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LITERATURE SURVEY OF POROUS AND FIBROUS LOW-TEMPERATURE INSULATION

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ABSTRACT

The literature on porous and fibrous insulations suitable for use in liquefied gas containers has been reviewed and an annotated bibliography is presented.

OBJECTIVE

The primary objectives of the literature were to determine the insulating materials that have been used or proposed, and the information available on the effect of certain variables on rates of heat transfer through layers of granular insulation.

INTRODUCTION

The theoretical analysis of rates of heat transfer to the inner container of the Aro 5-liter oxygen container presented in a previous report* showed that the major portion of the heat leak occurred by radiation. It was suggested in that report that the use of granular insulation in the annular space between the inner and outer shells of the container be considered and a number of references were cited in support of this view. Since only a cursory examination of the literature had been made at that time, it was decided to prepare as complete a literature survey as possible of published information on low-temperature insulation, with special emphasis on finely-divided solid materials for use in a space under vacuum.

The primary objectives of the survey were to determine the insulating materials that have been used or proposed and the information available on the effect of the following variables on rates of heat transfer through layers of granular insulation:

1. Pressure on the insulated space.
2. Particle size and orientation.
3. Gas initially present in the insulated space.
4. Thickness of the insulated space.
5. Temperature of the retaining walls.
6. Addition of powdered metals (such as silicon) to the insulation.
7. Materials and surface treatment of the containing walls.

The results of the literature survey are presented in two parts. Part I includes a discussion of the theory of heat transfer through porous insulation, summarizes the pertinent information found, and presents recommendations. Part II is an annotated bibliography of the literature on which Part I is based. For convenience, all references except patents are given in Part II-A and are arranged in alphabetical order according to author. Patents are listed in Part II-B and are presented in chronological order for each country. United States patents are listed first, followed by British, French, and German patents.

*Banchero, J. T., Calvert, S., Elzinga, E. R., and Sinek, J. R., The University of Michigan Engineering Research Institute Report 2254-6-P, November, 1956.

PART I. ANALYSIS OF LITERATURE DATA AND RECOMMENDATIONS

THEORY OF HEAT TRANSFER THROUGH POROUS AND FIBROUS INSULATION

Heat transfer through porous and fibrous insulation is due mainly to conduction through the gas, conduction through the solid, and radiation. Convection is a negligible factor in this type of insulation.

Conduction.—From kinetic gas theory, the thermal conductivity k of a gas is proportional to the absolute pressure P of the gas. It is also proportional to the length of the mean path its molecules actually traverse; this is the mean free path L of the gas, or the distance D between the solid barriers confining the gas, whichever is smaller:

$$k \propto PL \qquad (L/D \leq 1) \qquad (1)$$

$$k \propto PD \qquad (L/D \geq 1) \qquad (2)$$

Equation (1) may be simplified, since the mean free path L is inversely proportional to P. Equation (2) may also be simplified, since D is constant for a given piece of equipment.

$$k = \text{const} \qquad (L/D \leq 1) \qquad (1A)$$

$$k \propto P \qquad (L/D \geq 1) \qquad (2A)$$

Usually, the mean free path L is smaller than D at atmospheric pressure. Upon gradually lowering the pressure of a body of gas, thereby increasing its mean free path L, the conductivity remains constant (Eq. 1A) until L becomes equal to D. From this transition point on, a decrease in pressure brings with it a proportional decrease in conductivity (Eq. 2A).

In a Dewar vessel, D is the inter-wall distance, and generally varies from 5 to 50 cm in commercial vessels. The transition pressure (at which L = D) is 10^{-3} to 10^{-4} mm Hg, and the pressure at which the conductivity is 10% of its normal value is therefore 10^{-4} to 10^{-5} mm Hg.[22] Such pressures are attained in a Dewar vessel.

The chief advantage of porous and fibrous insulators is the fact that here D, the distance between the solid barriers confining the gas, is no longer the inter-wall distance but the mean pore diameter, and is therefore much smaller.

Consequently, the transition pressure is much higher than in a Dewar vessel (and therefore easier to attain), and is also independent of the size of the vessel.

Although porous or fibrous insulating materials are poor thermal conductors, solid conduction through points of contact between granules or fibers is unavoidable. It varies with the kind of material used, especially in terms of the density of the material, and in fibrous material it also varies according to the directional arrangement of the fibers. Solid conduction generally accounts for only a small fraction of the total heat transfer.

Radiation.—At low pressures, where gas conduction is relatively small, radiative heat transfer contributes considerably towards the total heat transfer. In a Dewar vessel, it is a function of the reflectivity of the surfaces with respect to thermal radiation. A third reflecting wall cuts down radiation considerably. A layer of porous or fibrous insulating material in the interwall space also reduces radiation. A recent investigation[28] showed that this effect may be calculated by determining the absorption and scattering parameters of the material. It has also been correlated for powders by a fourth-power absolute temperature term multiplied by a Beer's-law extinction term.[14]

Overall Heat Transfer.—The overall heat transfer through porous or fibrous insulation is generally expressed as an apparent conductivity, applying Fourier's law to the overall heat transfer rate. The only inconvenience of this practice is that the radiative contribution to overall heat transfer does not vary with insulation thickness according to Fourier's law. This should be kept in mind when using the apparent overall conductivity. This conductivity has been correlated in the literature[1,21,24,42,44,46] as a function of the properties and pore dimensions of the material and of the gas properties.

PHYSICAL PROPERTIES OF POROUS AND FIBROUS INSULATION UNDER VACUUM

General.—The porous and fibrous insulations whose conductivity under vacuum has been investigated are listed in Table I, with the temperatures and pressures at which they were tested.

In general, the experimental results correspond to theory in that, below a critical pressure, conductivity decreases linearly with pressure. When very low pressures are reached, overall conductivity levels out to an approximately constant value, since the only remaining heat transfer is substantially by solid conduction and by radiation.

The most decisive property of porous insulating material seems to be the pore diameter. For porous material of uniform particle size, there seems to be no appreciable effect when the particle size is varied from 4/10 mesh to 100/200 mesh.[48] However, noticeable differences can be obtained by mixing small particles with larger particles.[48] For fibrous insulation, orientation

TABLE I

INSULATING MATERIALS USED UNDER VACUUM

Material	Temperature Range, °C Hot Side	Cold Side	Pressure Range	Ref.
Silox powder in air, CO_2, H_2	R.T.*		1 mm—Atm	1
Santocel	14.5	-196	2×10^{-6} mm—Atm	5
Santocel	R.T.	-183	10^{-2} mm	10
Mipora	R.T.	-196	10^{-2} mm—Atm	13
Celite	150	10	3 mm—Atm	15
Santocel	R.T.	-183	10^{-4} mm	17
Santocel Fiberglas Rock wool Kapok Charcoal Cork	R.T.	-253	10^{-4} mm—Atm	18
Santocel Slag wool Basic Mg carbonate Brelite	20	-183	10^{-3} mm—1000 mm	22
Santocel	R.T.		10^{-4} mm—Atm	24
Santocel in air, CO_2, Freon-12	R.T.		2 mm—Atm	25
Glass wool in air, CO_2, Freon-12	65	- 60	10^{-4} mm—Atm	27
Perlite Charcoal Glass wool Plastic microballoons Santocel Santocel AO	R.T.	-197	10^{-5} mm 2×10^{-3} mm—10^{-1} mm 2×10^{-3} mm—10^{-1} mm 2×10^{-3} mm—10^{-1} mm 2×10^{-3} mm 2×10^{-3} mm	35
Corkboard Cotton Glass wool Hair felt Kapok Rock wool Santocel Wood fiber	52	24	10^{-4} mm—Atm	36

*R.T. - room temperature

and fiber density have an effect on heat transfer, but data have been reported mainly at atmospheric pressure.

There has been relatively little research on the influence of different gases on the conductivity of porous and fibrous insulation under vacuum. Besides gaseous heat transfer, adsorption plays an important part. Carbon dioxide has a 20-30% lower conductivity than air when used with porous and fibrous insulators at atmospheric pressure and between 25 and 50°C, and Freon-12 has a 30-50% lower conductivity than air under similar conditions, as reported by one author.[27]

The effect of thickness of the insulation depends on whether heat transfer is mainly conductive or radiant under the given conditions. In conduction, heat transfer is inversely proportional to thickness. Where radiation is the main factor, as when the pressure is very low and the temperature drop large (low-temperature insulation), heat transfer is independent of thickness unless the insulation layer is so thin that it is no longer opaque to thermal radiation.

Some porous and fibrous material may be rendered more opaque to thermal radiation by adding powdered silicon, aluminum, or graphite.[48] The addition of 10% silicon to Santocel reduces conductivity by 10-20%.[35] The reflectivity of the walls does not significantly reduce heat transfer when porous or fibrous insulation is used.[5]

Examination of the data in the literature (see Table I) shows that Santocel has the most outstanding properties of the insulating materials for which data have been reported. Consequently, a more detailed account of this material and its properties are given in the following section.

Properties of Santocel.—Santocel is a porous silica aerogel manufactured by the Monsanto Chemical Company. The grade used for thermal insulation (Santocel A) is a granular material containing 93-96 weight percent silica and 2-3% sodium sulfate, so that for most purposes it may be considered as chemically inert. It has excellent temperature stability since it has been reported that it will withstand temperatures as high as 800°C apparently indefinitely without breakdown of structure.[24] It is a free-flowing powder with a bulk density of 4.5-5 lb/cu ft. A given mass of the aerogel contains 94 volume percent air in the form of minute air cells. It has been estimated that it has a mean pore size of 5×10^{-5} mm[25] and a specific adsorption surface of 400 sq m/g.[26]

The thermal conductivity of Santocel at room temperature and atmospheric pressure is 10% less than that for still air, and it has the lowest thermal conductivity at atmospheric pressure of any insulator so far reported.[24] At low pressures (0.1 μ), only a few types of glass wool have lower thermal conductivities at a mean temperature of 100°F.[27]

The thermal conductivity of Santocel at various pressures and for different conditions of temperature is listed in Table II. For purposes of comparison, the apparent thermal conductivity of a Dewar-type vacuum space was calculated as 0.011 Btu/(hr)(sq ft)(°F/in.), based on the data reported for the heat leak into a spherical, 8-liter, liquid oxygen container having silvered surfaces and a vacuum of 1×10^{-6} mm Hg.[52]

TABLE II

THERMAL CONDUCTIVITY OF SANTOCEL IN AIR
Btu/(hr)(sq ft)(°F/in.)

Pressure, mm Hg	Between Liquid N_2 and Room Temperatures			At Approximately Room Temperature		
				Ref. 24		Ref. 36
	Ref. 5	Ref. 22	Ref. 35	20-40 mesh	100-200 mesh	
1	0.0357	0.025		0.074	0.043	
10^{-1}	0 0218	0.017		0.044	0.027	
10^{-2}	0.0125	0.012		0.036	0.025	
10^{-3}	0.0087		0.01	0.035	0.024	
$\leq 10^{-4}$	0.0073				0.023	0.0255

Although Santocel is a gel, it has a sufficiently open structure so that moisture absorption is small even at large relative humidities,[24] being about 3 to 4 percent. Its behavior under mechanical pressure up to 15 psi (repeated compression and release) shows an initial reduction in volume of about 10 percent due to closer packing of the particles and no further change.[24]

RECOMMENDATIONS RESULTING FROM LITERATURE SURVEY

Porous or fibrous insulators have certain advantages over a simple Dewar-type vacuum space. These are:

1. For pressures in the vacuum space from atmospheric down to about 1×10^{-3} mm Hg absolute, such insulators have a lower heat-leak rate than a Dewar-type space. This holds for vacuum spaces as low as 1 cm in thickness.

2. The reflectivity of the confining metal walls has little, if any, effect when porous or fibrous insulators are used at temperatures below room temperature.

3. The porous or fibrous insulators provide a certain degree of mechanical support to the confining walls.

On the other hand, if the vacuum space is maintained at a pressure of about 0.5×10^{-4} mm Hg absolute or less, the Dewar-type vacuum space has a lower rate of heat leak than porous or fibrous insulators in thicknesses of 3 cm or less. Few data are available for porous insulators on the effect of the insulation thickness on the apparent thermal conductivity. For example, for Santocel, Blat et al.[5] report the same apparent thermal conductivity for thicknesses of 1 and 3 cm, while Reynolds et al.[35] report an increase in the apparent thermal conductivity as the thickness increases. The latter investigators reported data for 0.5-, 1.0-, and 1.8-inch thickness. The corresponding values of the apparent thermal conductivity were in the proportion 1.0:1.14:1.50. Consequently, it is not certain at what thickness the rates of heat leak become equal for the two cases. It appears that for any given design problem there is some critical distance below which the Dewar-type high-vacuum space has a lower rate of heat leak than a space filled with porous insulation (also under vacuum, but of the order of 10^{-3} - 10^{-4} mm Hg), and that this value may vary with the geometry of the system.

Of the various powders and fibers for which data are reported in the literature surveyed, silica aerogel (Santocel) has the best combination of properties, such as low apparent thermal conductivity, chemical inertness and temperature stability, ease of handling, and commercial availability. Therefore, Santocel appears to be the best choice for further investigation.

Since Santocel has a high specific surface, its gas adsorptivity deserves consideration. This property is advantageous in that it aids in creating and conserving vacuum when the desorbed powder is at low temperature. To accomplish this most effectively, the insulation should be heated under vacuum to desorb gases from the aerogel before the vacuum space is sealed off. Since the powder may be fluidized by the desorbed gases, evacuation may have to proceed slowly at the start, and a filter should be provided to prevent entry of the powder into the main vacuum system. It has also been suggested that other gases, such as carbon dioxide and various Freons be used to displace air from the aerogel. The gases used should have lower thermal conductivities and higher liquefaction temperatures than air. It is reasoned that adsorption of the gas by the aerogel and the lower thermal conductivity of the gas will lower the apparent thermal conductivity of the powder. This effect is quite pronounced at atmospheric pressure but should decrease as the pressure is decreased.

PART II. ANNOTATED BIBLIOGRAPHY

A. LITERATURE REFERENCES OTHER THAN PATENTS

1. Aberdeen, J., and Laby, T. H., Proc. Royal Soc. (London), A, 113, 459 (1926).

The conductivity of Silox powder (a very pure silica with some silicon) in air, CO_2, and H_2 was measured at room temperature and pressures from 1-760 mm Hg. The conductivity was found to depend on the nature of the gas but not on that of the solid (except for pore size). The results obtained were similar to those of Smoluchowski,[42] but authors criticized his experimental procedure and theoretical derivation. An empirical correlation for their own data was formulated. Stanley's patent[53] was analyzed and a thermal conductivity of 0.02 Btu/(hr)(sq ft)(°F/in.) was predicted for the insulated space. The use of Silox under vacuum is suggested as thermal insulation for liquid oxygen vessels, using a relatively low vacuum and unpolished retaining walls. The heat leak for such a vessel was calculated as 14×10^{-4} and 5.6×10^{-4} cal/(sq cm)(sec) for insulating spaces of 1.0 and 2.5 cm, respectively.

2. Anon., Mech. Eng., 69, 772 (1947).

Santocel is described.

3. Anon., Product Eng., 18, 168 (1947).

Santocel is described.

4. Austin, J. B., Symposium on Thermal Insulating Materials, American Society for Testing Materials, Philadelphia, 1939. Pages 3-67.

Detailed review of the factors influencing the thermal conductivity of nonmetallic materials, including powders.

5. Blat, M. I., Bresler, S. E., and Ryabinin, Y. N., J. Tech. Phys. (USSR), 15, 916 (1945).

Two concentric hollow steel spheres, the inner filled with liquid O_2 or N_2 and the outer at 14.5°C, were used to determine the apparent thermal conductivity of a vacuum space and of Santocel. Pressures from 2×10^{-6} to 760 mm Hg were used. Without Santocel in the vacuum space, the rate of heat transfer into the inner sphere depended on the conditions of the walls, being considerably less with polished walls. With Santocel, no such difference was obtained. The apparent thermal conductivity of the Santocel was found to be the same for a 1-cm and a 3-cm gap. The adsorption of air by Santocel over the range 10-760 mm Hg was determined.

6. Codegone, C., Atti accad. sci. Torino, Classe sci. fis. mat. e nat., 81, 75 (1945-46).

A method for measurement of thermal conductivity at low temperatures and atmospheric pressure is described, in which material to be tested is shaped into a hollow sphere, with a metal sphere full of solid CO_2 within its cavity. Applied to "cellular cement."

7. Codegone, C., Ricerca Sci., 20, 68 (1950).

The above method was applied to silica gel, vermiculite, $MgCO_3$ powder, and rock wool, using liquid O_2 in the inner sphere.

8. Dewar, J., Proc. Roy. Inst., 15, 815 (1898).

First to report that the introduction of finely powdered solids into the vacuum space of a Dewar vessel increases the effectiveness of the insulation. Also studied effects of metallic foil as reflective insulation. Results are qualitative only.

9. Edberg, E. A., Refrig. Eng., 64, 38 (1956).

Described expandable polystyrene insulation.

10. Eichstaedt, H. A., NBS Report No. 3517, 8 (1955).

Report on the liquid O_2 container manufactured by Ronan and Kunzel, Inc. It is a cylindrical aluminum container, built in 50-, 150-, and 500-gallon sizes. In the 500-gallon container, an 8-in. vacuum space is filled with Santocel, held at 10 microns or less. Conductivity was roughly 0.015 Btu/(sq ft)(hr)(°F/in.).

11. Finck, J. L., Bur. Standards J. Research, 5, 973 (1930).

Investigated conductivity of fibrous materials as a function of bulk density. Investigated conductivity of mixed fibers, of randomly arranged and orderly fibers, and the influence of moisture. Observed radiation decrease due to the effect of Al powder as opacifying agent when dusted on kapok, etc.

12. Finck, J. L., Ind. Eng. Chem., 31, 824 (1939).

Expressed solid conduction and radiation in fibrous insulators as functions of bulk density, and thus determined optimum density.

13. Fradkov, A. B., Doklady Akad. Nauk S.S.S.R., 81, 549 (1951).

Made conductivity measurements on liquefied gas containers insulated with a 25-mm layer of Mipora (a urea-formaldehyde resin) at pressures between 10 microns and atmospheric.

14. Fulk, M. M., Reynolds, M. M., and Park, O. E., NBS Report No. 3517, 151 (1955).

Absorptivities of many kinds of metal surfaces at 76°K for 300°K black-body radiation are listed. Radiative heat transfer through powders was found to follow the T^4 law multiplied by a Beer-Lambert **exponential** damping factor due to the powder.

15. Glaser, P. E., Ph.D. thesis, Columbia University (1955),as reported in: Glaser, P. E., and Kayan, C. F., Refrig, Eng., 64, 31 (1956).

Measured conductivity of Celite (dry, calcined diatomaceous earth) in He, NH_3, and air between 3 and 760 mm Hg and between 50 and 300°F.

16. Goalwin, D. S., Refrig. Eng., 59, 251 (1951).

Tested techniques and equipment for the transfer of liquid O_2, found glass fiber and silica gel to be preferable as insulation.

17. Hallett, N. C., Altman, H. W., Yeager, M. L., and Newton, C. L., NBS Report No. 3517, 5 (1955).

Constructional details are given for the cylindrical liquefied gas containers made by H. L. Johnstone, Inc., holding 2000 to 10,000 liters. Annular space of 7 in., filled with Santocel (vacuum pre-dried) at 0.1 micron, has conductivity of 0.015 Btu/(sq ft)(hr)(°F/in.) in practical operation.

18. Johnstone, H. L., Hood, C. B., and Bigeleisen, J., NBS Report No. 3517, 139 (1955).

Made experimental comparison of different insulations between 20°K and room temperature, and at pressures between 0.1 micron and atmospheric. In general, powder insulation was found to be more effective than fibrous or cellular insulation, and to vacuum alone. Of all insulations tried out, Santocel proved by far the most efficient.

19. Johnson, V. J., and Wilson, W. A., NBS Report No. 3517, 246 (1955).

The NBS hydrogen liquefier plant is described. Silica gel at liquid N_2 temperature was used to adsorb N_2 and O_2 from gas to the liquefier.

20. Jones, C. L., Paint Ind. Mag., 62, 390 (1947).

Described Santocel and its preparation.

21. Kannulick, W. G., and Martin, L. H., Proc. Roy, Soc. (London) A, 141, 144 (1933).

Made conductivity measurements on powdered magnesia, glass, and carborundum at 1-80 mm, using air, H_2, and CO_2. Found correlation of Aberdeen and Laby[1] to be unsatisfactory in this range. Derived a second-degree algebraic expression.

22. Katan, L. L., Vacuum, 1, No. 3, 191 (1951).

Made conductivity measurements on liquid O_2 vacuum-jacketed container, using Santocel, slag wool, basic magnesium carbonate, and brelite, an expanded volcanic mineral. Pressure range, 1 micron to 1000 mm; temperature range, 70 to -360°F. Detailed experimental procedure is described.

23. Kistler, S. S., Nature, 127, 741 (1931).

First prepared silica aerogel that retains its porous structure by supercritical evaporation of the alcogel.

24. Kistler, S. S., and Caldwell, A. G. , Ind. Eng. Chem., 26, 658 (1934).

Measured conductivity of silica aerogel, between 0.1 micron and atmospheric pressure and at room temperature, for different particle sizes. Although they confirmed the data of Aberdeen and Laby,[1] they did not feel justified in deriving the latters' correlation. Derived theoretical correlation based on solid and gas conduction within the granules, and on solid-solid and solid-gas-solid conduction between granules. Radiation not included in derivation, since temperature drop in tests was low.

25. Kistler, S. S., J. Phys. Chem., 39, 79 (1935).

Based on his theoretical correlation,[24] he calculated pore size of silica aerogel; conductivity measurements were made between 2 and 740 mm and at room temperature, using air, CO_2, and Freon.

26. Kistler, S. S., J. Phys. Chem., 46, 19 (1942).

Based on his theoretical correlation,[24] he calculated the specific surface area of silica aerogel. Found it to agree with results from adsorption measurements.

27. Lander, R. M., Trans. A. S.H.A.E., 61, 151 (1955).

The tests of Rowley et al.[36] were continued, substituting air partially or totally by CO_2 and Freon-12. Glass wool was investigated further by varying the mean temperature between -50° and +100°F, and by using specimens of different densities.

28. Larkin, B. K., A Study of the Rate of Thermal Radiation Through Porous Insulating Materials, Ph.D. thesis, The University of Michigan (1957).

Studied radiant heat transfer through Styrofoam, Foamglass and Fiberglas boards at atmospheric pressure and at temperatures between 70°F and 400°F, using thicknesses from 1/8 to 1 in. Analyzed scattering and absorption parameters. Found that the optimum fiber diameter is 2-5 microns, and the optimum foam-bubble diameter is of the same order of magnitude, for minimum radiation. Found that increasing foam density decreases radiation if absorption is more important, but increases radiation if scattering is more important; with fibers, increasing density decreases radiation. Surface emissivity was found to have influence on radiant heat transfer.

29. Macormack, J. G., Refrig. Eng., 41, 311 (1941).

Described "Ferrotherm," a lead-tin-alloy coated steel, having a dull surface but having a higher reflectivity for infrared radiation than polished stainless steel, and hence used as reflective insulation.

30. Mason, R. B., Ind. Eng. Chem., 25, 245 (1933).

Described experiments on the use of reflective insulation, especially Al foil, at normal temperatures. Heat transfer may be made to approximate that of still air by appropriate use of Al foil.

31. McIntire, O. R., and Kennedy, R. N., Chem. Eng. Progr., 44, 727 (1948).

A list of plastics used for the production of foam insulators is given, both U.S. and foreign. Styrofoam, manufactured by Dow Chemical Company, is described.

32. McIntire, O. R., and McCuaig, D. W., Refrig. Eng., 52, 217 (1946).

Described Styrofoam, a foam insulator made by the Dow Chemical Company. Cut down radiation considerably by adding 1 percent of Al powder as opacifying agent.

33. Moench, G. C., Glas u. Hochvakuum-Tech., 1, 1, 9 (1952).

Discussed the use of carbon black as insulation between the glass walls of a Dewar vessel, and the installation of a third wall in the vacuum space.

34. Ogden, F. F., and White, J. F., Refrig. Eng., 52, 411 (1946).

Described Santocel. Discussed use of opacifying agents with Santocel and concluded that they were only effective above 100°F.

35. Reynolds, M. M., Brown, J. D., Fulk, M. M., Park, O. E., and Curtis, G. W., NBS Report No. 3517, 142 (1955).

Thermal conductivity of perlite at 0.01 micron and of Santocel at 2 microns or less was measured. Cylindrical jacketed-wall N_2 containers were used. Found that with perlite, finer particles are better insulators. The addition of Al powder or the use of black surfaces decreases conductivity. However, vacuum and no powder at 0.01 micron is superior to any of the above uses of perlite. Santocel proved superior to pure vacuum. Annular spaces of 0.5, 1.0, and 1.8 in. were used. Santocel AO, containing 10 percent Si, has roughly 80 percent conductivity of Santocel. Influence of outer shell-temperature was also measured.

36. Rowley, F. B., Jordan, R. C., Lund, C. E., and Lander, R. M., Trans. A.S.H.V.E., 58, 155 (1952).

Tested conductivity of several fibrous and porous insulators at a mean temperature of 100°F, at 10^{-4} and at 760 mm Hg. Readings of intermediate pressures were not considered accurate enough. Substances tested were corkboard, cotton, glass wool, hair felt, kapok, rock wool, Santocel, and wood fiber. Apparatus used was guarded hot-plate, with a temperature difference of 50°F and a mean temperature of 100°F.

37. Sanders, V., Chem. Eng. Progr, 44, 804 (1948).

Described Foamglass, a cellular glass insulator made by Pittsburgh Corning Corp.

38. Scott, R. B., NBS Report No. 5027 (1956).

Empty vacuum space, with clean annealed Cu, Ag, or Al as highly reflective surfaces, competes with powdered insulation, since the latter needs enough width to decrease solid conduction.

39. Shand, L. D., Offic. Dig. Federation Paint and Varnish Production Clubs, 277, 185 (1948).

Described Santocel.

40. Simonova, L. K., J. Applied Chem. (USSR), 16, 87 (1943)(French summary).

Investigated conductivity and heat capacity of porous insulators as function of particle size. Worked with activated carbon and silica gel at atmospheric pressure and room temperature. Contrary to Kistler and Caldwell[24] and to Smith and Wilkes,[41] found conductivity to increase on powdering the sample.

41. Smith, W. R., and Wilkes, J. B., Ind. Eng. Chem., 36, 1111 (1944).

Investigated conductivity of carbon black as function of particle size, at atmospheric pressure. Found conductivity to be independent of particle size, similar to Kistler and Caldwell[24] and contrary to Simonova,[40] except when carbon black is pelletized, where conductivity increases.

42. Smoluchowski, M., Bull. Intern. acad. sci. de Cracovie, 5B, 129 (1910), and ibid., 8A, 548 (1911).

First to conduct quantitative thermal conductivity measurements for different powders in air, at room temperature, between 1 and 760 mm. Plotted conductivity versus log P. Found solids conduction to be negligible, and that conductivity depended on the nature of the gas but not on that of the solid, except for pore size. Derived theoretical expression of conductivity based on analogy to slip-flow under vacuum. Recognized the importance of radiation.

43. Taylor, C. S., and Edwards, J. D., Heating, Piping, and Air Conditioning, 11, 59 (1939).

Studied the effect of an oxide layer on the reflectivity of Al.

44. Topper, L., Ind. Eng. Chem., 47, 1377 (1955).

Derived a mathematical expression for the conductivity of a porous insulation as function of pore size, pore form, and number of pores per unit volume.

45. Van Buskirk, E. C., and Surland, C. C., Chem. Eng. Progr., 44, 803 (1948).

Described Flotofoam, an urea-formaldehyde foam insulator made by the U. S. Rubber Company.

46. Verschoor, J. D., and Greebler, P., Trans. Amer. Soc. Mech. Engrs., 74, 961 (1952).

Derived a theoretical correlation of conductivity vs pressure for fibrous insulation as function of fiber density and diameter. Made conductivity measurements on glass fiber insulation between 1 micron and atmospheric pressure, at room temperature. The mathematical model considers solid conduction, gas conduction, and radiation.

47. Waite, H. J., NBS Report No. 3517, 158 (1955).

Lists mechanical and thermal properties of Styrofoam, a polystyrene cellular insulation made by the Dow Chemical Company, and its uses.

48. White, J. F., Ind. Eng. Chem., 31, 827 (1939).

Tested Si, Al, and graphite as opacifying agents with Santocel, at atmospheric pressure and between 100 and 600°F.

49. White, J. F., Refrig. Eng., 41, 171 (1941).

Described Santocel.

50. White, J. F., Chem. Eng. Progr., 44, 647 (1948).

Described Santocel, and commented on work of Blat et al.[5]

51. Wilkes, J. B., Ind. Eng. Chem., 31, 832 (1939).

Described use of reflective insulation.

52. Wilson, W. A., NBS Report 5041 (1957).

A complete report on the design and performance of an 8-liter spherical liquid oxygen tank with a 1-in. Dewar-type vacuum space.

B. PATENT LITERATURE

U. S. Patents

53. U. S. Patent 1,071,817, Sept. 2, 1913 (Stanley, W.).

Double-wall metallic vacuum flank, in which gas-absorbing powder at reduced pressure is contained in the annular space. Insulating power thus obtained claimed to be the same as that of a conventional Dewar flask having an absolute pressure 100 times lower, when used for storing liquefied gases.

54. U. S. Patent 1,694,967, Dec. 11, 1929 (Coolidge, W. D., to General Electric Co.).

Use of lamp black and P_2O_5 as adsorbent in vacuum space of vessels used for storing liquefied gases.

55. U. S. Patent 2,093,454, Sept. 21, 1937 (Kistler, S. S.).

Preparation of silica aerogel by supercritical evaporation of silica aquagel directly, without passing through the alcogel stage.

56. U. S. Patent 2,164,143, June 27, 1939 (Munters, C. G., to Aktiobolaget Termisk Isolation).

Flexible double-jacketed container, with powder under vacuum in jacket.

57. U. S. Patent 2,396,459, March 12, 1946 (Dana, L. J., to The Linde Air Products Co.).

Large, rigid, double-walled tank, not less than 2 ft ID, annular space not less than 3 in., at pressures between 1 micron and 10 mm, filled with finely divided solid material. When used as liquid oxygen tank, a heat flux of less than 5 Btu/(sq ft)(hr) is specified.

British Patent

58. Brit. Patent 252,385, May 20, 1925 (Union Générale Cooperative).

Use of charcoal as adsorbent in vacuum space of vessels used for storing liquefied gases.

French Patent

59. Fr. Patent 657,440, July 12, 1928 (I.G. Farbenindustrie A.-G.).

Use of silica as adsorbent in vacuum space of vessels used for storing liquefied gases.

German Patents

60. Ger. Patent 510,577, June 20, 1927 (I.G. Farbenindustrie A.-G.).

Use of silica as adsorbent in vacuum space of vessels used for storing liquefied gases.

61. Ger. Patent 528,497, July 4, 1930 (Societé l'air liquide).

Heat-insulating means for apparatus at very low temperatures.

62. Ger. Patent 806,786, June 18, 1951 (Perlick, A., to Gesellschaft für Linde's Eismachinen A.-G.).

Use of finely divided silica as filler for liquefied gas vessels. The silica forms a solid gel, which, since it will not flow, provides greater safety in the event of damage to the vessels.

63. Ger. Patent 809,561, July 30, 1951 (Kahle, H., to Gesellschaft für Linde's Eismachinen A.-G.).

Jacketed-wall insulating case for storage of liquefied gases, with an adsorbent contained in the vacuum jacket.