



(51) International Patent Classification:

G01N 27/26 (2006.01) C25D 21/12 (2006.01)

(21) International Application Number:

PCT/CN2016/083925

(22) International Filing Date:

30 May 2016 (30.05.2016)

(25) Filing Language:

English

(26) Publication Language:

English

(30) Priority Data:

15/162,609 23 May 2016 (23.05.2016) 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, BN, 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, IR, IS, JP, KE, KG, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SA, 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, RW, SD, SL, ST, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, 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, KM, ML, MR, NE, SN, TD, TG).

Published:

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

(54) Title: STATIC DISK ELECTRODE FOR ELECTROPLATING BATH ANALYSIS

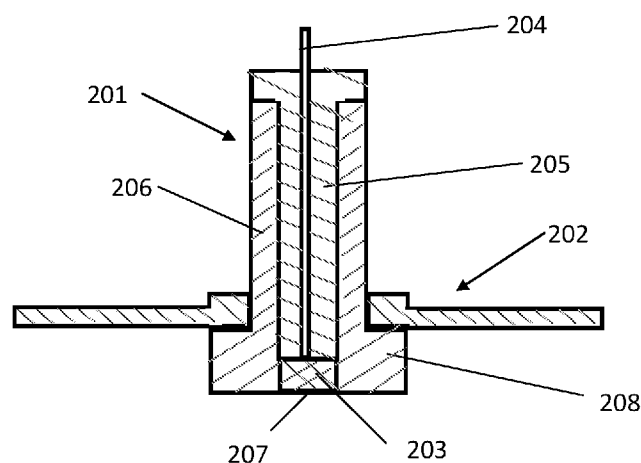


FIG. 2B

(57) Abstract: The presently claimed invention provides an electrochemical analytical apparatus for electrochemical bath analysis. The apparatus comprise a static electrode and a rotatable unit. As steady liquid flow can be generated on the electrolytic surface of the static electrode by the rotatable unit through rotation, the static disk electrode does not involve any movement during the bath analysis such that the design of the electrical contact in the electrode can be substantially simplified.



Static Disk Electrode for Electroplating Bath Analysis

Field of the Invention:

[0001] The present invention relates to electrochemical bath analysis. More particularly, the present invention relates to a static electrode for electrochemical bath analysis.

Background:

[0002] Cyclic voltammetric stripping (CVS) analyzer with a rotating disk electrode is an established method to evaluate the composition concentration in plating baths. The CVS applies a reversible potential sweep to firstly deposit metal onto the surface of the rotating disk electrode, and then strips the metal back into solution. These cycles of ramps in potential may be repeated as many times as desired. The current at the working electrode is plotted versus the applied voltage to give a cyclic voltammogram trace.

[0003] The rotating disk electrode rotates during experiments and induces a flux of analyte to the electrode. The electrode includes a conductive disk embedded in an inert non-conductive polymer or resin that can be attached to an electric motor providing fine control of the electrode's rotation rate. The disk is mostly made of a noble metal or glassy carbon. As the disk turns, some of the solution is dragged by the spinning disk and the resulting centrifugal force flings the solution away from the center of the electrode. Solution flows up, perpendicular to the electrode, from the bulk to replace the boundary layer as shown in FIG. 1A. It results a laminar flow of solution towards and across the electrode. The rate of the solution flow can be controlled by the electrode's angular velocity and modeled mathematically. This flow can quickly achieve conditions in which the steady-state current is controlled by the solution flow rather than diffusion.

[0004] Nevertheless, as the electrode has to be rotated during analysis, a simple electrical wire, connecting the conductive disk and CVS analyzer for transmitting current, can not be used since the electrical wire is twisted under rotation. Thus, special design of the electrical contact has to be used. To solve this problem, the electrical contact is designed as

shown in FIG. 1B. A conventional RDE 100 comprises a brush contact 101, a lower bearing assembly 102, a motor coupling 103, an attachment structure 104, and an electrode shaft 105. The brush contact 101 is a spring-loaded silver-carbon brush providing electrical contact with the rotating electrode shaft 105. The lower bearing assembly 102 can stabilize the rotating electrode shaft 105 at the point where the electrode shaft 105 exits a motor unit. The motor coupling 103 is used to attach the electrode shaft 105 to the motor. The attachment structure 104 is used to attach the electrode shaft 105 to the motor and comprises hex screws located on either side of the motor coupling 103 and being tightened to hold the electrode shaft 105 inside the motor coupling 103. The top end of the rotating electrode shaft 105 is mounted in the motor coupling 104 and the active electrode surface is located at the bottom end of the electrode shaft 105. However, the drawbacks of such electrical contact using the brush contact and the electrically conductive electrode shaft are expensive due to precision rotating control parts required, and short working life due to rotation wearing at the rotating electric contact. Furthermore, it is also difficult to fabricate the electrical contact since vibration or drifting of the electrode pole frequently appears.

[0005] Therefore, there is an unmet need to provide an electrode for electrochemical bath analysis having simplified electrical contact design.

Summary:

[0006] Accordingly, the presently claimed invention provides an electrochemical analytical apparatus for electrochemical bath analysis.

[0007] According to an embodiment of the present invention, an electrochemical analytical apparatus for electrochemical bath analysis comprises: a static working electrode having at least one electrolytic surface for being inserted into a liquid electrolyte; a rotatable unit comprising at least one blade and located near the static working electrode for creating flow of the liquid electrolyte by rotation; and a control unit for controlling the rotation of the rotatable unit for arousing a relative motion between the electrolytic surface of the working electrode and a bulk portion of the liquid electrolyte.

[0008] Preferably, the aroused relative motion is symmetrical relative to the at least one electrolytic surface of the static working electrode.

[0009] Preferably, the static working electrode is connected with an electrical contact.

[0010] Preferably, the static working electrode is a disk electrode or a ring disk electrode.

[0011] Preferably, the rotatable unit is located along a vertical axis of the at least one electrolytic surface of the static working electrode and rotates above the at least one electrolytic surface of the static working electrode.

[0012] Preferably, wherein the rotatable unit further comprises two or more blades being symmetrically arranged relative to the at least one electrolytic surface of the static working electrode.

[0013] Preferably, the control unit controls rotation speed of the rotatable unit.

[0014] Preferably, the rotatable unit is a magnetic stir bar freely mounted on a body of the static working electrode, and the control unit is a magnetic stirrer detachably coupled to the magnetic stir bar.

[0015] Preferably, the rotatable unit and the control unit are integrated into a motorized system.

[0016] According to a specific embodiment of the present invention, an electrochemical analytical apparatus for electrochemical bath analysis comprises: a static working electrode having at least one electrolytic surface located at bottom of the static working electrode for being inserted into a liquid electrolyte; an electrical contact connected to the static working electrode for transmitting current; a magnetic stir bar comprising a hole and at least two blades and located on top of the electrolytic surface for creating flow of the liquid electrolyte by rotation, wherein the hole is for accommodating the static working electrode, and the at least two blades are arranged in a symmetrical way to each other; and a magnetic stirrer for generating electric field for controlling the rotation of the magnetic stir bar to arouse a relative motion between the electrolytic surface of the static working electrode and a bulk portion of the liquid electrolyte; wherein the aroused relative motion is symmetrical relative to the at least one electrolytic surface of the static working electrode.

[0017] According to another specific embodiment of the present invention, an electrochemical analytical apparatus for electrochemical bath analysis comprises: a static working electrode having at least one electrolytic surface located at bottom of the static working electrode for being inserted into a liquid electrolyte; an electrical contact connected to the static working electrode for transmitting current; and a motorized stirrer comprising a motor, a hole and at least two blades and located on top of the electrolytic surface for creating flow of the liquid electrolyte by rotation to arouse a relative motion between the electrolytic surface of the static working electrode and a bulk portion of the liquid electrolyte, wherein the motor is for driving the rotation of the motorized stirrer, and the hole is for accommodating the static working electrode, and the at least two blades are arranged in a symmetrical way to each other; wherein the aroused relative motion is symmetrical relative to the at least one electrolytic surface of the static working electrode.

[0018] As steady liquid flow can be generated on the electrolytic surface of the static electrode by the rotatable unit through rotation, the static disk electrode does not involve any movement during the bath analysis such that the design of the electrical contact in the electrode can be substantially simplified.

Brief Description of the Drawings:

[0019] Embodiments of the present invention are described in more detail hereinafter with reference to the drawings, in which:

[0020] FIG. 1A depicts solution flow under the operation of a conventional rotating disk electrode;

[0021] FIG. 1B depicts an electrical contact design of a conventional rotating disk electrode;

[0022] FIG. 2A depicts a electrochemical analytical apparatus with a magnetic stir bar according to an embodiment of the presently claimed invention;

[0023] FIG. 2B depicts a cross section of the electrochemical analytical apparatus of FIG. 2A;

[0024] FIG. 2C depicts the magnetic stir bar of FIG. 2A;

[0025] FIG. **2D** depicts operation of the electrochemical analytical apparatus of FIG. **2A**;

[0026] FIG. **3** depicts flow simulation results between (A) a convention rotating disk electrode and (B) an electrochemical analytical apparatus of the present invention respectively at 900 rpm;

[0027] FIG. **4** depicts flow simulation results between (A) a convention rotating disk electrode and (B) an electrochemical analytical apparatus of the present invention respectively at 1500 rpm;

[0028] FIG. **5** depicts an electrochemical analytical apparatus with a motorized stirrer according to an embodiment of the presently claimed invention; and

[0029] FIG. **6** depicts a cross section of an electrochemical analytical apparatus with a motorized stirrer according to another embodiment of the presently claimed invention.

Detailed Description:

[0030] In the following description, an electrochemical analytical apparatus for electrochemical bath analysis is set forth as preferred examples. It will be apparent to those skilled in the art that modifications, including additions and/or substitutions may be made without departing from the scope and spirit of the invention. Specific details may be omitted so as not to obscure the invention; however, the disclosure is written to enable one skilled in the art to practice the teachings herein without undue experimentation.

[0031] The foregoing description of the present invention has been provided for the purposes of illustration and description. It is not intended to be exhaustive or to limit the invention to the precise forms disclosed. Many modifications and variations will be apparent to the practitioner skilled in the art.

[0032] FIG. **2A** depicts an electrochemical analytical apparatus with a magnetic stir bar according to an embodiment of the presently claimed invention. The electrochemical analytical apparatus **200** comprises a static disk electrode **201** and a magnetic stir bar **202**. FIG. **2B** shows a cross section of the apparatus **200**. The static disk electrode **201** comprises a disk electrode **203**, an electrical contact **204**, an electrode support **205**, and an inert material

sheath **206**. The disk electrode **203** has an electrolytic surface **207** located at the bottom of disk electrode **203** for exposing to a liquid electrolyte. The electrical contact **204** is connected to the disk electrode **203** for transmitting current. The electrode support **205** is in contact with the disk electrode **203** and encloses the electrical contact **204** for provide structural support of the static disk electrode **201**. The inert material sheath **202**, being cylindrical, encloses both of the disk electrode **203** and the electrode support **205**, and comprises an extending structure **208** for holding the magnetic stir bar **202**. As shown in FIG. 2C, the magnetic stir bar **202**, made from a magnetic material, comprises a connecting structure **209** and two blades **210a**, **210b**. The connecting structure **209** has a circular hole **211** located at the center of the connecting structure **209** for accommodating the cylindrical inert material sheath **206**. The two blades **210a**, **210b** are connected with the connecting structure **209** in a symmetrical way and arranged in parallel to the electrolytic surface **207** for generating steady liquid flow. The rotation center of the rotatable unit **202** is located along the vertical axis of the center of the electrolytic surface **207** for providing symmetrical liquid flow on the electrolytic surface **207**.

[0033] FIG. 2D depicts the operation of the apparatus **200**. The apparatus **200** is connected to an electrochemical analyzer **212** in a three electrode system via the electrical contact **204**. The apparatus **200** is inserted into a liquid electrolyte **213** in a tank **214**. A reference electrode **215** and a counter electrode **216**, connected with the electrochemical analyzer **212**, are inserted into the liquid electrolyte **213**. Then, the tank **214** is placed on a magnetic stirrer plate **217** of a magnetic stirrer **218**. The magnetic stirrer **218** can generate rotating magnetic field by a rotating magnet or a set of stationary electromagnets. Once the magnetic stirrer **218** is switched on, the generated rotating magnetic field drives the magnetic stir bar **202** to rotate. The rotation of the magnetic stir bar **202** generates flow of the liquid electrolyte **213**, which can create a relative motion symmetrically between the electrolytic surface **207** of the disk electrode **203** and a bulk portion of the liquid electrolyte **213**. As the magnetic stir bar **202** is located on top of the electrolytic surface **207** in parallel with each other, the magnetic stir bar **202** is able to create a flow condition on the electrolytic surface **207** substantially similar to that created by the conventional rotating disk electrode. As such, a laminar flow of the liquid electrolyte **213** towards and across the electrolytic surface **207** is

created. This flow can achieve a condition that a steady-state current is obtained, and mainly dictated by the solution flow rather than diffusion during chemical deposition. Under chemical deposition on the electrolytic surface **207** and the transmission of the corresponding current via the electrical contact **204**, the composition concentration of the liquid electrolyte **213** can be evaluated. Meanwhile, since there is mutual magnetic attraction between the magnetic stir bar **202** and the magnetic stirrer **218**, floating up of the magnetic stir bar **202** during rotation can be avoided such that the steady flow condition can be maintained.

[0034] Flow simulation of the present invention is conducted to verify the corresponding performance. Several liquid flow simulations on an electrolytic surface are performed under a convention rotating disk electrode and the apparatus of the present invention at rotational speeds of 900 rpm and 1500 rpm respectively. As shown in FIGs. **3A-B**, the simulation results show that the apparatus of the present invention is able to provide the flow condition similar to that of the traditional rotating disk electrode. Similarly, as shown in FIGs. **4A-B**, the simulation results show that the apparatus of the present invention is also able to provide the flow condition similar to that of the traditional rotating disk electrode even at a higher rotational speed.

[0035] The rotational speed of the rotatable unit can be in a range of 50 to 1500 rpm, depending on the type of electrochemical bath to be analyzed. The rotation speed of the magnetic stirrer can be in a range of 0 to 1700 rpm. The rotatable unit, preferably, comprises from two to five blades being arranged in a symmetrical way such that the center of the gravity can keep at the center of the rotatable unit during rotation so as to avoid swinging, which may affect generation of steady liquid flow. The blade can be a straight blade or twisted blade for generating steady liquid flow. The electrical contact can be a simple electrical wire or a bundle of electrical wires for transmitting current since the electrode is always kept static during measurement. The shape of the static working electrode is cylindrical for holding the rotatable unit and facilitating its rotational movement.

[0036] According to the present invention, as the electrode provides static axis with electrical lines going through, the design of the electrical contact is substantially simplified, comparing with that of the rotating disk electrode. For example, a simple electrical wire,

connected between the disk electrode and the chemical analyzer, is able to perform effective current measurement. The complex electrical contact mechanism required by the rotating disk electrode is avoided in the present invention.

[0037] According to another specific embodiment of the present invention, an electrochemical analytical apparatus comprising a motorized stirrer is provided. As shown in FIG. 5, the apparatus **500** comprises a static disk electrode **501** and a motorized stirrer **502**. The static disk electrode **501** is similar to that of FIG. 2A. The motorized stirrer **502** has a connecting structure **503** and two blades **504a**, **504b**, and further comprises a motor incorporated inside the connecting structure **503** to drive the rotation of the motorized stirrer **502**. In this embodiment, there is no need to have an additional magnetic stirrer for driving the motorized stirrer, and plastic or metal blades can be used.

[0038] As shown in FIG. 6, according to an embodiment of the present invention, an electrochemical analytical apparatus **600** comprises a disk electrode **601**, an inert material sheath **602** of the electrode, a structural support **603** of the electrode, an electrical contact **604** of the electrode, and a motorized stirrer **605**. The motorized stirrer **605** comprises blades **606**, a connecting structure **607**, and an electric motor **608**. The connecting structure **607** connects the blades **606** and the motor **608** together. The motor **608** is mounted on the structural support **603**, and located above the surface of an electrochemical bath during operation. The motor **608** can drive the blades **606** to rotate mechanically below the surface of the electrochemical bath.

[0039] The embodiments were chosen and described in order to best explain the principles of the invention and its practical application, thereby enabling others skilled in the art to understand the invention for various embodiments and with various modifications that are suited to the particular use contemplated. It is intended that the scope of the invention be defined by the following claims and their equivalence.

Claims:

What is claimed is:

1. An electrochemical analytical apparatus for electrochemical bath analysis, comprising:
a static working electrode having at least one electrolytic surface for being inserted into a liquid electrolyte;
a rotatable unit comprising at least one blade and located near the static working electrode for creating flow of the liquid electrolyte by rotation; and
a control unit for controlling the rotation of the rotatable unit for arousing a relative motion between the electrolytic surface of the working electrode and a bulk portion of the liquid electrolyte.
2. The apparatus of claim 1, wherein the aroused relative motion is symmetrical relative to the at least one electrolytic surface of the static working electrode.
3. The apparatus of claim 1, wherein the static working electrode is connected with an electrical contact.
4. The apparatus of claim 1, wherein the static working electrode is a disk electrode or a ring disk electrode.
5. The apparatus of claim 1, wherein the rotatable unit is located along a vertical axis of the at least one electrolytic surface of the static working electrode and rotates above the at least one electrolytic surface of the static working electrode.
6. The apparatus of claim 1, wherein the rotatable unit further comprises two or more blades being symmetrically arranged relative to the at least one electrolytic surface of the static working electrode.

7. The apparatus of claim 1, wherein the control unit controls rotation speed of the rotatable unit.
8. The apparatus of claim 1, wherein the rotatable unit is a magnetic stir bar freely mounted on a body of the static working electrode.
9. The apparatus of claim 8, wherein the control unit is a magnetic stirrer detachably coupled to the magnetic stir bar.
10. The apparatus of claim 1, wherein the rotatable unit and the control unit are integrated into a motorized system.
11. The apparatus of claim 1, further comprising a reference electrode and a counter electrode.
12. An electrochemical analytical apparatus for electrochemical bath analysis, comprising:
 - a static working electrode having at least one electrolytic surface located at bottom of the static working electrode for being inserted into a liquid electrolyte;
 - an electrical contact connected to the static working electrode for transmitting current;
 - a magnetic stir bar comprising a hole and at least two blades and located on top of the electrolytic surface for creating flow of the liquid electrolyte by rotation, wherein the hole is for accommodating the static working electrode, and the at least two blades are arranged in a symmetrical way to each other; and
 - a magnetic stirrer for generating electric field for controlling the rotation of the magnetic stir bar to arouse a relative motion between the electrolytic surface of the static working electrode and a bulk portion of the liquid electrolyte;

wherein the aroused relative motion is symmetrical relative to the at least one electrolytic surface of the static working electrode.

13. The apparatus of claim 12, wherein the static working electrode is a disk electrode or a ring disk electrode.

14. The apparatus of claim 12, wherein the electrical contact is an electrical wire or a bundle of electrical wires.

15. The apparatus of claim 12, wherein a center of the magnetic stir bar is located along a vertical axis of the electrolytic surface.

16. An electrochemical analytical apparatus for electrochemical bath analysis, comprising:
a static working electrode having at least one electrolytic surface located at bottom of the static working electrode for being inserted into a liquid electrolyte;
an electrical contact connected to the static working electrode for transmitting current; and
a motorized stirrer comprising a motor, a hole and at least two blades and located on top of the electrolytic surface for creating flow of the liquid electrolyte by rotation to arouse a relative motion between the electrolytic surface of the static working electrode and a bulk portion of the liquid electrolyte, wherein the motor is for driving the rotation of the motorized stirrer, and the hole is for accommodating the static working electrode, and the at least two blades are arranged in a symmetrical way to each other;
wherein the aroused relative motion is symmetrical relative to the at least one electrolytic surface of the static working electrode.

17. The apparatus of claim 16, wherein the static working electrode is a disk electrode or a ring disk electrode.

18. The apparatus of claim 16, wherein the electrical contact is an electrical wire or a bundle of electrical wires.

19. The apparatus of claim 16, wherein a center of the motorized stirrer is located along a vertical axis of the electrolytic surface.

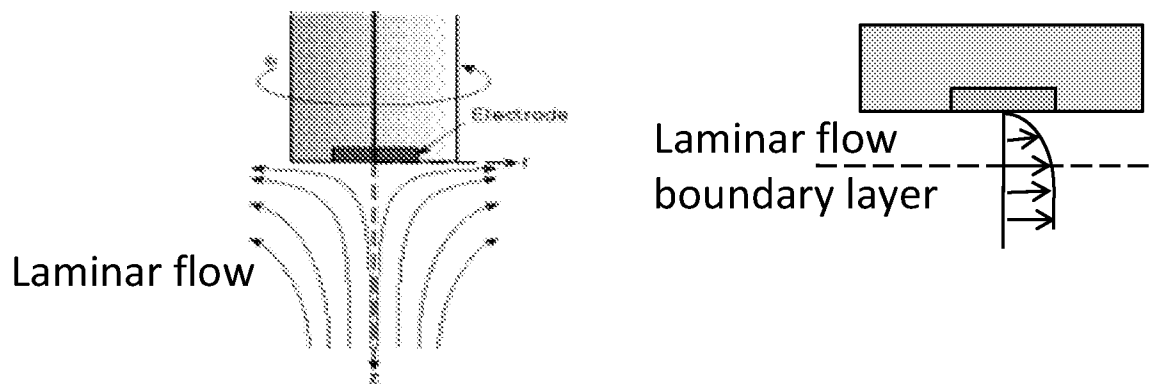


FIG. 1A (Prior Art)

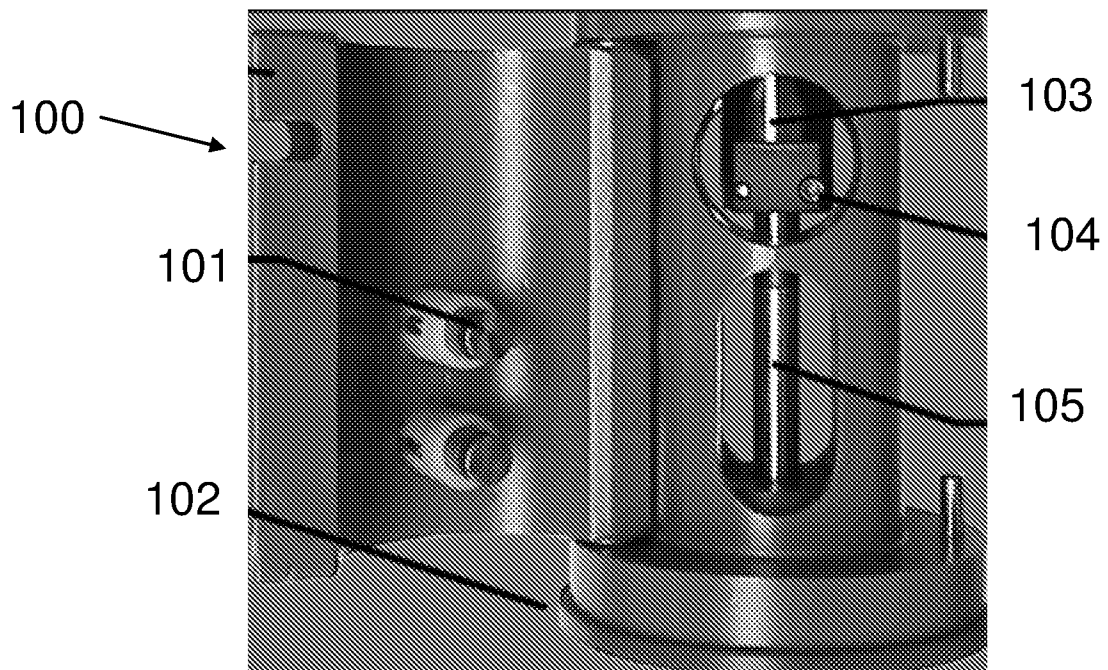


FIG. 1B (Prior Art)

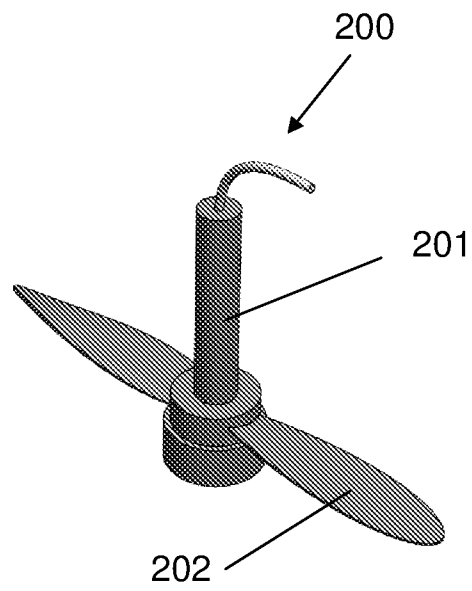


FIG. 2A

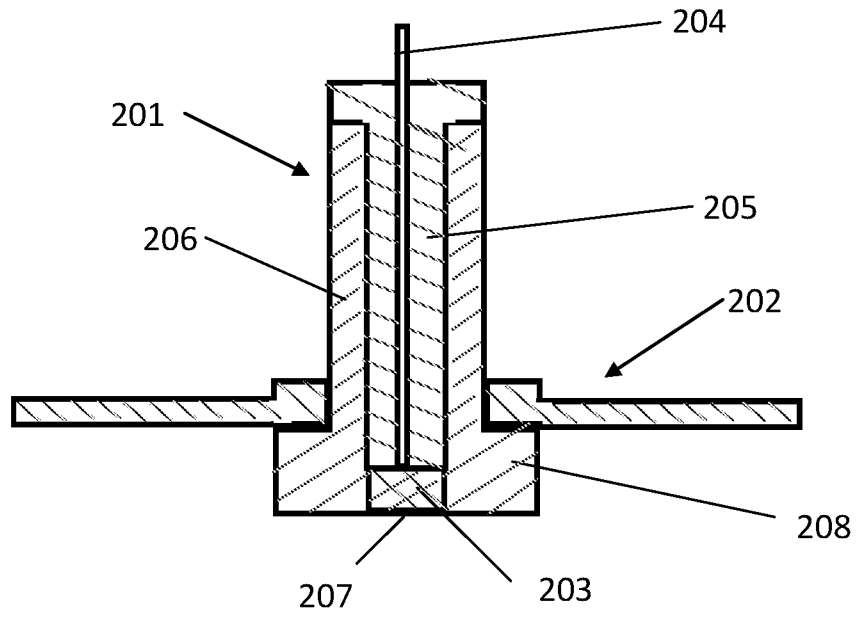


FIG. 2B

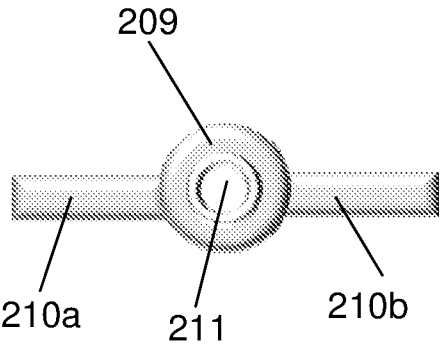


FIG. 2C

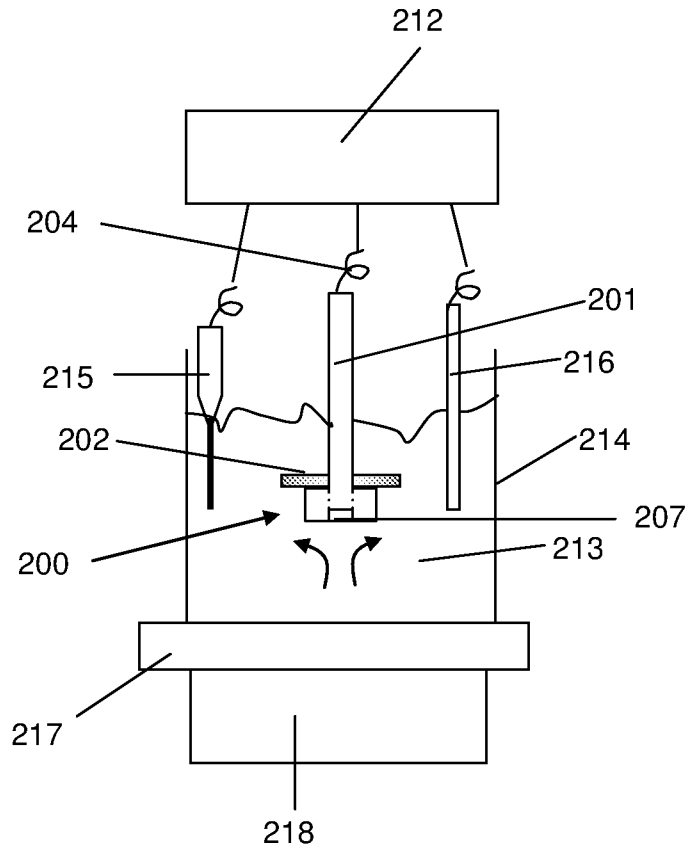
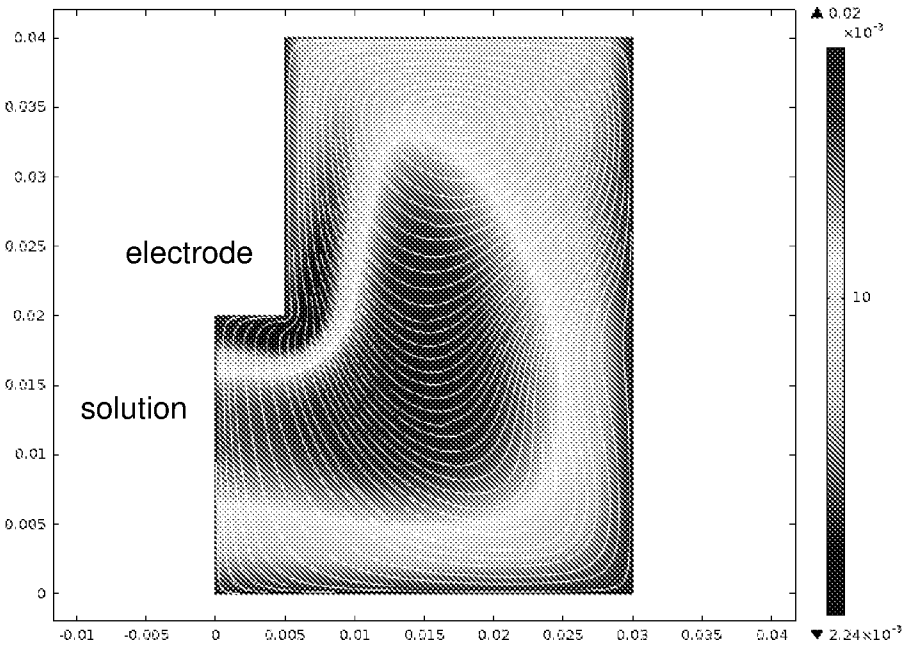
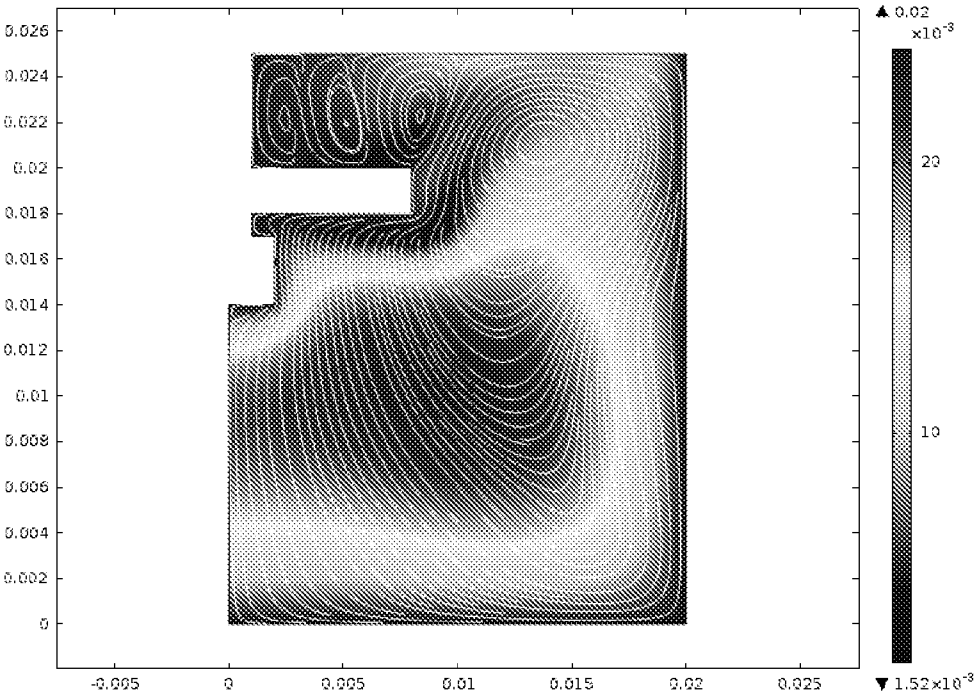


FIG. 2D

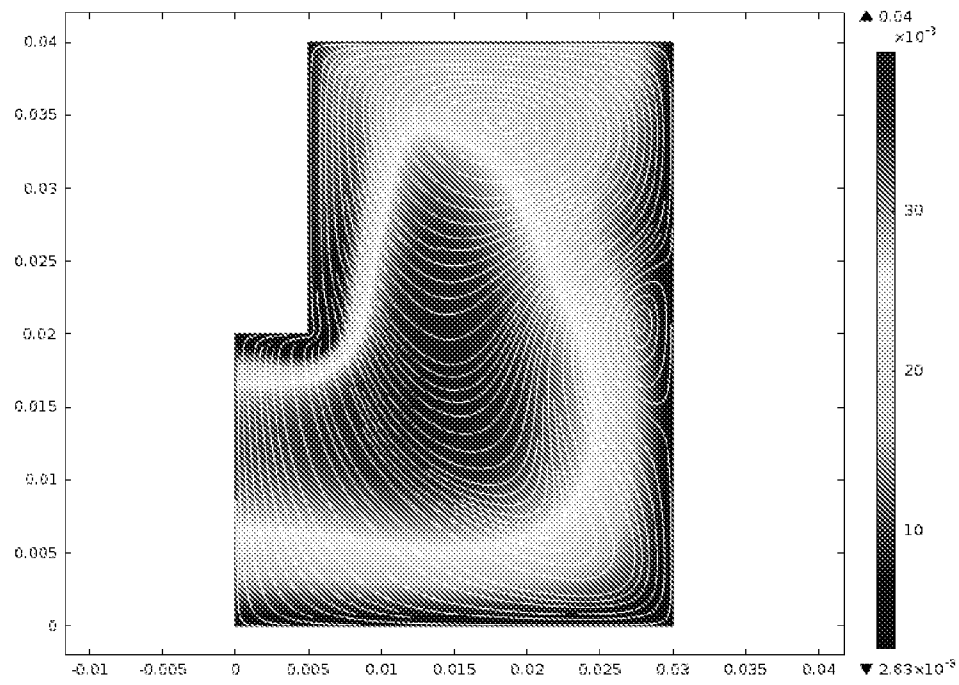


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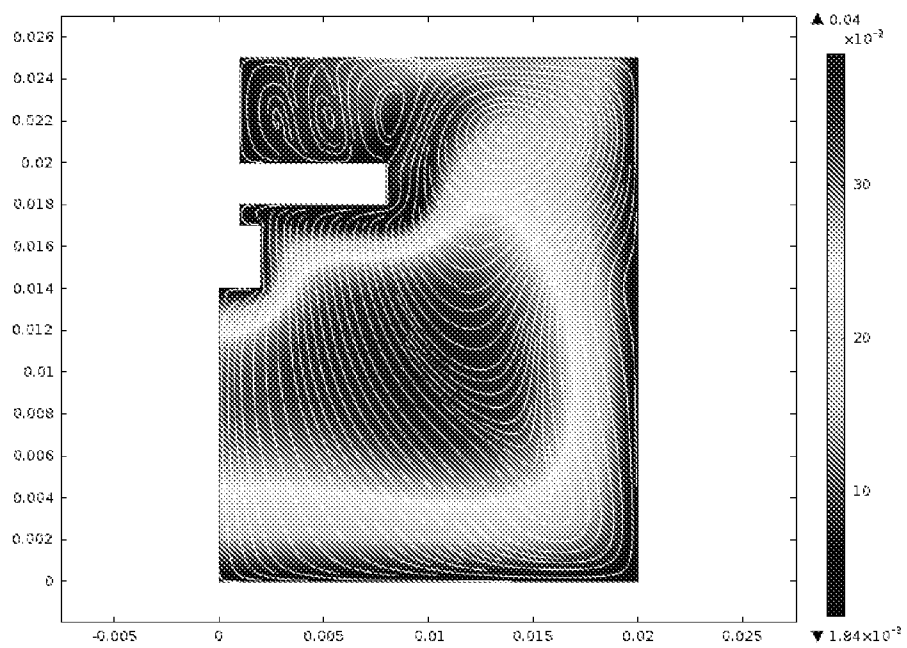


(B)

FIG. 3



(A)



(B)

FIG. 4

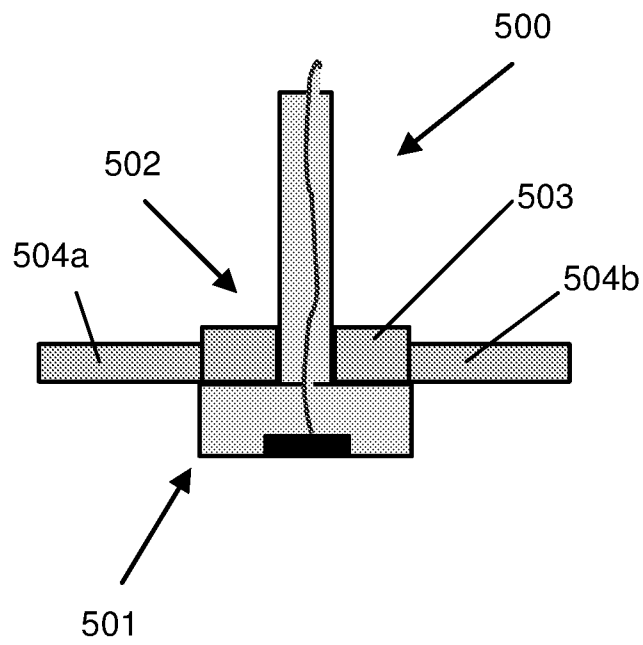


FIG. 5

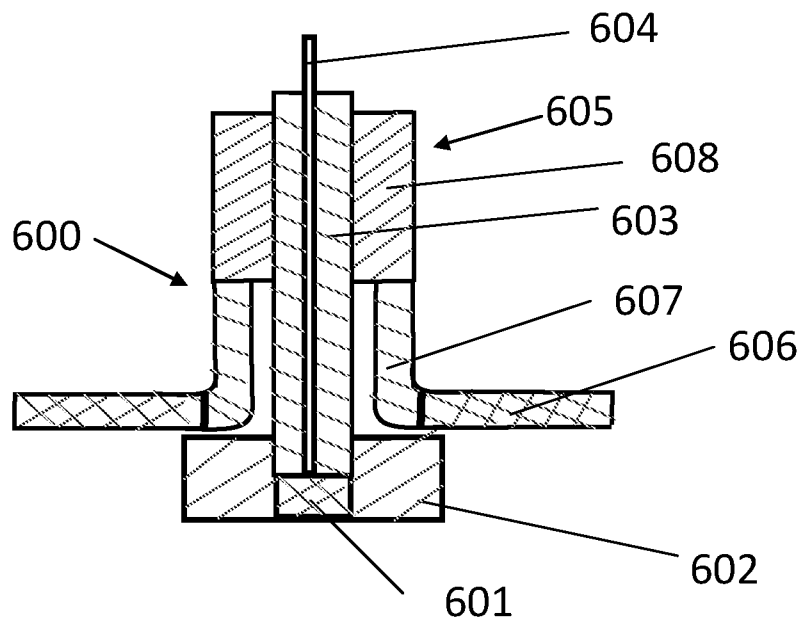


FIG. 6

INTERNATIONAL SEARCH REPORT

International application No.

PCT/CN2016/083925

A. CLASSIFICATION OF SUBJECT MATTER

G01N 27/26(2006.01)i; C25D 21/12(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)

G01N, C25D

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

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

CNABS,CNKI,EPODOC,WPI,:electrochem+,electroplat+,electroly+,analy+,electrode?,rotat+,blade?,stir+

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2015167195 A1 (HK APPLIED SCIENCE & TECH RES) 18 June 2015 (2015-06-18) description, paragraphs 33-35, 61-63	1-4, 6-7, 10-11
A	CN 101470097 A (BYD CO LTD) 01 July 2009 (2009-07-01) the whole document	1-19
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A	EP 1503169 A2 (KANSAI PAINT CO LTD) 02 February 2005 (2005-02-02) the whole document	1-19



Further documents are listed in the continuation of Box C.



See patent family annex.

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“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

22 September 2016

Date of mailing of the international search report

08 October 2016

Name and mailing address of the ISA/CN

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INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.

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