

GOVERNMENT OF INDIA : THE PATENT OFFICE, 214, LOWER CIRCULAR ROAD, CALCUTTA-17.

Specification No. 107624. Application No. 107624, dated 25th October 1966. Complete—
Specification left on 23rd August, 1967. (Application accepted 24th July 1968).

Index at acceptance—7002[LVIII(5)], 103[XLV(I)]. 144A[XII(3)].

PROVISIONAL SPECIFICATION

IMPROVEMENT IN OR RELATING TO ANODIC PHOSPHATING.

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH, RAJI MARG, NEW DELHI-1, INDIA, AN INDIAN REGISTERED BODY INCORPORATED UNDER THE REGISTRATION OF SOCIETIES ACT. (ACT XXI OF 1860).

The following specification describes the nature of this invention.

THIS IS AN INVENTION BY KUMMATTITHIDAL SANTHANAM RAJAGOPALAN, SCIENTIST, CHAKRAVARTHI RAJAGOPAL, JUNIOR SCIENTIFIC ASSISTANT AND BALASUBRAMANIAM DANDAPANI, JUNIOR SCIENTIFIC ASSISTANT ALL OF THE CENTRAL ELECTROCHEMICAL RESEARCH INSTITUTE, KARAİKUDI-3, INDIA, ALL INDIAN CITIZENS.

This invention relates to Improvement in or Relating to Anodic Phosphating.

A new method of electro phosphating called Anodic Phosphating was proposed in an earlier patent (Indian Patent No. 96133). The new method consisted in making the metal to be phosphated as anode in electrolytic bath containing alkali metal phosphate solution as the electrolyte and by adjustment of metal solution potential at an appropriate value by means of a potential controlling instrument e.g., electronic potentiostat, stabilised D.C. voltage supply.

The drawback of the earlier invention is that it requires potential—controlling equipment, which are costly.

The object of the present invention is to show that phosphating to get any desired coating weight upto 700 mg/sq. decimetre can also be carried out at constant current.

The invention consists in the development of a constant current method of Anodic Phosphating wherein phosphating is carried out by passing a constant current through the object as anode in a steel tank containing the preferred electrolyte. The tank itself may be used as cathode or separate cathodes may be employed. Any other arrangement such as electrolytic brush coating, electrolytic barrelling can also be used.

Alkali metal phosphate solution is the electrolyte preferred. However, solutions of heavy metal phosphate can also be employed. The phosphoric acid is preferred for adjustment of pH to the desired value. However, other acids e.g., nitric acid can also be used. The ranges to be employed in respect of concentration, current density, pH etc., are :

concentration of phosphate solution ... 1 to 10% (weight by volume)
pH ... 3.5 to 6
current density ... 1 mA to 25 mA/cm²
temperature ... 25 to 50°C.

The time of treatment to be employed and the coating weight obtained are illustrated below :—

Bath No.	Time of Treatment (minutes)	Coating weight mgm sq. dm.	Cost of treatment Paise/sq. dm.
I	25	50	0.05
II	20	70	0.05
III	40	100	0.08
IV	60	200	0.25
V	8	500	0.5

The compositions of the baths I to V are given below :—

Bath No.	Concentration of phosphate solution weight volume	Concentration of phosphoric acid weight volume
I	5.0	1.8
II	5.0	1.4
III	2.5	0.8
IV	2.5	0.9
V	5.0	1.8

The following are the advantages of the improved processes :—

- (1) One such equipment as is necessary for carrying out electro-plating has to be used.
- (2) Any coating weight upto 700 mgm/sq.dm., can be obtained by choosing suitable bath conditions as indicated above.
- (3) Low recurring cost of phosphating as compared to known phosphating processes, though initial investment on equipment to supply D.C. power is necessary.
- (4) The same bath may be used indefinitely provided pH is maintained by addition of phosphoric acid and periodical removal of sludge is carried out.
- (5) Any type of steel surface or article made of steel can be phosphated to get any desired coating weight.

Phosphate coating obtained by this process can be made use of to increase paint life, improve adhesion of paint to metal surface and reduce friction in wire making, tube making, body pressing and other deep drawing operations as well as reduce wear and galling of mating surfaces.

R. BHASKAR PAI,

Patents Officer.

Council of Scientific and Industrial Research.

Dated this 17th day of October, 1966.

COMPLETE SPECIFICATION.

IMPROVEMENT IN OR RELATING TO ANODIC PHOSPHATING.

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH, RAJI MARG, NEW DELHI-1, INDIA, AN INDIAN REGISTERED BODY INCORPORATED UNDER THE REGISTRATION OF SOCIETIES OF ACT (ACT XXI OF 1860).

The following specification particularly describes and ascertains the nature of this invention and the manner in which it is to be performed.

This is an invention by KUMMATTITHIDAL SANTHANAM RAJAGOPALAN, Scientist, CHAKRAVARTHI RAJAGOPAL, Junior Scientific Assistant and BALASUBRAMANIAM DANDAPANI, Junior Scientific Assistant, all of the Central Electrochemical Research Institute, Karaikudi-3, India, all Indian citizens.

3. This invention relates to improvement in or relating to Anodic Phosphating.

4. Phosphate coatings are widely employed to improve the adhesion of paint to metal and retard spreading of corrosion under paint film and reduce friction in

wire-making, tube-making, body pressing and other deep drawing operations. Only chemical phosphating processes based on solutions of heavy metal phosphate have found commercial application. Attempts made to produce phosphate coatings from alkali metal phosphates

Price : TWO RUPEES.

have met with limited success. It was shown earlier (Indian Patent No. 96133) that by making the metal to be phosphated as anode in alkali metal phosphate solution and by adjustment of metal-solution potential at an appropriate value by means of potential controlling instrument e.g., electronic potentiostat, stabilised D.C. voltage supply, phosphating can be carried out and this new method was referred to as Anodic Phosphating.

5. The draw back of this earlier invention is that it requires potential-controlling equipment which is costly.

6. The object of the present invention is to show that Anodic Phosphating can also be carried out at constant current.

7. The main finding is that phosphate-coating in the range of 50 mg., to 500 mg., per sq. decimetre can be produced from cold solution of phosphate by making the metal anode and passing a constant current for a given period.

8. The new result is that an electrolyte like sodium phosphate which is much cheaper than heavy metal phosphate may be used for producing phosphate coatings having desirable characteristics.

9. Cold rolled bright steel can be phosphated by the new method after degreasing. Steel carrying mill-scale or rust can be pickled in cold HCl or hot sulphuric acid and thoroughly rinsed free of acid can be phosphated by the new method.

10. The invention consists in the development of a constant current method of Anodic Phosphating wherein phosphating is carried out by passing a constant current through the object as anode in an steel tank containing sodium phosphate solution. The tank itself may be used as cathode or separate cathodes may be employed. Any other arrangements such as electrolytic brush coating, electrolytic barrelling can also be used. The sludge formed is removed by circulating the solution through a filter press. pH of bath can be regulated by circulating a small portion of the solution through an ion exchange column and mixing into the main lot. The concentration, current density cell voltage and pH ranges to be employed are:—

Concentration of sodium phosphate solution (w/v)	...	1-10%
pH	...	3.5-6
current density	...	1 mA to 25 mA
cell voltage	...	1.5 V-2.5 V/cm ²
temperature	...	25-50°C.

11. Examples :

The compositions of baths 1-5, the time of treatment to be employed in each of these baths coating weight obtained and the cost of treatment are illustrated in Table I.

TABLE I.

Bath No.	Concentration of Phosphate solution (w/v)	Concentration of Phosphoric acid %(w/v)	Current density mA/sq. cm.	Cell voltage Vol.	Energy consumption per sq. cm. milli-watts.	Time of treatment.	Coating weight mgm./treat. sq. cm.	Cost of treatment.
1	5	1.8	3.75	1.5	5.625	25	50	0.05
2	5	1.4	2.5	1.5	3.75	20	70	0.05
3	2.5	0.8	5	1.5	7.5	40	100	0.08
4	2.5	0.9	3.75	1.5	5.625	60	200	0.25
5	5	1.8	2.5	1.5	3.75	80	500	0.5

12. Main advantages of the invention :

(1) Sodium phosphate which is a by-product of other industries can be used as electrolyte in the im-

proved process in place of heavy metal phosphates used in chemical phosphating.

- (2) The improved process is carried out at room temperature, while chemical phosphating requires heating to 160-170°F.
- (3) Treatment cost of the improved process is low compared to processes using heavy metal phosphates as shown by following Table II.
- (4) The improved process can be applied to all types of steel surfaces free from rust, mill scale grease and other contaminants.
- (5) By the improved process, phosphate coatings in the range 50-500 mgms/sq.dm., can be obtained.

TABLE II

Phosphating of	Cost/ft. ² surface treated in paise			
	Anodic Phosphating		Chemical Phosphating	
	Total Cost	Chemical Cost	Total Cost	Chemical Cost
Steel cabinets (components)	3.0	1.0	9.4	7.8
Fabricated parts (for bicycles)	7.0	1.5	12.5	8.0

It is shown that by passing a current in the range of 1 mA to 25 mA per sq.cm. through the item to be phosphated, in sodium phosphate solution held in a steel tank, in accordance with our invention, phosphate coatings from 50 mgms. per sq. decimetre to 500 mgms per sq. decimetre can be produced. The cost of phosphating is reduced by using a cheap by-product viz., tri-sodium phosphate as the electrolyte in place of heavy metal phosphates.

We claim :

1. A method of phosphate coating of steel by anodic treatment in sodium phosphate solution wherein the metal part to be phosphated is made anode which is characterised in that the anodic treatment is carried out by passing a known current in the range of 1 mA to 25 mA per sq. cm., through the metal part to be phosphated.

2. A method as claimed in Claim 1, when carried out at cell voltages as low as 1.5 to 2.5 V.

3. A method as claimed in Claim 1 wherein the tank itself is used as cathode or separate steel cathodes are provided.

4. A method as claimed in Claim 1 when employed for phosphating of small metal parts by using a rotating barrel holding the phosphating solution and loaded with the small metal parts to be phosphated.

5. A method as claimed in Claim 1 when employed for touching up phosphated metal parts (at places where phosphate coating is not fully formed or removed) by using an absorbant pad carrying the phosphate solution and the cathode as in brush plating.

6. A method as claimed in Claim 1 wherein sludge is formed which is removed by circulation of the solution through filter press.

7. A method as claimed in Claim 1 wherein the pH of the bath is regulated by circulating a portion of the solution through an ion exchange column and mixing it with the main lot.

R. BHASKAR PAI,

Patent Officer,

Council of Scientific and Industrial Research.

Dated this 17th day of August 1967.