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IMPROVEMENTS IN OR RELATING TO THE FORMATION OF LEAD DIOXIDE ON LEAD AND ITS ALLOYS IN CHLORIDE MEDIUM.

PROVISIONAL SPECIFICATION

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

The following specification describes the nature of this invention.

THIS IS AN INVENTION BY HANDADY VENKATAKRISHNA UDUPA, DIRECTOR AND KAPISTHALAM CHETLUR NARASIMHAM, SCIENTIST, BOTH OF THE CENTRAL ELECTROCHEMICAL RESEARCH INSTITUTE, KARAIKUDI, INDIA, BOTH INDIAN CITIZENS.

This invention relates to improvements in or relating to the formation of lead dioxide on lead and its alloys in chloride medium.

Hitherto it has been the practice to form lead dioxide on lead and its alloys in chloride medium by introducing a platinum microelectrode into the lead surface and these have been used as anodes for cathodic protection.

The objection to the current practice is that a costly material like platinum has to be employed as a microelectrode in order to have lead dioxide formation on lead in chloride medium.

The object of this invention is to obviate the above disadvantage by suitably introducing a cheap solid lead dioxide microelectrode into the lead or lead-alloy surface.

To these ends, the invention broadly consists in the introduction of lead dioxide microelectrode in the place of platinum into lead or lead alloy surfaces. The lead dioxide microelectrode is prepared from solid pieces of lead dioxide deposited from lead nitrate—copper nitrate bath on a suitable substrate like graphite, stainless steel or nickel (as described in Indian Patents 66185 and 124215) and were ground on the emery belt of 60 and 100 to the desired size and shape. Alternatively the microelectrode is prepared by exposing the desired size on the substrate and stopping off the rest of the portion during electrodeposition of lead dioxide from nitrate bath. Suitable holes are drilled on the lead surface and the lead dioxide microelectrodes are pressed to fit them tightly in the holes. When the lead or lead alloy having the lead dioxide microelectrode is polarised in 0·5 to 3% sodium chloride or synthetic sea water, at anode current densities of 0·5 to 6 amp/dm^2, temperature of 15 to 50°C, the lead dioxide is formed on the surface of the lead or lead alloys. The ratio of area of lead surface to lead dioxide microelectrode can vary from 10 to 600 to get good deposit.

The following typical Examples are given to illustrate the invention ;

Example 1

Anode	Lead
Area of anode (cm^2)	2·2
Area of lead dioxide microelectrode (cm^2)	0·12
Bath voltage (volts)	3·65
Anode current density (amp/sq.dm.)	3
Temperature (°C)	30± 1

Example 2

Anode	Lead
Area of anode (cm^2)	19·54
Area of microelectrode (cm^2)	0·26
Anode current density (amp/sq.dm.)	2·3
Bath voltage (volts)	4·2—7·0
Temperature (°C)	30—32
Weight loss in 3% NaCl after 512 hours (mg/cm^2/A hr.)	0·330
Weight loss in synthetic sea water after 483 hours (mg/cm^2/A-hr.)	0·072

Example 3

Anode	Lead-antimony (6%)
Area of anode (cm^2)	19·54
Area of microelectrode (cm^2)	26
Anode current density (amp/sq.dm.)	2·3
Bath voltage (volts)	4·2—6.0
Temperature (°C)	30—32
Weight loss in 3% NaCl after 445 hrs. (mg/cm^2/A-hr.)	0·035
Weight gain in synthetic sea water after 482 hours (mg/cm^2/A-hr.)	0·071

Example 4

Anode	Lead-silver (1%)
Area of anode (cm^2)	20·33
Area of microelectrode (cm^2)	0·252
Anode current density (amp/sq.dm.)	2·5
Bath voltage (volts)	4·1—4·5
Temperature (°C)	30—35
Wt. loss in 3% NaCl after 524 hrs. (mg/cm^2/A-hr.)	0·180
Wt. gain in synthetic sea water after 482 hours (mg/cm^2/A-hr.)	0·066

The following are among the main advantages of the invention :

(1) A protective film of lead dioxide can be formed on lead and lead alloys in chloride solutions by incorporating lead dioxide microelectrodes into the surface instead of costly platinum microelectrodes.

(2) The ratio of lead to lead dioxide can vary widely to get lead dioxide film on to the surface of lead—lead dioxide bielectrodes as in the case of platinum microelectrode.

(3) Weight loss is less with alloys of lead containing microelectrode than with pure lead both in sodium chloride (3%) solution and synthetic sea water which favourably compares with the use of platinum microelectrode.

(4) The above system can be advantageously used as anodes in chloride media in general and for cathodic protection in particular.

R. BHASKAR PAI

Patents Officer,

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH.

Dated this 10th day of May 1972.

Price: TWO RUPEES.

COMPLETE SPECIFICATION

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

THIS IS AN INVENTION BY HANDADY VENKATAKRISHNA UDUPA, DIRECTOR AND KAPISTHALAM CHETLUR NARASIMHAM, SCIENTIST, BOTH OF THE CENTRAL ELECTROCHEMICAL RESEARCH INSTITUTE, KARAIKUDI, INDIA, BOTH INDIAN CITIZENS.

This invention relates to improvements in or relating to the formation of lead dioxide on lead and its alloys in chloride medium.

Hitherto it has been the practice to form lead dioxide on lead and its alloys in chloride medium by introducing platinum microelectrode into the lead surface and these have been used as anodes for cathodic protection.

The drawbacks to the current practice is that a costly material like platinum has to be employed as a microelectrode in order to have lead dioxide formation of lead in chloride medium.

The object of this invention is to obviate the above disadvantage by suitably introducing the cheap solid lead dioxide microelectrode into the lead or lead alloy surface.

To these ends the invention broadly consists in the introduction of lead dioxide microelectrode in the place of platinum into lead or lead alloy surface. The lead dioxide microelectrode is prepared from solid pieces of lead dioxide deposited from lead nitrate—copper nitrate bath on a suitable substrate like graphite, stainless steel or nickel (as described in Indian Patent Nos. 66185, 124215) and were ground on the 60 and 100 emery belt to the desired size and shape. Alternatively the microelectrode is prepared by exposing the desired size on the substrate and stopping off the rest of the portion during electrodeposition of lead dioxide from nitrate bath. Suitable holes are drilled on the lead surface and the lead dioxide microelectrode pressed to fit them tightly in the holes and the lead or lead alloys having the lead dioxide microelectrodes polarised in 0·5 to 3% sodium chloride or synthetic sea water at anode current densities of 0·5 to 6 A/dm^2, temperature of 15 to 50°C, the lead dioxide is formed on the surface of the lead or lead alloys. The ratio of lead surface to lead dioxide microelectrode can vary from 10 : 1 to 600 : 1 to get good deposit.

The introduction of lead dioxide microelectrode into lead or lead alloy surface makes it possible to get lead dioxide on the surface of lead or lead alloy when lead or lead alloys having the lead dioxide microelectrode is polarised in 0·5 to 3% sodium chloride or synthetic sea water at an anode current density of 0·5 to 6 A/dm^2, temperature of 15 to 50°C. The ratio of area of lead surface to lead dioxide microelectrode can vary from 10 : 1 to 600 : 1 to get good deposit.

If the lead or lead alloy containing lead dioxide microelectrode is polarised in a solution containing sulphates along with chlorides (1 to 3%) a lead dioxide deposit formed on the surface of lead or lead alloys is very adherent.

The present invention consists of a process for the formation of lead dioxide on lead and its alloys in chloride medium wherein lead dioxide microelectrode prepared from solid pieces of lead dioxide deposited from lead nitrate—copper nitrate bath on suitable substrates, is fitted to lead or lead alloy and is polarised in 0·5 to 3% sodium chloride or synthetic sea water at an anode current density of 0·5 to 6 A/dm^2, temperature of 15 to 50°C with the ratio of area of lead surface to lead dioxide microelectrode varying from 10 : 1 to 600 : 1.

The formation of lead dioxide on lead and lead alloys having lead dioxide microelectrode is better in solutions containing sulphate along with chloride.

The process does not involve any elaborate arrangement except electrolysis and hence no flow diagram is needed.

The following typical Examples are given to illustrate the invention :

Example 1

Electrolyte	3% (W/V) sodium chloride solution
Anode	Lead
Area of anode (cm^2)	2·2
Area of lead dioxide microelectrode (cm^2)	0·12
Cathode	Mild steel
Area of cathode (cm^2)	20
Inter electrode distance (cm)	6
Cell voltage (volts)	3·65
Anode current density (A/dm^2)	3
Temperature (°C)	30±1

Example 2

Electrolyte	3% (W/V) sodium chloride solution
Anode	Lead
Area of anode (cm^2)	2·2
Area of lead dioxide microelectrode (cm^2)	0·12
Cathode	Mild steel
Area of cathode (cm^2)	20
Inter electrode distance (cm)	6
Cell voltage (volts)	2·7—2·9
Anode current density (A/dm^2)	3
Temperature (°C)	50±2

Example 3

Electrolyte	3% (W/V) sodium chloride solution
Anode	Lead
Area of anode (cm^2)	2·2
Area of lead dioxide microelectrode (cm^2)	0·12
Cathode	Mild steel
Area of cathode (cm^2)	20
Inter electrode distance (cm)	6
Cell voltage (volts)	3·6—3·9
Anode current density (A/dm^2)	3
Temperature (°C)	15

Example 4

Anode	Lead
Area of anode (cm^2)	19·54
Area of microelectrode (cm^2)	0·26
Anode current density (A/dm^2)	2·3
Cathode	Mild steel
Area of cathode (cm^2)	60
Inter electrode distance (cm)	15

Example 4—contd.

Cathode	Mild steel
Cell voltage (volts)	4·2—7·0
Temperature (°C)	30—32
Weight loss in 3% NaCl after 512 hours (mg/cm²/A.hr.)	0·330
Weight loss in synthetic sea water after 483 hours (mg/cm²/A.hr.)	0·072

Example 5

Anode	Lead-antimony (6%)
Area of anode (cm²)	19·54
Area of microelectrode (cm²)	0·26
Anode current density (A/dm²)	2·3
Cathode	Mild steel
Area of cathode (cm²)	60
Inter electrode distance (cm)	15
Cell voltage (volts)	4·2—6·0
Temperature (°C)	30—32
Weight loss in 3% NaCl after 445 hrs. (mg/cm²/A.hr.)	0·035
Weight gain in synthetic sea water after 482 hrs. (mg/cm²/A.hr.)	0·071

Example 6

Anode	Lead-silver (1%)
Area of anode (cm²)	20·33
Area of microelectrode (cm²)	0·252
Anode current density (A/dm²)	2·5
Cathode	Mild steel
Area of cathode (cm²)	60
Inter electrode distance (cm)	15
Cell voltage (volts)	4·1—4·5
Temperature (°C)	30—35
Wt. loss in 3% NaCl after 524 hours (mg/cm²/A.hr.)	0·180
Wt. gain in synthetic sea water after 482 hrs. (mg/cm²/A.hr.)	0·066

The following are the main advantages of the invention :

1. A protective film of lead dioxide can be formed on lead and lead alloys in chloride solutions by incorporating lead dioxide microelectrodes into the surface instead of costly platinum microelectrodes.

2. The ratio of lead dioxide can vary widely to get lead dioxide film on to the surface of lead-lead dioxide bielectrodes as in the case of platinum microelectrode.

3. Weight loss is less with alloys of lead containing microelectrode than with pure lead both in sodium chloride (3%) solution and synthetic sea water (*vide* Examples 4 to 6) which favourably compares with the use of platinum microelectrode.

4. The above system can be advantageously used as anodes in chloride media and for cathodic protection.

Summary :

The process relates to the formation of lead dioxide on lead and lead alloys in chloride medium wherein lead dioxide microelectrode prepared from solid pieces of lead dioxide deposited from lead nitrate-copper nitrate bath on a suitable substrate is introduced in the place of platinum into lead or lead alloy surface. When these lead or lead alloys having the lead dioxide microelectrodes is polarised in 0·5 to 3% NaCl or in synthetic sea water at an anode current density of 0·5 to 6 A/dm², temperature of 15 to 50°C lead dioxide is formed on the surface of lead or lead alloys. The ratio of area of lead surface to lead dioxide microelectrode can vary from 10 : 1 to 600 : 1 to get a good deposit.

We claim :

1. A process for the formation of lead dioxide on lead and its alloys in chloride medium wherein lead dioxide microelectrode prepared from solid pieces of lead dioxide deposited from lead nitrate-copper nitrate bath on suitable substrates, is fitted to lead or lead alloy and is polarised in 0·5 to 3% sodium chloride solution or synthetic sea water at an anode current density of 0·5 to 6 A/dm², temperature of 15 to 50°C with the ratio of area of lead surface to lead dioxide microelectrode varying from 10 : 1 to 600 : 1.

2. A process as claimed in Claim 1 wherein lead dioxide microelectrode prepared from solid pieces of lead dioxide deposited from lead nitrate-copper nitrate bath on a suitable substrate like graphite, stainless steel or nickel are used as microelectrodes to be introduced in lead or lead alloy.

3. A process as claimed in Claim 1 wherein a lead or lead alloy having the lead dioxide microelectrode is polarised in 0·5 to 3% sodium chloride solution or synthetic sea water.

4. A process as claimed in Claims 1 and 2 wherein a lead or lead alloy having the lead dioxide microelectrode is polarised at an anode current density of 0·5 to 6 A/dm².

5. A process as claimed in Claims 1 and 2 wherein the polarisation of lead or lead alloy having lead dioxide microelectrode is carried at a temperature of 15 to 50°C.

6. A process as claimed in Claim 1 wherein the lead or lead alloy fitted with lead dioxide microelectrode is used as anode in cathodic protection.

7. A process for the formation of lead dioxide on lead and its alloys in chloride medium as substantially hereinbefore described.

R. BHASKAR PAI

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Dated this 29th day of June 1973.

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