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D. L. McElroy and J. F. Kimpflen, editors



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The quality of the papers in this publication reflects not only the obvious efforts of the authors and the technical editor(s), but also the work of these peer reviewers. The ASTM Committee on Publications acknowledges with appreciation their dedication and contribution of time and effort on behalf of ASTM.

Foreword

The ASTM Symposium on Insulation Materials, Testing, and Applications was held in Bal Harbour, Florida, on December 6-9, 1987. The event was sponsored by ASTM Committee C-16 on Thermal Insulation, the Department of Energy (DOE), the Building Thermal Envelope Coordinating Council (BTECC), and the Oak Ridge National Laboratory (ORNL).

The Symposium was co-chaired by J. F. Kimpflen, Certain-Teed Corporation, and I. S. Seigler, Ralph M. Parsons Company. Mr. Kimpflen has co-edited the present volume with D. L. McElroy, Oak Ridge National Laboratory.

Introduction

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Since ASTM Committee C-16 on Thermal Insulation was founded in March 1938, its policy has been to sponsor programs periodically that provide the latest information regarding test methods and insulation materials. As a result of these programs, more than a dozen ASTM Special Technical Publications have been published on thermal insulation materials and technology, most of them in the last decade, based on symposia sponsored by the committee.

Activity and interest in thermal insulation have increased as a result of the space program in the 1960s and energy conservation in the 1980s. In 1978, over 500 people participated in a symposium when the topic was enlarged to include not only technical measurement but also all the parameters related to thermal insulation for energy conservation. The trend has continued with sponsorship of conferences and symposia alternating between ASTM and the American Society of Heating, Refrigerating, and Air-Conditioning Engineers (ASHRAE), with additional sponsorship by both the U.S. Department of Energy and Oak Ridge National Laboratory. Proceedings of the ASHRAE-sponsored conferences are available as ASHRAE Special Publications. The ASTM-sponsored conferences and symposia continue to be published as ASTM Special Technical Publications.

A public awareness of rising energy prices has continued to focus attention on the use of thermal insulation as an effective energy-conserving measure. Research and development in thermal insulation materials, testing methods and apparatus, and innovative applications provide the basis for obtaining substantially improved thermal performance. This volume presents the insulation community with an update on the technical database in residential, commercial, and industrial applications.

ASTM Committee C-16 on Thermal Insulation is pleased to continue to provide the format for discussion and documentation of this complex subject. Also, this conference provides excellent input to Committee C-16 in discharging its responsibility for promulgating standards for insulation materials, systems, and test methods.

D. L. McElroy

Oak Ridge National Laboratory, Oak Ridge, Tennessee: editor

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Certain-Teed Corporation, Valley Forge, Pennsylvania; symposium co-chairman and editor

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Evaluation of Fumed-Silica Insulation for a Thermal Conductivity Standard Reference Material

REFERENCE: Rennex, B. G., Somers, T. A., Faison, T. K., and Zarr, R. R., "Evaluation of Fumed-Silica Insulation for a Thermal Conductivity Standard Reference Material," *Insulation Materials, Testing, and Applications, ASTM STP 1030*, D. L. McElroy and J. F. Kimpflen, Eds., American Society for Testing and Materials, Philadelphia, 1990, pp. 3-14.

ABSTRACT: Standard Reference Materials (SRMs) used for thermal conductivity measurements are required by industry, academic, and government laboratories for calibrating heat-flux-meter apparatus or checking the accuracy of guarded-hot-plate apparatus. New thermal conductivity SRMs are sought to improve the accuracy and extend the operational range of these apparatus. An advisory panel recommended the development of a low thermal conductivity SRM having approximately the same thermal conductivity as blown fluorocarbon foams, suitable for a temperature range of -175 to 900° C. Fumed-silica insulation was recommended by the advisory panel as the most suitable material.

The National Bureau of Standards (NBS) examined four lots of fumed-silica insulation and recommended one candidate for further development as a low thermal conductivity SRM. The four lots of fumed-silica insulation were examined for their relative handleability, material uniformity, variability of thermal conductivity measurements, and effect of heat treatment on the measured thermal conductivity of the materials. Thermal conductivity measurements were conducted using the NBS 1-m Guarded Hot Plate for each lot of fumed-silica insulation at a mean specimen temperature of 24°C. Analysis of the thermal conductivity data was performed using the NBS statistical analysis program, Dataplot.

KEY WORDS: standard reference material, fumed-silica insulation, thermal conductivity measurements

Thermal conductivity Standard Reference Materials (SRMs) are used by private and government laboratories for calibrating or verifying the accuracy of thermal conductivity apparatus. Materials of known thermal properties are required for calibration of relative heat-flux devices such as the heat-flow-meter apparatus (ASTM C 518). Recent SRM requirements have emphasized a material which is stable over time, capable of withstanding high temperature, and has a thermal conductivity similar to blown fluorocarbon foams.

Previous work identified the need for a Standard Reference Material having a low thermal conductivity of approximately 0.023 W/m · K (0.16 Btu · in./h · ft² · °F) and suitable for a temperature range of -175 to 900° C (-280° F to 1700° F) [1,2]. An advisory panel consisting of representatives of the Mineral Insulation Manufacturers Association, the Department of Energy, Oak Ridge National Laboratory, and ASTM decided that a fumed-silica insulation product was the most promising material. This study evaluates four fumed-silica insulation products and recommends a candidate for further development as a low thermal conductivity SRM.

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Four different fumed-silica insulation products were obtained and tested at the National Bureau of Standards (NBS) using the 1-m Guarded Hot Plate. The insulation products were obtained from the following manufacturers: Manville Services Corporation (U.S.), Micropore International LTD (U.K.), Grunzwick & Hartman und Glasfaser Ag (FRG), and Wacker-Chemie GmbH (FRG).⁴ Addresses for the manufacturers are provided in the Appendix.

Evaluation of the four products is based on the following criteria: (1) relative handleability of each material, (2) variability of the material's physical properties, (3) variability of the apparent thermal conductivity measurements for each lot of material, (4) material stability under heat treatment, and (5) economic considerations. Test data for each product are presented anonymously. Hereafter, references to each insulation product are identified simply as Product A, B, C, and D. One of these products is selected based on the overall results of the evaluation criteria. This product is now a candidate material for further SRM development.

Sample Description

The fumed-silica insulation products were manufactured in the form of square insulation boards. The material obtained from three of the manufacturers was 600 by 600 mm, 25 mm thick (24 by 24 by 1 in.). Material from the fourth manufacturer was 680 by 680 mm, 25 mm thick (27 by 27 by 1 in.). All specimens from each manufacturer were from the same lot of material. A total of 33 specimens was obtained.

Fumed-silica insulation is a silica-aerogel composite. The material is micro-porous, comprised primarily of submicron particles of amorphous silica bonded together in a cellular structure. The submicron distance between silica particles effectively reduces gas conduction by inhibiting molecular collisions of gases within the cell walls. Manufacturer's literature states the distance to be less than 0.1 μ m, which is less than the mean-free-path length for O_2 and N_2 molecules at standard temperature and pressure. Due to the micro-porous nature of the material, the apparent thermal conductivity depends not only on the type of gas within the interstices of the material but also on the gas pressure.

The composite material contains additional materials for other desirable properties. Metallic opacifiers are added to reduce radiative heat-transmission at high temperatures. Structural support for the material is enhanced by strengthening fibers such as quartz.

Handleability

All specimens were extremely fragile and required care when handling. The size of the specimens handled ranged from 600 mm (24 in.) square to 680 mm (27 in.) square. With careful handling, most of the specimens could be lifted by the edges without the material breaking. The specimens were transported by first lifting one edge of the specimen and then sliding a flat support board underneath the material. The support board was then used to transport the specimen.

The material itself was friable. Abrasion from sliding the support board under the specimen produced a thin layer of "dust" material on the support board. A residual layer of dust material was also deposited on the individual's hands working with the material.

As part of the overall evaluation, the specimens were heat-treated in a gas-fired oven. For testing the specimens in the oven, a stainless steel support-rack that encased the edges of the specimens was used. The support-rack was designed to transport the specimens in either a horizontal or vertical orientation. The support-rack held specimens 600 by 600 mm (24 by 24 in.),

⁴Because the products are potential candidates for SRMs, it is necessary to identify the manufacturers so that other laboratories may obtain the materials. This in no way represents an endorsement of these products by NBS.

requiring the larger size specimens to be cut. Manufacturers recommend following normal precautions associated with nuisance dusts when machining the material. Further information on the health issues concerning fumed-silica is available in Ref 3.

An index rating the relative durability of each material is presented in Table 1. Each specimen was rated for its ability to withstand movement when lifted and transferred onto a support board. The relative durability of the material was rated before and after the heat treatment. Overall, Product B was considered the most durable product, with C and D a close second. Even before heat treatment, the edges of Product A crumbled easily when lifted.

Variability of Physical Properties

Prior to heat treatment, measurements of thickness and density were made on all specimens. The specimen thickness was measured at ten locations using a special thickness caliper capable of 0.1 mm resolution. Mass measurements were made with a digital laboratory scale, uncertain by $\pm 0.1\%$. The density measurements were estimated to be uncertain by $\pm 0.5\%$. The average thickness and density for Products B, C, and D are presented in Table 2. Each average includes the standard deviation. For Product A, the lot size was too small for statistical evaluation and only the measured values are given. Product C was found to be the most uniform.

Product		After Heat Treatment						
	Room Temperature (21°C)	550°C 7.6 h	+	600°C 6.7 h	+	625°C 5.3 h	+	650°C 5.8 h
A	3	3		3		3		
В	1	1		1		1		1
C	1	1		1		2		2
D	1	1		1		2		2

TABLE 1-Durability of specimens."

- Specimen(s) did not crack when transferred to support board; however, a support board is still advised.
- (2) Specimen(s) cracked when transferred to support board; support always required.
- (3) Specimen edge(s) crumbled easily when transferred to support board; support always required and extra-careful handling necessary.

TABLE 2-Material uniformity prior to heat-treatment.

Product	Lot Size	Average Lot Thickness, mm	Average Lot Density, kg/m³	Range of Density, kg/m ³
A	2	24.7	361.2	361.2
		26.3	330.1	330.1
В	10	25.7 ± 0.2^{a}	379.3 ± 3.8^{a}	±6.2
С	10	25.5 ± 0.1	301.4 ± 2.0	±2.8
D	11	26.4 ± 0.8	280.4 ± 9.5	±16.4

[&]quot;Standard deviation of average. For Product A, the standard deviation and range of density are not provided due to the statistically small lot size. Measured values are given instead.

[&]quot;Durability Index:

Variability of Specimen Apparent Thermal Conductivity

Test Method

The apparent thermal conductivity of the fumed-silica specimens was measured using the NBS 1-m Guarded Hot Plate. The guarded hot plate was operated in a one-sided mode at a mean-specimen temperature of 23.9°C (75°F). Tests were performed in accordance with procedures described in ASTM Test Method C 177 and ASTM Practice C 1044. The apparent thermal conductivity (λ) of the specimen was determined by the equation

$$\lambda = \frac{L}{R} \tag{1}$$

where L is the specimen thickness (m), and R is the thermal resistance of the specimen (K \cdot m²/W). The average thickness for each specimen was computed by averaging ten thickness measurements made within a 400 mm (16 in.) diameter circle corresponding to the metering-area of the guarded hot plate. An uncertainty of $\pm 0.8\%$ was estimated for the thickness measurement. The uncertainty in the thermal resistance measurement was estimated to be $\pm 0.5\%$ [4]. Assuming the two uncertainties to be equally probable in both directions, an overall uncertainty of $\pm 0.9\%$ was determined for the thermal conductivity measurement.

Pressure Dependence

Early work on fumed-silica material by Tye [5] showed that the apparent thermal conductivity of the material depends on the pressure of the gas and the type of gas used in the hot plate measurement. For tests reported here, the apparent thermal conductivity measurements were conducted with air at atmospheric pressure. Variations in atmospheric pressure occurred throughout the test period. The apparent thermal conductivity of the material varied linearly with these variations of barometric pressure. A variation of 15 mbar (0.44 in. Hg) produced an approximate change of 0.7% in the specimen apparent thermal conductivity. The specimen apparent thermal conductivity was determined by averaging measurements taken over several hours when the barometric pressure was stable within ± 1 mbar (± 0.03 in. Hg). Pressure measurements were made with a barometric stripchart recorder calibrated at NBS to within ± 0.4 mbar (± 0.01 in. Hg). A summary of individual apparent thermal conductivity measurements, as well as material density, average thickness of the metered area, and barometric pressure, is presented in Table 3.

Analysis of Data

For comparison purposes, all apparent thermal conductivity measurements were conducted at a mean-specimen temperature of 23.9°C (75°F). The apparent thermal conductivity measurements of the fumed-silica material were found to depend on two independent parameters: material density (ρ) and barometric pressure (P). A linear regression equation dependent on these two parameters was used to predict the thermal conductivity for each lot of material. For the initial analysis the following form was used:

$$\lambda(\rho, P) = a_0 + a_1 \cdot (\rho - \rho_{ref}) + a_2 \cdot (P - P_{ref}) \tag{2}$$

The term a_0 is the thermal conductivity at the reference value of material density, standard pressure, and ambient temperature application. The two coefficients a_1 and a_2 are regression coefficients for the material density and barometric pressure, respectively. The following values

TABLE 3—Summary of specimen measurements at a mean temperature of 23.9°C.

Product & Sample No.		,		Barometric Pressure, mbar	Measured Thermal Conductivity, W/m·K	Predicted Thermal Conductivity W/m·K	
A	A21 A22	361.2 330.1	25.0 26.0	1011 1005	0.02267 0.02342	а	
В	A11	381.9	25.5	998	0.02153	0.02158	
	A12	384.0	25.8	1019	0.02159	0.02192	
	A13	371.6	25.9	1003	0.02191	0.02196	
	A14	377.1	26.3	1004	0.02206	0.02183	
	A15	381.5	26.0	999	0.02116	0.02161	
	A16	383.0	26.1	1000	0.02148	0.02159	
	A17	377.7	25.4	1007	0.02135	0.02187	
	A18	380.2	25.6	1002	0.02261	0.02170	
	A19	375.4	26.1	998	0.02160	0.02176	
	A20	380.9	26.0	1012	0.02241	0.02187	
С	A1	299.0	25.5	997	0.02173	0.02171	
	A2	298.9	25.4	993	0.02172	0.02166	
	A3	300.9	25.5	1012	0.02200	0.02191	
	A4	300.7	25.7	1002	0.02173	0.02181	
	A 5	300.6	25.6	1008	0.02186	0.02187	
	A 6	302.3	25.5	1000	0.02182	0.02184	
	A 7	299.7	25.5	1005	0.02172	0.02181	
	A8	303.4	25.7	1002	0.02191	0.02189	
	A9	304.4	25.7	994	0.02179	0.02185	
	A10	304.0	2 5.5	996	0.02192	0.02185	
D	A31	290.6	26,2	1012	0.02093	0.02114	
	A32	286.8	25.6	1006	0.02148	0.02103	
	A33	282.4	26.0	1008	0.02102	0.02098	
	A34	299.6	25.2	986	0.02101	0.02107	
	A35	281.7	25.9	999	0.02078	0.02091	
	A36	274.1	27.3	1016	0.02105	0.02092	
	A37	280.5	25.9	1016	0.02087	0.02102	
	A38	266.9	27.0	1007	0.02066	0.02075	
	A39	275.7	26.9	994	0.02084	0.02078	
	A40	269.5	27.9	1010	0.02078	0.02081	
	A41	276.9	27.0	988	0.02074	0.02075	

^aThe analysis of measurements for Product A was not done due to the statistically small lot size.

for the reference parameters were used in the analysis: ρ_{ref} = average density of the lot of material (kg/m³) (see Table 2), and P_{ref} = standard pressure of 1013.25 mbar (29.921 in. Hg) [6].

The regression analysis was accomplished using the NBS statistical analysis program, Dataplot [7]. The measured data were fit to Eq 2 using the method of least squares. The material density and barometric pressure coefficients computed using Dataplot are presented in Table 4. An insufficient sample number precluded analysis of the measurements of Product A.

The residual standard deviation (δ_{res}) in units W/m·K is shown as the last line in Table 4. The value is the smallest for Product C, indicating the curve fit is best for these measurements. The calculated regression coefficients in Table 4 are preliminary, based on small ranges of the independent parameters, ρ and P. The negative sign for the density coefficient of Product B is attributed to the large variation in thermal conductivity within the lot of material for a small range in material density.

Regression Coefficients	Product B	Product C	Product D
2 ₀	0.02194	0.02194	0.02100
ı ₁	-0.2783E-04	0.3076E-04	0.1473E-04
i ₂	0.1889E-04	0.9936E-05	0.7621E-05
δ_{res}	0.506E-03	0.069E-03	0.197E-03

TABLE 4-Regression coefficients for the thermal conductivity model.

Measured values of apparent thermal conductivity are presented in Table 3. Using the regression coefficients in Table 4, predicted thermal conductivity values were calculated using Eq 2 and are also presented in Table 3. The percent deviation for each apparent thermal conductivity data point was calculated from the equation

% deviation =
$$\frac{(\lambda_{\text{meas}} - \lambda_{(\rho, P)})}{\lambda_{(\rho, P)}} \cdot 100$$
 (3)

where

 $\lambda_{\text{meas}} = \text{apparent thermal conductivity of measured data point } (W/m \cdot K), \text{ and } \lambda_{(\rho,P)} = \text{predicted thermal conductivity at the same density and barometric pressure } (W/m \cdot K).$

Percent deviation versus material density, barometric pressure, and apparent thermal conductivity are shown in Figs. 1, 2, and 3, respectively. Each plot indicates the percent deviation of the measurement point with the product letter. In the worst case, Eq 1 is able to predict the thermal conductivity of Product B within $\pm 2.1\%$, Product C within $\pm 0.2\%$, and Product D within $\pm 1.0\%$. The percent deviation for Product C is extremely low and within the uncertainty of the measurement. The plots show the smallest scatter for Product C.

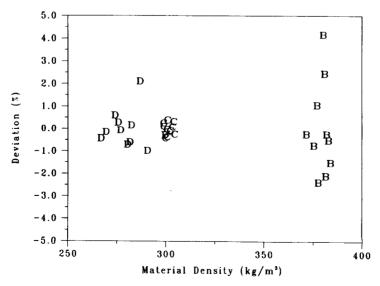


FIG. 1—Deviation (%) as a function of material density.