



(51) International Patent Classification:

H01G 9/004 (2006.01) *H01G 9/028* (2006.01)
H01G 9/00 (2006.01) *H01G 9/04* (2006.01)

(21) International Application Number:

PCT/JP2011/001840

(22) International Filing Date:

28 March 2011 (28.03.2011)

(25) Filing Language:

English

(26) Publication Language:

English

(30) Priority Data:

12/751,429 31 March 2010 (31.03.2010) US

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(81) Designated States (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PE, PG, PH, PL, PT, RO, RS, RU, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

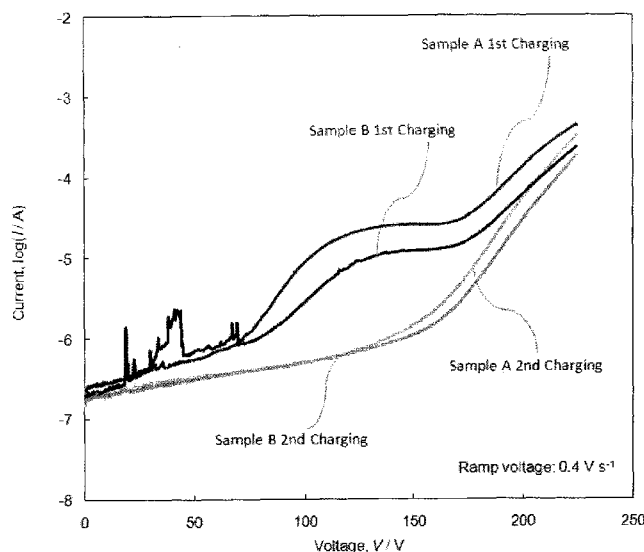
(84) Designated States (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, GH, GM, KE, LR, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Published:

— with international search report (Art. 21(3))

(54) Title: SOLID ELECTROLYTIC CAPACITOR

[Fig. 1]



(57) Abstract: Provided is a high-voltage solid electrolytic capacitor having a rated voltage of several hundreds of volts. After an amorphous anodic oxide film layer is formed on a roughened surface of an aluminum foil by way of a first conversion treatment, a hydrated film is formed by way of boiling water immersion (or a suitable alternative) and partial consumption of the amorphous anodic oxide film layer; the hydrated film and the remaining amorphous anodic oxide film layer are provided with a second conversion treatment at a formation voltage lower than that of the first conversion treatment such that an anodic foil is formed from reoxidation of the partially consumed amorphous anodic oxide film layer; and a conductive polymeric layer is formed on a surface of hydrated film located upon the anodic foil.

Description

Title of Invention: SOLID ELECTROLYTIC CAPACITOR

Technical Field

[0001] The present invention relates to a solid electrolytic capacitor having a conductive polymeric compound as a solid electrolyte, and more particularly to a solid electrolytic capacitor that has been improved for better withstand voltage characteristics, and a method for producing the same.

Background Art

[0002] Electrolytic capacitors comprising a metal having a valve action like aluminum and the like can obtain smaller size and higher capacitance by way of changing the shape of a valve metal used as an anodic electrode to be an etched foil and the like so as to enlarge the surface area of a dielectric material, and thus are in widespread general use. In particular, solid electrolytic capacitors comprising a solid electrolyte as an electrolyte have features of smaller size and higher capacitance and also have a low equivalent series resistance.

[0003] Examples of the known solid electrolytes used for a solid electrolytic capacitor include a conductive polymer having a high conductance and a remarkable adhesion to the oxide film layer of the anodic electrode. Examples of the known conductive polymers include polyaniline, polythiophene and polyethylenedioxy thiophene.

[0004] In particular, polyethylenedioxy thiophene (hereinafter, PEDOT) has received attention as a conductive polymer capable of having greater withstand voltage because PEDOT can have greater withstand voltage relative to the thickness of an oxide film. A capacitor comprising the PEDOT is fabricated by way of a chemical oxidative polymerization in the following manner.

[0005] Namely, a capacitor element is formed by the anode and cathode electrode foils wound together with an intervening separator, and the capacitor element is impregnated with a solution containing EDOT and oxidizing agent, and is heated to form a PEDOT polymeric layer between the two electrode foils such that a solid electrolytic capacitor is formed. One such solid electrolytic capacitor is described in Japanese Laid-open Patent Publication No. H09-293639.

[0006] Unlike a conventional electrolytic solution capacitor, this type of solid electrolytic capacitor has a poor capacity to form an oxide film, and thus has limited withstand voltage characteristics. Accordingly, an electrolytic capacitor has been suggested that employs both electrolytic solution and solid electrolyte as electrolyte such that an oxide film formation capacity of the electrolytic solution is retained while remarkable characteristics of solid electrolytic capacitors are maintained. One such electrolytic

capacitor is described in Japanese Laid-open Patent Publication No. 2000-021689.

Citation List

Patent Literature

[0007] PTL 1: Patent H09-293639

[0008] PTL 2: Patent 2000-021689

Summary of Invention

Technical Problem

[0009] This type of solid electrolytic capacitor has merely several tens of volts in rated voltage.

Solution to Problem

[0010] In this context, the present invention is intended to provide a high-voltage solid electrolytic capacitor having a rated voltage of several hundreds of volts.

Advantageous Effects of Invention

[0011] A solid electrolytic capacitor as a first aspect of the present invention, to address the above-mentioned problems, is an electrolytic capacitor wherein: an amorphous anodic oxide film layer is formed on a roughened surface of an aluminum foil by way of a first conversion treatment; a hydrated film is formed by way of boiling water immersion (or suitable alternative) and partial consumption of the amorphous anodic oxide film layer; the hydrated film and the partially consumed amorphous anodic oxide film layer thereunder are provided with a second conversion treatment at a formation voltage lower than that of the first conversion treatment such that an amorphous anodic foil is formed from the partially consumed amorphous anodic oxide film layer (i.e., the partially consumed amorphous anodic oxide film layer is reformed as an amorphous anodic foil); and a conductive polymeric layer is formed on a surface of the hydrated film that is located upon the amorphous anodic foil.

[0012] A method for producing a solid electrolytic capacitor as a second aspect of the present invention, to address the above-mentioned problems, is a method comprising the steps of: forming an amorphous anodic oxide film layer on a roughened surface of an aluminum foil by way of a first conversion treatment; forming a hydrated film by way of boiling water immersion (or suitable alternative) and partial consumption of the amorphous anodic oxide film layer; providing the hydrated film and the partially consumed amorphous anodic oxide film layer thereunder with a second conversion treatment at a formation voltage lower than that of the first conversion treatment such that an amorphous anodic foil is formed from the partially consumed amorphous anodic oxide film layer; and forming a conductive polymeric layer on a surface of the hydrated film that is formed upon the amorphous anodic foil.

[0013] Employment of the solid electrolytic capacitor according to the present invention

would achieve a high-voltage solid electrolytic capacitor having a rated voltage of several hundreds of volts while remarkable characteristics of solid electrolytic capacitors are maintained.

Brief Description of Drawings

[0014] [fig.1]Fig 1 is a view of the current-voltage behavior of Embodiment 1 of the solid electrolytic capacitor according to the present invention.

[fig.2]Fig. 2 is a view of the current-voltage behavior of Embodiment 2 of the solid electrolytic capacitor according to the present invention.

[fig.3]Fig. 3 is a view of the current-voltage behavior of Embodiment 3 of the solid electrolytic capacitor according to the present invention.

Description of Embodiments

[0015] A detailed explanation of an embodiment will be given below.

[0016] Following is a description of the method for producing the solid electrolytic capacitor according to the present invention.

[0017] First, an amorphous anodic oxide film layer (i.e. an amorphous aluminum containing oxide layer) having a thickness from about 420 to about 1260 nanometers is formed on a roughened surface of an aluminum foil (i.e., an aluminum containing substrate) by way of a first conversion treatment using a first conversion voltage from about 300 to about 900 volts.

[0018] Second, the amorphous anodic oxide film layer is hydrated by way of boiling water immersion (or suitable alternative) to be partially converted into a hydrated film (i.e., a hydrated aluminum containing oxide layer) having a thickness from about 200 to about 500 nanometers. Suitable alternatives to boiling water immersion include: (1) a heated water immersion at a temperature greater than about 40 degrees centigrade, preferred 60 degrees centigrade, or more preferred 90 degree centigrade; and (2) a steam immersion treatment.

[0019] Third, the hydrated film and the remaining amorphous anodic oxide film layer thereunder are provided with a second conversion treatment at a formation voltage also from about 200 to about 800 volts, but lower than that of the first conversion treatment such that an amorphous anodic foil (i.e., also an amorphous aluminum containing oxide layer) having a thickness from about 280 to about 1150 nanometers is formed (i.e., the amorphous anodic foil comprises an anodically reformed thinned amorphous anodic oxide film layer). The second conversion treatment allows for formation of a dielectric oxide film (i.e., the amorphous anodic foil that comprises the anodically reformed thinned amorphous anodic oxide film layer) having a withstand voltage equivalent to the formation voltage of the second conversion treatment (i.e., greater than about 200 volts), and further, a hydrated film is formed on the surface of the

amorphous anodic foil.

[0020] Fourth, the hydrated film contains water. Accordingly, damage to the dielectric oxide film caused in the fabrication process of an electrolytic capacitor can be repaired by way of anodic oxidation using the water during the aging process or the voltage application process.

[0021] Fifth, the present invention takes the advantage of the healing effect even with the withstand voltage of the dielectric oxide film being several hundreds of volts, resulting in achievement of a high-voltage solid electrolytic capacitor having a rated voltage of several hundreds of volts.

[0022] In this context, the applicant focused on the low reformability of the electrolytic solution, which causes the withstand voltage of the Al/PEDOT capacitor to be quite low in comparison with a conventional electrolytic capacitor. Electrolytic solution of conventional electrolytic capacitors contains a great amount of oxygen source required for reformation of a film, with the examples of the oxygen source including water (several %) and ethylene glycol. However, PEDOT contains only a slight amount of water. Hence, it cannot be stated that the PEDOT has an excellent reformability. The water simply added to the PEDOT would very likely be evaporated during the PEDOT polymerization process (at 150 degrees centigrade for 60 minutes), and thus, a type of water is required that can stably subsist even at relatively higher temperatures. Besides, in case where water is added to the PEDOT, the water in the bulk PEDOT would need to diffuse as far as the barrier film/PEDOT interface when the reformation by the electrolytic solution is in progress, leading to inefficient reformation. The applicant thus suggested, as a method for providing efficient reformability, to form a water-rich layer on the barrier film/PEDOT interface. In the present invention, after forming a conventional anode oxide film, a foil was immersed in boiling water and part of the barrier film was hydrated such that a Pseudoboehmite (PB) was formed. The method according to the present invention allows the electrodes to comprise Al/barrier film/PB/PEDOT/silver, carbon paste and the like. The PB stands for aluminum oxide that amply contains water, and can be represented as $\text{Al}_2\text{O}_3 \cdot n\text{H}_2\text{O}$ in a chemical formula.

[0023] (Embodiment 1)

In the solid electrolytic capacitor according to the present invention, a foil, etched with direct current, was immersed in a boric acid/borax aqueous solution at 300 V, and a film was formed. The withstand voltage of the film was 300 V.

[0024] The etched foil was then immersed in boiling water for three minutes to form a hydrated film. Under the experimental condition of Embodiment 1, the withstand voltage of the film dropped to 200 V. This means that the withstand voltage was reduced by 100 V, which reduction was caused by the conversion of some part of the barrier film into PB.

[0025] Further, the etched foil on which a hydrated film was formed was reformed at 200 V, whereby an oxide film having a withstand voltage of 200 V in the boric acid/borax aqueous solution was obtained.

[0026] After forming the oxide film, the PEDOT was prepared under the conditions of 60 degrees centigrade for 30 minutes, and 150 degrees centigrade for 60 minutes, and the current-voltage behavior was measured. The results are shown in FIG. 1. FIG. 1 depicts the first and second measurements of the current-voltage behavior. The two measurements were conducted with respect to each of the samples A and B, respectively.

[0027] According to Embodiment 1, the samples had a withstand voltage of 200 V in the boric acid/borax aqueous solution without generating any short circuit. A short circuit would be generated at around 90 to 100 V without any hydrated film layer.

[0028] At the second measurement of the current-voltage behavior, the current value dropped, and an aging effect was observed.

[0029] The applicant focused on the initial characteristics, and a very reproducible withstand voltage was obtained.

[0030] (Embodiment 2)

The etched foil was immersed in oxalic acid solution at 60 V and a porous anodic film was formed (first formation). The foil was immersed in a boric acid/borax solution at 550 V and an amorphous phase film was formed (second formation). The obtained material was then immersed in boiling water to form a hydrated film. The third formation was conducted at 450 V, in a manner similar to Embodiment 1. FIG. 2 shows the current-voltage behavior of 550 V to 450 V etched foil-based capacitors made using in-situ polymerized PEDOT. The withstand voltage of the V_f to V_{rf} oxide is 450 V. The capacitors show a low, very stable leakage current below the withstand voltage, and exhibit short circuit at voltages above 450 V. The oxide formation method according to the present application is very important for attaining such high voltages. The method consists of first forming an amorphous oxide of 550 V_f . An outer portion of the oxide is then hydrated in boiling water. The remaining barrier oxide is then reinforced by reforming the amorphous oxide to 450 V_{rf} .

[0031] (Embodiment 3)

The third and fourth formations were conducted at 900 V and 800 V, respectively, in a manner similar to Embodiment 2. The fifth and sixth formations were both conducted at 800 V. FIG. 3 shows the current-voltage behavior of 900 V to 800 V etched foil-based capacitors made using PEDOT. The withstand voltage of the V_f to V_{rf} oxide is 800 V. The capacitors show a low, very stable leakage current below the withstand voltage, and exhibit short circuit at voltages above 800 V. The oxide formation method according to the present application is very important for attaining such high voltages.

The method consists of first forming an amorphous oxide of 900 V_{rf}. An outer portion of the oxide is then hydrated in boiling water. The remaining barrier oxide is then re-inforced by reforming the oxide to 800 V_{rf}.

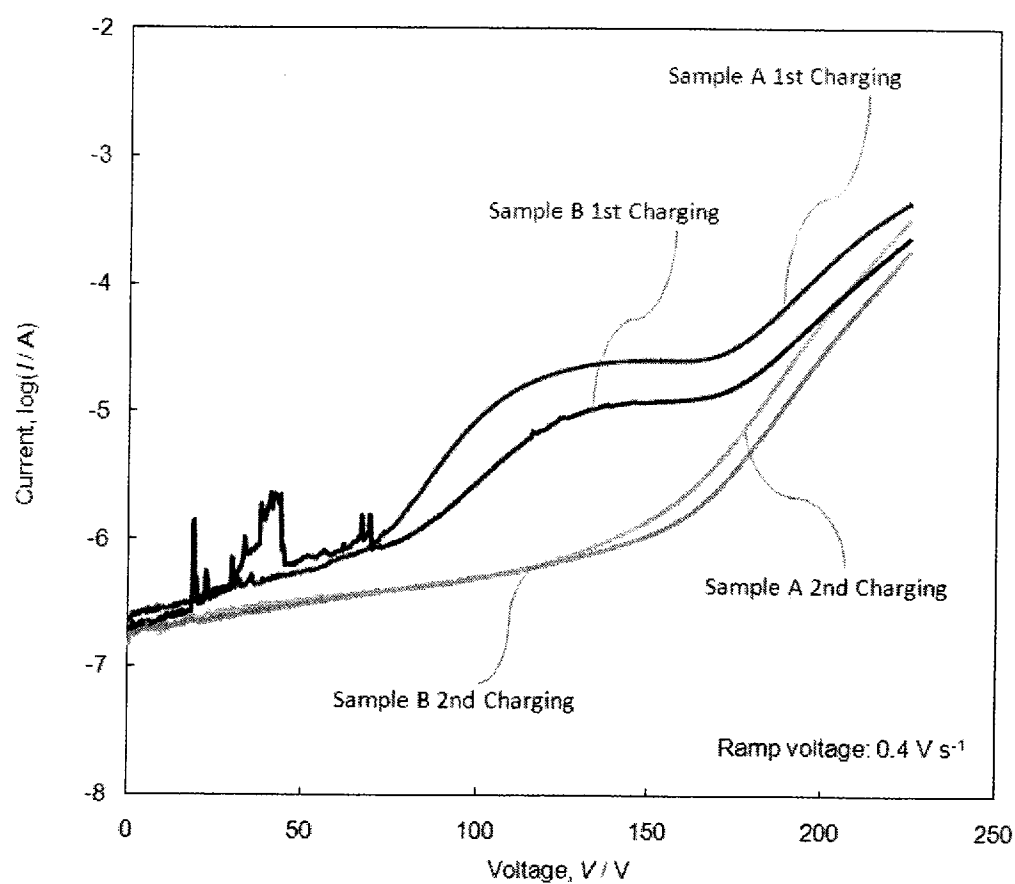
- [0032] The structure of the anodic layer where the barrier oxide also has a hydrated film according to the present application is novel because the layer is capable of withstanding several hundreds of volts and is also amorphous. In common art, an amorphous barrier layer capable of withstanding several hundred volts is not partially or wholly hydrated. In a separate example of common art, a high voltage foil anode is made by first immersion in boiling water to produce pseudoboehmite and then a crystal phase dielectric oxide is formed. The present structure of a high voltage amorphous phase dielectric oxide having a hydrated film layer on the surface of the dielectric oxide is therefore new.
- [0033] The structure of the barrier oxide layer is also important for achieving high voltage in Al/PEDOT capacitors. In the case where the 800 V dielectric is an amorphous oxide, a PEDOT capacitor capable of withstanding 600 V can be made without the conversion treatments of immersion in boiling water and second formation to a lower formation voltage. To achieve 600 V, the PEDOT electrolyte should be applied as a slurry and dried, rather than by chemical oxidative polymerization. An example of a suitable PEDOT slurry is described in US patent 5,300,575 and US laid-open patent application 0,152,832. If chemical oxidation polymerized PEDOT electrolyte is used in this case, the capacitor breakdown voltage is about 300 - 400 V. The PEDOT slurry is pH neutral and does not corrode the dielectric oxide, but the chemical oxidation polymerization of EDOT generates strong acid, which is corrosive to the anodic aluminum oxide layer.
- [0034] The PEDOT slurry is a dispersion element comprising particles of poly (3,4-ethylenedioxythiophene) and/or aggregate and solvent thereof. Examples of the solvent include water and organic solvent, as well as a mixture solvent of water and other solvent. However, water is the most preferable in view of its handleability and the dispersability of the conductive solid electrolyte. Examples of the method of preparing a dispersion element include: a method of preparing a dispersion element by dispersing a conductive solid electrolyte such as conductive polymers into a solvent; and a method of polymerizing a monomer in a solvent that forms a dispersion element, the monomer being a precursor of conductive polymers, such that the conductive polymers are synthesized and a dispersion element comprising the synthesized conductive polymers can be obtained.

Claims

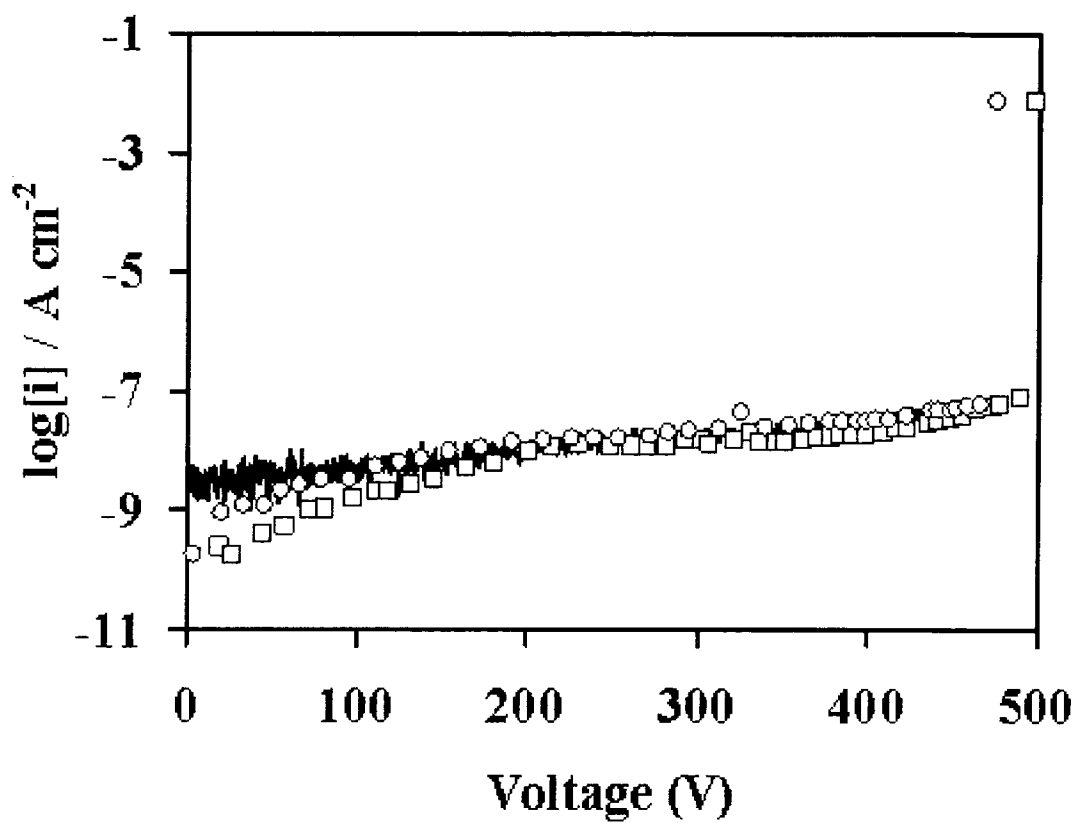
- [Claim 1] An aluminum composite comprising:
an aluminum containing substrate;
an amorphous aluminum containing oxide layer having a thickness from about 100 to about 1900 nanometers located over the aluminum containing substrate; and
a hydrated aluminum containing oxide layer having a thickness from about 10 to about 1000 nanometers located upon the amorphous aluminum containing oxide layer.
- [Claim 2] The aluminum composite of claim 1 wherein the aluminum composite comprises a capacitor electrode.
- [Claim 3] A capacitor comprising the capacitor electrode of claim 2.
- [Claim 4] The capacitor of claim 3 wherein the capacitor has a withstand voltage of at least about 70 volts.
- [Claim 5] The aluminum composite of claim 1 further comprising a conductive polymer layer located upon the hydrated aluminum containing oxide layer.
- [Claim 6] The aluminum composite of claim 1 wherein the hydrated aluminum containing oxide layer comprises a pseudoboehmite material.
- [Claim 7] An aluminum composite comprising:
an aluminum containing substrate;
an amorphous aluminum containing oxide layer having a thickness from about 100 to about 1900 nanometers located over the substrate;
and
a conductive polymer layer located upon the amorphous aluminum containing oxide layer.
- [Claim 8] The aluminum composite of claim 7 wherein the aluminum composite comprises a capacitor electrode.
- [Claim 9] A capacitor including the capacitor electrode of claim 8.
- [Claim 10] The capacitor of claim 9 wherein the capacitor has a withstand voltage of at least about 70 volts.
- [Claim 11] A method for forming an aluminum composite comprising sequentially treating an aluminum containing substrate with:
a first oxidation conversion treatment;
a hydrating treatment; and
a second oxidation conversion treatment, to form a composite amorphous and hydrated oxide dielectric layer over the aluminum

- containing substrate.
- [Claim 12] The method of claim 11 further comprising forming a conductive polymer layer upon the composite amorphous and hydrated oxide dielectric layer.
- [Claim 13] The method of claim 11 wherein:
the first oxidation conversion treatment uses a first anodic conversion voltage; and
the second oxidation conversion treatment uses a second anodic conversion voltage no greater than the first anodic conversion voltage.
- [Claim 14] The method of claim 13 wherein:
the first anodic conversion voltage is from 70 to 1500 volts; and
the second conversion voltage is from 70 to 1500 volts.
- [Claim 15] The method of claim 13 wherein the hydrating treatment is selected from the group consisting of:
a heated water immersion treatment at a temperature at least about 40 degrees centigrade; and
a boiling water immersion treatment.
- [Claim 16] The method of claim 13 wherein the hydrating treatment comprises a steam immersion treatment.
- [Claim 17] The method of claim 13 wherein:
the first oxidation conversion treatment forms an amorphous anodic oxide film layer over the aluminum containing substrate;
the hydrating treatment forms a hydrated film upon a thinned amorphous anodic oxide film layer formed from the amorphous oxide film layer; and
the second oxidation conversion treatment forms an anodic foil from the thinned amorphous anodic oxide film layer.
- [Claim 18] A method for forming an aluminum composite comprising:
treating an aluminum containing substrate with an anodic oxidation conversion treatment to form an amorphous aluminum containing oxide layer over the aluminum containing substrate and having a thickness from about 100 to about 1900 nanometers; and
forming a conductive polymer layer upon the amorphous aluminum containing oxide layer.
- [Claim 19] The method of claim 18 wherein the anodic oxidation conversion treatment uses a conversion voltage from about 70 to about 1500 volts.

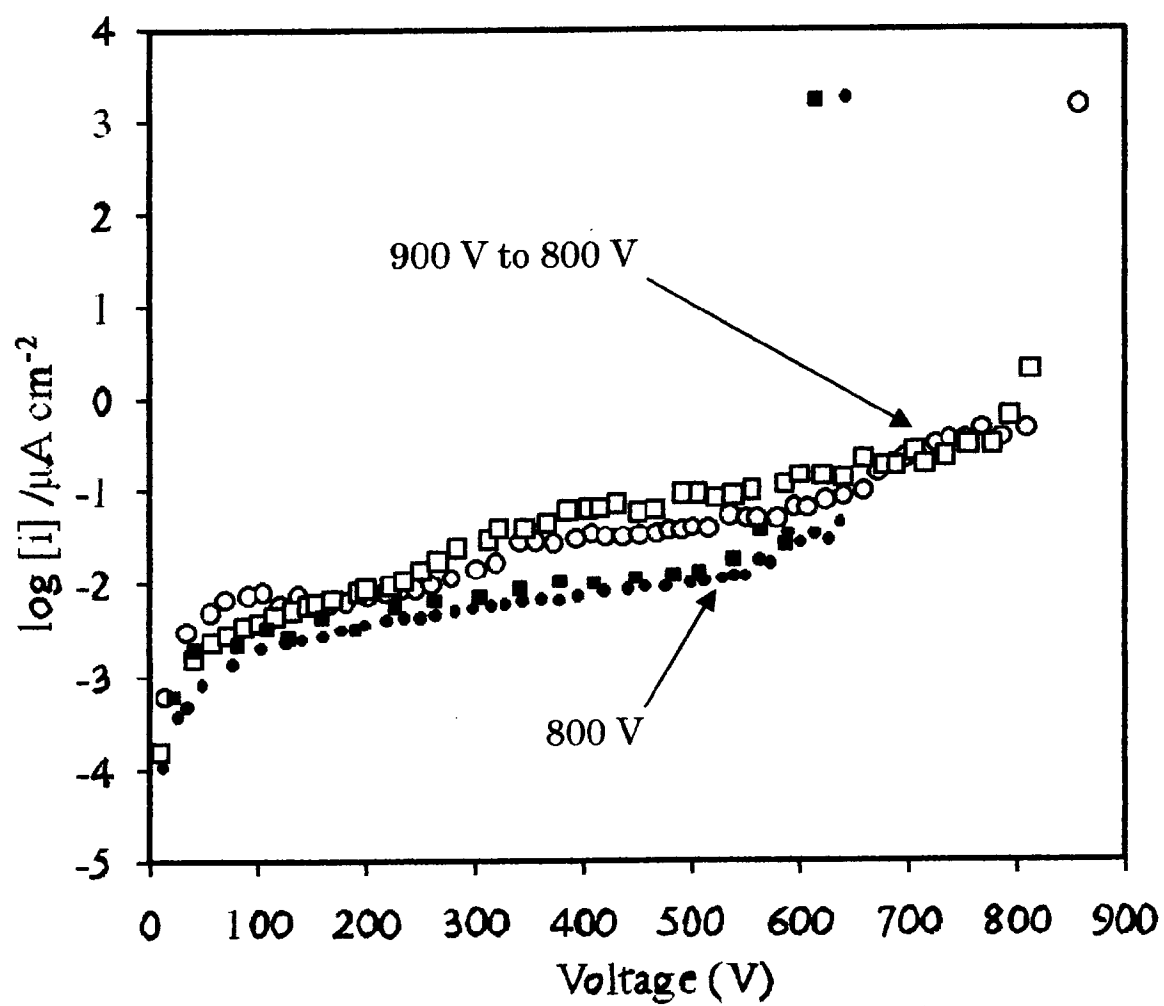
[Fig. 1]



[Fig. 2]



[Fig. 3]



PATENT COOPERATION TREATY

PCT

INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference PF-3742	FOR FURTHER ACTION see Form PCT/ISA/220 as well as, where applicable, item 5 below.	
International application No. PCT/JP2011/001840	International filing date(<i>day/month/year</i>) 28.03.2011	(Earliest) Priority Date (<i>day/month/year</i>) 31.03.2010
Applicant NIPPON CHEMI-CON CORPORATION		

This international search report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This international search report consists of a total of 3 sheets.

☐ It is also accompanied by a copy of each prior art document cited in this report.

1. Basis of the report

a. With regard to the **language**, the international search was carried out on the basis of:

☒ the international application in the language in which it was filed.

☐ a translation of the international application into _____ which is the language of
a translation furnished for the purposes of international search (Rules 12.3(a) and 23.1(b)).

b. ☐ This international search report has been established taking into account the **rectification of an obvious mistake** authorized by or notified to this Authority under Rule 91 (Rule 43.6**bis**(a)).

c. ☐ With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, see Box No. I.

2. ☐ **Certain claims were found unsearchable** (see Box No. II).

3. ☐ **Unity of invention is lacking** (see Box No. III).

4. With regard to the **title**,

☒ the text is approved as submitted by the applicant.

☐ the text has been established by this Authority to read as follows:

5. With regard to the **abstract**,

☒ the text is approved as submitted by the applicant.

☐ the text has been established, according to Rule 38.2, by this Authority as it appears in Box No. IV. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.

6. With regard to the **drawings**,

a. the figure of the **drawings** to be published with the abstract is Figure No. 1

☒ as suggested by the applicant.

☐ as selected by this Authority, because the applicant failed to suggest a figure.

☐ as selected by this Authority, because this figure better characterizes the invention.

b. ☐ none of the figures is to be published with the abstract.

INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2011/001840

A. CLASSIFICATION OF SUBJECT MATTER

Int.Cl. H01G9/004 (2006.01) i, H01G9/00 (2006.01) i, H01G9/028 (2006.01) i,
H01G9/04 (2006.01) i

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

Int.Cl. H01G9/004, H01G9/00, H01G9/028, H01G9/04

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Published examined utility model applications of Japan 1922-1996
Published unexamined utility model applications of Japan 1971-2011
Registered utility model specifications of Japan 1996-2011
Published registered utility model applications of Japan 1994-2011

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	JP 8-296088 A (Matsushita Electric Industrial Co., Ltd.) 1996.11.12, Par.No.[0007][0009] (No Family)	1-19
Y	JP 2005-264288 A (Matsushita Electric Industrial Co., Ltd.) 2005.09.29, Par.No.[0015][0085] (No Family)	1-19
Y	JP 9-293639 A (Nippon Chemi-Con Corporation) 1997.11.11, Par.No.[0026] & US 5914852 A & EP 833352 A1 & WO 1997/041577 A1	5,7-10,12,18 ,19
Y	WO 2006/106703 A1 (Showa Denko K.K.) 2006.10.12, Par.No.[0044] & JP 3974645 B & US 2008/0250621 A1 & EP 1876612 A1	13-17



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier application or patent but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search

03.06.2011

Date of mailing of the international search report

14.06.2011

Name and mailing address of the ISA/JP

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INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2011/001840

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	JP 8-293442 A (Matsushita Electric Industrial Co., Ltd.) 1996.11.05, Par.No.[0003][0007] [0014] (No Family)	15