flakes of cobalt or other metal become isolated as the zinc is attacked by the acid. When a single film of cobalt is plated, it is possible to make use of an excessively minute film of zinc upon the cathode but when more than one film is to be plated on the cathode, the deposit of zinc separating any two of the cobalt films should be preferably somewhat thicker than the film of zinc first deposited on the cathode in order to insure a more ready and complete separation of the cobalt flakes. If it were intended to make use of as thin a film of zinc on the cobalt film as might be effectively used if plated directly on the cathode, there would be danger of the zinc depositing unevenly upon the cobalt, thereby permitting the succeeding cobalt film to be deposited directly upon the previous cobalt film and resulting in an objectionable thickness of the latter, as will be obvious.

The acid treatment in the tank 29 preferably takes place when the cathode is stationary, or is very slowly rotating. The cobalt films peel off generally in small flakes, most of which float on the acid solution, owing to the large quantities of entrained hydrogen gas contained within the flakes which may be skimmed off of the solution by a sieve, or otherwise removed. These flakes are washed in a filter bag with water, dried, and annealed in a hydrogen atmosphere, so as to prevent oxidation. After this, the flakes are rubbed through a screen to reduce them to substantially uniform size, being then ready for use.

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

1. In apparatus for plating metallic films,

the combination with an electrolytic bath in which a readily soluble metal film is to be plated, and a second electrolytic bath in which a permanent film is to be plated, of a cathode arranged to be moved successively into said baths, an electric motor for rotating said cathode, and a switch for connecting the motor armature successively with a high and low tension circuit, substantially as set forth.

2. In apparatus for plating metallic films, the combination with a cathode on which the metal is plated, of an electric motor for rotating said cathode, two circuits of high and low tension respectively, a permanent connection between the field of said motor and the high tension circuit, a permanent connection between the low tension circuit and the cathode, and a switch for connecting the motor armature with either of said circuits, substantially as set forth.

3. In apparatus for plating metallic films, the combination with a rotatable cathode on which the metal is deposited electrolytically, of a support for said cathode, movable horizontally, means for elevating and lowering said support, and a source of power and a permanent connection between said source and said cathode, substantially as set forth.

4. In apparatus for plating metallic films, the combination with an electrolytic bath, a rotatable cathode on which the metal is deposited electrolytically, a support for the cathode movable horizontally, means for lowering and elevating said support to immerse the cathode into said bath and remove it therefrom, and means carried by the support for rotating said cathode throughout the whole period of immersion, substantially as set forth.

5. In apparatus for plating metallic films, the combination with a series of tanks, of a rotatable cathode movable into and out of said tanks successively, a horizontally movable support for the cathode, spring pressed means for locking said support in successive positions corresponding to said tanks, and means for rotating said cathode throughout the whole period of immersion, substantially as set forth.

6. In an electro-plating apparatus, the combination of a rotatable drum-like cathode, a vertically movable support therefor, and means for rotating said cathode comprising a motor mounted on said support, substantially as set forth.

7. In an electro-plating apparatus, the combination of a support arranged to rotate on a vertical axis, a rotatable drum-like cathode carried thereby, a series of tanks arranged in a circle concentric to the axis of said vertical support, and means for raising and lowering said cathode, substantially as set forth.

8. In an electro-plating apparatus, the combination of a vertical shaft, a frame mounted upon said shaft and extending outwardly therefrom, means for raising and lowering said frame, a drum-like cathode rotatably mounted in said frame, and a motor for rotating said cathode, substantially as set forth.

9. In an electro-plating apparatus, the combination of a frame capable of movement about a vertical axis, means for raising and lowering said frame, a cathode rotatably mounted in said frame, and means for locking said frame against rotation, substantially as set forth.

10. In an electro-plating apparatus, the combination of a frame capable of movement about a vertical axis, means for raising and lowering said frame, a cathode rotatably mounted in said frame, and a spring-pressed bolt for locking said frame against rotation, substantially as set forth.

11. In an electro-plating apparatus, the combination of a vertically movable frame, a drum-like cathode rotatably mounted in said frame, an electric conductor secured to said frame and making a rubbing contact with said cathode, and means for rotating said cathode, substantially as set forth.

12. In an electro-plating apparatus, the combination of a cathode supported so as to be capable of movements of rotation and translation, two sources of electric current of different potential, an electric motor for rotating said cathode, a permanent connection between the circuit of lower potential and the cathode, and means for connecting the said motor with either of said sources of current, substantially as set forth.

13. In an electro-plating apparatus, the combination of a cathode supported so as to be capable of movements of rotation and translation, a source of current of low potential connected to said cathode, an electric motor for rotating the cathode, a source of current of higher potential, and means for connecting said motor with either of said sources of current for driving said motor at different speeds, substantially as set forth.

14. In an electro-plating apparatus, the combination of a movable cathode, an electric motor for moving said cathode in the bath, a source of current for plating, a second source of current, and means whereby current from either source may be passed through the motor, substantially as set forth.

15. In an electro-plating apparatus, the combination of a movable cathode, an electric motor for moving said cathode in the bath, a source of current for plating, a second source of current, and means whereby current from either source may be passed through the motor armature, substantially as set forth.

16. In an electro-plating apparatus, the

ode, an electric motor for rotating said cathode, a source of current for plating, a separate source of current for exciting the field of said motor, and means whereby the plating current may be passed through the motor armature, substantially as set forth.

17. In an electro-plating apparatus, the combination of a rotatable cathode, an electric motor comprising two elements; field and armature, two sources of electric current, means for directing one of the currents through an element of the motor and means for directing the other current through the remaining motor element and the cathode, substantially as set forth.

This specification signed and witnessed this 10th day of October 1905.

THOS. A. EDISON.

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

Corrections in Letters Patent No. 950,227.

It is hereby certified that in Letters Patent No. 950,227, granted February 22, 1910, upon the application of Thomas A. Edison, of Llewellyn Park, Orange, New Jersey, for an improvement in "Apparatus for Making Metallic Films or Flakes," errors appear in the printed specification requiring correction as follows: Page 3, line 61, the words "washed off, after which the cathode" should be stricken out and the words *off any adhering cobalt solution* inserted instead; page 4, line 128, after the article "the" the words *combination of a rotatably supported cath-* should be inserted; and that the said Letters Patent should be read with these corrections therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 22d day of March, A. D., 1910.

[SEAL.]

C. C. BILLINGS,
Acting Commissioner of Pa