(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property

Organization

International Bureau



(43) International Publication Date 27 June 2013 (27.06.2013)

- (51) International Patent Classification: D06M 13/282 (2006.01) D06M 11/44 (2006.01) D06M 11/77 (2006.01)
- (21) International Application Number: PCT/US20 12/070660
- (22) International Filing Date:
 - 19 December 2012 (19. 12.2012)
- (25) Filing Language: English
- (26) Publication Language: English
- (30) Priority Data: 61/577,320 19 December 201 1 (19. 12.201 1) US
- (71) Applicant: UNIFRAX I LLC [US/US]; 2351 Whirlpool Street, Niagara Falls, NY 14305-2413 (US).
- (72) Inventors: ZOITOS, Bruce, K.; 48 Tristan Lane, Williamsville, New York 14221 (US). ANDRE JCAK, Mi chael, J.; 137 Koster Row, Amherst, New York 14226 (US).
- (74) Agents: CURATOLO, Joseph, G. et al; 24500 Center Ridge Road - Suite 280, Cleveland, OH 44145 (US).

(10) International Publication Number

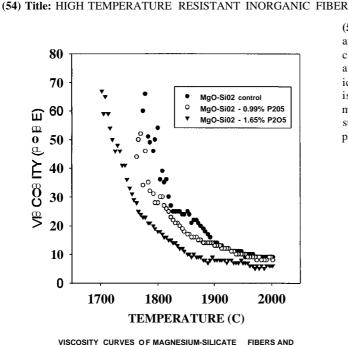
WO 2013/096471 Al

- (81) Designated States (unless otherwise indicated, for every kind *d* national protection available): AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.
- (84) Designated States (unless otherwise indicated, for every kind *f* regional protection available): ARIPO (BW, GH, GM, KE, LR, LS, MW, MZ, NA, RW, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Published:

— with international search report (Art. 21(3))

[Continued on next page]



VISCOSITY CURVES OF MAGNESIUM-SILICATE FIBERS AND MAGNESIUM-SILICATE PHOSPHOROUS CONTAINING FIBERS

FIG.1

(57) Abstract: Provided is an inorganic fiber containing silica and magnesia as the major fiber components which further includes a phosphate additive to the melt of fiber ingredients, or as a coating on the surfaces of the fiber, or both. The inorganic fiber exhibits improved thermal performance properties and is non-durable in physiological fluids. Also provided are methods of preparing the inorganic fiber and of thermally insulating articles using thermal insulation prepared from a plurality of the inorganic fibers.



- before the expiration *f* the time limit for amending the claims and to be republished in the event *f* receipt *f* amendments (Rule 48.2(h))

HIGH TEMPERATURE RESISTANT INORGANIC FIBER

TECHNICAL FIELD

10

15

5

[0002] A high temperature resistant inorganic fiber that is useful as a thermal, electrical, or acoustical insulating material, and which has a use temperature of 1400°C and greater is provided. The high temperature resistant inorganic fiber is easily manufacturable, exhibits low shrinkage after prolonged exposure to the use temperature, retains good mechanical strength after exposure to the use temperature, and is soluble in physiological fluids.

BACKGROUND

[0003] The insulation material industry has determined that it is desirable to utilize fibers in thermal, electrical and acoustical insulating applications, which are not durable in physiological fluids, that is, fiber compositions which exhibit a low biopersistence or a high solubility in physiological fluids. While candidate materials have been proposed, the use temperature limit of these materials have not been high enough to accommodate many of the applications to which high temperature resistant fibers, including synthetic vitreous fibers and ceramic fibers, are applied. Many other compositions within the synthetic vitreous fiber family of materials have been proposed which are non-durable or decomposable in a physiological medium.

[0004] The high temperature resistant fibers should also exhibit minimal linear shrinkage at expected exposure temperatures, and after prolonged or continuous exposure to the expected use temperatures, in order to provide effective thermal protection to the article being insulated.

5

10

[0005] In addition to temperature resistance as expressed by shrinkage characteristics that are important in fibers that are used in insulation, it is also required that the fibers have mechanical strength characteristics during and following exposure to the use or service temperature, that will permit the fiber to maintain its structural integrity and insulating characteristics in use.

[0006] One characteristic of the mechanical integrity of a fiber is its after service friability. The more friable a fiber, that is, the more easily it is crushed or crumbled to a powder, the less mechanical integrity it possesses. In general, inorganic fibers that exhibit both high temperature resistance and non-durability in physiological fluids also exhibit a high degree of after service friability. This results in the fiber lacking the strength or mechanical integrity after exposure to the service temperature to be able to provide the necessary structure to accomplish its insulating purpose. Other measures of mechanical integrity of fibers include compression strength and compression recovery.

20

25

15

[0007] Thus, it is desirable to produce an improved inorganic fiber composition that is readily manufacturable from a fiberizable melt of desired ingredients, which exhibits low shrinkage during and after exposure to service temperatures of 1400°C or greater, which exhibits low brittleness after exposure to the expected use temperatures, and which maintains mechanical integrity after exposure to use temperatures of 1400°C or greater.

DETAILED DESCRIPTION

[0008] Provided is a low shrinkage, high temperature resistant inorganic fiber having a 30 use temperature of 1260°C or greater, which maintains mechanical integrity after exposure to the use temperature, and which is non-durable in physiological fluids

PCT/US2012/070660

[0009] According to illustrative embodiments, provided is a low shrinkage, high temperature resistant inorganic fiber having a use temperature of 1400°C or greater, which maintains mechanical integrity after exposure to the use temperature, and which is non-durable in physiological fluids. The inorganic fiber comprises the fiberization product of a melt comprising about 65 to about 86 weight percent silica and about 14 to about 35 weight percent magnesia, and an addition of a phosphorous containing compound. The phosphorous containing compound may be incorporated throughout the fiber, or as a coating on at least a portion of the fiber, or both.

- 10 [0010] Also provided are methods for preparing a low shrinkage, high temperature resistant inorganic fiber having a use temperature of 1260°C, or greater, which maintains mechanical integrity up to the use temperature and which is non-durable in physiological fluids.
- 15 [001 1] According to illustrative embodiments, the method for preparing a low shrinkage, high temperature resistant inorganic fiber having a use temperature of 1400°C, or greater, which maintains mechanical integrity up to the use temperature and which is non-durable in physiological fluids. The method comprises forming a melt with ingredients comprising greater than about 65 to about 86 weight percent silica, about 14 to about 35 weight percent magnesia, and a phosphorous containing compound, producing fibers from the melt.

[0012] According to other illustrative embodiments, the method for preparing a low shrinkage, high temperature resistant inorganic fiber having a use temperature of 1400°C,
or greater, which maintains mechanical integrity up to the use temperature and which is non-durable in physiological fluids. The method comprises forming a melt with ingredients comprising greater than about 65 to about 86 weight percent silica and about 14 to about 35 weight percent magnesia and producing fibers from the melt. At least a portion of the resulting fibers are coated with a phosphorous containing compound. The inorganic fibers may be coated with the phosphorous containing compound at the point of fiberization or after fiberization.

10

30

PCT/US2012/070660

[0013] Also provided is a method for preparing a low shrinkage, high temperature resistant inorganic fiber having a use temperature of 1400°C, or greater, which maintains mechanical integrity up to the use temperature and which is non-durable in physiological fluids. The method comprises forming a melt with ingredients comprising greater than about 65 to about 86 weight percent silica, about 14 to about 35 weight percent magnesia, and a phosphorous containing compound, producing fibers from the melt, and coating at least a portion of the resulting fibers with a phosphorous containing compound. The inorganic fibers may be coated with the phosphorous containing compound at the point of fiberization or after fiberization. According to this method, the fibers include a phosphorous compound within the fiber and also on at least a portion of the exterior surface of the fiber.

[0014] According to an illustrative embodiment, the method for preparing a low shrinkage, high temperature resistant inorganic fiber comprises forming a melt with ingredients comprising greater than 65 to about 86 weight percent silica and about 14 to about 35 weight percent magnesia, producing fibers from the melt, and; coating at least a portion of the resulting fibers at the point of fiberization or after fiberization with a phosphorous containing compound.

20 [0015] Also provided is a method of insulating an article with fibrous insulation prepared from the inorganic fibers. The method includes disposing on, in, near or around the article, a thermal insulation material having a use temperature of 1260°C, or greater, which maintains mechanical integrity up to the use temperature and which is non-durable in physiological fluids, the insulation material comprising the fiberization product of a melt of ingredients comprising about 65 to about 86 weight percent silica, about 14 to about 35 weight percent magnesia, and a phosphorous containing compound.

[0016] Also provided is a method of insulating an article with fibrous insulation prepared from the inorganic fibers. The method includes disposing on, in, near or around the article, a thermal insulation material having a use temperature of 1400°C, or greater, which maintains mechanical integrity up to the use temperature and which is non-durable

in physiological fluids, the insulation material comprising the fiberization product of a melt of ingredients comprising about 65 to about 86 weight percent silica, about 14 to about 35 weight percent magnesia, and a phosphorous containing compound.

5 [0017] According to alternative embodiments, the method of insulating an article includes disposing on, in, near or around the article, a thermal insulation material having a use temperature up to at least 1260°C, or greater, which maintains mechanical integrity up to the use temperature and which is non-durable in physiological fluids, said insulation material comprising the fiberization product of a melt of ingredients comprising greater than 71.25 to about 86 weight percent silica and about 14 to about 35 weight percent magnesia, and a coating of a phosphorous containing compound.

[0018] According to alternative embodiments, the method of insulating an article includes disposing on, in, near or around the article, a thermal insulation material having a use temperature up to at least 1400°C, or greater, which maintains mechanical integrity up to the use temperature and which is non-durable in physiological fluids, said insulation material comprising the fiberization product of a melt of ingredients comprising greater than 71.25 to about 86 weight percent silica and about 14 to about 35 weight percent magnesia, and a coating of a phosphorous containing compound.

20

25

[0019] According to alternative embodiments, the method of insulating an article includes disposing on, in, near or around the article, a thermal insulation material having a use temperature up to at least 1400°C, or greater, which maintains mechanical integrity up to the use temperature and which is non-durable in physiological fluids, said insulation material comprising the fiberization product of a melt with ingredients comprising greater than about 65 to about 86 weight percent silica, about 14 to about 35 weight percent magnesia, and a phosphorous containing compound, producing fibers from the melt, and coating at least a portion of the resulting fibers with a phosphorous containing compound.

[0020] Also provided is an inorganic fiber containing article, as described above, comprising at least one of bulk fiber, blankets, needled blankets, papers, felts, cast shapes, vacuum cast forms, or compositions.

5 [0021] FIG. 1 is a viscosity vs. temperature curve of a melt chemistry for a commercially available magnesium-silicate fiber and magnesium-silicate fiber which includes a phosphorous containing compound.

[0022] FIG. 2 is a graph showing the dissolution rate of magnesium-silicate fibers which 10 include varying amounts of a phosphorous containing compound.

[0023] FIG. 3 is a graph which shows the linear shrinkage at 1260°C of magnesium-silicate fibers which include varying amounts of a phosphorous containing compound.

15 [0024] FIG. 4 is a graph which shows the linear shrinkage at 1400°C of magnesium-silicate fibers which include varying amounts of a phosphorous containing compound.

[0025] FIG. 5 is a graph which shows the linear shrinkage at 1500°C of magnesium-silicate fibers which include varying amounts of a phosphorous containing compound.

20

[0026] FIG. 6 is a graph which shows linear shrinkage at 1400°C of magnesium silicate fibers prepared from about 75 to about 79 weight percent silica and which include varying ranges of a phosphorous containing compound.

25 [0027] FIG. 7 is a graph showing the dissolution rate of magnesium silicate fibers prepared from about 75 to about 79 weight percent silica and which include varying amounts of a phosphorous containing compound.

[0028] FIG. 8 is a graph which shows linear shrinkage at 1260°C of magnesium silicate
30 fibers prepared having different target levels of silica and which include varying ranges of a phosphorous containing compound.

[0029] FIG. 9 is a graph which shows linear shrinkage at 1400^oC of magnesium silicate fibers prepared having different target levels of silica and which include varying ranges of a phosphorous containing compound.

5 [0030] FIG. 10 is a graph which shows linear shrinkage at 1400°C of high aluminacontaining magnesium silicate fibers prepared from about 75 to about 79 weight percent silica and which include varying ranges of a phosphorous containing compound.

[0031] FIG. 11 is a graph showing the dissolution rate of high alumina-containing
magnesium silicate fibers prepared from about 75 to about 79 weight percent silica and which include varying amounts of a phosphorous containing compound.

[0032] FIG. 12 is a graph which shows the linear shrinkage at 1400°C of magnesiumsilicate fibers which include varying amounts of a phosphorous containing compound as a coating on the exterior surfaces of the fiber.

[0033] FIG. 13 is a graph which shows the linear shrinkage at 1260°C of magnesiumsilicate fibers which include varying amounts of a phosphorous containing compound as a coating on the exterior surfaces of the fiber.

20

15

[0034] An inorganic fiber that is useful as a thermal, electrical, and acoustical insulation material is provided. The vitreous inorganic fiber has a continuous service or use temperature of 1260°C or greater. According to other embodiments, the vitreous inorganic fiber has a continuous service or use temperature of 1400°C or greater.

25

30

[0035] In order for a glass composition to be a viable candidate for producing a satisfactory high temperature resistant fiber product, the fiber to be produced must be manufacturable, sufficiently soluble in physiological fluids, and capable of surviving high temperatures with minimal shrinkage and minimal loss of mechanical integrity during exposure to the high service temperatures.

[0036] The present inorganic fiber is non-durable in physiological fluids. By "non-durable" in physiological fluids, it is meant that the inorganic fiber at least partially dissolves in such fluids, such as simulated lung fluid, during in vitro tests. The inorganic vitreous fiber also exhibits a linear shrinkage, as determined by the test method described below, of less than about 3.5 percent in response to exposure to a use temperature of 1260°C for 24 hours and less than 4.0 percent in response to exposure to a use temperature of 1400°C for 24 hours.

5

10

[0037] Durability may be tested by measuring the rate at which mass is lost from the fiber (ng/cm²-hr) under conditions which simulate the temperature and chemical conditions found in the human lung. This test consists of exposing approximately O.lg of de-shotted fiber to 50 ml of simulated lung fluid (SLF) for 6 hours. The entire test system is maintained at 37°C, to simulate the temperature of the human body.

[0038] After the SLF has been exposed to the fiber, it is collected and analyzed for glass constituents using Inductively Coupled Plasma Spectroscopy. A "blank" SLF sample is also measured and used to correct for elements present in the SLF. Once this data has been obtained, it is possible to calculate the rate at which the fiber has lost mass over the time interval of the study. The fibers of the present invention are significantly less durable than normal refractory ceramic fiber in simulated lung fluid.

20

25

[0039] FIG. 2 is a graph which illustrates the fiber dissolution rate of various magnesiumsilicate phosphorous containing fiber compositions. The fiber compositions of FIG. 2 generally comprise from about 75.4 to about 79.2 weight percent silica, from about 0.17 to about 0.4 weight percent calcia impurity, from about 17.1 to about 20.7 weight percent magnesia, from about 1.1 to about 1.7 weight percent alumina and varying amounts of a phosphorous containing compound (i.e., up to 3.0 weight percent). As is shown in FIG. 2, the rate of dissolution (ng/cm²hr) generally increases when the amount of phosphorous containing compound is increased within the magnesium-silicate fiber composition.

WO 2013/096471

PCT/US2012/070660

[0040] "Viscosity" refers to the ability of a glass melt to resist flow or shear stress. The viscosity-temperature relationship is critical in determining whether it is possible to fiberize a given glass composition. An optimum viscosity curve would have a low viscosity (5-50 poise) at the fiberization temperature and would gradually increase as the temperature decreased. If the melt is not sufficiently viscous (i.e. too thin) at the fiberization temperature, the result is a short, thin fiber, with a high proportion of unfiberized material (shot). If the melt is too viscous at the fiberization temperature, the resulting fiber will be extremely coarse (high diameter) and short.

[0041] Viscosity is dependent upon melt chemistry, which is also affected by elements or 10 compounds that act as viscosity modifiers. Viscosity modifiers permit fibers to be blown or spun from the fiber melt. It is desireable, however, that such viscosity modifiers, either by type or amount, do not adversely impact the solubility, shrink resistance, or mechanical strength of the blown or spun fiber.

15

30

5

[0042] One approach to testing whether a fiber of a defined composition can be readily manufactured at an acceptable quality level is to determine whether the viscosity curve of the experimental chemistry matches that of a known product which can be easily fiberized. Viscosity-temperature profiles may be measured on a viscometer, capable of operating at elevated temperatures. In addition, an adequate viscosity profile may be inferred by routine 20 experimentation, examining the quality of fiber (index, diameter, length) produced. The shape of the viscosity vs. temperature curve for a glass composition is representative of the ease with which a melt will fiberize and thus, of the quality of the resulting fiber (affecting, for example, the fiber's shot content, fiber diameter, and fiber length). Glasses generally 25 have low viscosity at high temperatures. As temperature decreases, the viscosity increases. The value of the viscosity at a given temperature will vary as a function of the composition, as will the overall steepness of the viscosity vs. temperature curve. The viscosity curve of a magnesium-silicate phosphorous containing fiber has a viscosity that approximates the target viscosity curve of the FIG. 1 for the commercially available, spun magnesium-silicate fiber.

[0043] Linear shrinkage of an inorganic fiber is a good measure of a fiber's high temperature resistance or of its performance at a particular continuous service or use temperature. Fibers are tested for shrinkage by forming them into a mat and needle punching the mat together into a blanket of approximately 8 pounds per cubic foot density and a thickness of about 1 inch. Such pads are cut into 3 inch x 5 inch pieces and platinum pins are inserted into the face of the material. The separation distance of these pins is then carefully measured and recorded. The pad is then placed into a furnace, ramped to temperature and held at the temperature for a fixed period of time. After heating, the pin separation is again measured to determine the linear shrinkage that pad has experienced.

10

15

5

[0044] In one such test, the length and width of this piece were carefully measured, and the pad was placed in a furnace and brought to a temperature of 1400°C for 24, 168, or 672 hours. After cooling, the lateral dimensions were measured and the linear shrinkage was determined by comparing "before" and "after" measurements. If the fiber is available in blanket form, measurements may be made directly on the blanket without the need to form a pad.

[0045] Experimentation of the present magnesium-silicate phosphorous containing fibers disclosed herein has shown a linear shrinkage as low as 0.83 % after exposure to 1260°C for 24 hours and 3.5 % or less after exposure to 1400°C for 24 hours. Thus, the addition of a phosphorous containing compound, either as a direct additive to the melt chemistry or as a coating applied to the fiber at the point of fiberization or after fiberization, to a magnesium-silicate fiber improves the shrinkage performance of the inorganic fiber at temperatures of 1400°C.

25

30

[0046] FIGS. 3-5 are graphs which illustrate the linear shrinkage of various magnesiumsilicate phosphorous containing fiber compositions at temperatures of 1260°C, 1400°C and 1500°C respectively. The fiber compositions of FIGS. 3-5 generally comprise from about 75.4 to about 79.2 weight percent silica, from about 0.17 to about 0.4 weight percent calcia impurity, from about 17.1 to about 20.7 weight percent magnesia, from about 1.1 to about 1.7 weight percent alumina and varying amounts of a phosphorous

containing compound (i.e., up to about 2.5 weight percent and up to about 4.5 weight percent). As is shown in FIGS. 3-5, the amount of fiber shrinkage at 1260°C, 1400°C and 1500°C generally decreases with increasing amounts of a phosphorous containing compound.

5

[0047] Mechanical integrity is also an important property since the fiber must support its own weight in any application and must also be able to resist abrasion due to moving air or gas. Indications of fiber integrity and mechanical strength are provided by visual and tactile observations, as well as mechanical measurement of these properties of after-service
temperature exposed fibers. The ability of the fiber to maintain its integrity after exposure to the use temperature may also be measured mechanically by testing for compression strength and compression recovery. These tests measure, respectively, how easily the pad may be deformed and the amount of resiliency (or compression recovery) the pad exhibits after a compression of 50%. Visual and tactile observations indicate that the present inorganic
fiber remains intact and maintains its form after exposure to a use temperature of at least 1400°C.

[0048] The low shrinkage, high temperature resistant inorganic fiber comprises the fiberization product of a melt containing magnesia and silica as the primary constituents.
20 The non-durable inorganic fibers are made by standard glass and ceramic fiber manufacturing methods. Raw materials, such as silica, any suitable source of magnesia such as enstatite, forsterite, magnesia, magnesite, calcined magnesite, magnesium zirconate, periclase, steatite, or talc, and, if zirconia is included in the fiber melt, any suitable source of zirconia such as baddeleyite, magnesium zirconate, zircon or zirconia, are introduced into a suitable furnace where they are melted and blown using a fiberization nozzle, or spun, either in a batch or a continuous mode.

[0049] The inorganic fiber comprising the fiberization product of magnesia and silica is referred to as a "magnesium-silicate" fiber. The low shrinkage, high temperature
30 resistant inorganic fiber also comprises a phosphorous containing compound as part of the melt chemistry of the fiber composition or as a coating that is applied to the fiber at

the point of fiberization or after fiberization. In alternative embodiments, the inorganic fiber may comprise a phosphorous containing compound as both part of its melt chemistry and as a coating which is applied to at least a portion of the exterior surface of the inorganic fiber.

5

[0050] According certain embodiments, the present inorganic fiber comprises the fiberization product of about 65 to about 86 weight percent silica, about 14 to about 35 weight percent magnesia, and greater than 0 to about 10 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of about 65 to about 86 weight percent silica, about 14 to about 35 weight percent magnesia, and includes a coating of greater than 0 to about 10 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of about 65 to about 86 weight percent silica, about 10 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of about 65 to about 86 weight percent silica, about 14 to about 35 weight percent magnesia, and includes a coating of from about 5 to about 10 weight percent
15 based on the total fiber weight of a phosphorous containing compound. In alternative embodiments, the inorganic fiber comprises the fiberization product of about 14 to about 35 weight percent silica and about 14 to about 35 weight percent magnesia, and greater than 0 to about 10 weight percent silica and about 14 to about 35 weight percent magnesia, and greater than 0 to about 10 weight percent silica and about 14 to about 35 weight percent magnesia, and greater than 0 to about 10 weight percent silica and about 14 to about 35 weight percent magnesia, and greater than 0

- chemistry and as a coating based on the total fiber weight. In alternative embodiments, the 20 inorganic fiber comprises the fiberization product of about 65 to about 86 weight percent silica and about 14 to about 35 weight percent magnesia, and from about 5 to about 10 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.
- [0051] According other embodiments, the fiber comprises the fiberization product of greater than 71.25 to about 86 weight percent silica, about 14 to about 28.75 weight percent magnesia and greater than 0 to about 10 weight percent of a phosphorous containing compound. In alternative embodiments, the magnesium-silicate phosphorous containing fiber comprises the fiberization product of greater than 71.25 to about 86 weight percent silica and about 14 to about 28.75 weight percent magnesia, and includes a coating in an amount of greater than 0 to about 10 weight percent based on the total fiber weight of a

WO 2013/096471

5

PCT/US2012/070660

phosphorous containing compound. In alternative embodiments, the inorganic fiber comprising the fiberization product of greater than 71.25 to about 86 weight percent silica and about 14 to about 28.75 weight percent magnesia also comprises greater than 0 to about 10 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

[0052] According other embodiments, the fiber comprises the fiberization product of about 71.25 to about 86 weight percent silica, about 14 to about 28.75 weight percent magnesia, and greater than 0 to about 7 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of about 71.25 to about 86 weight percent silica and about 14 to about 28.75 weight percent magnesia, and includes a coating in an amount of greater than 0 to about 7 weight percent based on the total fiber weight of a phosphorous containing compound. In alternative embodiments, the fiberization product of about 7 weight percent based on the total fiber weight of a phosphorous containing compound. In alternative embodiments, the fiber comprising the fiberization product of about 71.25 to about 86 weight percent silica 15 and about 14 to about 28.75 weight percent magnesia also comprises greater than 0 to about 7 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

[0053] According other embodiments, the fiber comprises the fiberization product of greater than 71.25 to about 86 weight percent silica, about 14 to about 28.75 weight percent magnesia and greater than 0 to about 6 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of greater than 71.25 to about 86 weight percent silica and about 14 to about 28.75 weight percent magnesia, and includes a coating in an amount of greater than 0 to about 6 weight percent based on the total fiber weight of a phosphorous containing compound. In alternative embodiments, the fiberization product of greater than 71.25 to about 86 weight percent silica and about 14 to about 28.75 weight percent based on the total fiber comprising the fiberization product of greater than 71.25 to about 86 weight percent silica and about 14 to about 28.75 weight percent magnesia also comprises greater than 0 to about 6 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

30

10

PCT/US2012/070660

[005] According other embodiments, the fiber comprises the fiberization product of greater than 71.25 to about 86 weight percent silica, about 14 to about 28.75 weight percent magnesia, and greater than 0 to about 5 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of greater than 71.25 to about 86 weight percent silica and about 14 to about 28.75 weight percent magnesia, and includes a coating in an amount of greater than 0 to about 5 weight percent based on the total fiber weight of a phosphorous containing compound. In alternative embodiments, the fiberization product of greater than 0 to about 5 weight percent based on the total fiber weight of a phosphorous containing compound. In alternative embodiments, the fiber comprising the fiberization product of greater than 71.25 to about 86 weight percent silica and about 14 to about 28.75 weight percent based on the total fiber weight of a phosphorous containing compound. In alternative embodiments, the fiber comprising the fiberization product of greater than 71.25 to about 86 weight percent silica and about 14 to about 28.75 weight percent magnesia also comprises greater than 0 to about 5 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

[0055] According other embodiments, the fiber comprises the fiberization product of greater than 71.25 to about 86 weight percent silica, about 14 to about 28.75 weight percent magnesia, and greater than 0 to about 4 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of greater than 71.25 to about 86 weight percent silica andabout 14 to about 28.75 weight percent magnesia, and includes a coating in an amount of greater than 0 to about 4 weight percent based on the total fiber weight of a phosphorous containing compound. In alternative embodiments, the fiberization product of greater than 71.25 to about 86 weight of a phosphorous containing compound. In alternative embodiments, the fiber comprising the fiberization product of greater than 71.25 to about 86 weight percent silica and about 14 to about 28.75 weight percent based on the total fiber comprising the fiberization product of greater than 71.25 to about 86 weight percent silica and about 14 to about 28.75 weight percent magnesia also comprises greater than 0 to about 4 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

[0056] According other embodiments, the fiber comprises the fiberization product of greater than 71.25 to about 86 weight percent silica, about 14 to about 28.75 weight percent magnesia, and greater than 0 to about 3 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of greater than 71.25 to about 86 weight percent silica and about 14 to about 28.75 weight percent of greater than 71.25 to about 86 weight percent silica and about 14 to about 28.75 weight percent magnesia, and includes a coating in an amount of greater than 0 to about 3 weight percent based on the total fiber weight of a phosphorous containing compound. In

alternative embodiments, the fiber comprising the fiberization product of greater than 71.25 to about 86 weight percent silica and about 14 to about 28.75 weight percent magnesia also comprises greater than 0 to about 3 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

5

[0057] According other embodiments, the fiber comprises the fiberization product of greater than 71.25 to about 86 weight percent silica, about 14 to about 28.75 weight percent magnesia, and greater than 0 to about 2 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of greater than 71.25 to about 86 weight percent silica and about 14 to about 28.75 weight percent magnesia, and includes a coating in an amount of greater than 0 to about 2 weight percent based on the total fiber weight of a phosphorous containing compound. In alternative embodiments, the fiberization product of greater than 71.25 to about 86 weight of a phosphorous containing compound. In alternative embodiments, the fiber comprising the fiberization product of greater than 71.25 to about 86 weight percent silica and about 14 to about 28.75 weight percent based on the total fiber weight of a phosphorous containing compound. In alternative embodiments, the fiber comprising the fiberization product of greater than 71.25 to about 86 weight percent silica and about 14 to about 28.75 weight percent magnesia also comprises greater than 0 to about 2 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

[0058] According other embodiments, the fiber comprises the fiberization product of about 70 to about 80 weight percent silica, about 15 to about 25 weight percent magnesia, and greater than 0 to about 10 weight percent of a phosphorous containing compound. In alternative embodiments, the magnesium-silicate phosphorous containing fiber comprises the fiberization product of about 70 to about 80 weight percent silica and about 15 to about 25 weight percent magnesia, and includes a coating of a phosphorous containing compound in an amount of greater than 0 to about 10 weight percent based on the total fiber weight. In alternative embodiments, the inorganic fiber comprising the fiberization product of about 70 to about 15 to about 25 weight percent silica and about 15 to about 25 weight percent silica and about 10 weight percent based on the total fiber weight. In alternative embodiments, the inorganic fiber comprising the fiberization product of about 70 to about 80 weight percent silica and about 15 to about 25 weight percent magnesia also comprises greater than 0 to about 10 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

10

PCT/US2012/070660

[0059] According other embodiments, the fiber comprises the fiberization product of about 70 to about 80 weight percent silica, about 15 to about 25 weight percent magnesia, and greater than 0 to about 7 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of about 70 to about 80 weight percent silica and about 15 to about 25 weight percent magnesia and includes a coating of a phosphorous containing compound in an amount of greater than 0 to about 7 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprises greater than 0 to about 7 weight percent silica and about 15 to about 20 weight percent silica and about 15 to about 25 weight percent silica and about 15 to about 7 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprising the fiberization product of about 70 to about 80 weight percent silica and about 15 to about 25 weight percent silica and about 15 to about 70 to about 80 weight percent silica and about 15 to about 25 weight percent silica and about 15 to about 70 to about 80 weight percent silica and about 15 to about 25 weight percent magnesia also comprises greater than 0 to about 7 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

[0060] According other embodiments, the fiber comprises the fiberization product of about 70 to about 80 weight percent silica, about 15 to about 25 weight percent magnesia, and greater than 0 to about 6 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of about 70 to about 80 weight percent silica and about 15 to about 25 weight percent magnesia, and a includes coating of a phosphorous containing compound in an amount of greater than 0 to about 6 weight percent based on the total fiber weight. In alternative embodiments, the fiber 20 comprising the fiberization product of about 70 to about 15 to about 25 weight percent silica and about 15 to about 70 to about 80 weight percent silica and about 15 to about 25 weight percent silica and about 15 to about 25 weight percent silica and about 16 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

[0061] According other embodiments, the fiber comprises the fiberization product of about 70 to about 80 weight percent silica, about 15 to about 25 weight percent magnesia, and greater than 0 to about 5 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of about 70 to about 80 weight percent silica and about 15 to about 25 weight percent magnesia, and includes a coating of a phosphorous containing compound in an amount of greater than 0 to about 5 weight percent based on the total fiber weight. In alternative embodiments, the fiber

comprising the fiberization product of about 70 to about 80 weight percent silica and about 15 to about 25 weight percent magnesia also comprises greater than 0 to about 5 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

5

10

15

[0062] According other embodiments, the fiber comprises the fiberization product of about 70 to about 80 weight percent silica, about 15 to about 25 weight percent magnesia, and greater than 0 to about 4 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of about 70 to about 80 weight percent silica and about 15 to about 25 weight percent magnesia, and includes a coating of a phosphorous containing compound in an amount of greater than 0 to about 4 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprises the fiberization product of about 4 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprising the fiberization product of about 70 to about 80 weight percent silica and about 15 to about 25 weight percent silica and about 15 to about 20 to about 80 weight percent silica and about 15 to about 20 to about 80 weight percent silica and about 15 to about 20 to about 80 weight percent silica and about 15 to about 25 weight percent magnesia also comprises greater than 0 to about 4 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

[0063] According other embodiments, the fiber comprises the fiberization product of about 70 to about 80 weight percent silica, about 15 to about 25 weight percent magnesia, and greater than 0 to about 3 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of about 70 to about 80 weight percent silica and about 15 to about 25 weight percent magnesia, and includes a coating of a phosphorous containing compound in an amount of greater than 0 to about 3 weight percent based on the total fiber weight. In alternative embodiments, the fiber 25 comprising the fiberization product of about 70 to about 80 weight percent silica and about 15 to about 25 weight percent magnesia also comprises greater than 0 to about 3 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

10

PCT/US2012/070660

[0064] According other embodiments, the fiber comprises the fiberization product of about 70 to about 80 weight percent silica, about 15 to about 25 weight percent magnesia, and greater than 0 to about 2 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of about 70 to about 80 weight percent silica and about 15 to about 25 weight percent magnesia, and includes a coating of a phosphorous containing compound in an amount of greater than 0 to about 2 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprises greater than 0 to about 2 weight percent silica and about 15 to about 20 weight percent silica and about 15 to about 20 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprising the fiberization product of about 70 to about 80 weight percent silica and about 15 to about 25 weight percent silica and about 15 to about 20 weight percent silica and about 16 to about 20 weight percent silica and about 170 to about 20 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprising the fiberization product of about 70 to about 80 weight percent silica and about 15 to about 25 weight percent magnesia also comprises greater than 0 to about 2 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

[0065] According other embodiments, the fiber comprises the fiberization product of about 70 to about 80 weight percent silica, about 15 to about 25 weight percent magnesia, and greater than 0 to about 1.5 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of about 70 to about 80 weight percent silica and about 15 to about 25 weight percent magnesia, and includes a coating of a phosphorous containing compound in an amount of greater than 0 to about 1.5 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprises greater than 0 to about 1.5 weight percent silica and about 16 to about 70 to about 1.5 weight percent silica and about 15 to about 25 weight percent silica and about 1.5 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprising the fiberization product of about 70 to about 80 weight percent silica and about 15 to about 25 weight percent silica and about 15 to about 25 weight percent silica and about 15 to about 25 weight percent magnesia also comprises greater than 0 to about 1.5 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

[0066] According other embodiments, the fiber comprises the fiberization product of about 70 to about 80 weight percent silica, about 15 to about 25 weight percent magnesia, and greater than 0 to less than 1 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of about 70 to about 80 weight percent silica and about 15 to about 25 weight percent magnesia, and includes a coating of a phosphorous containing compound in an amount of greater than 0 to less than 1 weight percent based on the total fiber weight. In alternative embodiments, the fiber veight.

comprising the fiberization product of about 70 to about 80 weight percent silica and about 15 to about 25 weight percent magnesia also comprises greater than 0 to less than 1 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

5

10

15

[0067] According other embodiments, the fiber comprises the fiberization product of about 72 to about 80 weight percent silica, about 15 to about 25 weight percent magnesia, and greater than 0 to about 10 weight percent of a phosphorous containing compound. In alternative embodiments, the magnesium-silicate phosphorous containing fiber comprises the fiberization product of about 72 to about 80 weight percent silica and about 15 to about 25 weight percent magnesia, and includes a coating of a phosphorous containing compound in an amount of greater than 0 to about 10 weight percent based on the total fiber weight. In alternative embodiments, the inorganic fiber comprising the fiberization product of about 72 to about 80 weight percent silica and about 15 to about 25 weight percent magnesia also comprises greater than 0 to about 10 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

[0068] According other embodiments, the fiber comprises the fiberization product of about 72 to about 80 weight percent silica, about 15 to about 25 weight percent magnesia, and greater than 0 to about 7 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of about 72 to about 80 weight percent silica and about 15 to about 25 weight percent magnesia, and includes a coating of a phosphorous containing compound in an amount of greater than 0 to about 7 weight percent based on the total fiber weight. In alternative embodiments, the fiber 25 comprising the fiberization product of about 72 to about 15 to about 25 weight percent magnesia also comprises greater than 0 to about 7 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

10

PCT/US2012/070660

[0069] According other embodiments, the fiber comprises the fiberization product of about 72 to about 80 weight percent silica, about 15 to about 25 weight percent magnesia, and greater than 0 to about 6 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of about 72 to about 80 weight percent silica and about 15 to about 25 weight percent magnesia, and includes a coating of a phosphorous containing compound in an amount of greater than 0 to about 6 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprises the fiberization product solut 80 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprising the fiberization product of about 72 to about 80 weight percent silica and about 15 to about 25 weight percent silica and about 15 to about 26 weight percent silica and about 15 to about 60 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprising the fiberization product of about 72 to about 80 weight percent silica and about 15 to about 25 weight percent silica and about 15 to about 25 weight percent magnesia also comprises greater than 0 to about 6 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

[0070] According other embodiments, the fiber comprises the fiberization product of about 72 to about 80 weight percent silica, about 15 to about 25 weight percent magnesia, and greater than 0 to about 5 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of about 72 to about 80 weight percent silica and about 15 to about 25 weight percent magnesia, and includes a coating of a phosphorous containing compound in an amount of greater than 0 to about 5 weight percent based on the total fiber weight. In alternative embodiments, the fiber 20 comprising the fiberization product of about 72 to about 15 to about 25 weight percent silica and about 15 to about 72 to about 80 weight percent silica and about 15 to about 25 weight percent silica and about 15 to about 50 weight percent based on the total fiber weight. In alternative embodiments, the fiber 20 comprising the fiberization product of about 72 to about 80 weight percent silica and about 15 to about 25 weight percent magnesia also comprises greater than 0 to about 5 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

[0071] According other embodiments, the fiber comprises the fiberization product of about 72 to about 80 weight percent silica, about 15 to about 25 weight percent magnesia, and greater than 0 to about 4 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of about 72 to about 80 weight percent silica and about 15 to about 25 weight percent magnesia, and includes a coating of a phosphorous containing compound in an amount of greater than 0 to about 4 weight percent based on the total fiber weight. In alternative embodiments, the fiber

comprising the fiberization product of about 72 to about 80 weight percent silica and about 15 to about 25 weight percent magnesia also comprises greater than 0 to about 4 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

5

15

[0072] According other embodiments, the fiber comprises the fiberization product of about 72 to about 80 weight percent silica, about 15 to about 25 weight percent magnesia, and greater than 0 to about 3 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of about 72 to about 80 weight percent silica and about 15 to about 25 weight percent magnesia, and includes a 10 coating of a phosphorous containing compound in an amount of greater than 0 to about 3 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprising the fiberization product of about 72 to about 80 weight percent silica and about 15 to about 25 weight percent magnesia also comprises greater than 0 to about 3 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

[0073] According other embodiments, the fiber comprises the fiberization product of about 72 to about 80 weight percent silica, about 15 to about 25 weight percent magnesia, and greater than 0 to about 2 weight percent of a phosphorous containing compound. In 20 alternative embodiments, the fiber comprises the fiberization product of about 72 to about 80 weight percent silica and about 15 to about 25 weight percent magnesia and a coating of a phosphorous containing compound in an amount of greater than 0 to about 2 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprising the fiberization product of about 72 to about 80 weight percent silica and about 15 to about 25 25 weight percent magnesia also comprises greater than 0 to about 2 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

10

PCT/US2012/070660

[0074] According other embodiments, the fiber comprises the fiberization product of about 72 to about 80 weight percent silica, about 15 to about 25 weight percent magnesia, and greater than 0 to about 1.5 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of about 72 to about 80 weight percent silica and about 15 to about 25 weight percent magnesia, and includes a coating of a phosphorous containing compound in an amount of greater than 0 to about 1.5 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprises greater than 0 to about 1.5 weight percent silica and about 15 to about 25 weight percent silica and about 15 to about 25 weight percent silica and about 1.5 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprising the fiberization product of about 72 to about 80 weight percent silica and about 15 to about 72 to about 80 weight percent silica and about 15 to about 72 to about 80 weight percent silica and about 15 to about 25 weight percent silica and about 15 to about 25 weight percent silica and about 15 to about 25 weight percent silica and about 15 to about 25 weight percent silica and about 15 to about 25 weight percent silica and about 15 to about 25 weight percent silica and about 15 to about 25 weight percent magnesia also comprises greater than 0 to about 1.5 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

[0075] According other embodiments, the fiber comprises the fiberization product of about 72 to about 80 weight percent silica, about 15 to about 25 weight percent magnesia, and greater than 0 to less than 1 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of about 72 to about 80 weight percent silica and about 15 to about 25 weight percent magnesia, and includes a coating of a phosphorous containing compound in an amount of greater than 0 to less than 1 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprises greater than 0 to less than 1 weight percent silica and about 72 to about 80 weight percent silica and about 15 to about 25 weight percent silica and about 15 to about 25 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprising the fiberization product of about 72 to about 80 weight percent silica and about 15 to about 25 weight percent silica and about 15 to about 25 weight percent magnesia also comprises greater than 0 to less than 1 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

[0076] According other embodiments, the fiber comprises the fiberization product of about 75 to about 79 weight percent silica, about 15 to about 20 weight percent magnesia, and greater than 0 to about 10 weight percent of a phosphorous containing compound. In alternative embodiments, the magnesium-silicate phosphorous containing fiber comprises the fiberization product of about 75 to about 79 weight percent silica and about 15 to about 30 weight percent magnesia, and includes a coating of a phosphorous containing compound

22

in an amount of greater than 0 to about 10 weight percent based on the total fiber weight. In

alternative embodiments, the inorganic fiber comprising the fiberization product of about 75 to about 79 weight percent silica and about 15 to about 20 weight percent magnesia also comprises greater than 0 to about 10 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

5

10

15

[0077] According other embodiments, the fiber comprises the fiberization product of about 75 to about 79 weight percent silica, about 15 to about 20 weight percent magnesia, and greater than 0 to about 7 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of about 75 to about 79 weight percent silicaand about 15 to about 20 weight percent magnesia, and includes a coating of a phosphorous containing compound in an amount of greater than 0 to about 7 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprises greater than 0 to about 7 weight percent silica and about 15 to about 79 weight percent silica and about 15 to about 79 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprising the fiberization product of about 75 to about 79 weight percent silica and about 15 to about 79 weight percent silica and about 15 to about 79 weight percent silica and about 15 to about 79 weight percent silica and about 15 to about 20 weight percent silica and about 15 to about 20 weight percent fiberization product of about 79 weight percent silica and about 15 to about 20 weight percent fiberization product of about 79 weight percent silica and about 15 to about 20 weight percent magnesia also comprises greater than 0 to about 7 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

[0078] According other embodiments, the fiber comprises the fiberization product of about 75 to about 79 weight percent silica, about 15 to about 20 weight percent magnesia, and greater than 0 to about 6 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of about 75 to about 79 weight percent silica and about 15 to about 20 weight percent magnesia and a coating of a phosphorous containing compound in an amount of greater than 0 to about 6 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprises greater than 0 to about 15 to about 20 weight percent of a bout 15 to about 6 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprising the fiberization product of about 75 to about 79 weight percent silica and about 15 to about 20 weight percent magnesia also comprises greater than 0 to about 6 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

10

PCT/US2012/070660

[0079] According other embodiments, the fiber comprises the fiberization product of about 75 to about 79 weight percent silica, about 15 to about 20 weight percent magnesia, and greater than 0 to about 5 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of about 75 to about 79 weight percent silica and about 15 to about 20 weight percent magnesia, and includes a coating of a phosphorous containing compound in an amount of greater than 0 to about 5 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprises greater than 0 to about 5 weight percent silica and about 15 to about 79 weight percent silica and about 15 to about 20 weight percent silica and about 15 to about 20 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprising the fiberization product of about 75 to about 79 weight percent silica and about 15 to about 20 weight percent silica and about 15 to about 5 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

[0080] According other embodiments, the fiber comprises the fiberization product of about 75 to about 79 weight percent silica, about 15 to about 20 weight percent magnesia, and greater than 0 to about 4 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of about 75 to about 79 weight percent silica and about 15 to about 20 weight percent magnesia, and includes a coating of a phosphorous containing compound in an amount of greater than 0 to about 4 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprises greater than 0 to about 4 weight percent silica and about 15 to about 79 weight percent silica and about 15 to about 20 weight percent silica and about 15 to about 20 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprising the fiberization product of about 75 to about 79 weight percent silica and about 15 to about 20 weight percent magnesia also comprises greater than 0 to about 4 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

[0081] According other embodiments, the fiber comprises the fiberization product of about 75 to about 79 weight percent silica, about 15 to about 20 weight percent magnesia, and greater than 0 to about 3 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of about 75 to about 79 weight percent silica and about 15 to about 20 weight percent magnesia, and includes a coating of a phosphorous containing compound in an amount of greater than 0 to about 3 weight percent based on the total fiber weight. In alternative embodiments, the fiber

comprising the fiberization product of about 75 to about 79 weight percent silica and about 15 to about 20 weight percent magnesia also comprises greater than 0 to about 3 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

5

10

15

[0082] According other embodiments, the fiber comprises the fiberization product of about 75 to about 79 weight percent silica, about 15 to about 20 weight percent magnesia, and greater than 0 to about 2 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of about 75 to about 79 weight percent silica and about 15 to about 20 weight percent magnesia, and includes a coating of a phosphorous containing compound in an amount of greater than 0 to about 2 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprises the fiberization product of about 2 weight percent silica and about 15 to about 79 weight percent silica and about 15 to about 79 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprising the fiberization product of about 75 to about 79 weight percent silica and about 15 to about 20 weight percent silica and about 15 to about 20 weight percent silica and about 35 to about 20 weight percent silica and about 35 to about 20 weight percent fiberization product of about 75 to about 20 weight percent silica and about 15 to about 20 weight percent magnesia also comprises greater than 0 to about 2 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

[0083] According other embodiments, the fiber comprises the fiberization product of about 75 to about 79 weight percent silica, about 15 to about 20 weight percent magnesia, and greater than 0 to about 1.5 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of about 75 to about 79 weight percent silica and about 15 to about 20 weight percent magnesia, and includes a coating of a phosphorous containing compound in an amount of greater than 0 to about 1.5 weight percent based on the total fiber weight. In alternative embodiments, the fiber 25 comprising the fiberization product of about 75 to about 79 weight percent silica and about 15 to about 79 weight percent silica and about 15 to about 20 weight percent silica and about 1.5 weight percent 5 to about 1.5 weight percent based on the total fiber weight. In alternative embodiments, the fiber 25 comprising the fiberization product of about 75 to about 79 weight percent silica and about 15 to about 20 weight percent silica and about 15 to about 20 weight percent silica and about 35 to about 79 weight percent silica and about 1.5 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

10

PCT/US2012/070660

[0084] According other embodiments, the fiber comprises the fiberization product of about 75 to about 79 weight percent silica, about 15 to about 20 weight percent magnesia, and greater than 0 to less than 1 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of about 75 to about 79 weight percent silica and about 15 to about 20 weight percent magnesia, and includes a coating of a phosphorous containing compound in an amount of greater than 0 to less than 1 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprises greater than 0 to less than 1 weight percent silica and about 15 to about 79 weight percent silica and about 15 to about 20 weight percent silica and about 15 to about 20 weight percent than 0 to less than 1 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprising the fiberization product of about 75 to about 79 weight percent silica and about 15 to about 20 weight percent silica and about 15 to about 20 weight percent silica and about 15 to about 79 weight percent silica and about 15 to about 20 weight percent silica and about 15 to about 79 weight percent silica and about 15 to about 20 weight percent magnesia also comprises greater than 0 to less than 1 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

[0085] According other embodiments, the fiber comprises the fiberization product of about 72 to about 80 weight percent silica, about 20 to about 28 weight percent magnesia, and greater than 0 to about 10 weight percent of a phosphorous containing compound. In alternative embodiments, the magnesium-silicate phosphorous containing fiber comprises the fiberization product of about 72 to about 80 weight percent silica and about 20 to about 28 weight percent magnesia, and includes a coating of a phosphorous containing compound in an amount of greater than 0 to about 10 weight percent based on the total fiber weight. In alternative embodiments, the inorganic fiber comprising the fiberization product of about 72 to about 80 weight percent silica and about 20 to about 28 weight percent magnesia also comprises greater than 0 to about 10 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

[0086] According other embodiments, the fiber comprises the fiberization product of about 72 to about 80 weight percent silica, about 20 to about 28 weight percent magnesia, and greater than 0 to about 7 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of about 72 to about 80 weight percent silica and about 20 to about 28 weight percent magnesia, and includes a coating of a phosphorous containing compound in an amount of greater than 0 to about 7 weight percent based on the total fiber weight. In alternative embodiments, the fiber

comprising the fiberization product of about 72 to about 80 weight percent silica and about 20 to about 28 weight percent magnesia also comprises greater than 0 to about 7 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

5

10

15

[0087] According other embodiments, the fiber comprises the fiberization product of about 72 to about 80 weight percent silica, about 20 to about 28 weight percent magnesia, and greater than 0 to about 6 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of about 72 to about 80 weight percent silica and about 20 to about 28 weight percent magnesia, and includes a coating of a phosphorous containing compound in an amount of greater than 0 to about 6 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprises greater than 0 to about 6 weight percent silica and about 72 to about 72 to about 6 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprising the fiberization product of about 72 to about 80 weight percent silica and about 20 to about 72 to about 80 weight percent silica and about 20 to about 72 to about 80 weight percent silica and about 72 to about 6 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

[0088] According other embodiments, the fiber comprises the fiberization product of about 72 to about 80 weight percent silica, about 20 to about 28 weight percent magnesia, and greater than 0 to about 5 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of about 72 to about 80 weight percent silica and about 20 to about 28 weight percent magnesia, and including a coating of a phosphorous containing compound in an amount of greater than 0 to about 5 weight percent based on the total fiber weight. In alternative embodiments, the fiber 25 comprising the fiberization product of about 72 to about 20 to about 28 weight percent magnesia also comprises greater than 0 to about 5 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

10

PCT/US2012/070660

[0089] According other embodiments, the fiber comprises the fiberization product of about 72 to about 80 weight percent silica, about 20 to about 28 weight percent magnesia, and greater than 0 to about 4 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of about 72 to about 80 weight percent silica and about 20 to about 28 weight percent magnesia, and includes a coating of a phosphorous containing compound in an amount of greater than 0 to about 4 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprises greater than 0 to about 4 weight percent silica and about 72 to about 20 to about 28 weight percent silica and about 20 to about 28 weight percent silica and about 20 to about 4 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprising the fiberization product of about 72 to about 80 weight percent silica and about 20 to about 28 weight percent silica and about 20 to about 72 to about 80 weight percent silica and about 20 to about 28 weight percent silica and about 72 to about 80 weight percent silica and about 20 to about 28 weight percent magnesia also comprises greater than 0 to about 4 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

[090] According other embodiments, the fiber comprises the fiberization product of about 72 to about 80 weight percent silica, about 20 to about 28 weight percent magnesia, and 15 greater than 0 to about 3 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of about 72 to about 80 weight percent silica and about 20 to about 28 weight percent magnesia, and includes a coating of a phosphorous containing compound in an amount of greater than 0 to about 3 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprises greater than 0 to about 3 weight percent silica and about 72 to about 72 to about 30 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprising the fiberization product of about 72 to about 80 weight percent silica and about 20 to about 28 weight percent silica and about 20 to about 30 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprising the fiberization product of about 72 to about 80 weight percent silica and about 20 to about 28 weight percent magnesia also comprises greater than 0 to about 3 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

[0091] According other embodiments, the fiber comprises the fiberization product of about 72 to about 80 weight percent silica, about 21 to about 28 weight percent magnesia, and greater than 0 to about 2 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of about 72 to about 80 weight percent silica and about 20 to about 28 weight percent magnesia, and includes a coating of a phosphorous containing compound in an amount of greater than 0 to about 2 weight percent based on the total fiber weight. In alternative embodiments, the fiber

comprising the fiberization product of about 72 to about 80 weight percent silica and about 20 to about 28 weight percent magnesia also comprises greater than 0 to about 2 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

5

10

15

[0092] According other embodiments, the fiber comprises the fiberization product of about 72 to about 80 weight percent silica, about 20 to about 28 weight percent magnesia, and greater than 0 to about 1.5 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of about 72 to about 80 weight percent silica and about 20 to about 28 weight percent magnesia, and includes a coating of a phosphorous containing compound in an amount of greater than 0 to about 1.5 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprises greater than 0 to about 1.5 weight percent silica and about 20 to about 72 to about 28 weight percent silica and about 20 to about 28 weight percent silica and about 1.5 weight percent based on the total fiber weight. In alternative embodiments, the fiber comprising the fiberization product of about 72 to about 80 weight percent silica and about 20 to about 28 weight percent silica and about 20 to about 72 to about 80 weight percent silica and about 20 to about 28 weight percent silica and about 72 to about 80 weight percent silica and about 20 to about 28 weight percent magnesia also comprises greater than 0 to about 1.5 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

[0093] According other embodiments, the fiber comprises the fiberization product of about 72 to about 80 weight percent silica, about 20 to about 28 weight percent magnesia, and greater than 0 to less than 1 weight percent of a phosphorous containing compound. In alternative embodiments, the fiber comprises the fiberization product of about 72 to about 80 weight percent silica and about 20 to about 28 weight percent magnesia, and includes a coating of a phosphorous containing compound in an amount of greater than 0 to less than 1 weight percent based on the total fiber weight. In alternative embodiments, the fiber 25 comprising the fiberization product of about 72 to about 28 weight percent silica and about 20 to about 72 to about 80 weight percent silica and about 20 to about 72 to about 80 weight percent based on the total fiber weight. In alternative embodiments, the fiber 25 comprising the fiberization product of about 72 to about 80 weight percent silica and about 20 to about 28 weight percent silica and about 20 to about 28 weight percent magnesia also comprises greater than 0 to less than 1 weight percent of a phosphorous containing compound as part of the fiber's melt chemistry and as a coating based on the total fiber weight.

[0094] In addition to magnesia, silica and phosphorous, the magnesium-silicate phosphorous containing fiber may contain a number of impurities. In certain embodiments, the magnesium-silicate phosphorous containing fiber may contain up to about 10 weight percent of impurities. Such impurities may include calcia and iron oxides. In certain embodiments, the fiber does not contain more than about 1 weight percent calcia impurity. In other embodiments, the fiber contains less than 0.5 weight percent calcia. According to other embodiments, the fiber does not contain more than about 2 weight percent iron oxides impurity (calculated as $Fe_2C''3$).

10

15

5

[0095] The magnesium-silicate phosphorous containing fibers exhibit a linear shrinkage after exposure to a service temperature of 1260°C for 24 hours of less than 3.5 percent. In other embodiments, the magnesium-silicate phosphorous containing fibers exhibit a linear shrinkage after exposure to a service temperature of 1260°C for 24 hours of less than 2.0 percent.

[0096] In other embodiments, the magnesium-silicate phosphorous containing fibers exhibit a linear shrinkage after exposure to a service temperature of 1400°C for 24 hours of less than 10 percent. In other embodiments, the magnesium-silicate phosphorous containing fibers exhibit a linear shrinkage after exposure to a service temperature of 20 1400°C for 24 hours of less than 5 percent. In other embodiments, the magnesiumsilicate phosphorous containing fibers exhibit a linear shrinkage after exposure to a service temperature of 1400°C for 24 hours of less than 4 percent. In other embodiments, the magnesium-silicate phosphorous containing fibers exhibit a linear shrinkage after 25 exposure to a service temperature of 1400°C for 24 hours of less than 3.5 percent. In other embodiments, the magnesium-silicate phosphorous containing fibers exhibit a linear shrinkage after exposure to a service temperature of 1400°C for 24 hours of less than 2.5 percent. In other embodiments, the magnesium-silicate phosphorous containing fibers exhibit a linear shrinkage after exposure to a service temperature of 1400°C for 24 30 hours of less than 2 percent.

10

30

PCT/US2012/070660

[0097]According to certain embodiments, a fiber comprising the fiberization product comprising of about 65 to about 86 weight percent silica and about 14 to about 35 weight percent magnesia, greater than 0 to about 10 weight percent of a phosphorous containing compound, and greater than 0 to about 1.5 weight percent alumina exhibits a linear shrinkage of about 5% or less at 1260°C for 24 hours.

[0098] According to certain embodiments, a fiber comprising the fiberization product comprising of about 65 to about 86 weight percent silica and about 14 to about 35 weight percent magnesia, greater than 0 to about 10 weight percent of a phosphorous containing compound, and greater than 0 to about 3 weight percent alumina exhibits a linear shrinkage of about 15% or less at 1260°C for 24 hours.

[0099]According to certain embodiments, a fiber comprising the fiberization product comprising of about 65 to about 86 weight percent silica and about 14 to about 35 weight percent magnesia, greater than 0 to about 10 weight percent of a phosphorous containing compound, and greater than 0 to about 4 weight percent alumina exhibits a linear shrinkage of about 20% or less at 1260°C for 24 hours.

[0100] Thus, the magnesium-silicate phosphorous containing fibers are useful for thermal insulating applications at continuous service or operating temperatures of at least 1260°C or greater. According to certain embodiments, the magnesium-silicate phosphorous containing fibers are useful for thermal insulating applications at continuous service or operating temperatures of at least 1400°C or greater. Furthermore, it has been found that the magnesium-silicate phosphorous containing fibers do not melt until they are exposed to a temperature of 1500°C or greater.

[0101] The magnesium-silicate phosphorous containing fiber may be prepared by fiber blowing or fiber spinning techniques. A suitable fiber blowing technique includes the steps of mixing the starting raw materials containing magnesia, silica and, phosphorous containing compound together to form a material mixture of ingredients, introducing the material mixture of ingredients into a suitable vessel or container, melting the material

mixture of ingredients for discharge through a suitable nozzle, and blowing a high pressure gas onto the discharged flow of molten material mixture of ingredients to form the magnesium-silicate phosphorous containing fibers.

5 [0102] A suitable fiber spinning technique includes the steps of mixing the starting raw materials containing magnesia, silica and phosphorous containing compound together to form a material mixture of ingredients, introducing the material mixture of ingredients into a suitable vessel or container, melting the material mixture of ingredients for discharge through a suitable nozzle onto spinning wheels. The molten stream then 10 cascades over the wheels, coating the wheels and being thrown off through centripetal forces, thereby forming fibers.

[0103] In some embodiments, the fiber is produced from a melt of raw materials by subjecting the molten stream to a jet of high pressure/high velocity air or by pouring the melt onto rapidly spinning wheels and spinning fiber centrifugally. If phosphorous pentoxide is provided as an additive to the melt, then a suitable phosphorous pentoxide bearing raw material is simply added at the proper amount to the raw materials being melted. Phosphorous pentoxide may be added as magnesium phosphate, ammonium phosphate or any other form of phosphate compatible with the overall chemistry. The addition of phosphorous pentoxide to the melt may range from greater than 0 to about 5 weight percent or greater.

[0104] The addition of a phosphorous containing compound as a component of the raw materials which are fiberized or as a coating which is applied to the exterior surfaces of the fiber results in a decrease of linear shrinkage of the resulting fiber after exposure to the use temperature. In addition to improvements in shrinkage, the addition of a phosphorous containing compound as a component of the raw materials which are fiberized decreases the temperature of solidification and results in an improved viscosity of the fiberization melt. In certain embodiments, the addition of a phosphorous containing compound to the fiberization melt decreases the solidification temperature about 50°C. Thus, the phosphorous containing compound may function as a viscosity

modifier in certain embodiments.

[0105] In addition to the phosphorous containing compound, the viscosity of the material melt of ingredients may optionally be controlled by the presence of viscosity modifiers, in an amount sufficient to provide the fiberization required for the desired applications. The viscosity modifiers may be present in the raw materials which supply the main components of the melt, or may, at least in part, be separately added. Desired particle size of the raw materials is determined by furnacing conditions, including furnace size (SEF), pour rate, melt temperature, residence time, and the like.

10

15

5

[0106] A compound containing a lanthanide series element may be utilized to enhance the viscosity of a fiber melt containing silica and magnesia as major components, thereby enhancing the fiberizability of the fiber melt. Other compounds which may be utilized to enhance the viscosity of the fiber melt include alumina, boria or combinations of alumina and boria. In certain embodiments, it is desirable to limit the amount of alumina present in the fiber melt chemistry to at least below about 2 weight percent, and, if possible, with raw materials used, to less than about 1 weight percent. Other elements or compounds may be utilized as viscosity modifiers which, when added to the melt, affect the melt viscosity so as to approximate the profile, or shape, of the viscosity/temperature curve of a melt that is readily fiberizable.

20

[0107] While it is not necessary that the entire exterior surface area of the individual fibers be coated with a phosphorous containing compound, a sufficient portion of the surface area may be coated with the phosphorous compound coating to provide a magnesium-silicate fiber having a continuous use or service temperature of at least 25 1400°C. Thus, according certain embodiments, a portion of the exterior surfaces of the fiber is coated with a phosphorous containing compound. According to other embodiments, substantially all of the exterior surface of the fiber is coated with a phosphorous containing compound. According to yet further embodiments, the entire 30 exterior surface of the fiber is coated with the phosphorous containing compound.

10

15

PCT/US2012/070660

[0108] The phosphorous containing compound precursor that is used to form the coating on the at least a portion of the surface of the magnesium-silicate fiber may include phosphoric acid in its various forms, such as a metaphosphoric acid, orthophosphoric acid, polyphosphoric acid, superphosphoric acid, any water soluble salt of phosphoric acid that includes the -PO $_{4}$ group, and mixtures thereof.

[0109] Metal phosphates are suitable for forming the coating of the surfaces of the magnesium-silicate fibers to increase the temperature resistance of the fibers. According to certain embodiments, the metal phosphates that may be utilized to coat the surfaces of the magnesium-silicate fibers include the alkali metal phosphates and the alkaline earth metal phosphates, ammonium phosphates, or mixtures thereof. Without limitation, suitable alkali metal phosphates. Without limitation, suitable alkali metal phosphates. Without limitation, suitable alkaline earth metal phosphates. Without limitation, suitable alkaline earth metal phosphates and calcium phosphate. Ammonium phosphate may also be used to coat the surfaces of the magnesium-silicate fiber.

[01 10] The magnesium-silicate fibers having a phosphorous containing compound coating that is derived from a phosphorous containing compound precursor on at least a portion of the exterior fiber surfaces exhibit a linear shrinkage after exposure to a service
20 temperature of 1400°C for 24 hours of less than 4 percent. Thus, the coated magnesium-silicate fibers are useful for thermal insulating applications at continuous service or operating temperatures of at least 1400°C or greater.

[01 11] The coating containing a phosphorous compound may be applied to the exterior
surfaces of the fiber either during the fiberization process (at the point of fiberization), or
after the magnesium-silicate fibers have been fiberized. It is useful to apply the coating
of the phosphorous compound on the fibers surfaces during the fiberization process.
According to this technique, the coating containing the phosphorous compound is
sprayed onto the surfaces of the fibers at the point of fiberization with a suitable spray
apparatus having a nozzle for discharging the coating composition onto the fibers. That
is, the coating composition containing a phosphorous compound is applied to the fibers as

PCT/US2012/070660

the fibers are discharged from the molten mixture of ingredients.

[01 12] The coating containing the phosphorous compound may also be applied to the fiber surfaces after completion of the fiberization process by a number of techniques including, without limitation, dipping, immersing, impregnating, soaking, spraying, or splashing the fibers with the coating composition containing a phosphorous compound.

[01 13] A method for preparing a low shrinkage, high temperature resistant, non-durable magnesium-silicate phosphorous containing fiber having a use temperature of at least 1400°C or greater is provided. 10 The method of forming the magnesium-silicate phosphorous containing fiber includes forming a material melt of ingredients comprising magnesia, silica, and a phosphorous containing compound and forming fibers from the melt of ingredients. In other embodiments, the method of forming the magnesiumsilicate phosphorous containing fiber includes forming a material melt of ingredients 15 comprising magnesia and silica, forming fibers from the melt of ingredients and coating the resulting fiber at the point of fiberization or after fiberization with a phosphorous containing compound. In other embodiments, the method of forming the magnesiumsilicate phosphorous containing fiber includes forming a material melt of ingredients comprising magnesia, silica, and a phosphorous containing compound, forming fibers from the melt of ingredients and coating the resulting fiber at the point of fiberization or 20 after fiberization with a phosphorous containing compound. The magnesium-silicate phosphorous containing fibers may be produced from the melt of ingredients by standard melt spinning or fiber blowing techniques.

25 [01 14] The fiber may be manufactured with existing fiberization technology and formed into multiple thermal insulation product forms, including but not limited to bulk fibers, fiber-containing blankets, boards, papers, felts, mats, blocks, modules, coatings, cements, moldable compositions, pumpable compositions, putties, ropes, braids, wicking, textiles (such as cloths, tapes, sleeving, string, yarns, etc.), vacuum cast shapes and composites.
30 The fiber may be used in combination with conventional materials utilized in the production of fiber-containing blankets, vacuum cast shapes and composites, as a

PCT/US2012/070660

substitute for conventional refractory ceramic fibers. The fiber may be used alone or in combination with other materials, such as binders and the like, in the production of fiber-containing paper and felt.

- 5 [01 15] A method of insulating an article using a thermal insulation containing the magnesium-silicate phosphorous containing fibers is also provided. The method of insulating an article includes disposing on, in, near, or around the article to be insulated, a thermal insulation material that contains the magnesium-silicate phosphorous containing fibers. The magnesium-silicate phosphorous containing fibers included in the thermal 10 insulation material comprise the fiberization product of about 65 to about 86 weight percent silica, about 14 to about 35 weight percent magnesia and greater than 0 to about 10 weight percent of a phosphorous containing compound.
- [01 16] A method of insulating an article using thermal insulation containing
 phosphorous coated magnesium-silicate fibers is also provided. The method of insulating an article includes disposing on, in, near, or around the article to be insulated, a thermal insulation material that contains phosphate coated magnesium-silicate fibers prepared in accordance with this process. The thermal insulation article comprises inorganic fibers comprising a fiberization product of about 65 to about 86 weight percent silica and about
 14 to about 35 weight percent magnesia and wherein at least a portion of the fiber surface is coated with a phosphorous containing compound.

[01 17] The high temperature resistant refractory inorganic fibers are readily manufacturable from a melt having a viscosity suitable for blowing or spinning fiber, are non-durable in physiological fluids, exhibit good mechanical strength up to the service temperature, exhibit excellent linear shrinkage up to 1400°C, and improved viscosity for fiberization.

EXAMPLES

[01 18] The following examples are set forth to describe illustrative embodiments of the magnesium-silicate phosphorous containing fibers in further detail and to illustrate the methods of preparing the inorganic fibers, preparing thermal insulating articles containing the fibers and using the fibers as thermal insulation. However, the examples should not be construed as limiting the fiber, the fiber containing articles, or the processes of making or using the fibers as thermal insulation in any manner.

10 Linear Shrinkage

5

Example 1

[01 19] Magnesium-silicate phosphorous containing fibers were produced by a fiber blowing process from a melt comprising about 20.5 weight percent magnesia, about 78 weight percent silica, about 1.5 weight percent alumina impurity and magnesium phosphate in an amount sufficient to yield 3 weight percent measured as P2O5 of the fiberization product.

[0120] A shrinkage pad was prepared by mixing the blown fibers, a phenolic binder and water. The mixture of fibers, binder and water was poured into a sheet mold and the water was allowed to drawn through openings in the bottom of the mold. A 3 inch x 5 inch test piece was cut from the pad and was used in the shrinkage testing. The length and width of the test pad was carefully measured. The test pad was then placed into a furnace and brought to a temperature of 1400°C for 24 hours. After heating for 24 hours, the test pad was removed from the test furnace and cooled. After cooling, the length and width of the test pad were measured again. The linear shrinkage of the test pad was determined by comparing the "before" and "after" dimensional measurements. The test pad comprising fibers of Example 1 exhibited a linear shrinkage of about 1.9 %.

30

PCT/US2012/070660

Example 2

[0121] Magnesium-silicate phosphorous containing fibers were produced by a fiber blowing process from a melt comprising about 20.5 weight percent magnesia, about 78 weight percent silica, about 1.5 weight percent alumina impurity and magnesium phosphate in an amount sufficient to yield 2 weight percent measured as P2O5 of the fiberization product.

[0122] The magnesium-silicate phosphorous containing fibers were formed into a test pad and the shrinkage characteristics of the test pad were determined according to the methods described for Example 1. The test pad comprising fibers manufactured from a melt of ingredients of Example 2 exhibited a linear shrinkage of from about 1.6 % to about 1.9 % after exposure to a use temperature 1260°C for 24 hours and exhibited a linear shrinkage of from about 2.5 % to about 3.1 % after exposure to a use temperature of 1400°C after 24 hours.

Example 3

- [0123] Magnesium-silicate phosphorous containing fibers were produced by a fiber blowing process from a melt comprising about 20.5 weight percent magnesia, about 78 weight percent silica, about 1.5 weight percent alumina impurity and magnesium phosphate in an amount sufficient to yield 1.5 weight percent measured as P205 of the fiberization product.
- 25 [0124] The magnesium-silicate phosphorous containing fibers were formed into a test pad and the shrinkage characteristics of the test pad were determined according to the methods described for Example 1. The test pad comprising fibers manufactured from a melt of ingredients of Example 3 exhibited a linear shrinkage of from about 3.1 % after exposure to a use temperature 1260°C for 24 hours and exhibited a linear shrinkage of from about 3.6 % 30 after exposure to a use temperature of 1400°C after 24 hours.

PCT/US2012/070660

Example 4

[0125] Magnesium-silicate fibers were produced by a fiber blowing process from a melt comprising from about 18 to about 27 weight percent magnesia and from about 70 to about 80 weight percent silica. A solution of ammonium phosphate was prepared and sprayed onto the surface of the fibers during fiberization, thus coating a plurality of the fibers. The ammonium phosphate solution consisted of 160g/l of diammonium phosphate and was sprayed onto the fibers at a rate of 200 ml/min. Melt pour rate was maintained at approximately 75 - 100 lb/hr. This was determined to be sufficient to provide a coating of 4.5 wt. % measured as P₂0 ₅ on the fibers. Needled fiber pads were prepared from this fiber and then tested at 1400°C for 24 hours for shrinkage. Two shrinkage tests were conducted. Certain test pads of these fibers exhibited a shrinkage of 2.5% in the first test. Other test pads comprising these same fibers exhibited a shrinkage of 2.6% in the second test.

15 Viscosity

Example 5

[0126] Magnesium-silicate fibers were produced by a fiber blowing process from a melt comprising from about 18 to about 27 weight percent magnesia and from about 70 to about 80 weight percent silica and about 2 wt. % measured as P_20_5 . The addition of phosphate pentoxide modified the viscosity of the melt and resulted in a decrease in the solidification temperature from approximately 1780°C to approximately 1730°C, thereby extending the working range of the melt by 50 degrees.

25

[0127] Additional samples of magnesium-silicate phosphorous containing fibers were prepared and tested for performance. The performance tests conducted included tests for linear shrinkage, compression recovery and dissolution rate within a physiological medium. The melt composition of these fiber samples and their corresponding test results for linear

30 shrinkage, compression recovery and dissolution rate are provided in Table I and Table II below.

TABLE I

5

Sample	SiO ₂	CaO	MgO	Al ₂ O ₃	Fe ₂ O ₃	P ₂ O ₅
6	79	0.4	18.1	1.7	0.1	0.66
7	78	0.4	18.7	1.6	0.1	1.04
8	77.4	0.3	19	1.6	0.1	1.34
9	77.9	0.3	18.1	1.6	0.1	1.67
10	78.5	0.3	17.1	1.5	0.1	2
11	75.902	0.268	20.321	1.302	0.126	1.75
12	77.392	0.231	20.694	1.342	0.106	0.066
13	77.285		20.067	1.274		0.986
14	75.888	0.197	19.06	1.139	0.094	2.77
15	75.365	0.379	18.812	1.37	0	4.06
16	75.643	0.221	18.826	1.388	0.109	2.665
17	78.791	0.203	17.219	1.293	0.05	1.81
18	79.199	0.167	18.133	0.117	0.049	1.72
19	78.142	0.151	15.551	3.707	0.038	1.53
C20	62.66	0.25	35.63	0.96	0.23	0
C21	63.25	0.25	34.35	1.05	0.11	0.96
C22	61.83	0.25	34.86	0.98	0.13	1.91
23	69.5	0.54	28.23	1.53	0.15	0
24	72.5	0.19	24.99	1.01	0.09	1.01
25	71.99	0.18	24.64	1.1	0.09	1.95
26	77.5	0.36	20.3	1.23	0.16	0.4
27	77.4	0.36	20.2	1.23	0.16	0.62
28	78.2	0.33	18.5	1.26	0.15	1.56
29	77.8	0.32	18.5	1.29	0.15	1.92
30	76.7	0.33	19.3	1.31	0.15	2.22
31	77.4	0.33	18.7	1.29	0.16	2.09
32	77	0.33	19.1	1.3	0.16	2.14
33	78.2	0.32	18	1.27	0.15	2.07
34	77.8	0.32	18.1	1.31	0.16	2.36
C35*	53-57			43-47	trace	
C36**	53-55			29-31		
C37***	70-80		18-27			

^ABlanket commercially available from Unifrax ILLC (Niagara Falls, NY, USA) under the designation DURABLANKET S.

** Blanket commercially available from Unifrax I LLC (Niagara Falls, NY, USA) under the designation DURABLANKET 2600; includes 15-17 weight percent Zr0 ₂.

5

***Blanket commercially available from Unifrax I LLC (Niagara Falls, NY, USA) under the designation ISOFRAX Blanket.

	Sh	rinkage (%	o of origina	l)	Compression Recovery (% of original)				
Sample	1260C 24hrs	1260C 168hrs	1400C 24hrs	1500C 24hrs	1260C 24hrs	1260C 168hrs	1400C 24hrs	1500C 24hrs	Diss. Rate ng/cm2 hr
6	4.75		5.85	11.66	6.99		2.40	4.33	603
7	3.70		4.25	7.94	6.31		2.45	3.21	702
8	2.09			6.71	8.44			5.15	926
9			2.70				6.09		
10	2.07		2.58	5.20	8.02		6.65	4.45	1367
11		3.30	4.30			7.14	4.27		1069
12		7.00	9.90			16.39	6.32		669
13		4.00	5.70			8.20	5.88		915
14		1.80	2.80			8.50	6.32		3334
15			4.90						
16	3.22								
17			3.54						1545
18			4.00						6012
19			5.16						
C20	39.4		43				C20	39.4	
C21	60.1						C21	60.1	
C22	32						C22	32	
23	11.8		31.1				23	11.8	
24	5.6		9.8		7.9		24	5.6	
25	3.8		9		7.7		25	3.8	
26	21.1		26.7		13		26	21.1	
27	12.3		15.9		10		27	12.3	
28	3.5		4.6	11.8	10		28	3.5	
29	2.4		3.3	8.2	9		29	2.4	
30	2.4		3.2	9.6	9		30	2.4	
31	3		4.9	13.3	11		31	3	
32	3.7		9.6	20.2	13		32	3.7	
33	3.3		5.1	10.9	9		33	3.3	

TABLE II

34	3.3	—	5.3	13.1	9	—	34	3.3	—
C35	4.5	—	11.5	—	24.2	—	16.9	—	0
C36	—	2.2	2.3	—	—	33.4	30	—	—
C37	5.7	7.2	9.1	13.3	9.8	11.9	3.3	_	375

C = comparative

[0128] As is shown in Tables I and II above, magnesium-silicate fiber samples which included a phosphate addition, measured as P2O5, generally exhibited excellent linear shrinkage values. Compression recovery and dissolution rate remained satisfactory. The results for fiber composition examples containing high levels of alumina exhibit excellent linear shrinkage (less than 5.2%) and dissolution in physiological fluid. This is quite surprising given the fact that it is known in the thermal insulating fiber art that the inclusion of high levels of alumina, such as at a level of 1.5 weight percent or more, in an alkaline earth silicate fiber results in high linear shrinkage and lower solubility as compared to fibers having lower levels of alumina.

[0129] Additional samples of magnesium-silicate fibers coated with a phosphorous containing compound were prepared and tested for performance. The performance tests conducted included tests for linear shrinkage, compression recovery and dissolution rate within a physiological medium. The melt composition of these fiber samples and their corresponding test results for linear shrinkage, compression recovery and dissolution rate are provided in Table III and Table IV below.

15

10

Sample	SiO ₂	CaO	MgO	Al ₂ O ₃	Fe ₂ O ₃	P ₂ O ₅
38	77.75	0.35	19.58	1.29	0.17	0.80
39	77.19	0.36	20.02	1.31	0.17	0.87
40	75.1	0.22	20.1	1.29	0.10	3.10
41	76.95	0.23	20.38	1.23	0.10	0.97
42	77.49	0.23	20.35	1.22	0.10	0.49
C43	62.66	0.25	35.63	0.96	0.23	0
C44	62.66	0.25	35.63	0.96	0.23	2.86
C45	62.66	0.25	35.63	0.96	0.23	5.61
46	69.5	0.54	28.23	1.53	0.15	0
47	69.5	0.54	28.23	1.53	0.15	1.48
48	69.5	0.54	28.23	1.53	0.15	4.69
49	77.99	0.18	20.43	1.24	0.09	0.72
50	77.99	0.18	20.43	1.24	0.09	1.5
51	77.99	0.18	20.43	1.24	0.09	2.2
C52	78.6	0.3	19.7	1.2	0.2	0
53	78.6	0.3	19.7	1.2	0.2	0.8
54	78.6	0.3	19.7	1.2	0.2	0.8
55	78.6	0.3	19.7	1.2	0.2	1.8
C56*	53-57			43-47	trace	
C57**	53-55			29-31		
C58***	70-80		18-27			

TABLE III

[^]Blanket commercially available from Unifrax ILLC (Niagara Falls, NY, USA) under the designation DURABLANKET S.

***Blanket commercially available from Unifrax I LLC (Niagara Falls, NY, USA) under the designation ISOFRAX Blanket.

^{**} Blanket commercially available from Unifrax I LLC (Niagara Falls, NY, USA) under the designation DURABLANKET 2600; includes 15-17 weight percent ZrO_2 .

10

	Shri	inkage (%	of origin	nal)	Compression Recovery (% of original)			
Sample	1260C	1260C	1400C	1500C	1260C	1260C	1400C	1500C
Sample	24hrs	168hrs	24hrs	24hrs	24hrs	168hrs	24hrs	24hrs
38	8.093	11.198		23.580	15.073	6.431	1.723	
39	3.652	5.247		22.120	12.770	6.299	1.569	
40			11.340					
41			3.460					
42			5.530					
C43	39.4	43						
C44	10.9	17.7			14.4			
C45	14.6	20.5			1.4			
46	11.8	31.1			29.6			
47	16.3	29.8			11			
48	14.3	25			12.9			
49	2.3	2.7			17.9		9.9	
50	1.5	2.3			21.5		12.9	
51	0.8	5			25.5		14.2	
C52		8.63					4.8	
53		2.77					15.8	
54		1.98					11.3	
55		1.87					16.4	
C56	4.5	11.5	11.7		24.2	16.9		0
C57			3.6					
C58	5.7	9.1	11.4	13.3	9.5	1.8		375

TABLE IV

[0130] As is shown in Tables III and IV above, magnesium silicate fiber samples which were coated with a phosphorous containing compound, measured as P_20_5 generally exhibited excellent shrinkage values. Compression recovery and dissolution rate remained satisfactory.

[0131] The inorganic fiber of all embodiments may further include that the phosphorous containing compound as a component of the fiberization product, a coating on at least a portion of the exterior surface of the fiber, or combinations thereof.

[0132] The inorganic fiber of all embodiments may further include that the phosphorous containing compound component of the fiberization product or the phosphorous

10

PCT/US2012/070660

containing compound coating may comprise a phosphorous pentoxide bearing material. The fiberization product and/or coating may comprise greater than 0 to about 10 weight percent of a phosphorous pentoxide bearing material, measured as P2O5, based on the total weight of the fiber. The fiberization product and/or coating may comprise greater than 0 to about 5 weight percent, measured as P2O5, based on the total weight of the fiber. The fiberization product and/or coating may comprise greater than 0 to about 1.5 weight percent of a phosphorous pentoxide bearing material, measured as P2O5, based on the total weight of the fiber. The fiberization product and/or coating may comprise greater than 0 to about 1.5 weight percent of a phosphorous pentoxide bearing material, measured as P2O5, based on the total weight of the fiber. The phosphorous containing compound may comprise at least one of ammonium phosphate or magnesium phosphate. The phosphorous containing compound may comprise magnesium phosphate.

[0133] The inorganic fiber of all embodiments may further include that the coating may comprise a solution of a phosphorous containing compound. The solution of a phosphorous containing compound may be derived from a precursor compound of phosphoric acid, a salt of phosphoric acid, or mixtures thereof. The phosphoric acid may be selected from the group consisting of metaphosphoric acid, orthophosphoric acid, polyphosphoric acid, superphosphoric acid, and mixtures thereof. The salt of phosphoric acid may be selected from the group consisting of alkali metal phosphates, alkaline earth metal phosphates, ammonium phosphates, and mixtures thereof. The salt of phosphoric acid may be selected from the group consisting of alkali metal phosphate, magnesium phosphate and mixtures thereof. The salt of phosphoric acid may comprise magnesium phosphate. The salt of phosphoric acid may comprise diammonium phosphate.

[0134] The inorganic fiber of all embodiments may further include that the fiber exhibits a linear shrinkage of less than 3.5 percent or less when exposed to 1260°C for 24 hours; a linear shrinkage of less than 5.0 percent or less when exposed to 1400°C for 24 hours; and/or a linear shrinkage of less than 2.5 percent or less when exposed to 1400°C for 24 hours.

30 [0135] The inorganic fiber of all embodiments may further include that the fiber has a solidification temperature of from about 1730°C to less than 1780°C.

PCT/US2012/070660

[0136] It will be understood that the embodiment(s) described herein is/are merely exemplary, and that one skilled in the art may make variations and modifications without departing from the spirit and scope of the invention. All such variations and modifications are intended to be included within the scope of the invention as described hereinabove. Further, all embodiments disclosed are not necessarily in the alternative, as various embodiments may be combined to provide the desired result.

PCT/US2012/070660

WE CLAIM:

An inorganic fiber comprising the fiberization product of about 65 to about 86 weight percent silica, about 14 to about 35 weight percent magnesia, and (i) a
 phosphorous containing compound which is a component of the fiberization product, or (ii) as a coating on at least a portion of the exterior surface of the fiber, or (iii) as both a component of the fiberization product and as a coating.

The fiber of claim 1, wherein the phosphorous containing compound component
 of the fiberization product and/or the phosphorous containing compound coating comprises a phosphorous pentoxide bearing material.

3. The fiber of claim 2, wherein the fiberization product and/or coating comprises greater than 0 to about 10 weight percent of a phosphorous pentoxide bearing material, measured as P2O5, based on the total weight of the fiber.

4. The fiber of claim 3, wherein the fiberization product and/or coating comprises greater than 0 to about 5 weight percent of a phosphorous pentoxide bearing material, measured as P2O5, based on the total weight of the fiber.

20

15

5. The fiber of claim 4, wherein the fiberization product and/or coating comprises greater than 0 to about 1.5 weight percent of a phosphorous pentoxide bearing material, measured as P2O5, based on the total weight of the fiber.

25 6. The fiber of claim 2, wherein said phosphorous containing compound comprises at least one of ammonium phosphate or magnesium phosphate.

7. The fiber of claim 1, wherein the coating comprises a solution of a phosphorous containing compound.

30

8. The fiber of claim 7, wherein said phosphorous containing compound is derived from a precursor compound of phosphoric acid, a salt of phosphoric acid, or mixtures thereof.

5 9. The fiber of claim 8, wherein said phosphoric acid is selected from the group consisting of metaphosphoric acid, orthophosphoric acid, polyphosphoric acid, superphosphoric acid, and mixtures thereof.

10. The fiber of claim 8, wherein the salt of phosphoric acid is selected from the
 group consisting of alkali metal phosphates, alkaline earth metal phosphates, ammonium
 phosphates, and mixtures thereof.

11. The fiber of claim 10, wherein the salt of phosphoric acid comprises magnesium phosphate.

15

12. The fiber of claim 10, wherein the salt of phosphoric acid comprises diammonium phosphate.

13. The fiber of claim 1, wherein the fiber exhibits a linear shrinkage of less than 3.5
20 percent or less when exposed to 1260°C for 24 hours.

14. The fiber of claim 1, wherein the fiber exhibits a linear shrinkage of less than 5.0 percent or less when exposed to 1400° C for 24 hours.

15. The fiber of claim 1, wherein the fiber has a solidification temperature of from about 1730°C to less than 1780°C.

PCT/US2012/070660

16. A method for preparing the inorganic fiber of any one of claims 1 to 15 comprising:

(a) forming a melt with ingredients comprising about 65 to about 86 weight percent silica, about 14 to about 35 weight percent magnesia, and a phosphorous
 5 containing compound;

producing fibers from the melt; and

optionally coating at least a portion of the surface of the resulting fibers at the point of fiberization or after fiberization with a phosphorous containing compound; or

(b) forming a melt with ingredients comprising from about 65 to about 8610 weight percent silica and about 14 to about 35 weight percent magnesia;

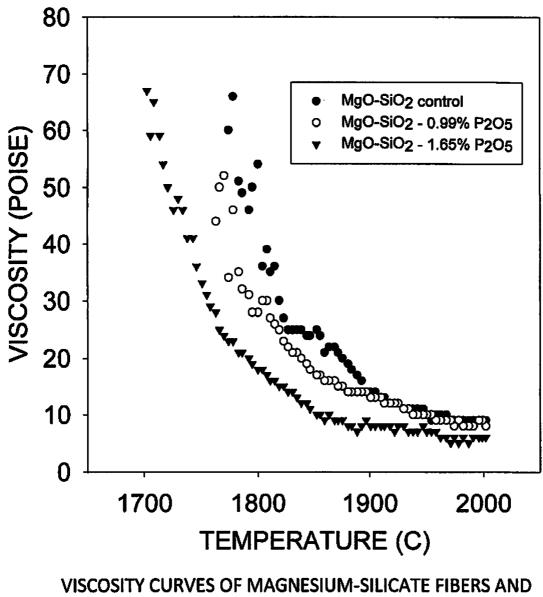
producing fibers from the melt; and

coating at least a portion of the surfaces of the resulting fibers at the point of fiberization or after fiberization with a phosphorous containing compound.

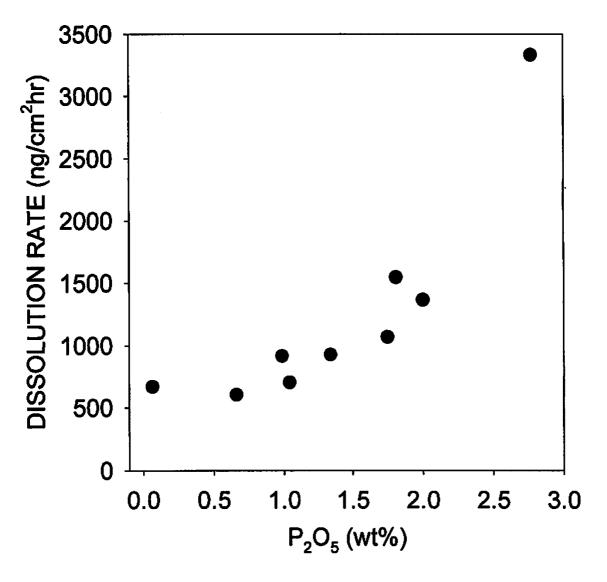
15 17. A method of insulating an article, including disposing on, in, near or around the article, a thermal insulation material, said insulation material comprising a plurality of inorganic fibe5s of any one of claims 1 to 15, wherein the inorganic fiber comprises a phosphorous containing compound incorporated throughout the fiber, or as a coating on at least a portion of said fiber, or both.

20

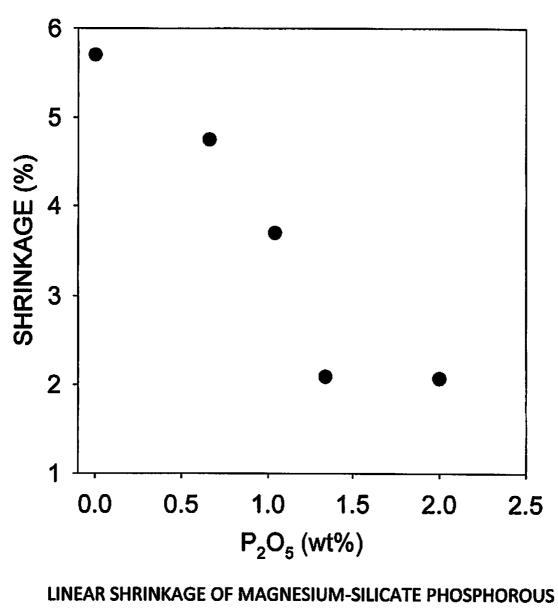
18. An inorganic fiber containing article comprising at least one of blankets, needled blankets, papers, felts, cast shapes, vacuum cast forms, or compositions, said fiber containing article comprising a plurality of the inorganic fibers of any one of claims 1 to 15.



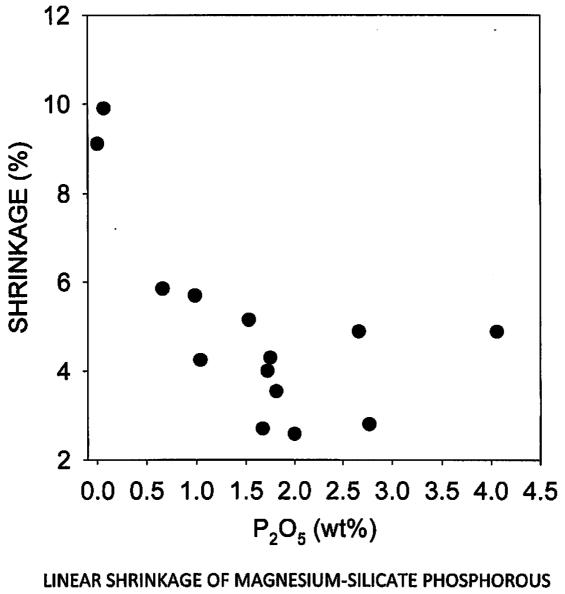
MAGNESIUM-SILICATE PHOSPHOROUS CONTAINING FIBERS



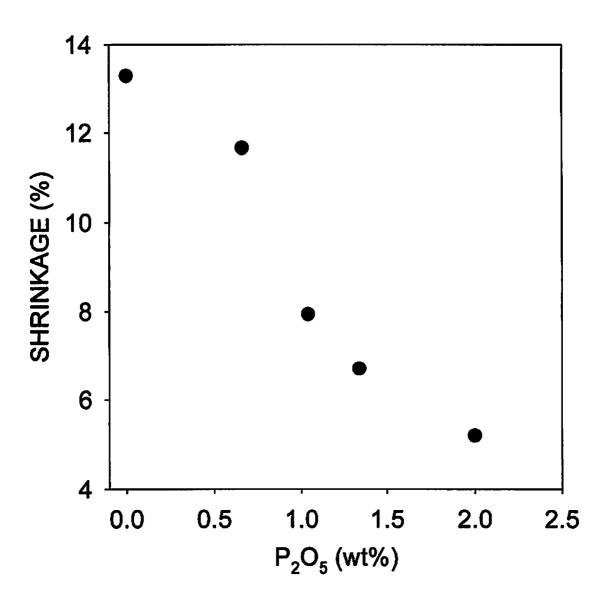




CONTAINING FIBERS AT 1260°C



CONTAINING FIBERS AT 1400°C





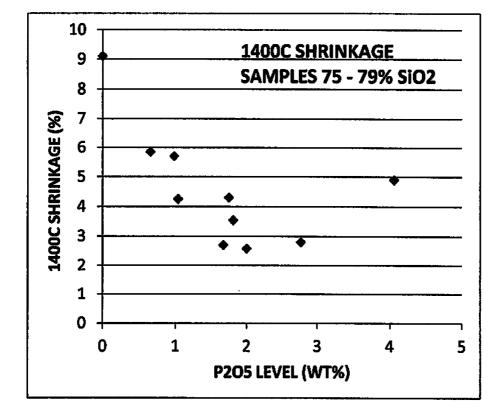


FIG. 6

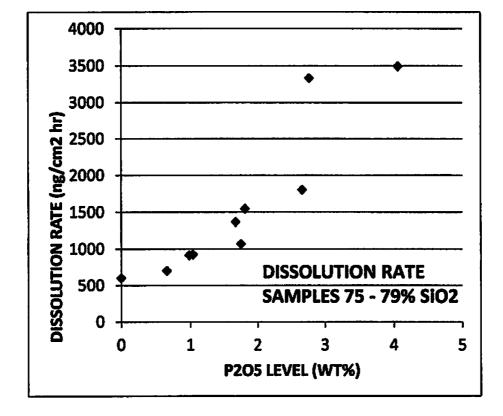


FIG. 7

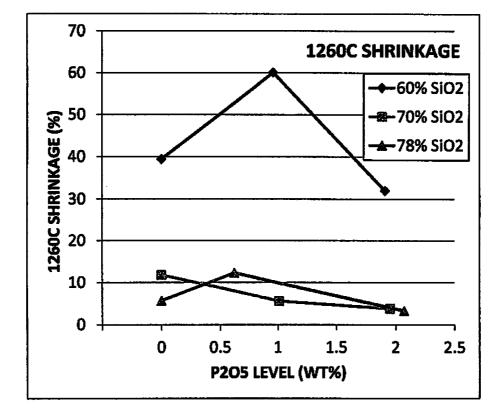


FIG. 8

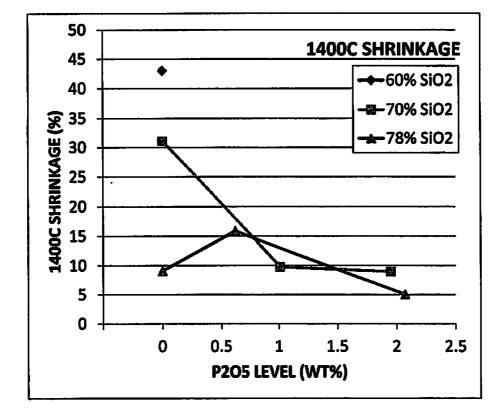


FIG. 9

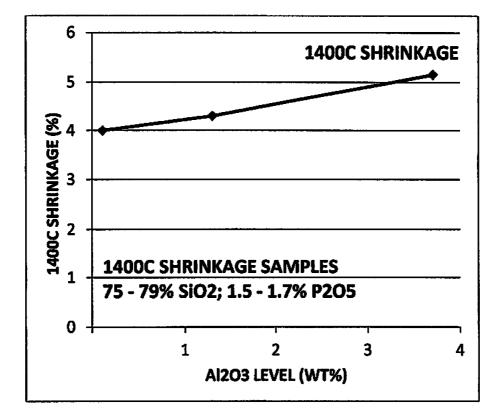


FIG. 10

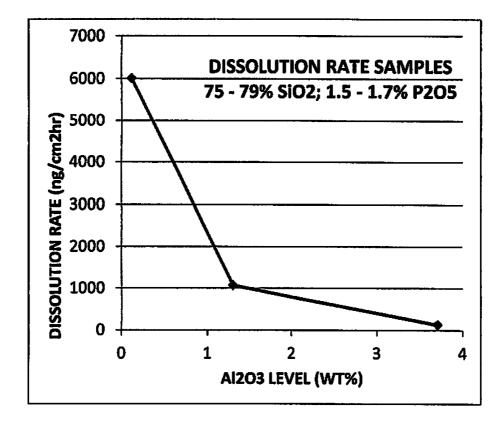


FIG. 11

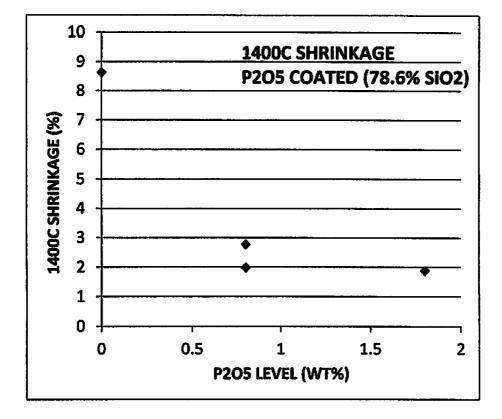


FIG. 12

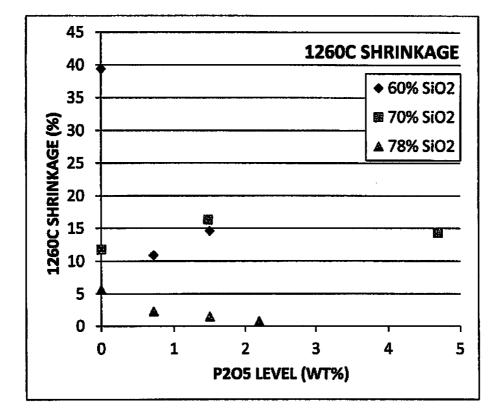


FIG. 13

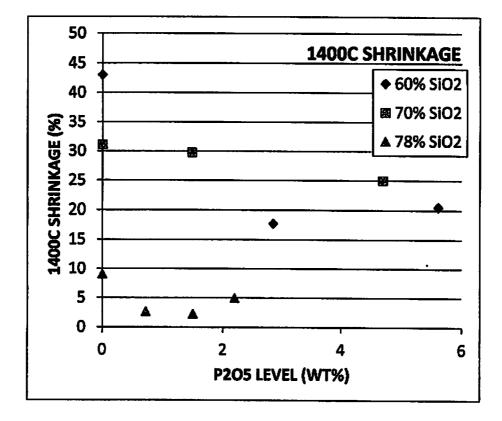


FIG. 14

A. CLASSIFICATION OF SUBJECT MATTER

D06M 13/282(2006.01)i, D06M 11/77(2006.01)1, D06M ll/44(2006.01)i

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols) D06M 13/282; C03C 13/00; C03C 3/04; C03C 13/06; C03C 25/42; C03C 13/02

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched Korean utility models and applications for utility models Japanese utility models and applications for utility models

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) eKOMPASS(KIPO internal) & Keywords: insulate, high temperature, resistant, inorganic fiber, phosphorous, silica, magnesia

c. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where app	propriate, of the relevant passages	Relevant to claim No.
X Y	US 7468336 B2 (ZOITOS, L.K.et al.) 23 Dec See abstract; claims 1,4,16,22,37.	cember 2008	1,13-18 2-12
Y	<pre>KR 10-2007-0114767 A (SAINT-GOBAIN ISOVER) See abstract; claims 1-4, 26-27.</pre>	4 December 2007	2-12
А	EP 0074655 Al (TOSHIBA MONOFRAX COMPANY LTI See abstract; claims 1-4.	D.) 23 March 1983	1-18
А	WO 2007-005836 A2 (UNKFRAX CORPORATION) 11 See abstract; claims 1-21.	January 2007	1-18
А	US 2011-0172077 Al (LEWIS A.) 14 July 2011 See abstract; claims 1-41.		1-18
Further	documents are listed in the continuation of Box C.	See patent family annex.	
"A" document to be of pau "E" earlier app filing date "L" document cited to es special rea "O" document means "P" document	tegories of cited documents: defining the general state of the art which is not considered rticular relevance dication or patent but published on or after the international which may throw doubts on priority claim(s) or which is tablish the publication date of citation or other uson (as specified) referring to an oral disclosure, use, exhibition or other published prior to the international filing date but later iority date claimed	 "T" later document published after the internation date and not in conflict with the application the principle or theory underlying the invent "X" document of particular relevance; the claime considered novel or cannot be considered the step when the document is taken alone "Y" document of particular relevance; the claime considered to involve an inventive step who combined with one or more other such docu being obvious to a person skilled in the art "&" document member of the same patent family 	h but cited to understand tion d invention cannot be o involve an inventive ed invention cannot be hen the document is
Date of the actu	ual completion of the international search	Date of mailing of the international search rep	port
25	6 April 2013 (25.04.2013)	26 April 2013 (26.04	.2013)
Name and mai	ling address of the ISA/KR	Authorized officer	
	Korean Intellectual Property Office 89 Cheongsa-ro, Seo-gu, Daejeon Metropolitan Sity' 302-70 1' Republic of Korea 82-42-472-7140	YANG, In Su Telephone No. 82-42-481-8131	SPER

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.

PCT/US2012/070660

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 7468336 B2	23.12.2008	US 2005-0326 19 A1	10. 02 .2005
kr 10-2007-0 114767 A	04. 12.2007	CA 2603292 A1	05. 10.2006
	01.12.2007	EP 1868954 A2	26. 12.2007
		EP 1868954 B1	17.09.2008
		JP 2008-534420 A	28. 08.2008
		US 2008-019 1179 A1	14. 08.2008
		Wo 2006-103375 A2	05. 10.2006
EP 0074655 A1	23.03.1983	AU 540095 B2	01.11.1984
		AU 8837082 A	24.03.1983
		CA 1189091 A1	18.06.1985
		DE 326941 1 D1	03. 04. 1986
		EP 0074655 B1	26. 02 . 1986
		JP 02029615 B	02.07.1990
		JP 1606212 C	31.05.1991
		JP 58-04612 1 A	17.03.1983
WO 2007-005836 A2	11.01.2007	BR P10613080 A2	11.01.2011
		CN 101263250 AO	10.09.2008
		CN 101263250 B	06.07.2011
		EP 1910595 A2	16.04.2008
		мх 2008-000323 А	07. 04. 2008
		US 2007-0020454 A1	25.01.2007
		US 201 1-01 18 102 A1	19.05.201 1
		US 7887917 B2	15.02.201 1
		wo 2007-005836 A3	04. 10.2007
US 201 1-0172077 A 1	14.07.2011	None	