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


Index at acceptance—70C6(LVIII(5)).

PROVISIONAL SPECIFICATION.

IMPROVEMENTS IN OR RELATING TO OXALATE COATINGS ON STEEL

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH, RAPTI 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 KUMMATHITHIDAL SANTHANAM RAJAGOPALAN AND MARGASAHAYAM VISWANATHAN.

The invention relates to improvements in or relating to oxalate coatings on steel.

Hitherto it has been proposed that oxalate coatings on steel can be formed only by treatment of steel in proprietary solutions containing an oxidizing agent, a wetting agent, oxalate ion and other ingredients, for five minutes and longer.

This is open to the objection that—

(i) proprietary special formulations have to be employed for producing oxalate coating;

(ii) treatment time is five minutes and more; and

(iii) a black film is not obtained.

The object of this invention is to produce black oxalate coatings on steel in less than a minute.

The invention consists in electrolytic treatment of steel in oxalic acid solution. The steel is given a cathodic treatment for $\frac{1}{2}$ to 1 minute followed by anodic treatment of the duration required. The treatment can be carried out in a stainless steel tank.

The conditions to be employed are:

- Conc. of oxalic acid: 1.0% (w/w)
- Current density: 1.0 amp/sq.ft.
- Time of treatment: about 1 minute.

**EXAMPLES.**

<table>
<thead>
<tr>
<th>No.</th>
<th>Condition of steel specimen</th>
<th>Time of treatment</th>
<th>Coating weight</th>
<th>Appearance</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Ground on 1/2&quot; emery wheel</td>
<td>1 minute</td>
<td>1.009 g/m²</td>
<td>Uniform jet black coating adheres well and resistant to scratching</td>
</tr>
<tr>
<td>2.</td>
<td>Etched in HCl</td>
<td>1 minute</td>
<td>920</td>
<td>do</td>
</tr>
<tr>
<td>3.</td>
<td>Mirror polished</td>
<td>1 minute</td>
<td>950</td>
<td>do</td>
</tr>
<tr>
<td></td>
<td>Chem cut Method</td>
<td>15 minutes</td>
<td>450</td>
<td>OLIVE GREEN</td>
</tr>
</tbody>
</table>

The following are the main advantages of the invention:

1. A black coating which is useful for masking Defence equipment can be produced on steel;
2. The treatment is at room temperature unlike browning, pickling, etc.;
3. The duration of treatment is very short unlike other processes producing a black colour on steel;
4. An inexpensive chemical is used to formulate the bath.

R. BHASKAR PAL
Patent Officer,
Council of Scientific & Industrial Research.

Dated this 16th day of March 1967.

COMPLETE SPECIFICATION.

IMPROVEMENTS IN OR RELATING TO OXALATE COATINGS ON STEEL

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH, RAPTI 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 KUMMATHITHIDAL SANTHANAM RAJAGOPALAN AND MARGASAHAYAM VISWANATHAN, BOTH OF THE CENTRAL ELECTROCHEMICAL RESEARCH INSTITUTE, KARAKURU-3, INDIA, BOTH INDIAN CITIZENS.

This invention relates to improvements in or relating to oxalate coatings used in the steel industry.

Oxalate coatings on steel have been employed to facilitate cold forming operations.

Hitherto it has been proposed that oxalate coatings on steel can be formed only by treatment of steel in proprietary solutions containing an oxidizing agent, a wetting agent, oxalate ion and other ingredients for five minutes and longer and an olive green coating of 100-200 milligrams/sq.ft. is usually produced.

The treatment time of five minutes is too long for the purpose of using oxalate coatings for cold drawing of steel. The rate of production of steel wires, rods and threads is so fast that only very rapid treatment can be considered. Another drawback of the existing process is that it gives rise to coating of varying colour and black film is not obtained.

The main feature of the invention:
Uniform jet black oxalate coating having good adhesion is obtained on steel in less than a minute.

The new result—

The speed of oxalate coating formation has been increased and the time of coating formation brought down from five minutes to less than a minute and thicker coating than what can be obtained by known process is obtained obtained.

Price: TWO RUPEES.
A statement of the invention:—

The invention consists in electrolytic treatment of steel in oxalic acid solution. The concentration of oxalic acid to be used is in the range of 1-10 per cent. weight by volume. The current density to be employed is in the range of 1-10 Amp/sq.ft.

The electrolytic treatment consists in making the steel cathodic for 10-60 seconds and anodic for 10-60 seconds so that the total time of treatment is approximately one minute.

The cell voltage is maintained in the range of 1 to 5 volts.

EXAMPLES.

<table>
<thead>
<tr>
<th>No.</th>
<th>Condition of steel specimen</th>
<th>Time of treatment in minutes</th>
<th>Coating weight gm/sq. ft.</th>
<th>Appearance</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>Ground on 1 minute 1/20 emery wheel</td>
<td>1,000</td>
<td>Uniform jet black coating adheres well and resistant to scratch.</td>
<td></td>
</tr>
<tr>
<td>II</td>
<td>Etched in HCl</td>
<td>1 minute</td>
<td>900</td>
<td>do</td>
</tr>
</tbody>
</table>

Note worthy features,

(1) An electrolytic treatment of steel in oxalic acid solution which gives a uniform black oxalate coating in less than a minute.

(2) The treatment can be carried out in a stainless steel tank.

(3) The treatment can be carried out at room temperature.

We claim:

1. A process for the rapid formation of black oxalate coatings on steel by the electrolytic treatment of steel in oxalic acid solution wherein the steel is made cathodic for 10-60 seconds and anodic for 10-60 seconds so that the total time of treatment is approximately one minute.

2. A process as claimed in Claim 1 wherein the concentration of oxalic acid used is in the range of 1-10 per cent. weight by volume.

3. A process as claimed in Claim 1 or 2 wherein the current density employed is in the range of 1-10 Amp/sq.ft.

4. A process as claimed in any of the preceding claims wherein the cell voltage is maintained in the range of 1 to 5 volts.

5. A process as claimed in any of the preceding claims wherein the treatment is carried out in a stainless steel tank.

6. A process as claimed in any of the preceding claims wherein the treatment is carried out at room temperature.

R. BHASKAR PAL,

Patent Officer,

Council of Scientific and Industrial Research,

Dated this 5th day of January 1968.