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### PROVISIONAL SPECIFICATION

Index at acceptance—70C6[LVIII(5)] 103[XLV(1)]

“IMPROVEMENTS IN OR RELATING TO ELECTROPHOSPHATING PROCESS FOR THE PRODUCTION OF PHOSPHATE COATING ON STEEL”

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH, RAFI 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, Narayanaswamy KRITHIVASAN, Senior Scientific Assistants, Rengachari SRINIVASAN, Scientist, Mohamad Abdullah TAJUDEEN, Anayath UNNIKRISHNAN, Senior Laboratory Assistant, Handady Venkatakrishna UDUPA, Director all from Central Electrochemical Research Institute, Karaikudi and Indian Nationals.

HITHERTO IT HAS BEEN PROPOSED THAT Anodic Phosphating can be carried out by making the metal e.g., steel, zinc as anode in an alkali metal phosphating solution and adjusting its potential to the desired value by means of a potential controlling equipment e.g., potentiostat and this is covered by specification No. 96133 (1964).

Anodic Phosphating can also be carried out by passing a constant current through the metal e.g., steel, zinc as anode and dipping in a solution of trisodium phosphate, the pH of which has been previously regulated by passing through an ion exchange column and this is covered by specification No. 107624 (1966).

THIS IS OPEN TO THE OBJECTION THAT

- (a) for the phosphating of a large number of sheets or fabricated items at the same time, a current which is directly proportional to the area of the metal surface to be treated has to be passed necessitating the use of high current low voltage rectifiers e.g., 8 V 1000 amp. and heavier conductors with a high current carrying capacity and
- (b) the cost of fabrication of the phosphating tanks is increased by incorporation of cathode screens to enable parallel distribution of current.

THE OBJECT OF THIS INVENTION IS TO OBVIATE THESE DISADVANTAGES BY the adoption of bipolar electrode arrangement. In this arrangement the electrical connections are given only to the two end electrodes of the electrolytic cell and the current flowing between these end electrodes is made to pass through the items to be phosphated e.g., steel sheets by suspending the items between the end electrodes. The use of the bipolar electrode arrangement results in the application of much smaller currents to phosphate the same area in series when current passes from one end electrode to the other thereby making it possible to use lighter conductors (2 sq. cm, as against 15 sq. cm, in the older process) for carrying the current and high voltage low current rectifiers or A.C. transformers. The cost of fabrication of the phosphating tank is reduced to half since cathode screens are not required and the volume of the phosphating tank can be more fully utilised.

TO THESE ENDS, THE INVENTION BROADLY CONSISTS IN passing A.C. or D.C. current through two end electrodes placed in an electrolytic cell containing the solution specified in Indian Patent No. 96133 (1964) or 107624 (1966) and dipping the items to be phosphated e.g., steelsheets, fabricated items of steel or zinc such that these items are regularly arranged between the end electrodes. The terminals from A.C. or D.C. source are connected to the end electrodes. In the case of D.C. the current

corresponding to 1 ma/sq.cm. to 25 ma/cm<sup>2</sup>, of one side of the item to be phosphated (preferably 2 to 10 mA/sq., cm.,) is passed through the cell for the duration (20 to 30 minutes) as specified in Indian Patent No. 107624. This current is passed in one direction for the specified time and the same current is passed in the opposite direction for the same time when the other side also gets phosphated, if phosphating of both sides is required. The voltage required for D.C. is approximately 1-3 volt/sheet or fabricated item arranged in the phosphating tank, the actual voltage drop being fixed by the inter electrode distance. When A.C. is used the current corresponding to 10 to 100 mA/sq. cm., (preferably 15 to 50 mA/sq. cm.,) of one side of the item to be phosphated is passed, at which values of A.C. current D.C. component of the potential falls within the desired range. The A.C. current is passed for a duration of 15 to 30 minutes. The A.C. voltage drop required is approximately 2-6 volts/sheet or fabricated item arranged in the phosphating tank, the actual voltage drop being fixed by the inter electrode distance.

THE FOLLOWING TYPICAL EXAMPLES ARE GIVEN TO ILLUSTRATE THE INVENTION :

#### Example 1

Phosphating solution to Specification No. 96133 or 107624 taken in a 2½ litre PVC container, 15 cm., × 10 cm., end electrodes of steel are inserted along the walls of the container. In the centre, steel plate to be phosphated was inserted such that 12½ cm. × 10 cm., of steel specimens (approx.,) immersed in the solution. The end electrodes were connected to the D.C. source and 0.46 Amps., passed for a period of 30 minutes in one direction and another 30 minutes in the opposite direction. The voltage drop through the cell was 3 volts. The central plate was taken out and examined. A coating of 7 gms/sq., metre was obtained.

#### Example 2

Phosphating solution to specification No. 96133 or 107624 was taken in a 400 c.c., PVC container and 7.5 cms., × 5 cms., end electrodes were inserted. In the centre 4 Nos., of 7.5 cms., × 5 cms., of steel plates were suspended on a PVC rod. 1.5 Amps., A.C., was passed through the cell. The voltage drop was 9 volts at the end of 15 minutes. Plates were taken out and examined for phosphating. A coating of 7 grams/sq. metre was obtained in each plate.

THE FOLLOWING ARE THE MAIN ADVANTAGES OF THE INVENTION :—

- (1) By the use of the bipolar electrode arrangement, current to be drawn from D.C. or A.C. source of supply is reduced and lighter conductors can be used for electrical connections.
- (2) Area of steel item phosphated per unit volume of phosphating tank is increased.
- (3) Capital expenditure on current source can be reduced by 30 to 40% when A.C. is used.

(4) Time of phosphating is reduced when the A.C. is employed.

(5) Phosphating could be restricted to one side without resorting to marking when the bipolar arrangement is used with D.C.

Dated this 1st day of September, 1971.

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## COMPLETE SPECIFICATION

### "IMPROVEMENTS IN OR RELATING TO ELECTROPHOSPHATING PROCESS FOR THE PRODUCTION OF PHOSPHATE COATING ON STEEL"

COUNCIL OF SCIENTIFIC & 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 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, Senior Scientific Assistant, Narayanaswamy KRITHIVASAN, Senior Scientific Assistant, Rengachari SRINIVASAN, Scientist, Mohammed Abdullah TAJUDEEN, Anayath UNNIKRISHNAN, Senior Laboratory Assistants, Handady VENKATAKRISHNA UDUPA, Director — all from Central Electrochemical Research Institute, Karakudi-3, Tamil Nadu, India and Indian citizens.

This invention relates to Improvements in or relating to electrophosphating process for the production of phosphate coating on steel.

Hitherto it has been proposed that phosphating can be carried out by making metals like steel and zinc as anode in alkaline metal phosphating solution and passing direct current through the solution and the specification is covered by Indian Patent No. 96133 (1964) and 107624 (1966).

This is open to the objection that (a) the current required is directly proportional to the area of the metal surface to be treated, (b) high current low voltage rectifiers involving high cost are required for carrying out the operation, (c) conductors with high current carrying capacity are required.

The object of the present invention is to obviate these disadvantages.

We have found that this can be achieved by the adoption of bipolar electrode arrangements.

According to the present invention, there is provided a process of electrophosphating for the production of phosphate coating on steel items which comprises of introducing two end electrodes in an electrolytic cell containing sodium phosphate solution (according to I.P. No. 107624) and introducing the item to be phosphated between the two end electrodes and passing a current (A.C. or D.C.) in the range of 3 mA/sq.cm., to 50 mA/sq.cm. in the bipolar arrangement for a period of 15 to 45 minutes.

The items to be phosphated need not be given any electrical connection individually.

Thus steel sheets to be phosphated are arranged in the bipolar system in the phosphating tank made of P.V.C. or F.R.P. or any insulating material or lined with insulating material and current (AC or DC) is passed through the end electrodes for a period of 15-45 minutes at a current density of 3 mA per sq.cm., to 50 mA per sq.cm. thereby producing phosphate coated steel sheets.

Thus steel tubes to be phosphated are arranged in the phosphating tank made of P.V.C. or F.R.P. or any insulating material or lined with insulating material such that the tubes are surrounded by bigger perforated steel tubes and steel rods introduced centrally, all the items resting on an insulated base and passing current (AC or DC) in the bipolar arrangement for a period of 15-45 minutes at a current density of 3 mA per sq.cm., to 50 mA per sq.cm., thereby producing phosphate coated steel tubes.

In this arrangement electrical connections are given to the end electrodes immersed in the solution used for phosphating and the items to be phosphated are immersed in the phosphating solution and current passed between these end electrodes.

Steel sheets and tubes are phosphated by introducing one or more numbers of these items between the end electrodes in an electrolytic cell and connected to an A.C. source of supply.

The full holding capacity of the electrolytic cell can be fully utilised and no electrical connection to the items phosphated are required.

The invention will now be described with the help of accompanying drawings wherein Figure 1 shows the set-up employed. Two end electrodes (1) are introduced in the electrolytic cell (2) containing sodium phosphate solution according to (I.P. No. 107624). The item (3) to be phosphated is introduced between the two end electrodes and a current (A.C. or D.C.) in the range of 3 mA/cm<sup>2</sup> to 50 mA/cm<sup>2</sup> is passed by connecting the end electrodes to the A.C./D.C. source of supply (not shown) for a period of 15-45 minutes.

The items to be phosphated need not be given any electrical connection individually.

#### Example 1

Phosphating solution according to Specification No. 96133 or 107624 is taken in a 2½ litre PVC container. 15 cm × 10 cm., end electrodes of steel are inserted along the walls of the container. In the centre, one 12½ cm. × 10 cm., specimen was inserted such that the steel specimen was immersed in the solution. The end electrodes were connected to the D.C. source and 0.46 Amps., passed for a period of 30 minutes in one direction and another 30 minutes in the opposite direction. The voltage drop through the cell was 3 volts. The steel specimen was taken out and examined. A coating of 7 gms/sq., metre was obtained.

#### Example 2

Phosphating solution according to specification No. 96133 or 107624 was taken in a 400 c.c. PVC container and 7.5 cms. × 5 cms., end electrodes were inserted at the two ends of the cell. In the centre 4 Nos., of 7.5 cms. × 5 cms., of steel specimens provided with dispersion holes at the top, were immersed such that the specimens do not touch each other. 1.5 Amps., A.C. was passed through the cell. The voltage drop was 9 volts at the end of 15 minutes. Specimens were taken out and examined for phosphating. A coating of 7 gms/sq., metre was obtained in each plate.

The main advantages of the invention are that by the use of bipolar electrode arrangement ferrous items can be phosphated by the anodic phosphating method covered by I.P. No. 96133 (1964) and 107624 (1966) by connecting the end electrodes to the A. C. or D. C. source of supply and immersing the items to be phosphated between these end electrodes.

**NOTEWORTHY FEATURES :**

- (a) the area of ferrous items phosphated by the anodic phosphating process is higher for the new process than what has been claimed for Anodic Phosphating earlier,
- (b) the capital expenditure on power supply equipment can be reduced by the use of A. C. by about 30%,
- (c) where necessary only one side can be phosphated by the new process, and
- (d) phosphating time can be reduced by the new process.

**WE CLAIM :**

1. A process of electrophosphating for the production of phosphate coating on steel items such as herein described consists in introducing two end electrodes in an electrolytic cell containing sodium phosphate solution (according to I.P. No. 107624) and introducing the item to be phosphated between the two end electrodes and passing a current (A.C. or D.C.) in the bipolar arrangement in the range of 3 mA/sq.cm. to 50 mA/sq.cm. for a period of 15 to 45 minutes.

2. A process as claimed in claim 1 wherein the items to be phosphated are not given any electrical connection individually.

3. A process as claimed in claim 1 above wherein steel sheets to be phosphated are arranged in the bipolar system in the phosphating tank made of PVC or F.R.P. or any insulating material or lined with insulating material and current (AC or DC) is passed through the end electrodes for a period of 15-45 minutes at a current density of 3 mA per sq.cm., to 50 mA per sq.cm. thereby producing phosphate coated steel sheets.

4. A process as claimed in claim 1 or 2 above wherein steel tubes to be phosphated are arranged in the phosphating tank made of P.V.C. or F.R.P. or any insulating material or lined with insulating material containing sodium phosphate solution (according to Indian Patent No. 107624) such that the tubes are surrounded by bigger perforated steel tubes and steel rods introduced centrally, all the items resting on an insulated base and passing current (AC or DC) in the bipolar arrangement for a period of 15-45 minutes at a current density of 3 mA per sq.cm., to 50 mA per sq.cm., thereby producing phosphate coated steel tubes.

5. A process of electrophosphating for the production of phosphate coated steel sheets and tubes substantially as hereinbefore described.

Dated this 28th day of June, 1972.

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No 132761 of 1971

*R.B. Pai*

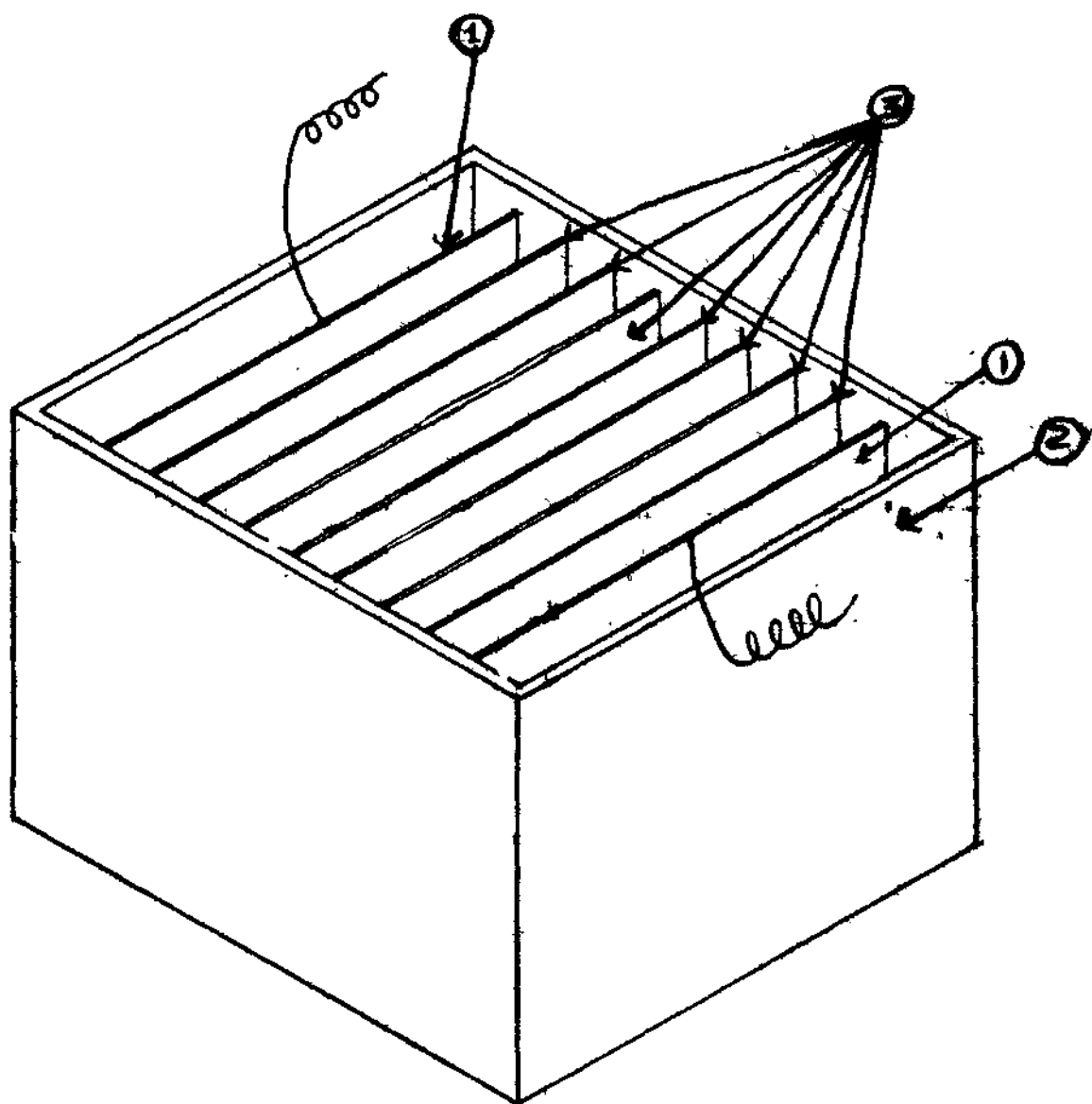


Fig. 1

*R. B. Shankaribai*

R. B. PAI  
PATENTS OFFICER,  
.S.I.R.