

Published:
— with international search report (Art. 21(3))
— before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments (Rule 48.2(h))

(88) Date of publication of the international search report: 13 November 2014

(54) Title: FABRICATION OF NANOPORES IN ATOMICALLY-THIN MEMBRANES BY ULTRA-SHORT ELECTRICAL PULSING

FIG. 5

Electrical Fabrication of Nanopore in Graphene Membranes

(57) Abstract: In a method for forming nanopores, two opposing surfaces of a membrane are exposed to an electrically conducting liquid environment. A nanopore nucleation voltage pulse, having a first nucleation pulse amplitude and duration, is applied between the two membrane surfaces, through the liquid environment. After applying the nanopore nucleation voltage pulse, the electrical conductance of the membrane is measured and compared to a first prespecified electrical conductance. Then at least one additional nanopore nucleation voltage pulse is applied between the two membrane surfaces, through the liquid environment, if the measured electrical conductance is no greater than the first prespecified electrical conductance.
### A. CLASSIFICATION OF SUBJECT MATTER

**IPC (8) -** B02Y 30/00; C25F 7/00 (2014.01)

**CPC -** B01D 67/009, B01D 2325/02

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)

CPC - B01D 67/009, 2325/02

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

USPC- 204/520, 627

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

PubWEST (USPT,PGPB,JPAB,EPAB, Google): Search Terms: Nanopore membrane nanostructure graphene electrolyte conductive liquid nucleation seed current electricity voltage pulse duration amplitude frequency second multiple plural

### C. DOCUMENTS CONSIDERED TO BE RELEVANT

<table>
<thead>
<tr>
<th>Category</th>
<th>Citation of document, with indication, where appropriate, of the relevant passages</th>
<th>Relevant to claim No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>WO 2008/060324 A2 (White et al.) 22 May 2008 (22.05.2008), entire document</td>
<td>1-22</td>
</tr>
</tbody>
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* Further documents are listed in the continuation of Box C.

**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

02 September 2014 (02.09.2014)

### Date of mailing of the international search report

19 SEP 2014

### Name and mailing address of the ISA/US

Mail Stop PCT, Attn: ISA/US, Commissioner for Patents
P.O. Box 1450, Alexandria, Virginia 22313-1450
Facsimile No. 571-273-3201

### Authorized officer:

Lee W. Young

PCT Helpdesk: 571-272-4300
PCT OSP: 571-272-7774
This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. □ Claims Nos.: because they relate to subject matter not required to be searched by this Authority, namely:

2. □ Claims Nos.: because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3. □ Claims Nos.: because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

This International Searching Authority found multiple inventions in this international application, as follows:

--- See Continuation Sheet ---

1. □ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.

2. □ As all searchable claims could be searched without effort justifying additional fees, this Authority did not invite payment of additional fees.

3. □ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:

4. □ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.: 1-30

Remark on Protest

□ The additional search fees were accompanied by the applicant's protest and, where applicable, the payment of a protest fee.

□ The additional search fees were accompanied by the applicant's protest but the applicable protest fee was not paid within the time limit specified in the invitation.

□ No protest accompanied the payment of additional search fees.
Box III: Observations where unity of invention is lacking:

This application contains the following inventions or groups of inventions which are not so linked as to form a single general inventive concept under PCT Rule 13.1. In order for all inventions to be examined, the appropriate additional examination fees must be paid.

Group I: claims 1-30: directed to a method for forming a nanopore in a membrane.

Group II: claims 31-50: directed to a method for wetting a surface.

The inventions listed as Groups I-II do not relate to a single general inventive concept under PCT Rule 13.1 because, under PCT Rule 13.2, they lack the same or corresponding special technical features for the following reasons:

Group I includes the special technical features of exposing two opposing surfaces of a membrane to an electrically conducting liquid environment; applying a nanopore nucleation voltage pulse, having a first nucleation pulse amplitude, between the two membrane surfaces, through the liquid environment, the nanopore nucleation voltage pulse having a pulse duration; after applying the nanopore nucleation voltage pulse, measuring the electrical conductance of the membrane and comparing the measured electrical conductance to a first prespecified electrical conductance; applying at least one additional nanopore nucleation voltage pulse between the two membrane surfaces, through the liquid environment, each additional nanopore nucleation voltage pulse being applied if the measured electrical conductance is no greater than the first prespecified electrical conductance; and applying at least one nanopore diameter tuning voltage pulse, having a tuning pulse voltage amplitude, between the two membrane surfaces, through the liquid environment, each nanopore diameter tuning voltage pulse being applied if the measured electrical conductance is greater than the first prespecified electrical conductance and no greater than a second prespecified electrical conductance, each nanopore diameter tuning voltage pulse having a pulse duration, which are not required by Group II.

Group II includes the special technical features of disposing a surface to be wetted in a local wetting environment; flushing the local wetting environment with a flushing gas for a flushing duration sufficient to remove substantially all air from the local wetting environment and replace the air with the flushing gas; preventing air from entering back into the local wetting environment; and introducing a liquid wetting solution into the local wetting environment, the flushing gas having at least one condition selected from the group consisting of 1) being chemically reactive with the liquid wetting solution to produce a chemical reaction product that is dissolvable in the liquid wetting solution and 2) being dissolvable in the liquid wetting solution, which are not required by Group I.

Groups I and II do not share any common technical features.

Groups I-II therefore lack unity under PCT Rule 13 because they do not share a same or corresponding special technical feature.