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

Specification No. 100313, application No. 100313, dated 29th June 1965. Complete Specification left on 28th April 1966. (Application Accepted 14th December 1966.)

Index at Acceptance—31A [LVIII(2)], 70C5 [LVIII(5)].

IMPROVEMENTS IN OR RELATING TO ETCHING OF FOILS OF ALUMINIUM OR ALUMINIUM ALLOYS FOR CAPACITORS.

PROVISIONAL 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 describes the nature of this invention.

This is an invention by Sarvashri Balkunje Anantha Shenoi, Kandadai Rajagopalachrari Narasimhan, Ven-kataraman Shinivasan and Ananthanarayanan Anantharaman, all of the Central Electrochemical Research Institute, Karaikudi-3, Madras State, India, all Indian citizens.

This invention relates to improvements in etching of aluminium or aluminium alloys for capacitors.

Hitherto it has been proposed to use pulsating current to obtain a favourable etch ratio for use as anode in high voltage capacitors.

This is open to objection that it requires complicated set up for obtaining the pulsating currents. Further the equipment has to be imported at heavy costs.

The object of this invention is (i) to obviate these disadvantages by using a suitable electrolyte (ii) to provide improved capacitor employing etched aluminium electrodes giving higher etch ratio even at higher voltages.

To these ends, the invention broadly consists in etching aluminium in an electrolyte consisting of a chloride of sodium, potassium or ammonium and a film forming substance, an organic acid belonging to the group of dibasic acid or hydroxy acid or the salts of the acids referred to above.

The following examples are given to illustrate the invention:

EXAMPLE 1.

Electrolyte:

25 gms. of sodium chloride and 25gms, of tartaric acid in 250 ml. of water

'l'emperature :

95° C. to 102° C.

Etching is caried out using aluminium or alloy of aluminium as anode and stainless or alloy of aluminium as cathode and at a current density of 250-400 ma/cm² for 1 to 3 minutes.

EXAMPLE 2.

Electrolyte:

5 to 15% sodium chloride and 5 10% sodium tartarate dissolved in 250 ml. of water.

Temperature:

95° C. to 102° C.

Etching is carried out as conditions indicated in the electrolye (1).

With Aluminium or alloy of aluminium as anode it is possible to obtain etch ratio of 5-6 at 450 Volts and above

The following are the main advantages of the invention:

It is not possible to obtain etch ratio more than four by using direct current at 450 V. The use of pulsating current for obtaining higher etch ratios at voltages more than 450 is known but this involves costly equipment which has to be imported. The present method enables one to attain fairly high etch ratio without using these equipments. Added advantage is that the same method enables one to attain etch ratio of more than 10 at lower voltages and hence there will be considerable saving in the use of aluminium foil to be used as anode in the manufacture of the electrolytic capacitors.

Sd./-Illegible

INDUSTRIAL LIAISON & EXTENSION OFFICER, COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH.

Dated this 23rd day of June 1965.

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 1880).

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 Sarvashri Balkunje Anantha Shenoi, Kandadai Rajagopalachari Narasimhan, Venkatarama Shinivasan and Ananthanarayanan Annantharaman, all of the Central Electrochemical Research Institute, Karaikudi-3, Madras State, India, all Indian citizens.

This invention relates to a method of etching of foils of aluminium or aluminium alloys for capacitors. Electrolytic capacitors are known for their small size and the capacity per unit volume is sufficiently high compared to paper, ceramic or other types of capacitors. This high capacitance to volume ratio is made possible by the use of etched foils.

A few firms have started manufacturing aluminium electrolytic capacitors using etched foils with foreign know how. With the recent import restrictions many new entrants are unable to get the machinery from abroad and especially for the manufacture of etched foils useful for high voltage capacitors.

Electrolytic etching is usually done with direct current and the high etch ratio of 1-10 have been obtained for forming voltages upto 100 volts. This high etch ratio is lowered as the forming voltage is increased due to the filling up of the pores or the etch pits by the oxide film and at 450 volts and above, the etch ratio is usually between 2-3. So, etching of aluminium for use in high voltage capacitors must be such that the pores are deeper and should not be affected during the forming process. One patented process makes use of uni directional pulsed currents and claims high etch ratio even at high forming voltages. The main disadvantage of the process is that it involves the use of costly machinery which have to be imported.

The method described in the application comprises of electrolytically etching aluminium or its alloys in a suitable electrolyte using direct current. Reasonably, high etch ratio 5-6 have been obtained by the procedure.

Price: TWO RUPEES,

By etch ratio, we mean the ratio of the capacitance of etched and formed foil at a given voltage to the capacitance of a plain and formed foil of the same geometric area and also formed at the same voltage.

The dielectric medium in aluminium electrolytic capacitors is the oxide layer and this can be built up by anodising in suitable electrolytes comprising boric acid alone or borax. The thickness increases with the forming voltage and hence, the capacitance is inversely proportional to the forming voltage.

The following examples illustrate the use of this process:

EXAMPLE 1.

Electrolyte:

25 grams or sodium chloride and 25 grams of tartaric acid in 250 ml. of distilled water.

Temperature:

95° to 102° C.

Etching is carried out using superpurity aluminium or alloys of aluminium and stainless steel as cathode and at a current density of 0.39 to 0.62 amp/cm² for one to three minutes.

The etch ratio obtainable at 450 volts is 5-6.

EXAMPLE 2.

Electrolyte:

5-15% Sodium Chloride and

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Temperature:

95° to 102° C.

Etching is carried out as indicated in Example 1 and the etch ratio obtainable at 450 volts is also between

The following are the main advantages of the invention:

- (i) it is possible to get reasonably high etch ratio 5-6 at 450 voltages by using direct current and the electrolytes referred to in this patent;
- (ii) precipitation during etching is trouble some process and this can be avoided by proper choice of the electrolyte, i.e., sodium chloride and tartaric acid; and
- (iii) since the method gives very high etch ratio at lower voltages, considerable saving can be effected in the use of aluminium to be used as anode in the capacitors. The following are the etch ratio values at different voltages:

TABLE.

Electrolyte	Etch Ratio values			
	30V	▶ 100V	300V	450V
 Sodium Chloride 10% 10% Sodium Chloride & 10% tartaric acid. 	1 0 1 7	8.2 13.0	4.0 7.0	3.0 5.0

and

(iv) it is always desirable to keep down the weight loss of the aluminium during the etching process, as otherwise, the foil will lose its mechanical strength during subsequent winding operations. The weight loss during the process adopted by our method has always been less than 27 per cent.

We claim:

- 1. The method of etching aluminium and its alloys employing direct current and an electrolyte consisting of hydrochloric acid or soluble chlorides like sodium, potassium, magnesium or aluminium and a film forming organic acid.
- 2. The method of etching aluminium or its alloys using direct current and an electrolyte consisting of hydrochloric acid or soluble chlorides like sodium,

potassium, magnesium or aluminium and suitable organic acids like dibasic acids or hydroxy acids or its salts.

3. The method of etching aluminium or its alloys using direct current and an electrolyte consisting of hydrochloric acid or soluble chlorides like, sodium, potassium, magnesium or aluminium and suitable organic acids like dibasic or hydroxy acids or its salts and at temperature 95-102° C.

R. BHASKAR PAI

Patents Officer,

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH.

Dated this 14th day of April 1966.