We, GLAVERBEL, being the person(s) identified below as the Applicant, request the grant of a standard patent to the person identified below as the Nominated Person, for an invention described in the accompanying complete specification.

Full application details follow.

Applicant: GLAVERBEL
Address: Chaussee de la Hulpe, 166 B-1170 Brussels, BELGIUM
Nominated Person: GLAVERBEL
Address: Chaussee de la Hulpe, 166 B-1170 Brussels, BELGIUM
Invention Title: "PROCESS AND MIXTURE FOR FORMING A COHERENT REFRACTORY MASS ON A SURFACE"
Name(s) of Actual Inventor(s): Jean-Pierre MEYNCKENS; Leon-Philippe MOTTET
Address for service is: F.B. RICE & CO., 28A Montague St, Balmain N.S.W. 2041
Attorney Code: RI

BASIC CONVENTION APPLICATION(S) DETAILS

<table>
<thead>
<tr>
<th>Application No</th>
<th>Country</th>
<th>Country Code</th>
<th>Date of Application</th>
</tr>
</thead>
<tbody>
<tr>
<td>87969</td>
<td>Luxemburg</td>
<td>LU</td>
<td>3 July 1991</td>
</tr>
</tbody>
</table>

We are not an eligible person described in Section 33 - 36 of the Act.

Dated this 17 day of June 1992

GLAVERBEL

By: [Signature]

Registered Patent Attorney

57697
Commonwealth of Australia
The Patents Act 1952

DECLARATION IN SUPPORT

In support of the (Convention) Application made by: GLAVERBEL
of 166, Chasse de la Hulpe, 166 B-1170 Brussels BELGIUM

for a patent for an invention entitled: PROCESS AND MIXTURE FOR FORMING A
COHERANT REFRACTORY MASS ON A SURFACE

I (We) L. Tytgat-Vandenberghen and J. De Keersmaecker
of and care of the applicant company do solemnly and sincerely declare as follows:

a) I am (We are) the applicant(s) for the patent
or
b) I am (We are) authorised by the applicant(s) for the patent to make this declaration on its behalf.

The basic application(s) as defined by section 141 (42) of the Act was (were) made
on 3 July 1991 in Luxemburg

by GLAVERBEL

The basic application(s) referred to in this paragraph is (are) the first application(s) made in
a Convention country in respect of the invention the subject of the application.

a) I am (We are) the actual inventor(s) of the invention

or

b) Jean-Pierre Meynckens of Rue du Chateau, 17 B 6210 Villers-
Perwin BELGIUM
Leon-Philippe Mottet of Rue Grand Douze Bois, 19 B 6120
Nalinnes BELGIUM

is (are) the actual inventor(s) of the invention and the facts upon which
THE APPLICANT COMPANY
is (are) entitled to make the application are as follows:

The Applicant is a person who would if a patent were granted
upon an application made by the actual inventors be entitled
to have the patent assigned to it

Declared at Brussels this 9th day of June 1992

Signed
Declarant's Name

Status

F. B. RICE & CO PATENT ATTORNEYS
This form is suitable for any type of Patent Application. No legalisation required.
1. A process for forming a coherent refractory mass on a surface based on a silicon compound, wherein there is projected against the surface, simultaneously with oxygen, a mixture comprising refractory particles and combustible particles which react in an exothermic manner with the projected oxygen by releasing sufficient heat to form the refractory mass, under the action of heat of combustion, characterised in that the mixture comprises:

(i) combustible silicon particles;

(ii) as a major proportion by weight of the mixture, refractory particles of one or a plurality of substances; and

(iii) additive particles of another substance which, during the formation of the refractory mass, causes incorporation of silica, formed by combustion of the silicon particles, into a crystalline lattice, and/or

(iii) additive particles of a non-metallic compound which, during the formation of the refractory mass, generates said another substance which causes the incorporation of silica, formed by the combustion of silicon particles, into a crystalline lattice.

10. A mixture of particles intended for a process for forming a coherent refractory mass on a surface based on a silicon compound by projecting the mixture and oxygen against said surface, the mixture comprising refractory particles and combustible particles capable of reacting exothermically with oxygen to release sufficient heat to form, under the action of the heat of combustion, said refractory mass, characterised in that the mixture comprises:
(11) 18404/92

(i) combustible silicon particles;
(ii) as a major proportion by weight of the mixture, refractory particles of one or a plurality of substances; and

(iii) additive particles of another substance which, during the formation of the refractory mass, causes incorporation of silica, formed by combustion of the silicon particles, into a crystalline lattice, and/or

(iii) additive particles of a non-metallic compound which, during the formation of the refractory mass, generates said another substance which causes the incorporation of silica, formed by the combustion of silicon particles, into a crystalline lattice.
Invention Title:

PROCESS AND MIXTURE FOR FORMING A COHERENT REFRACTORY MASS ON A SURFACE

The following statement is a full description of this invention including the best method of performing it known to us:-
The present invention relates to a process for forming a coherent refractory mass on a surface wherein is projected against this surface, simultaneously with oxygen, a mixture of refractory particles and combustible particles which react in an exothermic manner with the projected oxygen by releasing sufficient heat to form, under the action of the heat of combustion, the aforesaid refractory mass. The invention also relates to a mixture of particles intended for a process for forming a coherent refractory mass on a surface by projecting the mixture and oxygen against the surface, the mixture comprising refractory particles and particles of a combustible substance which are capable of reacting exothermically with the oxygen in order to release sufficient heat to form, under the heat of combustion, the aforesaid refractory mass.

If it is desired to form a refractory mass in situ on a surface, one can choose between two known principal processes.

According to the first of these processes, sometimes called "ceramic welding", illustrated in Patent GB 1,330,894 (Glaverbel) and GB 2,170,191 (Glaverbel), a coherent refractory mass is formed on a surface by projecting onto the latter a mixture of refractory particles and combustible particles in the presence of oxygen. The combustible particles are particles whose composition and granulometry are such that they react in an exothermic manner with the oxygen while forming a refractory oxide and while releasing the necessary heat for melting, at least superficially, the projected refractory particles. Aluminium and silicon are examples of such combustible substances. It is known that silicon, properly speaking, must be classified as a half metal, but because silicon behaves like certain metals (it is capable of undergoing considerable exothermic oxidation in forming a refractory oxide), these combustible elements are called combustible metallic substances for reason of simplicity. In general it is recommended to perform the projection of particles in the presence of a high concentration of oxygen, for example, by using oxygen of commercial quality as a gas carrier. In this manner a coherent refractory mass is formed that adheres to the surface onto which the particles are projected. Because of the very high temperatures that the ceramic welding reaction can reach, it can penetrate slag which might be present on the surface of the refractory substance being treated, and it can soften or melt.
the surface in such a way that a good bond is produced between the treated surface and the newly formed refractory mass.

These known ceramic welding processes can be employed for forming a refractory article, for example, a block having a particular shape, but they are most widely used for forming coatings or for repairing bricks or walls and are particularly useful for repairing or reinforcing existing refractory structures, for example, for repairing walls or coating refractory equipment such as furnace walls in glassmaking or coke furnaces.

This operation is generally performed when the refractory base is hot. This makes it possible to repair eroded refractory surfaces while the equipment remains substantially at its working temperature and, in certain cases, even while it is operating.

The second known process for forming a refractory mass on a surface is called "flame spraying process." It involves directing a flame to the site where a refractory mass is to be formed and spraying refractory powder across this flame. The flame is fed by a gaseous fuel or liquid or even coke powder. It is apparent that the efficient utilisation of this flame spraying technique requires complete combustion of the fuel in order to generate the hottest flame possible and to attain maximum efficiency. In general, the temperature of the flame obtained with a flame spraying process is not so high as that which may be obtained with a ceramic welding technique, with the result that coherence of the formed refractory mass is not so good, and since the bond between the new refractory mass and the surface of the refractory base is formed at a lower temperature, this bond will not be so firm. Moreover, such a flame is less apt than a ceramic welding reaction to penetrate slag which might be present on the refractory surface being treated.

The composition of the mixture used in a ceramic welding process is generally chosen in such a way as to produce a repair mass which has a chemical composition similar or close to that of the basic refractory. This helps to ensure compatibility with and adhesion to the new material and the base material on which it is formed.

We have observed, however, that problems occur if it is desired to repair certain types of refractory structures and this, even if a refractory mass of a chemical composition is formed which is similar to that of the basic refractory mass.

For example, repairing refractory surface structures having a silicon carbide base with the help of a mixture containing primarily carbon silicon particles
and also particles of metallic combustible substances, such as aluminium and silicon particles, produces a refractory mass which does not always demonstrate sufficient adhesion to the base refractory.

Refractories having a base of silicon carbide are used in certain metallurgy equipment, in particular, in blast furnaces in the iron industry or in zinc distillation columns. During the operation of this equipment, certain portions of the refractory structures may have a rather low minimum operating temperature, for example, on the order of 700°C, and may additionally be subjected to significant variations of ambient temperature. It has been observed that the refractory masses produced by known techniques on these parts of refractory structures do not always demonstrate sufficient adhesion to the base refractory mass and, in certain cases, particularly when the repair is made on a block or a refractory wall whose temperature is low, the new refractory mass becomes completely separated from the base refractory mass and detaches itself during the operation of the equipment.

Similar problems present themselves if one desires to repair refractory structures having a high density silica base (so named in order to distinguish them from traditional silica refractories whose density is lower), used in certain coke furnaces; even though one can form a refractory similar in chemical composition to the base refractory mass, the new mass does not always sufficiently adhere and may even separate rapidly from the base refractory mass when the furnace is in operation.

A process is known from International Patent Application WO 90/03848 (Willmet/Willard) for the repair of, for example, furnace linings, wherein an inert carrier gas and particles of refractory oxide and combustible oxidisable material are delivered to a flame spraying apparatus, wherein high pressure oxygen aspirates and accelerates the carrier gas/particle mixture. Willard applies this process to the repair of refractory blocks/bricks in the tuyere line of a copper smelting converter as well as to the repair of silicon carbide tray columns. For example, one projects a mixture containing 79% silicon carbide, 16.25% silicon, 4% aluminium and 0.75% magnesium through a double venturi air oxygen system to a silicon carbide tray column.

However, the use of magnesium metal powder in this process is disadvantageous, at least in that since magnesium metal is relatively volatile, there is a degree of uncertainty about the composition of the formed refractory coating.

One of the objectives of the present invention is to solve these
problems.

The present invention relates to a process for forming a coherent refractory mass on a surface based on a silicon compound, wherein there is projected against the surface, simultaneously with oxygen, a mixture comprising refractory particles and combustible particles which react in an exothermic manner with the projected oxygen by releasing sufficient heat to form the refractory mass, under the action of heat of combustion, characterised in that the mixture comprises: (i) combustible silicon particles; (ii) as a major proportion by weight of the mixture, refractory particles of one or a plurality of substances; and (iii) additive particles of another substance which, during the formation of the refractory mass, causes incorporation of silica, formed by combustion of the silicon particles, into a crystalline lattice, and/or (iiib) additive particles of a non-metallic compound which, during the formation of the refractory mass, generates said another substance which causes the incorporation of silica, formed by the combustion of silicon particles, into a crystalline lattice.

The present invention also relates to a mixture of particles intended for a process for forming a coherent refractory mass on a surface based on a silicon compound by projecting the mixture and oxygen against said surface, the mixture comprising refractory particles and combustible particles capable of reacting exothermically with oxygen to release sufficient heat to form, under the action of the heat of combustion, said refractory mass, characterised in that the mixture comprises: (i) combustible silicon particles; (ii) as a major proportion by weight of the mixture, refractory particles of one or a plurality of substances; and (iii) additive particles of another substance which, during the formation of the refractory mass, causes incorporation of silica, formed by combustion of the silicon particles, into a crystalline lattice, and/or (iiib) additive particles of a non-metallic compound which, during the formation of the refractory mass, generates said another substance which causes the incorporation of silica, formed by the combustion of silicon particles, into a crystalline lattice.

Such a mixture and such a process are useful for forming high quality refractory masses for the repair of surfaces based on a silicon compound, such as, for example, refractory structures of furnaces as well as for welding pieces together. It is possible to obtain a refractory mass which demonstrates excellent adherence to the base refractory when the repaired surface undergoes repeated variations of thermal conditions during the operation of the equipment and/or when the repair is made on a surface whose temperature is relatively low, such as
between 600°C and 1000°C (for example, 700°C), although the invention is applicable to surfaces having a temperature outside this range.

The refractory masses produced according to the invention present thermal expansion properties at the interface between the surface and the formed refractory mass which are different from those that would be obtained if the starting mixture did not contain any substance causing the incorporation into a crystalline lattice of silica formed by combustion of silicon. We believe that the advantages obtained by the invention are due, at least in part, to this difference at the interface and that the refractory masses obtained demonstrate thermal expansion properties at the interface which are well adapted to those of the refractory structures in question.

The combustible silicon particles (i) may be used as the only combustible material or they may be mixed with particles of a further combustible material, such as aluminium. Thus, the mixture preferably further comprises combustible aluminium particles. Aluminium particles may be rapidly oxidised with a significant release of heat and form refractory oxides themselves. The adaptation of this characteristic thus favours the formation of high quality refractory masses.

The mixtures according to the invention, preferably comprise not more than 15% by weight silicon. This is important for limiting the amount of unreacted silicon which may remain in the formed refractory mass. We have found that the presence of unreacted silicon in the formed refractory mass may detract from the qualities thereof.

The refractory particles (ii) may be present in an amount of at least 70% by weight, most preferably at least 75% by weight, in order to obtain a homogeneous mass.

The additive particles (iii) preferably make up the remainder of the mixture and may comprise up to 25% by weight of the mixture, preferably from 5 to 15% by weight.

The combustible particles (i) used in the mixture preferably have an average particle size of less than 50μm.

The refractory particles (ii) preferably comprise substantially no particles with a size greater than 4mm, most preferably not greater than 2.5mm in order to facilitate the formation of a regular jet of powder.

The additive particles (iii) preferably used in the mixture preferably have a particle size of less than or equal to 500μm. If particles which are too large are employed, there is a risk that they will not play an effective role. Preferably,
these particles have a size of at least 10μm. If particles which are too small are employed, there is a risk that they will volatilise during the reaction.

Various substances are suitable for inducing, during the formation of the refractory mass, the incorporation of silica, formed by the combustion of silicon, into a crystalline lattice.

The aforesaid additive substance (iiia) causing the incorporation of silica, formed by the combustion of silicon, into a crystalline lattice, is preferably introduced into the mixture in the form of magnesia particles.

The presence of this compound into the mixture which is projected onto the refractory surface to be repaired helps to ensure the correct heat resisting properties of the refractory mass formed.

Moreover, the introduction of magnesia into the mixture permits the formation of a refractory mass in which one portion at least of the silica formed by the combustion of silicon is incorporated into a crystalline lattice of the forsterite type. This also helps to ensure the correct heat resisting properties of the refractory mass formed.

If the mixture contains aluminium as well as magnesia, a refractory mass may be formed in which one portion at least of the silica formed by the combustion of silicon is incorporated into a crystalline lattice of the forsterite structure and/or into a crystalline lattice of the spinel structure and/or into a crystalline lattice of the cordierite structure.

The presence of a crystalline lattice of the cordierite structure in the refractory mass formed helps to ensure excellent resistance to thermal shocks of this mass. The presence of a crystalline lattice of the forsterite structure and/or spinel structure, on the other hand, favourably influences the heat resistance of the formed refractory mass.

Other oxides such as calcium oxide or iron (II) oxide, may also be used as the additive substance (iiia) causing the incorporation of silica, formed by the combustion of silicon, in a crystalline lattice.

A mixture of particles may be used which additionally or alternatively comprises an additive substance or substances (iiib) whose composition is such that, when the refractory mass is formed, it/they generate a substance causing the incorporation of silica formed by the combustion of silicon, into a crystalline lattice.

For example, peroxides such as calcium peroxide, nitrides, carbides may be used.

An oxide, for example calcium oxide, may be introduced in the form of a compound, for example, in the case of calcium oxide, in the form of wollastonite
CaO.\(\text{SiO}_2\).

The present invention is particularly useful for repairing refractories having a silicon carbide base or refractories having a high density silica base. Consequently, it is preferred for ceramic welding to be carried out with the help of a mixture whose major portion by weight comprises silicon carbide or silica, respectively.

It goes without saying that the invention may also be useful for repairing other types of refractories based on a silicon compound than the ones previously mentioned, such as normal silica bricks and silica-alumina bricks.

The present invention will now be further illustrated in more detail with the help of the following examples:

**EXAMPLE I**

A refractory mass is formed on a wall of the zinc distillation column. This wall comprises bricks having a silicon carbide base. A mixture of refractory particles, particles of a combustible substance which are exothermically oxidisable by forming a refractory oxide, and of magnesia particles is projected onto these bricks. The temperature of the wall is 800°C. The mixture is projected at the rate of 60 kg/h into a stream of pure oxygen. The mixture has the following composition:

- SiC: 79% by weight
- Si: 8%
- Al: 5%
- MgO: 8%

The silicon particles have a dimension below 45 μm and a specific surface area comprised between 2,500 and 8,000 cm²/g. The aluminium particles have a dimension below 45 μm and a specific surface area comprised between 3,500 and 6,000 cm²/g. The dimension of the silicon carbide particles is less than 1.47 mm with 60% by weight from 1 to 1.47 mm, 20% from 0.5 to 1 mm, and 20% below 0.125 mm. The MgO particles have an average dimension of approximately 300 μm. "Average dimension" designates a dimension such that
50% by weight of the particles have a smaller dimension than this average.

The wall which has been repaired in this manner is subjected to significant variations of ambient temperature and it is observed that the new refractory mass adheres durably to the support.

The structure of the formed mass is examined under the microscope. Excellent continuity is observed between the new refractory mass and the base refractory mass. It is also observed that the silica formed by the combustion of silicon is incorporated into the crystalline lattices of forsterite, cordierite and aluminous spinel.

For purpose of comparison, a mixture not containing magnesia is projected under the same conditions. The composition of this mixture is as follows:

<table>
<thead>
<tr>
<th>Element</th>
<th>Weight Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiC</td>
<td>87%</td>
</tr>
<tr>
<td>Si</td>
<td>12%</td>
</tr>
<tr>
<td>Al</td>
<td>1%</td>
</tr>
</tbody>
</table>

It is observed that the refractory mass formed separates rapidly from the wall and detaches itself in solid blocks if the zinc distillation column continues to operate.

In a modification of this example, the mixture is used to repair the bottom of a coke furnace formed of normal silica bricks and silica-alumina bricks. One obtains a repair mass having good resistance to abrasion which adheres well to the wall, even when subjected to significant thermal variations.

**EXAMPLE 2**
As a variation of Example 1 a mixture having the following composition is used:

<table>
<thead>
<tr>
<th>Element</th>
<th>Weight Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiC</td>
<td>82%</td>
</tr>
<tr>
<td>Si</td>
<td>8%</td>
</tr>
<tr>
<td>Al</td>
<td>5%</td>
</tr>
<tr>
<td>MgO</td>
<td>5%</td>
</tr>
</tbody>
</table>

The wall being repaired comprises bricks having a silicon carbide base and it has a temperature of 700°C. The refractory mass obtained also adheres durably to the wall.

**EXAMPLE 3**
The object is to form a refractory mass on a wall of a coke furnace comprising high density silica bricks. While the apparent density of traditional silica bricks is on the order of 1.80, the apparent density of high density bricks is
approximately 1.89. Such bricks have recently appeared on the refractory material market, presenting advantageous characteristics by comparison with traditional silica bricks, notably with respect to their properties of gas permeability and thermal conductivity.

The repair is performed on a wall whose temperature is approximately 750°C with the help of the following mixture:

- SiO₂: 80.5% by weight
- Si: 11.1%
- Al: 1%
- MgO: 7.4%

The dimension of the SiO₂ particles is less than 2 mm, with a maximum of 30% by weight from 1 to 2 mm, and less than 15% by weight below 100 μm.

The formed mass adheres durably to the wall.

By contrast, projection under the same operating conditions, of a similar mixture but not containing magnesia, furnished a refractory mass which easily separates itself from the wall if the latter is subjected to various thermal conditions which are present when the furnace is in operation.

**EXAMPLE 4**

The object is to form a refractory mass on a wall of a coke furnace made of a refractory based on a silicon compound which is subject to significant variations of ambient temperature and of which the temperature does not exceed 900°C. The repair is performed on a wall whose temperature is approximately 750°C with the help of the following mixture:

- SiO₂: 80% by weight
- CaO.SiO₂ (wollastonite): 8%
- Si: 8%
- Al: 4%

The average dimension of the wollastonite particles is about 300μm.

The metal particle size is as given in Example 1 and the silica particle size is as given in Example 3.
THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:-

1. A process for forming a coherent refractory mass on a surface based on a silicon compound, wherein there is projected against the surface, simultaneously with oxygen, a mixture comprising refractory particles and combustible particles which react in an exothermic manner with the projected oxygen by releasing sufficient heat to form the refractory mass, under the action of heat of combustion, characterised in that the mixture comprises:
   (i) combustible silicon particles;
   (ii) as a major proportion by weight of the mixture, refractory particles of one or a plurality of substances; and
   (iiia) additive particles of another substance which, during the formation of the refractory mass, causes incorporation of silica, formed by combustion of the silicon particles, into a crystalline lattice, and/or
   (iiib) additive particles of a non-metallic compound which, during the formation of the refractory mass, generates said another substance which causes the incorporation of silica, formed by the combustion of silicon particles, into a crystalline lattice.

2. A process for forming a refractory mass according to claim 1, characterised in that said substance (iiia) causing the incorporation of silica, formed by the combustion of the silicon particles, into a crystalline lattice, is introduced into the mixture in the form of magnesia particles.

3. A process for forming a refractory mass according to claim 2, characterised in that at least one portion of the silica formed by the combustion of silicon is incorporated into a crystalline lattice of the forsterite structure.

4. A process for forming a refractory mass according to one of claims 1 to 3, characterised in that said combustible particles (i) further comprise aluminium particles.

5. A process for forming a refractory mass according to claims 2 and 4, characterised in that at least a portion of the silica formed by the combustion of silicon is incorporated into a crystalline lattice of the forsterite structure and/or into a crystalline lattice of the spinel structure and/or into a crystalline lattice of the cordierite structure.

6. A process for forming a refractory mass according to any preceding claim, characterised in that said non-metallic compound (iiib) is introduced into the mixture in the form of particles of a peroxide or a silicate.

7. A process for forming a refractory mass according to one of claims
1 to 6, characterised in that said refractory particles (ii) constituting the major portion by weight of the mixture are silicon carbide particles.

8. A process for forming a refractory mass according to one of claims 1 to 6, characterised in that said refractory particles (ii) constituting the major portion by weight of the mixture are silica particles.

9. A process according to any preceding claim, characterised in that the temperature of the surface is less than 1000°C.

10. A mixture of particles intended for a process for forming a coherent refractory mass on a surface based on a silicon compound by projecting the mixture and oxygen against said surface, the mixture comprising refractory particles and combustible particles capable of reacting exothermically with oxygen to release sufficient heat to form, under the action of the heat of combustion, said refractory mass, characterised in that the mixture comprises:

(i) combustible silicon particles;
(ii) as a major proportion by weight of the mixture, refractory particles of one or a plurality of substances; and

(iii) additive particles of another substance which, during the formation of the refractory mass, causes incorporation of silica, formed by combustion of the silicon particles, into a crystalline lattice, and/or

(iii) additive particles of a non-metallic compound which, during the formation of the refractory mass, generates said another substance which causes the incorporation of silica, formed by the combustion of silicon particles, into a crystalline lattice.

11. A mixture according to claim 10, characterised in that said mixture contains particles of magnesia as said particles of another substance (iii).

12. A mixture according to claim 10 or 11, characterised in that said mixture contains particles of a peroxide or a silicate as said non-metallic compound (iii).

13. A mixture according to one of claims 10 to 12, characterised in that said combustible particles (i) further comprise aluminium particles.

14. A mixture according to one of claims 10 to 13, characterised in that said refractory particles (ii), constituting the major portion by weight of the mixture, are silicon carbide particles.

15. A mixture according to one of claims 10 to 13, characterised in that said refractory particles (ii), constituting the major portion by weight of the mixture, are silica particles.
16. A mixture according to one of claims 10 to 15, characterised in that said additive particles (iiia) or (iiib) have a particle size of less than or equal to 500μm.

17. A mixture according to any one of claims 10 to 16, characterised in that the additive particles (iiia) or (iiib) have a particle size of at least 10μm.

18. A mixture according to any one of claims 10 to 17, characterised in that the level of silicon therein is not more than 15% by weight.

DATED THIS 17TH DAY OF JUNE 1992

GLAVERBEL

Patent Attorneys for the Applicant:-

F.B. RICE & CO.