

# PATENT COOPERATION TREATY

From the  
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

To:  
SUSAN L. SMITH  
OWENS-ILLINOIS, INC.  
ONE MICHAEL OWENS WAY  
PERRYSBURG, OH 43551  
USA

Owens-Illinois, Inc.

JUN 09 2017

Environmental and Intellectual Property

## PCT

NOTIFICATION OF TRANSMITTAL OF  
INTERNATIONAL PRELIMINARY  
REPORT ON PATENTABILITY  
(Chapter II of the Patent Cooperation Treaty)

(PCT Rule 71.1)

Date of mailing  
(day/month/year)

Applicant's or agent's file reference

19318 WO

### IMPORTANT NOTIFICATION

International application No.

PCT/US16/24001

International filing date (day/month/year)

24 March 2016 (24.03.2016)

Priority date (day/month/year)

01 April 2015 (01.04.2015)

Applicant

OWENS-BROCKWAY GLASS CONTAINER INC.

1. The applicant is hereby notified that this International Preliminary Examining Authority transmits herewith the international preliminary report on patentability and its annexes, if any, established on the international application.
2. A copy of the report and its annexes, if any, is being transmitted to the International Bureau for communication to all the elected Offices.
3. Where required by any of the elected Offices, the International Bureau will prepare an English translation of the report (but not of any annexes) and will transmit such translation to those Offices.

#### 4. REMINDER

The applicant must enter the national phase before each elected Office by performing certain acts (filing translations and paying national fees) within 30 months from the priority date (or later in some Offices)(Article 39(1))(see also the reminder sent by the International Bureau with Form PCT/IB/301).

Where a translation of the international application must be furnished to an elected Office, that translation must contain a translation of any annexes to the international preliminary report on patentability. It is the applicant's responsibility to prepare and furnish such translation directly to each elected Office concerned.

For further details on the applicable time limits and requirements of the elected Offices, see Volume II of the *PCT Applicant's Guide*.

The applicant's attention is drawn to Article 33(5), which provides that the criteria of novelty, inventive step and industrial applicability described in Article 33(2) to (4) merely serve the purposes of international preliminary examination and that "any Contracting State may apply additional or different criteria for the purposes of deciding whether, in that State, the claimed invention is patentable or not" (see also Article 27(5)). Such additional criteria may relate, for example, to exemptions from patentability, requirements for enabling disclosure, clarity and support for the claims.

Name and mailing address of the IPEA/ US

Mail Stop PCT, Attn: IPEA/US  
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Authorized officer

Joe Del Sole

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# PATENT COOPERATION TREATY

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# PATENT COOPERATION TREATY

## PCT

### INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

(Chapter II of the Patent Cooperation Treaty)

(PCT Article 36 and Rule 70)

|                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                         |                                                                                   |                                                                                                                                                                  |                                     |           |                     |                          |            |          |                          |             |                                                                                                  |                                     |            |                            |                                     |           |                                                                                                                                                                  |                          |            |                         |                                     |             |                                                  |                          |              |                                                       |
|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------------------|-----------|---------------------|--------------------------|------------|----------|--------------------------|-------------|--------------------------------------------------------------------------------------------------|-------------------------------------|------------|----------------------------|-------------------------------------|-----------|------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------|------------|-------------------------|-------------------------------------|-------------|--------------------------------------------------|--------------------------|--------------|-------------------------------------------------------|
| Applicant's or agent's file reference<br>19318 WO                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                       | <b>FOR FURTHER ACTION</b>                                                         | See Form PCT/IPEA/416                                                                                                                                            |                                     |           |                     |                          |            |          |                          |             |                                                                                                  |                                     |            |                            |                                     |           |                                                                                                                                                                  |                          |            |                         |                                     |             |                                                  |                          |              |                                                       |
| International application No.<br>PCT/US16/24001                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                         | International filing date ( <i>day/month/year</i> )<br>24 March 2016 (24.03.2016) | Priority date ( <i>day/month/year</i> )<br>01 April 2015 (01.04.2015)                                                                                            |                                     |           |                     |                          |            |          |                          |             |                                                                                                  |                                     |            |                            |                                     |           |                                                                                                                                                                  |                          |            |                         |                                     |             |                                                  |                          |              |                                                       |
| International Patent Classification (IPC) or national classification and IPC<br>IPC: <b>C03B 9/193</b> ( 2006.01); <b>C03C 1/00</b> ( 2006.01)<br>CPC: C03B 9/193; C03C 1/006                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           |                                                                                   |                                                                                                                                                                  |                                     |           |                     |                          |            |          |                          |             |                                                                                                  |                                     |            |                            |                                     |           |                                                                                                                                                                  |                          |            |                         |                                     |             |                                                  |                          |              |                                                       |
| Applicant<br>OWENS-BROCKWAY GLASS CONTAINER INC.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |                                                                                   |                                                                                                                                                                  |                                     |           |                     |                          |            |          |                          |             |                                                                                                  |                                     |            |                            |                                     |           |                                                                                                                                                                  |                          |            |                         |                                     |             |                                                  |                          |              |                                                       |
| <p>1. This report is the international preliminary examination report, established by this International Preliminary Examining Authority under Article 35 and transmitted to the applicant according to Article 36.</p> <p>2. This REPORT consists of a total of <u>9</u> sheets, including this cover sheet.</p> <p>3. This report is also accompanied by ANNEXES, comprising:</p> <p style="margin-left: 20px;">a. <input checked="" type="checkbox"/> (<i>sent to the applicant and to the International Bureau</i>) a total of <u>25</u> sheets, as follows:</p> <div style="margin-left: 40px;"> <p><input checked="" type="checkbox"/> sheets of the description, claims and/or drawings which have been amended and/or sheets containing rectifications authorized by this Authority, unless those sheets were superseded or cancelled, and any accompanying letters (see Rules 46.5, 66.8, 70.16, 91.2, and Section 607 of the Administrative Instructions).</p> <p><input type="checkbox"/> sheets containing rectifications, where the decision was made by this Authority not to take them into account because they were not authorized by or notified to this Authority at the time when this Authority began to draw up this report, and any accompanying letters (Rules 66.4<i>bis</i>, 70.2(e), 70.16 and 91.2).</p> <p><input type="checkbox"/> superseded sheets and any accompanying letters, where this Authority either considers that the superseding sheets contain an amendment that goes beyond the disclosure in the international application as filed, or the superseding sheets were not accompanied by a letter indicating the basis for the amendments in the application as filed, as indicated in item 4 of Box No.I and the Supplemental Box (see Rule 70.16(b)).</p> </div> <p style="margin-left: 20px;">b. <input type="checkbox"/> (<i>sent to the International Bureau only</i>) a total of (indicate type and number of electronic carrier(s)) _____ containing a sequence listing, in the form of an Annex C/ST.25 text file, as indicated in the Supplemental Box Relating to Sequence Listing (see paragraph 3<i>ter</i> of Annex C of the Administrative Instructions).</p> |                                                                                   |                                                                                                                                                                  |                                     |           |                     |                          |            |          |                          |             |                                                                                                  |                                     |            |                            |                                     |           |                                                                                                                                                                  |                          |            |                         |                                     |             |                                                  |                          |              |                                                       |
| <p>4. This report contains indications relating to the following items:</p> <table style="width: 100%; border: none;"> <tr> <td style="width: 10%;"><input checked="" type="checkbox"/></td> <td style="width: 30%;">Box No. I</td> <td style="width: 60%;">Basis of the report</td> </tr> <tr> <td><input type="checkbox"/></td> <td>Box No. II</td> <td>Priority</td> </tr> <tr> <td><input type="checkbox"/></td> <td>Box No. III</td> <td>Non-establishment of opinion with regard to novelty, inventive step and industrial applicability</td> </tr> <tr> <td><input checked="" type="checkbox"/></td> <td>Box No. IV</td> <td>Lack of unity of invention</td> </tr> <tr> <td><input checked="" type="checkbox"/></td> <td>Box No. V</td> <td>Reasoned statement under Article 35(2) with regard to novelty, inventive step and industrial applicability; citations and explanations supporting such statement</td> </tr> <tr> <td><input type="checkbox"/></td> <td>Box No. VI</td> <td>Certain documents cited</td> </tr> <tr> <td><input checked="" type="checkbox"/></td> <td>Box No. VII</td> <td>Certain defects in the international application</td> </tr> <tr> <td><input type="checkbox"/></td> <td>Box No. VIII</td> <td>Certain observations on the international application</td> </tr> </table>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      |                                                                                   |                                                                                                                                                                  | <input checked="" type="checkbox"/> | Box No. I | Basis of the report | <input type="checkbox"/> | Box No. II | Priority | <input type="checkbox"/> | Box No. III | Non-establishment of opinion with regard to novelty, inventive step and industrial applicability | <input checked="" type="checkbox"/> | Box No. IV | Lack of unity of invention | <input checked="" type="checkbox"/> | Box No. V | Reasoned statement under Article 35(2) with regard to novelty, inventive step and industrial applicability; citations and explanations supporting such statement | <input type="checkbox"/> | Box No. VI | Certain documents cited | <input checked="" type="checkbox"/> | Box No. VII | Certain defects in the international application | <input type="checkbox"/> | Box No. VIII | Certain observations on the international application |
| <input checked="" type="checkbox"/>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     | Box No. I                                                                         | Basis of the report                                                                                                                                              |                                     |           |                     |                          |            |          |                          |             |                                                                                                  |                                     |            |                            |                                     |           |                                                                                                                                                                  |                          |            |                         |                                     |             |                                                  |                          |              |                                                       |
| <input type="checkbox"/>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                | Box No. II                                                                        | Priority                                                                                                                                                         |                                     |           |                     |                          |            |          |                          |             |                                                                                                  |                                     |            |                            |                                     |           |                                                                                                                                                                  |                          |            |                         |                                     |             |                                                  |                          |              |                                                       |
| <input type="checkbox"/>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                | Box No. III                                                                       | Non-establishment of opinion with regard to novelty, inventive step and industrial applicability                                                                 |                                     |           |                     |                          |            |          |                          |             |                                                                                                  |                                     |            |                            |                                     |           |                                                                                                                                                                  |                          |            |                         |                                     |             |                                                  |                          |              |                                                       |
| <input checked="" type="checkbox"/>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     | Box No. IV                                                                        | Lack of unity of invention                                                                                                                                       |                                     |           |                     |                          |            |          |                          |             |                                                                                                  |                                     |            |                            |                                     |           |                                                                                                                                                                  |                          |            |                         |                                     |             |                                                  |                          |              |                                                       |
| <input checked="" type="checkbox"/>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     | Box No. V                                                                         | Reasoned statement under Article 35(2) with regard to novelty, inventive step and industrial applicability; citations and explanations supporting such statement |                                     |           |                     |                          |            |          |                          |             |                                                                                                  |                                     |            |                            |                                     |           |                                                                                                                                                                  |                          |            |                         |                                     |             |                                                  |                          |              |                                                       |
| <input type="checkbox"/>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                | Box No. VI                                                                        | Certain documents cited                                                                                                                                          |                                     |           |                     |                          |            |          |                          |             |                                                                                                  |                                     |            |                            |                                     |           |                                                                                                                                                                  |                          |            |                         |                                     |             |                                                  |                          |              |                                                       |
| <input checked="" type="checkbox"/>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     | Box No. VII                                                                       | Certain defects in the international application                                                                                                                 |                                     |           |                     |                          |            |          |                          |             |                                                                                                  |                                     |            |                            |                                     |           |                                                                                                                                                                  |                          |            |                         |                                     |             |                                                  |                          |              |                                                       |
| <input type="checkbox"/>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                | Box No. VIII                                                                      | Certain observations on the international application                                                                                                            |                                     |           |                     |                          |            |          |                          |             |                                                                                                  |                                     |            |                            |                                     |           |                                                                                                                                                                  |                          |            |                         |                                     |             |                                                  |                          |              |                                                       |
| Date of submission of the demand<br>20 January 2017 (20.01.2017)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        | Date of completion of this report<br>28 May 2017 (28.05.2017)                     |                                                                                                                                                                  |                                     |           |                     |                          |            |          |                          |             |                                                                                                  |                                     |            |                            |                                     |           |                                                                                                                                                                  |                          |            |                         |                                     |             |                                                  |                          |              |                                                       |
| Name and mailing address of the IPEA/ US<br>Mail Stop PCT, Attn: IPEA/US<br>Commissioner for Patents<br>P.O. Box 1450<br>Alexandria, Virginia 22313-1450<br>Facsimile No. (571) 273-8300                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                | Authorized officer<br><br>Joe Del Sole<br><br>Telephone No. (571) 272-1700        |                                                                                                                                                                  |                                     |           |                     |                          |            |          |                          |             |                                                                                                  |                                     |            |                            |                                     |           |                                                                                                                                                                  |                          |            |                         |                                     |             |                                                  |                          |              |                                                       |

Form PCT/IPEA/409 (cover sheet) (January 2015)

**Box No. I Basis of the report**

1. With regard to the **language**, this report is based on:

- ☒ the international application in the language in which it was filed.
- ☐ a translation of the international application into English which is the language of a translation furnished for the purposes of:
- ☐ international search (Rules 12.3(a) and 23.1(b)).
  - ☐ publication of the international application (Rule 12.4(a)).
  - ☐ international preliminary examination (Rules 55.2(a) and/or 55.3(a) and (b)).

2. With regard to the **elements** of the international application, this report is based on (*replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report*):

- ☐ the international application as originally filed/furnished
- ☒ the description:  
pages 1,3-6,8,9,11-13,15,18,19 and 21-29 as originally filed/furnished  
pages\* 2,7,10,14,16,17 and 20 received by this Authority on 20 January 2017 (20.01.2017)  
pages\* NONE received by this Authority on \_\_\_\_\_
- ☒ the claims:  
Nos. NONE as originally filed/furnished  
Nos.\* NONE as amended (together with any statement) under Article 19  
Nos.\* 30-35 received by this Authority on 20 January 2017 (20.01.2017)
- ☒ the drawings:  
pages 1/3 and 3/3 as originally filed/furnished  
pages\* 2/3 received by this Authority on 20 January 2017 (20.01.2017)  
pages\* NONE received by this Authority on \_\_\_\_\_

☐ a sequence listing - see Supplemental Box Relating to Sequence Listing.

3. ☐ The amendments have resulted in the cancellation of:

- ☐ the description, pages \_\_\_\_\_
- ☐ the claims, Nos. \_\_\_\_\_
- ☐ the drawings, sheets/figs \_\_\_\_\_
- ☐ the sequence listing (*specify*): \_\_\_\_\_

4. ☐ This report has been established as if (some of) the amendments annexed to this report and listed below had not been made, since either they are considered to go beyond the disclosure as filed, or they were not accompanied by a letter indicating the basis for the amendments in the application as filed, as indicated in the Supplemental Box (Rules 70.2(c) and (c-bis)):

- ☐ the description, pages \_\_\_\_\_
- ☐ the claims, Nos. \_\_\_\_\_
- ☐ the drawings, sheets/figs \_\_\_\_\_
- ☐ the sequence listing (*specify*): \_\_\_\_\_

5. ☐ This report has been established:

- ☐ taking into account the **rectification of an obvious mistake** authorized by or notified to this Authority under Rule 91 (Rules 66.1(d-bis) and 70.2(e)).
- ☐ without taking into account the **rectification of an obvious mistake** authorized by or notified to this Authority under Rule 91 (Rules 66.4bis) and 70.2(e)).

6. ☒ With regard to top-up searches (Rules 66.1ter and 70.2(f)):

- ☐ A top-up search was carried out by this Authority on \_\_\_\_\_
- ☐ Additional relevant documents have been discovered during the top-up search.
- ☒ No top-up search was carried out by this Authority because it would serve no useful purpose.

7. ☐ Supplementary international search report(s) from Authority(ies) \_\_\_\_\_ has/have been received and taken into account in establishing this report (Rule 45bis.8(b) and (c)).

\* If item 4 applies, some or all of those sheets may be marked "superseded."

**Box No. IV Lack of unity of invention**

1. ☒ In response to the invitation to restrict or pay additional fees the applicant has, within the applicable time limit:
- ☐ restricted the claims.
  - ☒ paid additional fees.
  - ☐ paid additional fees under protest, and, where applicable, the protest fee.
  - ☐ paid additional fees under protest but the applicable protest fee was not paid.
  - ☐ neither restricted the claims nor paid additional fees.
2. ☐ This Authority found that the requirement of unity of invention is not complied with and chose, according to Rule 68.1, not to invite the applicant to restrict or pay additional fees.
3. This Authority considers that the requirement of unity of invention in accordance with Rules 13.1, 13.2 and 13.3 is:
- ☐ complied with.
  - ☒ not complied with for the following reasons:

Please See Continuation Sheet

4. Consequently, this report has been established in respect of the following parts of the international application:
- ☒ all parts.
  - ☐ the parts relating to claims Nos. \_\_\_\_\_

## INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

International application No.  
PCT/US16/24001**Box No. V Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**

## 1. Statement

|                               |                    |     |
|-------------------------------|--------------------|-----|
| Novelty (N)                   | Claims <u>1-29</u> | YES |
|                               | Claims <u>NONE</u> | NO  |
| Inventive Step (IS)           | Claims <u>NONE</u> | YES |
|                               | Claims <u>1-29</u> | NO  |
| Industrial Applicability (IA) | Claims <u>1-29</u> | YES |
|                               | Claims <u>NONE</u> | NO  |

2. Citations and Explanations (Rule 70.7)  
Please See Continuation Sheet

**Box No. VII    Certain defects in the international application**

The following defects in the form or contents of the international application have been noted:

The drawings contain the following defect(s) in the form or content thereof: Original figure on page 3/3 and amended figure on page 2/3 are both labeled FIG. 3.

## Supplemental Box

In case the space in any of the preceding boxes is not sufficient.  
Continuation of:

**IV. 3. This Authority considers that the requirement of unity of invention is accordance with Rules 13.1, 13.2 and 13.3 is not complied with for the following reasons:**

## REQUIREMENT FOR UNITY OF INVENTION

As provided in 37 CFR 1.475(a), an international application shall relate to one invention only or to a group of inventions so linked as to form a single general inventive concept ("requirement of unity of invention"). Where a group of inventions is claimed in an international application, the requirement of unity of invention shall be fulfilled only when there is a technical relationship among those inventions involving one or more of the same or corresponding special technical features. The expression "special technical features" shall mean those technical features that define a contribution which each of the claimed inventions, considered as a whole, makes over the prior art.

The determination whether a group of inventions is so linked as to form a single general inventive concept shall be made without regard to whether the inventions are claimed in separate claims or as alternatives within a single claim. See 37 CFR 1.475(e).

## WHEN CLAIMS ARE DIRECTED TO MULTIPLE PROCESSES, PRODUCTS, AND/OR APPARATUSES

Products, processes of manufacture, processes of use, and apparatuses are different categories of invention. When an application includes claims to more than one product, process, or apparatus, the first invention of the category first mentioned in the claims of the application and the first recited invention of each of the other categories related thereto will be considered as the "main invention" in the claims. In the case of non-compliance with unity of invention and where no additional fees are timely paid, the international search and/or international preliminary examination, as appropriate, will be based on the main invention in the claims. See PCT Article 17(3)(a), 37 CFR 1.475(d), 37 CFR 1.476(c) and 37 CFR 1.488(b)(3).

As provided in 37 CFR 1.475(b), an international application containing claims to different categories of invention will be considered to have unity of invention if the claims are drawn only to one of the following combinations of categories:

- (1) A product and a process specially adapted for the manufacture of said product; or
- (2) A product and process of use of said product; or
- (3) A product, a process specially adapted for the manufacture of the said product, and a use of the said product; or
- (4) A process and an apparatus or means specifically designed for carrying out the said process; or
- (5) A product, a process specially adapted for the manufacture of the said product, and an apparatus or means specifically designed for carrying out the said process.

Otherwise, unity of invention might not be present. See 37 CFR 1.475(c).

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.

**Supplemental Box**

Group 1, claim(s) 1-5, drawn to a glass gel.

Group 2, claim(s) 6-20, drawn to a method of making glass products.

The groups of inventions listed above 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:

Groups 1 and 2 lack unity of invention because even though the inventions of these groups require the technical feature of a glass gel comprising 60-85% silica, 8-18% sodium oxide, and 5-15% calcium oxide, this technical feature is not a special technical feature as it does not make a contribution over the prior art in view of Hatcher (US 2004/0052861). Hatcher teaches a glass gel having 0-70% silica, 0-40% sodium oxide, and 0-40% calcium oxide (para. 0073).

**V. 2. Citations and Explanations:**

Claims 1-5 lack an inventive step under PCT Article 33(3) as being obvious over Beall (US 4,239,519) in view of Hench (US 5,196,382).

Beall teaches a glass gel having 50-70% silica, 2-10% sodium oxide, and 0-10% calcium oxide (col. 21 lines 48-52). Beall is silent to the density of the gel.

Hench teaches a glass gel. Hench teaches that a specific density of glass gels can be achieved by the heating conditions and suggest a glass gel density of 1.3 to 1.4 g/cm<sup>3</sup> (col. 3 lines 25-28). It would have been obvious to one of ordinary skill in the art to try the heating conditions of Hench to achieve a density of less than 2 g/cm<sup>3</sup> because Beall teaches that the glass gel may have varying densities (col. 8 lines 60-62).

Regarding claim 2, Beall teaches that the gel comprises aluminum oxide (col. 21 line 50).

Regarding claim 3, Hench teaches that the glass gel may have a coloring agent (col. 2 lines 3-4).

Regarding claim 4, Hench teaches a glass gel density of 1.3 to 1.4 g/cm<sup>3</sup> (col. 3 lines 25-28) but teaches that the glass may be further densified by heating (col. 3 lines 27-28). Therefore it would have been obvious to one of ordinary skill in the art that a density of 1.6 to 1.85 g/cm<sup>3</sup> could have been achieved by Hench's suggestion of further densifying the gel. Hench teaches that the gel has a surface area of 800 m<sup>2</sup>/g (col. 3 line 26).

Regarding claim 5, Hench teaches heating to 500°C to dispel gases (col. 3 lines 22-23).

Claim 6 lacks an inventive step under PCT Article 33(3) as being obvious over the prior art as applied in the immediately preceding paragraph and further in view of Jeanvoine (US 2002/0162358) and Rue (US 2011/0236846).

Beall as modified by Hench teaches a glass gel.

Jeanvoine teaches a method of melting glass using a submerged combustion melter (figure 1). Jeanvoine teaches that a feed material is fed into the melter and melted into a molten glass (para. 0032). Jeanvoine teaches that the molten glass is formed into a product (para. 0002).

Jeanvoine teaches that the method can be used to melt and form glass products having a highly varied types of compositions (para. 0047).

Rue teaches a method of melting glass using a submerged combustion melter. Rue teaches that the submerged melter can be fed with feed materials that are solids, liquids, or gels (para. 0022). It would have been obvious to one of ordinary skill in the art that the method of Jeanvoine could have fed the glass gel of modified Beall into the melter because Jeanvoine teaches that the starting material may be liquid or solid (para. 0030, 0032) and Rue teaches that gel starting materials are suitable for use in submerged combustion melters (para. 0022).

Claims 7-10 lacks an inventive step under PCT Article 33(3) as being obvious over the prior art as applied in the immediately preceding paragraph and further in view of Sugahara (US 4,018,616).

Beall as modified by Hench, Jeanvoine, and Rue teaches melting a glass gel and forming a product therefrom.

## Supplemental Box

Sugahara teaches a method of forming a glass precursor gel. Sugahara teaches providing a silicate solution that includes a dissolved alkali silicate, the dissolved alkali silicate having a molar ratio of an alkali metal oxide to silica (col. 10 lines 39-46); adding an alkaline earth salt to the silicate solution to displace some of the alkali oxide with an alkaline earth metal oxide (col. 12 lines 35-37) and to derive a wet precipitate having a molar ratio of the alkali metal oxide to the alkaline earth metal oxide to silica and removing liquid solvent from the wet precipitate to obtain the glass precursor gel (col. 10 lines 57-59).

It would have been obvious to one of ordinary skill in the art that the method of modified Beall could have used the gel of Sugahara because Sugahara teaches that the gel can be used for the production of molded articles (col. 13 lines 46-47).

Regarding claim 8, Sugahara teaches the molar ratio of the alkali oxide to silica in the dissolved alkali silicate can be adjusted by neutralizing some of the alkali oxide with an acid such that the molar ratio of the alkali oxide to silica is reduced (col. 8 lines 5-12).

Regarding claim 9, Jeanvoine teaches that the method can be used to melt and form glass products having a highly varied types of compositions (para. 0047) and teaches a variety of example compositions in paragraphs 0077, 0078, 0079, 0081, and 0084 therefore it would have been obvious to one of ordinary skill in the art from the variety of compositions taught by Jeanvoine that one of ordinary skill in the art could have achieved a molar ratio of the alkali oxide to the alkaline earth oxide to silica between 0.8:0.8:6 and 1.4:1.3:6 by routine experimentation.

Regarding claim 10, Jeanvoine teaches the step of melting the glass precursor gel is performed below 1400°C (para. 0039) which overlaps with the claimed range of 1200°C to 1450°C. Jeanvoine teaches that the method allows for better control of the residence time in the melting furnace (para. 0032) therefore it would have been obvious to one of ordinary skill in the art that a residence time of 30 minutes to 16 hours could have been achieved through routine experimentation.

Claims 11, 13-20 an inventive step under PCT Article 33(3) as being obvious over Jeanvoine (US 2002/0162358) in view of Rue (US 2011/0236846) and Sugahara (US 4,018,616).

Jeanvoine teaches a method of melting glass using a submerged combustion melter (figure 1). Jeanvoine teaches that a feed material is fed into the melter and melted into a molten glass (para. 0032). Jeanvoine teaches that the molten glass is formed into a product (para. 0002).

Jeanvoine teaches that the method can be used to melt and form glass products having a highly varied types of compositions (para. 0047). Jeanvoine teaches an example in paragraph 0077 that the glass composition may comprise 70-74.3% silica, which falls into the claimed range of 60-85%, and 11.1-13.3% sodium oxide, which overlaps with the claimed range of 8-18%, and 7.5-10% calcium oxide, which overlaps with the claimed range of 5-15%.

Rue teaches a method of melting glass using a submerged combustion melter. Rue teaches that the submerged melter can be fed with feed materials that are solids, liquids, or gels (para. 0022). It would have been obvious to one of ordinary skill in the art that the method of Jeanvoine could have feed glass gel precursor into the melter because Jeanvoine teaches that the starting material may be liquid or solid (para. 0030, 0032) and Rue teaches that gel starting materials are suitable for use in submerged combustion melters (para. 0022).

Sugahara teaches a method of forming a glass precursor gel. Sugahara teaches providing a silicate solution that includes a dissolved alkali silicate, the dissolved alkali silicate having a molar ratio of an alkali metal oxide to silica (col. 10 lines 39-46); adding an alkaline earth salt to the silicate solution to displace some of the alkali oxide with an alkaline earth metal oxide (col. 12 lines 35-37) and to derive a wet precipitate having a molar ratio of the alkali metal oxide to the alkaline earth metal oxide to silica and removing liquid solvent from the wet precipitate to obtain the glass precursor gel (col. 10 lines 57-59). Sugahara teaches that the silicate is sodium silicate (col. 10 lines 39-40) and that calcium hydroxide is added (col. 12 line 39).

It would have been obvious to one of ordinary skill in the art that the method of Jeanvoine could have used the gel of Sugahara because Sugahara teaches that the gel can be used for the production of molded articles (col. 13 lines 46-47).

Regarding claim 13, Sugahara teaches the molar ratio of the alkali oxide to silica in the dissolved alkali silicate can be adjusted by neutralizing some of the alkali oxide with an acid such that the molar ratio of the alkali oxide to silica is reduced (col. 8 lines 5-12).

Regarding claim 14, Jeanvoine teaches that the method can be used to melt and form glass products having a highly varied types of compositions (para. 0047) and teaches a variety of example compositions in paragraphs 0077, 0078, 0079, 0081, and 0084 therefore it would have been obvious to one of ordinary skill in the art from the variety of compositions taught by Jeanvoine that one of ordinary skill in the art could have achieved a molar ratio of the alkali oxide to the alkaline earth oxide to silica between 0.8:0.8:6 and 1.4:1.3:6 by routine experimentation.

Regarding claim 15, Jeanvoine teaches thermally conditioning the molten glass (para. 0003) at a temperature not exceeding 1500 °C (para. 0039) which incorporates the claimed range of about 1050°C to about 1200°C. Jeanvoine teaches delivering a gob of the molten glass to an individual section machine and forming the molten glass into a glass container in the individual section machine, the glass container having a glass body defining an interior accessible at a mouth located at one axial end of the glass body (para. 0002, 0110).

Regarding claim 16, Sugahara teaches the calcium salt is calcium hydroxide (col. 12 line 39).

Regarding claim 17, Jeanvoine teaches an example in paragraph 0077 that the glass composition may comprise 70-74.3% silica, therefore it would be expected that the silicate solution is concentrated to at least 5 wt.% of the dissolved sodium silicate at the time the soluble calcium salt is added to the silicate solution.

Regarding claim 18, Jeanvoine teaches the step of melting the soda-lime-silica glass precursor gel evolves oxygen gas (para.

**Supplemental Box**

0015) and teaches that the melting occurs below 1400°C (para. 0039) which overlaps with the claimed range of between 450°C and 1500°C.

Regarding claim 19, Jeanvoine teaches the step of melting the soda-lime-silica glass precursor gel does not release carbon dioxide gas (para. 0024).

Regarding claim 20, Jeanvoine teaches the step of melting the soda-lime-silica glass precursor gel evolves water vapor (para. 0037) and teaches that the melting occurs below 1400°C (para. 0039) which overlaps with the claimed range of above 125 °C.

Claim 12 lacks an inventive step under PCT Article 33(3) as being obvious over the prior art as applied in the immediately preceding paragraph and further in view of HU et al. (US 5,487,882).

Jeanvoine as modified by Rue and Sugahara teaches a method of forming glass articles by melting a glass gel. Sugahara teaches that the starting material for the silicate can be quartz (col. 6 lines 16-17). Modified Jeanvoine is silent to the dissolving conditions of the quartz.

Hu teaches that sodium silicate solutions can be prepared by dissolving quartz sand in a sodium hydroxide solution at a pressure of at least 100 psig (col. 4 lines 16-20) or at least 6.8 atm, which incorporates the claimed range of 10 atmospheres to 100 atmospheres, and a temperature of at least 130 °C (col. 4 lines 16-20), which overlaps with the claimed range of between 25°C and 300°C. It would have been obvious to one of ordinary skill in the art that time necessary to dissolve the sand would depend on the temperature and pressure conditions therefore it would have been obvious to one of ordinary skill in the art that a dissolving time of 3 hours to 24 hours could have been achieved through routine experimentation to optimize the dissolving conditions.

It would have been obvious to one of ordinary skill in the art that the sodium silicate solution of modified Jeanvoine could have been prepared by the method taught by Hu because Hu teaches that this is a suitable process for preparing sodium silicate (col. 4 lines 16-20).

----- NEW CITATIONS -----

US 4,239,519 (Beall et al.) 16 December 1980

US 5,196,382 (Hench) 23 March 1993

US 2002/0162358 (Jeanvoine et al.) 7 November 2002

US 2011/0236846 (Rue et al.) 29 September 2011

US 4,018,616 (Sugahara et al.) 19 April 1977

US 5,487,882 (Hu et al.) 30 January 1996

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including intermediate crystalline phases, are generally present up to about 1200°C, as melting and dissolution of these materials does not occur instantaneously. Second, the glass feedstock needs to be dispersed and homogeneously mixed by convection after being melted to produce molten glass, which is a time-consuming process. Quartz sand, in particular, takes the longest to disperse on account of its slow dissolution rate and the tendency to agglomerate into SiO<sub>2</sub>-rich regions within the glass melt known as “cord.” The presence of cord is indicative of glass inhomogeneity and may result in imperfections or defects in the finished glass product. Third, some of the virgin raw material ingredients—*e.g.*, soda ash and limestone for soda-lime-silica glass—are carbonate-containing materials that, when melted, release carbon dioxide (CO<sub>2</sub>). The evolution of carbon dioxide during feedstock melting introduces bubbles in the resultant molten glass, which, in turn, can cause a thin spot or bubble defect in the finished glass product. Any such bubbles are typically removed from the molten glass in a process known as “refining the glass.” To address the challenges associated with melting and homogenizing crystalline raw materials and to remove bubbles caused by carbon dioxide evolution, among other factors, conventional glass feedstocks are usually subjected to high temperatures and heating times of 24 hours or more in the glass furnace in order to obtain suitably-refined and chemically homogenized molten glass.

[0004] The melting of the glass feedstock can be made less taxing if some of the virgin raw materials are replaced with cullet in the feedstock. The cullet accelerates the melting of the feedstock and lowers furnace energy consumption as compared to a feedstock that contains all virgin raw materials. Cullet has this effect because it has already been melted, mixed, and formed into a glass product and will not release carbon dioxide when re-melted since it is not an intrinsic carbonate-containing material. But cullet is not widely available as a commodity in

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3 mol% to 15 mol% potassium oxide, 2 mol% to 10 mol% sodium oxide, or 4 mol% to 10 mol% barium oxide, as the primary constituent oxides.

[0016] In addition to the primary constituent oxides, the amorphous oxide-based matrix may optionally include a number of secondary materials that are commonly used in the glass manufacturing industry. Examples of such secondary materials are up to 25 mol% total of other glass network-formers, up to 2 mol% total of coloring and/or decoloring agents, and up to 20 mol% total of other materials that can affect the physical properties and/or the redox state of the final glass product composition. Specific other glass network-formers that may be added include one or more of phosphorus oxide ( $P_2O_5$ ) and germanium oxide ( $GeO_2$ ). Specific colorants and decolorants that may be added include the elemental forms or oxide compound forms of one or more of selenium, chromium, manganese, iron, cobalt, nickel, copper, niobium, molybdenum, silver, cadmium, indium, tin, gold, cerium, praseodymium, neodymium, europium, gadolinium, erbium, and uranium. And specific materials that can affect the physical properties and/or the redox state of the glass include one or more of carbon (0 mol% to 3 mol%), nitrates (0 mol% to 3 mol%), selenium (0 mol% to 1 mol%), titanium oxide ( $TiO_2$ ) (0 mol% to 5 mol%), arsenic oxide ( $As_2O_3$ ) (0 mol% to 2 mol%), vanadium oxide ( $V_2O_5$ ) (0 mol% to 5 mol%), fluorines (0 mol% to 2 mol%), chlorines (0 mol% to 2 mol%), and sulfates (0 mol% to 2 mol%).

[0017] The exact composition of the amorphous oxide-based matrix including the proportions of its primary constituent oxides and optional secondary materials can be varied to achieve any of the large variety of glass chemistries that may be desired in the final glass product. Both the physical and chemical properties of the resultant glass derived from the glass precursor gel can be affected by variances in the relative proportions of the primary constituent oxides as well as the inclusion or exclusion of certain secondary materials in the amorphous oxide-based matrix.

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therebetween, and a surface area of at least 20 m<sup>2</sup>/g, preferably about 25 m<sup>2</sup>/g to about 40 m<sup>2</sup>/g including all ranges and sub-ranges therebetween, as measured by nitrogen BET adsorption. The glass precursor gel is thus less dense and has a higher surface area than cullet.

[0020] The composition of the glass precursor gel facilitates rapid melting into molten glass while avoiding long residence times in the glass furnace. The glass precursor gel does not include large quantities of crystalline materials and, most notably, the amorphous oxide-based matrix does not contain any crystalline precursor materials of silica such as, for example, quartz sand. The absence of quartz sand is noteworthy here. Unlike conventional glass feedstocks that contain a fair amount of quartz sand, which generally has a slow dissolution rate, the glass precursor gel does not have to be maintained in a molten state at high temperatures for long durations in order to achieve satisfactory SiO<sub>2</sub> dissolution. The amorphous oxide-based matrix also includes virtually no carbonate-containing materials and, as such, will produce no more than a negligible amount of CO<sub>2</sub> upon melting and hence no more than a negligible amount of bubbles within the molten glass. The molten glass obtained from the glass precursor gel therefore does not have to be refined by removing substantial amounts of entrained bubbles through extended heating times. Given that the amorphous oxide-based matrix is already homogeneously chemically mixed and does not contain crystalline precursor materials of the primary constituent oxides or more than an insignificant amount of carbonate-containing materials, the glass precursor gel does not have to be heated to as high of a temperature or maintained at an elevated temperature for as long of a timeframe as conventional glass feedstocks in order to obtain a molten glass that is suitable for downstream processing into a glass product.

[0021] To be sure, as mentioned above, conventional soda-lime-silica glass feedstocks are typically melted in a furnace and maintained at 1400°C or higher for at least about 24 hours in

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[0026] The SLS glass precursor gel can be obtained in step 12 in numerous ways. In one embodiment, as depicted here in FIG. 1, the SLS glass precursor gel is obtained by (1) providing a silicate solution that includes a dissolved sodium silicate (step 12a); (2) adding a soluble calcium salt to the silicate solution to derive a wet precipitate that has the same proportions of the primary constituent oxides as the amorphous oxide-based matrix of the SLS glass precursor gel (step 12b); and (3) deriving the SLS glass precursor gel from the wet precipitate (step 12c). A specific implementation of steps 12a–12c will be described below in more detail. It should be understood, however, that the SLS glass precursor gel can also be obtained through other mechanisms not expressly described, but nonetheless known to skilled artisans, including alternative ways to provide the silicate solution in step 12a such as by purchasing the silicate solution or chemically preparing the solution by melting a mixture of quartz sand and soda ash and then dissolving the resultant product in water.

[0027] The silicate solution can be provided in step 12a by hydrothermally dissolving quartz sand in a caustic aqueous sodium-based solvent. The caustic aqueous sodium-based solvent may be a strong sodium base, such as sodium hydroxide (NaOH), which is preferably concentrated to greater than 10 wt.% (of the sodium base) so as to make dissolution of the quartz sand easier. One example of a suitable strong sodium base is 18 wt.% NaOH. The hydrothermal dissolution of quartz sand may be accomplished in a pressure vessel at a pressure above atmospheric pressure. There, the quartz sand may be dissolved in the caustic aqueous sodium-based solvent at a temperature between about 25°C and about 300°C including all ranges and sub-ranges therebetween, preferably between about 200°C and about 300°C, and a pressure of about 10 atmospheres to about 100 atmospheres including all ranges and sub-ranges therebetween, preferably about 30 atmospheres to about 50 atmospheres, over the course of about 3 hours to

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oxide, magnesium oxide, potassium oxide, iron oxide, titanium oxide, zirconium oxide, barium oxide, strontium oxide (SrO), sulfur trioxide (SO<sub>3</sub>), and oxides of selenium, cobalt, chromium, manganese, and lead. Aluminum oxide, in particular, which enhances the chemical durability of soda-lime-silica glass and reduces the tendency of devitrification in the molten glass, may be introduced by adding an alumina-bearing mineral to the caustic aqueous sodium-based solvent along with quartz sand under hydrothermal conditions. Adding the alumina-bearing mineral at this time results in aluminum oxide being chemically integrated into the dissolved sodium silicate during hydrothermal dissolution of the quartz sand. Some examples of alumina-bearing minerals that may be used here are nepheline syenite, aplite, and calumite slag. The aluminum oxide may also be introduced into the silicate solution in the form of dissolved sodium aluminate.

[0030] The list of secondary materials that may be introduced into the silicate solution during step 12a is more extensive than those just mentioned. Colorants and decolorants may be added such as one or more of iron oxides (*e.g.*, FeO and/or Fe<sub>2</sub>O<sub>3</sub>), chromium oxides (*e.g.*, CrO or Cr<sub>2</sub>O<sub>3</sub>), cobalt oxides (*e.g.*, CoO or Co<sub>2</sub>O<sub>3</sub>), nickel, copper, selenium, manganese dioxide, cerium oxide, titanium, and a combination of sulfur, iron, and carbon. Another class of secondary materials that can be added to the silicate solution is fining agents such as, for example, the combination of sodium sulfate (Na<sub>2</sub>SO<sub>4</sub>), carbon, arsenic oxide, and antimony oxide. Still further, oxidizers or reducers can be added to the silicate solution to modify, if desired, the redox number of the molten glass derived from the SLS glass precursor gel. Examples of common oxidizers and reducers include calcium sulfate (CaSO<sub>4</sub>), sodium nitrate (NaNO<sub>3</sub>), potassium nitrate (KNO<sub>3</sub>), iron pyrite (FeS<sub>2</sub>), and graphite.

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[0031] After the silicate solution has been formulated in step 12a, the soluble calcium salt may be added to the silicate solution in step 12b to derive a wet precipitate that has the same proportions of the primary constituent oxides (*e.g.*, silica, sodium, and calcium) desired in the amorphous oxide-based matrix of the SLS glass precursor gel. This step involves adding the soluble calcium salt to the silicate solution in an amount that brings the molar ratio of  $\text{Na}_2\text{O}:\text{CaO}:\text{SiO}_2$  in the wet precipitate to approximately 1:1:6, which is the desired molar ratio of the amorphous oxide-based matrix and, in turn, the soda-lime-silica glass produced from the SLS glass precursor gel. The soluble calcium salt may include, for example, calcium nitrate ( $\text{Ca}(\text{NO}_3)_2$ ), calcium chloride ( $\text{CaCl}_2$ ), calcium hydroxide ( $\text{Ca}(\text{OH})_2$ ) or any other soluble calcium salt or combination of soluble salts that provides a source of calcium ions. When added to the silicate solution, the calcium ions provided by the soluble calcium salt displace sodium in the dissolved sodium silicate, thus introducing calcium oxide into the silicate, which causes the newly-modified sodium silicate to precipitate out of solution as the wet precipitate. And since every 1 mole of calcium ions (which results in a corresponding mol of CaO) displaces 1 mole of  $\text{Na}_2\text{O}$  in the dissolved sodium silicate, the amount of the soluble calcium salt that needs to be added to the silicate solution to provide the wet precipitate with the 1:1:6 molar ratio of  $\text{Na}_2\text{O}:\text{CaO}:\text{SiO}_2$  can be easily calculated based on the molar ratio of  $\text{Na}_2\text{O}:\text{SiO}_2$  in the silicate solution from step 12a.

[0032] The displacement of  $\text{Na}_2\text{O}$  with CaO, and the precipitation of the wet precipitate, is preferably carried out with the silicate solution being as concentrated as possible. It has been found, in fact, that as the dissolved sodium silicate becomes more dilute in the silicate solution, the amount of  $\text{Na}_2\text{O}$  in the wet precipitate decreases while the amounts of  $\text{SiO}_2$  and CaO are not similarly affected. This, in turn, can increase the respective mole percentages of  $\text{SiO}_2$  and CaO

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materials, and it has already been chemically homogenized to the desired glass formulation before melting. In one embodiment, to melt the SLS glass precursor gel into molten glass that is suitable for subsequent glass forming operations, the gel needs only to be heated at a temperature of about 1450°C or greater for a time period of about 30 minutes to about 4 hours including all ranges and sub-ranges therebetween. Such a heating time is about one-sixth or less than the time it takes to satisfactorily melt conventional glass feedstocks. Longer heating times may of course be practiced, if desired, but they generally are not required. As an example of a longer heating time, the SLS glass precursor gel may be heated to 1200°C for about 16 hours to about 24 hours, which still produces a homogeneous, bubble-free melt yet consumes less energy than melting conventional soda-lime-silica glass feedstocks.

[0036] The SLS glass precursor gel can be melted in a glass furnace. For example, the glass furnace may be a refractory brick-lined vessel that is constructed with gas combustion and thermal radiation heat sources to heat the interior of the vessel. In use, the SLS glass precursor gel may be introduced into the furnace and on top of a glass bath from a side doghouse. The heat sources heat the SLS glass precursor gel from the top and/or bottom and the glass bath heats the floating gel from the bottom. The SLS glass precursor gel eventually melts and assimilates as molten glass into the glass bath. This occurs rather quickly for the SLS glass precursor gel, as compared to conventional soda-lime-silica glass feedstocks, because quartz sand dissolution, bubble removal, and homogenization of the molten glass derived from the SLS glass precursor gel has already been accomplished by low-temperature chemical reactions. As such, the molten glass derived from the SLS glass precursor gel does not have to stay in the furnace in a melted state for very long before it is ready for subsequent formation into a glass product, as indicated by the heat cycle times and temperatures recited above. For this reason, furnaces for melting the

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Claims

1.

A glass precursor gel comprising:

a bulk amorphous oxide-based matrix having an inorganic network of primary constituent oxides and water that is chemically entrained within the bulk amorphous oxide-based matrix, the primary constituent oxides comprising 60 mol% to 85% silica, 8 mol% to 18 mol% sodium oxide, and 5 mol% to 15 mol% calcium oxide; wherein the bulk amorphous oxide-based matrix is homogeneously chemically mixed and, further, wherein the glass precursor gel has a density of less than 2.0 g/cm<sup>3</sup>.

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2.

The glass precursor gel set forth in claim 1, wherein the amorphous oxide-based matrix further comprises aluminum oxide.

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3.

The glass precursor gel set forth in claim 1, wherein the amorphous oxide-based matrix further comprises at least one of glass network formers, coloring agents, redox agents, or combinations thereof.

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4.

The glass precursor gel set forth in claim 1, wherein the glass precursor gel has a density of 1.6 g/cm<sup>3</sup> to 1.85 g/cm<sup>3</sup> and a surface area of at least 20 m<sup>2</sup>/g as measured by nitrogen BET adsorption.

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5.

The glass precursor gel set forth in claim 1, wherein, upon heating, the glass precursor gel evolves oxygen gas between 450°C and 1500°C.

6.

A method of making a glass product, the method comprising:

obtaining a glass precursor gel as set forth in claim 1;

melting the glass precursor gel into molten glass; and

forming the molten glass into a glass product.

7.

The method set forth in claim 6, wherein the step of obtaining the glass precursor gel comprises:

providing a silicate solution that includes a dissolved alkali silicate, the dissolved alkali silicate having a molar ratio of an alkali metal oxide to silica;

adding an alkaline earth salt to the silicate solution to displace some of the alkali oxide with an alkaline earth metal oxide and to derive a wet precipitate having a molar ratio of the alkali metal oxide to the alkaline earth metal oxide to silica; and

removing liquid solvent from the wet precipitate to obtain the glass precursor gel.

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8.

The method set forth in claim 7, further comprising:

adjusting the molar ratio of the alkali oxide to silica in the dissolved alkali silicate by neutralizing some of the alkali oxide with an acid such that the molar ratio of the alkali oxide to silica is reduced.

9.

The method set forth in claim 6, wherein the molar ratio of the alkali oxide to the alkaline earth oxide to silica is between 0.8:0.8:6 and 1.4:1.3:6.

10.

The method set forth in claim 6, wherein the step of melting the glass precursor gel is performed at 1200°C to 1450°C for a period of 30 minutes to 16 hours.

11.

A method of making a glass product, the method comprising:

providing a silicate solution that includes a dissolved sodium silicate, the dissolved sodium silicate comprising a molar ratio of  $\text{Na}_2\text{O}:\text{SiO}_2$ ;

adding a soluble calcium salt to the silicate solution to displace some of the sodium oxide in the dissolved sodium silicate with calcium oxide and to derive a wet precipitate that comprises a molar ratio of  $\text{Na}_2\text{O}:\text{CaO}:\text{SiO}_2$ ;

removing solvent from the wet precipitate to obtain a soda-lime-silica glass precursor gel, the gel comprising a bulk amorphous oxide-based matrix having an inorganic

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network of oxides that comprises 60 mol% to 85 mol% silica, 8 mol% to 18 mol% sodium oxide, and 5 mol% to 15 mol% calcium oxide;

melting the soda-lime-silica glass precursor gel into molten glass; and  
forming the molten glass into a glass product.

12.

The method set forth in claim 11, wherein providing the silicate solution comprises dissolving quartz sand in a sodium-based solvent at a temperature between 25°C and 300°C and a pressure of 10 atmospheres to 100 atmospheres for 3 hours to 24 hours.

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13.

The method set forth in claim 11, further comprising:

adjusting the molar ratio of  $\text{Na}_2\text{O}:\text{SiO}_2$  in the dissolved sodium silicate by neutralizing some of the sodium oxide with an acid such that the molar ratio of  $\text{Na}_2\text{O}:\text{SiO}_2$  is  
reduced.

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14.

The method set forth in claim 13, wherein the step of adding a soluble calcium salt includes displacing sodium oxide in the dissolved sodium silicate with calcium oxide such  
that the molar ratio of  $\text{Na}_2\text{O}:\text{CaO}:\text{SiO}_2$  in the wet precipitate is between 0.8:0.8:6 and 1.4:1.3:6.

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15.

The method set forth in claim 11, wherein forming the molten glass into a glass product comprises:

thermally conditioning the molten glass at a temperature of about 1050°C to about 1200°C after the molten glass has been removed from the furnace;

delivering a gob of the molten glass to an individual section machine; and

forming the molten glass into a glass container in the individual section machine, the glass container having a glass body defining an interior accessible at a mouth located at one axial end of the glass body.

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16.

The method set forth in claim 11, wherein the soluble calcium salt includes at least one of calcium nitrate, calcium chloride, or calcium hydroxide.

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17.

The method set forth in claim 11, wherein the silicate solution is concentrated to at least 5 wt.% of the dissolved sodium silicate at the time the soluble calcium salt is added to the silicate solution.

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18.

The method set forth in claim 11, wherein the step of melting the soda-lime-silica glass precursor gel evolves oxygen gas between 450°C and 1500°C.

REPLACEMENT SHEET

19.

The method set forth in claim 11, wherein the step of melting the soda-lime-silica glass precursor gel does not release carbon dioxide gas.

20.

The method set forth in claim 11, wherein the step of melting the soda-lime-silica glass precursor gel evolves water vapor above 125°C.

## REPLACEMENT SHEET

2/3

| Component<br>(mol%)            | Soda-Lime-Silica<br>Glass | Borosilicate<br>Fiber (E-Glass) | Borosilicate Glass<br>(Flat Panel<br>Display) | Borosilicate Glass<br>(Low Thermal<br>Expansion) | Borosilicate Glass<br>(Fiber Wool) | Lead<br>Sealing<br>Glass |
|--------------------------------|---------------------------|---------------------------------|-----------------------------------------------|--------------------------------------------------|------------------------------------|--------------------------|
| SiO <sub>2</sub>               | 63-81                     | 43-74                           | 60-80                                         | 65-85                                            | 50-78                              | 30-67                    |
| Al <sub>2</sub> O <sub>3</sub> | 0-2                       | 6-10                            | 5-15                                          | 1-5                                              | 0-4                                | 0-7                      |
| B <sub>2</sub> O <sub>3</sub>  | -                         | 0-8.5                           | 5-15                                          | 8-15                                             | 2.5-9                              | 0-25                     |
| MgO                            | 0-6                       | 0.5-9                           | -                                             | -                                                | 1.5-8                              | 0-2                      |
| CaO                            | 7-14                      | 15-28                           | 2-10                                          | 0-2.5                                            | 5-15                               | 0-5                      |
| SrO                            | -                         | -                               | 0-5                                           | -                                                | -                                  | -                        |
| BaO                            | -                         | -                               | 0-3                                           | -                                                | -                                  | 4-10                     |
| Li <sub>2</sub> O              | 0-2                       | 0-1                             | -                                             | -                                                | -                                  | -                        |
| Na <sub>2</sub> O              | 9-15                      | 0-2.5                           | -                                             | 3-9                                              | 12-18                              | 2-10                     |
| K <sub>2</sub> O               | 0-1.5                     | 0-0.5                           | -                                             | -                                                | 0-1.5                              | 3-15                     |
| Fe <sub>2</sub> O <sub>3</sub> | 0-0.6                     | 0-0.3                           | -                                             | -                                                | 0-0.3                              | -                        |
| Cr <sub>2</sub> O <sub>3</sub> | 0-0.2                     | 0-0.2                           | -                                             | -                                                | -                                  | -                        |
| MnO <sub>2</sub>               | 0-0.2                     | -                               | -                                             | -                                                | -                                  | -                        |
| Co <sub>3</sub> O <sub>4</sub> | 0-0.1                     | -                               | -                                             | -                                                | -                                  | -                        |
| TiO <sub>2</sub>               | 0-0.8                     | 0-1                             | -                                             | -                                                | -                                  | -                        |
| SO <sub>3</sub>                | 0-0.2                     | -                               | -                                             | -                                                | 0-0.2                              | -                        |
| Se                             | 0-0.1                     | -                               | -                                             | -                                                | -                                  | -                        |
| F                              | 0-0.1                     | 0-2                             | -                                             | -                                                | 0-2.5                              | -                        |
| ZrO <sub>2</sub>               | -                         | -                               | -                                             | -                                                | -                                  | 0-3                      |
| PbO                            | -                         | -                               | -                                             | -                                                | -                                  | 15-80                    |

**FIG. 3**

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE  
PATENT COOPERATION TREATY**

International Application No.: PCT/US2016/024001  
Applicant: Owens-Brockway Glass Container Inc.  
International Filing Date: 24 March 2016  
Attorney Docket No.: 19318 WO  
In Response to: Search Report and Written Opinion  
Mailed 23 June 2016

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**RULE 34 AMENDMENT AND RESPONSE TO WRITTEN OPINION**

Mail Stop PCT  
Commissioner for Patents  
PO Box 1450  
Alexandria, VA 22313-1450

This is in response to the Search Report and Written Opinion mailed 23 June 2016 in connection with the subject application. The period for response expires twenty-two months from the 01 April 2015 priority date (*i.e.*, on 01 February 2017).

**I. Amendments to the Specification**

Please amend the subject PCT application by substituting enclosed new pages 2, 7, 10, 14, 16, 17, and 20 for original pages 2, 7, 10, 14, 16, 17, and 20 of the specification as-filed. New pages 2, 7, 10, 14, 16, 17, and 20 are being submitted to correct obvious typographical errors or other formalities. In particular, on page 2, the word "to" has been inserted at line 4 between the words "longest" and "disperse." On page 7, the chemical name "boric oxide" at line 2 has been replaced with the chemical name "boron oxide" as lead sealing glass may include anywhere from 4 mol% to 10 mol% of BaO (and not necessarily B<sub>2</sub>O<sub>3</sub>) as shown in FIG. 3 of the as-filed specification. On page 7, the

misspelling of the metal element "molybdenum" at line 11 has been corrected. On page 10, the spelling of the word "temperatures" at line 20 has been corrected to "temperature." On pages 14 and 17, the word "the" has been added at lines 4 and 2, respectively, between the words "has" and "same." On page 16, the word "material" at line 6 has been replaced with the word "mineral" for consistency with the surrounding text. And finally, on page 20, the misspelling of the term "brick-line" at line 12 has been corrected to "brick-lined." The Applicant submits that the above-noted amendments to the specification does not introduce any new matter into the application.

## II. Amendments to the Drawings

Please amend the subject PCT application by substituting enclosed new drawing sheet 2 for original drawing sheet 2 of the as-filed application. New drawing sheet 2 corrects a typographical error in the recited mol% of Na<sub>2</sub>O in the "Soda-Lime-Silica Glass" column of FIG. 3. As can be seen in FIG. 3, the mol% of each of the components listed is provided as a range bound by the minimum mol% and the maximum mol%. The Na<sub>2</sub>O listing for "Soda-Lime-Silica Glass" in originally-filed FIG. 3 mistakenly recites a maximum of "1.5" mol%, which is less than the recited minimum of "9" mol%. Clearly, as supported in paragraph 0015 (on page 6) of the as-filed specification, the recited range for Na<sub>2</sub>O should read "9–15" mol%, not "9–1.5" mol%. New drawing sheet 2 makes this correction.

## III. Amendments to the Claims

Please amend the subject PCT application by substituting enclosed new pages 30–35, which contain new claims 1–20, for original pages 30–36 containing original claims 1–23 of the as-filed application.

Claim 1 corresponds to original claim 1 of the as-filed application and has been amended to incorporate the subject matter of original dependent claims 2–3 and 8. Accordingly, each of dependent claims 2–3 and 8 has been canceled. Further support for the amendments to claim 1 can be found in paragraphs 0015, 0019, and 0025 of the as-filed application.

Claims 2–5 correspond to claims 4–7 of the as-filed application and are unchanged apart from being renumbered due to the cancellation of dependent claims 2–3.

Claim 6 corresponds to claim 9 of the as-filed application and has been amended to state that the glass precursor gel “as” set forth in claim 1 is obtained. The phrases “that comprises a bulk amorphous oxide base matrix” and “in a homogeneously chemically mixed form” have been deleted since those limitations are already present in claim 1.

Claims 7–20 correspond to claims 10–23 of the as-filed application and are unchanged apart from being renumbered due to the cancellation of dependent claims 2–3 and 8.

#### IV. Response to the Written Opinion of the International Search Authority

The present amendments render each of claims 1–20 patentable over the prior art applied in the Written Opinion. Referring now specifically to claim 1, the recited glass precursor gel includes (1) a bulk amorphous oxide-based matrix having an inorganic network of primary constituent oxides and (2) water that is chemically entrained within the bulk amorphous oxide-based matrix. The bulk amorphous oxide-based matrix is homogeneously chemically mixed and the primary constituent oxides included therein comprise 60 mol% to 85% silica ( $\text{SiO}_2$ ), 8 mol% to 18 mol% sodium oxide ( $\text{Na}_2\text{O}$ ), and 5

mol% to 15 mol% calcium oxide (CaO). Moreover, the glass precursor gel has a density of less than  $2.0 \text{ g/cm}^3$ , which is less dense than cullet.

The glass precursor gel set forth in claim 1 has been developed as an alternative to traditional glass-making procedures in which virgin raw materials along with optional cullet are melted, homogenized, and refined in a glass furnace at relatively high temperatures for long periods of time. This traditional process typically involves introducing the virgin raw materials (many of which are crystalline) and optional cullet onto a molten glass bath within a glass furnace. The added materials are melted and dissolved into the glass bath—with quartz sand taking the longest to melt—and are kept inside the furnace for a residence time of 24 hours or more to ensure the melted materials become homogeneously mixed while allowing enough time for carbon dioxide produced from the melting of carbonate-containing raw materials to be removed. The melting of the virgin raw materials and optional cullet and the homogenizing and refining of the molten glass is a long process that requires a substantial energy commitment.

The claimed glass precursor gel is synthesized into a chemically homogeneous form at relatively low temperatures of, for example, below  $300^\circ\text{C}$ , outside of a glass furnace before being melted. See Applicant's As-Filed Specification at paragraph 0022. In other words, "the three rate-limiting steps of conventional glass making—dissolution of quartz sand, bubble removal, and homogenization/mixing of the primary constituent oxides—are accomplished at low temperatures by way of chemical reaction, not the standard procedure in which crystalline raw materials (and optionally come cullet) are physically mixed, melted, and maintained in a molten state to facilitate dissolution." *Id.*

And because the glass precursor gel is chemically homogeneously mixed and carbonate free upon being synthesized, it can be rapidly melted into a chemically homogenized and

virtually bubble free molten glass composition during the production of soda-lime-silica glass products such as, for example, glass containers, as explained in the as-filed specification:

The composition of the glass precursor gel facilitates rapid melting into molten glass while avoiding long residence times in a glass furnace. The glass precursor gel does not include large quantities of crystalline materials and, most notably, the amorphous oxide-based matrix does not contain any crystalline precursor materials of silica such as, for example, quartz sand. The absence of quartz sand is noteworthy here. Unlike conventional glass feedstocks that contain a fair amount of quartz sand, which generally has a slow dissolution rate, the glass precursor gel does not have to be maintained in a molten state at high temperatures for long durations of time in order to achieve satisfactory  $\text{SiO}_2$  dissolution. The amorphous oxide-based matrix also includes virtually no carbonate-containing materials and, as such, will produce no more than a negligible amount of  $\text{CO}_2$  upon melting and hence no more than a negligible amount of bubbles within the molten glass. The molten glass obtained from the glass precursor gel therefore does not have to be refined by removing substantial amounts of entrained bubbles through extended heating times. Given that the amorphous oxide-based matrix is already homogeneously chemically mixed and does not contain crystalline precursor materials of the primary constituent oxides or more than an insignificant amount of carbonate-containing materials, the glass precursor gel does not have to be heated to as high of a temperature or maintained at an elevated temperature for as long of a timeframe as conventional glass feedstocks in order to obtain a molten glass that is suitable for downstream processing into a glass product.

*Id.* at paragraph 0020 (emphasis added).

The technical effect attributed to the Applicant's claimed glass precursor gel is thus related to the fact that the gel is chemically synthesized at low temperatures prior to being melted, as opposed to conventional glass making practices in which melting of the raw materials and optional cullet plus homogenizing and refining of the glass melt are all carried out at high temperatures over prolonged periods of time in a glass furnace. In this regard, when used during the production of a soda-lime-silica glass product, the glass precursor gel can be rapidly melted into a chemically homogenized, bubble free molten

glass, which can lower the furnace energy consumption, reduce the infrastructure and furnace footprint of each glass product produced, and even allow for smaller furnace designs since the molten glass derived from the glass precursor gel does not have to remain in the furnace for very long before it is ready for downstream production. *Id.* at paragraphs 0022 and 0036. The claimed glass precursor gel also has the tendency to produce glass with good clarity due to its ability to suppress  $\text{Fe}^{2+}$  absorption. *Id.* at paragraph 0038.

None of the D1 to D5 references cited in the Written Opinion discloses a glass precursor gel that includes the bulk amorphous oxide-based matrix having an inorganic network of primary constituent oxides that comprises  $\text{SiO}_2$ ,  $\text{Na}_2\text{O}$ , and  $\text{CaO}$  in their respective mole percentages, along with water that is chemically entrained within the bulk amorphous oxide-based matrix. Nor do any of the D1 to D5 references appreciate the technical effect associated with the Applicant's claimed chemically-synthesized glass precursor gel and its ability to be rapidly melted into a molten glass suitable for glass manufacturing at substantial energy savings compared to the heavy up-front energy investment required to melt crystalline feed materials and thereafter homogenize and refine the molten glass in a furnace according to conventional melt processing techniques for soda-lime-silica glass products. For at least those reasons, which are described in more detail below, the Applicant contends that each of claims 1–20 is novel and possesses an inventive step over the references cited and applied in the Written Opinion.

The primary D1 reference (Kaşgöz et al.) cited in the Written Opinion as teaching the glass precursor gel of claim 1 is no longer applicable. That reference teaches borosilicate gel plates (the Applicant is assuming the gel plates are being equated to the claimed glass precursor gel) derived by aging a concentrated polymer solution of a

polyborosiloxane. D1 at 168–69. The borosilicate gel plates do not include a bulk amorphous oxide-based matrix having an inorganic network of primary constituent oxides that comprises 60 mol% to 85% silica, 8 mol% to 18 mol% sodium oxide, and 5 mol% to 15 mol% calcium oxide. In fact, the  $\text{SiO}_2\text{-B}_2\text{O}_3$  oxide network of the borosilicate gel plates is not even “inorganic” since, according to Kaşgöz, the polyborosiloxane in the polymer solution that produces the borosilicate gel plates “is composed of Si-O-Si and Si-O-B bonds with hydroxy(trimethylsilyl) and butoxy groups as pendants.” *Id.* at 170. And there is no articulation in the Written Opinion that supports the proposition that the borosilicate gels “can implicitly be assumed [to] have a density of less than 2.0 g/cm<sup>3</sup>” simply because they are formed by the reaction sequence described in D1.

The D2 and D3 references to Hench et al. also fail to disclose a glass precursor gel as set forth in claim 1. D2 is directed to using ketomalonic acid during a sol-gel process in order to introduce copper ions into a silica-containing sol that is subsequently processed into a  $\text{SiO}_2$  containing monolith. D2 at col.1 l.56 – col.2 l.4. The silica containing sol may include  $\text{SiO}_2$  alone or in combination with one or more of  $\text{B}_2\text{O}_3$ ,  $\text{Na}_2\text{O}$ ,  $\text{Li}_2\text{O}$ ,  $\text{TiO}_2$ , and  $\text{Al}_2\text{O}_3$ . *Id.* at col.2 ll.22–26. The described  $\text{SiO}_2$ -containing gels thus do not fall within the scope of claim 1. Additionally, the  $\text{SiO}_2$ -containing gels are described in the context of  $\text{SiO}_2$  monolith formation, which does not include melting of the gel into a molten glass, but, rather, involves casting the solution into a mold, gelling the solution, aging and drying the gel, and then densifying or firing the dried gel over the course of one to three days. *Id.* at col.2 ll.10–14 and ll.44–61.

The D3 reference is directed to using drying control chemical additives (DCCA) during the mixing phase of a sol-gel process of silicon-containing monoliths that are used to make dense ceramic shapes. D3 at col.1 ll.13–19 and col.1 l.67 – col.2. l.2.

The DCCA that is added to solution may be glycerol, formamide, oxalic acid, and other acids having the formula  $C_nH_{2(n-1)}O_{2n}$ , *id.* at col.2 ll.22–30, and its presence helps facilitate quick drying, aging, and densifying of the gel. *Id.* at col.1 ll.13–19 and col.2 ll.57–64. As relevant here, the gels that are formed during the sol-gel process may include  $SiO_2-Na_2O$ ,  $SiO_2-LiO_2$ , and  $SiO_2-B_2O_3-Na_2O$ . *Id.* at col.5 ll.4–9, col.7 ll.58–59, and col.8 ll.60–64. The described gels thus do not fall within the scope of claim 1. Moreover, as before with the D2 reference, the gels taught by D3 are described in the context of silicon-containing monolith formation, which does not include melting the gel into a molten glass, but, rather, involves casting the solution into a mold, gelling the solution, aging and drying the gel, and then densifying or firing the aged dried gel. *Id.* at col.2 ll.57–61.

The D4 and D5 references have been cited in the Written Opinion because when combined with the D1 to D3 references they allegedly teach a method of making a glass product by providing a glass precursor gel, melting the glass precursor gel into a molten glass, and obtaining a glass product from the molten glass. Thus, according to the Written Opinion, a person skilled in the art would have applied the same process to the gels taught in the D1 to D3 references to solve the technical problems addressed by the present application and to ultimately arrive at the Applicant's recited method claims (i.e., claims 6–20). But, as already discussed above, none of the D1 to D3 references discloses a glass precursor gel that includes an inorganic network of primary constituent oxides that comprises  $SiO_2$ ,  $Na_2O$ , and  $CaO$ —let alone in the specified proportions of those three oxides as set forth in claim 1. Nor do the D1 to D5 references attempt to solve the technical problem addressed by the present application relating to the time-consuming and energy intensive process of creating homogenized and refined molten glass of the soda-lime-silica variety through conventional melt processing. None of the D1 to D3

references even wishes to melt its disclosed gel as part of forming a glass product, so the D4 and D5 references are not particularly relevant to the D1 to D3 references on that issue.

The D4 and D5 references, when viewed as a whole, also would not have prompted a skilled person to apply their particularized teachings to a glass precursor gel that includes an inorganic network of primary constituent oxides that comprises 60 mol% to 85 mol% SiO<sub>2</sub>, 8 mol% to 18 mol% Na<sub>2</sub>O, and 5 mol% to 15 mol% CaO. The D4 reference is directed to glass composition that includes SiO<sub>2</sub>, B<sub>2</sub>O<sub>3</sub>, PbO, and optionally Al<sub>2</sub>O<sub>3</sub> or ZnO or a mixture of Al<sub>2</sub>O<sub>3</sub> and ZnO, with a significantly lower SiO<sub>2</sub> component (i.e., less than 30 wt% SiO<sub>2</sub>) than what is called for in claims 6 and 11. The D4 reference is also specifically concerned with removing unwanted black coloration from the specific disclosed glass composition when a inorganic, oxidic precursor having the same oxide content is heated. D4 at col.2 ll.1–9 and ll.48–59. The D5 reference is directed to forming a silica gel from a high-purity synthetic quartz powder prepared by a sol-gel reaction. D5 at paragraph 0009. The silica gel taught by D5 does not include an an inorganic network of primary constituent oxides; rather, it includes only SiO<sub>2</sub>. The glass compositions of D4 and D5 and the technical challenges solved by those references are clearly distinct from the Applicant's claimed methods and supporting disclosure.

Still further, neither the D4 nor the D5 reference teaches a process in which a chemically homogenized gel is synthesized and then rapidly melted to produce molten glass in an effort to circumvent the conventional process of melting a batch of virgin raw materials (and optionally cullet) and then homogenizing and refining the glass melt, which is an prolonged and energy-intensive process. A skilled person would not immediately assume or understand that the teachings of D4 and D5 could be applied to soda-lime-silica glass precursor gels. Indeed, the D1 reference is relatively vague on how the borosilicate

gel plates can be further processed, and both of the D2 and D3 references age, dry, and then densify their gel products in a mold instead of melting and further processing them into a glass product. D2 at col.2 ll.10–14; D3 at col.2 ll.57–61. The notion that the problem solved by the present application as stated in the Written Opinion is “to develop a method for obtaining a glass precursor gel and a glass product comprising the bulk amorphous oxide-based matrix” as set forth in the Applicant's claims is an oversimplification that fails to appreciate the intricacies of glass-making and the different approaches described in the present application and the D1 to D5 references to solve problems affecting their different glass chemistries.

Apart from the arguments presented above, the Applicant further submits that independent claim 11 is novel and possess inventive step over D1 to D5 references for a separate reason as well. In particular, claim 11 sets forth a method of making a glass product. The claimed method involves forming a glass precursor gel in which a silicate solution comprising a dissolved sodium silicate is provided followed by adding a soluble calcium salt to the silicate solution to displace some of the sodium oxide in the dissolved sodium silicate and to thereby derive a wet precipitate having a molar ratio of  $\text{Na}_2\text{O}:\text{CaO}:\text{SiO}_2$ . Solvent is then removed from the wet precipitate to obtain a soda-lime-silica glass precursor gel that includes a bulk amorphous oxide-based matrix having an inorganic network of oxides that comprises 60 mol% to 85 mol%  $\text{SiO}_2$ , 8 mol% to 18 mol%  $\text{Na}_2\text{O}$ , and 5 mol% to 15 mol%  $\text{CaO}$ . The soda-lime-silica glass precursor gel is then melted and formed into a glass product.

None of the D1 to D5 references disclose the totality of the method recited in claim 11 including the use of dissolved sodium silicate as a precursor to  $\text{SiO}_2$  in the ultimately-prepared soda-lime-silica glass precursor gel. In the D1 reference, silicic acid is

reacted with boron tri-*n*-butoxide under reflux of tetrahydrofuran followed by alkoxylation with 1-butanol to produced highly polymerized polyborosiloxanes. D1 at 168–69. Furthermore, in each of the D2 to D5 references, an alkoxysilane that can undergo hydrolysis and polycondensation is used as precursor to SiO<sub>2</sub>. D2 at col.2 ll.14–22 (tetramethoxysilane); D3 at col.2 l.65 – col.3 l.9 (silicon tetraalkoxides having the genral formula Si(OR)<sub>4</sub> where R is an alkoxide group); D4 at col.5 ll.36–44 (a tetra-alkoxy silane or silcon alkoxide having the formula Si(OR)<sub>4</sub>); D5 at paragraph 0026 (an alkoxysilane). As such, none of the D1 to D5 references, when considered individually or in combination, teaches or otherwise suggests the method of claim 11. These same arguments also apply to dependent claims 7–10 for similar reasons.

It therefore is believed and respectfully submitted that all claims meet the novelty, inventive step, and industrial applicability requirements of the PCT, as well as all form and procedural requirements raised in the Written Opinion.

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SBW/MJD/dim

## HOJA DE REEMPLAZO

que incluyen fases cristalinas intermedias, presentan generalmente hasta aproximadamente 1200 °C, ya que la fundición y disolución de estos materiales no ocurre instantáneamente. En segundo lugar, la materia prima del vidrio necesita ser dispersada y mezclada homogéneamente mediante convección después de ser fundida para producir vidrio fundido, que es un proceso que consume tiempo. La arena de cuarzo, en particular, toma mucho tiempo para dispersarse debido a su lenta velocidad de disolución y la tendencia a aglomerarse en regiones ricas en SiO<sub>2</sub> dentro del fundido de vidrio conocido como “cordón”. La presencia del cordón es indicadora de falta de homogeneidad del vidrio y puede resultar en imperfecciones o defectos en el producto de vidrio terminado. Tercero, algo de los ingredientes de materia prima virgen, por ejemplo, sosa y cal para vidrio de sílice-cal-sosa, son materiales que contienen carbonato que, cuando se funde, libera dióxido de carbono (CO<sub>2</sub>). La evolución del dióxido de carbono durante la fundición de la materia prima introduce burbujas en el vidrio fundido resultante, que, a su vez, puede provocar un defecto de burbuja o punto delgado en el producto de vidrio terminado. Cualquiera de dichas burbujas se retira normalmente del vidrio fundido en un proceso conocido como “refinamiento del vidrio”. Para superar los retos asociados con la fundición y homogeneización de materias primas cristalinas y retirar las burbujas provocadas por la evolución del dióxido de carbono, entre otros factores, las materias primas de vidrio convencionales usualmente se someten a altas temperaturas y tiempos de calentamiento de 24 horas o más en el horno de vidrio con el fin de obtener vidrio fundido homogeneizado químicamente y refinado adecuadamente.

[0004] La fundición de la materia prima de vidrio se puede hacer menos gravosa si algunas de las materias primas vírgenes se reemplazan con casco en la materia prima. El casco acelera la fundición de la materia prima y reduce el consumo de energía del horno en comparación con una materia prima que contiene solo materia

prima virgen. El casco tiene este efecto porque ya ha sido fundido, mezclado, y formado en un producto de vidrio y no liberara dióxido de carbono cuando se vuelve a fundir en razón a que no es un material intrínseco que contenga carbonato. Pero el casco no solo está disponible ampliamente como un producto básico en

3% de moles a 15% de moles de óxido de potasio y 2% de moles a 10% de moles de óxido de sodio 4% de moles a 10% de moles de óxido de bario, como los óxidos constituyentes primarios.

5 [0016] Además de los óxidos constituyentes primarios, la matriz basada en óxido amorfo puede incluir opcionalmente una serie de materiales secundarios que se utilizan comúnmente en la industria de fabricación del vidrio. Ejemplos de dichos materiales secundarios son hasta 25% de moles total de otros formadores de red de vidrio, hasta 2% de moles de total de agentes colorantes y decolorantes y hasta  
10 20% de moles total de otros materiales que pueden afectar las propiedades físicas y/o el estado redox de la composición del producto de vidrio final. Otros formadores red de vidrio específicos que se pueden agregar incluyen uno o más de óxido fosforoso ( $P_2O_5$ ) y óxido de germanio ( $GeO_2$ ). Los colorantes y decolorantes específicos que se pueden agregar incluyen formas elementales o formas de  
15 compuestos de óxido de uno o más de selenio, cromo, manganeso, hierro, cobalto, níquel, cobre, niobio, molibdeno, plata, cadmio, indio, estaño, oro, cerio, praseodimio, neodimio, europio, gadolinio, erbio, y uranio. Y materiales específicos que pueden afectar las propiedades físicas y/o el estado redox del vidrio incluyen uno o más de carbono (0% de moles a 3% de moles), nitratos (0% de moles a 3%  
20 de moles), selenio (0% de moles a 1% de moles), óxido de titanio ( $TiO_2$ ) (0% de moles a 5% de moles), óxido de arsénico ( $As_2O_3$ ) (0% de moles a 2% de moles), óxido de vanadio ( $V_2O_5$ ) (0% de moles a 5% de moles) , flúor (0% de moles a 2% de moles) cloro (0% de moles a 2% de moles) y sulfatos (0% de moles a 2% de moles).

25

[0017] La composición exacta de la matriz basada en óxido amorfo incluye las proporciones de sus óxidos constituyentes primarios y materiales secundarios opcionales que se pueden variar para alcanzar cualquiera de la gran variedad de químicas de vidrio que se pueden desear en el producto de vidrio final. Las

propiedades físicas y químicas del vidrio resultante derivado del gel precursor de vidrio se pueden afectar mediante varianzas en las proporciones relativas de los óxidos constituyentes primarios, así como la inclusión o exclusión de determinados materiales secundarios en la matriz basada en óxido amorfo.

entre ellos, y un área de superficie de por lo menos 20 m<sup>2</sup>/g, preferiblemente aproximadamente 25 m<sup>2</sup>/g a aproximadamente 40 m<sup>2</sup>/g que incluye todos los rangos y subrangos entre ellos, según se mide por la adsorción BET de nitrógeno. El gel precursor de vidrio por lo tanto es de esta manera menos denso y tiene una mayor  
5 área de superficie que el casco.

[0020] La composición del gel precursor de vidrio facilita la fundición rápida en vidrio fundido mientras que evita largos tiempos de residencia en el horno de vidrio. El gel precursor de vidrio no incluye grandes cantidades de materiales cristalinos y, más  
10 notablemente, la matriz basada en óxido amorfo no contiene ningún material precursor cristalino de sílice tal como, por ejemplo, arena de cuarzo. La ausencia de arena de cuarzo es notable aquí. Por el contrario, la materia prima de vidrio convencional que contienen una cantidad justa de arena de cuarzo, que generalmente tiene una velocidad de disolución lenta, el gel precursor de vidrio no  
15 se tiene que mantener en un estado fundido a altas temperaturas durante mucho tiempo con el fin de alcanzar disolución satisfactoria del SiO<sub>2</sub>. La matriz basada en óxido amorfo también incluye materiales que virtualmente no contienen carbonato y, como tal, producirán no más de una cantidad insignificante de CO<sub>2</sub> luego de fundición y por lo tanto, no más de una cantidad insignificante de burbujas dentro  
20 del vidrio fundido. Por lo tanto el vidrio fundido obtenido a partir del gel precursor de vidrio no tiene que ser refinado al retirar cantidades sustanciales de burbujas atrapadas a través de tiempos de calentamiento extendidos. Dado que la matriz basada en óxido amorfo ya se mezcla químicamente homogéneamente y no contiene materiales precursores cristalinos de los óxidos constituyentes primarios o  
25 más de una cantidad insignificante de materiales que contiene carbonato, el gel precursor de vidrio no se tiene que calentar a una temperatura tan alta o mantenerse a una temperatura elevada durante un tiempo largo como las materias primas de vidrio convencionales con el fin de obtener un vidrio fundido que sea adecuado para el procesamiento posterior de un producto de vidrio.

[0021] Para asegurarse, como se mencionó anteriormente, normalmente se funde materias primas de vidrio de sílice-cal-sosa convencional en un horno y se mantienen a 1400 °C o más durante por lo menos aproximadamente 24 horas con

[0026] El gel precursor de vidrio SLS se puede obtenerse en la etapa 12 en numerosas formas. En una realización, como se describe aquí en la figura 1, el gel precursor de vidrio SLS es obtenido al (1) proporcionar una solución de silicato que incluye un silicato de sodio disuelto (etapa 12a); (2) agregar una sal de calcio soluble a la solución de silicato para derivar un precipitado húmedo que tiene las mismas proporciones de los óxidos constituyentes primarios como la matriz basada en óxido amorfo del gel precursor de vidrio SLS (etapa 12b); y (3) derivar el gel precursor de vidrio SLS del precipitado húmedo (etapa 12c). Se describirá adelante una implementación específica de las etapas 12a-12c en más detalle. Cabe entender, sin embargo, que el gel precursor de vidrio SLS también se puede obtener a través de otros mecanismos no descritos expresamente, pero no obstante conocido por expertos, que incluyen formas alternas de proporcionar la solución de silicato en la etapa 12a tal como al comprar la solución de silicato o preparar la solución químicamente al fundir una mezcla de arena de cuarzo y sosa y luego disolver el producto resultante en agua.

[0027] La solución de silicato se puede proporcionar en la etapa 12a al disolver hidrotérmicamente arena de cuarzo en un disolvente basado en sodio acuoso cáustico. El disolvente basado en sodio acuoso cáustico puede ser una base de sodio fuerte, tal como hidróxido de sodio (NaOH), que está preferiblemente concentrado a más del 10% en peso (de la base de sodio) con el fin de hacer la disolución de la arena de cuarzo más fácil. Un ejemplo de una base de sodio fuerte adecuada es 18% en peso de NaOH. La disolución hidrotérmica de arena de cuarzo se puede lograr en un recipiente a presión a una presión por encima de la presión atmosférica. Aquí, la arena de cuarzo se puede disolver en el disolvente basado en sodio acuoso cáustico a una temperatura entre aproximadamente unos 25 °C y aproximadamente 300 °C incluyendo todos los rangos y sub rangos entre ellos, preferiblemente entre aproximadamente 200 °C y aproximadamente 300 °C, y una presión de aproximadamente 10 atmósferas hasta aproximadamente 100

atmósferas incluyendo todos los rangos y sub rangos entre ellos, preferiblemente aproximadamente 30 hasta aproximadamente 50 atmósferas, sobre el curso de aproximadamente 3 horas hasta aproximadamente 24 horas incluyendo todos los rangos y sub rangos entre ellos. La solución de silicato formada bajo estas

5 condiciones hidrotérmicas contiene una fase de sólidos disuelta de silicato de sodio. El silicato de sodio disuelto producido tiene la fórmula química general  $\text{Na}_2\text{O} \cdot x\text{SiO}_2$  con x que varía de 1,5 a 3,75 incluyendo todos los rangos y sub rangos entre ellos.

- óxido, óxido de aluminio, óxido de magnesio, óxido de potasio, óxido de hierro, óxido de titanio, óxido de circonio, óxido de bario, óxido de estroncio ( $\text{SrO}$ ), trióxido de azufre ( $\text{SO}_3$ ) y óxidos de selenio, cobalto, cromo, manganeso y plomo. Óxidos de aluminio, en particular, que mejoran la durabilidad química del vidrio de sílice-cal-
- 5 sosa y reducen la tendencia de la desvitrificación en el vidrio fundido, se pueden introducir al agregar un mineral que contiene alúmina al disolvente basado en sodio acuoso cáustico junto con arena de cuarzo bajo condiciones hidrotérmicas. Agregar material que tiene alúmina en este momento resulta en un óxido de aluminio que se integra químicamente en el silicato de sodio disuelto durante disolución hidrotérmica
- 10 de la arena de cuarzo. Algunos ejemplos de minerales que tienen alúmina que se pueden utilizar aquí son sienita nefileno, aplita, y escoria de calumita. El óxido de aluminio también se puede introducir en la solución de silicato en la forma de aluminato de sodio disuelto.
- 15 [0030] La lista de materiales secundarios que se pueden introducir en la solución de silicato durante la etapa 12a es más extensa que aquella recién mencionada. Se pueden agregar colorantes y decolorantes tal como uno o más de óxidos de hierro (por ejemplo,  $\text{FeO}$  y  $\text{Fe}_2\text{O}_3$ ), óxidos de cromo ( $\text{CrO}$  o  $\text{Cr}_2\text{O}_3$ ), óxidos de cobalto ( $\text{CoO}$  o  $\text{Co}_2\text{O}_3$ ), níquel, cobre, selenio, dióxido de manganeso, óxido de cerio, titanio y
- 20 combinaciones de azufre, hierro y carbón. Otra clase de materiales secundarios que se pueden agregar a la solución de silicato son agentes de refinación, tal como, por ejemplo, la combinación de sulfato de sodio ( $\text{Na}_2\text{SO}_4$ ), carbono, óxido de arsénico y óxido de antimonio. Todavía adicionalmente, se pueden agregar oxidantes o reductores a la solución de silicato para modificar, si lo desea, el número redox del
- 25 vidrio fundido derivado del gel precursor de vidrio SLS. Ejemplos de reductores y oxidantes comunes incluyen sulfato de calcio ( $\text{CaSO}_4$ ), nitrato de sodio ( $\text{NaNO}_3$ ), nitrato de potasio ( $\text{KNO}_3$ ), pirita de hierro ( $\text{FeS}_2$ ) y grafito.

[0031] Después que se ha formulado la solución de silicato en la etapa 12a, se puede agregar sal de calcio soluble a la solución de silicato en la etapa 12b para derivar un precipitado húmedo que tiene las mismas proporciones de los óxidos constituyentes primarios (por ejemplo, sílice, sodio y calcio) deseados en la matriz basada en óxido amorfo del gel precursor de vidrio SLS. Esta etapa implica agregar la sal de calcio soluble a la solución de silicato en una cantidad que lleva la relación molar de  $\text{Na}_2\text{O}:\text{CaO}:\text{SiO}_2$  en el precipitado húmedo a aproximadamente 1:1:6, que es la relación molar deseada de la matriz basada en óxido amorfo y, a su vez, el vidrio de sílice-cal-sosa producido a partir del gel precursor de vidrio SLS. La sal de calcio soluble puede incluir, por ejemplo, nitrato de calcio ( $\text{Ca}(\text{NO}_3)_2$ ), cloruro de calcio ( $\text{CaCl}_2$ ), hidróxido de calcio ( $\text{Ca}(\text{OH})_2$ ) o cualquier otra sal de calcio soluble o combinación de sales solubles que proporciona una fuente de iones de calcio. Cuando se agrega a la solución de silicato, los iones de calcio proporcionados por la sal de calcio soluble desplazan el sodio en el silicato de sodio disuelto, introduciendo de esta manera óxido de calcio en el silicato, lo que provoca que el silicato de sodio recientemente modificado se precipite fuera de la solución como precipitado húmedo. Y en razón a que cada 1 mol de iones de calcio (que resultan en un mol correspondiente de  $\text{CaO}$ ) desplazan 1 mol de  $\text{Na}_2\text{O}$  en el silicato de sodio disuelto, la cantidad de sal de calcio soluble que se necesita agregar a la solución de silicato para proporcionar el precipitado húmedo con la relación molar 1:1:6 de  $\text{Na}_2\text{O}:\text{CaO}:\text{SiO}_2$  que se puede calcular fácilmente basado en la relación molar de  $\text{Na}_2\text{O}:\text{SiO}_2$  en la solución de silicato de la etapa 12a.

[0032] El desplazamiento de  $\text{Na}_2\text{O}$  con  $\text{CaO}$ , y la precipitación del precipitado húmedo, se lleva a cabo preferiblemente con la solución de silicato que es tan concentrada como sea posible. Se ha encontrado, de hecho, que como la solución de silicato de sodio disuelto se hace más diluida en la solución de silicato, la cantidad de  $\text{Na}_2\text{O}$  en el precipitado húmedo se reduce mientras que la cantidad de  $\text{SiO}_2$  y

CaO no se afectan similarmente. Esto, a su vez, puede aumentar los porcentajes de mol respectivos de  $\text{SiO}_2$  y CaO en

y ya se ha homogeneizado químicamente a la formulación vítrea deseada antes de fundición. En una realización, para fundir el gel precursor de vidrio SLS en vidrio fundido que es adecuado para operaciones de formación de vidrio posteriores, el gel solo necesita ser calentado a una temperatura de aproximadamente 1450 °C o más durante un periodo de tiempo de aproximadamente 30 minutos a aproximadamente 4 horas incluyendo todos los rangos y subrangos entre ellos. Dicho tiempo de calentamiento es de aproximadamente un sexto o menos del tiempo que toma fundirse satisfactoriamente las materias primas de vidrio convencional. Por supuesto se pueden practicar temperaturas de calentamiento mayores, si se desea, pero generalmente no se requieren. Como un ejemplo de un mayor tiempo de calentamiento, el gel precursor de vidrio SLS se puede calentar a 1200 °C durante aproximadamente 16 horas hasta aproximadamente 24 horas, lo que aún produce un fundido homogéneo, libre de burbujas que consume menos energía que fundir las materias primas de vidrio de sílice-cal-sosa convencionales.

[0036] El gel precursor de vidrio SLS se puede fundir en un horno de vidrio. Por ejemplo, el horno de vidrio puede ser un recipiente de línea de ladrillos refractarios que se construye con fuentes de calor de radiación térmica y de combustión de gas para calentar el interior del recipiente. En uso, el gel precursor de vidrio SLS se puede introducir en un horno y en la parte superior de un baño de vidrio de una boca de carga lateral. Las fuentes de calor calientan el gel precursor de vidrio SLS desde la parte superior y/o inferior y el baño de vidrio calienta el gel flotante desde el fondo. El gel precursor de vidrio SLS eventualmente se funde y se asimila como vidrio fundido en el baño de vidrio. Esto ocurre más bien rápidamente para el gel precursor de vidrio SLS, en comparación con las materias primas de vidrio de sílice-cal-sosa convencional, debido a que la distribución de arena de cuarzo, el retiro de burbujas y la homogeneización del vidrio fundido derivado del gel precursor de vidrio SLS ya se ha logrado mediante reacciones químicas de baja temperatura. Como tal, el vidrio fundido derivado del gel precursor de vidrio SLS no tiene que permanecer en el

horno en un estado fundido durante mucho tiempo antes que esté listo para formación posterior en un producto de vidrio, como se indica por los tiempos de ciclos de calor y temperaturas mencionados anteriormente. Por esta razón, los hornos para fundir el gel

| Componente<br>(% de moles)     | Vidrio de sílice-cal-sosa | Fibra de borosilicato (Vidrio E) | Vidrio de Borosilicato (visualiza panel plano) | Vidrio de borosilicato (baja expansión térmica) | Vidrio de borosilicato (lana de vidrio) | Vidrio de sellado de plomo |
|--------------------------------|---------------------------|----------------------------------|------------------------------------------------|-------------------------------------------------|-----------------------------------------|----------------------------|
| SiO <sub>2</sub>               | 63-81                     | 43-74                            | 60-80                                          | 65-85                                           | 50-78                                   | 30-67                      |
| Al <sub>2</sub> O <sub>3</sub> | 0-2                       | 6-10                             | 5-15                                           | 1-5                                             | 0-4                                     | 0-7                        |
| B <sub>2</sub> O <sub>3</sub>  | -                         | 0-8,5                            | 5-15                                           | 8-15                                            | 2,5-9                                   | 0-25                       |
| MgO                            | 0-6                       | 0,5-9                            | -                                              | -                                               | 1,5-8                                   | 0-2                        |
| CaO                            | 7-14                      | 15-28                            | 2-10                                           | 0-2,5                                           | 5-15                                    | 0-5                        |
| SrO                            | -                         | -                                | 0-5                                            | -                                               | -                                       | -                          |
| BaO                            | -                         | -                                | 0-3                                            | -                                               | -                                       | 4-10                       |
| Li <sub>2</sub> O              | 0-2                       | -1                               | -                                              | -                                               | -                                       | -                          |
| Na <sub>2</sub> O              | 9-15                      | 0-2,5                            | -                                              | 3-9                                             | 12-18                                   | 2-10                       |
| K <sub>2</sub> O               | 0-1,5                     | 0-0,5                            | -                                              | -                                               | 0-1,5                                   | 3-15                       |
| Fe <sub>2</sub> O <sub>3</sub> | 0-0,6                     | 0-0,3                            | -                                              | -                                               | 0-0,3                                   | -                          |
| Cr <sub>2</sub> O <sub>3</sub> | 0-0,2                     | 0-0.2                            | -                                              | -                                               | -                                       | -                          |
| MnO <sub>2</sub>               | 0-0,2                     | -                                | -                                              | -                                               | -                                       | -                          |
| Co <sub>3</sub> O <sub>4</sub> | 0-0,1                     | -                                | -                                              | -                                               | -                                       | -                          |
| TiO <sub>2</sub>               | 0-0,8                     | 0-1                              | -                                              | -                                               | -                                       | -                          |
| SO <sub>3</sub>                | 0-0,2                     | -                                | -                                              | -                                               | 0-0,2                                   | -                          |
| Se                             | 0-0,1                     | -                                | -                                              | -                                               | -                                       | -                          |
| F                              | 0-0,1                     | 0-2                              | -                                              | -                                               | 0-2,5                                   | -                          |
| ZrO <sub>2</sub>               | -                         | -                                | -                                              | -                                               | -                                       | 0-3                        |
| PbO                            | -                         | -                                | -                                              | -                                               | -                                       | 15-80                      |