

Government of India : The Patent Office, 214, Acharya Jagadish Bose Road, Calcutta-700017.
Complete Specification No. 140220 dated 13th August 1973. Application No. 1859/Cal/1973
dated 13th August 1973. Acceptance of the Complete Specification advertised on 2nd October 1976.

Index at acceptance—31A [LVIII(2)].

International classification—H01g 9/00.

IMPROVEMENTS IN OR RELATING TO THE FABRICATION OF SOLID FOIL TYPE ALUMINIUM ELECTROLYTIC CAPACITORS.

COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH, RAJ 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 BALKUNJE ANANTHA SHENOI, Scientist, KANDADAI RAJAGOPAL-ACHARI NARASIMHAN, Scientist and Mrs. VIJAYALAKSHMI RAMAKRISHNAN, Senior Laboratory Assistant, all of the Central Electrochemical Research Institute, Karaikudi-623006, Tamil Nadu, India, and all Indian citizens.

This invention relates to improvements in or relating to the method of producing solid foil type aluminium electrolytic capacitors which are likely to be in great demand in the country in future.

Hitherto it has been proposed to make solid foil type capacitors as per the following procedure : Etched and formed anode foil and etched aluminium foil having the tab connection are rolled up using fibreglass tape as the separator. The roll is then impregnated in the manganese nitrate solution, dried in vacuum and then decomposed to get the manganese dioxide layer (MnO_2). The roll is then reformed to get low leakage current. Satisfactory values of capacitance and power factor could be obtained only if impregnation in manganese nitrate solution, drying and decomposition and reforming are repeated for two to three times.

In the hitherto known processes, the rolls are reformed for 15-20 hours after each pyrolysis step. This process is time-consuming and very laborious.

The main object of this invention is to reform the roll in a suitable electrolyte after giving the necessary number of coatings of manganese dioxide.

The main finding of this invention is that the values of capacitance, power factor and leakage current are obtained within the limits using single reforming step after giving the necessary number of coatings of manganese dioxide. The reforming of the roll after each pyrolysis step is avoided. If the reforming is carried out only once after giving the necessary number of coatings of manganese dioxide, the number of hours involved to get a finished capacitor is reduced.

According to the present invention, there is provided a process for making solid type etched aluminium foil capacitors by rolling up (a) an etched and formed aluminium anode foil having a lead connection with (b) an etched cathode using fibreglass tape as a separator, (ii) impregnating the roll in manganese nitrate solution, drying and decomposing by pyrolysis at 200-350°C to get manganese dioxide coating, followed by (iii) reforming the roll characterised in that after obtaining a sufficiently thick manganese coating by repeating step (ii) 3 or 4 times in one stretch step (iii) of reforming the thick manganese coated roll is performed only once, whereby satisfactory values of capacitance, power factor and leakage current are obtained by adopting the single reforming step, and the conventional step of repeatedly reforming the roll after each pyrolysis step is avoided resulting in saving of time.

Reforming of the manganese dioxide coated roll is done only once in a suitable non-aqueous electrolyte containing ethylene glycol and 10-30% film forming inorganic acid or ammonium salt of a film forming organic acid.

Thus, the reforming of the roll, after giving the necessary number of coatings of manganese dioxide is carried out in a non-aqueous electrolyte containing either ethylene glycol and a film forming inorganic acid or ammonium salt of a film forming organic acid.

The invention is carried out as follows :

The 99.99% pure etched aluminium foil is formed at a suitable voltage to form the dielectric aluminium oxide,

the forming solution being ammonium dihydrogen orthophosphate at 90°C. The anode foil is rolled up with etched cathode using fibreglass tape as the separator. The roll is then impregnated in the manganese nitrate solution for 5 minutes to 30 minutes dried in vacuum at 80-100°C then decomposed at 200-350°C to get the coating of manganese dioxide. At this stage, the power factor is high. Hence, the impregnation in manganese nitrate solution, drying in vacuum and pyrolysis are repeated for three times or more to get better values of capacitance and power factor. After giving the necessary number of coatings the roll is reformed in the electrolyte containing either ethylene glycol and a film forming inorganic acid of ammonium salt of a film forming organic acid.

The following are the typical examples given to illustrate the invention :

EXAMPLE 1

The anode foil is prepared by forming the etched foil in 0.25% dihydrogen ammonium orthophosphate solution at 30 volts till the minimum leakage current is reached. This is then washed in deionised water and dried before it is rolled up with etched cathode foil and fibreglass tape as separator. The roll so prepared is impregnated in manganese nitrate solution. It is then dried in vacuum at 80-100°C and decomposed at 300°C for 2-10 minutes. The operation is repeated three or four times and when measured the following values are obtained at 2 volts DC.

Capacity	Power factor	Leakage current
17.8 mfd	0.12	200 μ A

It could be seen from the values that the oxide film is damaged during the pyrolysis step as the important electrical characteristic namely the leakage current is high though of course the power factor is normal. So, the roll is reformed, a step in which the roll is kept polarised at 15 volts in an electrolyte of the following composition :

Glycol :	85%
Boric acid :	15%

for 20-25 hours during which period the damaged oxide film gets healed up. The roll is then taken out, washed and dried and the following values are obtained at 10 volts DC.

Capacity	Power factor	Leakage current
17 mfd	0.12	30 μ A

As mentioned in page 1, the hitherto known practice is to carry out reforming after each pyrolysis step which takes totally about 60-70 hours for completion and this explains the novelty in the present process.

EXAMPLE 2

The procedure adopted for giving the manganese dioxide coatings was the same as in Example 1.

Capacity	Power factor	Leakage current
18 mfd	0.12	200 μ A at 2V DC

Price : TWO RUPEES.

The capacitor is reformed in the electrolyte containing ;

Glycol : 80 %
Ammonium formate : 21 %

After reforming the capacitor gives the following value at 10V DC.

Capacity	Power factor	Leakage current
18 mfd	0.12	Less than 30 μ A

The main advantage of this invention is that considerable amount of reforming time is reduced in as much as the reforming is carried out only once.

We claim :

1. A process of making solid type etched aluminium foil capacitors by rolling up (a) an etched and formed aluminium anode foil having a lead connection with (b) an etched cathode using fibreglass tape as a separator, (ii) impregnating the roll in manganese nitrate solution, drying and decomposing by pyrolysis at 200-350°C to get manganese dioxide coating, followed by

(iii) reforming the roll characterised in that after obtaining a sufficiently thick manganese dioxide coating by repeating step (ii) 3 or 4 times in one stretch, step (iii) of reforming the thick manganese coated roll is performed only once, whereby satisfactory values of capacitance, power factor and leakage current are obtained by adopting the single reforming step, and the conventional step of repeatedly reforming the roll after each pyrolysis step is avoided resulting in saving of time.

2. A process as claimed in Claim 1 wherein reforming of the manganese dioxide coated roll is done only once in a suitable non-aqueous electrolyte containing ethylene glycol and 10-30% film forming inorganic acid or ammonium salt of a film forming organic acid.

3. A process of making solid type etched aluminium foil capacitor substantially as hereinbefore described.

Sd./ R. BHASKAR PAI

Patent Officer,

Council of Scientific & Industrial Research.

Dated this 7th day of August 1973.