

Electrodeposition of lead dioxide on titanium substrates

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The present paper describes an electrolytic process for the deposition of lead dioxide and the various steps involved in obtaining a very good, smooth, fine and adherent deposit on titanium substrate.

Key words: Titanium substrate, lead dioxide deposition, perchlorate

INTRODUCTION

A thin coating of lead dioxide over graphite or carbon [1-3] is adequate to be employed as anode in industrial scale cells for the production of perchlorates [4] directly from chloride in a single step [5]; but the anode loss reported is heavy. Hence an alternative base was thought of.

Titanium, because of its physical properties, cannot be used directly as substrate. The following stages are involved: (a) Mechanical abrasion and sand blasting of the smooth surface; (b) pretreatment either chemically or electrochemically to get an activated surface; (c) application of a proper undercoat to protect the surface from getting oxidised prior to deposition of lead dioxide [6]; (d) electrodeposition of lead dioxide from usual nitrate bath and (e) evaluation of the life of these anodes in the preparation

of perchlorates from chlorate or directly from chlorides.

EXPERIMENTAL

Several pieces of titanium were emiered and sandblasted after sizing the edges. They were etched either chemically in sulphuric or oxalic or hydrofluoric or (1.18 Sp.gr.) hydrochloric acids or electrochemically in 1:1 sulphuric or hydrochloric acid. Actual deposition of lead dioxide was carried on these substrates employing them as anodes and copper as cathode from lead nitrate-copper nitrate bath at predetermined operating conditions.

The deposit, without a protective undercoat was not uniform. Prior to actual deposition of lead dioxide, the pretreated and activated titanium plates were given a copper undercoat from conventional copper plating bath or an undercoat of stannic oxide by thermal decomposition of a mixture of stannic chloride, antimony oxide and isopropyl

TABLE-I: Electrodeposition of lead dioxide on titanium substrates without undercoat

pH < 4; Temperature: 318 to 333K; Flow of electrolyte: 1 l.hr⁻¹

Anode: 15 cm (l) × 5 cm (b) × 0.3 cm Ti plate

Electrolyte: 275 g.l⁻¹ lead nitrate; 25 g.l⁻¹ copper nitrate

Sl No.	Pretreatment	Anode C. D. (A.dm ⁻²)	Voltage (V)	Duration (Hrs)	Nature of the deposit	Life in perchlorate cell (A.Hrs)
1.	Chemical (H ₂ SO ₄)	2.5	2.7	6	Good, fine	250
2.	Chemical (Oxalic)	2.0 initial	2.7 to 2.8	5	Good	300
3.	Chemical (1 HF)	2.5	2.7	5	Not good	—
4.	Chemical (1.18 Sp.gr. HCl)	2.5 to 5.0	2.7	6	Good	700
5.	Electrochemical (1:1 HCl)	3.5	2.7	6	Good	450
6.	Electrochemical (1:1 H ₂ SO ₄)	2.5	2.7	6	Good	400

TABLE—II: Electrodeposition of lead dioxide with either Cu or SnO₂ undercoat
Anode c.d.:1.5 to 5.0 A.dm⁻² (other conditions same as Table I).

Sl No	Pretreatment	Undercoat	Duration (Hrs)	Nature of the deposit	Life in ClO ₄ ⁻ cell (A.Hrs)
1.	Chemical (1.18 Sp.gr.HCl)	Copper	6	Good	375
2.	Elec.chemical (1:1HCl)	Copper	7	Good	250
3.	Chemical (1.18 /Sp.gr.HCl)	Stannic oxide	8	Good	600
4.	Elec.chem.(1:1HCl)	Stannic oxide	8	Good	650

alcohol at 673K. Further deposition of lead dioxide was carried out as usual.

These titanium substrate lead dioxide anodes served for different lengths of time in perchlorate preparation and failed either due to cracking or increase in voltage.

RESULTS AND DISCUSSION

Typical experimental results for the preparation of TSLD electrodes are given in Tables I and II.

CONCLUSION

From the results, the selection of a proper and protective intermediate electro or thermal coating to prevent the easy oxidation of titanium base to insulative TiO₂, gives encouraging results for the preparation of TSLD electrodes. Literature survey also reveals that coatings of silver, platinum and mixtures of oxides of noble metals could enhance the life of these electrodes as anodes in preparation of perchlorates.

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