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ADSORBENT-COATED THERMAL PANELS

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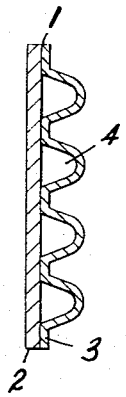


FIG. 1

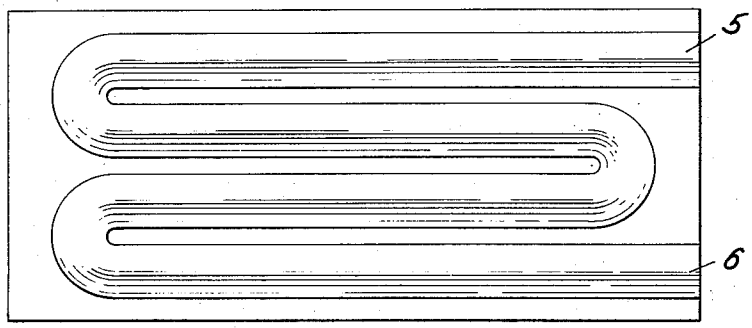


FIG. 1A

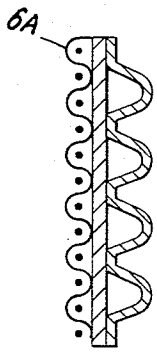


FIG. 2.

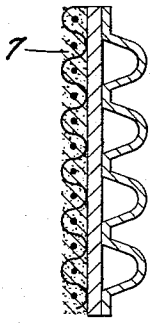


FIG. 3

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ADSORBENT-COATED THERMAL PANELS

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This invention relates to panels coated with gas adsorbents in thermal contact therewith.

The production and maintenance of extremely high vacuums in chambers (e.g., in space simulation chambers) are becoming an increasingly important technological area. One typical means presently employed to produce extremely high vacuums in chambers is the use of an oil or mercury diffusion pump, and another typical means is the use of a trap containing a cryogenic liquid-cooled gas adsorbent. Such diffusion pumps and traps are in gaseous communication with the chamber via a conduit, and the performance of these pumps and traps is limited by the diameters of the conduits, particularly where the vacuum is so low that the gas molecules in the chamber are in free molecular flow. Under the latter conditions, practical diffusion pumps and known adsorbent traps are often scarcely able to maintain a given vacuum owing to the gassing of the metals in the system and further increase in vacuum cannot be achieved. Moreover, diffusion pumps of the oil variety have the disadvantage that the oil tends to migrate into the chamber unless precautions are taken, while diffusion pumps of the mercury variety have relatively low pumping speeds. Known gas adsorbent traps have the further disadvantage that an elaborate construction is required to make efficient use of the adsorbent (e.g., the adsorbent must be spread on a plurality of thermally conductive trays which are in thermal contact with a cryogenic liquid coolant).

It is an object of this invention to provide a means for rapidly pumping gases that are in free molecular flow to very low pressures.

Further objects of this invention will be apparent from the following description of this invention.

FIGURE 1 is an edge view in section of an adsorbent-coated thermal panel of this invention.

FIGURE 1–*a* is a front view of a panel wall which can be employed in producing a coated panel of this invention.

FIGURE 2 is an edge view in section of the panel wall of FIGURE 1 covered with a screen suitable for retaining an adsorbent in a coated panel of this invention.

FIGURE 3 is an edge view in section of the screen-covered panel wall of FIGURE 2 wherein the screen retains an adsorbent. Thus, FIGURE 3 depicts a coated panel of this invention.

This invention provides a coated panel which comprises a non-porous panel wall coated with a thin layer of a gas adsorbent. The panel wall is adapted for rapid heating and cooling and is covered, on at least one level surface, with a thin layer of a gas adsorbent. The gas adsorbent is in good thermal contact with said wall, so as to rapidly attain about the same temperature as the panel wall.

The panel walls in the coated panels of this invention are adapted for rapid heating and cooling. This is readily and preferably achieved by providing channels within the panel wall or by attaching cooling conduits to the panel wall. Heat transfer media can be passed through such channels or cooling conduits to heat or cool the panel wall as desired. Such heat transfer media include refrigerated brine, liquid nitrogen, liquid hydrogen, liquid helium, cold gaseous nitrogen, cold gaseous hydrogen, and cold gaseous helium in those cases where it is desired to cool the panel wall. When it is desired to heat the panel wall, heat transfer media such as hot water, hot organic

liquids (e.g., Dowtherm), or hot gases (e.g., hot air, hydrogen, nitrogen helium, or steam) can be employed. Heating and cooling the panel wall results in a corresponding heating or cooling of the adsorbent since the adsorbent is in good thermal contact with the panel wall. Heating serves to release adsorbed gases from the adsorbent, while cooling serves to increase the gas adsorbent properties of the adsorbent. Good heat transfer between the panel wall and the adsorbent is achieved by bonding the adsorbent to the wall in such a manner that no voids separate the adsorbent from the wall surface.

The panel wall suitable for use in the coated panels of this invention can be composed of a single sheet or of a plurality of sheets suitably affixed together (e.g., by bolts or by welding or by brazing, or by soldering). Provided good heat transfer to the adsorbent and good bonding of the adsorbent can be achieved, the particular materials of which the panel walls are constructed are not critical. However, because of their excellent properties, metals—and particularly stainless steel, aluminum, and copper—are preferably the principal or only materials present in the panel walls. The particular configuration of the panel walls is not narrowly critical, and can be selected in view of the particular application for which the wall is intended. Thus, the panel walls can be curved or flat, and regularly or irregularly shaped.

In general, any gas adsorbent can be employed as a coating on the coated panels of this invention. Thus, the adsorbent can be a material such as charcoal (preferably coconut charcoal) or silica gel. However, it is preferred that the adsorbent be a crystalline zeolitic molecular sieve. Suitable zeolitic molecular sieves include both the naturally-occurring zeolitic molecular sieves and the synthetic zeolitic molecular sieves. Among the naturally-occurring zeolitic molecular sieves are chabazite, erionite, mordenite, and faujasite, these being adequately described in the chemical art. Synthetic zeolitic molecular sieves include zeolites A, D, L, R, S, T, X, and Y, as well as the mordenite-type material known commercially as Zeolon and described in Chemical and Engineering News, Mar. 12, 1956, pages 52–54.

The pore size of the zeolitic molecular sieves may be varied by employing different metal cations. For example, sodium zeolite A has a pore size of about 4 angstrom units whereas when calcium cations have been exchanged for at least about 40 percent of the sodium cations calcium zeolite A has a pore size of about 5 angstrom units.

Zeolite A is a crystalline zeolitic molecular sieve which may be represented by the formula:

$$1.0\pm0.2M_{2-n}O:Al_2O_3:1.85\pm0.5SiO_2:yH_2O$$

wherein M represents a metal, n is the valence of M, and y may have any value up to about 6. The as-synthesized zeolite A contains primarily sodium ions and is designated sodium zeolite A, described in more detail in U.S. Patent No. 2,882,243, issued Apr. 14, 1959.

Zeolite X is a synthetic crystalline zeolitic molecular sieve which may be represented by the formula:

$$0.9\pm0.2M_{2-n}O:Al_2O_3:2.5\pm0.5SiO_2:yH_2O$$

wherein M represents a metal, particularly alkali and alkaline earth metals, n is the valence of M, and y may have any value up to about 8, depending on the identity of M and the degree of hydration of the crystalline zeolite. Sodium zeolite X has an apparent pore size of about 10 angstrom units. Zeolite X, its X-ray diffraction pattern, its properties, and methods for its preparation are described in detail in U.S. Patent No. 2,882,244, issued Apr. 14, 1959.

Zeolite Y is described and claimed in U.S. patent ap-

plication Ser. No. 109,487, filed May 12, 1961, in the name of D. W. Breck.

The thickness of the coating of adsorbent on the coated panels of this invention is not narrowly critical, and can be varied as desired to meet the particular requirements of any area of application. In general, it has been found that adsorbent coatings having a thickness from 0.01 to 0.25 inch are particularly satisfactory.

The adsorbent coating can be bonded to the panel wall in the coated panels of this invention by any suitable means. In general, it is preferred to bond the coating to the wall by a procedure which involves cleaning the panel wall, roughening the panel wall, and then applying to the panel wall a mixture of the adsorbent and an inorganic binder. The cleaning step can be accomplished employing steam, pickling solutions, organic solvents, aqueous solvents, and the like alone or in various combinations, depending upon the initial condition of the panel wall and depending upon the particular material that comprises the panel wall. The roughening step can be accomplished in any of a variety ways, including grit blasting, or milling the panel walls, or attaching a screen to the panel wall. All of these methods provide raised metal portions on the panel wall which serve to lock the subsequently applied adsorbent to the panel wall. When a screen is used, it can be suitably fastened to the panel wall by first covering the panel wall with thin sheets of a brazing alloy, placing a metallic screen—preferably of relatively fine mesh—over the sheets of brazing alloy, heating the assembly to a temperature at which the brazing alloy melts, and then cooling the assembly. The brazing alloy then serves as a bonding agent between the panel wall and the screen. Alternately, the screen can be welded or soldered to the panel wall.

Suitable inorganic binders which can be employed to bond the adsorbent to the roughened panel wall include finely divided attapulgite, kaolin, sepiolite, polygarskite, kaolinite, plastic ball clays, clays of the attapulgite or kaoline types, bentonite, montmorillonite, illite, chlorite, and bentonite-type clay. Such binders are pre-mixed with the adsorbent, which is also preferably finely divided, and then applied to the roughened panel wall, preferably in the form of a slurry whose concentration can be varied to suit the thickness of the coating desired. It is important that the adsorbent coating be substantially free of any organic binder (e.g., epoxy resin binders). Such organic binders impair the adsorbent properties of the adsorbent, are not thermally stable, and possess other deleterious properties.

After the adsorbent-binder mixture has been applied to the surface of the panel wall, it is desirable to heat the adsorbent to temperatures sufficiently elevated to cause the binder to set or cure so as to effectively bond the adsorbent to the panel wall. The temperature will be dependent upon the particular binder and adsorbent employed, but generally temperatures from 500° to 650° C. are suitable, particularly where the adsorbent is a crystalline zeolitic molecular sieve. In the latter case, the heating also serves to liberate water adsorbed by the zeolitic molecular sieve. The heating to set the adsorbent-binder mixture is preferably done in an atmosphere of an inert gas such as nitrogen or argon.

FIGURE 1 is an edge view in section of a panel wall suitable for use in producing a coated panel of this invention. The panel wall **1** consists of flat sheet **2** welded to corrugated sheet **3**. The corrugations (or channels) **4** provide channels for the flow of a heat transfer medium through the panel wall.

FIGURE 1–*a* shows how the corrugations **4** of FIGURE 1 join at the ends of the panel wall so as to define a continuous conduit through panel wall **1**. Conduits **5** and **6** provide means for the flow of a heat transfer medium into and out of the corrugations.

FIGURE 2 shows a screen **6***a* attached to the outer surface of sheet **2** of the panel wall of FIGURES 1 and 1–*a*.

FIGURE 3 shows an adsorbent **7** bonded to the screen-covered panel wall of FIGURE 2.

A coated panel of this invention of the type depicted by FIGURES 1, 1–*a*, 2, and 3 was prepared as follows:

(1) The surface of the panel wall to which the absorbent was to be bonded was cleaned with a commercially available pickling solution. Both the flat sheet **2** and the corrugated sheet **3** were composed of stainless steel having a thickness of 1/16 inch. The corrugations were 3/8 inch high and 1¼ inch wide at the base. The panel wall was Type S–16, sold by the Dean Products Company. A stainless steel screen of 16 mesh having a wire diameter of about $^{18}/_{1000}$ inch was similarly cleaned with a standard pickling solution.

(2) The panel wall and the screen were further cleaned by washing with ethanol, contacting them with steam, and then rinsing them with distilled water.

(3) Excess moisture was removed from the panel wall and the screen by passing dry nitrogen gas over them.

(4) Thin strips of a silver-copper brazing alloy were laid on the surface of the panel wall.

(5) The wire screen was placed over the sheets of the brazing alloy, and the entire assembly was clamped together.

(6) The entire assembly was placed in an oven and was heated to 1300° F. to melt the brazing alloy.

(7) The assembly was removed from the oven and washed with hot water and acid.

(8) The assembly was further cleaned by passing steam over the surface, and then distilled water.

(9) Excess surface moisture was removed from the assembly by passing dry nitrogen gas over it.

(10) An aqueous slurry containing a mixture of 80 weight-percent calcium zeolite A and 20 weight-percent kaolin was applied to the surface of the panel wall coated with the screen.

(11) The entire assembly was again placed in an oven in which was maintained an atmosphere of dry nitrogen gas. The temperature of the oven was raised slowly to approximately 600° C. The oven was maintained at 600° C. for two hours, and then the assembly was cooled to room temperature by passing cooled dry nitrogen gas over the assembly. The thickness of the coating of the zeolitic molecular sieve-kaolin mixture on the panel wall was from 1/64 to 1/32 inch.

The coated panels of this invention can be used in any of a number of applications. They are especially adapted for use in pumping systems employed to produce vacuums as low as 10^{-10} torr. In such applications they are conveniently employed in conjunction with conventional pumping means. By way of illustration, the coated panels can be employed along with mechanical pumps, diffusion pumps, and cryogenic condensation pumps to achieve such vacuums. In such areas the mechanical pumps can be used to lower the pressure to 10^{-3} torr, then the diffusion pumps can be used to lower the pressure further—to 10^{-6} to 10^{-7} torr, and the combination of the cryogenic condensation pumps and the coated panels of this invention can be employed to still further reduce the pressure to 10^{-10} torr. In such systems it is preferred that the adsorbent on the coated panels of this invention is a zeolitic molecular sieve bonded to the panel wall with one of the above-described inorganic binders.

It will be apparent that the coated panels of this invention are generally useful in the same areas as are the adsorbents themselves (e.g., in selectively separating gases, in retaining catalysts, and the like).

The non-porous (i.e., gas-impermeable) nature of the panels of this invention makes them suitable for use as the outer walls in vacuum enclosures. In such cases the coated surface of the panel is oriented toward the inside of the enclosure so as to aid in producing or maintaining the vacuum.

The gases which can be adsorbed by the coated panels of this invention include oxygen, nitrogen, hydrogen, helium, argon, vaporized organic liquids, carbon dioxide, water, etc.

What is claimed is:

1. A coated panel comprising a non-porous metal wall adapted for rapid heating and cooling, said wall having a roughened first lateral surface with raised portions and a smooth second lateral surface; and a thin agglomerate layer consisting of finely divided adsorbent material and finely divided inorganic clay binder material uniformly dispersed in said adsorbent, said binder material being a component different from said adsorbent material, said agglomerate covering said roughened first lateral surface and bonded thereto.

2. The coated panel of claim 1 in which the raised portions of said roughened first metal surface have a milled contour.

3. The coated panel of claim 1 in which a metallic screen is bonded to said metal wall to form said roughened first lateral surface coated by said thin agglomerate layer.

4. The coated panel of claim 1 in which said finely

divided adsorbent material is a crystalline zeolitic molecular sieve.

5. The coated panel of claim 1 in which fluid conduit means with an inlet opening and an outlet opening are attached to the smooth second lateral surface of said wall for said rapid heating and cooling.

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