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2,808,338

THERMAL INSULATING BODIES AND METHOD OF MANUFACTURE

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This invention relates to handleable heat insulation shapes which have low thermal conductivity comparable to that of free still air, and methods of manufacturing such insulating bodies.

This application is related to copending U. S. patent applications Serial No. 326,778, filed December 18, 1952, and Serial No. 270,748, filed February 8, 1952, the latter of which is a continuation-in-part of U. S. patent application Serial No. 226,548, filed May 15, 1951, now abandoned.

A primary object of the invention is to provide a heat insulation shape which is handleable and is retained in its form without the presence of any added bonding material, and which contains a maximum number of minute pore spacings between the finest structural units, such spacings averaging effectively below the approximate mean free path of air.

An additional and more specific object is to provide a shape-retaining, handleable heat insulation body having a thermal conductivity factor k of approximately 0.2 B. t. u./sq. ft./hour/inch ° F. at 350° F. mean.

Another object of the invention is to provide a handleable heat insulation shape having a chiefly inorganic composition adapted for insulating service at temperatures ranging as high as 1500° F.

With the above and other objects and features in view, the invention consists in the thermal insulation material and method of manufacture which are hereinafter described and more particularly defined by the accompanying claims.

An important novel feature of the herein-disclosed thermal insulating shapes is that, in general, the preferred shapes having densities in the range between approximately 12 and 30 lbs./cu. ft. and moduli of rupture in the range of approximately 10 to 120 lbs./sq. in. exhibit thermal insulating characteristics comparable to a correspondingly dimensioned volume of free still air which has a thermal conductivity of 0.26 at 350° F. mean; that is, the thermal insulating bodies of this invention, within the density range indicated, exhibit a thermal conductivity factor k of approximately 0.2–0.4 at 350° F. mean.

In the copending applications Serial No. 270,748 and Serial No. 326,778 there are disclosed and claimed thermal insulating shapes of comparable densities and thermal conductivity factors k to the insulating shapes of this invention. The preferred insulating shapes disclosed and claimed in copending application Serial No. 270,748 essentially comprise a reinforcing skeleton or network of fine staple reinforcing fibers which may be either organic or inorganic; a substantial amount of particulate filler material having an ultimate structural unit with a diameter finer than 100 millimicrons; and an inorganic or organic binder distributed throughout and holding the body in shape-retaining form. A suitable, disclosed binder for such blocks is a thermosetting phenolic-aldehyde resin. In copending application Serial No. 326,778 there are disclosed and claimed thermal insulating shapes generally similar to those described above,

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but containing, instead of an added inorganic or organic binder, a ceramic binder having a composition comprising the heat reaction product of the ingredients initially present in the aerogel particles.

We have now found that insulating bodies having the heretofore-mentioned low densities and very low thermal conductivities may be fabricated without the use of an added binder matrix and without the use of such a ceramic binder. We have found that excellent shape-retaining handleable thermal insulating bodies may be molded from a mixture essentially comprising a reinforcing skeleton of fine staple reinforcing fibers which may be either organic or inorganic, a substantial amount, and preferably at least approximately 45% by weight, of a particulate filler material having a porous or fibrillate structure such as aerogels of, for example, silica, chromic oxide, thoria, magnesium hydrate, alumina, and mixtures thereof, and, preferably, a substantial amount of finely divided opacifier materials.

Staple reinforcing fibers suitable for use in the insulating shapes of this invention may be inorganic or organic and may comprise such materials as various types of asbestos fibers of reinforcing grade, cleaned mineral fibers, fine diameter glass fibers, preferably pretreated, as with acid, to roughen the surface or otherwise to improve the surface adhesion characteristics, organic fibers, either natural such as cotton or the like, or synthetic such as acetate, rayon, acrylic fibers, the latter preferably employed in the heat-stable condition, etc. The preferred inorganic fiber is a well-opened fine staple amosite asbestos classifying as to length not less than at least 25% longer than 1/4". All of such fibers should preferably classify finer than approximately 20 microns diameter and further finer than approximately 10 microns. The amount of fiber present in the bonded insulation shape is usually small but may vary over a considerable range, depending upon the strength requirements for the particular insulation service. For most applications the fiber content may comprise up to approximately 15% by weight, and preferably approximately 5% by weight of the insulating shape is used.

As heretofore indicated, the low conductivity insulating shapes of this invention must contain a substantial amount of a particulate filler material having the structural and chemical characteristics which are necessary to this invention and which are possessed by an aerogel. The particulate filler material is exemplified by inorganic aerogels such as those of silica, chromic oxide, thoria, magnesium hydrate, alumina, etc., and mixtures thereof. Such aerogels in particle form have a "straw stack" agglomerate fibril structure, with the fibrils composing the ultimate or finest structural unit of diameter finer than 100 millimicrons. The average aerogel particles should embrace a total void or dead air space of 75–99% by volume, and may be treated to render them hydrophobic in nature.

The aerogel particles utilized to form the hydrous gel bonded insulating shapes of this invention are formed by a procedure generally similar to that outlined in the patent to Kistler, No. 2,093,454. The method generally comprises forming a gel, confining the resulting product in a pressure vessel, applying heat thereto until the liquid in the gel has reached a temperature at which the surface dimension of the liquid is so small as to produce no substantial shrinkage of the gel when the liquid is allowed to evaporate, maintaining such a temperature, and then releasing the vapor from the pressure vessel at a rate insufficient to injure the gel. Dry, porous, fibrillate aerogel particles are thus obtained. The composition of these aerogels is substantially that of the materials forming the gel. For example, the composition is almost entirely silica when a silica gel is used as the starting material.

The amount of particulate filler material of the above-defined nature which must be utilized is dependent upon the nature of the insulating shape formed and its desired use. Preferably, filler in the amount of approximately 45-95% by weight of the insulating body is employed. If the shapes are to be utilized for high temperature insulation, the preferred proportion of particulate filler of this nature is approximately 45-75% by weight, and a substantial amount of opacifying material is used.

Below 150° F., the opacity of an aerogel, such as silica aerogel, is usually adequate for insulating against heat transfer by radiation. However, above this temperature, the insulating shape should be composed in part of finely divided inorganic or organic opacifiers. These opacifiers may be of the radiation reflective type, such as metallic aluminum or silicon powder; of the radiation absorbing type, such as finely divided carbon black, powdered metals, or finely divided pigments, as for example, chromium oxide; or of the radiation scattering type, such as zircon, titanium dioxide, or other materials of high index of refraction in the infrared. Various of these materials, and mixtures thereof, may advantageously be used as opacifiers for the aerogel type filler in amounts up to approximately 100% by weight of the total aerogel content of the shape. It will be appreciated that the amount of opacifier required is usually determined by the severity of the radiation problem, which increases with the increase in temperature.

As heretofore indicated, neither an added binder matrix nor a heat-developed ceramic binder is employed to obtain the shape-retaining, handleable characteristics of the molded insulating bodies formed in accordance with the invention. We have found that strong, shape-retaining insulating bodies may be formed from the above-defined molding compositions containing staple reinforcing fibers and a particulate filler having the characteristics of aerogel particles if there is distributed substantially uniformly throughout the aerogel particles at least a relatively small amount of moisture. We have found that a mixture of staple reinforcing fibers and aerogel particles which contain at least approximately two percent by weight of water may be cold molded to densities within the range of approximately 12-30 lbs./cu. ft., to form relatively strong, hydrous gel bonded, shape-retaining bodies having excellent thermal insulating characteristics. While this minimum amount of moisture must be associated with the aerogel particles to enable formation of such handleable bodies by the molding procedure, we have further found that substantially greater amounts of moisture may be present in the composition. For example, it has been found that moisture in the amount of approximately 35% by weight of aerogel particles may be employed without detrimentally affecting the strength or insulating characteristics of the bodies thus formed. Obviously, the maximum amount of water allowable in the composition is less than that which will substantially destroy the structure of the filler particles. Any excess moisture in the molded body may be removed by drying either with or without heat.

Any suitable means may be employed to moisturize the aerogel particles prior to their incorporation in the molding mixture or to moisturize the intimately mixed molding composition containing such particles. Any conventional moisturizing equipment and conditions may be employed to obtain the desired water content in the molding mixture. For example, a molding mixture containing approximately 17% by weight of moisture based upon the aerogel content was obtained by moisturizing a mixture comprising approximately 5 parts by weight of amosite fiber, approximately 10 parts by weight of zircon particles, and approximately 85 parts by weight of silica aerogel particles in a humidifying chamber wherein the mass was exposed to air with a relative humidity of approximately 95% and a temperature of approximately 100° F. for 24

hours. A thermal insulating body having a density of approximately 13 lbs./cu. ft., a modulus of rupture of approximately 13 lbs./sq. in., and a thermal conductivity factor k of approximately 0.16 at 150° F. mean was formed by molding the above mixture at room temperature to the desired thickness.

In order to compare the aforementioned thermal insulating body with the type of shape formed in accordance with the disclosure in copending application Serial No. 270,748, a block was formed in accordance with the therein-disclosed procedure having the same composition as defined in the above paragraph but additionally containing 5 parts by weight of a phenolaldehyde resin binder. The block was molded to have the same density, that is, a density of approximately 13 lbs./cu. ft., and exhibited approximately the same thermal conductivity factor k of approximately 0.16 at 150° F. As pointed out in application Serial No. 270,748, the resin-bonded insulating blocks exhibit better thermal insulating characteristics than a loose bulk mixture of the same ingredients as present in the block. By analogy, therefore, it would appear that the blocks formed in accordance with this invention would also exhibit this characteristic. It has been found, however, that it is not possible to obtain handleable insulating blocks by the method of this invention at densities below approximately 12 lbs./cu. ft., and it is also impossible to compact by vibration a loose bulk mixture of the aerogel and opacifier particles to a density greater than approximately 10 lbs./cu. ft. For this reason, it is not possible to compare, at the same density, the thermal conductivity of a thermal insulating body of this invention and a loose bulk mixture of the ingredients employed therein.

It is immediately apparent that the procedure outlined herein is advantageous over those disclosed in the above-mentioned related copending applications. Since no added binder is present in the blocks formed in accordance with this invention, it is obvious that no substantial time must be provided during the molding procedure in order to allow the binder matrix to cure or set to its desired strength. In the same manner, no firing procedure is required as is required to develop the ceramic bond defined in application Serial No. 326,778. It is apparent, therefore, that the herein-defined procedure will be more rapid and hence more economical than those defined in these related disclosures.

Blocks formed in accordance with this invention from a molding mixture containing staple reinforcing fiber and aerogel particles which contain at least 2% by weight of moisture may be used in accordance with copending application Serial No. 326,778, filed December 18, 1952, to form the therein-disclosed ceramic bonded insulating bodies. As disclosed in that application, blocks formed in accordance with the herein-disclosed procedure may be heated for a time and at an elevated temperature, as for example 600-1600° F., sufficient to develop therein a ceramic bond essentially comprising the heat reaction product of the ingredients initially present in the aerogel particles.

It will be understood that the details given herein are for the purpose of illustration, not restriction, and that variations within the spirit of the invention are intended to be included in the scope of the appended claims.

What we claim is:

1. The method of forming a light weight, handleable insulating body having a low thermal conductivity, which comprises forming an intimate moldable mixture consisting essentially of staple reinforcing fibers in amount sufficient to form a reinforcing skeleton up to approximately 15% by weight and approximately 45 to 95% by weight of inorganic aerogel particles containing at least approximately 2% up to approximately 35% by weight of water, and molding said mixture to form a hydrous gel bonded, handleable, self-sustaining body.

ing essentially of asbestos fibers in amount sufficient to form a reinforcing skeleton up to approximately 15% by weight, approximately 45 to 95% by weight of inorganic aerogel particles containing approximately 2 to 35% by weight of water, and finely divided opacifier particles in amount up to 100% by weight of said aerogel particles, and molding said mixture to form a hydrous gel bonded, handleable, self-sustaining body.

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