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PROVISIONAL SPECIFICATION

"IMPROVEMENT IN OR RELATING TO ELECTROPOLISHING OF MILD STEEL."

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

This is an invention by

BALKUNJE ANANTHA SHENOI, SCIENTIST AND ARUMUGAM PALAMALAI, JUNIOR SCIENTIFIC
ASSISTANT, BOTH ARE INDIAN NATIONALS EMPLOYED IN THE CENTRAL ELECTROCHEMICAL
RESEARCH INSTITUTE, KARAKKODE-3, INDIA.

The following specification describes the nature of:

This invention relates to improvements in or relating to
electropolishing of mild steel.

Hitherto, it has been proposed to adopt the following electrolytes/processes for electropolishing of mild steel:

1. Phosphoric-sulfuric-chromic acid solutions
2. Phosphoric-sulfuric acid solutions (Faust)
3. Phosphoric-sulfuric-lactic acid electrolyte (Levin’s
bath and its modifications)
4. Mixtures of sulphuric and phosphoric acid + Inhibi-
tors.

The processes employing electrolytes are open to the
objectives as described below:

Chrome acid baths (1) have to be operated at a higher
temperature (65-75°C). The use of hot electrolytes leads to
corrosion of the lead cathode and celllining due to slow
attack on prolonged use. Evaporation of solution com-
ponents is harmful to the health of the operators. Short-
service life due to cathodic reduction of Cr³⁺, use of
diaphragm to prevent this reduction and additional
operating steps in the regeneration of the bath are other
disadvantages.

In the simple phosphoric-sulfuric acid solution (2),
water content is critical. Hence evaporation at the high
operating temperature (40-80°C) is practically being
55-75°C) requires control of the bath composition. The
results are not satisfactory in that the surface obtained is
not very bright (about 20% reflectivity). Relatively, longer
time of polishing is required (5-40 minutes).

Levin’s bath (3) in spite of being operated at low tempe-
rate (18-27°C) and current density (0-7 Amp/lit/lit),
requires 1-2 hours and the high concentration of lactic
acid (33% by wt.) makes it costly. Even the modified ones
with smaller amounts of lactic acid (about 20 ml/l) are operated at
relatively high temperatures (40°-60°C) and requires
initial ageing, to the extent of 5 g/l of iron.

Electrolytes (4) are operated at higher temperatures (ex-
cepting, one two-baths employed in foreign countries, con-
taining proprietary inhibitors. Thus, solutions containing
“dextrone” or “teepol” are operated at 60°C-70°C and
those containing glycerol, at 50°-70°C.

The object of this invention is to obviate disadvantages by
adding inhibitors which could be easily prepared by its
constituents by conventional methods to the phosphoric-
sulfuric acid solutions for electropolishing mild steel.

To these ends, the invention broadly consists in adding
any one of the condensation products of formaldehyde
with a number of aromatic primary amines, such as p-
sulphocian, beta naphthalmine to mixture of phosphoric
and sulphuric acids. This enables these baths to be used
for electropolishing mild steel, even at lower temperatures
(20-30°C) and relatively low current densities (25-75
A/dm²) as compared with the plain baths (i.e., phosphoric-
sulfuric acid mixtures without inhibitors). The addition of
inhibitors enables that A.C. can also be used for
polishing.

Drossing and mechanical polishing from the necessary
pretreatment before electropolishing.

The composition of the electrolyte and the anodic polishing
conditions are as follows:

<table>
<thead>
<tr>
<th>Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Phosphoric acid (Sp. gr. 1.75): 55-75% (v/v)</td>
</tr>
<tr>
<td>Sulphuric acid (Sp. gr. 1.348): 45-25% (v/v)</td>
</tr>
<tr>
<td>Water: 0-5% (v/v)</td>
</tr>
<tr>
<td>Inhibitor: 0.2-1.5% (v/v)</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Polishing conditions</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature: 34-3°C</td>
</tr>
<tr>
<td>Time: 5-10 minutes</td>
</tr>
<tr>
<td>Bath voltage: 8-23 V (approx.)</td>
</tr>
<tr>
<td>Current density: 10-100 A/dm²</td>
</tr>
<tr>
<td>Range of current density: 25-75 A/dm²</td>
</tr>
<tr>
<td>Cathode: Lead</td>
</tr>
</tbody>
</table>

First, the inhibitor is added to the measured amount of sulphuric acid, stirred and to this rest of the components
are added and stirred vigorously. The heat of mixing itself
may be sufficient to dissolve the inhibitor. If not dissolved,
the bath may be heated to be about 80°C.

The specular reflectivity of the polished mild steel surfaces
is measured using a gloss meter, with a silver mirror as the
standard and the percentage reflectivity is calculated in
each case.

The reflectivity of the polished specimens in the new baths
ranges from 30-45%. This is somewhat relatively less than
that obtainable from the chromic acid bath (40-60% approx.).

In view of the fact that the mild steel is electropolished
mainly as a preplating treatment rather than as a finishing
for appearance, the reflectivity obtainable with the new
baths may be sufficient for practical purposes. Air agita-
tion and cooling arrangements are required. To obtain
good quality of the polished surface, agitation may be
stopped in the last one or two minutes.

Price : TWO RUPEES
The following typical examples are given to illustrate the invention:

**EXAMPLE 1**

Specimens of mild steel are denatured and mechanically polished and degreased with trichloroethylene. It is then anodically polished in the following electrolyte:

- **Phosphoric acid**: 65% (v/v)
- **Sulphuric acid**: 32% (v/v)
- **Water**: 3% (v/v)
- **Inhibitor A (condensate of HCHO and p-silica)**: 0.3% (w/v)

At the same temperatures as in Example 1 these are polished at 77 A/dm² for 10 minutes and at 50 A/dm² for 5 minutes. The reflectances under these conditions are 36% and 41% respectively.

**EXAMPLE 5**

As in example 1, mild steel specimens are pretreated and electropolished in the following bath using both AC and DC:

- **Phosphoric acid**: 70% (v/v)
- **Sulphuric acid**: 30% (v/v)
- **Inhibitor B**: 0.2% (w/v)

**Polishing conditions:**
- **Temperature**: 30±3°C
- **Time**: 10 minutes

<table>
<thead>
<tr>
<th>Type of current</th>
<th>Current density (A/dm²)</th>
<th>Specular reflectance (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>AC</td>
<td>60</td>
<td>39</td>
</tr>
<tr>
<td>AC</td>
<td>75</td>
<td>42</td>
</tr>
<tr>
<td>DC</td>
<td>50</td>
<td>41</td>
</tr>
</tbody>
</table>

The following are among the main advantages of the invention.
1. The new baths employ inhibitors which could be easily prepared from its constituents by conventional methods.
2. The new baths can be operated at room temperature using either DC or AC.
3. The use of room temperature enables that cheap non-metallic materials like plastics may be used for cell linings due to the relatively lower corrosiveness.

**PATENT OFFICER**  
St. Eligible  
Council of Scientific and Industrial Research.

*DATED THIS 1ST DAY OF APRIL, 1972.*

**COMPLETE SPECIFICATION**

COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH  
RAI MAULI, NEW DELHI, INDIA, AN INDIAN  
REGISTERED BODY INCORPORATED UNDER THE REGISTRATION OF SOCIETIES ACT (ACT XXI) OF 1860

This is an invention by  
BALKUNDE ANANTHA SHENOI, SCIENTIST AND ARUMUGAM PALAMALAI, SENIOR SCIENTIFIC  
ASSISTANT, BOTH ARE INDIAN NATIONALS EMPLOYED IN THE CENTRAL ELECTROCHEMICAL  
RESEARCH INSTITUTE, KARAKORUMI-2, INDIA.

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

This invention relates to improvements in or relating to electropolishing of mild steel.

Hitherto it has been proposed to adopt the following electrolyte processes for electropolishing mild steel:
1. Phosphoric-sulphuric-chrome acid solution  
2. Phosphoric-sulphuric acid solution

The processes employing these electrolytes are open to the objections are described below:

(iii) Phosphoric-sulphuric-lactic acid electrolyte (Levin’s bath and its modifications)

(iv) Mixtures of sulphuric and phosphoric acids and inhibitors

The following are among the main advantages of the invention.
1. The new baths employ inhibitors which could be easily prepared from its constituents by conventional methods.
2. The new baths can be operated at room temperature using either DC or AC.
3. The use of room temperature enables that cheap non-metallic materials like plastics may be used for cell linings due to the relatively lower corrosiveness.
Chronic acid bath (1) have to be operated at a higher temperature (65-75°C). The use of hot electrolytes leads to corrosion of the lead cathode and cell lining due to slow attack on protracted use. Fumes from the solution components are harmful to the health of the operator. Short service life due to cathodic reduction of Cu²⁺, use of diaphragm to prevent this reduction and additional operating steps in the regeneration of the bath are the disadvantages.

In the simple phosphoric-sulphuric acid solutions (2), water content is rather critical. Hence, evaporation at the high operating temperature (40-80°C) requires rigorous control of the bath composition. The results are not satisfactory in that the surface obtained is not very bright (about 20% reflectivity). Relatively longer time of polishing is required.

In Levin's bath (3), in spite of the low operating temperature at 0-7 A/m² in current density, it requires longer time (15-20 minutes) and moreover, the higher lactic acid content (33% by wt) makes it very costly. Even the modified composition with smaller amounts of lactic acid (about 20 mol/l) are operated at relatively high temperatures (40-60°C) and requires initial aging, to the extent of 5 gpl of iron.

While bath (iv) comprising mixture of sulphuric acid and phosphoric acid with inhibitor overcomes the disadvantages of the bath's explained in the prior art (bath (i) to (iii), it still has the disadvantage that the inhibitors used in the bath are to be imported.

The object of the present invention is to overcome the disadvantages cited above by using a bath comprising sulphuric acid and phosphoric acid with an inhibitor prepared independently for the electropolishing mild steel after derusting and mechanical polishing in order to obtain a surface suitable for electropolishing.

The main finding underlying the invention is for the preparation of indigenous inhibitors which can be easily prepared from its constituents and to use these inhibitors in the phosphoric acid sulphuric acid electrolyte for electropolishing of mild steel at room temperature 30-35°C.

In the present invention, the condensation products of formaldehyde with an aromatic amine, such as para toluidine or beta naphthylamine are used as inhibitors in the phosphoric acid sulphuric acid mixture electrolyte for electropolishing of mild steel.

The electropolishing bath thus obtained by using the inhibitors of this invention can be operated at lower temperature (20-35°C) and at relatively lower current densities (25-35 A/m²) as compared with the plain baths (viz. phosphoric acid sulphuric acid mixture without inhibitors) to get good electropolishing of mild steel with good reflectivity.

Other new finding of this invention is that alternating current can also be used in addition to direct current for electropolishing mild steel using the above bath. Moreover, the polishing time required is lower for this new bath compared with the baths known earlier.

The present invention consists in adding any one of the condensation products of formaldehyde with aromatic primary amines, such as para toluidine or beta naphthylamine to a mixture of phosphoric and sulphuric acids. This enables these baths to be used for electropolishing mild steel at lower temperature (20-35°C) and at lower current densities 25-75 A/dm² as compared with the plain bath (viz. phosphoric acid sulphuric acid mixture without inhibitor of this patent). The addition of inhibitors of the present invention enables the use of AC for electropolishing mild steel after derusting and mechanical polishing.

The composition of the electrolyte and the nodice polishing are as follows:

<table>
<thead>
<tr>
<th>Composition</th>
<th>Phosphoric acid (sp. gr. 1.75)</th>
<th>55-75% (v/v)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Sulphuric acid (sp. gr. 1.34)</td>
<td>45-25% (v/v)</td>
</tr>
<tr>
<td></td>
<td>Water</td>
<td>0-5% (v/v)</td>
</tr>
<tr>
<td></td>
<td>Inhibitor</td>
<td>0.01-0.3% (v/v)</td>
</tr>
</tbody>
</table>

Polishing conditions:
- Temperature: 20-35°C
- Time: 5-10 minutes
- Bath voltage: 8-23V (approx)
- Current density: 10-150 A/dm²
- Range of current density giving the best polishing: 25-75 A/dm²
- Cathode: Lead

To the measured amount of sulphuric acid, the inhibitor is added and stirred well till complete dissolution. Later, the other ingredients of the bath are added and stirred vigorously. The heat of mixing itself may be sufficient to dissolve the inhibitor. Otherwise, the bath is heated to about 80°C.

The specular reflectivity of the polished mild steel surfaces is measured using a goniometer, with a silver mirror as the standard and the percentage reflectivity is calculated in each case. The reflectivity of the specimens after mechanical polishing ranges from 10 to 15%. The reflectivity of the polished specimens in the new baths ranges from 30-45%. This is somewhat relatively less than that obtainable from the chronic acid bath (40-60% approx.). Since mild steel is electropolished only as prepolishing operation, the reflectivity obtained from the inventive bath is sufficient for practical purposes. Air agitation and cooling arrangements are required for maintaining optimum temperature. To obtain good quality of the polished surface, agitation may be stopped in the last one or two minutes.

The following are the typical examples given to illustrate the invention and not to limit the scope of this invention:

**Example 1**

Specimens of low carbon mild steel are derusted and mechanically polished (reflectivity 12%) and degreased with trichlorethylene. It is then anodically polished in the following electrolyte:

| Phosphoric acid | 65% (v/v) |
| Sulphuric acid  | 30% (v/v) |
| Water           | 5% (v/v)  |
| Inhibitor B (condensation product HCHO and beta naphthylamine) | 0.2% (w/v) |

The polishing is carried at 30-35°C for 10 minutes, under the current densities (d.c) of 37.5 A/dm² and 50 A/dm² the reflectivities, under these two conditions being respectively 34% and 42%.

**Example 2**

Mild steel specimens are pretreated as in Example 1 and anodically polished in the following electrolyte:

| Phosphoric acid | 60% (v/v) |
| Sulphuric acid  | 40% (v/v) |
| Inhibitor B     | 0.2% (w/v) |

The polishing is carried out at 30-35°C for 10 minutes, under the current densities of 37.5 A/dm² and 50 A/dm² the reflectivities, under these two conditions being respectively 34% and 42%.

**Example 3**

Mild steel specimens are treated as in Example 1 and electrolytically polished in the following bath:

Composition:

<table>
<thead>
<tr>
<th>Composition</th>
<th>Phosphoric acid (sp. gr. 1.75)</th>
<th>55-75% (v/v)</th>
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<tr>
<td></td>
<td>Inhibitor</td>
<td>0.01-0.3% (v/v)</td>
</tr>
</tbody>
</table>
Sulphuric acid : 35% (v/v)
Phosphoric acid : 65% (v/v)
Inhibitor B: condensation
product of HCHO and beta naphthylamine : 0.15% (w/v)

At 30±3°C they are polished for 10 minutes at a current density (A/dm²) of 25 A/dm² for 5 minutes, at 50 A/dm² the specular reflectances being 31% and 40% respectively.

**EXAMPLE 4**

Specimens of mild steel are pretreated as in Example 1 and polished anodically in the following bath :

Phosphoric acid : 65% (v/v)
Sulphuric acid : 32% (v/v)
Water : 3% (v/v)
Inhibitor A : (condensation product of HCHO and p-toluidine) 0.2% (w/v)

At the same temperature as in Example 1, these are polished at a current density (A/dm²) of 37.5 A/dm² for 10 minutes and at 50 A/dm² for five minutes. The reflectances under these conditions are 36% and 41% respectively.

**EXAMPLE 5**

As in Example 1, mild steel specimens are pretreated. Electropolishing is done in the following bath using AC or DC :

Phosphoric acid : 70% (v/v)
Sulphuric acid : 30% (v/v)
Inhibitor B : 0.2% (w/v)

**Polishing conditions :**

Temperature : 30±3°C
Time : 10 minutes

<table>
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<tr>
<th>Type of current</th>
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</tr>
</tbody>
</table>

The following are among the main advantages of the invention :

1. The new baths employ inhibitors which could be easily prepared from their constituents by conventional methods.
2. The new baths can be operated at room temperature using either DC or AC.
3. The use of room temperature enables the use of cheap non-metallic materials like plastics for cell-lining. In this invention condensation products of formaldehyde with beta naphthylamine or para toluidine are used as inhibitors in the conventional phosphoric and sulphuric acid bath for electropolishing of mild steel. This enables the bath to operate at room temperatures (30–35°C) unlike the conventional baths and AC or DC can be used at comparatively low current densities to get polishing suitable for electropolishing.

We claim :

1. A process for electropolishing of mild steel in a bath comprising sulphuric acid and phosphoric acid characterized in that an inhibitor is added which is a condensation product of formaldehyde and beta naphthylamine or formaldehyde and para toluidine.
2. A process for electropolishing of mild steel using a bath as claimed in Claim 1 wherein the sulphuric acid concentration ranges from 25–40% phosphoric acid 60–75%. Water 0–5% and the inhibitor concentration ranges from 0.01% to 1% w/v
3. A process as claimed in claims 1 and 2 wherein the electropolishing bath is operated at 20–35°C using a current density of 25–75 A/dm².
4. A process as claimed in claims 1 to 3 wherein electropolishing of mild steel can be carried out using direct or alternating current.

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