Abstract:

A gasket material for high pressure high temperature presses, comprising: - a proportion of a clay mineral - a proportion of a hard material for increasing the viscosity of the clay mineral - a proportion of a binder selected from the group of borate binders, phosphate binders, and mixtures thereof.
SYNTHETIC GASKET MATERIALS FOR USE IN HIGH PRESSURE HIGH TEMPERATURE PRESSES

This invention relates to synthetic gasket materials for high pressure high temperature (HPHT) presses and encompasses gaskets made from such materials.

HPHT presses are used in a number of applications but the principal one of note is for the manufacture or growth of synthetic diamond. In such applications gasket materials are required that can withstand the high pressures and temperatures involved. [For example of the order of 1500°C and pressures of 6GPa or more].

Typically such gaskets may be of varying form [for example, without limitation, being cubic, cylindrical or of more complex form to suit press geometry].

Such gasketing materials and methods of making same are disclosed in US5858525 and US6338754 the contents of which are hereby incorporated by way of reference.

US5858525 discloses the use binders in gasket composition, the binders comprising "the group of materials including sodium silicate, acrylic copolymers, arabic gum, portland cement and the like". US6338754 used sodium silicate binders. The applicants propose improved binder systems.

The present invention comprises a gasket material comprising:-

• a proportion of a clay mineral
• a proportion of a hard material for increasing the viscosity of the clay mineral
• a proportion of a binder selected from the group of borate binders, phosphate binders, and mixtures thereof.

By clay mineral is meant a silicate material of fine particle size. A non-exhaustive list of clay minerals is provided in US5858525, which list is incorporated by reference.

The proportions of clay mineral to hard material may (but does not have to) lie in the respective ratios 1-10:10-1.

The proportion of binder to the combined amount of clay mineral and hard material may (but does not have to) lie in the respective ratios 1:1-100.

The total amount of clay mineral, hard material, and binder may lie in the range 70-100wt% of the gasket material.

The clay mineral may be (but does not have to be) clays selected from the group consisting of akermanite, bertrandite, kaolinite, pyrophyllite, prehnite, pyrope, scolecite, serpentine, talc, zoisite, and mixtures thereof.
The hard material may be (but does not have to be) selected from the group consisting of silica, zircon, garnet, silicon carbide, boron carbide, alumina, zirconia, kyanite, mullite, and mixtures thereof.

A borate binder system may be (but does not have to be) a solution of boric acid and water comprising both liquid and solid form. The solution formed from such mixture may be super-saturated in form, heated above room temperature, and combined with other ingredients at elevated temperature.

A phosphate binder may be (but does not have to be) formed by reaction of an acid phosphate with a sparsely soluble oxide or an oxide mineral. Phosphate binders may comprise both solid and liquid components but conveniently binders comprising only solid components may be used. Typical phosphate binders include magnesium phosphate and aluminium phosphate but other phosphate binders may be used.

An advantage of borate and phosphate binders, versus sodium silicate is equipment cleanability - once sodium silicate sets, it needs to be chipped out. With borate binders such as boric acid, or phosphate binders, hot water will clean the equipment of unset material.

Manufacture of the gasket material may include the steps of dispersing the solid constituents, blending them, and pressing.

The solid constituents may be dispersed in a solution (e.g. a water solution of Boric Acid/Borax) and spray dried to provide a dispersed and intimately mixed material that will flow under pressing. Once the binder sets, such easy flow will be prevented.

The solid constituents may be granulated to ensure uniformity of distribution and improved pressing properties.

Pressing may include uniaxial pressing or isostatic pressing, or any form of pressing that provides the required density in the pressed product.

A typical composition might be:-

Clay mineral  60-90wt%
Hard material  5-35wt%
Binder        5-25wt%

and for example may comprise:-

Clay mineral  65±4wt%
Hard material 15±5wt%
Binder        5-25wt%.

Typical combinations of clay mineral and hard material include but are not limited to:-

- Talc and garnet
- Pyrophyllite and garnet
- Talc and zircon
- Pyrophyllite and zircon
However, generally the combination of:
• each and every clay mineral or combination thereof; with
• each and every hard material and combinations thereof; with
• each and every borate or phosphate binder and combinations thereof;
should be considered as falling within the scope of the invention.

The clay and hard material may have a fine particle size, for example being -200 mesh or -230 mesh or -270 mesh or -325 mesh or even finer. However coarser particle sizes are not excluded.

**Examples**

In the following examples:-
• talc was used as the clay mineral [the commercial grade "Nicron 554" from Emerys. It is a -325 mesh material with a mean particle size of 3 microns];
• -220 mesh silicon carbide was used, although finer material is preferred;
• examples #1 and #3 are prophetic examples.

Powders of examples #2, #4, and #5 indicated below were manufactured by using hot water to dissolve the boric acid, then adding the other ingredients, then drying to form a powder.

This powder was subsequently pressed in a laboratory uni-axial Carver press to a pressure of about 103MPa (15,000psi) to form sample discs for evaluation.

The discs prepared were found to be consolidated.

Assessment of the brittleness of the discs by crushing in a mortar and pestle back to a pressable powder showed that higher clay content (in the case of these examples, talc) improved the materials, in that Examples #4, and #5 appeared more brittle than Example #2.

<table>
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<th>#1</th>
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<th>#3</th>
<th>#4</th>
<th>#5</th>
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<tr>
<td>Talc</td>
<td>65%</td>
<td>65%</td>
<td>45%</td>
<td>45%</td>
<td>60%</td>
</tr>
<tr>
<td>B₃C</td>
<td>15%</td>
<td>0%</td>
<td>25%</td>
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<td>0%</td>
<td>15%</td>
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<tr>
<td>Boric acid</td>
<td>20%</td>
<td>20%</td>
<td>30%</td>
<td>30%</td>
<td>20%</td>
</tr>
<tr>
<td>Total:</td>
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<td>100%</td>
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For phosphate binders prospective embodiments include:

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<td>SiC</td>
<td>10%</td>
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<td>0%</td>
</tr>
<tr>
<td>Total dry mix</td>
<td>100%</td>
<td>100%</td>
<td>100%</td>
<td>100%</td>
</tr>
<tr>
<td>Phosphate binder (as % of dry mix)</td>
<td>10%</td>
<td>15%</td>
<td>15%</td>
<td>15%</td>
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These examples and prospective embodiments are indicative of materials that may be made in accordance with the invention and the invention is not restricted thereto.
A gasket material for high pressure high temperature presses, comprising:-

i. a proportion of a clay mineral

ii. a proportion of a hard material for increasing the viscosity of the clay mineral

iii. a proportion of a binder selected from the group of borate binders, phosphate binders, and mixtures thereof.

1. A gasket material as claimed in Claim 1, in which the proportions of clay mineral to hard material lie in the respective ratios 1-10:10-1.

2. A gasket material as claimed in Claim 1 or Claim 2, in which the proportion of binder to the combined amount of clay mineral and hard material lies in the respective ratios 1:1-100.

3. A gasket material as claimed in any of Claims 1 to 3, in which the total amount of clay mineral, hard material, and binder lies in the range 70-100 wt% of the gasket material.

4. A gasket material as claimed in any of Claims 1 to 4, comprising:
   - Clay mineral 60-90wt%
   - Hard material 5-35wt%
   - Binder 5-25wt%

5. A gasket material as claimed in Claim 5, comprising 61wt% or more clay mineral.

6. A gasket material as claimed in Claim 6, comprising:
   - Clay mineral 65±4wt%
   - Hard material 15±5wt%
   - Binder 5-25wt%

7. A gasket material as claimed in any of Claims 1 to 7, in which the clay mineral is selected from the group consisting of akermanite, bertandite, kaolinite, pyrophyllite, prehnite, pyrope, scolecite, serpentine, talc, zoisite, and mixtures thereof.

8. A gasket material as claimed in any of Claims 1 to 8, in which the hard material is selected from the group consisting of silica, zircon, garnet, silicon carbide, boron carbide, alumina, zirconia, kyanite, mullite, and mixtures thereof.

9. A method of making a gasket material as claimed in any of Claims 1 to 9, comprising the steps of dispersing the solid constituents, blending them, and pressing.
11. A method as claimed in Claim 10, in which dispersing and blending the solid constituents comprises dispersal of the solid constituents in a solution to form a suspension, and spray drying the suspension.

12. A method as claimed in Claim 10 or Claim 11, in which the solid constituents are granulated.

13. A gasket formed from the gasket material of any of Claims 1 to 9.
AMENDED CLAIMS
received by the International Bureau on 04 August 2015 (04.08.2015)

CLAIMS

1. A gasket material for high pressure high temperature presses, comprising:-
   i. a proportion of a clay mineral
   ii. a proportion of a hard material for increasing the viscosity of the clay mineral
   iii. a proportion of a binder selected from the group of borate binders, and mixtures of borate binders and phosphate binders.

2. A gasket material as claimed in Claim 1, in which the proportions of clay mineral to hard material lie in the respective ratios 1:10:1-10.

3. A gasket material as claimed in Claim 1 or Claim 2, in which the proportion of binder to the combined amount of clay mineral and hard material lies in the respective ratios 1:1-100.

4. A gasket material as claimed in any of Claims 1 to 3, in which the total amount of clay mineral, hard material, and binder lies in the range 70-100wt% of the gasket material.

5. A gasket material as claimed in any of Claims 1 to 4, comprising: -
   Clay mineral 60-90wt%
   Hard material 5-35wt%
   Binder 5-25wt%

6. A gasket material as claimed in Claim 5, comprising 61wt% or more clay mineral.

7. A gasket material as claimed in Claim 6, comprising: -
   Clay mineral 65±4wt%
   Hard material 15±5wt%
   Binder 5-25wt%

8. A gasket material as claimed in any of Claims 1 to 7, in which the clay mineral is selected from the group consisting of akermanite, bertandite, kaolinite, pyrophyllite, prehnite, pyrope, scolecite, serpentine, talc, zoisite, and mixtures thereof.

9. A gasket material as claimed in any of Claims 1 to 8, in which the hard material is selected from the group consisting of silica, zircon, garnet, silicon carbide, boron carbide, alumina, zirconia, kyanite, mullite, and mixtures thereof.

10. A method of making a gasket material as claimed in any of Claims 1 to 9, comprising the steps of dispersing the solid constituents, blending them, and pressing.

AMENDED SHEET (ARTICLE 19)
11. A method as claimed in Claim 10, in which dispersing and blending the solid constituents comprises dispersal of the solid constituents in a solution to form a suspension, and spray drying the suspension.

12. A method as claimed in Claim 10 or Claim 11, in which the solid constituents are granulated.

13. A gasket formed from the gasket material of any of Claims 1 to 9.
**INTERNATIONAL SEARCH REPORT**

**International application No**

PCT/EP2015/057275

**A. CLASSIFICATION OF SUBJECT MATTER**

INV. C04B 28/00 C04B 28/34 C04B 33/04 C04B 33/13 C04B 35/626
C04B 35/63 F16J 15/00 F16J 15/10

**ADD.**

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

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)

C04B F16J

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

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

EPO-Internal, WPI Data

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

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<td>CN 102 773 043 A (ZHONGNAN DIAMOND CO LTD) 14 November 2012 (2012-11-14) abstract; claims 1-6; examples 1-3</td>
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<td>US 6 045 885 A (CHENEY JAMES E [US]) 4 April 1 2000 (2000-04-04) column 1, line 62 - column 2, line 44; claims 1-5</td>
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Further documents are listed in the continuation of Box C.

See patent family annex.

* Special categories of cited documents :
  * "A" document defining the general state of the art which is not considered to be of particular relevance
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**Date of the actual completion of the international search**

24 June 2015

**Date of mailing of the international search report**

07/07/2015

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**Authorized officer**

Bonneau, Sebastien
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