

(12) PATENT
(19) AUSTRALIAN PATENT OFFICE

(11) Application No. AU 199710009 B2
(10) Patent No. 702549

(54) Title
Cementitious gypsum-containing compositions and materials made therefrom

(51)⁶ International Patent Classification(s)
C04B 011/30 C04B 014/04

(21) Application No: 199710009 (22) Application Date: 1997 .01 .03

(30) Priority Data

(31) Number	(32) Date	(33) Country
713727	1996 .09 .13	US
710264	1996 .09 .13	US

(43) Publication Date : 1997 .03 .13
(43) Publication Journal Date : 1997 .03 .13
(44) Accepted Journal Date : 1999 .02 .25

(62) Divisional of:
199526019

(71) Applicant(s)
National Gypsum Company

(72) Inventor(s)
Elisha Stav; Edward A. Burkard; Ronald S. Finkelstein; Daniel A. Winkowski ; Lambert J. Metz; Patrick J. Mudd

(74) Agent/Attorney
SPRUSON FERGUSON,GPO Box 3898,SYDNEY NSW 2001

(56) Related Art
US 4494990
US 4661159



AU9710009

(12) PATENT ABSTRACT (11) Document No. AU-A-10009/97
(19) AUSTRALIAN PATENT OFFICE

(54) Title
CEMENTITIOUS GYPSUM-CONTAINING COMPOSITIONS AND MATERIALS MADE THEREFROM

International Patent Classification(s)

(51)⁶ **C04B 011/30 C04B 014/04**

(21) Application No. : **10009/97**

(22) Application Date : **03.01.97**

(30) Priority Data

(31) Number	(32) Date	(33) Country
713727	13.09.96	US UNITED STATES OF AMERICA

(43) Publication Date : **13.03.97**

(62) Related to Division(s) : **26019/95**

(71) Applicant(s)
NATIONAL GYPSUM COMPANY

(72) Inventor(s)
**ELISHA STAV; EDWARD A. BURKARD; RONALD S. FINKELSTEIN; DANIEL A. WINKOWSKI;
LAMBERT J. METZ; PATRICK J. MUDD**

(74) Attorney or Agent
SPRUSON & FERGUSON , GPO Box 3898, SYDNEY NSW 2001

(57) Claim

1. A cementitious composition comprising:

- (a) about 20wt% to about 75wt% calcium sulphate beta-hemihydrate;
- (b) about 10wt% to about 50wt% Portland cement or a blend of Portland cement with fly ash and/or ground blast slag;
- (c) about 4wt% to about 20wt% silica fume.

Cementitious Gypsum-Containing Compositions And Materials Made Therefrom

Abstract

A cementitious composition useful for water-resistant construction materials includes about 20wt% to about 75wt% calcium sulphate beta-hemihydrate, about 10wt% to about 50wt% Portland cement, and about 4wt% to about 20wt% silica fume. The Portland cement component may comprise a blend of Portland cement with fly ash and/or ground blast slag. The cementitious composition is useful as a binder in fibre-containing compositions comprising about 10wt% to about 35wt% fibre and about 65wt% to about 90wt% binder. Optionally cementitious compositions of the invention may further comprise about 1wt% to about 50wt% pozzolanic aggregate as a filler

5
10

S & F Ref: 356706D1

AUSTRALIA
PATENTS ACT 1990

COMPLETE SPECIFICATION

FOR A STANDARD PATENT

ORIGINAL

Name and Address
of Applicant: National Gypsum Company
2001 Rexford Road
Charlotte North Carolina 28211-3498
UNITED STATES OF AMERICA

Actual Inventor(s): Elisha Stav, Edward A. Burkard, Ronald S. Finkelstein,
Daniel A. Winkowski, Lambert J. Metz and Patrick J. Mudd

Address for Service: Spruson & Ferguson, Patent Attorneys
Level 33 St Martins Tower, 31 Market Street
Sydney, New South Wales, 2000, Australia

Invention Title: Cementitious Gypsum-containing Compositions and
Materials Made Therefrom

The following statement is a full description of this invention, including the best method of performing it known to me/us:-

Cementitious Gypsum-Containing Compositions And Materials Made Therefrom

Background of the Invention

Field of the Invention

5 The invention relates to cementitious compositions, particularly to cementitious construction materials such as floor underlayments, backer boards, floor and road patching materials, fibreboard, roofing tiles, shingles, exterior sheathing, fire-proofing sprays, and fire-stopping materials made from a composition comprising gypsum, Portland cement and silica fume, more particularly to cementitious construction materials
10 containing fibre made from a composition comprising gypsum, Portland cement, silica fume, and fibre.

Description of Related Technology

Construction materials, such as backer boards for showers, floor underlayments, and exterior applications such as sheathing and shingles, typically do not contain gypsum
15 because gypsum-containing materials are usually not water resistant. However, gypsum is a desirable component in construction materials due to its rapid cure and early strength characteristics. Attempts to improve the water-resistance of gypsum boards by mixing Portland cement and gypsum (calcium sulphate hemihydrate) have met with limited success because such a mixture can result in the formation of ettringite, which may cause
20 expansion of the gypsum/Portland cement product and thus lead to its deterioration. Ettringites are formed when tricalcium aluminate ($3\text{CaO}\cdot\text{Al}_2\text{O}_3$) in the Portland cement reacts with sulphate.

A cementitious composition useful as a pavement patching compound which contains Portland cement and alpha gypsum is disclosed in Harris, US 4 494 990. The
25 composition also includes a pozzolan source, such as, for example, silica fume, fly ash or blast furnace slag. The Harris patent discloses that the pozzolan blocks the interaction between the tricalcium aluminate and the sulphate from gypsum. The Harris patent discloses mixing a three-component blend of Type I Portland cement, alpha gypsum and silica fume with a fine aggregate to prepare a mortar used to cast mortar cubes for
30 evaluating the strength of the resulting composition.

Ortega et al, US 4 661 159 discloses a floor underlayment composition that includes alpha gypsum, beta gypsum, fly ash and Portland cement. The patent also discloses that the floor underlayment material can be used with water and sand or other aggregate to produce a fluid mixture which may be applied to a substrate. Neither the Harris patent
35 nor the Ortega et al. patent discloses fibre - containing compositions.

Sattler et al., US Patent No. 5,030,289 discloses a first group of moulded construction parts made from waste paper or cellulose fibres and a binder made from (1) Portland cements, alumina cements, belite cements, or mixtures thereof; and (2) a



pozzolan such as amorphous silicic acid, powdered trass, fly ash, or mixtures thereof. Sattler et al. also discloses a second group of moulded construction parts made from fibre and a binder of (1) a latently hydraulic component such as blast sand or blast slag; (2) hemihydrate gypsum; and (3) Portland cement. However, the Sattler et al. patent does not disclose combining gypsum with the cement/pozzolan-containing mixtures used to make the first group of moulded construction parts.

Summary of the Invention

It is an object of the invention to overcome one or more of the problems described above.

10 It is another object of the invention to provide a cementitious composition comprising about 20wt% to about 75wt% calcium sulphate beta-hemihydrate, about 10wt% to about 50wt% Portland cement, and about 4wt% to about 20wt% silica fume. It is another object of the invention to provide a cementitious composition further comprising about 1wt% to about 50wt% pozzolanic aggregate as a filler.

15 It is a further object of the invention to provide a fibre-containing cementitious composition comprising about 10wt% to about 35wt% fibre and about 65wt% to about 90wt% of either of the above cementitious compositions as a binder. The Portland cement component of any of the compositions according to the invention may also be a blend of Portland cement with fly ash and/or ground blast slag.

20 It is a further object of the invention to provide construction compositions, materials made from the inventive cementitious compositions and methods for making the same. Other objects and advantages of the invention will be apparent to those skilled in the art from the following detailed description taken in conjunction with the appended claims.

Detailed Description of the Invention

25 Throughout the specification, the expression "Portland cement" includes blends of Portland cement with one or more pozzolanic materials such as fly ash and ground blast-furnace slag.

According to the invention, a composition for use in construction materials is provided which is particularly useful in areas where water resistance is an important 30 consideration for both interior and exterior applications, such as for backer boards for baths and showers, floor underlay applications, fibre board, sheeting, roofing tile, shingles, and exterior sheathing boards. Further uses of the inventive composition include materials such as self-leveling floors and road patching materials, fireproofing sprays, fire-stopping materials, and eaves.

35 According to a first embodiment of the invention there is provided a cementitious composition comprising:

- (a) about 20wt% to about 75wt% calcium sulphate beta-hemihydrate;



(b) about 10wt% to about 50wt% of a cement selected from the group consisting of Portland cement, a blend of Portland cement and fly ash, a blend of Portland cement and ground blast slag; and mixtures thereof;

(c) about 4wt% to about 20wt% silica fume; and

5 (d) about 1wt% to about 50wt% pozzolanic aggregate.

According to a second embodiment of the invention there is provided a self-leveling floor composition comprising:

(i) about 25wt% to about 75wt% of the composition of the first embodiment; and

10 (ii) about 75wt% to about 25wt% sand.

According to a third embodiment of the invention there is provided a road patching composition comprising:

(i) about 25wt% to about 100wt% of the composition of the first embodiment; and

15 (ii) about 75wt% to about 0wt% sand.

According to a fourth embodiment of the invention there is provided a water resistant construction material prepared by combining a cementitious composition with a slight stoichiometric excess of water, said cementitious composition comprising:

(a) about 20wt% to about 75wt% calcium sulfate beta-hemihydrate;

20 (b) about 10wt% to about 50wt% of a cement selected from the group consisting of Portland cement, a blend of Portland cement and fly ash, a blend of Portland cement and ground blast slag; and mixtures thereof;

(c) about 4wt% to about 20wt% silica fume; and

(d) about 1wt% to about 50wt% pozzolanic aggregate.

25 According to a fifth embodiment of the invention there is provided a water resistant construction material having a thickness of about $\frac{1}{8}$ inch, said material prepared by combining a cementitious composition with a slight stoichiometric excess of water, said cementitious composition comprising:

(a) about 20wt% to about 75wt% calcium sulfate beta-hemihydrate;

30 (b) about 10wt% to about 50wt% of a cement selected from the group consisting of Portland cement, a blend of Portland cement and fly ash, a blend of Portland cement and ground blast slag; and mixtures thereof;

(c) about 4wt% to about 20wt% silica fume; and

(d) about 1wt% to about 50wt% pozzolanic aggregate.

35 According to a sixth embodiment of the invention there is provided a cementitious composition comprising:

(a) about 30wt% to about 75wt% calcium sulfate beta-hemihydrate;

(b) about 10wt% to about 40wt% Portland cement;

(c) about 5wt% to about 20wt% silica fume; and



- (d) about 10wt% to about 40wt% pozzolanic aggregate.

According to a seventh embodiment of the invention there is provided a cementitious composition comprising:

- 5 (a) about 30wt% to about 75wt% calcium sulfate beta-hemihydrate;
(b) about 10wt% to about 40wt% Portland cement;
(c) about 4wt% to about 20wt% silica fume; and
(d) about 1wt% to about 40wt% pozzolanic aggregate.

The beta-gypsum component of the inventive composition is calcium sulphate beta hemihydrate, commonly referred to as stucco. Beta-gypsum is traditionally less expensive
10 than alpha-gypsum and is typically not as strong because it comprises very small crystals of hemihydrate held together in porous conglomerates, while alpha-gypsum comprises well crystallised prisms of hemihydrate. Alpha-hemihydrate powder has a higher
15 apparent density and smaller related surface area than beta-hemihydrate, resulting in a lower water requirement for the same workability and a higher compressive strength of the set material. However, boards made from the inventive composition exhibit more than adequate strength for interior applications such as backer boards and floor underlayments and exterior applications, such as exterior sheeting, roof tiles, shingles and eaves.

Typically, the Portland cement component of the composition according to the
20 invention may be any of Types I, II, III, IV, or V (or mixtures thereof) as set forth according to ASTM standards. However, Type III Portland cement is preferred. Type III Portland cement cures faster than Type I and Type II Portland cement and exhibits an early high strength.

Blended cements may advantageously be used in compositions according to the
25 invention. Blended cements are blends of Portland cement with one or more pozzolanic materials such as fly ash and blast-furnace slag. The pozzolanic materials that are added to produce a "blend" with Portland cement are distinguished from the pozzolanic aggregate filler component according to the invention of the application in that the components of the cement "blend" typically have a particle size which is in the same
30 range as the particle size range of Portland cement. Portland cement particle size may be defined as having approximately 15% of the particles retained on a 325 mesh screen. In other words, at least 85% of the Portland cement particles pass through a 325 mesh



screen (allows particles having a diameter of up to 45 microns to pass through). Thus, for example, blast furnace slag and certain fly ash must be ground prior to mixing with Portland cement to result in a "blend" for use in the invention.

The silica fume component of compositions according to the invention is an extremely active pozzolan and prevents the formation of ettringite. Silica fume is typically very fine (particle average diameter of between about 0.1 microns and about 0.3 microns), has a high surface area (between about 20 meter²/gram and about 30 meter²/gram), and is highly amorphous (between about 98wt% and about 100wt% amorphous SiO₂ (glassy material)).

The pozzolanic aggregate filler component optionally included in compositions according to the invention is typically a natural or man-made aggregate that contains a high percentage of amorphous silica. Preferred natural pozzolanic aggregates are of volcanic origin and include trass, pumice, and perlite. Preferred man-made pozzolanic aggregate fillers include fly ash and FILLITE (hollow silicate spheres which may be made from fly ash; produced by Fillite Division of Boliden Intertrade, Inc. Atlanta, Georgia). As compared to cement "blend" components of the invention, pozzolanic aggregates used as fillers according to the invention are defined herein as having an average particle size larger than that of Portland cement (i.e., average particle size larger than 45 microns).

Pozzolanic aggregate fillers contain a high percentage of amorphous silica which possesses little or no cementitious properties. However, in the presence of moisture, pozzolanic aggregates have surfaces that are chemically reactive with calcium hydroxide at standard temperatures to form hydrated calcium silicate (CSH) which, in compositions and methods according to the invention, are believed to become a homogeneous part of a cementitious system due also to the presence of the finely divided pozzolan of the invention, silica fume. Compositions according to the invention which include both a pozzolanic aggregate and a finely divided pozzolan result in cementitious materials wherein the transition zone between the aggregate and a cement paste is densified and thus produces a cured product of higher compressive strength than compositions which utilise a pozzolanic aggregate alone or a finely divided pozzolan alone. It is believed that the mechanism which causes changes in the microstructure of compositions according to the invention to result in higher compressive strengths is associated with two effects: a pozzolanic effect and a micro-filler effect (due to the fine size and spherical shape of the silica fume).

Compositions for construction materials such as backer boards and floor underlays according to the invention preferably include about 20wt% to about 75wt% calcium sulphate beta-hemihydrate (about 30wt% to about 50wt% is preferred), about 10wt% to about 50wt% Portland cement (about 6wt% to about 35wt% is preferred), about 4wt% to about 20wt% silica fume (about 4wt% to about 10wt% is preferred), and preferably further comprise about 10wt% to about 50wt% pozzolanic aggregate filler (about 25wt% to about 35wt% is preferred). A preferred aggregate filler for use in such construction

materials is pumice. Pumice is desirable as it is relatively light weight and can be sized to result in a product of desirable strength and physical properties. For example, Hess Pumice Products Inc. manufactures a size No. 10 pumice aggregate that measures about 93% greater than 1400 microns, while the size No. 5 pumice aggregate has a particle size measurement of about 23% greater than 1400 microns.

Although fillers such as calcium carbonate, crystalline silica and different types of clay could be included in the composition, it has been found that the use of a pozzolanic aggregate filler results in a product according to the invention having superior properties. As explained above, this is believed to occur because the surfaces of the pozzolanic aggregate filler react with free lime to form hydrated calcium silicate (pozzolanic reaction) which becomes part of the product matrix. Such a reaction is only possible with pozzolanic aggregate fillers.

Fibre-containing compositions according to the invention typically comprise a binder comprising about 20wt% to about 75wt% calcium sulphate beta-hemihydrate (ie., beta-gypsum), about 10wt% to about 50wt% Portland cement, and about 4wt% to about 20wt% silica fume. Optionally, the binder may further comprise about 1wt% to about 40wt% pozzolanic aggregate. About 65wt% to about 90wt% of the binder is then mixed with about 10wt% to about 35wt% of a fibre component to result in a fibre-containing composition according to the invention.

The fibre component of a fibre-containing composition according to the invention is preferably selected from wood and paper fibres, including recycled waste paper fibres, other ligneous materials such as flax and cotton, and mixtures of such fibres. Wood fibre is a preferred fibre component for a composition according to the invention.

Most preferably, the fibre is obtained from debarked wood which is refined to long thin flakes having a thickness of about 0.008 inches (about 0.2 mm) to about 0.013 inches (about 0.33 mm) and a length of up to about 1.18 inches (about 30 mm). The flaked wood is then milled and screened and possibly further refined using known processes in order to provide fibres or fibre flakes of substantially constant geometry.

If the wood fibre material used in a composition according to the invention is waste paper, such paper must first be processed to remove foreign material such as plastic, dirt and metals. The paper is then further processed by shredding, preferably with a heavy hammermill. The shredded paper is then preferably dry-refined to result in fibres of substantially constant geometry.

The binder for fibre-containing compositions according to the invention preferably includes about 20wt% to about 75wt% calcium sulphate beta-hemihydrate (about 36wt% to about 47wt% is preferred for fibreboard), about 10wt% to about 50wt% Portland cement (about 40wt% to about 50wt% is preferred), and about 4wt% to about 20wt% silica fume (about 10wt% to about 15wt% is preferred for fibreboard).

About 65wt% to about 90wt% (preferably about 70wt% to about 85wt%) of the binder is then mixed with about 10wt% to about 35wt% wood fibre (preferably about

15wt% to about 30wt% wood fibre) to form a fibreboard. Most preferably, a binder according to the invention for use in a fibreboard includes about 40wt% calcium sulphate beta-hemihydrate, about 46wt% Portland cement, and about 14wt% silica fume.

Compositions according to the invention advantageously produce building materials which set up quickly, exhibit high strength and durability, and are water resistant. Gypsum boards produced from compositions according to the invention may be produced on a continuous line. Because the composition according to the invention sets up quickly (typically in three minutes or less), building materials made from the composition can be handled (eg. sheets can be cut into smaller sheets or boards) much faster than products made from Portland cement alone. Unlike traditional gypsum board, boards or other products made from a composition according to the invention do not require kiln drying, and in fact, kiln drying should be avoided. For example, a benefit of a composition and process according to the invention is that a fibreboard made from the inventive process undergoes a total pressing/curing time of under seven hours (and as little as three hours) as compared to the seven to twelve-hour pressing/curing time required for some other Portland cement/fibre board processes.

Brief Description of the Drawings

Fig. 1 is a cross-sectional view of a covered board according to the invention.

Fig. 2 is a graph depicting compressive strength vs. curing time for a composition #1 according to the invention and a comparative composition #2.

Fig. 3 is a scanning electron microscope (SEM) micrograph (500x) of a board made from a composition according to the invention disclosed in Example 3.

Fig. 4 is an SEM micrograph (100x) of the board shown in Fig. 3.

Fig. 5 is an SEM micrograph (1000x) of the board shown in Fig. 3.

Best Mode and other modes for carrying out the invention

A fibre-containing construction material, such as a fibreboard according to the invention may be manufactured by the following process:

Raw gypsum may be calcined at about 160°C to about 175°C to form calcium sulphate hemihydrate. The calcined gypsum can be post-ground to a finer particle size if, for example, certain strengths, water requirements, and working properties are desired.

All components of the composition, including gypsum, cement, silica fume, water, wood fibre, and any other additives preferably are weighted batch-wise. Moisture in the wood fibre also is measured.

The gypsum powder is fed to a mixer, such as a large batch or continuous mixer, and blended with Portland cement and silica fume.

In a second mixer, the fibre is mixed with water to allow the fibre/water mixture to loosen. The gypsum/cement/silica fume binder is then added to the fibre/water mixture

and intensively mixed with the humid fibre. Although water may be added to the binder/fibre mixture (or to the binder prior to mixing with the fibre), preferably, the water is added to the fibre and then the binder is added to the water/fibre mixture.

Most preferably, the water addition to the fibre and the subsequent binder addition to the wetted fibre are performed with the aid of computer control so that it is possible to add to the fibre the total quantity of water required for the process (ie. a slight stoichiometric excess amount of water required for hydration), and then vigorously mix the wetted fibre with the binder.

Other ingredients, such as set control additives (eg. accelerators), water reducing agents, water repellent additives, retarders, and latex or polymer modifiers may be added to the fibre/binder mixture. Some additives may be added to the dry binder mixture prior to mixture with the wet fibre. Preferably, the composition includes about 0.01wt% to about 1.5wt% retarder, based upon the total weight of the composition.

The mixed composition is then conveyed directly to a forming machine which spreads an endless mat onto an elongated belt of a continuous press. The mat enters the press on the conveyor belt, is pressed and may be cut into sections, and exits on a conveyor belt in the form of an endless board-ribbon or panel sections. A pressing machine which can be used for this purpose is the Bison-Hydro-Dyn-Press (Bison GmbH, Springe, Germany). In such a press, the hydration of the board occurs quickly and may be hastened by warming the board in the press up to an optimal hydration temperature. Preferred processing conditions include pressing at room temperature (about 25°C) at pressures up to about thirty (30) kg/cm² for a press or clamping time of about three (3) to about eight (8) hours.

The board-ribbon (or panel sections) leaving the press has sufficient green strength so that it can be transferred onto a conveyor which will carry the board forward to a cutting station. Hydration may continue as the board-ribbon or panel sections are conveyed to the board cutter. The board-ribbon is then cut or sawed to a desired panel length. If necessary, the panels are then dried to a final moisture content.

Finally the board panels are trimmed and, if desired, split lengthwise to a final dimension. Boards are typically cut into 3 ft. (0.9 meter) x 5 ft. (1.5 meter) sheets, and have a thickness between about 1/2 inch (about 1.3 cm) and about 5/8 inch (about 1.6 cm).

With reference to Figure 1, a backer board according to the invention comprises a core made from a cementitious composition according to the invention and adjacent cover sheets disposed at either side thereof. Such a board may be manufactured by the following process:

Raw gypsum may be calcined at about 160°C to about 175°C to form calcium sulphate hemihydrate. The calcined gypsum can be post-ground to a finer particle size if, for example, certain strengths, water requirements, and working properties are desired. The gypsum powder is fed to a mixer and blended with Portland cement, silica fume, and

optionally a pozzolanic aggregate filler. The pozzolanic filler may be pumice, perlite, trass, or fly ash or a mixture thereof. Other ingredients that may be included in the composition are set control additives (eg. accelerators), water reducing agents, water repellent additives and latex or polymer modifiers. The resulting blend is combined with
5 a slight stoichiometric excess of water to produce a slurry. The slurry, which forms the core of the board, is poured onto a lower, continuous cover sheet which is disposed on a conveyor. Then, an upper continuous cover sheet is placed on the core as it moves on the conveyor. The cover sheets are preferably made from fibreglass matt, fibreglass scrim, or a composite of both. The cover sheets may also be made from polyethylene,
10 polypropylene or nylon; however, such materials are not as desirable as fibreglass as they are more expensive. As the slurry sets, dihydrate needles form and interlock with the cover sheets. As the covered board moves along the conveyor line in a continuous sheet, the board gains sufficient strength so that it can be handled. The board is then cut into sections, (for backer boards, usually either 3 ft. x 5 ft. or 3 ft. x 4 ft. sheets) and
15 transferred to pallets. The board thickness preferably ranges between about 1/8 inch and about 5/8 inch. The boards are then preferably stacked and cured from one to seven days (particularly preferred about three days) at a temperature of about 16°C to about 27°C (ie. room temperature) and a humidity of about 40% to about 70%, after which the boards may be sent to a customer. The stacking of the boards advantageously provides a moist
20 environment for curing. The boards may be cured at temperatures and humidities outside of the above-stated ranges resulting in an acceptable product. However, this may extend the curing time. A board according to the invention usually reaches its full strength about fourteen to about twenty-eight days after formation.

When preparing a board or other product according to the invention, the forced
25 drying required for gypsum board should be avoided. An alternative curing procedure is to cover or wrap the boards in plastic wrapping for about three days to retain moisture for continuous curing. Such covered or wrapped boards have exhibited about 50% higher strength than normal gypsum boards of the same density. Also, the wrapped boards develop about 70% to about 80% of their ultimate strength in three days.

30 When a board or other product having a thickness of about 1/8 inch is desired, the cementitious composition thereof preferably includes about 20wt% to about 75wt% calcium sulphate beta-hemihydrate, about 10wt% to about 50wt% Portland cement, about 4wt% to about 20wt% silica fume, and optionally about 1wt% to about 50wt% pozzolanic aggregate filler, resulting in a very strong thin product, especially useful, for example, for
35 floor underlayments. A preferred cementitious composition for use in very thin boards (ie. about 1/8 inch) and floor underlayments includes about 70wt% to about 75wt% calcium sulphate beta hemihydrate (about 74wt% is particularly preferred), about 15wt% to about 40wt% Portland cement (about 35wt% is particularly preferred), about 4wt% to about 10wt% silica fume (about 6wt% is particularly preferred), and optionally about
40 1wt% to about 25wt% pozzolanic aggregate filler.

Compositions according to the invention may also be used to prepare self - leveling floor compositions and road patching materials. In such materials, a master blend composition according to the invention is prepared which includes about 20wt% to about 75wt% calcium sulphate beta-hemihydrate (ie. beta-gypsum) (about 30wt% to about 50wt% is preferred), about 10wt% to about 50wt% Portland cement (about 6wt% to about 25wt% is preferred), about 4wt% to about 20wt% silica fume (about 4wt% to about 8wt% is preferred), and optionally about 1wt% to about 50wt% pozzolanic aggregate filler (about 1wt% to about 15wt% is preferred; about 1wt% to about 5wt% particularly preferred). The master blend is then mixed with silica aggregates (ie., predominantly quartz local sand) to form the floor or road patching material.

Preferably, a self-leveling floor composition according to the invention includes (i) about 25wt% to about 75wt% of the master blend; and (ii) about 75wt% to about 25wt% sand. Most preferably, a self-leveling floor composition master blend includes about 71wt% calcium sulphate beta-hemihydrate, about 20wt% Portland cement, about 6wt% silica fume and about 2wt% FILLITE pozzolanic filler. Because of its low density, FILLITE addition of amounts as low as about 1wt% of the composition provide a considerable volume of filler (see Example 2, Table II for FILLITE physical properties).

A road patching composition according to the invention includes (i) about 25wt% to about 100wt% of the master blend described herein with respect to the self-leveling floor compositions of the invention; and (ii) about 75wt% to about 0wt% sand.

The master blend described herein may also be used in fibreboards according to the invention. Such fibreboards include (i) about 65wt% to about 90wt% of the master blend described herein with respect to the self-leveling floor compositions and road patching compositions of the invention; and (ii) about 35wt% to about 10wt% of a fibre component. The fibre component is preferably selected from the following: wood fibres, paper fibres, glass fibres, polyethylene fibres, polypropylene fibres, nylon fibres, and other plastic fibres.

Most preferably, a master blend according to the invention for use in such a fibreboard includes about 74wt% calcium sulphate beta-hemihydrate, about 20wt% Portland cement, and about 6wt% silica fume.

Fire-proofing sprays and fire-stopping materials may also be prepared utilising compositions according to the invention. Such fire-proofing and fire-stopping materials include about 20wt% to about 75wt% calcium sulphate beta-hemihydrate (about 30wt% to about 50wt% is preferred), about 10wt% to about 50wt% Portland cement (about 6wt% to about 25wt% is preferred), about 4wt% to about 20wt% silica fume (about 4wt% to about 10wt% is preferred), and optionally about 1wt% to about 50wt% pozzolanic aggregate filler (about 1wt% to about 10wt% is preferred). Preferably, the pozzolanic filler is FILLITE or perlite or mixtures thereof. Fire-proofing sprays and fire-stopping materials according to the invention also preferably include about 1wt% to about 30wt% unexpanded vermiculite filler. Such fire-proofing and fire-stopping materials may also

include up to about 2wt% glass fibres and up to about 2wt% of a thickening agent. The thickening agent is preferably selected from the following: cellulose derivatives, acrylic resins and mixtures thereof.

Example 1

5 A cementitious composition according to the invention was prepared with components set forth in the amounts stated in Table I below:

Table I

Material	Weight Percent
Beta-gypsum (Stucco)	45.1
Type III Portland Cement	19.2
Silica Fume	9.5
Pumice Filler	24.6
Perlite	1.47
W.R.A. ¹	0.87
Water Repellent Agent ²	0.11
Accelerator ³	0.042

10 ¹Water reducing agent or wetting agent including lignosulphonates and/or naphthalene sulphonates manufactured by Georgia Pacific Corp. and Henkel Corp., respectively.

²A silicone product or like material, eg., Veoceal 2100 and Veoceal 1311 (both trade mark designations of products manufactured by Wacker Silicone Corp.)

15 ³Ball milled CaSO₄ · 2H₂O gypsum dihydrate. See US Patent No.s 3 920 465, 3 870 538 and 4 019 920

20 The materials identified in Table I were mixed and 100 grams thereof was mixed with 35.6 grams of water. About 1wt% to about 5wt% of a polymer latex (acrylic or SBR) was added to the mixture to improve flexibility. The mixture was then formed into boards according to the invention using a glass matt/scrim composite. The boards were tested for water absorption, nail holding properties, deflection, compression strength (wet and dry), water wicking characteristics and other ASTM specification requirements. The boards met the ASTM specifications with respect to each test.

Example 2

25 A self-leveling floor composition #1 according to the invention was prepared with the components set forth in the amounts stated in Table II below. A cementitious composition #2 with components also set forth in the amounts stated in Table II below was also prepared.

Table II

Material	Composition #1 (weight percent)	Composition #2 (weight percent)
Beta-Gypsum (Stucco)	36.1	40.0
Type III Portland Cement	9.8	10.8
Silica Fume	2.96	3.24
FILLITE 500 Pozzolanic Filler ¹	0.0	1.35
Sand (quartz; crystallised silica)	49.4	43.26
W.R.A. ²	0.82	0.9
Retarder ³	0.06	0.06
Anti-foaming agent ⁴	0.33	0.26

¹Fillite Division of Boliden Intertrade, Inc., Atlanta Georgia. Hollow silicate spheres with the following physical properties: average particle density of 0.6-0.8 g/cc; average bulk density of 0.35-0.45 g/cc; and typical particle size of 5-300 microns. The shell composition includes 27wt% to 33wt% Al₂O₃, 55wt% to 65wt% SiO₂, and a maximum of 4wt% Fe₂O₃.

²Water reducing agent or wetting agent including lignosulphonates and/or naphthalene sulphonates manufactured by Georgia Pacific Corp. and Henkel Corp., respectively.

³A natural protein-based material.

⁴A vegetable oil-based dry powder.

In order to form a floor composition of a smooth consistency, composition #1 was mixed with about 26wt% water and composition #2 was mixed with about 24wt% water. The density of composition #1 was 107 lbs./ft³. The density of composition #2 was 111.62 lbs./ft³.

Both compositions were allowed to dry at about 21°C and a relative humidity of about 50%. The compressive strengths of samples (2 inch by 2 inch by 2 inch cubes) of each of the compositions were tested after 2 hours of drying, and after 1, 3, 7 and 28 days by pressing in an Instron press according to ASTM C472-9A.

The results of the compressive strength tests are shown in Fig. 2. Composition #1 exhibited a greater compressive strength than Composition #2 for all samples tested. Although the compressive strengths of both compositions were similar after curing for 28 days, the advantage of Composition #1 is evident when the densities of the two compositions are taken into consideration. Typically, a composition having a higher density should also exhibit a higher compressive strength. However, in this instance, Composition #1 had a lower density than Composition #2, and yet exhibited a slightly higher compressive strength.

Example 3

A cementitious composition according to the invention was prepared with components set forth in the amounts stated in Table III below:

Table III

Material	Weight Percent
Beta-gypsum (Stucco)	35.9
Type III Portland Cement	15.6
Silica Fume	7.8
Pumice Filler	39.5
W.R.A. ¹	0.87
Water Repellent Agent ²	0.11
Accelerator ³	0.058

¹Water reducing agent or wetting agent including lignosulphonates and/or naphthalene sulphonates manufactured by Georgia Pacific Corp. and Henkel Corp., respectively.

²A silicone product or like material, eg., Veoceal 2100 and Veoceal 1311 (both trade mark designations of products manufactured by Wacker Silicone Corp.)

³Ball-milled CaSO₄ · 2H₂O gypsum dihydrate. See US 3 920 465, 3 870 538 and 4 019 920.

10 The materials identified in Table III were mixed and 100 grams thereof was mixed with 35.6 grams of water. About 1wt% to about 5wt% of a polymer latex (acrylic or SBR) was added to the mixture to improve flexibility. The mixture was then formed into boards according to the invention using a glass matt/scrim composite. The boards were tested for water absorption, nail holding properties, deflection, compression strength (wet
15 and dry), water wicking characteristics and other ASTM specification requirements. The boards met the ASTM specifications with respect to each test.

The scanning electron microscope (SEM) micrographs shown in Figs. 3, 4, and 5 were made of a cured sample of Example 3. An arrow 30 points to pumice in the sample, illustrating that in a composition according to the invention, the pumice becomes part of
20 the hydrated calcium silicate (CSH) matrix, substantially eliminating any transition zone 32 between the pumice filler and the cement paste.

Example 4

A cementitious master blend binder according to the invention was prepared with the components set forth in the amounts stated in Table IV below:

25

Table IV

Material	Approx. Weight Percent
Beta-gypsum (Stucco)	40
Type III Portland Cement	46
Silica Fume	14
Accelerator ¹	0.35

¹BMA (board milling accelerator, a fine-ground gypsum produced by National Gypsum Company).

The materials identified in Table IV were mixed to form the master blend binder. Then, about 75wt% of the binder was mixed with about 25wt% pumice aggregate (Hess Products, Inc., Malard City, Idaho) and 100 grams thereof was mixed with 43 grams of water. To improve the workability of the mixture, a water reducing agent (lignosulphonates and/or naphthalene sulphonates manufactured by Georgia Pacific Corp. and Henkel Corp., respectively) was added. The mixture was then formed into two-inch by two-inch (2" x 2") cubes to evaluate strength gain over the time lapse of twenty-eight days. The cubes were sealed in a plastic bag and kept at room temperature (about 25°C).

For the purpose of comparison, about 75wt% of the master blend binder of Table IV was mixed with about 25wt% of CaCO₃, a non-pozzolanic aggregate having about the same particle size as the pumice, and 100 grams thereof was mixed with 44 grams of water. This mixture also was formed into two-inch by two-inch (2" x 2") cubes to evaluate strength gain over the time lapse of twenty-eight days. The cubes were sealed in a plastic bag and kept at room temperature (about 25°C).

The density and wet compressive strengths for the samples made according to the invention and the comparative samples made with CaCO₃ were measured and are shown in Table V below:

Table V

Time Elapsed Days	Sample Made With Pozzolanic Aggregate		Sample Made With Non-Pozzolanic Aggregate	
	Density ¹	Wet Compressive Strength ²	Density ¹	Wet Compressive Strength ²
1	79.8	1151	87.0	725
3	83.3	1779	88.9	1329
7	83.3	2646	92.6	2155
28	84.8	4267	92.8	3983

¹Pounds/cubic foot. ² Pounds/square inch.

Table V illustrates the acceptable weight strength development of the samples made from a composition according to the invention.

A second test was performed on the composition made from 75wt% master blend binder of Table IV and the pumice aggregate to study durability. A four and one-half inch (4 1/2") diameter, one-half inch (1/2") thick patty of the composition was placed under running water for a period of two months. No deterioration of the patty was visible and the total weight loss of the patty after the two-month test was 0.5%.

In other tests, the master blend binder disclosed in Table IV was blended with up to about 50wt% pozzolanic aggregate filler (pumice or perlite), with and without foaming agent, to produce boards according to the invention. Such boards exhibited acceptable physical properties.

Example 5

A fibre-containing cementitious composition according to the invention was prepared with the binder components set forth in the amounts stated in Table I below:

Table VI

Material	Weight Percent
Beta-Gypsum(Stucco)	40
Type III Portland Cement	46
Silica Fume	14

5 About 75% by weight of the binder materials identified in Table VI were mixed with about 25wt% (dry weight) of fibre that had been mixed with water (slight stoichiometric excess). The wetted fibre and binder were vigorously mixed, formed into mats and pressed into sample boards using a Bison laboratory press (Bison GmbH, Springe, Germany). The pressing conditions included 30kg/cm² pressure; press
10 temperature of about 25°C; and a press time of three hours.

The samples exhibited excellent dry and wet durability even when subjected to a continuous water spray. The final products had an extremely smooth surface.

Certain physical properties of boards made according to Example 5 were tested, including percent linear variation (ASTM D 1037), percent water absorption (ASTM D
15 1037), Mor's 3-Point Loading (ASTM C 947; and nail pull (ASTM C 473). For each of these tests, gypsum/cement/silica fume (GCSF) boards made according to Example 5 were compared to cement fibre boards made of about 82wt% Portland cement and about 18wt% wood fibre. The press time required for the cement fibre boards ranged between seven to ten hours.

20 The results of the tests are set forth in the following tables:

TABLE VII
LINEAR VARIATION (%) (ASTM D 1037)

	70°F/50%RH ³ to 90°F/90%RH ³		70°F/50%RH ³ to 109°F (Bone Dry)	
	Long Direction	Short Direction	Long Direction	Short Direction
GCSF ¹	+0.125	+0.125	-0.155	-0.155
Cement ²	+0.177	+0.175	-0.195	-0.192

¹Gypsum/cement/silica fume fibre board.

²Cement fibre board.

25 ³Relative humidity.

TABLE VIII
WATER ABSORPTION (%) (ASTM D 1037)

	Percent by Weight		Percent Caliper Swell	
	2 Hours	24 Hours	2 Hours	24 Hours
GCSF ¹	4.5	8.4	0.47	1.1
Cement ²	12.4	21.6	0.64	1.1

¹Gypsum/cement/silica fume fibre board.

²Cement fibre board.

5

TABLE IX
MOR'S 3 POINT LOADING (DRY) (ASTM C 947)

	MOR ³		PEL ⁴	
	Long Direction	Short Direction	Long Direction	Short Direction
GCSF ¹	2146.5	2297.9	1269.5	1986.2
Cement ²	2123	1928.9	1458.8	1146.2

¹Gypsum/cement/silica fume fibre board.

²Cement fibre board.

³Modules of Rupture (lb/in²).

⁴Proportional Elastic Limit (lb/in²).

10

TABLE X
MOR'S 3 POINT LOADING (WET) (ASTM C 947)

	MOR ³		PEL ⁴	
	Long Direction	Short Direction	Long Direction	Short Direction
GCSF ¹	1141.4	1332	509.2	474.3
Cement ²	1402.7	1385.8	760.7	462.3

¹Gypsum/cement/silica fume fibre board.

²Cement fibre board.

³Modules of Rupture (lb/in²).

⁴Proportional Elastic Limit (lb/in²).

15

TABLE XI
NAIL PULL (ASTM C 473)

	Pounds Force (dry) ³	Pounds Force (wet) ³
GCSF ¹	688	356
Cement ²	615	439

20

¹Gypsum/cement/silica fume fibre board.

²Cement fibre board.

³Testing parameters included:

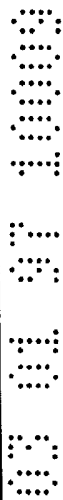
0.400 inch nail head diameter;

5 0.121 inch shank diameter; and

a loading rate of one foot/minute.

As shown in the tables above, boards made according to the invention exhibited comparable or improved physical properties as compared to cement fibre boards which did not include gypsum or silica fume. Furthermore, the boards according to the invention
10 advantageously took much less time to process (three hour press time) as compared to the cement fibre boards (seven to ten hour press time).

The foregoing detailed description is given for clearness of understanding only, and no unnecessary limitations should be understood therefrom, as modifications within the scope of the invention will be apparent to those skilled in the art.



The claims defining the invention are as follows:

1. A cementitious composition comprising:
 - (a) about 20wt% to about 75wt% calcium sulphate beta-hemihydrate;
 - (b) about 10wt% to about 50wt% of a cement selected from the group
 - 5 consisting of Portland cement, a blend of Portland cement and fly ash, a blend of Portland cement and ground blast slag; and mixtures thereof;
 - (c) about 4wt% to about 20wt% silica fume; and
 - (d) about 1wt% to about 50wt% pozzolanic aggregate.
2. The composition of claim 1 wherein said composition is free of alpha-gypsum.
- 10 3. The composition of claim 1 or claim 2 wherein the Portland cement is Type III Portland cement.
4. The composition of any one of claims 1-3 wherein the silica fume is about 4wt% to about 8wt% of the composition.
5. The composition of any one of claims 1-4 wherein the pozzolanic aggregate is
 - 15 about 10wt% to about 50wt% of the composition and comprises pumice.
 6. The composition of any one of claims 1-4 wherein the pozzolanic aggregate is about 1wt% to about 10wt% of the composition and comprises hollow silicate spheres.
 7. The composition of any one of claims 1-6 further comprising at least one
 - 20 agent and water repellent additives.
8. A self-leveling floor composition comprising:
 - (i) about 25wt% to about 75wt% of the composition of any one of claims 1-
 - 7; and
 - (ii) about 75wt% to about 25wt% sand.
 - 25 9. The self-leveling floor composition of claim 8 wherein said composition (i) comprises about 72wt% calcium sulphate beta-hemihydrate, about 20wt% Portland cement, about 6wt% silica fume and about 2wt% pozzolanic aggregate.
 10. The self-leveling floor composition of claim 9 wherein said pozzolanic aggregate comprises hollow silicate spheres.
 - 30 11. A road patching composition comprising:
 - (i) about 25wt% to about 100wt% of the composition of any one of claims 1-7; and
 - (ii) about 75wt% to about 0wt% sand.
 12. A water resistant construction material prepared by combining a cementitious
 - 35 composition with a slight stoichiometric excess of water, said cementitious composition comprising:
 - (a) about 20wt% to about 75wt% calcium sulfate beta-hemihydrate;
 - (b) about 10wt% to about 50wt% of a cement selected from the group
 - consisting of Portland cement, a blend of Portland cement and fly ash, a blend of Portland cement and ground blast slag; and mixtures thereof;



- (c) about 4wt% to about 20wt% silica fume; and
- (d) about 1wt% to about 50wt% pozzolanic aggregate.

13. The construction material of claim 12 wherein the cementitious composition is free of alpha-gypsum.

5 14. The construction material of claim 12 or claim 13 wherein the Portland cement is Type III Portland cement.

15. The construction material of any one of claims 12-14 wherein the pozzolanic aggregate is about 10wt% to about 50wt% of the composition and comprises pumice.

16. The construction material of any one of claims 12-15 wherein the silica fume
10 is about 4wt% to about 8wt% of the composition.

17. The construction material of any one of claims 12-16 wherein the cementitious composition further comprises at least one component selected from the group consisting of set control additives, water reducing agents and water repellent additives.

18. A water resistant construction material having a thickness of about $\frac{1}{8}$ inch
15 (3.2mm), said material prepared by combining a cementitious composition with a slight stoichiometric excess of water, said cementitious composition comprising:

- (a) about 20wt% to about 75wt% calcium sulfate beta-hemihydrate;
- (b) about 10wt% to about 50wt% of a cement selected from the group
consisting of Portland cement, a blend of Portland cement and fly ash, a blend of Portland

20 cement and ground blast slag; and mixtures thereof;

- (c) about 4wt% to about 20wt% silica fume; and
- (d) about 1wt% to about 50wt% pozzolanic aggregate.

19. The construction material of claim 18 wherein the cementitious composition is free of alpha-gypsum.

25 20. The construction material of claim 18 or claim 19 wherein the Portland cement is Type III Portland cement.

21. The construction material of any one of claims 18-20 wherein the cementitious composition comprises:

- (a) about 70wt% to about 75wt% calcium sulfate beta-hemihydrate;
- (b) about 15wt% to about 25wt% Portland cement;
- (c) about 4wt% to about 8wt% silica fume; and
- (d) about 1wt% to about 10wt% pozzolanic aggregate.

22. The construction material of claim 18 wherein the cementitious composition further comprises at least one component selected from the group consisting of set control
35 additives, water reducing agents and water repellent additives.

23. A cementitious composition comprising:

- (a) about 30wt% to about 75wt% calcium sulfate beta-hemihydrate;
- (b) about 10wt% to about 40wt% Portland cement;
- (c) about 5wt% to about 20wt% silica fume; and



(d) about 10wt% to about 40wt% pozzolanic aggregate.

24. A cementitious composition comprising:

(a) about 30wt% to about 75wt% calcium sulfate beta-hemihydrate;

(b) about 10wt% to about 40wt% Portland cement;

5 (c) about 4wt% to about 20wt% silica fume; and

(d) about 1wt% to about 40wt% pozzolanic aggregate.

Dated 6 January, 1999
National Gypsum Company

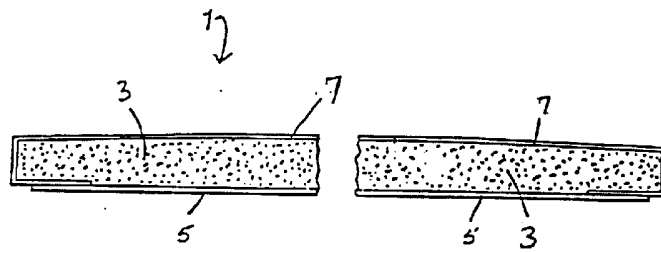
Patent Attorneys for the Applicant/Nominated Person
SPRUSON & FERGUSON

10

S
P
R
U
S
O
N
F
E
R
G
U
S
O
N

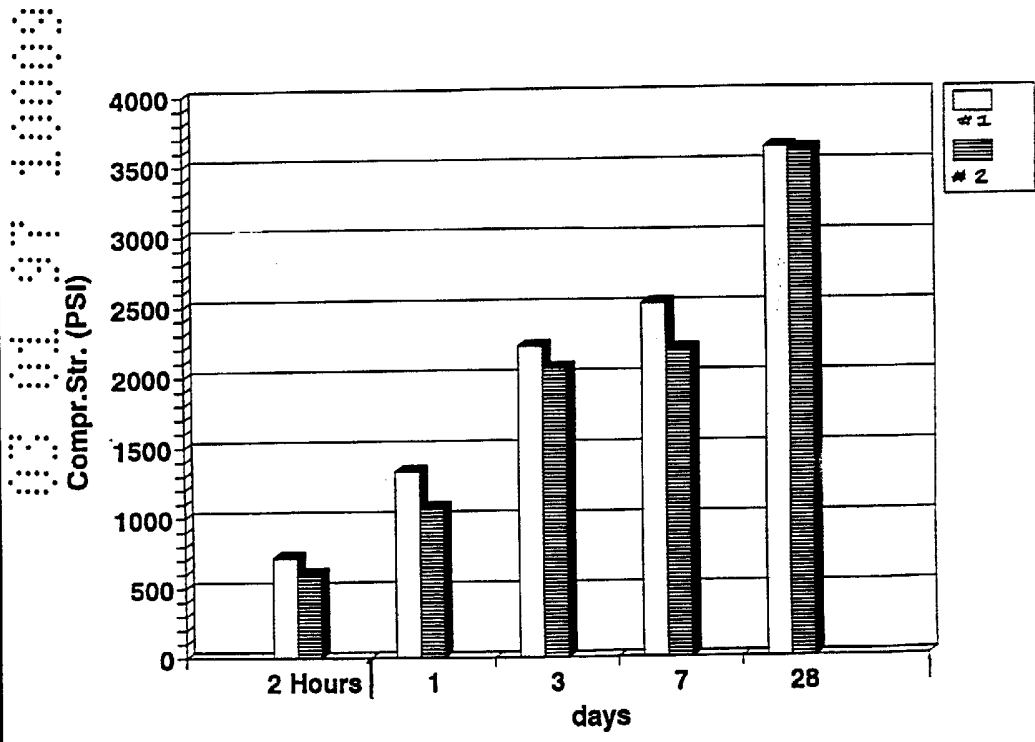


FIG. 1



5
5
5
5
5

FIG. 2



Curing Time

FIG. 3

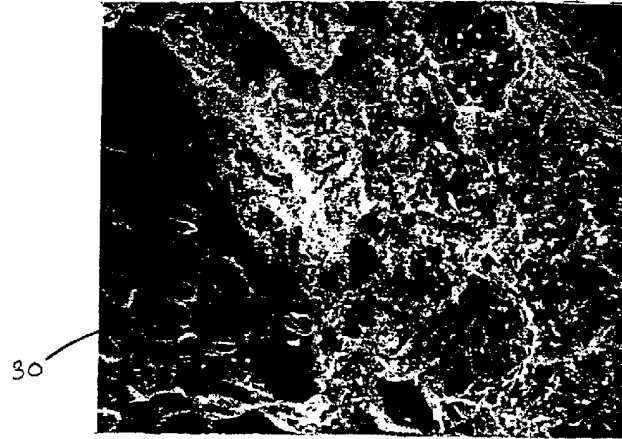
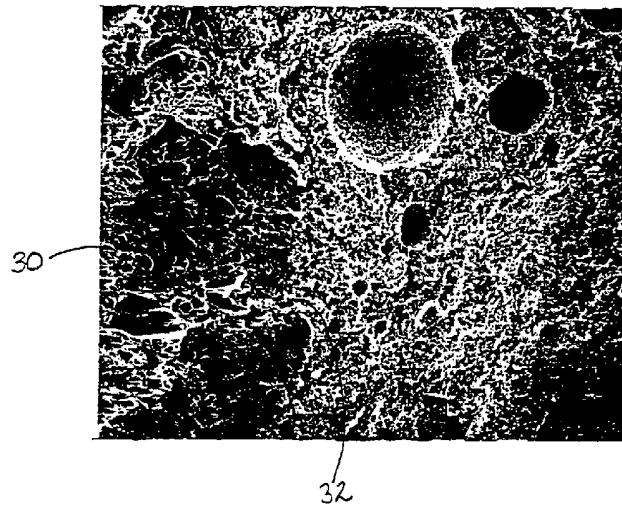
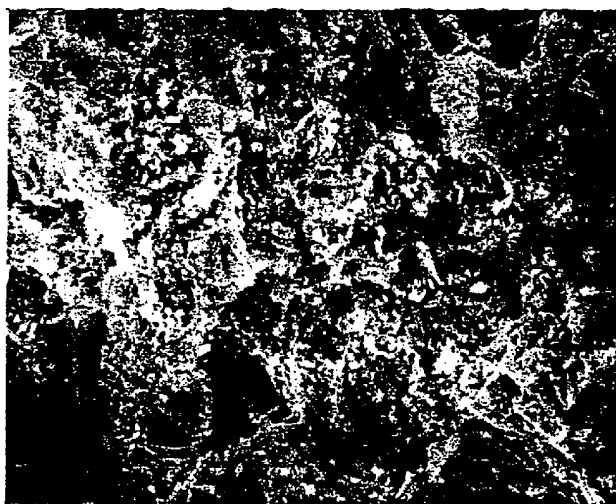


FIG. 4



3
0
4
3
0

FIG. 5



2
3
4
5
6
7
8
9
0