I\We, ISOVER SAINT-GOBAIN of "LES MIROIRS" 18 AVENUE D'ALSACE 92400 COURBEVOIE FRANCE hereby apply for the grant of a standard patent for an invention entitled: BINDER AND BINDER-BASED SIZE FOR MINERAL FIBRES. which is described in the accompanying complete specification.

Details of basic application(s):

<table>
<thead>
<tr>
<th>Number of basic application</th>
<th>Name of Convention country in which basic application was filed</th>
<th>Date of basic application</th>
</tr>
</thead>
<tbody>
<tr>
<td>88.14008</td>
<td>FR</td>
<td>27 OCT 88</td>
</tr>
</tbody>
</table>

My/our address for service is care of GRIFFITH HACK & CO., Patent Attorneys, 601 St. Kilda Road, Melbourne 3004, Victoria, Australia.

DATED this 26th day of October 1989

ISOVER SAINT-GOBAIN

GRIFFITH HACK\&CO.

TO: The Commissioner of Patents.
AUSTRALIA
PATENTS ACT 1952

DECLARATION IN SUPPORT OF AN APPLICATION
FOR A PATENT

In support of an application made by
ISOVER SAINT-GOBAIN
Siège Social : 18, avenue d'Alsace
92400 COURBEVOIE
Cedex 27 - 92696 PARIS LA DEFENSE

for a patent for an invention entitled:
BINDER AND BINDER-BASED SIZE FOR MINERAL FIBRES

I, S. Levaguerese
of Isover Saint-Gobain, "Les Miroirs",
18 Avenue d'Alsace, 92400 Courbevoie, France
do solemnly and sincerely declare as follows:

1. I am authorised by the above mentioned applicant for the patent to make
this declaration on its behalf.

2. The name and address of each actual inventor of the invention
is as follows:
   Bernard GICQUEL, 8, Allee des bouleaux,
   95350 SAINT-BRICE SOUS FORET, France

3. The facts upon which the applicant is entitled to make this application
are as follows:
The said applicant is the assignee of the
said inventor

4. The basic application(s) as defined by Section 141 of the Act was (were)
made as follows:
Country France on 27th October 1988
in the name(s) Isover Saint-Gobain
and in on
in the name(s)

5. The basic application(s) referred to in the preceding paragraph was
were the first application(s) made in a Convention country in respect of
the invention the subject of this application.

Declared at Courbevoie, France
this 18th day of September 1989
Signed S. LEVAGUERESSE
Position

GRIFFITH HACK & CO
PATENT AND TRADE MARK ATTORNEYS
MELBOURNE - SYDNEY - PERTH
1. Size for so-called insulating glass fibres based on epoxy resin of glycidyl ether type dispersable in water, of an amino setting agent with a flash point over 180°C and additives (in parts calculated per 100 parts of dry resin), namely 0.1 to 2% of silane and 0 to 15% of a mineral oil.
AUSTRALIA
PATENTS ACT 1952
COMPLETE SPECIFICATION (ORIGINAL)
FOR OFFICE USE

Short Title: 
Int. Cl: 
Application Number: 
Lodged: 
Complete Specification-Lodged: 
Accepted: 
Lapsed: 
Published: 

Priority: 
Related Art: 

TO BE COMPLETED BY APPLICANT

Name of Applicant: ISOVER SAINT-GOBAIN
Address of Applicant: "LES MIROIRS"
18 AVENUE D'ALSACE
92400 COURBEVOIE
FRANCE

Actual Inventor: 
Address for Service: GRIFFITH HACK & CO.,
601 St. Kilda Road,
Melbourne, Victoria 3004,
Australia.

Complete Specification for the invention entitled:
BINDER AND BINDER-BASED SIZE
FOR MINERAL FIBRES.

The following statement is a full description of this invention including the best method of performing it known to me:-
The invention concerns the use of a new type of thermosetting synthetic resin for the production of products based on mineral fibres, in particular glass fibres such as mineral fibre mats intended for thermal insulation and/or soundproofing of buildings.

Most mineral-fibre based insulating materials comprise a binder which ensures the mechanical strength of the material, i.e., a link between the fibres. This binder must be dispersed homogeneously over the fibres; to avoid forming clumps of fibres surrounded by a clog of binder within a group of more brittle and therefore dustier fibres, the binder is always used in diluted state in a size.

It is known procedure to use thermosetting phenolic moulding resins (phenol-formol) or amino-aldehydic resins (melamine formol or urea-formol). The most frequently used binders are resols, products of condensation - in the presence of alkaline or alkaline-earth catalysts - phenols, having ortho- and para-vacant sitings, and aldehydes (principally formaldehyde). These resins form a size which, in addition to water, contains urea which serves to reduce the free formol content and also acts as a binder, and various additives such as oil, ammonia, coloring agents and, if necessary, fillers.

There are very many selection criteria, and of different types, for a binder, without forgetting however that a binder must above all adhere correctly to the glass.

Firstly, it is essential that the binder be rheologically
compatible with the fibre manufacturing process. Without going into unnecessary details here, let us just say that the glass fibres are usually produced using a centrifuge with a vertically oriented axis into which a continual jet of molten glass is introduced. The glass is sprayed towards the peripheral wall of the centrifuge from which it escapes in the form of filaments through a multitude of tiny orifices; the said filaments being drawn out and pulled downwards by a high temperature high pressure gas current. The fibres obtained are collected on a gas permeable conveyor and thus form a mattress of varying thickness depending on the speed of the conveyor.

The size should ideally clad each fibre produced in this way perfectly; it is therefore preferable to spray the size compound whilst the fibres are still separate, i.e. before the mattress is formed. Consequently, the size is sprayed into the fibre reception hood, below the burners generating the gas current to draw out the fibres. As a corollary to this operation it is forbidden to use inflammable organic solvents and/or pollutants in formulating the size, since the risk of fire and/or pollution in the reception hood is too high. In addition, the resin serving as binder must not polymerise too rapidly before taking on the desired shape.

Furthermore, although this polymerisation should not be too rapid, it should not take too long. However, on this point, resins used in the current state of the art are not perfectly satisfactory since complete polymerisation is achieved in a time compatible with a high production speed only after remaining in a high temperature oven (at approx 250°C) which is a high power consumer.

Lastly, the resin and its implementation process must be of relatively moderate cost compatible with that of glass fibre manufacture and not lead either directly or indirectly to the formation of toxic or polluting effluents. In this respect, the applicant company has chosen to select resins not
necessitating the use of formaldehyde in their manufacture, and which in addition do not release significant quantities of formaldehyde when they decompose under the effects of great heat. In such conditions, the product is, of course, non-toxic and in addition does not give off an unpleasant odour if it should burn.

In an associated industry to the insulating glass fibre industry, i.e. the reinforcement glass fibre industry, it is known procedure to sheath the glass fibres with epoxy resins. The sizing operation has the double object of protecting the fibres individually so that they are thus less sensitive to friction and (offer) better bonding to the plastic material for which the fibres act as reinforcement. But in this case, we are not trying to link the glass fibres and nothing allowed concluding that glass/glass bonding was sufficiently solid (for good mechanical resistance) and localized (throughout the total thickness).

After many industrial tests, the applicant obtained very satisfactory results with a size intended for so-called insulating fibres, based on epoxy resin of glycidyl ether type, carried in an aqueous medium, and a non volatile amino setting agent comprising as additives calculated in parts by weight per 100 parts of dry resin - 0.1 to 2 parts of silane and 0 to 15 parts of mineral oil.

By carried in an aqueous medium we mean a resin directly dispersible in water or likely to be emulsified with or without the addition of an emulsifying agent.

The epoxy resins preferred for the invention have a mean polymerisation index \( n \) from 0 to 1 inclusive and preferably under 0.2, \( n \) being equal to the mean number of supplementary bisphenol A groups per glycidyl ether molecule obtained by condensation of epichlorhydrin \((\text{CH}_2\text{CH} = \text{CHCl})\) and of bisphenol A \((\text{HO} - \text{C}_6\text{H}_4 - \text{C} (\text{CH}_3)_{3} - \text{C}_6\text{H}_4 - \text{OH})\), the condensation reaction being carried out in strictly stoechiometric
conditions (2 moles of epichlorhydrin per 1 mole of bis-phenol A) giving a glycidylic ether of index \( n = 0 \). Resins with a low polymerisation index — and therefore shorter chains — usefully form a denser reticulated network which one finds leads experimentally to products of higher mechanical strength. However, an epoxy resin of index \( n = 0 \) is not preferred since it tends to crystallise during storage and is much more difficult to synthesise since it is purer and thus more costly.

By non volatile amino setting agent we mean a setting agent with a flash point over 180°C; indeed, in these conditions, no self-igniting is observed in the fibre reception hood where the binder is sprayed.

As amino setting agent, one can use primary, secondary, aliphatic, alicyclic, aromatic or araliphatic polyamines, polyaminoamides. The NH equivalent molar mass, i.e. the quantity of product necessary to obtain the equivalent of one amino-hydrogen link per mole, is chosen preferably at under 100 g which corresponds to a high number of reactive sites per molecule.

Furthermore, one can use a catalytic agent for example of the tertiary amino type.

The polymerisation index and NH equivalent molar mass are two fairly symmetrical conditions and it is possible to compensate — at least to an extent — a fairly poor polymerisation index by using a setting agent of suitable NH equivalent molar mass and vice versa.
Generally excluded from the scope of the present invention are the hardeners belonging to the group of polyacids and acid anhydrides, this being due to the fact of their low solubility in water which raises significant problems in their utilisation because of their corrosive nature, exacerbated by their high cost. Also excluded are phenolic and aminoplast resins - which release formol during their curing, and urea, melamine, guanamine types of hardeners - which are poorly soluble and require the use of an accelerator.

On the other hand, it is possible to use hardeners of the polyamide or amino-polyamide type, which are products of moderate cost and of which the conditions for their utilisation are relatively simple and they do not release formol during their curing. On the other hand this type of hardener has the disadvantage of undergoing reaction at a relatively low temperature, which entails the danger of pre-gelification. Furthermore, some of them are corrosive and when heated there is a substantial release of amine compounds which are a pollution risk.

These disadvantages are not found with hardeners of the dicyandiamine type (DCN) which are inexpensive, non-toxic, non-volatile and non-corrosive, slightly soluble in water and, above all, since they react almost exclusively only when heated, they may be stored for approximately one year without any observable hardening of the resin.

The resin sprayed onto the fibres must not harden before the mat is formed, that is to say pregelification must be as little as possible. We have found in experiments that this condition is satisfied if the epoxy resin gelling time is over 25 minutes at 100°C; the gelling time being, by definition, the time needed at a given temperature for a certain quantity of resin to attain a viscosity set at 3000 centipoises. With
the usual phenoplastic resins, a gelling time of 25 minutes is judged insufficient and we prefer resins with a gelling time of over 1 hour which is highly limiting. With epoxy resins, much shorter gelling times are possible, surprisingly, if necessary using more dilute resins. It seems, indeed, that phenoplastic epoxy resin/water systems are less stable in the oven than phenoplastic resin/water systems which enables eliminating the water more easily.

In this invention, preparation of the size does not require formol and neither does one see a significant amount of formol being given off in the oven. In addition, total polymerisation can be carried out at a temperature under 220°C which reduces the risk of producing pollutant products in the oven which require later elimination, by pyrolysis in particular.

Other characteristics of the invention are set out in detail below referring to comparative tests carried out on 3 resins A, B, C satisfying the following formulations:

**RESIN A** formo-phenolic resin (standard resin).
Dry part : 55% (by weight) of formol resin - whose phenol/formaldehyde ration is equal to 3.2, with mineral catalysis less than 20 mPa of viscosity at 20°C - and 45% of urea.

**RESIN B** bi-compound epoxy resin based on diglycidylether of bisphenol A (Euroepox 756, trademark of the SCHERRING company; epoxy index 0.54 ± 0.02 (epoxy mole for 100 g); epoxy equivalent 178 - 192 (g/mole); mean polymerisation index n = 0.1

**RESIN C** bi-compound epoxy resin Neoxyl 865, trademark of the SAVID company, epoxy index 0.33 (epoxy mole for 100 g); epoxy equivalent 300 g/mole, mean polymerisation index n = 0.91.

**SETTING AGENT D** Water-based polyamine, Euredur 36, patented
trademark of the SCHERRING company; flash point 190°C, dry extract 80%, NH equivalent mass 132 g (i.e. one NH active group for 132 g of dry product).

SETTING AGENT E Aliphatic polyamine XIONEL SP 3288 trademark of the SAVID company; NH equivalent mass 57 g.

SETTING AGENT F Dicyane diamide, in association with a polymerisation accelerator, preferably of the tertiary amino type (for example tri-demethylaminoethyl phenol)

Unless otherwise stipulated, the resins are mixed with the setting agents in a ratio (by weight) of 1 NH group for 1 epoxy equivalent group.

VICOSIMETRIC BEHAVIOUR

The size must have good rheological compatibility with the fibre manufacturing process. In particular, one must avoid gellification of resin occurring in the fibre reception hood before the mat is formed in order to avoid forming unhomogeneous fibrous masses.

To estimate the viscosimetric behaviour in a container thermostatically controlled at 100°C, 10 g of resin is placed in a 30% solution in de-ionised water. A viscosimetric probe is immersed in the container and the time taken for viscosity to reach 3000 centipoises is measured.
- Test n° 1 (resin A) : 60
- Test n° 2 (resin B + setting agent D) : 27
- Test n° 3 (resin C + setting agent E) : 39

The gelling times obtained during tests 2 and 3 are thus much shorter than for test n° 1; however one notes, surprisingly, that this has no significant effect, on line, on condition that the quantities of water used are increased, if necessary.

ON LINE TEST

For the invention, the size is intended to be sprayed onto the so-called insulating glass fibres, i.e. obtained using aerodynamic processes with drawing out of fibres by a high pressure high temperature gas current, as opposed to so-called textile fibres obtained by mechanical drawing out of filaments produces by a spinning machine. The size is particularly
suited to fibres obtained according to the TEL process, molten glass being introduced inside the centrifuge plate revolving at high speed from which it escapes in the form of filaments through a series of orifices practised on the plate wall, the filaments being drawn out in the form of fibres by a high speed high temperature gas current generated by the burners surrounding the plate. The size is usefully sprayed onto the fibres before they are collected by a reception device. The sizes are prepared by dispersion of the resin in a quantity of water calculated in order to bring the proportion of dry extract to 10%, then by adding a silane. For the standard resin, we also added to the size 3 parts of ammonia for 100 parts of formo-phenolic resin in compliance with standard practice.

We firstly carried out the different tests on a laboratory line producing glass fibres with drawing out at 12 kg/hour. The characteristics of the fibre producing unit comply with the teachings of patent FR-2 223 318 and the fibres produced thus are comparable to those obtained industrially. In the fibre-producing hood, we spray onto the fibres a size with 2% of resin in de-ionised water. We obtain squares with sides of 450 mm by 50 mm thick with a binder content of approximately 5% after polymerisation.

**TENSILE STRENGTH**

The tensile strength (or "TS" given in gf/g) is measured on test samples in the form of rings drawn out by two interior rods in compliance with standard ASTM C 686-71T. The results of these measurements are indicated in TABLE 1. Ageing is simulated by passing the test sample through an autoclave for 30 minutes at 107°C, under autogenous water pressure.
According to the invention the mechanical strengths after ageing are slightly less with resins but however remain satisfactory. The influence of the epoxy resin polymerisation index $n$ and of the amino setting agent NH equivalent molar mass is exemplified by the following tests (carried out with 0.5% of silane added systematically).

The resin formulations used for tests 7 to 10 of TABLE II (TS in g/f) are as follows:

Test n° 7: resin B + setting agent D,
Test n° 8: resin C + setting agent E,
Test n° 9: resin C + setting agent D,
Test n° 10: resin B + setting agent E.

Tests 5, 7 and 10 carried out with the resin having the smallest polymerisation index give the best results after ageing. Test 8 (and also test 6) indicate that a setting agent whose NH equivalent molar mass is high associated with a resin with a high polymerisation index leads to products initially
very correct, but whose mechanical strength deteriorates greatly.

The poorest results are obtained with test 9 corresponding to an epoxy resin with high polymerisation index and a setting agent with low NH equivalent molar mass.

The product preferred for the invention (test 10) has excellent behaviour after preparation and after ageing. In addition, an adequate quantity of silane enables optimisation of the properties as shown in tests 11 to 14 in TABLE III established by varying the quantity of silane in the different samples prepared from a type B resin and a setting agent type D.

**TABLE III**

<table>
<thead>
<tr>
<th>Test</th>
<th>Silane %</th>
<th>Binder %</th>
<th>TS after preparation</th>
<th>TS after autoclave</th>
</tr>
</thead>
<tbody>
<tr>
<td>11</td>
<td>0</td>
<td>6.6</td>
<td>707</td>
<td>258</td>
</tr>
<tr>
<td>12</td>
<td>0.5</td>
<td>5.6</td>
<td>724</td>
<td>392</td>
</tr>
<tr>
<td>13</td>
<td>1.0</td>
<td>5.8</td>
<td>692</td>
<td>395</td>
</tr>
<tr>
<td>14</td>
<td>1.5</td>
<td>6.5</td>
<td>712</td>
<td>422</td>
</tr>
</tbody>
</table>

The main effect of adding silane is the improvement of product ageing whereas at a nearby binder rate the tensile strength (given here in gf/g) measured immediately after product preparation is practically constant. The best results are obtained for a percentage of silane between 0.5 and 1% inclusive. In the case of insulating fibre materials, it therefore appears that the silane acts essentially by forbidding the insertion of water molecules between the glass and the resin and not as a promoter of glass/resin bonding.

As regards the influence of the ratio of epoxy resin to its setting agent we once again operated with a type B resin and type D setting agent, adding 0.5% of silane in compliance with experience gained from the previous tests. The results are summarised in Table IV below.
The best results (for an identical binder rate) are obtained when the ratio of number of epoxy resin groups to number of setting agent NH equivalent groups is close to the stoichiometric ratio (i.e. 53/43 by mass for the resins of tests 15 to 19).

We then checked these first results by realizing 9 samples including two reference samples based on a standard resin. These samples were obtained on a pilot production line in conditions very close to industrial conditions. In this line, the glass fibres are prepared according to the so-called "TEL" process as explained in patent EP-91 866. Production of drawn-out glass is 20 tonnes per day. The sizes are delivered by a dosing pump with a quantity of de-ionised water enabling the proportion of dry extract to be brought to 10%. As additive, they include a silane and, as softener and anti-dust agent, from 0 to 10% of mineral oil. On can also use other types of oil such as linseed oil, soybean oil, safflower oil fatty acid, fish oil or Chinese wood oil or a non drying oil such as coconut oil, palm oil or stearic acid. The sizes are sprayed into the fibre collection hood at an air pressure of 1.5 bar; a certain quantity of extra water, referred to as overspray, is sprayed at the same time to bring the proportion of dry extract in relation to the final quantity of water to a value of between 5 to 8 % inclusive and preferably between 6.5 and 7.5%. In compliance with the teachings of viscosimetric measurements, tests with the resin according to the invention were carried out with a quantity of extra water more than 50% more than that used for a standard size.
Polymerisation is carried out in a ventilated oven in which the glass fibre mat penetrates between two squeeze rollers which impose upon it a given thickness, greater than rated thickness, i.e. the minimum thickness guaranteed to the user.

Here below we give details of preparation conditions of the different samples, the rate of binder measured after polymerisation and dimensional characteristics (gsm substance, density, actual product thickness).

Sample n° 20

- Size formulation:
  - formo-phenolic control resin
  - type A: 100 parts
  - % of silane: 1 part
  - % of oil: 10 parts
  - % of liquid ammonia: 3 parts
- Flow rate:
  - size (kg/h): 540
  - overspray (kg/h): 200
- Temperature of product in oven: 250°C
- Percentage of binder in finished product: 5.04
- Gsm substance (g/m2): 878
- Density (kg/m3): 10.97
- Thickness (mm): 129.2

Sample n° 21

- Size formulation:
  - Resin B, setting agent D in ratio: 55/45
  - % of silane: 0.5
- Flow rate:
  - size (kg/h): 640
  - overspray (kg/h): 300
- Temperature of product in oven: 250°C
- Percentage of binder in finished product: 5.88
- Gsm substance (g/m2): 899
- Density (kg/m3): 11.24
- Thickness (mm): 129.3

Sample n° 22

- Size formulation:
  - Resin B, setting agent D in ratio: 55/45
<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Size Formulation</th>
<th>Resin B, Setting Agent D in Ratio</th>
<th>% of Silane</th>
<th>Flow Rate</th>
<th>Temperature of Product in Oven</th>
<th>Percentage of Binder in Finished Product</th>
<th>Gsm Substance (g/m²)</th>
<th>Density (kg/m³)</th>
<th>Thickness (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>23</td>
<td></td>
<td>55/45</td>
<td>0.5</td>
<td>840 kg/h</td>
<td>250°C</td>
<td>5.06</td>
<td>892</td>
<td>11.15</td>
<td>128.7</td>
</tr>
<tr>
<td>24</td>
<td>formo-phenolic control resin type A</td>
<td>55/45</td>
<td>0.5</td>
<td>840 kg/h</td>
<td>250°C</td>
<td>5.12</td>
<td>897</td>
<td>11.21</td>
<td>129.6</td>
</tr>
<tr>
<td>25</td>
<td></td>
<td>Resin B, Setting Agent D in Ratio</td>
<td>55/45</td>
<td>540 kg/h</td>
<td>218°C</td>
<td>4.5</td>
<td>916</td>
<td>11.4</td>
<td>129.5</td>
</tr>
</tbody>
</table>
- % of silane: 0.5
- **Flow rate:**
  - size (kg/h): 840
  - overspray (kg/h): 300
- **Temperature of product in oven:** 216°C
- **Percentage of binder in finished product:** 5.8
- Gsm substance (g/m²): 916
- **Density (kg/m³):** 12.0
- **Thickness (mm):** 129.1

**Sample nº 26**

- **Size formulation:**
  - Resin B, setting agent D in ratio: 55/45
  - % of silane: 0.5
  - % of oil: 9.5

- **Flow rate:**
  - size (kg/h): 840
  - overspray (kg/h): 300
- **Temperature of product in oven:** 218°C
- **Percentage of binder in finished product:** 6.5
- Gsm substance (g/m²): 908
- **Density (kg/m³):** 11.3
- **Thickness (mm):** 128.2

**Sample nº 27**

- **Size formulation:**
  - Resin B, setting agent D in ratio: 55/45
  - % of silane: 0.5
  - % of oil: 9.5

- **Flow rate:**
  - size (kg/h): 840
  - overspray (kg/h): 300
- **Temperature of product in oven:** 200°C
- **Percentage of binder in finished product:** 6.2
- Gsm substance (g/m²): 898
- **Density (kg/m³):** 11.2
- **Thickness (mm):** 129.0

**Sample nº 28**

- **Size formulation:**
  - Resin B, setting agent D in ratio: 50/50
  - % of silane: 0.5
<table>
<thead>
<tr>
<th>Sample no.</th>
<th>Size formulation</th>
<th>Flow rate</th>
<th>Temperature of product in oven</th>
<th>Percentage of binder in finished product</th>
<th>Gsm substance (g/m²)</th>
<th>Density (kg/m³)</th>
<th>Thickness (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>29</td>
<td>Resin C, setting agent E in ratio 84/16</td>
<td>size (kg/h): 840, overspray (kg/h): 300</td>
<td>193°C</td>
<td>6.0</td>
<td>911</td>
<td>11.4</td>
<td>131.3</td>
</tr>
<tr>
<td>30</td>
<td>Resin C, setting agent E in ratio 84/16</td>
<td>size (kg/h): 740, overspray (kg/h): 200</td>
<td>240°C</td>
<td>5.95</td>
<td>920</td>
<td>11.5</td>
<td>127.9</td>
</tr>
</tbody>
</table>

The size according to the invention therefore enables obtaining products which scarcely differ from standard products from point of view of their dimensional characteristics (density and thickness) and this without important modification of the manufacturing process.

Samples 20 and 24 obtained with a standard size are yellow
in the absence of specific coloring additives. Samples 21, 22 and 23 are very slightly brown however samples 25 to 28 are of white colour, it is therefore advantageous to maintain the temperature of the product in the oven at approximately 220°C, which enables a good polymerisation of the binder and in addition one can choose exactly the final colour one wishes the product to have. In addition, the risk of giving off pollutant emanations is less if the oven temperature is low.

For its packaging, on leaving the oven, the product is compressed with a compression rate equal by definition to the ratio of rated thickness to thickness under compression. The samples were tested for compression rates equal to 4 or 6. To check the good dimensional resistance of a sample, we indicate the thickness after unwrapping calculated as a percentage of rated thickness; this percentage referred to as thickness recovery, can therefore sometimes exceed 100.

Thickness recovery 24 hours after manufacture for a compression rate of 6 (and 4, respectively)

<table>
<thead>
<tr>
<th>Sample no</th>
<th>20</th>
<th>21</th>
<th>22</th>
<th>23</th>
<th>24</th>
<th>25</th>
<th>26</th>
<th>27</th>
<th>28</th>
<th>29</th>
<th>30</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness</td>
<td>135.8 (140.7)</td>
<td>125.8 (133.9)</td>
<td>126.0 (135.1)</td>
<td>104.7 (119.7)</td>
<td>143.1</td>
<td>127.5</td>
<td>129.4</td>
<td>129.2</td>
<td>131.7</td>
<td>141.8 (137.2)</td>
<td>143.7 (136.4)</td>
</tr>
</tbody>
</table>

Thickness recovery 12 days after manufacture for a compression rate of 6 (and 4, respectively)

<table>
<thead>
<tr>
<th>Sample no</th>
<th>20</th>
<th>21</th>
<th>22</th>
<th>23</th>
<th>24</th>
<th>25</th>
<th>26</th>
<th>27</th>
<th>28</th>
<th>29</th>
<th>30</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness</td>
<td>135.8 (140.7)</td>
<td>110.7 (124.6)</td>
<td>113.3 (122.7)</td>
<td>105.3 (106.6)</td>
<td>137.0 (133.6)</td>
<td>143.7 (132.5)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
The mechanical strength of the products was then tested immediately after preparation of the samples, after 24 hours then 12 days later, lastly we also carried out an artificial ageing test in the autoclave (TABLE V).

TABLE V

<table>
<thead>
<tr>
<th>No</th>
<th>Preparation</th>
<th>24 Hours</th>
<th>12 Days</th>
<th>Autoclave</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>292</td>
<td>257</td>
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<tr>
<td>30</td>
<td>264</td>
<td></td>
<td>247</td>
<td>232</td>
</tr>
</tbody>
</table>

The products according to the invention have, after preparation, a tensile strength close to that of standard products, however they age slightly better.

These tests demonstrate the possibility of realizing glass fibre products intended in particular for thermal insulation and/or soundproofing of buildings, particularly light products, replacing standard size with the size according to the invention, and this without modification of the glass fibre production line operating parameters, except as regards the temperature of the oven whose setting point is reduced by about 30 to 50°C, which saves energy.
Three additional tests have been carried out for the purpose of verifying the feasibility of a size containing a hardener of type F (dicyandiamide) with which an accelerator of the type of 2,4,6-tri(dimethylaminomethyl)-phenol is combined. Such a product is marketed under the Trade Mark DMP-30 by the registered French Company of ROHM and HAAS FRANCE.

These three tests have been on the industrial production line described previously (drawing of 20 tonnes of glass per day, without overspray). The following are the compositions of the sizes which have been utilised:

**SAMPLE No. 31**

- Formulation of the size:
  - sample resin of type A : 100 parts
  - silane : 0.3 part
  - mineral oil : 9.5 parts
  - ammonia solution : 6 parts
  - ammonium sulphate : 3 parts

**SAMPLE No. 32**

- Formulation of the size:
  - resin B : 72 parts
  - hardener E : 28 parts
  - silane : 0.5 part
  - mineral oil : 12 parts

**SAMPLE No. 33**

- Formulation of the size:
  - resin B : 88 parts
  - hardener F : 12 parts
  - Accelerator : 1.2 parts
  - silane : 0.5 part
  - mineral oil : 12 parts

The characteristics of the products obtained are presented in the following TABLE VI:

<table>
<thead>
<tr>
<th>Test</th>
<th>Microns</th>
<th>Grammage</th>
<th>Density</th>
<th>Thickness</th>
</tr>
</thead>
<tbody>
<tr>
<td>31</td>
<td>3.35</td>
<td>896</td>
<td>11.2</td>
<td>125.6</td>
</tr>
<tr>
<td>32</td>
<td>3.50</td>
<td>899</td>
<td>11.2</td>
<td>128.1</td>
</tr>
<tr>
<td>33</td>
<td>3.60</td>
<td>927</td>
<td>11.6</td>
<td>158.5</td>
</tr>
</tbody>
</table>
The hardener based on DCN gave complete satisfaction. Actually, the recovery of thickness is equal to that observed for the standard product after fabrication and is even better after ageing. The tensile strength is, on the other hand, slightly less after ageing but, all the same, it remains at a high value. Furthermore, we have noted from a comparison of tests 32 and 33 that the hardeners E and F give very similar results, but at only half the cost of manufacture for the size containing the hardener F, so that this incontestably yields the preferred product of the present invention.
THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. Size for so-called insulating glass fibres based on epoxy resin of glycidylic ether type dispersable in water, of an amino setting agent with a flash point over 180°C and additives (in parts calculated per 100 parts of dry resin), namely 0.1 to 2% of silane and 0 to 15% of a mineral oil.

2. Size according to claim 1, characterized in that the epoxy resin is a glycidylic ether of polymerisation index in under 1 and preferably under 0.2.

3. Size according to claim 2, characterized in that the polymerisation index n is strictly over 0.

4. Size according to one of the claims 1 to 3, characterized in that the NH equivalent molar mass of the setting agent is less than 100 g.

5. Size according to one of claims 1 to 4, characterized in that the setting agent is added to the epoxy resin at a ratio virtually identical to the stoechiometric ratio.

6. Size according to one of the claims 1 to 5, characterized in that the resin/setting agent couple presents a gelling time exceeding 25 minutes.

7. Size according to one of the claims 1 to 6 characterized in that the setting agent is based on dicyane diamide.

8. Treatment process of so-called insulating glass fibres obtained according to aerodynamic processes with drawing out by gas currents at high temperature, characterized in that a size according to one of claims 1 to 7, diluted in water, is sprayed onto the fibres in the fibre-making and collection hood of the said glass fibres.

9. Application of the process according to claim 8 for the obtention of insulating products whose density is between 4 to 30 kg/m² inclusive.

DATED THIS 26TH DAY OF OCTOBER 1989
ISOVER SAINT-GOBAIN
By its Patent Attorneys:
GRiffith HACK & CO.
Fellows Institute of Patent
Attorneys of Australia