# "INORGANIC FIBERS AND ARTICLES MADE FROM THEM"

## U. S. PATENT LITERATURE

# "INORGANIC FIBERS AND ARTICLES MADE FROM THEM"

## A Review of the American Patent Literature

The patent literature relating to inorganic fibers, their manufacture, composition, and fabrication into products has proved to be more voluminous than had been expected, and although a large number of American patents have now been surveyed, it is still considered that the review is incomplete. Indeed, the present review does not extend back much before 1940, whereas a complete review would extend back to the beginning of the century or earlier. A decision was made to issue the present admittedly incomplete review as soon as possible, as it is felt that it is urgent that the information obtained so far should be placed in the hands of all those who are experimenting with fiber-containing products. This review has been issued in loose leaf form with the thought that it should be possible, from time to time, to issue new pages giving abstracts of other patents as they come to hand.

Since the number of patents in this general area is large, an attempt has been made to classify them under a number of headings as follow:

1. Fiber Composition:

   (a) Alumino-silicate fibers and other common high temperature fibers.

   (b) Other highly refractory fibers.

   (c) Glass fibers.

   (d) Slag and mineral wool fibers.

2. Methods of Fiberization

   (a) Fiberization from the melt.

   (b) Fiberization from solution.

   (c) Other methods of fiberization.

3. Articles Made From Fibers and Methods for Their Manufacture

Some brief comments on the patents of principal interest under each of the above headings are given below. Since there are many patents which deal with more than one topic - for example, patents claiming compositions of fiber often give the method of manufacture - each patent abstract in the following has been classified under the topic of principal interest. To assist the reader, areas of subsidiary interest are cross-referenced under the appropriate section.

1. Fiber Compositions:

(a) Alumino-silicate Fibers and Other Common High Temperature Materials

Attention is particularly called to the three patents (Nos. 1, 2 and 4) issued to Babcock and Wilcox Company, Carborundum Company and Johns-Manville respectively, covering fibers of the Fiberfrax/Kaowool type. The compositions claimed are all very similar, overlapping to a considerable extent. It will be noted that Babcock and Wilcox were first in the field with Johns-Manville last. Only the Johns-Manville patent remains unexpired.

Another interesting patent is No. 3, assigned to Babcock and Wilcox, which claims a method for making mullite-containing fibers from the melt.

The other patents listed in this section do not appear to be of great significance.

(b) Other Highly Refractory Fibers

The compositions claimed in this group (which include a number of materials described in Section 2 and cross-referenced under Section 1) are probably not of major interest to Foseco, as they are likely to be extremely expensive. However, the methods of manufacture are in many cases of interest. It may be that the aluminum borate fiber claimed in No. 9 has merit from the point of view of riser sleeves and hot tops.

(c) Glass Fibers

Of the 3 or 4 patents listed under this heading, the only one of possible interest appears to be No. 12, which covers a high silica composition, which may be quite refractory and which is made by leaching out other constituents from a lower silica glass. This principal is also referred to in several other patents listed under Section 2.

(d) Mineral Wool

Only one patent covering mineral wool compositions has been turned up to date. This does not appear to be of particular interest.

2. Methods of Fiberization

(a) Fiberization from the Melt

The patents listed under this heading together form a fairly complete guide to methods of making fibers from the melt. One notable omission is that so far no patent has been found to cover the "spinning disc" used in a number of mineral wool manufacturing processes.

(b) Fiberization from Solution

This section includes quite a large number of patents and it appears that this is an area where considerable research has been done in the last few years, most of it by two or three corporations. It appears that solution methods of fiber manufacture, are mainly directed towards the manufacture of the more exotic fibers in highly refractory oxide mixtures; however, the principal is clearly equally applicable to the less expensive Kaowool type of fiber, as particularly indicated by No. 34. These solution processes are probably of little or no interest to Foseco at the present time, but may become more interesting in due course; it is, therefore, felt that the development of this type of process should be watched carefully.

(c) Other Methods of Fiberization

The methods described in this section breakdown into (a) fiber formation via the vapor phase and (b) conversion of the low melting point glasses to fibers of higher melting point by leaching. Only the second of these general processes is likely to be of much interest to Foseco.

3. Articles Made from Fibers and Methods for Their Manufacture

This section is probably of most immediate interest to Foseco, since the patents in it are all concerned either with articles made from fibers or with methods of manufacturing fibers into articles.

A number of the patents listed deal with the use of colloidal silica and colloidal alumina as binders for fibers and contain some interesting ideas which may be well worth checking out. Patents Nos. 60 and 63 are of particular interest; No. 60 appears to be a basic patent covering the use of colloidal silica, while No. 63 goes into considerable detail on the use of colloidal silica as a binder; it is here claimed that the use of positively charged colloidal starch to precondition the fibers is beneficial in securing a good bond.

Several methods of reducing the amount of colloidal silica used, by partial substitution of a less expensive binder are suggested. In Patent No. 61, it is suggested that resins may be used to reduce the amount of colloidal silica. Use of calcium aluminate cement is quoted in No. 65.

One Patent No. 57 claims a use for a method of generating colloidal silica in-situ by reaction of sodium silicate and boric acid or borates which lock up the sodium ions from the silicate; this may be worth considering as a cheaper alternative to colloidal silica.

Several patents claim the use of suspension agents in felting processes involving colloidal silica. For example, No. 59 claims the use of Lyofax SPK, dicyandamide-formaldehyde condensate. No. 64 suggests the use of bentonite and "Veegum T", a magnesium aluminum silicate. This patent is also interesting in that it is claimed that fiber dimensions are important and that short fibers confer exceptional strength on the felted product.

3. <u>Articles Made from Fibers and Methods for Their Manufacture</u> (Cont.)

The use of silica aerogel as a binder is claimed in patents Nos. 53, 54, and 55; the latter suggests that the presence of impurities such as sodium sulfate may be beneficial in products bonded with silica aerogel.

A continuous process for fiber manufacture, air-felting and bonding with colloidal silica is described in No. 56.

The other patents in this group cover a miscellaneous collection of products or manufacturing processes of which several are of interest. Patent No. 70 describes a method of making a duplex product consisting of a thin layer of high temperature fiber backed by cheaper fiber; colloidal silica is used as the binder in both instances.

Johns-Manville in Patent No. 69 claim a felted riser sleeve consisting of asbestos, fiber, bentonite, diatomaceous earth and basic calcium or magnesium carbonate binder. Patent No. 62 suggests a binder consisting of bentonite, ground glass and resin which may also be worth considering.

No. 52 is interesting in claiming mold liners which are sprayed directly on to the mold or alternatively made first on a separate former and then inserted into the mold.

Two patents assigned to Alcoa (Nos. 66 and 67) are of interest in claiming an unusual composition consisting of asbestos fiber, cement and cryolite together with other fibers.

A method for bonding Fiberfrax with silicon nitride or silicon carbide is claimed in No. 51 and appears novel and interesting, but the necessity for high temperature treatment is likely to be an objection.

RWR/dt

1. FIBER COMPOSITIONS:

(a) Alumino-Silicate Fibers

(See also Nos. 24, 25, 30, 34, 43, 58)

U.S. 2,467,889. "Mineral Wool"
Inventor: I. Harter, et al
Assignee: Babcock and Wilcox Co.
#1 Filed: December 19, 1944. Issued: April 19, 1949

The two claims in this patent cover a non-crystalline wool prepared from fused kaolin, having a fusion temperature in the range 2900 to 3350°F. and a devitrification temperature above 2000°F. The presence of 0.05 to 1.0% carbon is optional. Fibers have the approximate composition 45% alumina, 51% silica, 1% $Fe_2O_3$, 2% $TiO_2$. This appears to be the original patent covering high temperature fibers, antidating both the Carborundum No. 2,557,834 and the Johns-Manville Patent No. 2,669,397. Other possible raw materials such as fire-clays, kyanite, silliminite and topaz are referred to in the specification. A cupola is suggested for melting the charge and is illustrated in a drawing. Fiberization is accomplished by means of a high velocity steam, or air jet, blown across the falling stream of slag. The fibers have an average diameter of about 1 to 6 microns, with some in the range 10 to 15 and above. Recrystallization does not take place when the fibers are heated below about 2600°F. Large amounts of mullite crystal are present in the crystallized fiber. The fibers melt at 3300°F.

U.S. 2,557,834. "Refractory Glass Wool"
Inventor: J. C. McMullen
Assignee: Carborundum Co.
#2 Filed: February 23, 1949. Issued: June 19, 1951

This appears to be Carborundum Company's basic patent in the high temperature fiber field. The claims cover a wool consisting essentially of aluminum and silica in substantially equally proportions, containing boric oxide and an alkaline oxide totalling less than 10% by weight. The claimed alumina and silica ranges are 45 to 55 parts and 55 to 45 parts respectively. The fibers have a maximum diameter of 10 microns, and resist breakdown at 1400°C. for over 24 hours. Other claims refer to the raw material charged, which consists of silica and alumina in the above proportions, plus borax glass or equivalent. The silica to aluminum ratio is held to be very important, as is the presence of borax glass or other modifying agent. Up to 7½% of the silica can be replaced by zirconia. Impurities affect both maximum service temperature and ease of fiberization. The best aluminous raw material is Alcoa's T-21 tabular alumina; Alcoa's A-1 grade, white alumina ore is also considered to be satisfactory. The silica is derived either from high purity flint or from white sand. It is stated that kaolin or other clays are unsuitable. The importance of the borax glass or borax alone as a modifier is emphasized. Separate additions of borax are unnecessary if the A-1 grade of alumina is used, together with a small addition of boric oxide and soda ash.

(a) Alumino-Silicate Fibers (Cont.)

U.S. 2,674,539. "High Temperature Refractory Products".
Inventor: I. Harter and C. L. Norton
Assignee: Babcock and Wilcox Co.
Filed: January 29, 1949. Issued: April 6, 1954
#3 (Divided out of application originally filed July 19, 1946)

The claims cover "flame-melted" kaolin fibers of the same general type as those described in the earlier patent, U.S. 2,467,889. The specification includesmention of fibers prepared by melting other refractory materials such as fire clay, bauxite, kyanite, etc. The specification also refers to the poor economics of arc furnace melting and the disadvantage that these furnaces have an inherently reducing atmosphere. Mention is made of fibers produced by melting silliminite and topaz. The method of melting claimed consists in the use of a rotary kiln employing a low cost fluid fuel together with preheated air. The specification repeats much of the information on kaolin fibers given in U.S. Patent No. 2,467,889. It is also stated that mullite wool can be made using the rotary kiln, from a mixture of bauxite, kaolin grog and raw kaolin in about 4:3:2 to 6:2:3 proportion. One of the claims recognizes this, specifying the composition range alumina 45 to 69% and silica 51 to 26%. Alkali content is restricted to 0.75%.

U.S. 2,699,397. "Refractory Mineral Fiber"
Inventor: W. P. Hahn
Assignee: Johns-Manville Corporation
Filed: September 9, 1952. Issued: January 11, 1955
Application was divided out of an earlier application
#4 Filed: November 14, 1950

This is Johns-Manville's original patent on high temperature fibers. A refractory mineral wool is claimed which is stable at temperatures above 2000°F. and has the following composition: alumina 51 to 55%, silica 43 to 47%, balance chiefly ferric oxide and titania. The wool is produced by fusing kyanite and silica and subsequent fiberization. All are composition claims. In essence it differs from the Carborundum Patent No. 2,557,834, only in the source of raw materials. It is indicated that there is only a narrow range of alumino-silica ratios, which can be efficiently fiberized to give good yields of wool. In this range, shot is readily removed. The critical ratio range is 1.1 to 1.3 alumina to silica.

U.S. 3,007,806. "High Silica Refractory Wools"
Inventor: F. J. Hartwig
Assignee: Babcock and Wilcox Co.
#5 Filed: December 13, 1957. Issued: November 7, 1961

The claims cover a fiber for use at about 1500°F. having a viscosity substantially identical with that of pure silica at 2000°F. and consisting of 70 to 95% of silica and 5 to 30% of a viscosity-reducing modifier from the group magnesia, lime, alumina, titania, chromic oxide and zirconia. Melting temperature is above 2800°F. when in a crystalline form. The preferred composition contains 90% silica. Other claims cover the method of making continuous monofilament fiber from the melt. The preferred starting material is kaolin clay to which is added silica in amounts sufficient to increase the silica percentage into the claimed range.

(a) Alumino-Silicate Fibers (Cont.)

U.S. 3,007,806. (Cont.) Fiberization (monofilament) is accomplished at between 100 and 1000 poise. The molten mixture is tapped through a nozzle and the monofilament wound on a reel. In one particular instance the drawing speed was between 1000 and 4000 FPM, fiber diameter was about 20 microns. In another, a drawing speed of 8000 FPM was used. Fiberization may also be accomplished by blowing. The specification contains a good deal of information on the properties of siliceous fibers at high temperature. It is pointed out that kaolin fibers undergo partial crystallization very rapidly. Mullite crystals are formed in such fibers in a matter of seconds at 2000°F.; the remaining silica remains in vitreous form. Of the modifiers listed, alumina, titania, chromic oxide and zirconia yield fibers with the highest melting point.

U.S. 3,044,888. "Glass Fiber"
Inventor: J. D. Provance
Assignee: Houze Glass Corporation
#6 Filed: July 5, 1960. Issued: July 17, 1962

This patent claims a high temperature fiber with a minimum softening temperature between 1600 and 2400°F., and consisting essentially of 30-40% alumina, 40-50% silica, 0.5 to 8% zirconia, 3 to 24% magnesia and less than 6% soda. The presence of 0.3 to 0.5% fluorine is optional. The preferred composition ranges are 34 to 38% alumina, 45 to 49% silica, 2.5 to 6.5% zirconia, 8% to 15% magnesia and less than 1.3% soda. The fluorine increases the strength of the fiber without detrimental effect. The magnesia content has a considerable effect on minimum softening temperature, the highest melting temperature being associated with the lowest magnesia content. The claimed compositions are characterized by high strength, high modulus of elasticity and high durability together with good temperature resistance.

U.S. 2,674,539. " Refractory Fiber Composition"
Inventor: W. G. Ekdahl
Assignee: Johns-Manville Corporation
#7 Filed: December 13, 1965. Issued: October 24, 1967

The claims relate to an improved alumino-silicate fiber containing 0.5 to 1.5% manganese dioxide. The manganese dioxide addition results in superior fusion and fiberizing characteristics, thus facilitating fiber production by blowing or spinning.

1. FIBER COMPOSITIONS:

(b) Other Highly Refractory Fibers

(See also Nos. 28, 29, 30, 31, 32, 33, 34, 43, 45)

U.S. 3,080,242. "Fibrous Aluminum Borate and its Preparation"
Inventor: K. L. Berry
Assignee: E. I. Dupont Co.
#8 Filed: March 25, 1959. Issued: March 5, 1963

The claims include composition and methods claims covering aluminum borate fibers of the composition $(Al_2O_3)_3n \pm 0.4\ B_2O_3$, where "n" is not greater than 2. The length to width ratio of the fiber exceeds 10; the fibers are produced by reacting boric oxide with aluminum oxide or aluminum hydroxide at a temperature between 1000 and 1600°C. In a modification of the process, water is present at 500°C. under pressure, the amount of water being equal to at least 50% of the weight of the solid ingredients. In the process involving molten alumina and molten $B_2O_3$ , slow cooling from about 1000°C. to below 900°C. is vital. The amount of alumina can vary from 1 to 25% by weight of the boric oxide. The fibers crystallize from the melt after addition of the boric oxide. A good deal of information is presented on the crystallographic characteristics of the fibers and several examples are given on the preparation of different fiber compositions. Mechanical properties, etc. are described.

U.S. 3,386,840. "Article Containing Silicon Carbide Fibers"

Inventor: G. A. Gruber
Assignee: Monsanto Co.
Filed: March 31, 1959. Application Date: Nov. 24, 1964
(Division of Application filed January 3, 1961).
Now U.S. Patent No. 3,246,950. The earlier application
#9 itself a C.I.P. of an application filed March 31, 1959.

The single claim relates to fabricated articles comprising a continuous fibrous material selected from the group consisting of oxides, ceramics and polymerized organic monomers having dispersed in them fibrous beta silicon carbide. The production of silicon carbide fibers is described in detail. These fibers have diameters from 50 to 10,000,000 Angstroms and vary in length from 250 to two and one-half billion Angstroms. Fiber length to diameter ratio varies from 5:1 to 50,000,000:1. Tensile strength is about 10,000,000 p.s.i. The fibers are made by combining a mixture of carbon monoxide with silicon monoxide gas at high temperature. Various modifications of the process are described. Mats of silicon carbide fiber are excellent insulators and are usable at temperatures where no other materials are available. Mention is also made of products made from these fibers including resin bonded products which may be heat treated to burn out the resin and leave a carbon residue or skeleton associated with the fiber and thus produce extremely good refractory properties at high temperature. This type of material has been used for rocket nose cones.

(b) Other Highly Refractory Fibers (cont.)

U.S. 3,386,840. (Cont.) Production of metals strengthened with silicon carbide fiber is also mentioned. Another interesting example is the development of a wear resistant surface on aluminum castings. The mold is lined with a layer of silicon carbide fiber in paper form and poured with molten aluminum. Mention is made of improvement of ceramic articles by incorporation of silicon carbide fiber in the slip. Use as a high temperature filter is also mentioned. It is stated that carbon may also be introduced in pyrolitic form into a felt of the material.

U.S. 3,391,681. "Preparation of Aluminum and Chromium Sesquioxide Fibers"
Inventor: W. A. Westdorp
Assignee: E. I. Dupont & Co.
Filed: December 29, 1965. Issued: July 9, 1968
#10 (C.I.P. original application, December 31, 1964)

Claims a process for the preparation of single crystal metal oxide fibers having the corundum structure; the chemical composition of the fibers is aluminum sesquioxide, or chromium sesquioxide. In the process claimed water and the vapor of a sub-oxide of aluminum or chromium are brought into contact with a substrate composed of alumina, alumina containing silica, alumino-silicate, chromium sesquioxide or mixtures, in the presence of a catalyst consisting of molten particles of cobalt or nickel. The substrate is maintained at 1455 to 2300°C. The reaction vessel is such that there is a temperature gradient along the substrate. The nickel or cobalt is vaporized and condenses on a cooler part of the substrate; these tiny condensed particles serve as nuclei for the crystallization of the fiber. Other claims relate to methods preparing the sub-oxide which is accomplished by passing hydrogen over polycrystalline aluminum sesquioxide, chromium sesquioxide or by the reaction of oxygen and aluminum. Practical and theoretical details of the process are described in detail.

(c) Glass Fibers

(See also 22, 36, 37, 38, 39, 40, 42, 46)

U.S. 2,143,022. "Fiberized Mineral Felt".
Inventor: B. C. McClure
Assignor: None
#11 Filed: May 22, 1933. Issued: January 10, 1939

There is claimed a felted body of fiberized glass of a particular composition. The composition claim does not appear to be of particular significance from the present point of view.

(c) Glass Fibers (Cont.)

U.S. 2,494,259. "Fibrous Glass Articles"
Inventor: M. E. Nordberg
Assignee: Corning-Glass Works
Filed: March 20, 1946. Issued: January 10, 1940
#12 C.I.P. of U.S. 2,461,841

This patent covers articles made from glass fibers consisting of approximately 90.5% silica, 3.5% alumina, 2.7% lime and 0.7% magnesia with 2.5% boric oxide. Numerous modifications of this composition which is said to resist high temperatures are mentioned. The presence of moderate amounts of BeO, ZnO, CdO, BaO, $Al_2O_3$, $TiO_2$, $ZrO_2$ and $ThO_2$ are allowable. Both porous and non-porous "vitreous" fibers are mentioned. Although not mentioned in the claims, a major feature of this invention is the method of leaching glass fiber to extract non-siliceous constituents, thereby bringing the composition in line with the above. The preliminary heat treatment usually employed to condition glass for leaching cannot be used with these fibers owing to the danger of sintering and fusing. The fiber diameter is critical and must not exceed 0.001 inch. Leaching must be accomplished in acid or neutral solution. The glass must contain over 75% silica before leaching together with at least one glass flux consisting of an alkaline metal oxide or boric oxide and an oxide of one or more of the following: Be, Mg, Ca, Zn, Sr, Cb, Ba, Al, Ti, Zr, and Th. The leached fibers are porous, but may be consolidated by heat treatment. A good deal of information on leaching practice and chemistry is presented in the specification.

U.S. 2,876,120. "Glass Composition".
Inventor: G. R. Machlan.
Assignee: Owens-Corning Fiberglas Corporation
#13 Filed: December 3, 1954. Issued: March 3, 1959

This patent covers a glass composition of high elastic modulus and improved abrasion resistance. The glass is intended to be manufactured into fibers. The composition ranges mentioned do not appear to be of interest from the present point of view.

U.S. 3,138,561. "Radiation Resisting Glass Composition"
Inventor: D. Labino
Assignee: Johns-Manville Fiberglass, Inc.
Filed: July 1, 1957. Issued: June 23, 1964
Divided out of original application of Nov. 23, 1953.
#14 The original application is now issued as U.S. 2,830,000

Claims a glass for use as a radiation shield, containing silica, lead, oxide, alkaline metal oxide and boric oxide. The composition of glass claimed does not have any particular relevance to the present problems.

(d) Slag & Mineral Wool Fibers:

(See also Nos. 16, 18, 21)

#15 U.S. 2,051,279. "Mineral Wool"
Inventor: A. T. Thorndyke
Assignee: Alfred W. Knight
Filed: March 21, 1934. Issued: August 18, 1936

Claims the production of mineral wool by fusing a high calcium mineral selected from the pyroxene group and of general composition $RSiO_3$, the molecular proportion of silica being substantially equal to the moldecular proportion of the other metal oxide or oxides present. The material is blown to form fibrous strands. Particular reference is made to the mineral wollastonite as a raw material. It is stated that the claimed mineral wool, may be made to close tolerances in regard to color and physical properties.

2. METHODS OF FIBERIZATION:

(a) Fiberization from the Melt

(see also Nos. 1, 2, 3, 4, 5, 7, 15, 22, 35,49)

#16 U. S. 2,215,887. " Method of Operating Mineral Wool Furnace"
Inventor: W. R. Seigel, etal
Assignee: Johns-Manville Corporation
Filed: May 4, 1937. Issued: September 24, 1940

The invention claimed is a method of charging and operating a blast furnace for the manufacture of mineral wool. In the method proposed, the fuel is predominately located in the center of the combustion zone at the walls of the furnace, some interleaving is promoted, the fuel extending into the raw material area and vice versa. The method is said to produce a superior grade of mineral wool on an economic basis, without rapid deterioration of the blast furnace or cupola. Easy control of the melt is also claimed.

#17 U.S. 2,300,930. "Mineral Wool"
Inventor: L. C. Johnson
Assignee: Anthracite Industries, Inc.
Filed: August 8, 1939. Issued: November 3, 1942

There is claimed a method of producing mineral wool substantially free of iron, from coal ashes and a fluxing agent; in the method proposed the ashes are mixed with the fluxing agent and heated so that the carbon in the ashes is burned out. The wool produced is naturally very dependent on the analysis of the ash. A high alumina ash used together with limestone as a flux appears to be preferred. Iron removal may be accomplished by heating the molten ash under reducing conditions, e.g. in the presence of carbon.

U.S. 2,297,006. "Apparatus for Making Glass Wool"
Inventors: D. Kleist and G. Slayter
Assignee: Owens-Glass Fiberglas Corporation
#18 Filed: April 14, 1937. Issued: June 16, 1942

Claims a novel apparatus for fiberization of glass and formation of a mat. The apparatus consists essentially of a container for a molten glass which is fitted with nozzles at the bottom; opposite each nozzle is a jet from which a hot steam blast issues, and which causes the formation of fine fibers. The individual blast jets blow the fibers down into individual vertically arranged spouts beneath them. These spouts are of the venturi type, such that a considerable injection of air takes place; the attenuated fibers are thus all directed into the spout. The lower ends of the spouts open into an expansion hood or chamber, which is also of streamlined construction with the large end at the bottom. The fibers become matted together as they travel down this hood, and impinge on a conveyor at the base of the hood, there becoming further matted. The equipment is designed so that the flow of air is essentially streamline, thus avoiding undesirable turbulence, vibration and noise. The fibers collect on the conveyor and there are sprayed with a suitable oil or other coating. The mat thus formed passes beneath a pressure plate where it is further compacted. No special means for deshotting appear to exist in the apparatus described. The equipment permits a mat free from thick and thin spots or knots or lumps to be produced. The density of the mat is readily controlled.

U.S. 2,481,543. "Method and Apparatus for Producing Glass Fibers"
Inventor: C. J. Stalego
Assignee: Owens-Corning Fiberglas Corporation
#19 Filed: April 13, 1947. Issued: September 13, 1949

The patent describes equipment used to produce fibers from molten glass by blowing. It is stated that the equipment is more productive than that previously in use. The claims are not restricted to glass, but apply to any softenable material. The glass is melted in a small chamber above the nozzle and thin streams issue from orifices beneath the melting unit. The glass rods thus formed are fed into a hot, high velocity, blast with the rods disposed in a common plane extending diagonally to the blast. By the time the glass reaches the blowing zone, it is in an essentially solid condition, but is remelted by the force and heat of the blast.

U.S. 2,489,242. "Method and Apparatus for Making Fine Glass Fibers".
Inventor: G. Slayter, et al
Assignee: Owens-Corning Fiberglas Corporation
#20 Filed: April 27, 1944. Issued: November 22, 1949

In this process, glass fibers ranging in diameter from 1 to 2½ microns are produced by feeding a rod of glass into a very hot blast directed horizontally. The glass is remelted and formed into fibers, which are blown into a collector. The process is not unlike that of Stalego (U.S. Patent No. 2,481,543), but in this case the glass is fed into the blast in the horizontal direction, whereas in Stalego's process, the feed is in the vertical direction. A novel feature of the present process is that low pressure gas is used to heat the blast. Velocity of the blast is 500 to 700 ft. per second, and is largely determined by dimensions of the combustion chamber. The process is described in considerable detail.

U.S. 2,576,312. "Method of Making Mineral Wool"
Inventor: L. J. Minnick
Assignee: Baldwin-Hill Co.
#21 Filed: August 16, 1948. Issued: November 27, 1951

Claims the process of making mineral wool from a charge comprising agglomerated bodies in which particles of wool forming material are bonded in a matrix comprising glauconite having a particle size no greater than 100 microns; the matrix comprises between 3 and 70% of the total. Part of the charge may be mineral wool shot. Besides glauconite, the other constituents of the charge may include silica sand, silica rock, sandstone, crushed glass, silicon limestone, dolomite high in silica, slags such as iron blast furnace slag, copper and lead furnace slag and the like. Lime-bearing materials such as ordinary limestone, dolomite, etc. may also be present, as may be iron-bearing materials such as roll-scale. The practice of the invention is described in great detail in the specification. Much useful information is given on general features of the slag wool manufacturing process.

U.S. 3,081,179. - "Glass Fiber Composition"
Inventor: R. Charbat and A. D. Fentzke
Assignee: Union Carbide Corporation
#22 Filed: May 29, 1961. Issued: March 12, 1963

Claims include process and composition claims covering a low temperature glass fiber. It is stated that for the production of glass fibers by blowing, viscosity must be in the range of 5 to 200 poise. The purpose of the compositions claimed is to enable this viscosity range to be achieved over a relatively large temperature range.

U.S. Re. 24,060. "Apparatus for Forming Glass Fibers"
Inventor: R. G. Russell
Assignee: Owens-Corning Fiberglas Corporation
(Originally No. 2,634,553)
#23 Issued April 14, 1953. Reissued September 6, 1965

Claims an apparatus for producing glass filaments which consists of a container for the molten glass, fitted with a set of orifices in one wall and a fluid cooled plate with orifices corresponding to those in the wall; the plate is located close to the wall and serves to cool the filaments rapidly. The equipment includes a means for drawing streams of glass from the orifices and attentuating them in fine filaments. The claimed advantage of the apparatus is that cooling of the glass filaments is equalized.

(b) Fiberization from Solution

U.S. #2,866,404. "Spinning Amorphous Glass Fiber from a Viscous Aqueous System"
Inventor: J. D. Teja.
Assignee: Montecatini
#24 Filed: April 26, 1957. Issued: May 12, 1959

In the process claimed, a viscous aqueous system consisting essentially of water, colloidal silica &alkali metal oxides is made into a slurry with oxides of one or more of the following metals: boron, aluminum, zirconium, titanium, zinc, calcium, barium, arsenic, germanium, phosphorus, lead, thorium, beryllium and tungsten. The viscous slurry is formed into a stream which is contracted to less than 20 micron diameter by elongation in a gaseous atmosphere containing 10 mm. water vapor pressure. Elongation and syneresis takes place simultaneously, the water being concentrated at the surface of the stream and thus evaporated. There is thus produced a stable amorphous glass fiber containing more than 40% silica. Viscosity of the initial system must be at least 14 poise. The fiber is subsequently dried at above 200°F. Tensile strength exceeds 30,000 psi.

U.S. #2,908,545. "Spinning Non-fused Glass Fibers from an Aqueous Dispersion".
Inventor: J. D. Teja
Assignee: Montecatini
Filed: May 25, 1955. Issued October 13, 1959
#25 Divided from application filed originally on May 25, 1955(sic)

In the process claimed three aqueous mixtures are prepared; one contains a mixture of glass forming metal oxides, another a suspension of silica particles and a third, a transforming or stabilizing agent. The silica suspension is normally a silica sol. The slurry of glass forming oxides can vary considerably in composition, but typically would contain materials such as zirconia, zinc oxide, boric oxide, silica, titania, etc. The transforming agent is of complex composition. Basically, this comprises an organo-silicon reagent, an organic hydrophilic material and an organic hydrophobic material. The hydrophilic material has a molecular weight exceeding 10,000 and contains sufficient water solubilizing groups to furnish one such group to about 30 groups of the hydrophilic material. In addition, ethyl acetate, polyvinyl alcohol, polyvinyl acetate, polymethacrylic acid and copolymers are present. The mixture tends to form a gel material of neutral pH. The molecular weight of the hydrophobic material must exceed 5000; this can be a dispersion selected from natural latex, synthetic rubber, polymers of vinyl chloride and acrylonitrile, copolymers of butadiene and acrylonitrile, vinyl resins, vinylidene, chloride resins, starch ethers, starch esters and butyl acrylate. The method of preparation of the transforming solution is complicated, and fully described in the patent. A good deal of information on the chemical theory involved in given. Fiberization is achieved by inserting a pointed body into the syrup and withdrawing it at a controlled rate through an electrostatic field; alternatively extrusion or other methods may be employed. The electric field imposes an elongating force on the fiber as it is formed.

U.S. #2,919,996. "Aqueous Dispersions of High Molecular Weight Fibrils of Amorphous Silicates".
Inventor: J. D. Teja
Assignor: Montecatini
#26 Filed: April 26, 1957. Issued: January 5, 1960

There is claimed a composition and method for the preparation of colloidal particles of polysilicic acid in which colloidal particles are linear with molecular weights of between 5,000 and 20,000 instead of spherical.

(b) Fiberization from Solution (Cont.)

U.S. 2.919.996 (Cont.) Colloidal solutions of this kind are said to be useful in coatings, adhesives and in the preparation of glass fibers. The basic physical chemistry is described in detail. The process is as follows: boric acid is added to a colloidal silica solution and with this is incorporated alumina and zinc oxide. Sodium trisilicate solution is heated in a separate container to increase the solids content to about 40%. The second solution is added to the first, and the mixture heated to evaporate the water down to a solids contents of 40%.

U.S. 2.969.272. "Preparation of Siliceous Materials"
Inventor: J. D. Teja
Assignee: Montecatini
#27 Filed: April 26, 1957. Issued: January 24, 1961

Claims a method of preparing amorphous glass fibers from a viscous siliceous solution. (See U. S. No. 2,919,996). The viscous siliceous solution is extruded through a 45 micron orifice. As the result of gravitational force, it undergoes considerable stretching and 30 ft. below the orifice is sufficiently strong to permit gentle handling. After passing through a drying zone the fiber is reeled. The minimum stretching force is such that the square root of the ratio of length of the resulting siliceous product to the diameter of a theoretical sphere of the siliceous solution whose total solids content is equal to the solids content of the fiber, exceeds five.

U.S. 3,077,380. "Preparation of Sapphire Fibers".
Inventor: E. Wainer and A. Cunningham
Assignee: Horizons, Inc.
#28 Application Date: July 24, 1959. Issued: Feb. 12,1963

The patent claims a process for the manufacture of alpha alumina fibers which possess considerable strength at high temperature.

Aluminum is melted and heated to 1370-1510°C. The molten metal is reacted with hydrogen gas dried to a dew point between -30°C. and -90°C.; a condensing surface is in physical contact with the vapor phase, the surface containing at least one refractory oxide having a heat of formation smaller than the heat formation of alumina at the prevailing temperature; fibrous alumina is formed on this condensing surface. The hydrogen contains 3 to 5 ppm. of water vapor. The melt may contain up to 25 mole percent of one of the following elements: Ti, Zr, Ng, Ta, Si. The condensing surface may consist of silica, titania, zirconia, or of impure alumina contaminated by one of these oxides. The mechanism of fiber formation is said to be as follows: When the molten aluminum reacts with a trace of moisture vapor in the hydrogen atmosphere, either AlO or $Al_2O$ forms. Condensation of this oxide on the condensing surface results in disproportionation of the oxide, and formation of alpha alumina plus aluminum metal. If this takes place under the correct conditions, whiskers of alumina are formed. Addition of about 20% of titanium to the melt speeds up the process by a factor of eight. The rate at 1510°C. is 17 times that at 1370°C. The tensile strength of the fibers exceeds 1,000,000 p.s.i. and diameters range from 0.02 to 0.04 mm. It is held that the formation of the sub-oxide of aluminum takes place in the vapor phase by reaction between the aluminum vapor pressure at about 7 mm. Hg pressure with the water vapor in the atmosphere. Wool whose fiber diameters are in the range 0.1 to 7 microns can be produced;

(b) Fiberization from Solution (Cont.)

U.S. 3,077,380. (Cont.) alternatively whiskers whose diameters exceed 80 microns can be made by the process. Coarse fibers are formed from the finer wool fibers when the formation temperature is excessive or when the proper amount of oxygen is not available from the refractory condensing surface.

U.S. 3,082,051. "Fiber Forming Process"
Inventors: E. Wainer and R. M. Beasley.
Assignee: Horizons, Inc.
#29 Filed: July 24, 1959. Issued: March 19, 1963

In the process claimed, there is prepared a solution containing an organic salt, e.g. an acetate, of the following: aluminum, rare earth, zirconium, thorium, chromium, hafnium, niobium, tantalum, manganese, iron, cobalt, nickel and an organic salt of zinc, beryllium, copper, or cadmium. The solution must form a stable colloidal dispersion, after evaporation of most of the liquid. A thin film of the solution is deposited on a clean smooth surface and the film dried by heating between 20 to 1000°C. until the solvent is removed. The film spontaneously disrupts to form fibers. The fibers are subsequently fired at about 1200°C. to insure complete dehydration and to drive off residual acid. The solvent may be aqueous or organic. The solution may be acid or alkaline. Examples of the use of the process to prepare zirconia and other fibers are given.

U.S. 3,082,099. "Inorganic Fibers and Method of Preparation".
Inventors: R. M. Beasley & H. L. Jones
Assignee: Horizons, Inc.
#30 Filed: Feb. 26, 1960. Issued: March 19, 1963

The patent claims a non-vitreous fiber in the form of a ribbon of rectangular cross-section composed of 5 to 15% silica and 85 to 95% of an oxide of one of the following: aluminum, rare earths, zirconium, hafnium, thorium, iron, cobalt, manganese, nickel and vanadium. The fibers are 0.1 to 1.0 micron in thickness and 2 to 20 microns in width. The method of forming consists of taking a solution containing (1) a metal salt of a carboxylic acid whose dissociation constant is greater than $1.5 \times 10^{-5}$ (2) the metal salt of a strong mineral acid, and (3) a silica sol. The relative proportion of metal salts to silica lies in the range 85 to 95% metallic oxide to 5 to 15% silica. The pH is adjusted to between 1 and 2 by addition of acid. Acetic acid is the preferred carboxylic acid. A layer of the composition is formed about 50 microns thick, on an infrared absorbing supporting material to which the fibers do not adhere. The layer is exposed to infrared radiation to remove the solvent, whereby it dries and disintegrates into fibers. The fibers are subsequently fired at between 500 and 600°C., and finally at 1200°C. The silica sol contains 30 50% of silica dispersed in water. Considerable information is given on the changes taking place during the treatment cycle. The fibers made are strong, having tensile strengths up to 400,000 p.s.i. Little information on the properties of the fibers is given.

U.S. 3,096,144. "Method of Making Inorganic Fibers".
Inventors: E. Wainer & E. F. Mayer.
Assignee: Horizons, Inc.
#31 Filed: August 12, 1960. Issued: July 2, 1963

Claims a method for making fibers having porosities up to 75%. The starting materials are a metal salt of a strong acid and an alkali salt of a weak acid from which a sol stable in the presence of large amounts of ionized salts is produced. A double decomposition reaction takes place. The pH is adjusted to between 5 and 9. Fibers are produced by spreading the sol on a substrate and applying a succession of low temperature heat treatments in which the thin film of the fiber forming composition is caused to pass through an incipient gel stage. The fibers are subsequently heat treated at about 105°C. to remove water soluble constituents and are finally fired at a high temperature (above 600°C.) to promote shrinkage and drive off impurities, etc. The fibers range in length from 2.5 to 6 inches and in width from 5 to 25 microns; thickness varies from 0.2 to 0.5 microns. A final washing treatment may be given. Numerous examples of the process are quoted and a good deal of background information is given. Among the fiber species mentioned in the examples, are zirconia, barium titanate, titania, alumina, zirconia, boric oxide and urania.

U.S. 3,108,741. "Liquid Polymers, Solid Articles Made Therefrom, and Methods of Preparing Same:.
Inventors: E. Wainer, etal.
Assignee: Horizons, Inc.
#32 Filed: November 29, 1960. Issued: April 27, 1965.

This patent claims methods for the production of novel inorganic polymers in liquid form and methods for producing solid articles from them, including fibers. The materials used are metal salts which produce hydrated oxides, or hydrous oxide derivatives on neutralization with alkalies. The following metals are particularly suitable: Al, Ti, Va, Cr, Mn, Fe, Co, Ni, Y, Zr, Hf, Th and Rare Earths. The polymeric solutions are produced from a diluted aqueous solution of organic metal salts such as a mono-dicarboxylic acid salt. This solution is concentrated by vacuum evaporation. Polymerization of the concentrate, which contains at least 50% by weight of an oxygen-containing derivative of the metal is produced by a low temperature heat treatment. Fibers may be made from the polymerized material by drawing, extrusion, or other method. The fiber is subsequently reduced to the metal oxide by high temperature firing. The specification contains a great deal of information on the details of the process.

U.S. 3,311,481. "Refractory Fibers & Methods of Making Them.
Inventors: J. P. Sterry & A. Skurcenski.
Assignee: Hitco Corp.
#33 Filed: March 1, 1962. Issued: March 28, 1967.

The claims relate to a process for manufacturing ceramic fibers containing a proportion of zircon. Colloidal silica is added to zirconium diacetate in 1:1 molar ratio between the zirconia and silica constituents; acetic acid is added to maintain the pH between 3.5 and 4; the material is evaporated to between 150 and 400 poise. Fiberization is achieved by drawing through dies or by a combination of extrusion or blowing and

(b) Fiberization from Solution (Cont.)

U.S. 3,311,481. (Cont.) the fibers are then fired at successively higher temperatures, to decompose and eliminate unwanted constituents. Two claims relate to the product and the rest are process claims. Fibers made from other oxides including magnesia, thoria, hafnia, alumina and rare earth oxides are mentioned. Stabilizers, such as calcium acetate, may be added to the solution in amounts up to 10% calcia. The purpose of the calcia is to impart a desirable crystalline structure to the fiber. For example, zirconia fibers with 8% calcia present have a cubic structure; omission of the calcia often results in a proportion of monoclinic and tetragonal crystallization. Acid is added to keep the pH in the range 3 to 5. Atmosphere control is necessary during fiberization. A centrifugal spinning machine is a particularly useful device. Factors affecting the final fiber are discussed in detail. Drying is accomplished at temperatures successively higher up to 2000°F.

U.S. 3,322,865 "Process of Manufacturing Refractory Fibers".
Inventor: J. E. Blaze.
Assignee: Babcock & Wilcox Co.
#34 Filed: Nov. 10, 1966 (CIP of Application filed 2/19/64)
Issued: May 30, 1967

The claims in this patent cover a method for making fibers from aqueous solutions. A solution of a water soluble inorganic metal salt capable of decomposing to a metallic oxide when calcined at 800 to 2500°F. is concentrated to a viscosity of 1 to 1,000 poise and fiberized at a solids concentration less than 50% by weight and in a space whose relative humidity is less than 60% and preferably 20 to 30%. The fibers are heat treated to eliminate the solvent and to decompose the salts. Fiber diameter is between 20 microns and 0.5 micron. The fibers are crystalline. Salts are selected from the sulphates, chlorides, oxychlorides, phosphates and nitrates of aluminum, beryllium, chromium, magnesium, thorium, uranium and zirconium. Heat treatment atmosphere may be oxidizing. Whereas glassy fibers have their surface temperature limited to about 2000°F. because they lose much of their strength as the result of recrystallization at higher temperatures, fibers made from aqueous solution, on the other hand, are already in the crystalline form and, therefore, maintain their strength better. (Mention is made of an alternative process in which a film of the metal salt is formed from a colloidal dispersion and the film is subsequently heat treated, removed and torn into ribbons). The concentrated aqueous solution must have the fiberizing characteristics of a molten glass and must also be stable at room temperature. The solids content should be as high as possible. The compounds must decompose and densify into a refractory oxide readily on heating, preferably at 1000 to 1400°F. Control of heating in the fiberizing process is most important. A final sintering process is designed to give the fibers maximum density without crystal growth. Mention is made of alumina fibers which are usable at 3300°F. Zircon oxide fibers were also made with a theoretical melting temperature of about 4900°F. Specific examples refer to the manufacture of alumina fibers from aluminum chloride, aluminum phosphate fibers from a mixture of mono- and di-hydrogen aluminum phosphate, plus some aluminum chloride, stabilized zirconia fibers containing MgO as a stabilizer from zirconium oxychloride hexahydrate and zirconium acetate, alumina fibers from aluminum chloride plus alumina, alumina fibers made from aluminum metal only, mullite fibers made from aluminum chloride or aluminum and magnesium chloride, unstabilized zirconium fibers made from zirconium oxychloride

(b) Fiberization from Solution (Cont.)

U.S. 3,322,865. (Cont.) and zirconium acetate, stabilized zirconia fibers from the oxychloride and magnesium metal, stabilized zirconia fibers from the oxychloride and lime or magnesia, thoria fibers from thorium hydroxide, thoria fibers from thorium chloride.

#35 Canadian Patent 787,838. "Colloidal Alumina Monohydrate".
Inventor: D. R. Napier.
Assignee: Continental Oil Co.
Filed: June 18, 1964. Issued: June 18, 1968.

This patent describes claims processes for the manufacture of colloidal size particles and colloidal fibrous particles of alpha-alumina monohydrate (Boehmite). In essence the process consists of hydrolyzing an aluminum alcoholate and then reacting the alumina thus formed with acetic acid. The material is subsequently dried to an alumina content of 50%. In the modification which leads to colloidal fiber an aluminum alkyl is reacted with oxygen, aluminum alkoxide, thus being formed; the alkoxide is hydrolyzed and the alumina separated. The separated alumina is heated with acid at 150° to 300°C. Specific examples of the process are given. The fibers have an average length of about 300 to 30,000 Angstroms and a width of 10 to 400 Angstroms.

(c) Other Methods of Fiberization (See also Nos. 5, 8, 10, 12, 24, 58)

#36 U.S. 2,461,841. "Method of Making Fibrous Glass Articles".
Inventor: M. E. Nordberg.
Assignee: Corning Glass Works.
Filed: January 26, 1944. Issued: Feb. 15, 1949

A further development of U.S. 2,494,259. Some additional glass compositions are claimed; claims also relate to the diameter of the glass fibers which should be less than 0.001 inch to facilitate leaching.

#37 U.S. 2,494,259. "Fibrous Glass Articles".
Inventor: M. E. Nordberg.
Assignee: Corning Glass Works.
Filed: March 20, 1946. Issued: January 10, 1950.

This patent claims a number of particular compositions of glass fiber, and articles made from these fibers. The process involves leaching of the formed articles to extract the non-siliceous constituent.

#38 U.S. 2,577,936. "Colored Glass Fiber Product and Method for Making Same".
Inventor: J. H. Waggoner.
Assignee: Owens-Corning Fiberglas Corp.
Filed: May 7, 1949. Issued: Dec. 11, 1951.

Claims a method of coloring glass fibers which consists in reacting the fiber surfaces with the vapors of a decomposable salt with polyvalent metal whose oxides are colored.

#39 U.S. 2,593,817. "Colored Glass Fiber Product and Method for Making Same".
Inventor: J. H. Waggoner.
Assignee: Owens-Corning Fiberglas Corp.
Filed: June 1, 1949. Issued: April 22, 1952.

The patent claims a technique for coloring glass fibers by coating with polysiloxane and an organo-metallic compound, capable of producing colored reaction product on decomposition at elevated temperature.

(c) Other Methods of Fiberization

U.S. 2,593,818. "Colored Glass Fiber Product and Method for Making Same"
Inventor: J. H. Waggoner
Assignee: Owens-Corning Fiberglas Corporation
#40 Filed: June 1, 1949. Issued: April 22, 1952

A method of coloring glass fibers is claimed in which the fibers are coated with colored metal oxides in finely divided form, and then heat treated at just below the fusion temperature of the glass.

U.S. 2,754,224. "Method of Treatment of Glass Fibers with Colloidal Silica and Product Produced Thereby"
Inventor: R. F. Caroselli
Assignee: Owens-Corning Fiberglas Corporation
#41 Filed: January 31, 1952. Issued July 10, 1956

This patent applies primarily to coating and coloring fibers used for fabric production. In essence the treatment consists of applying colloidal silica to the glass fibers and following this by a treatment at 50° to 400° F. below fusion temperature of the glass. Numerous variants of the process are described together with methods of producing various colorations

U.S. 2,823,117. "Glass Paper-Calcium Silicate".
Inventor: D. Labino
Assignee: L.O.F. Glass Fibers, Inc.
#42 Filed: November 23, 1953. Issued: February 11, 1958

Claims a novel process for the production of glass fibers. The starting material is an alkali silicate glass fiber. This is leached with a solution of a soluble metallic salt with the result that the alkali present in the fiber is exchanged for the metal oxide. Useful salts are those which have an anion which forms a soluble salt with the metallic constituent of the leached alkali oxide. Leaching should be accomplished within 48 hours of blowing the fiber. A suitable glass contains 74.5% silica and 25% sodium oxide and the fiber diameter is in the range of 0.002 to 0.004 in. The fibers are heated in a hot blast having a temperature in the range of 3000 to 3300°F. and a velocity in the range of 1600 to 2000°f.p.s. This further attenuates the fibers to a diameter of 0.01 to 1.0 micron. The fibers are collected on a screen and leached for 15 minutes within 48 hours. The preferred reagent is boiling 5% calcium chloride solution. The fiber is washed and dried at 100°C. The resulting composition is about 74½% silica and 5.9% lime, together with small amounts of other oxides and some water.

(c) Methods of Fiberization (Cont.)

U.S. 3,023,115. "Refractory Material"
Inventor: E. Wainer, etal
Assignee: Horizons, Inc.
43 Filed: February 3, 1960. Issued: February 27, 1962.

One of several patents issued in the name of Horizons, Inc. relating to the formation of alumina fibers from the vapor phase. The present patent describes a method of considerably improving the yield, over that obtained using the technique described in U.S. Patent No. 3,082,051 (No. 29). In the presently described technique a gaseous sub-oxide of aluminum is reacted with silicon monoxide vapor in an atmosphere of hydrogen, maintained in a reaction zone held between 1300° and 1750°C. The aluminum sub-oxide is present in substantial excess over two moles for each three moles of silicon monoxide. In a modification of the process, molten aluminum is present in the heated reaction zone and is maintained between 1200° and 1500°C. The fiber generated obtains 95 to 99% by weight of alumina and 1 to 5% by weight of silica. The hydrogen may contain up to 5 p.p.m. of water vapor. Silicon metal formed may be recirculated. Fibers containing 95 to 99% alumina are claimed.

U.S. 3,092,531. "Process of Making Essentially Pure Silica Fiber Bats"
Inventor: D. Labino
Assignee: Johns-Manville Fiberglass, Inc.
Filed originally May 6, 1953
#44 Filed: February 1, 1956. Issued June 4, 1963

Describes a process for forming a fibrous bat of nearly pure silica. A compacted bat of siliceous fibers consisting of silica and an alkaline metal oxide in the ratio 4:1 (fiber diameter less than 2½ microns) is treated with aqueous leaching solution till all the alkali has been removed. Phosphoric acid is recommended.

U.S. 3,161,473. "Method of Making Beta-Silicon Carbide Fibers"
Inventor: W. W. Pultz
Assignee: Corning Glass Works
#45 Filed: June 6, 1962. Issued: December 16, 1964

An allegedly simple method for the manufacture of silicon carbide fibers is claimed in which carbon and silica in a mole ratio of 1:1 to 3.5:1, are heated in vacuo (less than 300 microns pressure), to a temperature between 1375 and 1550°C. for 1 to 50 hours. An atmosphere of cracked ammonia is introduced into the furnace during the heating process at a pressure between 400 and 700 mm. of mercury. Fibers form when the pressure ratio of hydrogen to nitrogen is varied between 2:1 and 9:1. High carbon to silica ratios up to 10:1 may be utilized if large fibers are required. Considerable information is furnished on properties of the product, reaction rates, etc.

U.S. 3,401,423. "Apparatus for the Continuous Formation of Filaments"
Inventor: R. L. Hough
Assignee: United States of America
Issued: September 17, 1968. Filed: May 7, 1965

This patent describes a novel method for the production of high temperature refractory filaments. The method is particularly directed towards the formation of fibers in materials such as pyrolytic graphite, borides, carbides and nitrides. The equipment consists of a reaction chamber in the middle of which there is a rotating disc which is heated if necessary. The chamber itself is evacuated to a few millimeters of mercury pressure. Electrodes are located at one or more points near the periphery of the disc. Ion-laden plasma containing the elements from which the refractory is made is blown through a nozzle on to the periphery of the disc at one or more locations distant from the electrode locations. In one instance, the plasma contained a mixture of titanium tetrachloride, nitrogen and hydrogen which led to the formation of titanium nitride. The refractory is formed on the disc at the point of impingement of the plasma as a result of a "plating" process. The formed refractory build-up on the disc then moves away from the plasma nozzle and after cooling somewhat passes through the arc. This causes removal of the refractory and fiberization; the continuous filament of refractory thus formed, is wound on a take up roll.

(c) Articles Made From Fibers and Their Manufacture: (See Nos. 44, 9, 11, 17)

U.S. 1,913,242. "Inorganic Resilient Fibrous Mass"
Inventor: B. C. McClure
Assignee: Gustin-Bacon Mfg. Co.
#46 Filed: July 16, 1931. Issued: June 6, 1933

This patent covers felted blankets made from fibers containing 58 to 72% silica and about 22 to 36% of alkali metal and alkaline earths. In some cases the compositions range up to 90% silica and less than 20% alkaline earth. Some calcium oxide is present in the fiber. The fiber itself is made by melting silica, soda, and lime and blowing this to form a fiber. The method of making the fiber is not claimed, nor is the felting technique.

U.S. 2,264,345. "Method of Making Slivers"
Inventor: J. L. Tucker, et al
Assignee: Owens-Corning Fiberglas Corporation
#47 Filed: November 20, 1935. Issued: December 2, 1941

This patent describes an elaborate machine for converting individual fibers of fiberglass, etc. into strands or slivers, which may then be used for the manufacture of textiles, etc. The process starts with fibers in the molten state

U.S. 2,633,428. "Process of Removing Size from Glass Fibers and the Subsequent Coating Thereof".
Inventor: G. Klug
Assignee: Alexander H. Kerr
#48 Filed: January 22, 1949. Issued March 31, 1953

In the manufacture of glass fabrics, size is often incorporated in the fibers to hold filaments together in strands. This must sometimes be later removed, and a heat treatment process for doing this is claimed.

U.S. 2,692,220. "Method for Making Glass Paper"
Inventor: D. Labino
Assignee: Glass Fibers, Inc.
#49 Filed: November 19, 1951. Issued October 19, 1954

Describes a method for the production of glass paper in which molten glass is fiberized employing techniques which yield fibers of closely controlled diameter and length. Hot gases are used in the blowing process. The fibers are loosely air felted onto a porous wire belt. The mat thus produced is passed through an acid bath having a pH between 2 and 6. This improves the matting of the fiber and densifies the mat. The mat is finally dried. The process is continuous. Resins may be incorporated in the paper as desired. The glass fibers have a diameter of 1 $\pm$ 0.45 micron.

U.S. 2,444,347. "Method of Treaing Glass Wool and Product Resulting Therefrom".
Inventor: H. H. Greger and R. F. Remler
Assignee: Briggs Filtration Co.
Filed: June 2, 1944. Issued: June 29, 1948

Claims a method for bonding glass wool with aluminum phosphate. The aluminum phosphate is in the colloidal form and additional material such as silica and other fillers, and reactive mterials such as talc, vermiculite asbestos, clays, bauxite, etc. and plasticizers such as glycerine, mineral oil and glucose may also be present. Wetting agents may be helpful in some cases. The aluminum phosphate is preferably either mono-aluminum phosphate or sesqui-aluminum phosphate. Additives containing $SO_4$, Cl or $NO_3$ ions may be necessary to insure maintenance of a colloidal solution. The binders are set by heating.

U.S. 2,649,388. "Manufacture of Silica Filled Materials:
Inventors: J. H. Wills and J. F. Hazel
Assignee: Philadelphia Quartz Co.
Filed: August 24, 1948. Issued: August 18, 1953.

The patent describes a process for impregnating materials with colloidal silica. The mixture of materials is frozen under conditions which produce complete freezing in less than an hour. Before any substantial gel formation takes place, the frozen mass is thawed. An irreversible reaction takes place as the result of which precipitation of silica occurs on the materiel. According to the specification, freezing is conveniently affected by addition of solid carbon dioxide. Rapid freezing is essential owing to the instability of the silica sol. Allegedly, the process has many applications, particularly in the rubber field; other applications include the bonding of cotton, woolen paper machine felt, asbestos fibers, asbestos sheet, cellulose acetate, kaolin clay, pigments, rayon, linen, wool, nylon, clay, carbon, metal oxide and many other materials. In many cases, the colloidal silica was generated in-situ from sodium silicate. Various methods of doing this are described in the examples.

3. Articles made from Fibers and their Manufacture (Cont.)

U.S. 2,693,668. "Poly-Phase Systems of Glassy Materials"
Inventor: G. Slayter
Assignee: Owens-Corning Fiberglas Corporation
#50 Filed: April 3, 1951. Issued: November 9, 1954

Claims a composite structure consisting of inorganic fibers bonded with fused glass. Numerous modifications of the basic invention are described utilizing various materials. In the practice of the invention, the fibers are formed from a glass. The two glassy materials must have similar working temperatures.

U.S. 2,731,359. "Refractory Fiber Body and Method of Making Same."
Inventor: K. C. Nicholson
Assignee: Carborundum Company
#51 Filed: August 22, 1952. Issued: January 17, 1956

The patent claims Fiberfrax fibers bonded with silicon nitride, or silicon carbide. Both composition and method claims are included in the patent. In the process described, silicon metal powder is mixed with the refractory fibers and the mixture heated in a nitrogen-containing atmosphere. Alternatively, a carbonaceous atmosphere is used if silicon carbide is desired as the bond. Bonding takes place at 1350 to 1400°C. (silicon nitride) or at 1250°C. (silicon carbide).

U.S. #2,757,426. "Method of Making Mold with Fibrous Liner"
Inventor: J. B. Brennan
Assignee: None
#52 Filed: October 3, 1951. Issued August 7, 1956

Claims a method of making metal castings which utilizes a fibrous mold liner. The liner is made by felting or otherwise fibers such as asbestos or fiber-glass to a shape which conforms to the interior of the mold cavity. The fibrous layer may be formed directly onto a porous metal-casting mold (e.g. one made from porous carbon or tungsten carbide or sintered metal aggregate); alternatively a separate former may be used and the liner backed up with any conventional foundry material. Spraying is recommended as a technique for making the liner. A binder such as plaster of Paris is present. Vacuum is generally used to dewater the liner. Pressure may also be employed to compact it. Desirable coatings may be applied to the interior surface of the liner and additional shaping operations may be used. May be useful prior art against more recent patents.

3. Articles made from Fibers and their Manufacture (Cont.)

U.S. 2,808,338. "Thermal Insulating Bodies and Method of Manufacture"
Inventor: A. J. Bruno
Assignee: Johns-Manville Corporation
#53 Filed: December 18, 1952. Issued: October 1, 1957

Claims insulating shapes made from inorganic fibers and inorganic aerogel particles containing 2 to 35% of water. The fibers are present in amounts up to 15% by weight and the aerogel in amounts up to between 45 and 95% by weight of the product. An opacifier may be present to improve effective thermal conductivity at high temperature. In particular examples, the fiber is amosite asbestos and the aerogel is silica aerogel. The mixture is moldable without addition of a fluid and the insulating body produced can have a density between 12 and 30 lbs./cu. ft. Thermal conductivity is less than 0.4 (units not specified) at 350°F. Phenol-formaldehyde resin is used as a temporary binder.

U.S. 2,881,457. "Inorganic Bonded Thermal Insulating Bodies and Method of Manufacture"
Inventor: S. Speil and I. Barnett
Assignee: Johns-Manville Corporation
#54 Filed: December 18, 1952. Issued: October 29, 1957

The patent describes a method for the ceramic bonding of asbestos and other inorganic fibers. The method described is generally similar to that described in U.S. 2,808,338 (No. 53). The main difference in the claims is that specific reference is made to the presence of a ceramic binder. This binder is derived from impurities in the form of soluble salts such as sodium sulfate, which are present in the aerogel particles. When the molded shape is heated to a sufficient temperature, these impurities form a ceramic bond, probably as the result of sintering of the aerogel or incipient ceramic reaction between the aerogel and the salts. The specification makes reference to very low thermal conductivities of the order of less than 0.55 (units not stated) at 1000°F. and densities of the order of 10 lbs. per cubic ft. Use of a PF resin temporary binder is unnecessary with the present technique, but is required in the technique described in U.S. 2,808,338 (No. 53).

U.S. 2,919,211. "Evaporator Plate and Method of Producing Same".
Inventor: D. Labino
Assignee: L.O.F. Glass Fibers Co.
#55 Filed: December 30, 1954. Issued: December 29, 1959

The patent claims a novel evaporator plate and process of producing it which involves formation of glass fibers into a mat, spraying with water to cause inter-felting of the fibers, removal of most of the water, and the addition of colloidal silica or colloidal alumina and final drying. In the process described, the glass fibers are air-felted immediately following the blowing process. The process envisaged is continuous. About 5% colloidal silica is used in the bonding operation. (Note: that this is the earliest reference so far found in the bonding of inorganic fibers with oolloidal silica, etc.).

3. Articles made from Fibers and their Manufacture (Cont.)

U.S. 3,001,362. "Insulator for Rocket Motor"
Inventor: D. A. Runton
Assignee: The Russell Mfg. Co.
Filed: July 26, 1957. Issued September 26, 1961

Claims an insulator for a rocket motor consisting of a bonded refractory fiber reinforced by numerous columns of refractory ( zirconium) cement extending through the bonded fibers.

U.S. 3,002,857. "High Temperature Inorganic Binder and Products Produced With Same". Inventor: J. Stalego. Assignee: Owens-Corning
Filed: November 14, 1955. Issued October 3, 1961

The patent claims a method of bonding fiberglass with sodium silicate from the group consisting of boric acid, borate, or acid salts of boric acid; these materials are present in the ratio 1 part of sodium silicate to 0.5 69 10 parts of the compound. The sodium silicate has a soda to silica ratio of less than 1 to 10 and is formed by reaction of commercial sodium silicate with water-soluble inorganic fillers such as the carbonates, hydroxides or oxides of aluminum, calcium, magnesium, sodium, iron, lead, zinc, copper, cadmium, tin, antimony, beryllium, molybdenum, chromium and clay may be present. The ratio of filler to sodium silicate varies from 1:1 to 10:1. The fiber used is normally prebonded with 3½ to 6.47% resin. Technical criteria, which have to be met if glass fibers are to be successfully bonded, are discussed in some detail. Bonding is accomplished by heating the material from 350° to 500°F. for 10 to 30 minutes. The purpose of the oxide addition is to neutralize or buffer the sodium ions to reduce their activity and form silicic acid and other compounds of a glass forming type at elevated temperatures.

U.S. 3,017,318. "High Temperature Resistant Siliceous Compositions and Method of Producing".
Inventor: D. Labino, etal
Assignee: Johns-Manville Corporation
Filed: November 2, 1957. Issued: January 16, 1962

Covers the composition and manufacture of board-like shapes for high temperature service from fibers containing from 50 to 98% silica, a granular filler such as talc, pyrophyllite, clay, flint, fluorspar, gypsum, mica or asbestos and an inorganic bonding agent such as colloidal silica or colloidal alumina. The filler amounts to 5 to 90% of the weight of the fibers. The amount of colloidal inorganic binder on a solids basis varies between 40 and 80% of the weight of the felt. The specification includes a full description of the equipment used in the production of the boards, and of the production of the fibers. The latter are produced by fiberizing an alkali silicate glass, and leaching this with acid to remove the soda. Glass of composition 72 to 80% silica and 20% alkaline metal oxide (preferably $Na_2O$) is recommended. After leaching with acid, these fibers are nearly pure silica. Production of the boards is accomplished by normal felting techniques. In the recommended practice, boards are felted without binder and are subsequently sprayed with colloidal silica or colloidal alumina. Particle size of the fillers should be in the range 80 to 300 mesh and preferably 80 to 140 mesh. Recommended drying temperature range is 100 to 250°F. A low drying temperature is advised to minimize migration of volloidal silica to the surface; in particular the range 110 to 120°F. is mentioned. The density of the dried board varies considerably from about 16 to 95 lbs. per cubic foot depending upon composition.

3. Articles Made from Fibers and their Manufacture (Cont.)

U.S. #3,022,213. "Conductive Web and Method of Making Same".
Inventor: D. K. Pattiloch, etal
Assignee: Michigan Research Laboratories & Electrochem Fiberseal Corporation
#59 Filed: February 13, 1958. Issued: Feb. 20, 1962

Claims a method of producing an electrically conducted web by the treatment of an aqueous suspension of fiber with a dicyandiamide-formaldehyde condensation product in the amount of 0.5 to 5%, and subsequently adding to the suspension aqueous colloidal silica containing 1 to 40% of silica, and finally adding finely divided conductive material in amounts up to 300%. The resulting suspension is felted into a web. The dicyandiamide-formaldehyde condensate is Lyofax SBK (Ciba Co.). The fibrous material may be short fibered asbestos or cellulose for low temperature applications. Glass fibers may also be employed. If incorporation of a laminating resin is desired, a powdered resin (B-stage phenolic) is dispersed in the colloidal silica.

U.S. #3,077,413. "Ceramic Fiber Products and Method and Apparatus for Manufacture Thereof".
Inventor: R. A. Campbell
Assignee: Carborundum Company
#60 Filed: ~~October~~ February 27, 1957. Issued February 12, 1963.

The claims cover articles of ceramic fibers bonded with at least 10% colloidal silica; casting molds consisting essentially of a hardened layer of the bonded fiber are also covered. This appears to be a basic patent covering the use of colloidal silica to make a rigid product from ceramic fiber, although use of colloidal silica is described in the prior art in connection with fiberglass. (U.S. No, 2,754,244 - No. 41.) Incorporation of the colloidal silica in the fiber by mixing, dipping, or spraying is described. Numerous methods for forming the product to shape are mentioned besides felting. The specification includes a good deal of information on the ratio of silica to fiber and its influence on the properties of the product. Typically, the mixes described contain about 30% of free silica derived from the colloidal silica in the finished product. Manufacture of mat by a technique similar to that used in paper making is described in some detail. Recommended drying temperature is 400 to 700°F. The dry product may be sprayed with additional colloidal silica to yield a smooth surface. Manufacture of shaped articles in metal molds lined with aluminum foil is discussed. Mention is made of a sandwich product consisting of fiberglass sheet lined on both sides with ceramic fiber sheets. Among other modifications are the following: glazing of the surface of the dry product by fusing in a kiln, reinforcement with wire screening, etc., incorporation of additives such as plaster of Paris, graphite, mica, powdered asbestos, cement, clay glass, metals, corundum and quartz. Mention is also made of the use of the fiber to produce reuseable molds for metal casting production. These molds are formed by pressing the wet mat around a pattern and drying in situ; these molds have their principal utility in the casting of aluminum and brass.

U.S. 3,024,145. "Process of Bonding Glass Articles"
Inventor: R. F. Nickerson
Assignee: Monsanto Chemical Co.
Filed: May 1, 1957. Issued: March 6, 1962.

Claims a process for bonding glass fiber insulation bat and other articles with silica sol and a water soluble organic polymer. The silica sol has a pH of about 8.5 to 10.5 and a $SiO_2/M_2O$ ratio of 50/1 to 200/1 (M is monovalent alkali cation); the sol contains colloidal silica particles in the size range 5 to 175 millimicrons. The organic polymer must be water soluble, non-ionic and film-forming and is present in from 5 to 100% by weight of the weight of the silica in the aquasol. Best results are obtained with polymers whose molecular weight is in the range 5,000 to 100,000. Polyvinyl alcohol is claimed in particular, as also are methyl-cellulose and dextrine; starches may also be used. The product is subsequently dried at a temperature sufficient to remove the organic polymer and its heat decomposition products; there is thus left a mat of glass fibers bonded with substantially pure silica. The solutions may be applied to the fibers by any convenient means such as immersion, dipping, spraying, etc. For most purposed, the binder solution should contain between 5 and 15% of solloidal silica by weight.

U.S. 3,092,247. "Refractory-Forming Products"
Inventor: R. K. Woodruff
Assignee: Refractory Products Co.
Filed: October 11, 1960. Issued: June 4, 1963.

This is the well-known patent covering "wet-pack" refractory-forming units which consist of alumino-silicate fiber mat and an unset high-temperature inorganic liquid binder; auxiliary organic binders may also be present. The inorganic binder as the specification indicates is usually colloidal silica but alternatives such as colloidal alumina, colloidal zirconia and sodium silicate are also mentioned. The claims do not restrict any particular refractory fiber, although a proportion of "alumina" fibers must be present. Materials such as mineral wool, asbestos fiberglass alumina, beryllia, zirconia, titania, slag wool and quartz fibers are quoted in the specification. The other novel feature of the invention is that the mat having been soaked in the inorganic binder is packed in a loose vapor-tight plastic envelop. In the manufacturing process described, a slurry of colloidal silica and fiber containing 22% solids is first made. The mat is felted out and after it is withdrawn from the tank, suction is maintained until the water content of the mat drops to about 20% by weight; at this stage the colloidal silica content of the mat is about 5% by weight. The mat is then sealed in a suitable impermeable bag, which may be plastic, rubber or even aluminum foil. The binder may be introduced into the mat after its formation, if this is preferred. The process may also be used with organic solvents, for example, polyvinyl acetate binder dispersed in trichlorethylene. Among ten auxiliary organic binders mentioned in the specification are materials such as casein, animal glue, zein, dextrin, shellac and asphalt; alternatively synthetic resins of almost any kind may be employed. Among envelope materials mentioned as suitable are polyethylene, cellulose acetate, regenerated cellulose (cellophane), polystryrene, polyvinyl chloride, rubber hydrochloride, polyvinylidene, methylmethacrylate. The specification also describes steps in utilizing the product. The mat is withdrawn from the plastic envelope, shaped to the desired form and retained in position until the binder hardens. Heat is applied to hasten this, and if necessary, the hardened mat can be rewetted with additional binder and solution to improve its strength.

FOS-00-0000001418

3. Articles Made from Fibers and their Manufacture (Cont.)

U.S. #3,100,734. "Furnace Combustion Chamber".
Inventor: J. P. Rex, Jr. etal
Assignee: Rex Roto Corporation
#61 Filed: September 23, 1960. Issued: August 13, 1963

Claims a fibrous refractory combustion chamber bonded with inorganic refractory colloidal binder. Specifically the fiber is alumino-silicate and the binder is colloidal silica. It is suggested that a secondary binder such as vinyl-phenolic polyester or epoxy resin may be used to reduce the amount of the expensive colloidal silica.

U.S. #3,118,807. "Bonded Fibrous Insulation".
Inventor: H. G. Holcomb
Assignee: Johns-Manville Corporation
#62 Filed: February 25, 1961. Issued October 21, 1964

There is claimed a lightweight refractory thermal-insulating blanket useable up to 2000°F. containing 80 to 85% of refractory mineral fiber felt and 15 to 20% of binder. The latter consists essentially of 10 to 15% of bentonite, 3 to 8% of fine ground soda-lime-silica glass, 2 to 7% of thermosetting resin; a PF resin is preferred. Claims also cover combustion chamber liners made from the above material. Combustion chamber liners are ultimately fired at 1600°F.

U.S. #3,224,927. "Forming Inorganic Fiber Material Containing Cationic Starch and Colloidal Silica".
Inventor: R. L. Brown and G. W. Cerose
Assignee: E. I. Dupont Company
#63 Filed: October 4, 1963. Issued: December 21, 1965

The claim covers a process for making products with inorganic fiber, selected from asbestos, paligorskite, attapulgite, quartz glass, alumina silicates, potassium titanate. The nub of the invention is the use of positively charged starch and colloidal silica as a binder. Starch content is 0.2 to 1.5% of the fiber content and preferably not more than 1%; colloidal silica content ranges between 10 and 20% of the weight of the fiber. The pH is controlled in the range 3 to 5. The positively charged colloidal starch first adsorbs on the surface of the fibers and then subsequently attracts the negatively charged silica particles to itself. The Cato starches of National Starch Products, Inc. are especially recommended. These starches can also be made by reacting ungelatinized starch with an etherifying agent, containing a tertiary amino radical (U.S. Patent #2,813,093). The starch also imparts considerable green strength. In practice, the starch is added first to the fiber slurry and stirred; the colloidal silica is added later after the starch has been adsorbed onto the fiber. The slurry is then acidified. The slurry is not felted for at least five minutes after acidification. All but one of the examples refer to use o the process for the treatment of asbestos fibers; the last example refers to alumino-silicate fibers.

3. Articles Made from Fibers and their Manufacture (Cont.)

U.S. #3,231,401. "Refractory Composition"
Inventor: C. E. Price and C. B. Walworth
Assignee: Carborundum Company
Filed: June 22, 1964. Issued: January 25, 1966
#64 C.I.P. of original application filed: February 3, 1958

All claims relate to a refractory composition containing 60-97% of ground (ball-milled) inorganic ceramic fibrous material, and a colloidal inorganic oxide from the group silica, zirconia, alumina, the oxide constituting from 3 to 40% of the product. The ground fibrous material consists of short fibers having a mean diameter in the range 2.5 to 20 microns, and length to diameter ratio from 10:1 to 50:1. Some claims refer to incorporation of 58 to 75% of non-fibrous particles. Other claims include a proportion (10 to 50%) of longer (staple) fiber. Alumino-silicate fibers are specifically referred to. Use of a suspension agent (bentonite clay) is mentioned. A material known as Veegum T, a magnesium alumina silicate, sold by the R. T. Vanderbilt Company, Inc. is particularly recommended as a suspension agent although not claimed. A novel feature of the invention appears to be the fiber length and fiber length to diameter ratio. Short fibers are said to confer exceptional strength on the product. Thermal shock resistance is good and the material can withstand temperatures of 2300°F. or higher. Mention is made of the part played by surface energy forces.

U.S. #3,253,936. "Castable Fibrous Refractory Compositions and Articles Obtained Therefrom".
Inventor: H. F. Weindel
Assignee: Socony Mobile Oil Co., Inc.
#65 Filed: June 10, 1963. Issued May 31, 1966

Claims relate to castable compositions and to the process for making shaped refractory articles. Compositions contain 20 to 90% of alumino-silicate or potassium titanate fiber, 2 to 60% of calcium aluminate hydration binder, 2 to 40% of colloidal silica (15 to 55% silica) and water in sufficient amount to form hydration products with the calcium aluminate. The process claims cover pouring the above mixture into molds, and allowing them to set. Fibers specifically mentioned are "Fiberfrax 100" and "Tipersul". Advantages claimed for the products made in this way include shrinkage less than 3% , short setting time, conventional simple technique, good mechanical properties. The product is dried at 100°F.

U.S. 3,269,849. "Fibrous Refractory"
Inventor: M. J. Caprio and R. F. Krisleit
Assignee: Alcoa
#66 Filed: December 11, 1964. Issued: August 13, 1966

Claims a fibrous heat insulator consisting of 40 to 60% of asbestos fiber, 25 to 20% of calcium aluminate cement, 5 to 2.5% of cryolite, 30 to 20% fibrous material, which may be alumino-silicate fiber or potassium titanate. The composition has a thermal conductivity of 2 to 4 BTU/hr./Ft.$^2$/°F./in. The preferred composition range is 45 to 55 of asbestos, 23 to 21 of calcium aluminate, 3.5 to 2.5% of cryolite, 28 to 22% of fiber. A method of manufacturing refractory slabs of the material is also claimed in which an addition of 300% of water is added to the above mixture and the resultant slurry is compressed under 1000 p.s.i.; this is followed by curing at 1400°F. for two hours for inch thickness. It may be advantageous to autoclave the slab prior to curing. The product is intended to resist temperatures up to 1000°C.

3. Articles Made from Fibers and their Manufacture (Cont.)

U.S. #3,294,562. "Refractory Composition"
Inventor: M. J. Caprio and R. F. Krisleit
Assignee: Alcoa
#67 Filed: December 11, 1964. Issued: December 27, 1966

This patents claims differ from that of the preceding patent only in that the cryolite is omitted. Preferred composition range is abestos 45 to 55%, calcium aluminate cement 28 to 22%, inorganic fiber 28 to 22%.

U.S. 3,377,229. "Artificial Logs for Fireplaces"
Inventor: L. E. Bryan
Assignee: Gem Clay Forming, Inc.
#68 Filed: February 12, 1965. Issued: April 9, 1968

The patent describes the use of alumino-silicate fibers bonded with colloidal silica in the manufacture of artificial logs for fireplaces.

U.S. 3,394,913. "Insulating Riser Sleeve Composition"
Inventor: M. E. Binkley.
Assignee: Johns-Manville Corporation
#69 Filed: October 23, 1965. Issued: July 30, 1968

Claims riser sleeves consisting essentially of asbestos fiber (5-20%), bentonite (5-20%), and diatomaceous earth (50-90%), plus a binder which is either calcium or basic magnesium carbonate (6-30%). The sleeves are said to be reusable. Sleeves are made by a felting process on a slurry which is heated to between 100°F. and 212°F. for at least 30 minutes prior to felting. After felting sleeves are fired at about 1400°F. for more than three hours. The sleeves are said to be satisfactory with aluminum, iron, stainless steel and brass. It is recommended that the sleeves be made in two halves which are joined bybanding with cloth, tape, plastic or steel strapping.

U.S. 3,395,068. "Laminated Heat Insulative Product"
Inventor: John P. Rex, Jr.
Assignee: Rex Roto Corporation
#70 Filed: June 26, 1964. Issued: July 13, 1968

Claims a duplex fiber product intended primarily for lining combustion chambers consisting of alumino-silicate fiber on the hot face backed by mineral wool, asbestos or glass fiber. Both materials are bonded with colloidal silica. The high temperature fiber is felted on to the tool first and is dewatered. The tool is then submerged in a second slurry containing inexpensive fiber and the operation repeated. It is stated that a very thin layer of alumino-silicate fiber is sufficient for a combustion chamber. Paper may be incorporated in the low temperature fiber layer to improve strength; alternatively this layer may be coated on the outside with a clay coating by spraying.

FOSECO, INC. RECIPE AND COST SHEET CONFIDENTIAL

| | | | |
|---|---|---|---|
| DATE | 11/5/68 | PRODUCT | E.P. 3900A/PROFAX |
| ORIGIN | USA | CLASS | |
| PREVIOUS ISSUE | 7/24/68 - #1 | CODE | F0030 |
| FORM | Profax Rings | PROCESS | |

CHANGE Replace of regular asbestos (2%) with short fiber white asbestos (2%) to increase green strength.

DESCRIPTION High heat resistant PROFAX.

| CODE NO. | % | MATERIAL Price per 100 lb. | COST Per 100 lb |
|---|---|---|---|
| 131D | 71.0 | .~~0085~~ | .60 |
| 447 | 1.25 | 20.50 | .26 |
| 177A | 4.75 | 17.48 | .83 |
| 265A | 7.0 | 10.30 | .72 |
| X-1790 | 4.0 | 3.78 | .15 |
| X-1791 | 10.0 | 3.36 | .34 |
| 319 | 2.0 | 7.79 | .16 |

BULK DENSITY 0.72 - 0.82 g/cc.

WT. PER GALLON ______ LBS.

| PRICE/POUND | |
|---|---|
| Under 100 | |
| 100 | |
| 400 | |
| 2,000 | |
| 10,000 | |
| Truckload | |

| PRICE/GALLON | |
|---|---|
| 1-4/1-4 | |
| 5-19/5-50 | |
| 20-99/55-165 | |
| 100-499/165-210 | |
| 500 and Over | |

NOTES:

| | |
|---|---|
| TOTAL | 3.06 |
| Cost (102%) | 3.12 |
| Direct Labor | |
| Mfg.Overhead ( %) | |
| Packaging | |
| Quality Control | |

| CONTRIBUTION | 40% | 50% | 60% |
|---|---|---|---|
| Cost | | | |
| Other Var. Expense % | | | |
| Variable Expense | | | |
| Margin | | | |
| Selling Price | | | |

Recipe Costing Price

RECIPE REVISED
Date: 8-19-69
By: EH

F05-00-0000003316