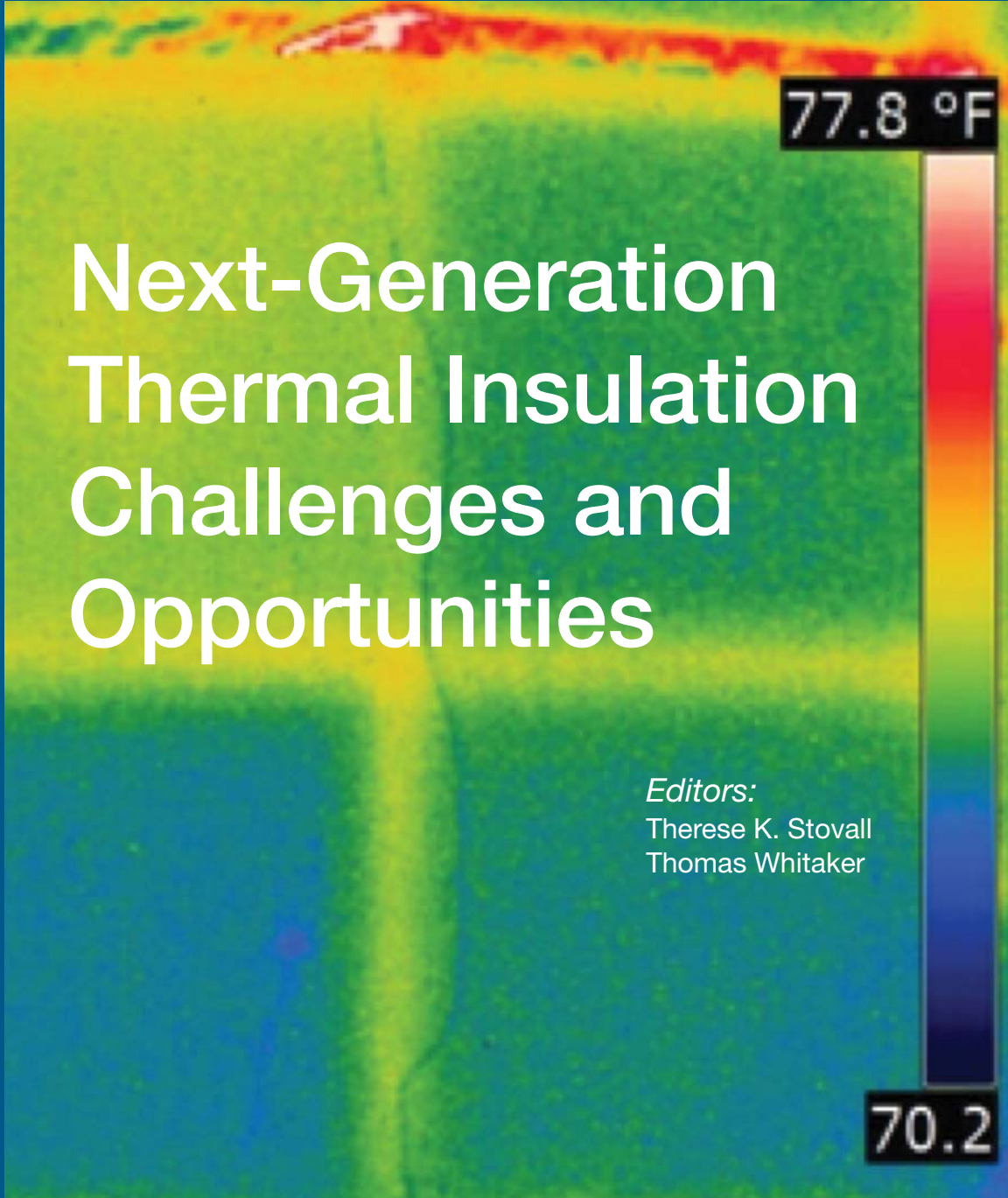


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STP 1574



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Next-Generation Thermal Insulation Challenges and Opportunities

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Editors:

Therese K. Stovall
Thomas Whitaker



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Foreword

This compilation of *Selected Technical Papers*, STP1574, *Next Generation Thermal Insulation Challenges and Opportunities*, contains peer-reviewed papers that were presented at a symposium held October 23–24, 2013 in Jacksonville, FL. The symposium was sponsored by ASTM International Committee C16 on Thermal Insulation.

The Symposium Co-Chairpersons and STP Co-Editors are Therese K. Stovall, Oak Ridge National Laboratory, Retired, Oak Ridge, TN, USA and Thomas Whitaker, Industrial Insulation Group, Retired, Grand Junction, CO, USA.

Contents

Overview	vii
Performance of Vacuum Insulation Panel Constructed With Fiber–Powder Composite as Core Material	1
P. Mukhopadhyaya, D. van Reenen, and N. Normandin	
Development of an Advanced Foam Insulation Based on Thermosetting Resins	11
F. A. Shutov, I. V. Scherbanev, and D. W. Yarbrough	
Full-Thickness Thermal Testing of Fiberglass Insulation Using an ASTM C518-10 Heat Flow Meter Apparatus	17
P. M. Noonan and T. R. Jonas	
Standard Reference Material 1450d, Fibrous Glass Board, for Thermal Insulation Measurements	39
R. R. Zarr and S. D. Leigh	
Development and Use of an Apparatus for In Situ Evaluation of the Thermal Performance of Building-Envelope Components	53
W. C. Thresher and D. W. Yarbrough	
Moisture Content Measurements in Wood and Wood-Based Materials—Advancements in Sensor Calibration and Low-Moisture-Content Regime	66
N. Shukla, D. Kumar, D. Elliott, and J. Kosny	
High-Performance External Insulation and Finish System Incorporating Vacuum Insulation Panels—Foam Panel Composite and Hot Box Testing	81
A. Seitz, K. Biswas, K. Childs, L. Carbary, and R. Serino	
An Innovative Low-Emissivity Insulation Developed in Korea	101
Y. C. Kwon, Y. O Kim, and G. Y. Lee	
Design Considerations for Sustainable Extruded Polystyrene (XPS) Thermal Insulation	119
R. E. Smith, J. M. Alcott, and M. H. Mazor	

An Investigation on Bio-Based Polyurethane Foam Insulation for Building Construction	131
P. Mukhopadhyaya, M.-T. Ton-That, T.-D. Ngo, N. Legros, J.-F. Masson, S. Bundalo-Perc, and D. van Reenen	
Lab-Scale Dynamic Thermal Testing of PCM-Enhanced Building Materials	142
N. Shukla, P. Cao, R. Abhari, and J. Kosny	
Presentation at ASTM C16 Symposium on Next-Generation Thermal Insulation Challenges and Opportunities	155
C. Petty	
Evaluation of Homogeneity Qualification Criteria in the Accelerated Aging of Closed-Cell Foam Insulation, Results after Five Years of Full-Thickness Aging	173
T. Stovall	

Overview

Founded in 1938, ASTM Committee C16 is celebrating 75 years of progress in the science and technology of insulation. George Santayana said that, “Those who cannot remember the past are condemned to repeat it.” In his keynote address at this most recent symposium, Dr. David McElroy, member emeritus, reminded us that our process of openly sharing technology advances in these symposia moves us forward only as long as we retain our awareness of the past accomplishments. The first C16 symposium, held in 1939, included only four papers but encompassed the development of new property test methods, modeling challenges, and insulation application problems. There have obviously been tremendous advances in the insulation industry over these 75 years, but these areas are still actively pursued at C16, as shown by the papers included in this publication representing the 21st symposium hosted by this committee. In 1939, property test methods were concerned with measuring thermal conductivity and specific heat of simple homogenous materials. Today’s work strives to measure similar performance indices for complex three-dimensional systems with multiple components, phase change materials, and in-situ systems. Application issues for advanced vacuum insulation systems include aging processes that occur over a 20- to 70-year time frame, even more challenging than the five-to-ten year time period studied for cellular plastic insulations.

Energy conservation, via improved insulation, is one of the most effective ways to reduce the environmental impacts of energy production. Considering the scope of that challenge, this symposium was organized to look at the “next-generation” of insulation, and the related challenges of supporting these technology advances with more sophisticated measurement systems. For some applications, the fields of computer modeling and property measurements are actually merging into an integrated process to provide the information needed to predict thermal performance. The work described in this publication was therefore organized in two areas: advanced materials and advanced measurement technology.

The Editors would like to thank the authors and reviewers who dedicated their time to ensure the high quality of the work reported here. We would also like to thank the staff at ASTM who shepherded us through this process, especially Heather Blasco, Susan Reilly, Mary Mikolajewski, Hanna Sparks, and Kathy Dernoga, and the ASTM staff manager for C16, Rick Lake. Finally, we would like to thank the industry and government support that helped 76 members attend the symposium, especially in light of the economic conditions.

Therese K. Stovall
Thomas E. Whitaker

STP 1574, 2014 / available online at www.astm.org / doi: 10.1520/STP157420130105

Phalguni Mukhopadhyaya,¹ David van Reenen,² and
Nicole Normandin²

Performance of Vacuum Insulation Panel Constructed With Fiber-Powder Composite as Core Material

Reference

Mukhopadhyaya, Phalguni, van Reenen, David, and Normandin, Nicole, "Performance of Vacuum Insulation Panel Constructed With Fiber-Powder Composite as Core Material," *Next-Generation Thermal Insulation Challenges and Opportunities*, STP 1574, Therese K. Stovall and Thomas Whitaker, Eds., pp. 1-10, doi:10.1520/STP157420130105, ASTM International, West Conshohocken, PA 2014.³

ABSTRACT

Buildings consume about 40 % of the national energy requirement in a developed country, and the addition of thermal insulation in building envelope construction is considered as the most primary and effective way to reduce energy consumption in buildings. Recent upgrades of energy codes in Europe and North America have also recommended higher levels of insulation in building envelopes. All these factors have provided a fresh impetus for the search for high-performance thermal insulation. Among various nonconventional insulations being introduced in the construction industry, as the next-generation thermal insulation, vacuum insulation panel (VIP) appears to be one of the most promising insulation materials, with the highest thermal insulating capacity (up to 10 times more thermally efficient than conventional thermal insulation materials). Quite naturally, the application of VIP in building envelope construction offers many advantages such as increased energy efficiency of

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¹National Research Council Canada, Construction Portfolio, 1200 Montreal Rd., Campus-Building M-24, Ottawa, ON, K1A 0R6, Canada (Corresponding author), e-mail: phalguni.mukhopadhyaya@nrc-cnrc.gc.ca

²National Research Council Canada, Construction Portfolio, 1200 Montreal Rd., Campus-Building M-24, Ottawa, ON, K1A 0R6, Canada.

³ASTM Symposium on *Next-Generation Thermal Insulation Challenges and Opportunities* on October 23-24, 2013 in Jacksonville, FL.

exterior building envelopes, thinner wall thickness, optimum space use, reduced material consumption, etc. However, the acceptance of VIP in the construction industry is critically dependent on the cost and long-term performance. The expensive core material (e.g., precipitated silica or fumed silica) is one of the main reasons for the higher cost of VIPs that offer a satisfactory long-term service life in building envelope applications. To overcome this cost barrier for the mass application of VIPs in the building industry, researchers at the National Research Council Canada – Construction Portfolio have developed a low-cost fiber-powder composite core material for the VIP. This paper briefly introduces the concept of fiber-powder composite and present performance assessment data from laboratory-scale trial VIPs (300 mm by 300 mm) constructed with fiber-powder composite core materials.

Keywords

vacuum insulation panel, core material, fiber-powder composite, nanoporous, thermal insulation

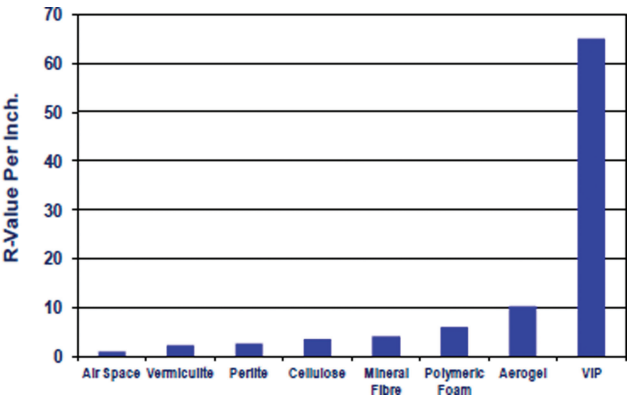
Introduction

The continuous increase in energy consumption by nations around the world [1], and more specifically by the industrially developed parts of the world, is a global concern that needs to be checked and reversed in order to avoid unprecedented environmental disaster. Thus, the reduction of energy consumption in every aspect of our daily life is considered the key to tackling the issues related to global warming and its adverse effects on the environment. Buildings consume up to 40 % of our total national energy requirement [2], and thermal insulation is a key component that determines the energy efficiency of the built environment.

One of the most promising types of high-performance thermal insulation currently being considered for building envelope construction by researchers and practitioners around the world is the vacuum insulation panel (VIP). VIPs can be more than 10 times as thermally efficient as conventional thermal insulation materials (Fig. 1). VIPs are made with open porous core materials enclosed in an impermeable gas barrier (Fig. 2) and have three major components: (1) an open porous core material that imparts mechanical strength and thermal insulating capacity, (2) a gas barrier/facer foil that provides the air- and vapor-tight enclosure for the core material, and (3) getter/desiccant inside the core material to adsorb residual or permeating atmospheric gases or water vapor in the VIP enclosure.

Based on the information available to date [3–6], it appears that the use of VIPs is an attractive technological option for substantially increasing the energy efficiency of the built environment. However, in Canada and elsewhere in the world, VIPs are rarely used for building envelope construction or are selectively used if space for traditional insulation is too expensive or not available. The primary reasons for the lack of real-life applications are cost and an absence of consumer confidence in the constructability and long-term performance of VIPs [7,8]. Researchers

FIG. 1 Thermal resistivity of VIP compared to those of several common insulating materials.



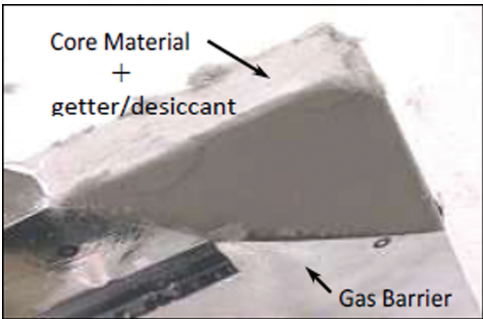
and manufacturers across the world are focusing their efforts at this moment to address these issues and integrate VIPs into building envelope construction.

This paper presents selected results from a research initiative that has developed the concept of alternative core materials with the aim of reducing the cost of VIPs. Reports about the concept of alternative core materials and the development of a fiber–powder composite core material have been published [9,10], and this paper moves one step further by assessing the thermal performance of a VIP made with an alternative fiber–powder composite core material.

Research Background: Why Focus on Core Materials?

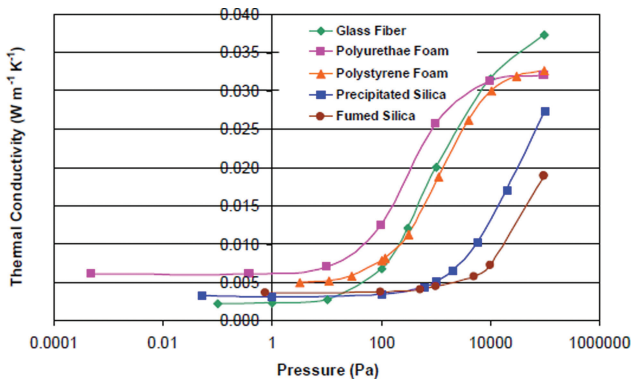
The long-term thermal performance of a VIP depends primarily on the gas barrier/facer foil and the core material. The gas barrier helps to maintain the air- and

FIG. 2 Vacuum insulation panel.



vapor-tight environment, and the core material provides the mechanical and insulating properties. Studies have indicated that commercially available vacuum technology and foil materials provide effective resistance against air and vapor permeation through the gas barrier (i.e., facer) and the facer seam [3,11]. However, studies have also indicated that, very slowly but steadily, air or vapor (or both) will penetrate into the core material to raise the internal pore pressure and thus increase the thermal conductivity of the core material [7]. The extent of this reduction in thermal insulating capacity depends at the initial stage on the capacity of the getter/desiccant material to adsorb residual or permeating atmospheric gases or water vapor inside the VIP enclosure and ultimately on the relationship between pore pressure and the thermal conductivity change of the core material. The most commonly used core materials, as reported in the literature, are glass fiber, open-cell polyurethane foam, open-cell polystyrene foam, precipitated silica, and fumed silica (e.g., carbon/silica aerogel). The relationship between the thermal conductivity and the pore pressure for these materials is shown in Fig. 3 [12]. It is very obvious from this figure that there is a threshold limit of pore pressure beyond which the thermal conductivity of the core materials increases almost exponentially; for some core materials this threshold value is as low as approximately 10 Pa, and for some others it is as high as 10 000 Pa. This phenomenon concerning the ability of the porous core material to maintain a lower thermal conductivity at higher pore pressures is directly related to the pore structure of the material [3,13]. Core materials with smaller open pores have a greater ability to maintain lower thermal conductivity at higher pore pressure (Fig. 3). For this reason, precipitated silica, fumed silica, and fumed silica materials with micro- or nanoporous structures maintain a very low vacuum-level thermal conductivity characteristic almost all the way up to a pressure level of 10 000 Pa, unlike glass fiber, open-cell polyurethane foam, and open-cell polystyrene foam. Incidentally, nanoporous thermal insulating core materials are

FIG. 3 Change of thermal conductivity of core material with pore pressure [12].



much more expensive than purely micro- or macroporous materials, not because the basic materials required for the construction of nanoporous material are expensive, but because the manufacturing process to impart the nanoporous open-cell structure is very cost intensive. Quite naturally, the expensive core material is one of the main reasons for the higher cost of VIPs that offer a satisfactory long-term service life of a building envelope.

To overcome this cost barrier for the mass application of VIPs in the building industry, National Research Council Canada – Construction Portfolio (NRC Construction) researchers are engaged in a research initiative that investigates the development of a low-cost core material for VIPs [9,10].

Development and Performance of Fiber-Powder Composite

The fiber-powder composite core materials (Fig. 4) developed in this study [9,10] were made with fiber (i.e., mineral oxide fiber and high-density glass fiber) and powder (pumice and zeolite) insulation materials. The fundamental principles behind the development of fiber-powder composite core materials and the thermo-physical properties (thermal properties, pore size distribution, etc.) of the fibers and powders considered in this study have been published elsewhere [9,10]. The fiber-powder composite core material has thin layers of mineral oxide/high-density glass fiber board and pumice/zeolite powder sandwiched together as shown in Fig. 4. The layered fiber-powder composite materials (density $\approx 340 \text{ kg/m}^3$) were placed inside an evacuation box (Fig. 5) for the thermal measurements. Thermal properties were measured using a vacuum guarded hot plate (VGHP) (Fig. 6). The relationships between the pore pressure and the thermal conductivity of these three newly introduced low-cost core materials are shown in Fig. 7. This figure also draws a

FIG. 4 Composite fiber-powder insulation.

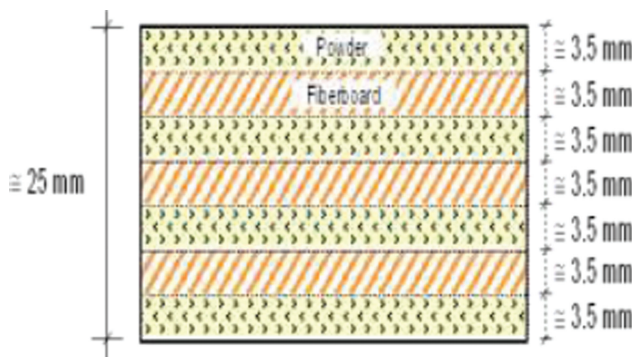
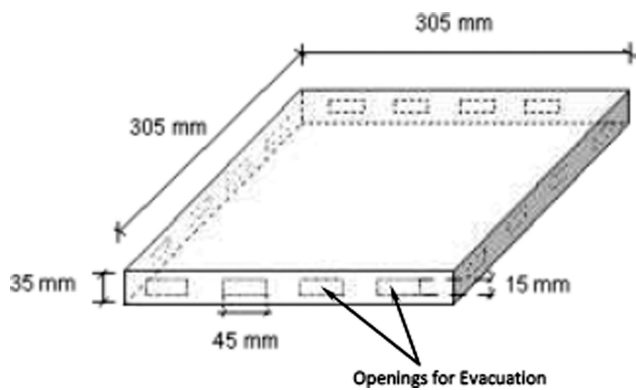


FIG. 5 Evacuation box for the powder material.



comparison between the thermal properties of traditionally expensive core materials (precipitated silica and fumed silica) and three newly developed low-cost core materials. Although at the atmospheric pressure level (100 000 Pa) the thermal conductivity values of the composite core materials are much higher than those of precipitated silica and fumed silica, the thermal conductivity values from vacuum to 10 000 Pa are very comparable with those of precipitated silica and fumed silica.

FIG. 6 Vacuum guarded hot plate.

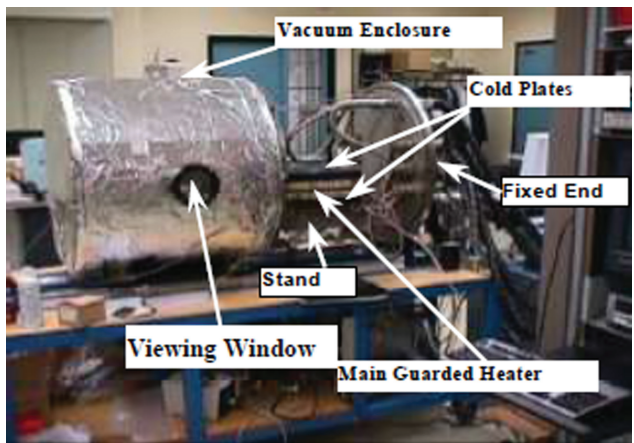
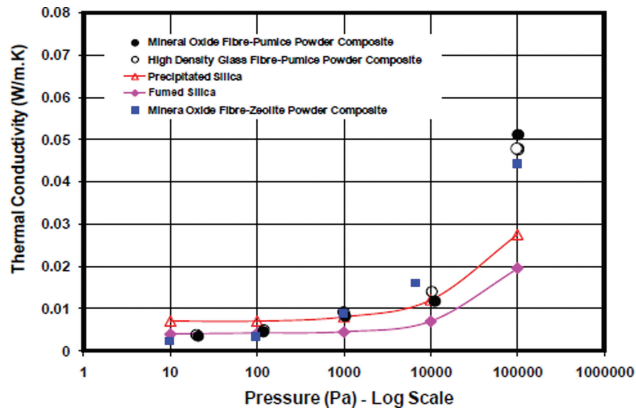


FIG. 7 Thermal properties of fiber-powder composite core material.



Performance of Vacuum Insulation Panel Constructed With Fiber-Powder Core Material

After desirable/satisfactory thermal performance of the newly developed fiber-powder composite core materials had been achieved, further investigations, as outlined below, were carried out to assess the performance of a VIP constructed with the fiber-powder composite core material. It is to be noted here that the core materials tested in this project, as reported in the previous section, were encased in a rigid evacuation box, made with hard laminated plastic sheets, during the tests. However, in reality, VIPs are made with a flexible thin gas barrier or facer foil that encases the core material and maintains the vacuum or low gas pressure during the service life.

A vacuum packaging machine primarily designed for the food packaging industry (HENKOVAC Model Basic-200 Series) (Fig. 8) was used in this study to construct VIPs in the laboratory. This equipment can reduce the internal pressure to 5 mbar (1 mbar = 100 Pa) or less. In this project it was used for producing VIPs of 300 mm by 300 mm. Fiber-powder composite core material, with eight slices of mineral oxide fiber boards (≈ 1.3 mm to 2.9 mm) and seven layers of pumice powders (≈ 1.5 mm), wrapped with an inner membrane (0.17 mm), was inserted into the impermeable gas barrier/facer envelope (0.10 mm) (Fig. 9) and placed inside the vacuum packaging chamber, and the machine itself evacuated and sealed the impermeable gas barrier/facer edge joint to produce the 300 mm by 300 mm VIP (Fig. 8).

The internal pressures of the VIP constructed in the laboratory were measured inside the VGHP using the foil lift-off technique [3,14]. The foil lift-off tests were carried out inside the VGHP chamber (Fig. 6). The VIP specimen was placed inside the VGHP chamber, and when the pressure inside the chamber diminished to less than the pore pressure of the VIP, the gas barrier/facer foil lifted off the core

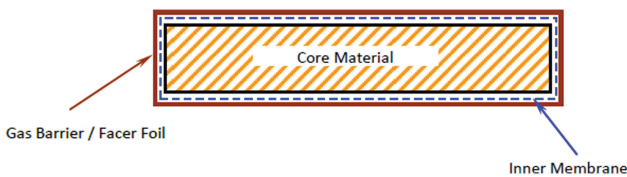
FIG. 8 Vacuum packaging equipment.



surface. This phenomenon was visually recorded through the viewing window (Fig. 6) of the VGHP, and the corresponding pressure was recorded as the internal pore pressure of the tested VIP.

The thermal property of the VIP constructed with newly developed core material was measured using a guarded hot plate apparatus inside the VGHP chamber

FIG. 9 Construction of vacuum insulation panel.



that conformed to ASTM C177 [15]. The thermal measurement was done at a mean temperature of $\approx 24^{\circ}\text{C}$.

The thermal conductivity value observed in the VIP specimen was $0.012 \text{ W/K} \cdot \text{m}$ (i.e., $\approx R 14.2$) at an internal pore pressure of 17 500 Pa. This thermal conductivity value is almost the same as that observed for the fiber–powder composite core materials evacuated inside the evacuation box at the corresponding pressure level. Thus, a logical extrapolation, based on the measurement done on the VIP specimen, would indicate that at an internal pore pressure level of approximately 100 Pa, an approximately 25-mm-thick VIP specimen constructed with the newly developed core materials could have a thermal conductivity of around $0.005 \text{ W/K} \cdot \text{m}$ (i.e., $\approx R 30$). This thermal characteristic of the newly developed VIP specimen is very comparable to that of VIPs made with fumed silica or precipitated silica.

Conclusions

The following conclusions can be drawn from the results and discussion presented in this paper.

- (1) The findings presented in this paper clearly confirm that there exists a significant opportunity to develop alternative core materials using locally available traditional thermal insulating fibers and powders with the ultimate goal of producing cost-competitive vacuum insulation panels (VIPs).
- (2) It has also been demonstrated that VIPs constructed with alternative fiber–powder composite core materials can have thermal insulating properties comparable with those of VIPs made with more traditional fumed silica or precipitated silica core materials.

Researchers at NRC Construction will be working further on these newly developed alternative core materials to optimize their short- and long-term thermal insulating capacity with a focus on the applications of VIPs in the construction industry.

ACKNOWLEDGMENTS

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STP 1574, 2014 / available online at www.astm.org / doi: 10.1520/STP157420130087

Fyodor A. Shutov,¹ Igor V. Scherbanev,¹ and David W. Yarbrough²

Development of an Advanced Foam Insulation Based on Thermosetting Resins

Reference

Shutov, Fyodor A., Scherbanev, Igor V., and Yarbrough, David W., "Development of an Advanced Foam Insulation Based on Thermosetting Resins," *Next-Generation Thermal Insulation Challenges and Opportunities*, STP 1574, Therese K. Stovall and Thomas Whitaker, Eds., pp. 11–16, doi:10.1520/STP157420130087, ASTM International, West Conshohocken, PA 2014.³

ABSTRACT

A new cellular foam for thermal insulation based on a mixture of thermosetting (nonpolyurethane) resins that has superior fire resistance and low apparent thermal conductivity has been developed. The resin components can be produced from either crude oil or coke as a starting material to produce rigid foams with densities in the range from 30 to 500 kg/m³. The new foam has very low volatile organic compound (VOC) emissions and excellent fire resistance. Properties and applications of the new foam in construction and shells for industrial heat- and oil-pipe line insulation, and for insulating hollow brick or concrete structures will be discussed in this paper.

Keywords

foam insulation, cellular plastic insulation, fire resistive insulation, thermal insulation

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¹Europanel Ltd, Moscow, Russia.

²R&D Services, Inc., Cookeville, TN 38501, United States of America.

³ASTM Symposium on *Next-Generation Thermal Insulation Challenges and Opportunities* on October 23–24, 2013 in Jacksonville, FL.

Introduction

A rigid cellular foam is being produced from the polymer resin components which can be produced either from crude oil or coke as the raw material. The foam can be formed as board stock by casting, self-foaming, and cold curing carried out in open or closed molds either at a manufacturing facility or in the field. The final product can be manufactured over a range of densities from 30 to 500 kg/m³. The fire resistance of the foam has been certified as grade G1 (self-retarding) by a Russian test method that determines weight loss during heating. The emission of VOCs during the manufacturing process and afterwards has been shown to be significantly less than allowed by environmental regulations. Applications for the new foam include use in the building envelope and use as above ambient temperature industrial insulation.

Production of the New Foam

Foam formulations consist of mixtures of two liquid components manufactured by the domestic industry. One component is a mixture of thermosetting (nonpolyurethane) resins with some special additives; the second component is a foaming and curing agent. To obtain the material they are mixed and the mixture is poured into an open or closed mold, where it foams and cures without any heat and pressure from the outside. This technology uses very little energy, no more than 5 kW/h.

The foaming and curing processes of the foam are completed within 2 to 3 min, and can be completed at temperatures from -20°C to 40°C . To reduce the direct cost and control the density, strength properties, and water resistance, some special multipurpose micro- and nanosized additives and solid fillers are added to the composition. It is important to note that the production of materials of different densities is achieved by changing only the ratio of components without changing the chemical nature of the components. Thus the developed foams produced by casting, self-foaming, and cold curing technology is like the well-known techniques for thermosetting resins such as polyurethane foams [1].

The final rigid foams can be produced by two techniques: foaming in open molds or foaming in closed molds of any sizes and configurations. In the first case (free foaming in an open mold) the composition is poured into products like structural-insulated panels (SIPs), sandwich panels, hollow brick and concrete masonry, or attic floors. This technology is applicable at the factory or in situ. When foaming proceeds in closed forms, such as shells for insulation of oil, steam, or heating pipes, building blocks with a hardened surface layer are fabricated.

Properties and Applications

Multilayer assemblies and sandwich panels based on the newly developed foam have successfully passed fire resistance tests and have demonstrated good sound absorption properties. For the open-cell product with density 55 kg/m³, the

apparent thermal conductivity is 0.036 W/m-K at 23.9°C and the compressive strength is 0.25 MPa. The foam is noncorrosive with a pH of 6. Ecology tests demonstrate that the foam generates toxic volatile organic compounds (VOCs) such as phenol, formaldehyde, toluene, benzene, styrene monomer, and others in amounts much lower than allowed by environmental regulations. Applications for the new foam include the production of structural-insulated panels (SIPs) using oriented strand board (OSB), sandwich panels with metal skins, shells for industrial heat- and oil-pipe line insulation, and for insulating hollow brick or concrete structures. Shells are produced in closed molds but SIPs, sandwich panels, and hollow structures are filled with the liquid formulation and it foams directly at the building field. It was shown that the foaming in situ might be at the range of ambient temperatures between -15°C and $+35^{\circ}\text{C}$. Fire-resistant door and window frames filled with the foam have been demonstrated. Several family houses that meet code requirements have been constructed in the Moscow region using SIP-OSB panels containing the new foam as the core layer inside the panels. The newly developed foams are innovative foamed composites that combine a number of unique features and are designed to meet the needs of the construction market for fire resistant, energy efficient materials produced using an environmentally sound technology.

Unlike some types of insulation materials, the foam meets the requirements of the new Russian Federal Law 123-FZ ("Technical Regulations of Fire Safety") and, according to the classification of materials regarding fire hazard, the material belongs to the flammability grade G1 self-retarding material that does not sustain flame and is a nonsmoldering material. The scale for this classification is NG (non-burning), G1, G2, G3, and G4 (easily burned like paper). With up to 2 h of exposure to a propane torch flame ($\sim 1500^{\circ}\text{C}$), the foam does not melt but instead it converts to porous coke. The material has much higher fire resistance and chemical and biological stability than polystyrene (EPS or XPS) and polyurethane foams. The foam is resistant to acids and alkalis, and does not attract rodents or other vermin.

Physical Property Data

The family developed foam materials have a range of densities from 30 to 500 kg/m³ and; therefore, a very wide range of thermal and physical properties. Table 1 contains apparent thermal conductivities measured as a function of temperature using a heat-flow meter apparatus operated in accordance with ASTM C518 [2].

TABLE 1 Apparent thermal conductivity data for the new foam product.

Average Specimen Temperature, °C	Apparent Thermal Conductivity, W/m-K
10.02	0.0339
23.91	0.0357
37.80	0.0375

TABLE 2 Apparent thermal conductivities for new foam product at selected temperatures.

Temperature, °C	λ_a , W/m·K
0	0.0326
10	0.0339
10	0.0352
30	0.0365
40	0.0378
50	0.0391

The density of the test specimens tested was 55.5 kg/m³. The test specimens were approximately 26 mm in thickness.

The data for the new foam are compared with data for rigid polystyrene thermal insulation taken from the standard specification [3]. The new foam has apparent thermal conductivities that fall between type XIII and type XIV polystyrene. The new foam apparent thermal conductivity as a function of temperature obtained from the test data using the method of least squares is given by Eq 1. Table 2 contains apparent thermal conductivities λ_a at selected temperatures.

(1)
$$\lambda_a = 0.03262 + 0.0001296 \cdot T$$

Type XIII polystyrene is an XPS insulation with density 26 kg/m³ while type XIV is an EPS insulation with typical density 38 kg/m³ (Fig. 1).

The material has a very high chemical and biological stability, it is resistant to acids and alkalis, and does not attract or provide nutrients for rodents or other vermin. Open-cell content depends on the density and equals 95 % for 30 kg/m³ insulation and 10 % for 200 kg/m³ insulation. The high open-cell content for the low density foams results in excellent sound adsorption properties.

FIG. 1 New foam compared with polystyrene.

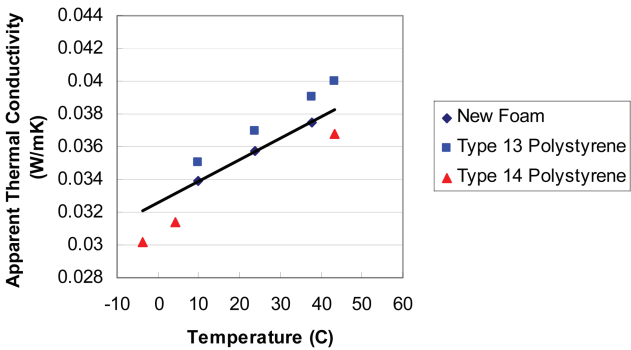


TABLE 3 VOCs generated in the foaming area during the foaming process.

Volatile Organic Compounds	VOCs Measured, mg/m ³	VOCs Maximum Permissible Content, mg/m ³	
		Foaming Area	Ambient Air
Polycyclic aromatic hydrocarbons (phenanthrene, anthracene, etc.)	0.0030	0.8	0.01
Mono- and dichlorophenol (total)	0.005	0.1	0.01
Phenol (hydroxybenzene)	0.0071	0.3	0.003
Formaldehyde	0.0002	0.5	0.003
Xylenes	<0.0001	50	0.2
Toluene (methylbenzene)	<0.0001	150	0.6
Benzene	<0.0001	5	0.1
Cymene	<0.0001	50	0.014
Cresol	0.006	0.5 to 1.5	0.02
Carboxylic acid esters (as ethyl acetate)	0.008	50 to 200	0.1
SOX	0.020	10	0.05 to 0.5
NOX	0.026	5	0.06 to 0.4
Styrene	0.0046	10 to 30	0.002 to 0.04
Mineral acids (as HCl)	0.5	–	0.15
Ethyl benzene	0.0006	50 to 150	0.02
Volatile solids	0.19	–	0.15 to 0.5

Environmental Considerations

The newly developed insulation has received Russian federal certifications of conformity, flammability, and sanitary safety. Due to the increased demands for environmental safety of thermal insulation in the construction industry, the new materials have been tested in accordance with the Russian Federal Agency for Technical Regulation and Metrology. According to the Agency, the materials are environmentally friendly at production as shown in Table 3.

Emissions after production are also very low as shown in Table 4. The concentration of such toxic volatile organic compounds (VOCs) as phenol, formaldehyde,

TABLE 4 VOC content at 25°C generated by foam 24 h after manufacturing.

Volatile Organic Compound	VOCs, Test Data, mg/m ³	VOCs, Maximum Permitted, mg/m ³
Phenol	0.003	0.01
Benzene	0.1	0.3
Ethyl benzene	<0.02	0.02
Toluene	0.1	0.6
Ethyl acetate	<0.1	0.1

styrene, benzene, toluene; for example, are much lower than the “maximum permissible content.” The VOC concentrations were measured using GC-MS equipment. VOC concentrations were well below permitted levels for every organic species observed.

Summary

The newly developed fire-resistant thermal insulation manufactured from readily available materials using advanced energy saving and environmentally friendly techniques address many of the problems of the construction industry in Russia. The new insulation materials have potential for application elsewhere. Industrial production of SIPs and shells started in 2011 at the facilities of the “Fachmann Group, Inc.,” a group of Russian companies located in the Moscow region demonstrating the usefulness of the foam in the construction industry [4].

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Patrick M. Noonan¹ and Timothy R. Jonas²

Full-Thickness Thermal Testing of Fiberglass Insulation Using an ASTM C518-10 Heat Flow Meter Apparatus

Reference

Noonan, Patrick M. and Jonas, Timothy R., "Full-Thickness Thermal Testing of Fiberglass Insulation Using an ASTM C518-10 Heat Flow Meter Apparatus," *Next-Generation Thermal Insulation Challenges and Opportunities*, STP 1574, Therese K. Stovall and Thomas Whitaker, Eds., pp. 17–38, doi:10.1520/STP157420130099, ASTM International, West Conshohocken, PA 2014.³

ABSTRACT

Can thermal conductivity tests be performed at thicknesses greater than that of the primary standard? As a test specimen thickness increases, the edge loss effects can also increase, causing a heat flux through the specimen that may become less one dimensional. New equipment designs in thermal comparators allow for greater guard-to-metered-area ratios, which should reduce this effect and allow for measurements on thicker specimens than before for commercially available equipment. Laboratory testing of roll and batt insulations at a representative thickness of 76mm has always been considered to be representative of the performance at full thickness. As more stringent building codes call for increasingly higher levels of insulation in homes, verification of material performance, such as loose-fill insulation, requires that it be tested at a representative thickness that is greater than that of the calibration standard. This paper describes a series of tests performed using a series of full-thickness fiberglass insulation standards that were delivered to a national laboratory for

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¹Manager of New Product Development and Testing Services, Knauf Insulation, North America (KINA) GmbH, USA Product Testing Laboratory, Shelbyville, IN 46176, United States of America.

²Engineer, Knauf Insulation, North America (KINA) GmbH, USA Product Testing Laboratory, Shelbyville, IN 46176, United States of America.

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thermal characterization and tested in a guarded hot plate apparatus conforming to ASTM C177-10 from December 2008 to July 2009. Tests were conducted on a total of six light density (12 kg/m^3) fiberglass standards varying in thickness from 76 mm to 203 mm of thickness for derived thermal conductivity values with associated uncertainties, characterized as calibrated transfer specimen (CTS) sections, and brought back to determine whether they could be effectively used in this “stacking exercise” study on one commercially available large-thickness opening ASTM C518-10 thermal test apparatus. From February 2011 through August 2011, a total of 40 tests were run in single and stacked configurations up to and including 305 mm (12.0 in.) in thickness, with and without septa.

Keywords

apparent thermal conductivity, ASTM, batt, bias, blanket, cellulose, CTS, fibrous glass, HFM, insulation, loose fill, mineral wool, perlite, precision, P.T. Lab, round robin, SOR, SRM, thermal resistance, thermal resistivity

Introduction

In recent years, advances in ASTM C518-10 [1] heat flow meter design technology has allowed research scientists, engineers, and technicians the ability to test thermal building materials at much greater thicknesses and, in some cases, greater than that of the primary reference standard. As demands increase from the energy-conservation community to require higher envelope efficiencies, the need to specify and deliver thermal insulation performance at full thickness, “as installed,” will continue to increase.

Scope

In September 2008, a special machine trial was performed at KINA plant 2 to produce several slabs of fiberglass insulation sections at various thicknesses. Six different thickness fiberglass slab lots were produced during the same production interval at a target density of 12 kg/m^3 (0.75 lb/ft^3) and ranging in thickness from 76 mm to 203 mm in 25 mm increments (3 in. to 8 in.). Sections were then weighed and categorized into groups centering on the material’s target nominal density. The next step was to select one specific specimen from each thickness category produced. One of each thickness and all as close to the nominal density produced were selected and transported to the National Institute of Standards and Technology on Dec. 1–2, 2008 for thermal measurements. The National Laboratory characterized and tested each of these individually using a guarded hot plate apparatus conforming to ASTM C177-10 [2], a primary test method. Materials were picked up and taken back to the KINA product testing laboratory to perform the stacked thermal specimen sensitivity study.

Purpose of In-House Study

The driving need for this study dealt with ambiguities in test methods ASTM C687-12 [3], Standard Practice for Determination of Thermal Resistance of Loose-Fill Building Insulation, and C518-10, Standard Test Method for Steady-State Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus, in terms of relevance in the testing of loose-fill thermal insulation. The item in question is the representative thickness required for testing for the R value of loose-fill thermal insulation materials.

Currently, there are no primary reference materials for loose fill in an ASTM C518-10 apparatus. Standard practice is to calibrate a C518-10 instrument using batt-type insulation materials at thicknesses near the representative thickness of 3 in. or 76 mm, which is generally accepted as being representative for this class of materials.

ASTM C687-12 is a practice for the determination of the R value, and includes all materials that are pneumatically applied or poured in place including fiberglass, rock wool, slag wool, cellulose, vermiculite, perlite, and pelletized materials. In Section 4.5 of ASTM C687-12 for significance and use, the practice specifies that if the specification or codes do not specify the nominal resistance level to be used for label comparison purposes, a recommended practice is to use the label density and thickness for an R-19 (IP units) for the determination.

In the case of fiberglass thermal insulation, ASTM C764-11 [4] does not specify an R value for thermal comparisons, so the recommendation applies. In Section 4.7 of ASTM C687-12, it states that thin sections of these materials are not uniform; thus, the test thickness must be greater than or equal to the product's representative thickness if the results are going to be consistent and typical of use.

ASTM C764-11 also states that "a representative thicknesses range of four to eight inches is considered typical for most products. The representative thickness shall be determined at the midpoint of the blown density range. Once this representative thickness determination is accomplished, all thermal testing is conducted at a thickness that is greater than or equal to the representative thickness."

Because most fiberglass thermal insulation materials have progressive design density inherent in the label, the midpoint of the density range is roughly the midpoint of the R value chart. For ASTM C764-11, the range for density covers the range of R value, which is mandated by ASTM C764-11 to include R values from 11 to 60 (IP units). In this case, the representative thickness would be roughly the midpoint of the R value table, roughly an R-26 (IP units). For most products, this would be greater than 10 in. in thickness.

This would guide the user of this practice to test at a representative thickness of at least an R-19, and preferably the midpoint R value of roughly an R-26 (IP units). Using the minimum representative thickness for R-19 for fiberglass thermal insulation materials, this would be roughly 8 in. in thickness. In Section 4.7.1 of ASTM

C764-11, the minimum thickness shall be 4 in., or the representative thickness, whichever is larger.

There are also exceptions in the case of instrument limitations, as stated in Section 4.8, because of cost of equipment; it states that it is acceptable to estimate the thermal resistance from *R*-value tests on the product at the minimum thickness from Section 4.7.1. This is, however, an estimate, as opposed to a determination of *R* value if the thickness is less than the representative of an R-19 (IP units).

The apparent thermal conductivity measurements themselves are performed using ASTM C177-10, C518-10, C1114-06 [5], and C136-11 [6], with C518-10 being preferred. Section 5 in ASTM C687-12 on apparatus, requires a device capable of measuring specimens up to at least 6 in. in thickness.

ASTM C518-10 is a test method as opposed to a practice, and has a higher degree of stringency than a practice. The scope of ASTM C518 in Section 1.6 states that the test method is applicable for thicknesses up to approximately 250 mm. Annex A1.8.2 of ASTM C518-10 describes the case of testing materials greater than that of the calibration standard, mandating that a series of calibration measurements be performed to insure that the equipment does not introduce additional systematic or random errors. One means mentioned is to use multiple thicknesses of calibration standards. If these are stacked with a radiation blocking septum between each of the standards, the first approximation is that the total thermal resistance is the sum of the individually stacked fiberglass slabs.

In Section 7.6.2, limitation on specimen thickness describes the maximum spacing between the hot and cold plates and the possible errors that may be introduced dependent upon the equipment design, "No suitable theoretical analysis is available to predict the maximum allowable thickness of specimens." It does, however, state that it is possible to use the results of an analysis for a primary method guarded hot plate apparatus as a guide.

In Adams and Hust [7], for the loose-fill analysis (cellulose, rock-slag, and unbounded glass fiber loose fill), there is no mention or reference to a calibration standard or SRM made. The standards themselves offer no explanation for the large spread between labs (10 % to 21 % versus 3.0 %). The reason for this was not clear at that time but states that it may have been caused by inadequate standardization of the technique for preparing samples. There is a reference to SRM 1451 in the glass fiber blanket data set. This SRM is of questionable value if the representative thickness is less than the recognized minimum of 76 mm (3.0 in.) as is the case in industry practice.

In McCaa and Smith [8], a round robin conducted in 1990, included 10 participating laboratories testing a fiberglass blanket and several types of loose-fill insulations. The blanket insulation had an interlaboratory imprecision of 2.8 % at the 2 standard-deviation level. The loose-fill interlaboratory imprecision was found to be 5.0 % for perlite, 5.8 % for cellulose, 9.4 % for unbonded fiberglass,

and 10.5 % for mineral wool at the 2 standard-deviation level. This represented a significant improvement over the 1987 results and is attributed to a more concise specimen preparation procedure in practice ASTM C687-12.

Materials

A bonded glass fiber blanket was produced with a nominal density of 0.75 lb/ft³ and at several thicknesses. A nominal thermal conductivity of 0.285 Btu in./h ft² °F was targeted for these materials with a thermal resistivity of R-3.5 (IP units) per inch. The range of thicknesses covered was from 3 to 12 in., which would represent a range of *R* values from 10.5 to 42.0 (IP units).

Objectives

The primary objective of this internal study is to investigate the stacked full-thickness effective thermal conductivity results of fiberglass insulation materials of known thermal conductivity on a commercially available apparatus. The measured experimental data will be compared with known data obtained from a nationally recognized laboratory. The data sets will then be compared statistically and a conclusion made on the ability to generate reliable, acceptable, and accurate measured data possible for fiberglass insulation specimens at test thicknesses greater than that of the primary calibration standard.

Report of Tests

The results for the primary calibration standards from the National Laboratory are shown in Table 1.

TABLE 1 Results of the individually characterized slabs used in the stacking study.

Primary Standard ID No.	Thickness mm (in.)	Mean Temperature °C (°F)	Thermal Conductivity (SI units) (IP) ^a	Thermal Resistance (SI units) (IP) ^b	Uncertainty (<i>k</i> = 2) (TR)
X-2009-2-005	76.22 (3.0)	23.9 (75.0)	0.04181 (0.2899)	1.823 (10.35)	0.027 (1.5 %)
X-2009-2-006	76.22 (3.0)	23.9 (75.0)	0.04144 (0.2874)	1.839 (10.44)	0.028 (1.5 %)
X-2009-2-007	101.58 (4.0)	23.9 (75.0)	0.03968 (0.2752)	2.560 (14.53)	0.051 (2.0 %)
X-2009-2-008	127.00 (5.0)	23.9 (75.0)	0.04093 (0.2838)	3.103 (17.62)	0.042 (2.0 %)
X-2009-2-009	152.40 (6.0)	23.9 (75.0)	0.04206 (0.2917)	3.623 (20.57)	0.091 (2.5 %)
X-2009-2-010	203.21 (8.0)	23.9 (75.0)	0.04180 (0.2899)	4.862 (27.60)	0.146 (3.0 %)

Note: National Laboratory—Report of test (July 2009); guarded hot plate apparatus (ASTM C177-10); fiberglass blanket slabs at a temperature difference of 22.2 °K (40.0 °F). Slabs were shipped measuring 1188 mm × 1188 mm × 88 mm (density of approximately 12 kg/m³ (0.75 lb/ft³)) (*k* = 2) is a statistical coverage factor consistent with international usage.

^aThermal conductivity SI units = W/m K (IP units) = Btu in./h ft² °F.

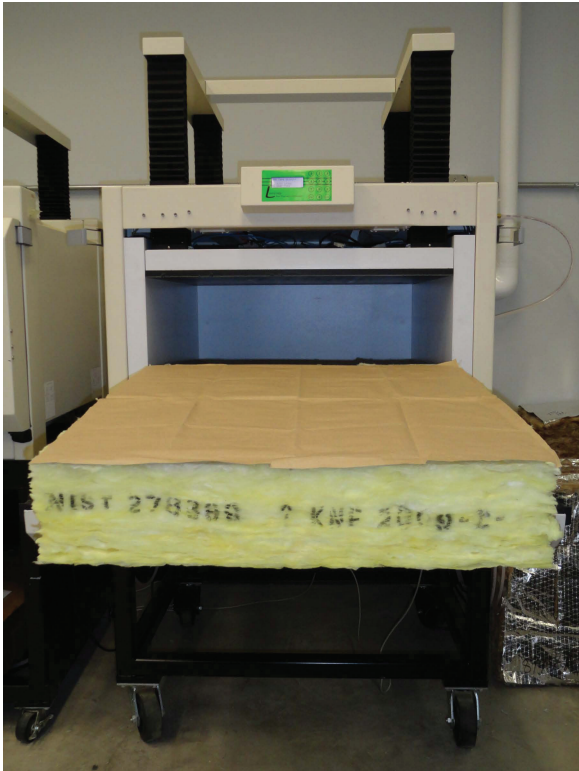
^bThermal resistance SI units = m²/K W (IP units) = h ft² °F/Btu. TR, thermal resistance (SI units).

TABLE 2 Thermal resistance for each of the measured specimens by the National Laboratory.

Thermal Resistance (SI units) (IP)	Uncertainty ($k = 2$) (TR)	Range of Measured Thermal Resistance (SI units) ($\text{m}^2 \text{ K/W}$)	Range of Measured Thermal Resistance (IP units) ($\text{h ft}^2 \cdot ^\circ\text{F/Btu}$)
1.823 (10.35)	0.027 (1.5 %)	1.850–1.796	10.50–10.20
1.839 (10.44)	0.028 (1.5 %)	1.867–1.811	10.60–10.28
2.560 (14.53)	0.051 (2.0 %)	2.611–2.509	14.82–14.24
3.103 (17.62)	0.042 (2.0 %)	3.145–3.061	17.85–17.38
3.623 (20.57)	0.091 (2.5 %)	3.714–3.532	21.08–20.05
4.862 (27.60)	0.146 (3.0 %)	5.008–4.716	28.43–26.77

Note: $k = 2$ is a statistical coverage factor used in the calculation of uncertainty and consistent with international practice.

FIG. 1 Single layer No. 1 is a 6-in.-thick primary CTS fiberglass slab with known thermal conductivity.



The National Laboratory derived results from measured experimental results and associated uncertainties on primary calibration standards (CRTs) used in the experiment.

Experimental Data

Stacking experiments with and without septa were performed on one commercially available ASTM C518-10 heat flow meter apparatus achieving a maximum thickness opening of 305 mm (12.0 in.) from February 2011 through September 2011. The plate area was 762 mm² (30 in.²) with a metered area of 254 mm² (10 in.²) centered in both the upper and lower plates. The data reported in Table 2 lists the raw data for the experiments using the primary fiberglass materials listed individually.

FIG. 2 Slab No. 2 added to No. 1. Slab No. 1 is 6-in.-thick primary CTS fiberglass standard with septum between next slab No. 2, a 3-in.-thick primary CTS fiberglass layer in the middle.



Individual and stacked experiments range in thickness from 76 mm (3.0 in.) to 305 mm (12.0 in.) on both apparatus. The septum used was flexible brown butcher paper measuring approximately 3 mm (0.118 in.) in thickness with a measured emissivity of approximately 0.85 (see Figs. 1 through 4 showing the ASTM C518-10 HFM apparatus with slab arrangement orientations).

For the range of materials tested under this study, the measured effective thermal conductivity was within $\pm 1.4\%$ of reference value.

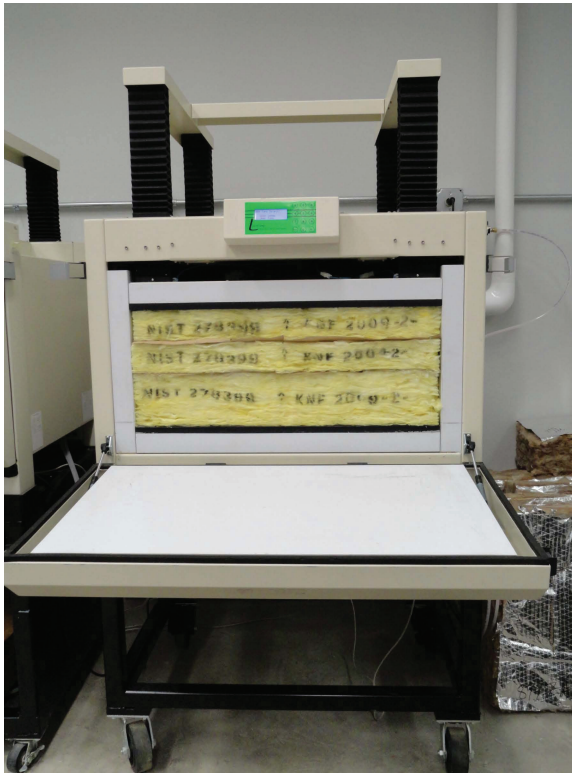
For part of this research, the impact of the thermal conductivity measurements with and without septa was investigated. Using a worse-case approach, a group of three stacked specimens with a total thickness of 305 mm (12.0 in.) were compared as shown in Table 3 (also see Table 4).

There was no significant measurable difference in this study relating to an improved effective apparent thermal conductivity caused by radiation effects for specimens tested with or without septa.

FIG. 3 Slab No. 3 added to the stack. Last 3 in. primary CTS fiberglass standard of known thermal conductivity added with septums between layers with a total stack thickness of 305 mm (12 in.).



FIG. 4 Entire stack inserted into the full-thickness ASTM C518-10 heat flow meter apparatus set at 305 mm (12 in.) for 2-day steady-state thermal test with bottom hot plate set at temperatures of 95°F, and top cold plate set at 55°F.



The next step was to run several steady-state tests on the 76 mm-thick primary CTS in a C177-10 guarded hot plate apparatus (at KINA P.T. Lab) at different mean temperatures. The testing was performed on the 76 mm (3.0-in.)-thick CTS primary fiberglass specimen (identity National Laboratory primary X-2009-2-006) used in the stack study as the calibration standard for the ASTM C518-10 heat flow meter apparatus. Because the stacked specimen thermal resistances are at an actual set mean temperature of 23.9°C (75°F), the individual specimens were corrected for the actual mean temperature of the total specimen. The data was then used to compare corrected versus non-corrected thermal resistances to determine the impact on the overall stacked thermal resistance. Table 5 contains the test results, and Table 6 is the ASTM C1045-07 [9] regression analysis.

The next step was to examine the worst-case stacking tests in the ASTM C518-10 HFM apparatus from an edge loss standpoint. This was 305-mm (12.0-in.)-thick stack and is the maximum opening the apparatus could achieve based on the equipment

TABLE 3 Lists the raw data for the experiments using the primary fiberglass materials listed individually.

Primary standard ID No. 0.75 lb/ft ³ × Thickness (Slab)	Thickness (in.)	Measured k Value ^a (IP units)	Known Estimate k Value (IP units)	Measured Difference (%)	Difference (+ or –) to KE by NL
X-2009-2-005	3.0	0.2908	0.2899	0.3 %	+0.0009
X-2009-2-006	3.0	0.2876	0.2874	0.1 %	+0.0002
X-2009-2-007	4.0	0.2714	0.2752	–1.4 %	–0.0038
X-2009-2-008	5.0	0.2823	0.2838	–0.5 %	–0.0015
X-2009-2-009	6.0	0.2876	0.2917	–1.4 %	–0.0041
X-2009-2-0010	8.0	0.2894	0.2899	–0.2 %	–0.0005

Note: These are KINA P.T. Lab measured test results compared to the known estimate reported by the National Laboratory. Thermal conductivity (SI units) = W/m K (IP units) = Btu in./h ft² °F.

^aMeasured results were obtained using an apparatus conforming to ASTM C518-10 with a thermal equilibrium criteria of 0.2 °C, between block count HFM equilibrium of 200 mV, HFM percent (%) change of 2.0 %, a minimum number of blocks of 100 (one block = 512 counts at 0.9 s), and calculation blocks of 30. HFM apparatus used was a 762 mm² (30 in.²) unit with a 254 mm² (10 in.²) metered area calibrated using a fiberglass CTS categorized by National Laboratory CTS ID No. X-2009-2-006 at 12 kg/m³ density with a measured estimated thermal conductivity = 0.04144 W/m °C (0.2874 Btu in./h ft² °F) at a measured thickness of 76.2 mm (3.0 in.). This standard’s calibration factor was then used throughout the remaining exercise for all individual measurements and all stacking tests used with and without septa up through and including 305 mm (12.0 in.) full-thickness tests.

design (Fig. 4). Table 7 lists the individual tests on the fiberglass slabs used in this stacking study including the 76 mm (3.0 in.) primary CTS ID No. X-2009-2-006 used as the calibration slab, and Table 8 shows the test results of 11 of a total possibility of 12 stack combination configurations using three stacked fiberglass slabs. Two slabs were 76 mm (3.0 in.) in thickness and one was 152 mm (6.0 in.) in thickness, all traceable to the National Laboratory. Table 8 lists the total added thermal resistance for comparison.

TABLE 4 Septa versus non-septa.

List of Primary Reference Standards Identification Test Numbers	Measured k Value (IP Units) Without Septum	Measured k Value (IP Units) With Septum	Difference (%)	Difference (+ or –)
31 X-2009-2-005/2009-2-006/2009-2-009	0.2887	0.2875	–0.42	+0.0012
32 X-2009-2-006/2009-2-005/2009-2-009	0.2883	0.2875	–0.28	+0.0008
33 X-2009-2-005/2009-2-009/2009-2-006	0.2899	0.2888	–0.38	+0.0011
34 X-2009-2-006/2009-2-009/2009-2-005	0.2890	0.2890	0.00	0.0000
35 X-2009-2-009/2009-2-005/2009-2-006	0.2889	0.2890	+0.03	–0.0001

Note: Most extreme thickness stack tested is 305 mm (12.0 in.) in all stacked triple layers (three layers) (Fig. 4). Thermal conductivity (IP units) = Btu in./h ft² °F.

TABLE 5 ASTM C1045-07 analysis on a lot specimen traceable to National Laboratory primary CTS No. X-2009-2-006 0.75 lb/ft³ × 3.0 in.

Standard 0.75 lb × 3 in. National Laboratory primary CRT	Mean Temperature SI °C	Hot Temperature SI °C	Cold Temperature SI °C	Delta T SI °C	Apparatus k SI W/m °C	Mean Temperature IP °F	Hot Temperature IP °F	Cold Temperature IP °F	Apparatus k IP Btu in./h ft ² °F	$(T_h + T_c) / 2$ C1045 Form	$(T_h^2 + T_h T_c + T_c^2) / 3$ C1045 Form	$(T_h + T_c) / (T_h^2 + T_c^2) / 4$ C1045 Form
ID No. X-2009-2-006	21.11	32.2	10.0	22.2	0.0408	70.0	90.0	50.0	0.2829	70.0	5033.026672	370 964.2013
Estimated $k = 0.04144$ W/m °C	23.89	35.0	12.8	22.2	0.0414	75.0	95.0	55.0	0.2871	75.0	5758.606672	451 903.551
Estimated $k = 0.2874$ Btu/h ft ² °F	26.67	37.8	15.6	22.2	0.0419	80.0	100.0	60.0	0.2906	80.0	6534.266704	544 111.2085
75°F or 23.9 °C	32.22	43.3	21.1	22.2	0.0432	90.0	110.0	70.0	0.2996	90.0	8232.586684	764 894.005
	37.78	48.9	26.7	22.2	0.0445	100.0	120.0	80.0	0.3086	100.0	10134.10668	11040 113.605

Note: The primary has an estimated $k = 0.04144 \text{ W/m}^2 \text{ °C} = 0.2874 \text{ Btu in./h ft}^2 \text{ °F}$. Thermal conductivity (SI units) = W/m K (IP units) = Btu in./h ft² °F.

Table 10 shows a very simplistic crude mean temperature gradient pattern within the C518-10 HFM apparatus of the temperature drops through the stacks from the bottom hot plate set at approximately 35°C (95°F) to the top plate set at approximately 12.8°C (55°F).

The Figure 5 plot is measured with apparent thermal conductivity (IP units) versus the thickness (inches) range for the entire stacking study. Figures 6 through 10 show the percent of measured apparent thermal conductivity versus thickness (inches) for each individual or stacked primary CTS fiberglass slab/slabs measured. Notice that for

TABLE 6 ASTM C1045 analysis on a lot specimen traceable to National Laboratory primary CTS No. X-2009-2-006 0.75 lb/ft³ × 3.0 in.

Start with Following Form:

$$k = a + bt + ct^2 + dt^3$$

But Regress Data to this Form:

$$k_m = a + b^*(T_h + T_c)/2 + c^*(T_h^2 + T_hT_c + T_c^2)/3 + d^*(T_h + T_c)(T_h^2 + T_c^2)/4$$

Summary Output: Lot Specimen from 0.75 lb × 3 in. 2006-2-006 Primary CRT Tested at Several Mean Temperatures

Regression Statistics					
Multiple R	0.999789126				
R ²	0.999578296				
Adjusted R ²	0.998313184				
Standard error	0.000423701				
Observations	5				

ANOVA	df	SS	MS	F	Significance F
Regression	3	0.000426	0.000142	790.1104	0.026145
Residual	1	1.8 × 10 ⁻⁷	1.8 × 10 ⁻⁷		
Total	4	0.000426			

	Coefficients	Standard Error	t Stat	p Value	Lower 95 %	Upper 95 %	Lower 95.0 %	Upper 95.0 %
Intercept	0.282181245	0.149484	1.8877	0.310135	-1.6172	2.181558	-1.6172	2.181558
x Variable 1	-0.001067297	0.005582	-0.1912	0.879729	-0.07199	0.069859	-0.07199	0.069859
x Variable 2	1.97821 × 10 ⁻⁵	6.75 × 10 ⁻⁵	0.292945	0.818581	-0.00084	0.000878	-0.00084	0.000878
x Variable 3	-6.47083 × 10 ⁻⁸	2.65 × 10 ⁻⁷	-0.24404	0.847617	-3.4 × 10 ⁻⁶	3.3 × 10 ⁻⁶	-3.4 × 10 ⁻⁶	3.3 × 10 ⁻⁶
a = 0.2822								
b = -0.0011								
c = 1.9782 × 10 ⁻⁵								
d = -6.4708 × 10 ⁻⁸								

The primary has an estimated $k = 0.04144 \text{ W/m}^2 \text{ }^\circ\text{C} = 0.2874 \text{ (IP units)}$.

TABLE 7 Worst-case stacks at maximum thickness of 305 mm (12.0 in.) layered in triplicate.

Primary Standard ID No. 0.75 lb/ft ³ × Thickness (Slab)	Test Thickness (in.)	75°F Mean			
		(KINA P.T. Lab)		National Laboratory	
		<i>k</i> Value (IP Units)	<i>R</i> Value (IP Units)	<i>k</i> Value (IP Units)	<i>R</i> Value (IP Units)
X-2009-2-005	3.0	0.2908	10.32	0.2899	10.35
X-2009-2-006	3.0	0.2876	10.43	0.2874	10.44
X-2009-2-009	6.0	0.2876	20.86	0.2917	20.57
Individual slabs	Added <i>R</i> totals	stack total target =	41.61	<i>R</i> totals known estimates =	41.36

Note: Individual slab sections used in worst-case 305 mm (12.0 in.) stacking study. Individual slabs used are listed in Table 3. Measured results in Table 5 are measured estimates. The apparatus used at the KINA product testing laboratory was a C518-10 heat flow meter apparatus. The apparatus used by the National Laboratory was a C177-10 guarded hot plate apparatus.

every primary CTS measured, all percentage data are less than 2 % except for one measurement greater than 2 % at a 9.0 in. stack thickness. Figure 11 is an overhead statistical box plot of the entire percentage spread of the effect of all the primary CTS standards thickness on the measured result of apparent thermal conductivity.

TABLE 8 Worst-case stacks tested at maximum thickness of 305 mm (12.0 in.) layered in triplicate in different stacked combinations.

Test Thickness (in.)	<i>k</i> Value (IP Units) at 75°F KI (U.S.)	<i>R</i> Value (IP Units) at 75°F KI (U.S.)
12.0	0.2887	41.57
12.0	0.2883	41.62
12.0	0.2899	41.39
12.0	0.2890	41.52
12.0	0.2889	41.54
12.0	0.2891	41.51
12.0	0.2875	41.74
12.0	0.2875	41.74
12.0	0.2888	41.55
12.0	0.2890	41.52
12.0	0.2886	41.58
Combinations of three layers	Average of 11 tests stacked <i>R</i> total =	41.57

Note: Individual slab sections used in worst-case 305 mm (12.0 in.) stacking study reference raw data (Table 12) for stack identification numbers/combinations. Thermal conductivity (IP units) = Btu in./h ft² °F. Thermal resistance (IP units) = h ft² °F/Btu.

TABLE 9 Total added thermal resistance corrected for mean temperature and non-corrected for mean temperature.

	Total <i>R</i> Value (IP Units)	National Laboratory <i>R</i> Value (IP Units)	Difference <i>R</i> Value (IP Units)
KINA P.T. Lab NC No. 1	41.61	41.36	0.25
KINA P.T. Lab No. 2	41.57	41.36	0.21
KINA P.T. Lab C	41.94	41.36	0.58

Note: Thermal resistance (IP units) = $h \text{ ft}^2 \text{ }^\circ\text{F/Btu}$. NC, non-corrected; C, corrected. NC No. 1 is the total added thermal resistance of each individual slab from Table 5 = 41.61. NC No. 2 is the average of 11 stacked tests from Table 6 = 41.57. C is the corrected *R* total sum of three different arrangements of same stack of three in Table 7 = 41.94.

TABLE 10 305 mm (12.0 in.) stacked fiberglass slabs (0.75 pcf) (mean temperature issue, worst case).

ASTM C518-10 HFM apparatus temperature gradient through stacked layers	
	Cold plate temperature = 55°F
	Layer No. 3 mean T3 = 65°F
	Layer No. 2 mean T2 = 75°F
	Layer No. 1 mean T1 = 85°F
	Hot plate temperature = 95°F

Note: Temperature gradient is an approximate mean temperature model of what each individual stacked slab section yields as a temperature drop through the stacks. Layers are a combination of two 3.0 in. thicknesses and one 6.0 in. thickness (worst case) when stacked to 305 mm (12.0 in.) in three different configurations.

FIG. 5 Apparent thermal conductivity versus thickness for the entire stacking study.

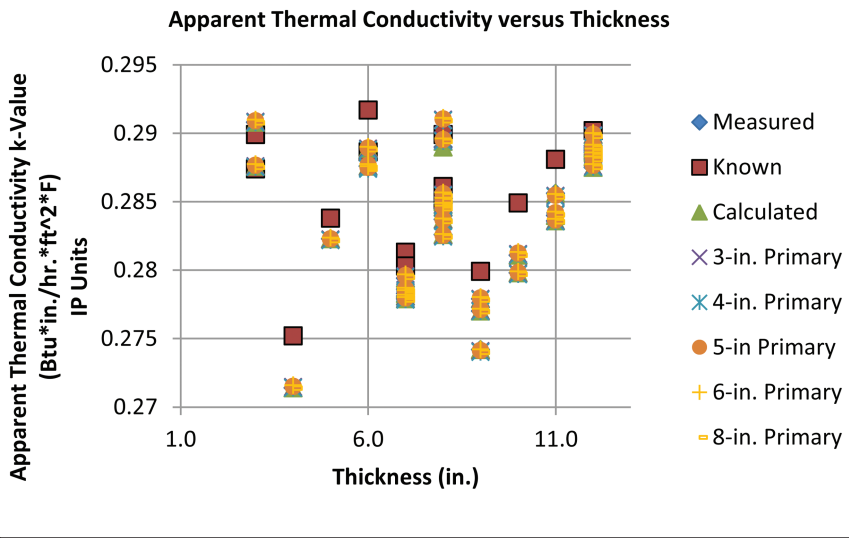


FIG. 6 Percentage of measured versus known as function of thickness for 3-in. primary CTS.

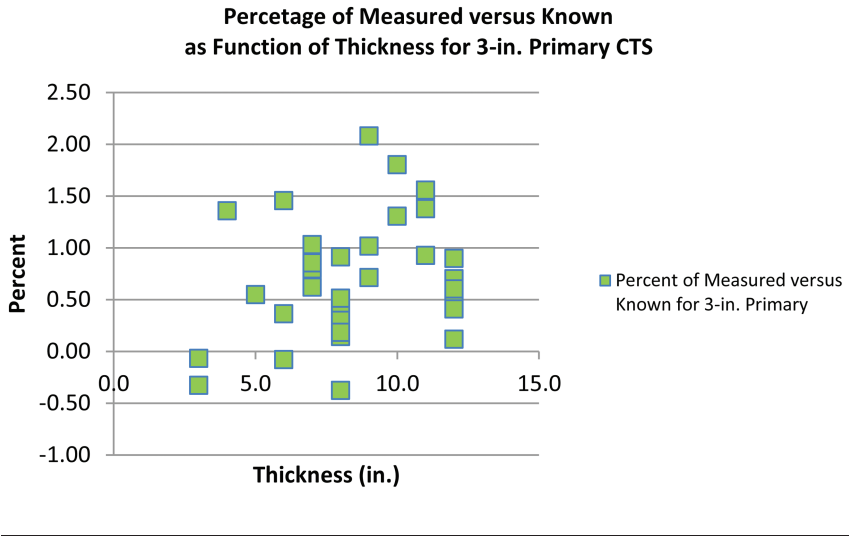


FIG. 7 Percent of measured versus known for 4-in. primary CTS.

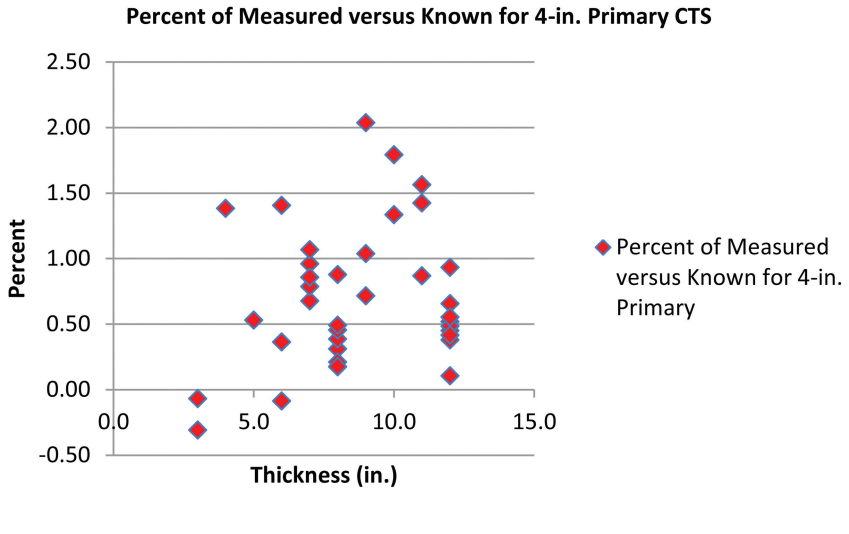


FIG. 8 Percent of measured versus known for 5-in. primary CTS.

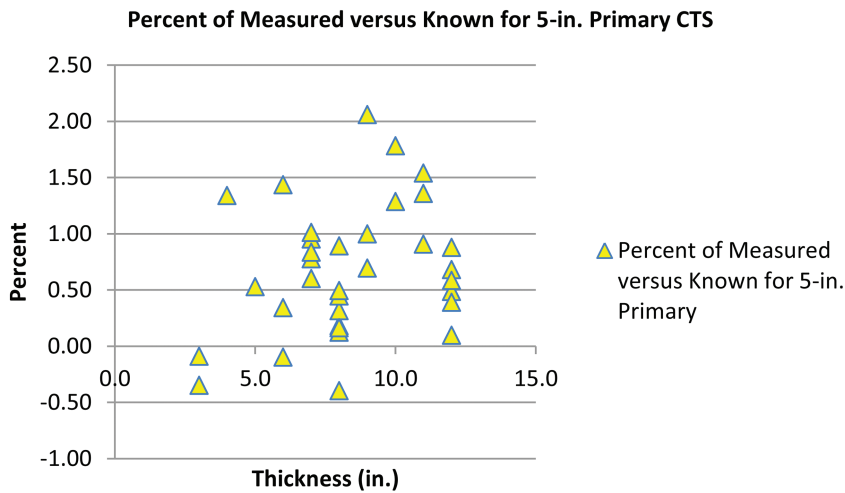


FIG. 9 Percent of measured versus known for 6-in. primary CTS.

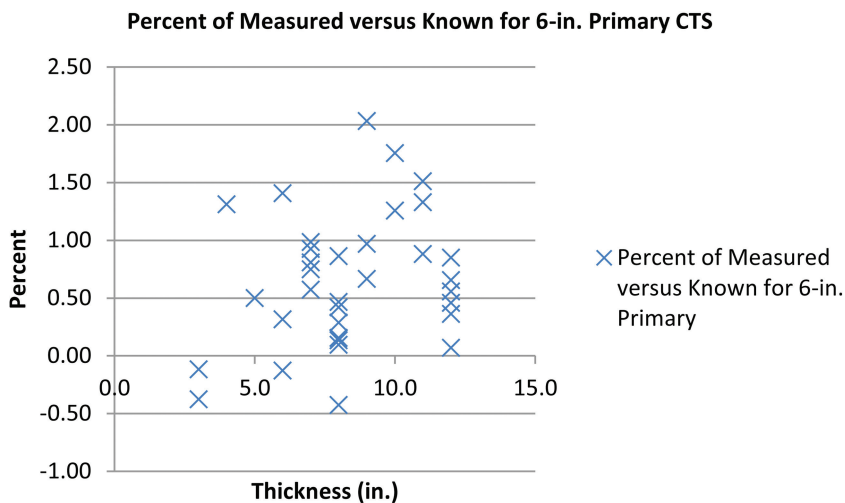


FIG. 10 Percent of measured versus known for 8-in. primary CTS.

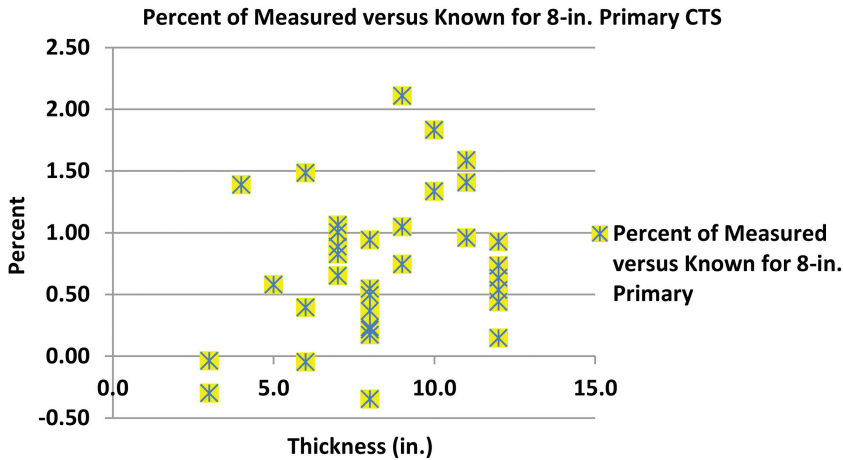


FIG. 11 Effect of standard thickness on measured result. The density is 12 kg/m³.



TABLE 11 305 mm (12.0 in.) six stacked fiberglass slabs (0.75 pcf) mean temperature (MT) (worst case).

Stack Configuration Combination Number	Thickness (in.) (12.0 in.) Stack	Mean Temperature (°F)	k Value (IP Units) of 12 °in. Stack	R Value Non-Corrected (IP Units)	k Value Corrected MT (IP Units)	R Value Corrected MT (IP Units)	Difference (%)
1	3	65			0.2786	10.77	
2	3	75	0.2887	41.57	0.2861	10.49	
3	6	85			0.2946	20.37	
						41.63	−0.06
1	3	65			0.2786	10.77	
3	6	75	0.2883	41.62	0.2861	20.97	
2	3	85			0.2946	10.18	
						41.92	−0.32
2	3	65			0.2786	10.77	
3	6	75	0.2899	41.39	0.2861	20.97	
1	3	85			0.2946	10.18	
						41.92	−0.53
3	6	65			0.2786	21.54	
2	3	75	0.2890	41.52	0.2861	10.49	
1	3	85			0.2946	10.18	
						42.21	−0.69
3	6	65			0.2786	21.54	
1	3	75	0.2889	41.54	0.2861	10.49	
2	3	85			0.2946	10.18	
						42.21	−0.67
2	3	65			0.2786	10.77	
1	3	75	0.2891	41.51	0.2861	10.49	
3	6	85			0.2946	20.37	
						41.63	−0.12

Note: All tests with no septa shows a comparison of calculated apparent thermal conductivity and thermal resistance results for non-corrected versus corrected for mean temperature through the stack. Also the percent difference in total added thermal resistance is compared for corrected versus non-corrected. Stack configuration No. 1 = KINA ID No. X-2009-2-005 thickness = 76.2 mm (3.0 in.). Stack configuration No. 2 = KINA ID No. X-2009-2-006 thickness = 76.2 mm (3.0 in.). Stack configuration No. 3 = KINA ID No. X-2009-2-009 thickness = 152.4 mm (6.0 in.). Thermal conductivity (SI units) = W/m K (IP units) = Btu in./h ft² °F). Thermal resistance (SI units) = m²/K W (IP units) = h ft² °F/Btu). MT, mean temperature.

Summary

The difference between the known and measured effective thermal conductivities range from −0.4 % to +2.1 % for the range of thicknesses and apparent thermal conductivities tested.

TABLE 12 Raw data.

Start Date	Test #	Single or Stacked	Fov 800 SN	Thickness (in.)	Cal Std 3-in primary used	Cal Calibration Constant (IP)	% Diff Cal Const. (IP)	PTLab Measured (IP)	Known NIST measured (IP)	Percent Difference %	Percent Difference U o I	Known Stacked Calculated (IP)	Septum used?	Q UPPER (μV)	Q LOWER (μV)	AVG GS	
2/10/2011	1 NIST 2003-2-005	single	S48	3.0	2003-2-006 SV348 b- 04144.9 (IP 2374 IP)	1.88578	0.12	0.2908	0.2899	0.31	0.03%	-	No	2051	2059	2055	
2/11/2011	2 NIST 2003-2-006	single	S48	3.0	2003-2-006 SV348 b- 04144.9 (IP 2374 IP)	1.88329	1.10	0.2916	0.2894	0.07	0.17%	-	No	2029	2076	2053	
2/21/2011	3 2003-2-005/2003-2-006	stacked double layer	S48	6.0	2003-2-006 SV348 b- 04144.9 (IP 2374 IP)	1.88731	0.09	0.2889	NA	0.09	4.27%	0.2886	No	1040	1001	1021	
2/21/2011	4 NIST 2003-2-006/2003-2-005	stacked double layer	S48	6.0	2003-2-006 SV348 b- 04144.9 (IP 2374 IP)	1.88778	0.06	0.2889	NA	0.09	3.70%	0.2886	No	1037	1004	1021	
2/22/2011	5 2003-2-005/2003-2-006 w/s	stacked double layer	S48	6.0	2003-2-006 SV348 b- 04144.9 (IP 2374 IP)	1.88657	0.12	0.2916	NA	0.05	4.33%	0.2886	Yes	1036	996	1016	
2/25/2011	6 NIST 2003-2-007	single	S48	4.0	2003-2-006 SV348 b- 04144.9 (IP 2374 IP)	1.88659	0.12	0.2914	0.2752	-1.38	1.07%	-	No	1463	1434	1439	
2/27/2011	7 NIST 2003-2-008	single	S48	5.0	2003-2-006 SV348 b- 04144.9 (IP 2374 IP)	1.88751	0.08	0.2823	0.2838	-0.53	2.71%	-	No	1210	1183	1197	
3/2/2011	8 NIST 2003-2-009	single	S48	6.0	2003-2-006 SV348 b- 04144.9 (IP 2374 IP)	1.88807	0.05	0.2916	0.2917	-1.41	4.46%	-	No	1036	995	1016	
4/25/2013	9 NIST 2003-2-000	single	S48	8.0	2003-2-006 SV348 b- 04144.9 (IP 2374 IP)	1.88857	0.13	0.2894	0.2899	-0.17	13.94%	-	No	819	715	767	
5/15/2013	10 NIST 2003-2-010	single	S48	8.0	2003-2-006 SV348 b- 04144.9 (IP 2374 IP)	1.87176	0.91	0.2890	0.2899	-0.31	14.58%	-	Yes	829	715	772	
											Measured Diffs						
Start Date	Test #	Single or Stacked	Fov 800 SN	Thickness (in.)	Cal Std 3-in primary used	Cal Calibration Constant (IP)	% Diff Cal Const. (IP)	PTLab Measured (IP)	Known NIST measured (IP)	Percent Difference %	Percent Difference U o I	Known Stacked Calculated (IP)	Septum used?	Q UPPER (μV)	Q LOWER (μV)	AVG GS	
3/17/2013	11 2003-2-005/2003-2-007	stacked double layer	S48	7.0	2003-2-006 SV348 b- 04144.9 (IP 2374 IP)	1.88809	0.15	0.2794	NA	-0.68	7.39%	0.2813	No	876	817	847	
3/18/2013	12 2003-2-007/2003-2-005	stacked double layer	S48	7.0	2003-2-006 SV348 b- 04144.9 (IP 2374 IP)	1.88737	0.09	0.2786	NA	-0.95	3.71%	0.2813	Yes	857	830	844	
3/19/2011	13 2003-2-007/2003-2-005 w/s	stacked double layer	S48	7.0	2003-2-006 SV348 b- 04144.9 (IP 2374 IP)	1.88846	0.13	0.2783	NA	-1.07	7.13%	0.2813	No	871	815	843	
3/24/2011	14 2003-2-006/2003-2-007	stacked double layer	S48	7.0	2003-2-006 SV348 b- 04144.9 (IP 2374 IP)	1.88734	0.09	0.2781	NA	-0.78	6.88%	0.2803	No	869	815	842	
3/25/2011	15 2003-2-007/2003-2-006	stacked double layer	S48	7.0	2003-2-006 SV348 b- 04144.9 (IP 2374 IP)	1.88711	0.10	0.2779	NA	-0.86	4.10%	0.2803	No	857	826	842	
NA	16 2003-2-006/2003-2-007	stacked double layer	S48	7.0	2003-2-006 SV348 b- 04144.9 (IP 2374 IP)	#VALUE!	#VALUE!	NA	NA	#VALUE!		0.2803	Yes	NA	NA	#DIV/0!	
											Measured Diffs						
Start Date	Test #	Single or Stacked	Fov 800 SN	Thickness (in.)	Cal Std 3-in primary used	Cal Calibration Constant (IP)	% Diff Cal Const. (IP)	PTLab Measured (IP)	Known NIST measured (IP)	Percent Difference %	Percent Difference U o I	Known Stacked Calculated (IP)	Septum used?	Q UPPER (μV)	Q LOWER (μV)	AVG GS	
4/18/2011	17 2003-2-005/2003-2-008	stacked double layer	S48	8.0	2003-2-006 SV348 b- 04144.9 (IP 2374 IP)	1.88698	0.11	0.2855	NA	0.13	7.09%	0.2851	No	782	731	757	
4/19/2011	18 2003-2-008/2003-2-005	stacked double layer	S48	8.0	2003-2-006 SV348 b- 04144.9 (IP 2374 IP)	1.88617	0.15	0.2850	NA	-0.05	6.94%	0.2851	No	780	731	756	
4/20/2011	19 2003-2-005/2003-2-008 w/s	stacked double layer	S48	8.0	2003-2-006 SV348 b- 04144.9 (IP 2374 IP)	1.88734	0.09	0.2848	NA	-0.12	7.37%	0.2851	Yes	781	728	755	
																#DIV/0!	
Start Date	Test #	Single or Stacked	Fov 800 SN	Thickness (in.)	Cal Std 3-in primary used	Cal Calibration Constant (IP)	% Diff Cal Const. (IP)	PTLab Measured (IP)	Known NIST measured (IP)	Percent Difference %	Percent Difference U o I	Known Stacked Calculated (IP)	Septum used?	Q UPPER (μV)	Q LOWER (μV)	AVG GS	
5/11/2011	20 2003-2-006/2003-2-008	stacked double layer	S48	8.0	2003-2-006 SV348 b- 04144.9 (IP 2374 IP)	1.88777	0.09	0.2846	NA	-0.19	7.64%	0.2851	No	781	727	754	
5/16/2011	21 2003-2-008/2003-2-006	stacked double layer	S48	8.0	2003-2-006 SV348 b- 04144.9 (IP 2374 IP)	1.88778	0.06	0.2826	NA	-0.89	5.34%	0.2851	No	767	749	749	
5/17/2011	22 2003-2-006/2003-2-008 w/s	stacked double layer	S48	8.0	2003-2-006 SV348 b- 04144.9 (IP 2374 IP)	1.88756	0.08	0.2879	NA	-0.50	7.04%	0.2851	Yes	776	727	752	
5/30/2011	23 2003-2-007/2003-2-008	stacked double layer	S48	9.0	2003-2-006 SV348 b- 04144.9 (IP 2374 IP)	1.88711	0.10	0.2877	NA	-0.72	7.12%	0.2799	No	676	633	655	
5/31/2011	24 2003-2-008/2003-2-007	stacked double layer	S48	9.0	2003-2-006 SV348 b- 04144.9 (IP 2374 IP)	1.88794	0.06	0.2742	NA	-2.04	7.97%	0.2799	No	670	621	646	
											Measured Diffs					#DIV/0!	

TABLE 12 (Continued)

Start Date	Test #	Single or Stacked	For 800 SN	Thickness (in.)	Cal Std 3-in primary used	Cal Calibration Constant (IP)	% Diff Cal Const. (IP)	PTLab Measured K (IP)	Known NIST measured K (IP)	Percent Difference %	Percent Difference U to L	Known Stacked Calculated K (IP)	Septum used?	QUPPER (uW)	QLOWER (uW)	AVG Qs
6/6/2011	25 2009-2-007/2009-2-008 w/s	stacked triple layer	548	10.0	2009-2-006 S1548 b= .04144 S12.28714 IP	1.88608	10.11	0.2770	NA	-1.04	6.93%	0.2799	Yes	614	631	653
6/21/2011	26 2009-2-007/2009-2-009	stacked double layer	548	10.0	2009-2-006 S1548 b= .04144 S12.28714 IP	1.88658	0.13	0.2811	NA	-1.32	8.83%	0.2849	No	621	571	596
6/21/2011	27 2009-2-009/2009-2-007	stacked double layer	548	10.0	2009-2-006 S1548 b= .04144 S12.28714 IP	1.88658	0.13	0.2811	NA	-1.32	11.88%	0.2849	No	630	562	596
6-23-2011	28 2009-2-007/2009-2-009 w/s	stacked double layer	548	10.0	2009-2-006 S1548 b= .04144 S12.28714 IP	1.88735	0.09	0.2798	NA	-1.78	9.39%	0.2849	Yes	620	566	593
Measured DIFFs																
Start Date	Test #	Single or Stacked	For 800 SN	Thickness (in.)	Cal Std 3-in primary used	Cal Calibration Constant (IP)	% Diff Cal Const. (IP)	PTLab Measured K (IP)	Known NIST measured K (IP)	Percent Difference %	Percent Difference U to L	Known Stacked Calculated K (IP)	Septum used?	QUPPER (uW)	QLOWER (uW)	AVG Qs
7/4/2011	29 2009-2-009/2009-2-009	stacked double layer	548	11.0	2009-2-006 S1548 b= .04144 S12.28714 IP	1.88626	0.15	0.2840	NA	-1.41	10.23%	0.2881	No	574	521	548
7/5/2011	30 2009-2-009/2009-2-008	stacked double layer	548	11.0	2009-2-006 S1548 b= .04144 S12.28714 IP	1.88826	0.04	0.2856	NA	-0.85	10.21%	0.2881	No	577	523	550
7/6/2011	31 2009-2-008/2009-2-009 w/s	stacked double layer	548	11.0	2009-2-006 S1548 b= .04144 S12.28714 IP	1.88705	0.10	0.2836	NA	-1.55	10.95%	0.2881	Yes	575	518	547
Measured DIFFs																
Start Date	Test #	Single or Stacked	For 800 SN	Thickness (in.)	Cal Std 3-in primary used	Cal Calibration Constant (IP)	% Diff Cal Const. (IP)	PTLab Measured K (IP)	Known NIST measured K (IP)	Percent Difference %	Percent Difference U to L	Known Stacked Calculated K (IP)	Septum used?	QUPPER (uW)	QLOWER (uW)	AVG Qs
8/20/2011	32 NIST 2009-2-005/2009-2-006/2009-2-009	stacked triple layer	548	12.0	2009-2-006 S1548 b= .04144 S12.28714 IP	1.88693	0.11	0.2887	NA	-0.50	10.61%	0.2902	No	536	484	510
8/23/2011	33 NIST 2009-2-006/2009-2-005/2009-2-009	stacked triple layer	548	12.0	2009-2-006 S1548 b= .04144 S12.28714 IP	1.88802	0.05	0.2883	NA	-0.64	11.89%	0.2902	No	538	480	509
8/25/2011	34 NIST 2009-2-005/2009-2-009/2009-2-006	stacked triple layer	548	12.0	2009-2-006 S1548 b= .04144 S12.28714 IP	1.88737	0.09	0.2899	NA	-0.09	12.80%	0.2902	No	544	480	512
8/26/2011	35 NIST 2009-2-006/2009-2-009/2009-2-005	stacked triple layer	548	12.0	2009-2-006 S1548 b= .04144 S12.28714 IP	1.88704	0.10	0.2890	NA	-0.40	11.09%	0.2902	No	538	483	511
8/27/2011	36 NIST 2009-2-009/2009-2-005/2009-2-006	stacked triple layer	548	12.0	2009-2-006 S1548 b= .04144 S12.28714 IP	1.88824	0.04	0.2889	NA	-0.44	10.68%	0.2902	No	536	484	510
8/28/2011	37 NIST 2009-2-009/2009-2-006/2009-2-005	stacked triple layer	548	12.0	2009-2-006 S1548 b= .04144 S12.28714 IP	1.88769	0.07	0.2881	NA	-0.37	10.25%	0.2902	No	535	486	511
8/31/2011	38 NIST 2009-2-005/2009-2-006/2009-2-009 w/s	stacked triple layer	548	12.0	2009-2-006 S1548 b= .04144 S12.28714 IP	1.88648	0.13	0.2875	NA	-0.92	13.09%	0.2902	Yes	540	476	508
9/3/2011	39 NIST 2009-2-006/2009-2-005/2009-2-009 w/s	stacked triple layer	548	12.0	2009-2-006 S1548 b= .04144 S12.28714 IP	1.88501	0.32	0.2875	NA	-0.92	10.97%	0.2902	Yes	535	481	508
9/4/2011	40 NIST 2009-2-009-2-003/2009-2-006 w/s	stacked triple layer	548	12.0	2009-2-006 S1548 b= .04144 S12.28714 IP	1.88889	0.01	0.2888	NA	-0.47	10.25%	0.2902	Yes	535	485	510
9/6/2011	41 NIST 2009-2-006/2009-2-009/2009-2-005 w/s	stacked triple layer	548	12.0	2009-2-006 S1548 b= .04144 S12.28714 IP	1.88463	0.24	0.2890	NA	-0.40	9.70%	0.2902	Yes	534	487	511
9/7/2011	42 NIST 2009-2-009/2009-2-005/2009-2-006 w/s	stacked triple layer	548	12.0	2009-2-006 S1548 b= .04144 S12.28714 IP	#REF!	#REF!	0.2886	NA	-0.54	7.77%	0.2902	Yes	528	491	510

Note: Lists all the raw data from February 2011 through August 2011. A total of 40 tests were run in single and stacked configurations up to and including 305 mm (12.0 in.) thickness with and without septa.

Conclusion

In this case, the use of this specific ASTM C518-10 apparatus for the measurement of effective apparent thermal conductivity at thicknesses greater than that of the calibration standard are possible, and do not introduce errors greater than the stated uncertainty of the primary CTS calibration standard. The measurement of loose-fill glass fiber insulation at representative thicknesses of an R-26 (IP units) installation is possible. One must use caution though as the potential exists that some ASTM C518-10 apparatus will not produce results similar to this example used in the study.

The data in Table 9 shows a worst-case triplicate stack with measured, individually added R values (IP units) and stacked R values (IP units), which were corrected and non-corrected for mean temperature. The resulting differences on total added R values (IP units) are fractions of 1 unit difference at 305 mm (12.0 in.) full thickness (see Tables 10–12). The data indicates that measurement differences in thermal resistance R values (IP units) are less than the associated uncertainty reported for the thermal resistance of the 203-mm (8.0-in.)-thick primary certified reference fiberglass standard (CTS) KINA product testing laboratory ID No. X-2009-1-010 at $k=2$ at 3.0 % from the National Laboratory. This stacking exercise follows the mandatory information requirements located in test method ASTM C518-10, Annex A1.8 through A1.8.2.

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Robert R. Zarr¹ and Stefan D. Leigh²

Standard Reference Material 1450d, Fibrous Glass Board, for Thermal Insulation Measurements

Reference

Zarr, Robert R. and Leigh, Stefan D., "Standard Reference Material 1450d, Fibrous Glass Board, for Thermal Insulation Measurements," *Next-Generation Thermal Insulation Challenges and Opportunities*, STP 1574, Therese K. Stovall and Thomas Whitaker, Eds., pp. 39–52, doi:10.1520/STP157420130063, ASTM International, West Conshohocken, PA 2014.³

ABSTRACT

Thermal conductivity measurements at and near room temperature are presented as the basis for certified values of thermal conductivity for Standard Reference Material 1450d, Fibrous Glass Board. The measurements have been conducted in accordance with a randomized full factorial experimental design with two variables, bulk density and temperature, using the National Institute of Standards and Technology 1016 mm line-heat-source guarded-hot-plate apparatus. The thermal conductivity of the specimens was measured over a range of bulk densities from 114 to 124 kg/m³ and mean temperatures from 280 to 340 K. Uncertainties of the measurements, consistent with current international guidelines, have been prepared. A summary of the physical properties and associated analyses of Standard Reference Material 1450d are presented.

Keywords

bulk density, certified reference material, guarded hot plate, heat flow meter, high density molded fibrous glass board, standard reference material, thermal conductivity, thermal insulation, thermal resistance

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¹Mechanical Engineer, National Institute of Standards and Technology, 100 Bureau Drive, Mail Stop 8632, Gaithersburg, MD 20899-8632, United States of America (Corresponding author), e-mail: robert.zarr@nist.gov

²Mathematical Statistician, National Institute of Standards and Technology, 100 Bureau Drive, Mail Stop 8632, Gaithersburg, MD 20899-8632, United States of America.

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Introduction

For 35 years, the National Institute of Standards and Technology (NIST) has issued thermal insulation Standard Reference Materials (SRMs)⁴ having certified value assignments for thermal resistance, thermal conductivity, and, most recently, bulk density. These Certified Reference Materials are provided by NIST as primary tools to assist user communities in achieving measurement quality assurance and metrological traceability. Thermal insulation SRMs, in particular, are utilized in standard test methods for the purposes of checking guarded-hot-plate apparatus [1], calibrating heat-flow-meter apparatus [2], and, when necessary, for checking or calibrating hot-box apparatus [3].

Standard Reference Material 1450 was originally issued by NIST (formerly the National Bureau of Standards⁵) in 1978 and was recently renewed in 2011 with the 1450d batch [4]. Like previous versions, SRM 1450d is a semi-rigid high-density, molded fibrous glass board consisting of fine glass fibers and a phenolic binding agent that limits the upper (test) temperature of the material to 473 K. Each unit is 26 mm thick and has lateral dimensions of 611 mm by 611 mm. The nominal thermal conductivity at 300 K is approximately 0.033 W/(m · K). This paper describes the production and certification of SRM 1450d and, in particular, summarizes the material lot requirements, bulk density homogeneity and thermal conductivity batch studies.

Materials

The general requirements for the renewal lot were based on recommendations from SRM customers and members of the ASTM Sub-committee C16.30 Task Group on Reference Materials. The material consignment was processed within a 3-day period and delivered to NIST in April 2009. Table 1 summarizes the manufacturing information for the SRM 1450d material lot. The consignment included 25 molded sheets having the same nominal density as the material described in Table 1; however, the finished lateral dimensions were larger, nominally 1200 mm by 1200 mm. These large sheets were from the same production run and were utilized later as lateral guarding for the thermal conductivity measurements.

Bulk Density Homogeneity Study

The objective of the homogeneity study was to quantify the density variability of the 1450d material lot (Table 1) by 100 % sampling of the panels and, thus,

⁴ The term “Standard Reference Material” and the diamond-shaped logo which contains the term “SRM,” are registered with the United States Patent and Trademark Office.
⁵ In 1901, Congress established the National Bureau of Standards (NBS) to support industry, commerce, scientific institutions, and all branches of government. In 1988, as part of the Omnibus Trade and Competitiveness Act, the name was changed to the National Institute of Standards and Technology (NIST) to reflect a broader mission for the agency.

TABLE 1 Manufacturing data for SRM 1450d.

Attribute	Value
Bulk density	128 kg/m ³ ± 10 %
Mold size	1245 mm by 1930 mm
Number of molded sheets	75
Number of panels per sheet	6 (die cut from molded sheet)
Number of panels	450
Nominal panel size	610 mm by 610 mm by 25.4 mm
Panel color	amber

determine bulk density rank (including upper and lower limits) and any anomalous panels (for possible exclusion). The 450 panels were randomly split into 9 groups of 50. Each group was randomly assigned to a 3-day interval in which the density of all 50 panels was determined. The sequence of events performed within the 3-day interval is shown in Table 2. The entire measurement process, which entailed several thousand mass and dimensional measurements for all 450 panels, required 30 days for completion [4].

BULK DENSITY MEASUREMENTS

The bulk density of the specimen (ρ_s), as defined in ASTM C177 [1], was determined by gravimetric and dimensional measurement procedures using Eq 1

(1)
$$\rho_s = \frac{m_s}{A_s \times L}$$

where:

- m_s = specimen mass, kg,
- A_s = specimen surface area, m², and
- L = specimen thickness, m.

MASS MEASUREMENTS

The mass measurement station consisted of: (1) digital weighing balance (32.1 kg range, 0.0001 kg resolution), (2) foot switch for manual activation, and (3) serial interface for automated data collection. As described in Table 2, each panel was

TABLE 2 Sampling plan for bulk density homogeneity study^a.

Day	Temperature, °C	Time, h	Event
1	100	24	50 panels conditioned in convection oven (100°C)
2	23	4	Mass time-history for each panel
2	23	17	50 panels conditioned in ambient (24°C)
3	23	4	Dimensional measurements of each panel

^aSequence of events for 3-day interval in which the density of all fifty panels are determined.

removed individually from an oven environment of 100°C and weighed every 20 s for 180 s. By measuring the panel mass at equal time intervals and establishing a mass history, the initial mass (m_0) for each panel at time zero (t_0) was determined by linear regression, thus correcting for the small mass change with time.

DIMENSIONAL MEASUREMENTS

The dimensional measurement station consisted of: (1) granite surface plate (1.2 m by 1.8 m), (2) electronic height gage with digital readout (330 mm range or 635 mm range, 0.01 mm resolution), (3) bi-directional touch probe (3-mm diameter carbide ball contact point, 0.4 N measuring force), and (4) output cable for automated data collection. As described in Table 2, the group of panels was conditioned in air at conditions near 24°C. Each panel was placed on the granite surface plate and lateral and thickness dimensional measurements were carried out by two operators using separate height gages. The lateral dimensions, identified as length (l) and width (w), were measured by clamping the insulation panel securely in the vertical position (one edge on the granite surface plate) between an aluminum plate and a right-angle support fixture. Linear dimensions were obtained at the center lines of the panel. Preliminary tests indicated that data acquired from the two middle axes were sufficient for an accurate determination of bulk density. One panel from each group, however, was selected at random for measurements at six locations (three equally spaced locations per side of panel) as a check.

For thickness dimensions, the insulation panel was placed in the horizontal position (lateral surface on the granite surface plate) and a modest load (approximately 43 N) was applied to the top of the panel. The panel thickness was measured at four locations, with each location representing the geometric center of a 200 mm by 200 mm sub-area of the panel. Preliminary tests indicated that the data acquired from four locations were sufficient for an accurate determination of the bulk density. For each group, approximately one-half of the panels were measured at the corner sub-areas and, for the other half, at the mid-center sub-areas. One panel from each group was selected at random for measurements at all eight locations.

SUMMARY STATISTICS

Table 3 provides summary statistics across the panels for the mass (m_s), length (l), width (w), area (A_s), thickness (L), and bulk density (ρ_s) of the 450 panels. The values for l and w are within acceptable limits. The large range and small standard deviation for L indicate that some of the panels were either unacceptably thin or thick. The mean and standard deviation values for ρ_s are acceptable, but the mean is near the low limit requirement (Table 1).

BULK DENSITY VARIATION

Figure 1 plots the bulk density for the 450 panels in rank order. There were two panels outside the upper and lower limits equal to the grand mean plus or minus

TABLE 3 Summary statistics for SRM 1450d bulk density homogeneity study.

Statistic	m_s , kg	l , mm	w , mm	A_s , m ²	L , mm	ρ_s , kg/m ³
Mean	1.1466	610.92	610.85	0.37318	25.88	118.7
Std. dev.	0.0250	0.62	0.56	0.00051	0.18	2.9
Range	0.1635	2.85	3.00	0.00243	1.74	18.0
Min	1.0697	609.50	609.55	0.37195	25.25	109.8
Max	1.2331	612.35	612.55	0.37438	26.99	127.8

three times the standard deviation ($\pm 3s$ where s equals 2.9 kg/m³ from Table 3). These panels and five other panels, identified from unacceptable thickness measurements, were removed from the production lot. The excluded panels represented only 1.6 % (7 of 450) of the 1450d material lot; 443 panels, or 98.4 %, were accepted.

Thermal Conductivity Batch Study

THERMAL CONDUCTIVITY MEASUREMENTS

The thermal conductivity measurements were determined using the NIST 1016 mm diameter guarded-hot-plate apparatus. Figure 2 shows the essential features of the guarded-hot-plate apparatus designed for operation in the double-sided mode near ambient temperature conditions. The apparatus is

FIG. 1 Panel bulk density variation, rank ordered. Control limits are three times the standard deviation ($\pm 3s$, where s equals 2.9 kg/m³ from Table 3).

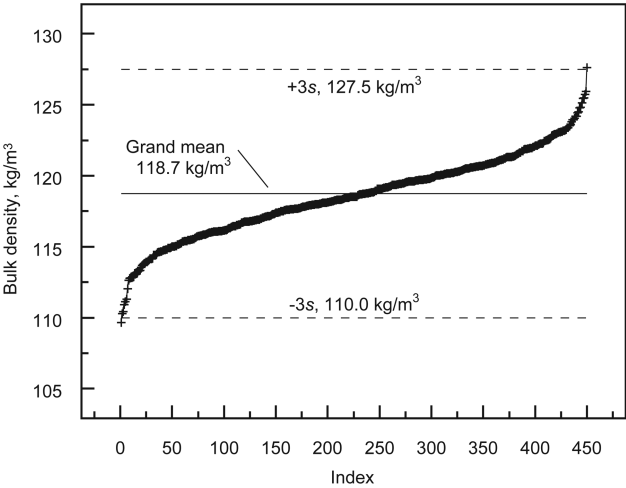
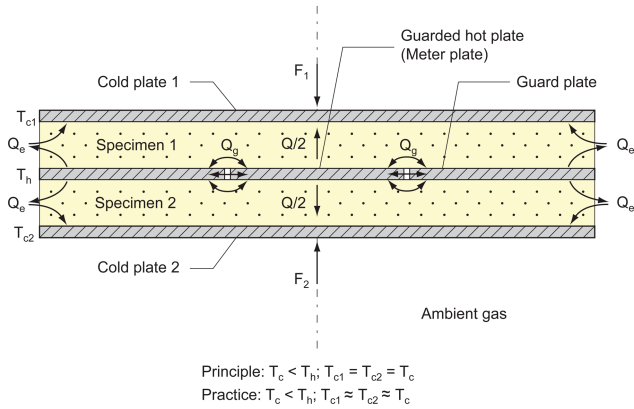


FIG. 2 Guarded-hot-plate schematic, double-sided mode of operation.

cylindrically symmetric about the axis indicated in Fig. 2, the plates are horizontal, and heat flow is vertical through the pair of specimens. The specimens, which have nearly the same density, size, and thickness, are placed on each surface of the guarded hot plate and clamped securely by the cold plates (F_1 and F_2). The guarded hot plate (T_h) and the cold plates (T_{c1} , T_{c2}) provide constant-temperature boundary conditions to the specimen surfaces. With appropriate guarding, lateral heat flows (Q_g and Q_e) are reduced to negligible proportions and, under steady-state conditions, the apparatus provides one-dimensional heat flow (Q) normal to the meter area of the specimen pair. A secondary guard is provided by an enclosed chamber that conditions the ambient air surrounding the plates to a temperature near to the mean specimen temperature (i.e., average surface temperatures of the hot and cold plates in contact with the specimens).

The experimental thermal conductivity (λ_{exp}), as defined in ASTM C1045 [6] was determined using Eq 2

$$(2) \quad \lambda_{\text{exp}} = \frac{QL_{\text{avg}}}{2A\Delta T_{\text{avg}}}$$

where:

Q = time rate of one-dimensional heat flow through the apparatus metering area (A), W,

A = metering surface area ($2A$ occurs because Q flows through two surfaces, Fig. 2), m^2 , and

L_{avg} , m, and ΔT_{avg} , K, are defined in Eqs 3 and 4, respectively.

$$(3) \quad L_{\text{avg}} = (L_1 + L_2)/2$$

$$(4) \quad \Delta T_{\text{avg}} = (\Delta T_1 + \Delta T_2)/2 = [(T_h - T_c)_1 + (T_h - T_c)_2]/2$$

The subscripts 1 and 2 in Eqs 3 and 4 correspond to the two specimens and respective cold plates illustrated in Fig. 2. In this study, the cold plate temperatures (T_{c1} , T_{c2}) and specimen thicknesses (L_1 , L_2) are nearly the same, respectively. The thermal conductivity property determined in Eq 2 corresponds to a mean test temperature T_m given by Eq 5.

$$(5) \quad T_m = (T_h + T_c)/2 = \left(T_h + \frac{T_{c1} + T_{c2}}{2} \right) / 2$$

EXPERIMENTAL DESIGN

In contrast to the 100 % sampling process for bulk density, the thermal conductivity of 1450d was batch certified. The experimental design for the thermal conductivity batch study was based on an initial model that was developed from the results of the previous version SRM 1450c [5]. The initial model for thermal conductivity (λ) as a function of bulk density (ρ) and temperature (T) was assumed to be bilinear in ρ and T as shown in Eq 6.

$$(6) \quad \lambda(\rho, T) = a_0 + a_1 \rho + a_2 T$$

Table 4 summarizes the experimental design for the thermal conductivity batch certification of 1450d. The design called for three levels for density qualitatively assigned as low, mid, and high and five levels for temperature from 280 to 340 K. The test sequence, shown as circled numbers ① in Table 4, was randomized to mitigate the influence of any systematic effects.

Each cell in Table 4 represents one measurement of a different pair of specimens (15 tests in total). The benefit of testing a unique pair of specimens at each combined level of temperature and density is that independent information is obtained at each such level. The experimental design given in Table 4 is balanced in the sense that an equivalent amount of information is obtained at each setting of the independent variables.

Results

Table 5 provides the measurement results for the 15 tests summarized in the experimental design (Table 4). The rows of data are arranged by T_m from 280 to 340 K and, within each level of T_m , the specimen densities (ρ_s) are ranked in ascending order. The data are partitioned in four sections: specimen bulk density (ρ_s), input quantities for Eqs 2 and 4; secondary test quantities, and (resultant) thermal conductivity (λ_{exp}). The notations “1” and “2” designate the top and bottom specimen, respectively, as illustrated in Fig. 2, and column 4 is the average of ρ_{s1} and ρ_{s2} . The “certified” density range for SRM 1450d is defined by the ultimate span for ρ (114 to 124 kg/m³).

During a test, the input temperature estimates, T_h , T_{c1} , and T_{c2} , were maintained within 0.01 K, or less, of their respective set-point temperatures. The estimates for $Q/2$ ranged from 3.9 W to 4.8 W for T_m at 280 and 340 K, respectively. However, for a given level of T_m , the variation of $Q/2$ due to changes in ρ_s was much smaller. The

TABLE 4 Experimental design for SRM 1450d batch study.

Density Level	Temperature Level, K				
	280	295	310	325	340
Low	1 obs. ^a ①	1 obs. ⑫	1 obs. ⑨	1 obs. ⑬	1 obs. ⑤
Mid	1 obs. ⑦	1 obs. ⑩	1 obs. ③	1 obs. ⑮	1 obs. ⑧
High	1 obs. ④	1 obs. ⑭	1 obs. ⑥	1 obs. ⑪	1 obs. ②

^aObservation; circled number represents random sequence.

estimates for A have been corrected for thermal expansion effects of the meter-plate radius [4] and the estimates for the in situ test thickness L were determined by averaging the digital outputs of eight linear position transducers (four for each cold plate) [4]. The estimates for λ_{exp} include an extra digit for display purposes.

During a test, the secondary (influence) quantities (T_a , p_a , RH , and f) were either controlled or only recorded. The chamber air temperature (T_a) was controlled to be the same temperature as T_m (within 0.1 K, or less) and the chamber air pressure (p_a) varied with the site barometric conditions (from 97.5 to 101.3 kPa). The chamber relative humidity (RH) was maintained less than 10 % RH by a dry-air purge and varied with T_a . The clamping pressure (f) was determined by averaging the loading force (F) applied by each cold plate divided by the surface area of the cold plate, which was corrected for thermal expansion effects. Table 6 provides summary statistics for test quantities given in Table 5 that were fixed at one value across all tests. The temperature difference (ΔT) of 25.0 K was based on ASTM C1058 [7], standard practice for selecting test temperatures.

Discussion

DATA SCREENING—GRAPHICAL ANALYSIS

Figures 3 and 4 plot values of λ_{exp} from Table 5 as a function of the design model (Eq 6) input variables ρ_s and T_m , respectively. For Fig. 3, the individual data points are plotted as filled circle symbols corresponding to T_m levels of 280, 295, 310, 325, and 340 K. The error bars represent expanded uncertainties (described later) of 0.86 %. For Fig. 4, the individual data points are plotted as filled circle, square, and triangle symbols (without error bars for clarity) corresponding to the three main levels selected for bulk density.

DATA EVALUATION—CHARACTERIZATION

The data in Fig. 3 strongly suggest that, in the range of ρ_s and T_m covered for the 450 panels comprising the current SRM:

- (1) λ_{exp} is insensitive to bulk density for the range studied (in contrast with previous 1450 version materials), and
- (2) the dependence of λ_{exp} on mean temperature is strongly linear.

TABLE 5 Thermal conductivity measurement data (sorted by T_m and ρ_s)

T_m , K	ρ_s , kg/m ³			Input Quantities for Eqs 2, 4, and 5							Secondary Quantities					λ_{exp}^a , W/(m·K)
	1	2	Avg.	T_h , K	T_{ch} , K	T_{cs} , K	$Q/2$, W	A , m ²	L_{avg} , mm	T_n , K	p_n , kPa	RH , %	f , Pa			
280	112.8	114.3	113.5	292.50	267.50	267.50	3.895	0.12980	25.93	280.0	99.3	8	477	0.03112		
280	118.3	118.7	118.5	292.50	267.50	267.51	3.894	0.12980	25.80	280.0	99.6	8	457	0.03096		
280	121.0	121.4	121.2	292.50	267.50	267.49	3.924	0.12980	25.56	280.0	100.6	9	478	0.03090		
295	113.5	115.2	114.3	307.50	282.50	282.50	4.050	0.12989	26.02	295.0	100.5	4	460	0.03245		
295	118.9	119.1	119.0	307.50	282.50	282.50	4.086	0.12989	25.82	295.0	100.6	4	461	0.03249		
295	121.1	121.8	121.5	307.50	282.50	282.50	4.105	0.12989	25.80	295.0	100.2	3	472	0.03261		
310	116.0	115.6	115.8	322.50	297.50	297.50	4.240	0.12998	26.13	310.0	100.1	2	484	0.03409		
310	118.5	118.9	118.7	322.50	297.50	297.50	4.324	0.12998	25.76	310.0	98.6	2	506	0.03428		
310	122.0	122.8	122.4	322.50	297.51	297.50	4.317	0.12998	25.76	310.0	99.7	2	488	0.03422		
325	115.2	116.1	115.7	337.50	312.50	312.50	4.494	0.13007	25.85	325.0	100.5	1	495	0.03572		
325	118.8	118.9	118.9	337.50	312.50	312.50	4.499	0.13007	26.00	325.0	101.3	1	491	0.03597		
325	122.5	123.9	123.2	337.50	312.50	312.50	4.549	0.13007	25.73	325.0	97.5	1	501	0.03599		
340	115.3	116.4	115.8	352.50	327.49	327.50	4.701	0.13016	25.98	340.0	99.7	1	466	0.03752		
340	118.6	119.7	119.1	352.50	327.50	327.50	4.747	0.13016	25.78	340.0	101.3	1	478	0.03760		
340	123.3	124.3	123.8	352.50	327.50	327.49	4.760	0.13016	25.85	340.0	99.1	<1	432	0.03782		

^aExtra digit included for display purposes.

TABLE 6 Summary statistics for fixed primary and secondary quantities.

	$\Delta T, \text{K}$	$L_{\text{avg}}, \text{mm}$	p_a, kPa	f, Pa
Grand mean	25.001	25.85	99.9	476
Std. dev.	0.003	0.14	1.0	19

The first assertion is borne out by linear least squares fits to the 3-point horizontal profiles of Fig. 3. Table 7 summarizes the slopes of the lines shown in Fig. 3 and their corresponding t -values, which are defined as the ratio of the slope and the standard error of the slope estimate. For the three middle profiles (Fig. 3), the slope of the fitted line is statistically indistinguishable from zero. The slopes of the extreme profiles are opposite in sign.

A fit of the no-intercept line model $\lambda_{\text{exp}} = a_2 T_m$ yields excellent fit statistics (R -square = 0.997) and is visually an excellent fit as well (Fig. 4). The residual standard deviation for the fit is 0.00012 W/(m · K). Table 8 summarizes the regression statistics for the no-intercept linear fit to temperature.

- For comparison and further model validation, other models were fit including:
- (1) bilinear in ρ and T , with and without constant term, and
 - (2) quadratic and cubic in T , with and without constant term.

For all the models assayed, one or more fitted coefficients were non-significant, with the exception of the pure third power model (T^3) with no constant, linear, or

FIG. 3 Graphical analysis of thermal conductivity data showing λ_{exp} dependence on ρ_s , error bars represent expanded uncertainties of 0.86 %.

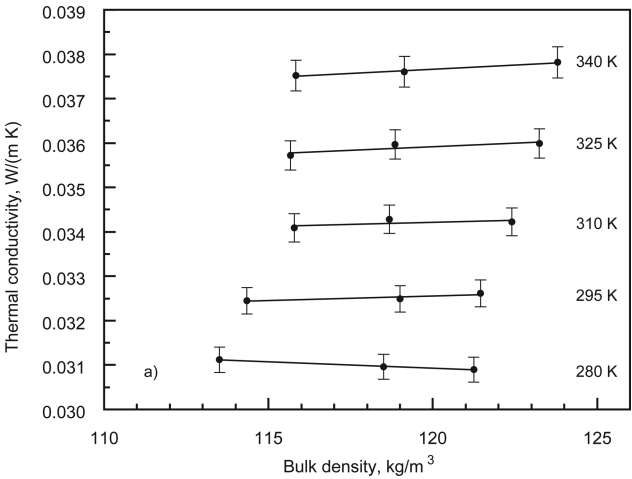
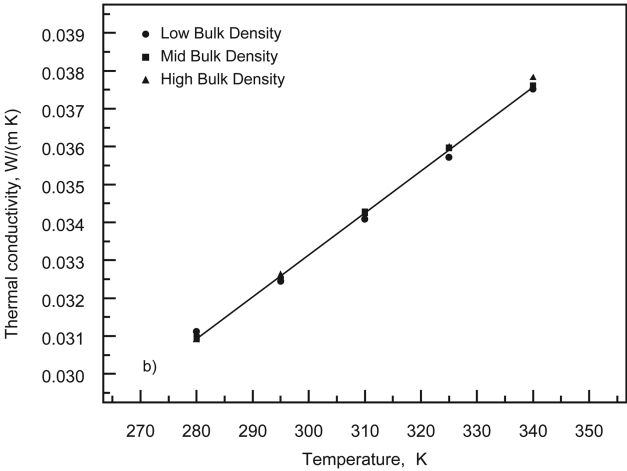


FIG. 4 Graphical analysis of thermal conductivity data showing λ_{exp} dependence on T_m (without error bars for clarity).



quadratic term. Both visually (superimposed and residual plots) and statistically (magnitudes of residual standard errors, fitted coefficients, standard errors of coefficients, and associated t -values), all other models, including T^3 , were found to be inferior to the simplest first power (linear) model in T with no constant term.

FINAL MODEL

Equation 7, derived by least squares linear regression of λ on T , gives the final certification model for SRM 1450d.

(7)
$$\hat{\lambda} = (1.10489 \times 10^{-4}) \times T_m$$

Figure 5 plots the deviations $(\lambda_{\text{exp}} - \hat{\lambda})/\hat{\lambda}$ (in %) versus T_m by bulk density (filled circle, square, and triangle symbols corresponding to low-, mid-, and high- levels of bulk density, respectively). The deviations for mid- and high-bulk densities are randomly and interchangeably scattered (i.e., no discernible pattern) around zero. The

TABLE 7 Summary of linear profiles for λ_{exp} versus ρ_s (Fig. 3).

T_m , K	Slope, (W · m ²)/(K · kg)	t -value
280	-2.939×10^{-5}	-12.1
295	2.114×10^{-5}	2.0
310	1.850×10^{-5}	0.8
325	3.314×10^{-5}	1.6
340	3.741×10^{-5}	6.4

TABLE 8 Summary of regression statistics for λ_{exp} versus T_m (Fig. 4).

Regression coefficient	Slope, W/(m · K ²)	s of slope, W/(m · K ²)	t-value
a_2	1.10489×10^{-4}	1.010×10^{-7}	1094

low-bulk densities, however, are biased low (about -0.5%), with the exception of one low-temperature point. The same pattern is visible on close inspection in Fig. 4. The bias magnitude, however, is dominated by the experimental uncertainty of $\pm 0.86\%$.

UNCERTAINTY BUDGET

For the multiplicative expression of Eq 2, the relative combined standard uncertainty in λ_{exp} can be expressed as the relative uncertainties associated with each factor combined in quadrature.

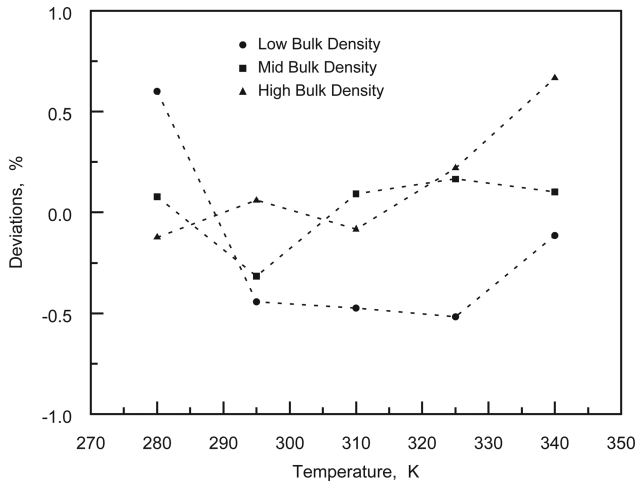
(8)
$$u_{c,\text{rel}}(\lambda_{\text{exp}}) = \frac{u_c(\lambda_{\text{exp}})}{\lambda_{\text{exp}}} = \sqrt{\left(\frac{u(Q)}{Q}\right)^2 + \left(\frac{u(\Delta T)}{\Delta T}\right)^2 + \left(\frac{u(L)}{L}\right)^2 + \left(\frac{u(A)}{A}\right)^2}$$

The standard uncertainties and input quantities used in Eq 8 are derived in Ref. [4]

(9)
$$u_{c,\text{rel}}(\lambda_{\text{exp}}) = \sqrt{\left(\frac{0.0074}{4.736}\right)^2 + \left(\frac{0.077}{25.001}\right)^2 + \left(\frac{0.065}{25.85}\right)^2 + \left(\frac{0.000043}{0.13016}\right)^2}$$

(10)
$$u_{c,\text{rel}}(\lambda_{\text{exp}}) = \sqrt{(0.00156)^2 + (0.00308)^2 + (0.00251)^2 + (0.00033)^2} = 0.0043$$

FIG. 5 Graphical analysis of deviations (in %) for the fit given in Eq 7.



The comparative contribution for the first term in Eq 10 is 13.3 %; for the second, 51.9 %; for the third, 34.2 %; and, for the fourth, 0.6 %. The major contributors to the uncertainty for SRM 1450d are the empirical determinations for specimen temperature difference (ΔT) and thickness (L_{avg}). These findings are consistent with results from previous NIST guarded-hot-plate analyses [5].

The relative expanded uncertainty, U_{rel} , for a coverage factor of k equals 2 is given in Eq 11.

$$(11) \quad U_{\text{rel}}(\lambda_{\text{exp}}) = 2u_{\text{c,rel}}(\lambda_{\text{exp}}) = 0.0086$$

Expressed as a % ($\times 100$), U_{rel} is equal to 0.86 %, which has been rounded to 1 % on the 1450d certificate for ease of use.

Summary

Thermal conductivity measurements of Standard Reference Material 1450d, Fibrous Glass Board, have been conducted using the NIST 1016 mm line-heat-source guarded-hot-plate apparatus following a full factorial experimental design with two variables, bulk density, and temperature. The thermal conductivity measurements were conducted over ranges of bulk densities from 114 to 124 kg/m³ and mean temperatures from 280 to 340 K. Analysis of the measurements revealed that the thermal conductivity of the material lot was insensitive to bulk density for the range exhibited by the 1450d material and strongly linearly dependent on temperature. The final model for the predicted thermal conductivity of 1450d was found to be a first power (linear) model in T with no constant term. The expanded uncertainty for thermal conductivity was prepared consistent with current international guidelines and was determined to be 0.86 %, which has been rounded to 1 % on the 1450d certificate for ease of use.

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Wayne C. Thresher¹ and David W. Yarbrough²

Development and Use of an Apparatus for In Situ Evaluation of the Thermal Performance of Building-Envelope Components

Reference

Thresher, Wayne C. and Yarbrough, David W., "Development and Use of an Apparatus for In Situ Evaluation of the Thermal Performance of Building-Envelope Components," *Next-Generation Thermal Insulation Challenges and Opportunities*, STP 1574, Therese K. Stovall and Thomas Whitaker, Eds., pp. 53–65, doi:10.1520/STP157420130083, ASTM International, West Conshohocken, PA 2014.³

ABSTRACT

R values determined in laboratory settings, according to adopted standard methods are reproducible quantities that predict the thermal resistance of various building materials under specified conditions. Insulation materials installed in walls, floors, and ceilings, however, are subject to a wide range of variables, including air and moisture ingress, biological challenges, settling, and other physical changes and temperature variations. Hence, there is a need for accurate in situ thermal measurements to quantify building system thermal resistance under actual environmental conditions. A self-supporting portable instrument that satisfies the requirements of ASTM C1046 has been developed to measure heat flux and *R* values in situ. The instrument consists of a housing that automatically positions a heat-flux transducer (HFT) on the region to be tested. The heat-flux transducer is a bismuth-telluride Peltier device that generates a voltage that is proportional to the heat flux. A temperature sensor is attached to the heat-flux transducer. A fan circulates air from the surroundings through the instrument housing to ensure that the interior of the housing is in

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¹Ph.D., Cambria, Ltd., Ashhurst, New Zealand.

²Professor Emeritus, Ph.D., R&D Services, Inc., Cookeville, TN 38501, United States of America.

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thermal equilibrium with the surroundings. The fan also creates a pressure difference that is sufficient to press the instrument firmly against the surface of a smooth wall or ceiling. A second temperature sensor, located on the opposite side of the building envelope, sends data to the instrument electronically to provide for a calculation of the temperature difference across the test region. A microcomputer receives the heat flux and temperature data needed for R -value calculations. This instrument can be used to evaluate heat flow and thermal resistance for walls, ceilings, floors, and other components that are part of the building envelope. In situ R values and heat-flux data obtained with this instrument will be discussed in this paper.

Keywords

in situ, thermal resistance, R value

Introduction

The thermal resistance of building elements and systems are important determinates of energy efficiency. Optimization of energy efficiency relies, in part, on maximizing the resistance to heat transfer across ceilings, walls, and floors within the limits of budget constraints and other practicalities. To help in the effort to achieve energy-efficient buildings, the Federal Trade Commission (FTC) enforces a number of consumer protection statutes, including “The R value Rule” [1]. The primary purpose of the rule is to ensure that the consumer is provided with comparable information to make cost-effective choices across the wide range of insulation materials that are available. One important outcome of the rule has been the appearance of a numerical indication of the laboratory-determined thermal resistance, or R value, clearly displayed on the label or via a “fact sheet” at the point of purchase of all residential thermal insulation materials.

The R value is an accurate indication of the thermal resistance of a material as it exists under controlled conditions of humidity, temperature difference, and air movement. It contributes to the prediction of heat flow through insulation, framing, cladding, and other building elements used in a building assembly. It is commonplace to assume that the label R value holds for installed insulations. However, it is recognized that the thermal resistance of most materials is influenced by environmental conditions, variable construction practices, physical degradation, and site-specific factors. A solution is to directly measure the performance of a building assembly with heat-flux transducers (HFTs) and temperature sensors. The results of these measurements can be interpreted as an “in situ R value,” a metric that includes the effect of environmental conditions on thermal performance. ASTM C1046 [2] prescribes the appropriate procedure for in situ determinations of thermal resistance. The standard, however, is not often applied because, in part, of the complexity of setting up the necessary equipment. This paper describes the development and application of an instrument specifically designed to monitor heat flux and obtain in situ thermal resistance over time scales relevant for useful

assessments. We describe a two-part instrument useful to measure the thermal properties of assemblies, including building elements and systems in either a laboratory setting under controlled conditions or in situ over time under the dynamically changing conditions of a real building or other “in-the-field” setting. One part is the module that is installed on an inside surface of the region to be measured. The second part is the module that is installed on the outside surface of the material to be measured. Hereafter, we will refer to the instrument as a “portable flux meter” (PFM).

Materials and Methods

INSULATION MATERIALS USED IN THIS RESEARCH

Fiberglass batts, expanded polystyrene, and polyester blanket were selected for use in this research. The apparent thermal conductivities for the selected insulations were measured as a function of temperature in accordance with ASTM C518 [3] for the selected insulations.

PORTABLE HEAT-FLUX METER APPARATUS

Because the PFM is currently under review with the USPTO, design details will be limited to a general description. The PFM consists of an inside module to be placed on a ceiling, wall, or floor on the interior of a building. The inside unit consists of a plastic shell that contains a heat-flux transducer and temperature sensor against the surface of the region to be evaluated. A fan located inside the shell exhausts air from the interior of the shell to the exterior, thus creating a pressure difference sufficient to push the module firmly against the building surface. The inside unit is self-supporting because the small pressure difference caused by the fan provides enough force to support the module against a smooth wall or ceiling. The module can also be held in place by other means, such as tape. A rubber seal installed around the perimeter of the plastic shell maintains the pressure difference created by the fan. A smooth surface is needed to ensure a good seal, although it has been tested successfully on a ceiling coated with textured paint. Another function of the fan is to ensure mixing of the air inside the plastic shell. A temperature sensor located in the exterior module measures the exterior surface temperature. An onboard microprocessor receives data from the temperature sensors and the HFT computes heat flux and the instantaneous value for the ratio $\Delta T/\text{flux}$ and outputs the results to a data logger and an LED display. The HFT is based on a bismuth-telluride Peltier plate as previously described [4]. In this application, the semiconductor was used to generate dc voltage that is proportional to the heat flux. The HFT was calibrated using various known materials to provide for the conversion of the electrical potential to heat flux. The outside module consists of a plastic shell fitted with a temperature sensor and a microprocessor combined with a miniature radio transmitter. The transmitter sends the outside temperature reading to a receiver in the inside module.

Test Assemblies

STEADY-STATE TEST ASSEMBLIES

A wall section was constructed using standard framing timber clad on both sides with 10-mm-thick plasterboard to test the PFM under steady-state conditions. The framing was 400-mm outside circumference (OC) with single top and bottom plates. The overall test wall dimensions were $1200 \times 1200 \times 110 \text{ mm}^3$. The wall cavity was insulated in one case with expanded polystyrene (EPS), and in the second case with fiber-glass batt insulation. These were selected to provide a range of resistances that would be typical of those encountered “in the field.” The wall assembly was also tested without insulation. The wall with the PFM mounted midway between studs and midway between the top and bottom plates was placed between a pair of temperature-controlled chambers. The air temperature of the two chambers was controlled to create and maintain a constant surface-to-surface temperature difference of 15°C across the test assembly. The system was allowed to operate for 2 h before the start of data logging. Data from the PFM were logged for approximately 30 h for each test.

DYNAMIC TESTS

In situ tests were undertaken in a wooden farmhouse located in the central area of New Zealand’s north island in late February. Two separate sections of a bedroom floor were fitted with thermal insulation. One was the EPS used in the test wall (nominal RSI, $1.4 \text{ m}^2 \cdot \text{K/W}$, 60 mm thick) and the other was a polyester blanket (nominal RSI, $1.8 \text{ m}^2 \cdot \text{K/W}$, 90 mm thick). The insulations were fitted under the subflooring according to the manufacturer’s instructions. A third floor section was left with no insulation. These insulations were chosen because they have very different physical properties. The polyester blanket is very open to air and moisture transport as compared to the closed-cell structure of the EPS. The floor was made of 20-mm-thick hardwood planks over joists. The joists were 120 by 50 mm and separated 430 mm OC. The floor joists were mounted on a pair of pile-supported bearers. The distance from the ground to the floor joists was $300 \pm 50 \text{ mm}$. Identical PFMs were located in the middle of each of three sections, positioned midway between joists. The outside unit was supported with the temperature sensor pressed gently against the bottom surface of the exposed insulation or in the case of no insulation, against the subflooring. The result is a side-by-side evaluation of three sections of floor with heat-flow down.

Results

APPARENT THERMAL CONDUCTIVITY MEASUREMENTS

The relationship between average temperature and apparent thermal conductivity (λ) of the insulation materials used in this study is shown in Table 1. The range of observed average temperatures for the insulations in the assembly tests is contained in Table 1 along with λ calculated at the average temperature.

TABLE 1 Measured thermal properties of insulations used in this study.

Insulation Type	Measured λ (W/m · K) Using C518 [3]	T (°C)	λ (W/m · K)	Thickness (m)	RSI (m ² · K/W)
Fiberglass batt	$\lambda = 0.000129 \cdot T + 0.029083$	23.51 (23.3/23.7)	0.0321	0.090	2.80
Expanded polystyrene	$\lambda = 0.000171 \cdot T + 0.037800$	22.38 (22.2/22.6)	0.0416	0.060	1.44
Polyester blanket	$\lambda = 0.000265 \cdot T + 0.037978$	24.69 (22.1/27.7)	0.0445	0.090	2.02

STEADY-STATE TESTS

Figures 1, 2, and 3 show the instantaneous in situ R values obtained using the PFM on the test wall system. The mean and standard deviation for each test (air gap, polyester, or expanded polystyrene) are listed in Table 2. Measurements were recorded every 10 min. The results for the test with no insulation are contained in Fig. 1. Figure 2 contains the results for the test with 90 mm of fiberglass batts. Figure 3 contains the results for the test with 60 mm of expanded polystyrene and a 30 mm air gap.

The RSI in column 1 includes the R value for two layers of 10-mm-thick gypsum ($0.118 \text{ m}^2 \cdot \text{K/W}$) from the *AIRAH Technical Handbook* [5]. The R values for fiberglass and polystyrene were computed using the measured thermal resistance values listed in Table 1. The R values for air gaps in the uninsulated wall and the partially insulated polystyrene wall were calculated using the procedure outlined by Desjarlais and Yarbrough [6]. Column 3 shows the measured in situ R values

FIG. 1 In situ R values obtained using PFM on test wall with no insulation.

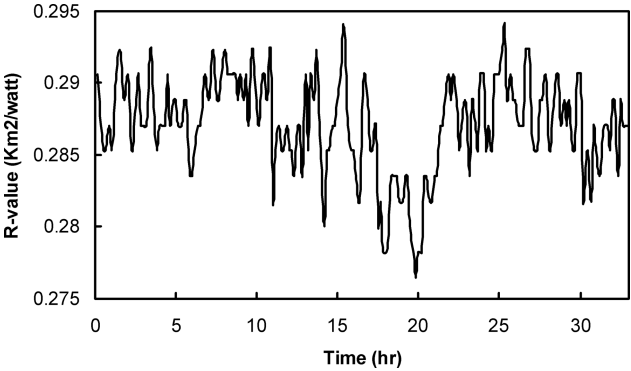
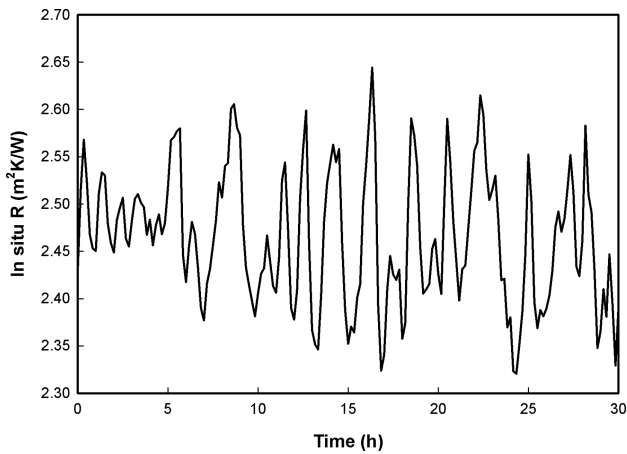


FIG. 2 In situ R values obtained using PFM on test wall with fiberglass batt.



obtained from the PFM. The standard deviations shown in Table 2 are for data sets consisting of all measurements used to establish the average for a 24-h period (after equilibration for the previous 6 h). The last column in Table 2 contains the percent difference between the PHM and the laboratory-measured components.

DYNAMIC MEASUREMENTS

The daily fluctuation in temperature during the trial is shown in Fig. 4. The starting time [0] for this trial was 17:00 h.

FIG. 3 In situ R values obtained using PFM on test wall with polystyrene.

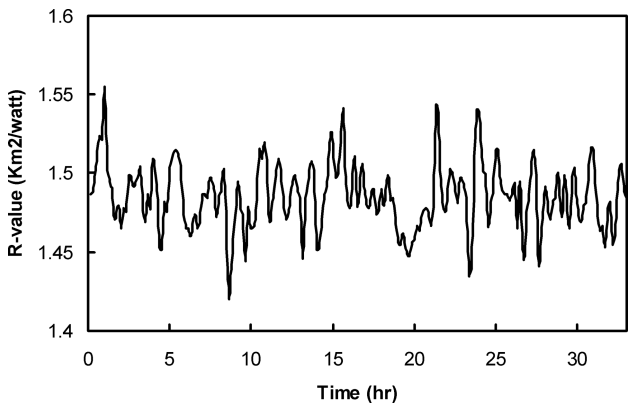


TABLE 2 RSI-values for the three wall tests.

Wall Insulation	RSI	In Situ <i>R</i> Value	Standard Deviation	% Difference ^a
Air gap	0.277	0.287	0.003	3.6
Fiberglass	2.92	2.47	0.07	−15.4
Expanded polystyrene	1.61	1.49	0.02	−12.0

^aIn situ *R*—RSI, 100/RSI.

Figure 5 shows the heat flux through the three floor sections monitored with the PFMs. The variation in heat flux displayed a daily cycle as the ambient temperature rose and fell.

The top curve in Fig. 5 shows that the largest heat flux was through the uninsulated floor area. The bottom curve shows the polyester-insulated floor section had the smallest heat flux. These are consistent with the relative thermal resistances for the assemblies.

The daily cycling of in situ *R* values is shown in Fig. 6. The thermal resistance of the uninsulated floor was much less than the polyester and expanded polystyrene-insulated floors. In situ *R* values for the three floor sections were calculated using the heat-flux data shown in Fig. 5 and the corresponding temperature differences.

FIG. 4 Temperature difference across three floor sections (NI, no insulation; PE, polyester; EPS, expanded polystyrene).

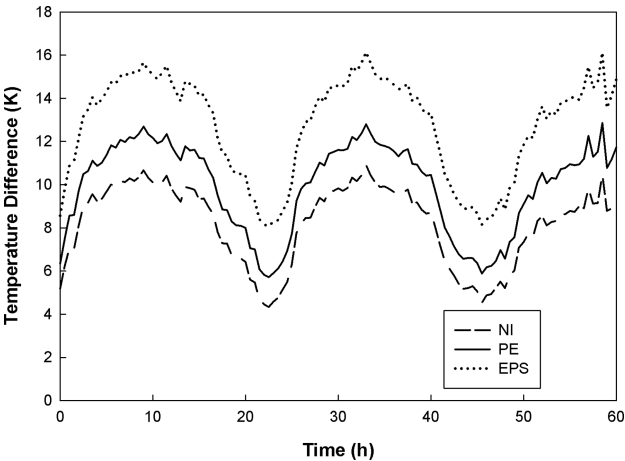
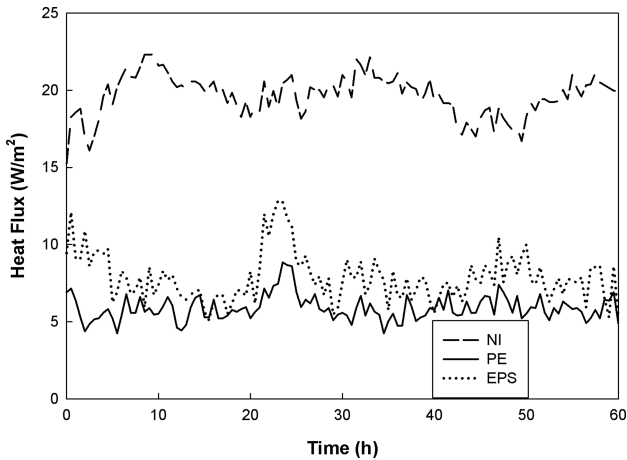


FIG. 5 Monitoring of heat flux over 60h period.



Each point shown in Figs. 4, 5, and 6 is the simple average of 10 readings taken in rapid succession by the PFMs at 3-min intervals. These averages were taken to be instantaneous R values. The average of the in situ R -value data collected during the 60 h test period is shown in Fig. 7.

FIG. 6 In situ R values for the three floor sections.

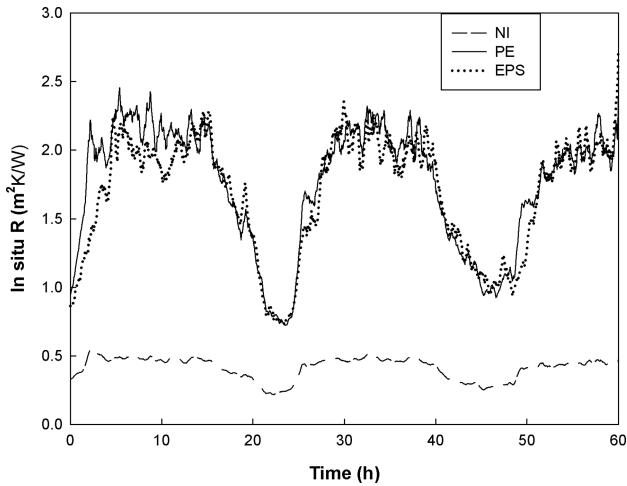
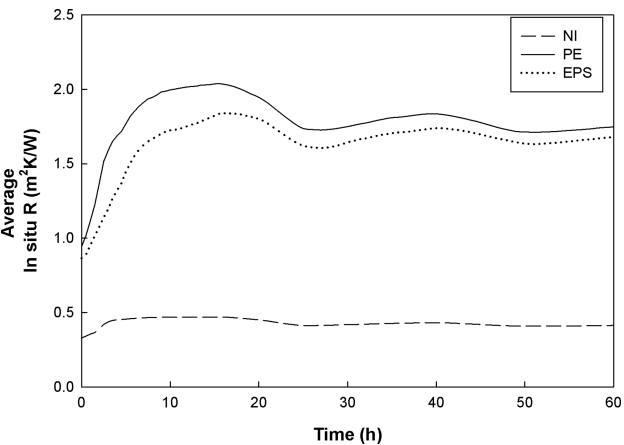


FIG. 7 Average in situ R value.



The results for in situ R values were computed using Eq 1

$$\begin{aligned} R_{\text{in situ}} &= (1/t) \cdot \int_0^t (\Delta T(\tau)/Q(\tau)) d\tau \approx (1/t) \sum_{j=1}^n (\Delta T/Q)_j \Delta t \\ &\approx (\Delta t/t) \cdot \sum_{j=1}^n R_j \\ &\approx (1/n) \cdot \sum_{j=1}^n R_j \end{aligned} \tag{1}$$

where:

$\Delta T(\tau)$ is the surface-to-surface temperature difference,

$Q(\tau)$ is the heat flux, and

ΔT and Q depend on time, τ .

In situ R values and standard deviations for the three floor sections are contained in Table 3 along with the estimated steady-state section R values.

TABLE 3 Summary of floor section thermal data.

Floor Insulation	RSI	In Situ R	In Situ R Standard Deviation	In Situ Heat Flux	In Situ Heat Flux Standard Deviation
None	0.14	0.40	0.02	19.72	0.56
Polyester	2.16	1.75	0.17	5.87	0.25
EPS	1.58	1.68	0.18	7.94	0.62

The RSI in Table 3 includes the 20-mm-thick medium density floor boards ($0.14 \text{ m}^2 \cdot \text{K/W}$ from the New Zealand Standard 4214, 2006) [7]. RSI for polyester and polystyrene were computed using the apparent thermal conductivity data in Table 1. Columns 3 and 4 show the measured in situ R values and standard deviations obtained from the PFM.

Discussion

The electrical potential that is produced when a temperature difference exists across a bismuth-telluride Peltier module is directly proportional to the heat flux [4]. Analysis of this relationship suggested that Peltier modules could be used as reliable, low-cost heat-flux transducers.

STEADY-STATE TESTS

The use of a controlled temperature chamber as described in this paper was intended to approximate the laboratory conditions recommended in ASTM C1046 [2]. To be useful, a field instrument must return measurements that match the actual value of the quantity being measured. Figures 1 through 3 provide a visual representation of the PFM output. The data summarized in Table 2 show that, under steady-state conditions, the PFM measurements were within 15 % of the RSI from the laboratory (Table 1). This comparison shows that, under these fixed conditions, the PFM data provides reasonable estimates that trend toward the thermal properties determined in the laboratory. One way to improve precision could be to use additional temperature sensors to achieve better certainty in the temperature difference. Taking the hysteresis of the temperature controllers ($\pm 0.5^\circ$) into account, it is likely that an even more stable signal is obtainable with more stringent temperature control. Also, improved accuracy could be achieved by careful adherence to the procedures called by ASTM C1130 [8] for thin heat-flux transducer calibration. However, the PFM concept is intended for use in the field to gauge in situ thermal performance over time. It is not intended to replace results from laboratory instrumentation currently used to measure the thermal resistance of building materials. Although it is important that the PFM results are comparable to RSI, it is also important to appreciate the potential applications to dynamic analysis of thermal performance. Rather than focusing on a single fixed resistance measure, the PFM can be used to gauge real-time changes in heat flux. The flow of heat into and out of buildings is, after all, a major if not the main concern with respect to energy utilization.

IN SITU TESTS

As indicated above, an occupied residence was fitted with two types of under-floor insulation: expanded polystyrene and polyester fiber blanket. Heat-flux and thermal resistance data were collected over 3 days totaling 60 h of continuous data logging. The in situ heat flux and in situ R value was strongly dependent on the diurnal

shifts in temperature that occurred during the course of this trial (Figs. 4 and 5). Overall, the insulated floor section heat flux was about 1/4 that of the uninsulated floor.

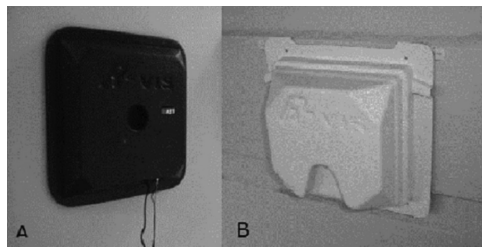
Many factors can affect the heat flux and temperature difference. The thermal mass of the building materials, large indoor and outdoor temperature fluctuations, small indoor–outdoor temperature differences, and the intrinsic thermal resistance of the insulation materials installed cause deviations from the laboratory-derived R value.

The curves in Figs. 4 through 6 illustrate the dynamic range of measurements obtained using the PFM in situ. Although averaging the thermal data (Table 3) will obtain results that trend toward the steady-state condition of a given building system as indicated in Fig. 7, monitoring the heat flux provides data that reflects the dynamics of heat flow under actual conditions. Heat flow can be significantly affected by the thermal mass of a building assembly. A large thermal mass tends to buffer changes in heat flux. Buffering causes a lag between changes in temperature and changes in heat flux. The consequence of this lag or phase shift is illustrated by the wide range of in situ R value shown in Fig. 6. The RSI is a material property that differs from in situ R value that changes value constantly. Heat flux, however, is expected to change and is directly linked to energy use. The PFM would be useful to assess the impact of thermal mass and other environmental factors on the flow of heat across the building envelope and contributes to assessment of energy efficiency.

PORTABLE HEAT-FLUX METER

The PFM is convenient to use because of the self-supporting feature of the inside module, and the serial output from the microprocessor is “spreadsheet ready” either by importation from a text file or as constantly updated data readable from the LED display. The inside module is self-supporting because a fan is used to make a small pressure difference between the inside and outside of the plastic shell. Figure 8 shows the modules as they would appear in use. The black module in Fig. 8 should be placed against the inside surface and the white module in Fig. 8 goes on

FIG. 8 Portable flux meter (A) inside module, and (B) outside module.



the outside surface. The inside module is 400 by 400 mm and 75 mm deep. The outside module is 200 by 200 mm and 75 mm deep.

The lightweight outside module facilitates installation with any reversible adhesive material. Data logging begins automatically once power is turned on from a 12 V wall transformer (or an onboard battery pack in the case of the outside module). This instrument is not intended to be used for product labeling or product classification. Rather it is intended as a useful method of determining heat flux and thermal performance “in the field.” Calls for in situ determinations have been made over the past 20+ years. Quoting Goss and Miller [9], “There is a need to develop a portable hot box or heat flow meter test method that can be used to make in situ measurements on the thermal resistance on field installed reflective insulation products. The results of in situ testing could be used to compare with laboratory hot box results and should give some indication of the need for good workmanship in installing these products.”

Although the PFM applications presented here develop the concept of in situ determinations of thermal performance, it should be used in combination with existing building assessment protocols. Quoting from ASTM C1046, 1.2 [2], “Use infrared thermography with this technique to locate appropriate sites for HFTs.”

Summary

A novel portable flux meter apparatus has been described in this paper. The PFM was used to measure three assembly R values with results that are within 20 % of the R values determined using conventional laboratory equipment. The PFM was also used to study heat flow through three floor sections with different levels of thermal resistance. The PFM provides a record of heat flux through the assembly and yields a value for an in situ R value for the region tested. This instrument may be used to determine the effect of environmental factors (moisture and air ingress, installation practices, biological damage, and other influences on thermal resistance) on in situ heat flux through building systems and elements.

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Nitin Shukla,¹ Devendra Kumar,¹ Diana Elliott,¹
and Jan Kosny¹

Moisture Content Measurements in Wood and Wood-Based Materials—Advancements in Sensor Calibration and Low-Moisture-Content Regime

Reference

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ABSTRACT

Wood is a major structural material used in the construction of residential and commercial buildings. In situ and continuous measurement of moisture in a wood element of the building envelope is critical in preventing the occurrence of moisture related damages to the building structure, and also helps in determining the thermal and energy performance of the system. Moisture content of a wood assembly may serve as an indicator of the effectiveness and potential condensation-related damage to the adjoining thermal insulation layers in the building envelope. Typically, the moisture content in a wood component is determined by measuring the electrical resistance across a pair of metallic pin sensors, with correlation or calibration between resistance and moisture content already established for the studied wood species. As the wood dries from fiber saturation point (FSP) to equilibrium moisture content (EMC), the resistance typically increases by 4 orders of magnitude from hundreds of k Ω to G Ω . Typical

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¹Fraunhofer Center for Sustainable Energy Systems, Boston, MA 02130, United States of America.

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resistance measurement instrumentation schemes such as voltage divider and multimeters are often incapable to cover this wide dynamic range of resistance. To further complicate the measurements, low EMC values of 5 %–10 % range correspond to very high resistances in the range of $M\Omega$ – $G\Omega$, a regime that has remained challenging to measure because of lower current and leakage issues. To circumvent these issues, we developed an instrumentation methodology based on a simple voltage divider circuit in combination with a data-logger with reference resistors selected in such a manner to maximize the dynamic range of the measurement with sufficient accuracy and resolution for higher resistance range. The results showed significant improvements in the dynamic range and resolution for the MC measurements.

Keywords

moisture content, wood and wood-based materials, moisture measurement methods

Introduction

Wood has an affinity for water, i.e., it is hygroscopic in nature. The amount of water, i.e., moisture content (MC) in wood depends on several factors such as temperature and relative humidity of the surrounding air. Several of the physical properties of the wood such as mechanical, thermal, and electrical properties are strongly dependent on the moisture content of wood [1]. In building applications, an increased level of MC in wood elements is highly undesirable as it adversely affects the structural strength and thermal insulating property of the element. Moisture content of a wood assembly may serve as an indicator of the effectiveness and potential condensation-related damage to the adjoining thermal insulation layers in the building envelope. In addition, a high level of MC causes health and safety issues to the building occupant [2]. Accurate measurement of MC in a wood element is critical in monitoring the performance and health of a building. Furthermore, an accurate knowledge of MC within wood structures of the building is needed as input in hygrothermal and building energy computer models to predict the moisture and energy performance of the building.

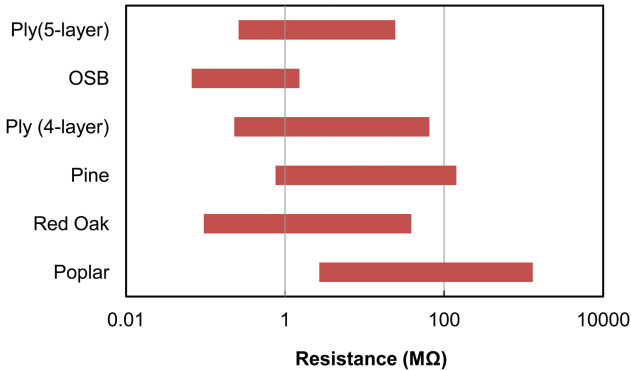
There are several commercial and lab-scale methods available to measure MC in wood samples [3–6], among which electric resistance based method is one of the most popular methods because of its simple and inexpensive design [7–10]. The method relies on the fact that the electrical resistance of wood is a decreasing function of MC. A pair of metal electrodes (pin, nail, or screw) that is inserted into the wood specimen is used as a sensor to measure the electrical resistance of the wood. A relationship between the electrical resistance and the true MC is pre-established for a wide range of MC under controlled laboratory conditions. This calibration is used to convert measured resistance during a field experiment into MC values. In a typical field experiment, a simple potential divider circuit, along with a data-logger is used to continuously record the data.

Because wood is an excellent electrical insulator while water is a moderate conductor, it is expected that introduction of water in wood would significantly decrease the electrical resistance of the wood sample. A previous research study conducted by authors showed that the electrical resistance across a pair of metallic screws spaced 1" apart increased by almost 2–3 orders of magnitude as MC decreased from the fiber saturation point (FSP) of MC ~0.3 [1] to commonly observed equilibrium moisture content (EMC) of MC ~0.1 [11]. Figure 1 provides the range of resistance for six different samples of wood and wood-based specimen as MC decreases from approximately 0.3 to 0.1. For higher MC region, the measured resistance lies in the range of kΩ to MΩ, a range that can be measured accurately using simple electrical circuits such as potential divider and voltage multimeter. However, as we approach lower MC region of 0.1 and below, the resistance is found to be in the range of high MΩ to GΩ, a range that is increasingly difficult to measure using before-mentioned methods [12,13]. In this paper, we develop a simple and inexpensive instrumentation scheme to measure resistance in MΩ–GΩ range, allowing accurate determination of low MC levels in the wood specimens.

Experimental Setup

Typically, a combination of a bridge circuit and a data-logger is used for the continuous resistance measurements. A Campbell Scientific CR1000 data-logger, consisting of a measurement and control module, and a wiring panel, was used for the purpose of this study. This data-logger module requires an external keyboard/display and power supply. The CR1000 provides reliable measurements as it suspends execution whenever the primary power drops below 9.6 V [14]. In a

FIG. 1 Observed range of electrical resistance as MC decreases from 0.3 to 0.1 for six different species of wood and wood products. A pair of metallic screws is inserted into the specimen to measure the resistance.



traditional measurement approach, a three-wire half-bridge terminal-input module is connected directly to the data-logger's input terminals to provide completion to resistive bridge measurements, voltage dividers, and precision current shunts.

We investigated two measurement circuits in combination with CR1000 data-logger.

Conventional Voltage Divider Circuit

A typical voltage divider is a linear circuit that divides input voltage among the components of the circuit. A simple voltage divider may be created by connecting two electrical impedances in series. The impedance may consist of any combination of elements such as resistors, capacitances, or inductors. In our case, impedance is in the form of resistance. The conventional circuit used for MC measurements contains two reference resistances, Z_1 and Z_2 , with the resistance of interest, i.e., load resistance, Z_L , applied across Z_2 . A schematic of the circuit is depicted in Fig. 2. Using simple circuit theory, a relationship between Z_L and output voltage, V_{out} , may be derived:

$$(1) \quad Z_L = \frac{Z_1 * Z_2}{\left(\left(Z_2 * \frac{V_{in}}{V_{out}} \right) - (Z_1 + Z_2) \right)}$$

Equation 1 is used to calculate the magnitude of V_{out} as Z_L is varied between $k\Omega$ and $G\Omega$, corresponding to high and low MC regimes. Figure 3 shows the theoretical predictions for V_{out} for several different ratios of Z_1 and Z_2 with Z_2 assumed to be

FIG. 2 Electrical circuit diagram for a typical potential divider. A parallel combination of the load (Z_L) and a reference (Z_{REF}) resistances is placed in series with another reference resistor. Output voltage (V_{out}) is measured across the load resistance as an input voltage (V_{in}) is supplied to circuit.

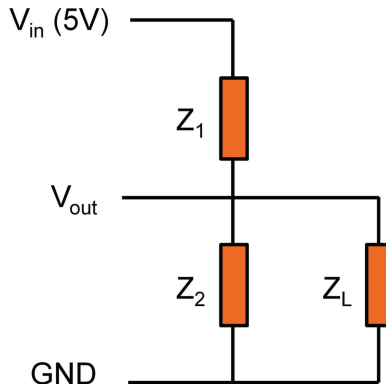
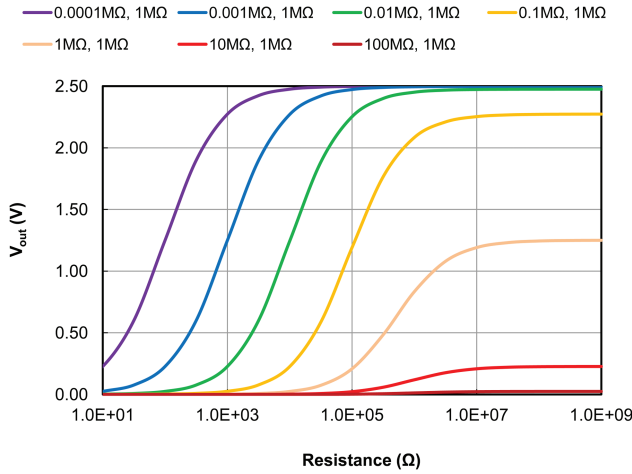


FIG. 3 Calculated output voltage, V_{out} , across load resistance, Z_L , using a conventional circuit with two reference resistors. Several combinations of resistances Z_1 and Z_2 are investigated. An input voltage, V_{in} , of 2.5 V is applied to the voltage divider circuit.



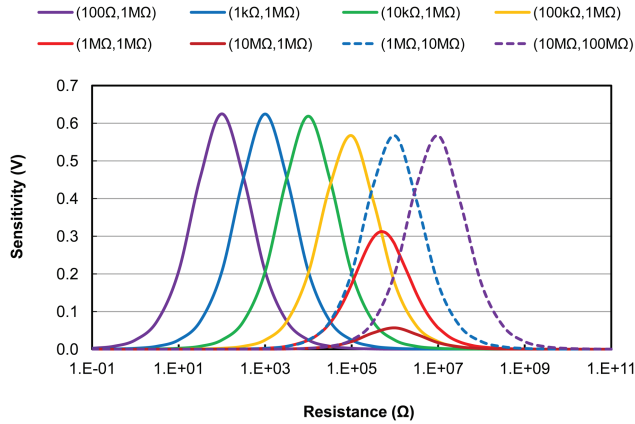
1 M Ω for all cases. V_{in} is assumed to be 2.5 V in all these calculations considering CR1000 data-logger supplies an input voltage of 2.5 V to the potential divider circuit. In general it is found that the curve shifts to higher load resistances as Z_1 is increased. V_{out} is sensitive to a load resistance range that varies by four decades. Full scale in voltage output is obtained as long as Z_1 is 2 orders lower than Z_2 . However, as Z_1 becomes comparable and larger than Z_2 , there is a loss in full-scale voltage. In fact, as Z_1 becomes 2 order higher than Z_2 , i.e., 100 M Ω , there is a negligible change in V_{out} value.

To determine the load resistance range in which this circuit will be useful, we calculated the sensitivity of V_{out} as a function of load resistance. Sensitivity may be defined as the change in the output voltage as a function of unit change in the load resistance. The greater the change in the voltage, the greater is the sensitivity. A higher sensitivity is desired to measure higher resistances with greater accuracy. Taking a derivative on both sides of Eq 1 and rearranging terms, we obtain sensitivity, S :

$$(2) \quad S = -\frac{\partial V_{out}}{\partial Z_L} = \frac{V_{in} Z_1 Z_2^2 Z_L}{(Z_1 Z_2 + Z_1 Z_L + Z_2 Z_L)^2}$$

A plot of S as a function of Z_L (on logarithmic scale) is shown in Fig. 4 for various combinations of Z_1 and Z_2 . For each combination, sensitivity is characterized by a

FIG. 4 Calculated sensitivity, S (volts), as a function of load resistance, Z_L , using a conventional circuit with two reference resistors. Several combinations of resistances Z_1 and Z_2 are investigated. An input voltage, V_{in} , of 2.5 V is applied to the voltage divider circuit.



symmetrical shape around a peak and falling off toward zero while moving away from peak. The peak of the sensitivity curve shifts to higher load resistance and decreases in magnitude as the magnitude of Z_1 increases. The decrease in the magnitude is more apparent as Z_1 becomes comparable and larger than Z_2 . For example, peak of the sensitivity changes from 0.625 V to 0.3125 V as Z_1 is changed from 100 Ω to 1 M Ω (i.e., a 4-order of magnitude increase), while it reduces significantly to 0.057 as Z_1 is decreased to 10 M Ω (a factor of 6 reduction as Z_1 is decreased by 1 order of magnitude).

The sensitivity analysis shows that any desired range of load resistances can be covered by this type of circuit by a proper selection of (Z_1, Z_2) combination. We looked at three different (Z_1, Z_2) combinations of (1 M Ω , 1 M Ω), (1 M Ω , 10 M Ω), and (10 M Ω , 100 M Ω).

Theoretical prediction for V_{out} is shown in Fig. 5. We find that (1 M Ω , 10 M Ω) and (10 M Ω , 100 M Ω) combinations provide almost full-scale voltage sensitivity in measurement of V_{in} (2.5 V in our case) in resistance ranges of 0.1–10 M Ω and 1–100 M Ω , respectively. It can be inferred that (10 M Ω , 100 M Ω) combination will be more suitable if low MC regime is of interest, although this combination will lack sufficient sensitivity for higher MC regions. These combinations of resistances were built using three-wire bridge modules, and a CR1000 data-logger was used to measure the voltage output for load resistances ranging between k Ω and G Ω . Results as shown in Fig. 5 show very good agreement between the theoretical predictions and the experiments. These experiments demonstrate that (Z_1, Z_2) combinations may be designed to maximize the sensitivity in a given resistance range.

FIG. 5 Calculated and measured output voltage, V_{out} , across load resistance, Z_L , using a conventional circuit with two reference resistors. Three combinations of reference resistors ($1\text{ M}\Omega$, $1\text{ M}\Omega$), ($1\text{ M}\Omega$, $10\text{ M}\Omega$), and ($10\text{ M}\Omega$, $100\text{ M}\Omega$) are investigated. An input voltage, V_{in} , of 2.5 V is applied to the voltage divider circuit.

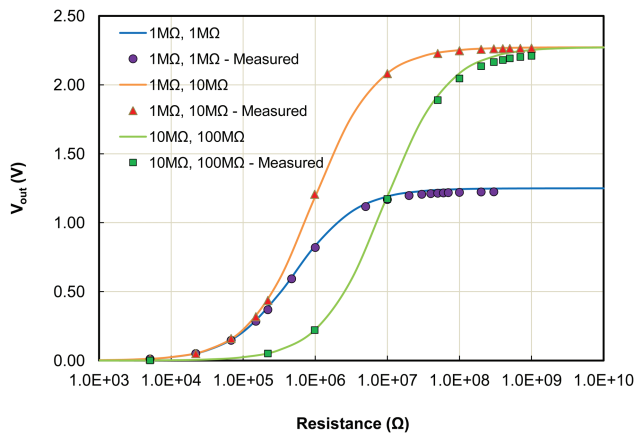
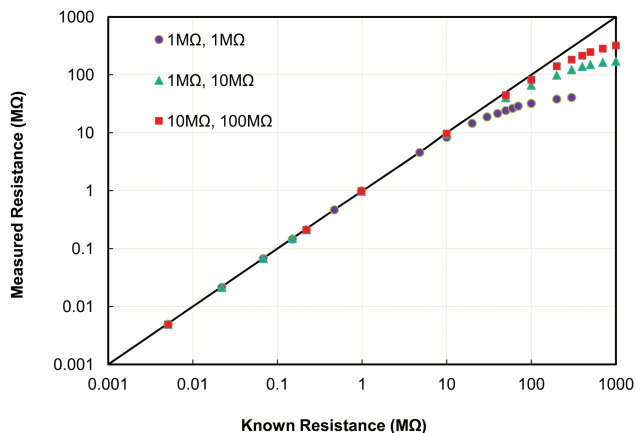


FIG. 6 Resistance measured using a conventional circuit with two reference resistors. Three combinations of reference resistors ($1\text{ M}\Omega$, $1\text{ M}\Omega$), ($1\text{ M}\Omega$, $10\text{ M}\Omega$), and ($10\text{ M}\Omega$, $100\text{ M}\Omega$) are used to measure a known value resistor.



Next, resistors of known values were tested with this potential divider circuit method. Figure 6 shows results for different combinations of (Z_1 , Z_2). As expected from theoretical predictions (1 M Ω , 1 M Ω) combination begins to show significant errors above 1 M Ω value. On the other hand (10 M Ω , 100 M Ω) combination displayed accurate results until 100 M Ω , with an error of approximately 18 % and 70 % at 100 M Ω and 1 G Ω load resistances, respectively. As a comparison (1 M Ω , 1 M Ω) and (1 M Ω , 10 M Ω) circuits produced an error of $\sim$ 68 % and 34 %, respectively, when measuring a 100 M Ω resistor.

It is to be noted that for the lowest resistor of 5.1 k Ω , the three circuits: (1 M Ω , 1 M Ω), (1 M Ω , 10 M Ω), and (10 M Ω , 100 M Ω) showed errors of $\sim$ 3 %, 3 %, and 4 %, respectively. MC of interest in most field monitoring experiments correspond to tens of k Ω to G Ω resistances, i.e., 5 to 6 orders of variation in magnitude. Theoretically, it is possible to use higher resistance combinations such as (100 M Ω , 1 G Ω) if the goal is to measure high resistances of G Ω . However, such combinations have disadvantage that it will increase the lower range of the measurement, i.e., resistances in k Ω range may not be accurately measured. In addition, such high resistance combination will result in current leakage losses, reducing the accuracy of the measurements. Therefore, this circuit may not be able to cover the whole range of MC of interest. Although two separate three-wire circuits may be designed to cover the range of interest; however, this will also double the instrumentation and data analysis needs.

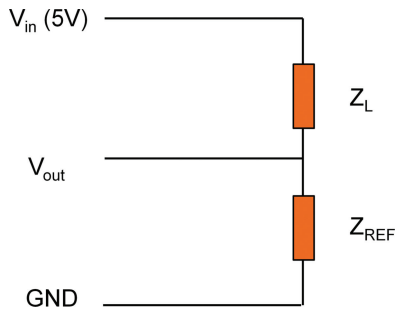
New Voltage Divider Circuit

The study performed on the conventional circuit showed that this type of commonly used potential divider is not suitable for the wood resistance measurement if goal is to cover both the low and high MC regions using a single sensor. We developed a new electrical circuit that provided much improved measurement sensitivity and accuracy. The new instrumentation scheme required changes to the data-logger program as well. The diagram of the new circuit is shown in Fig. 7. In this scheme, a continuous supply of 5 V is provided to the data-logger, and the program makes a single-ended voltage measurement on one or more analog channels. The resistive load is applied between high and low terminals, while a fixed reference resistance is connected between the low and ground terminals. Voltage measurements are performed across the reference resistance, and load resistance may be calculated as follows:

$$(3) \quad Z_L = Z_{\text{ref}} * \frac{(V_{\text{in}} - V_{\text{out}})}{V_{\text{out}}}$$

Theoretical predictions for V_{out} as a function of Z_L for various Z_{ref} are given in Fig. 8. It can be seen that full scale in voltage is obtained for the new circuit, implying improved measurement sensitivity. As Z_{ref} increases, V_{out} profile remains similar although it shifts to higher load resistances. We experimented with three

FIG. 7 Schematic of new voltage divider circuit. A load (Z_L) and fixed reference (Z_{ref}) resistances are placed in series, while output voltage (V_{out}) is measured across the reference resistance. An input voltage (V_{in}) of 5 V is supplied in the experiments.



different reference resistances: 59 k Ω , 150 k Ω , and 1 M Ω . Figure 9 shows theoretical prediction and measured V_{out} as a function of Z_L for the three reference resistance cases. Measured voltage matches very well with the predictions with a full scale of 5 V in voltage measurements for all three cases.

Sensitivity in V_{out} measurements with a differential change in load resistance may be calculated by taking derivative of Eq 3 on both sides:

FIG. 8 Calculated output voltage, V_{out} , across load resistance, Z_L , using the new circuit with one reference resistor. Relationships are obtained for several different reference resistances. An input voltage, V_{in} , of 5 V is applied to the voltage divider circuit.

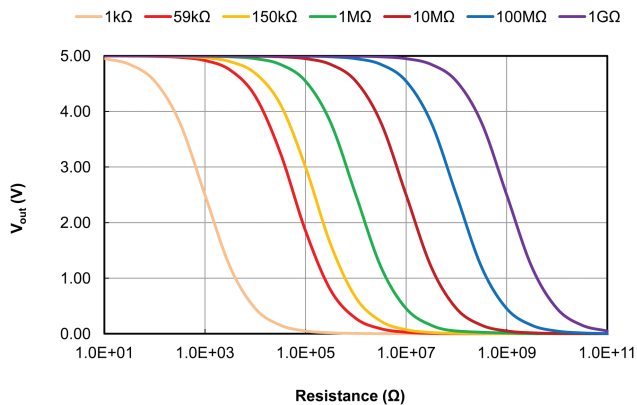
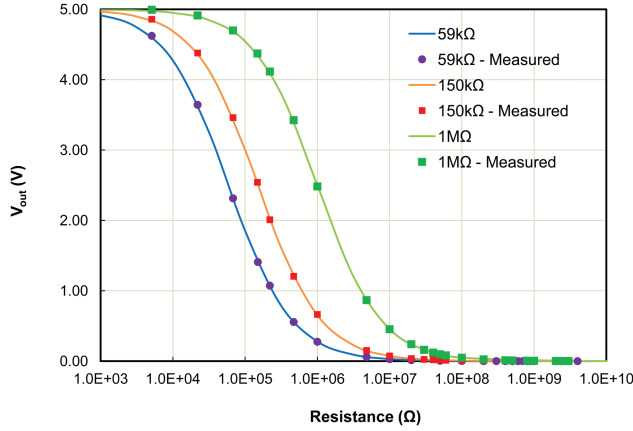


FIG. 9 Calculated and measured output voltage, V_{out} , across load resistance, Z_L , using the new circuit with one reference resistor. Relationships are obtained for three reference resistors: 59 k Ω , 150 k Ω , and 1 M Ω . An input voltage, V_{in} , of 5 V is applied to the voltage divider circuit.

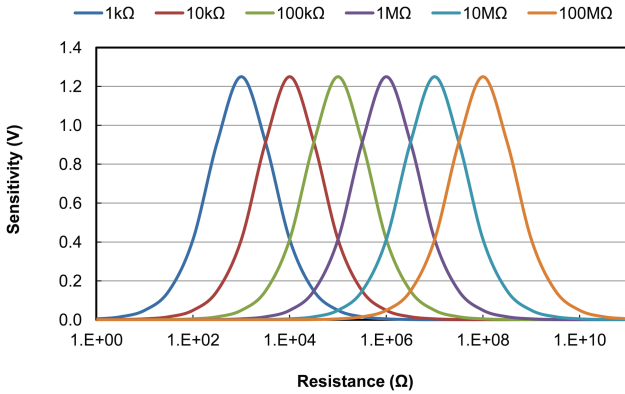


$$(4) \quad S = -\frac{\partial V_{out}}{\partial Z_L} = \frac{V_{in} Z_{ref} Z_L}{(Z_{ref} + Z_L) Z_L}$$

Sensitivity results as given in Fig. 10 indicate that magnitude of peak of sensitivity curve remains constant irrespective of the Z_{ref} . The sensitivity of 1.25 V for new circuit is 4 times larger than the conventional circuit using (1 M Ω , 1 M Ω) combination (Fig. 10). We also find that peak occurs at a load resistance equivalent to Z_{ref} . This new circuit offers a straightforward way to tune to the desired range of load resistance by selecting an appropriate reference resistor.

For both the conventional and new circuits, we may define a full width at half maximum (FWHM) as the distance between resistance points where sensitivity value is half of the peak. FWHM provides a way to compare the bandwidth or dynamic range of the resistance that can be measured with the circuit. We find that FWHM for both circuits is ~ 1.5 on log scale, suggesting that both circuits will measure the similar decades in the resistance. It is to be noted that in the new circuit, V_{out} transitions from 5 V to 0 V as load varies across the dynamic range as compared to the old circuit where V_{out} changes from 0 V to 2.5 V. Because basic resolution of data-logger becomes larger with the voltage magnitude range, it implies that the new circuit has improved resolution in voltage measurements toward the higher end of the dynamic resistance range. For example, CR1000 has basic resolutions of 6.67, 667, and 1333 μV for ± 2.5 , ± 2500 , and ± 5000 μV voltage ranges, respectively. In principle, voltage can be measured for resistances as long as the sensitivity

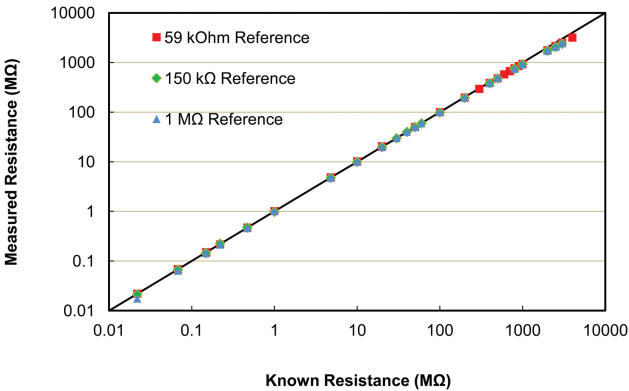
FIG. 10 Calculated sensitivity, S (volts), across load resistance, Z_L , using the new circuit with one reference resistor. Relationships are obtained for several different reference resistors. An input voltage, V_{in} , of 5 V is applied to the voltage divider circuit.



is above the resolution value. This implies that for the conventional circuit, resolution is 6.67 and 667 μV for lower and upper sides of the dynamic resistance range, respectively. Using Eq 2, we find that this corresponds to a measurable range of 0.4 Ω to 800 $\text{M}\Omega$. On the other hand, for new circuit, resolution is 1333 and 6.67 μV for lower and upper sides of the dynamic range, respectively. Using Eq 4, we obtain a corresponding measurable range of 250 Ω to 5000 $\text{G}\Omega$. This analysis shows that both circuits are capable to measure a dynamic resistance range that changes by 9–10 decades. However, upper limit of measurable resistance is much higher for the case of new circuit than the old circuit. In theory, the new circuit should be able to cover the whole range of $\text{k}\Omega$ to $\text{M}\Omega$ resistances corresponding to high and low MC.

Resistors of known values are measured using the new circuit and results are presented in Fig. 11. We observe an excellent accuracy in the measurement for load resistance range of $\text{k}\Omega$ to $\text{G}\Omega$ for all the three cases investigated. For all the three circuits, there was less than 1 % and 9 % errors at 100 $\text{M}\Omega$ and 1 $\text{G}\Omega$, respectively, showing usefulness of the new circuit for low MC measurements. Even for ultra high resistance of 3 $\text{G}\Omega$, we obtain a maximum error of 20 %. This shows that the new circuit is able to measure resistance even in high $\text{G}\Omega$ range, once the error values in resistance measurement is established in this range. Although the same level of accuracy is obtained in high $\text{G}\Omega$ regions for all three reference resistor cases, the output voltage signal was found to be on the order of 1 mV for 1 $\text{M}\Omega$ as compared to 0.1 mV for 59 and 150 $\text{k}\Omega$ references. This implies that 1 $\text{M}\Omega$ reference will display larger signal-to-noise ratio (SNR), therefore there is improved accuracy in low MC regimes compared to the other two cases.

FIG. 11 Resistance measured using a conventional circuit with two reference resistors. Three different reference resistors, 59 kΩ, 150 kΩ, and 1 MΩ, are used to measure a known value resistor.



To demonstrate the functioning of newly developed instrumentation scheme, a measurement setup using the newly developed instrumentation scheme was built to measure resistance in two wood-based samples—a plywood and wood specimen. The oven dry method was used to calibrate the resistance with true MC. Each sample was approximately 15 cm × 15 cm in size and 1.25 cm thick. The details of the

FIG. 12 Moisture sensor resistance calibration for the studied plywood. A linear fit is found to correlate resistance with MC for MC below FSP of ~0.3. A linear relation $\log(R) = -22.276 \cdot MC + 10.871$ with $R^2 = 0.9585$ is obtained.

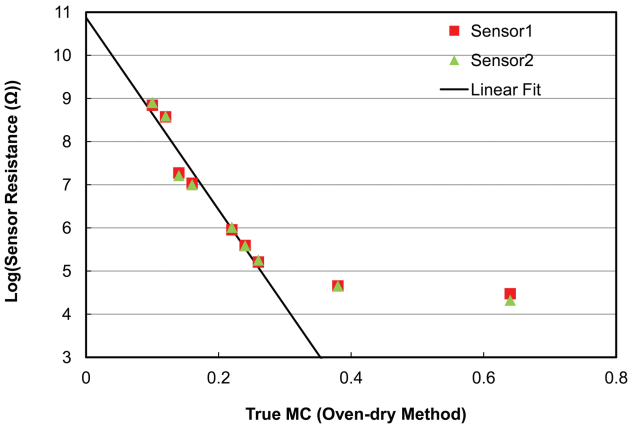
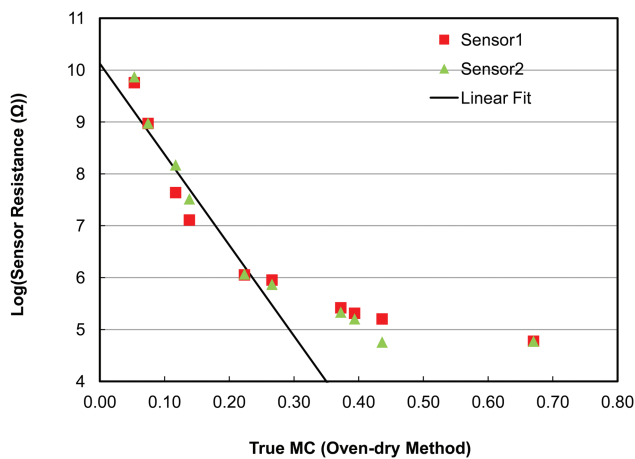


FIG. 13 Moisture sensor resistance calibration for the studied wood specimen. A linear fit is found to correlate resistance with MC for MC below FSP of ~0.3. A linear relation $\log(R) = -17.511x + 10.127$ with $R^2 = 0.9036$ is obtained.



steps involved in sample preparation have been described in a previous publication [11].

In summary, the two samples were completely dried in a Thermotron S-16 climate chamber under a temperature condition of $103^{\circ}\text{C} \pm 2^{\circ}\text{C}$. Once the dry mass was determined, samples were completely submerged in a water tank to take up water. Once MC reached ~0.5–0.7, samples were taken out and allowed to dry in ambient. Two pairs of metallic screws were inserted into each of the wood samples on different locations of the sample to see variability in the results. The sensors were placed along the grain direction of the wood. A reference resistor of $1\text{ M}\Omega$ was used in the circuit for improved accuracy in low MC regions, i.e., high resistance range. As the wood began to dry, the resistance of wood increased as shown in Figs. 12 and 13. Equilibrium moisture content (EMC) in the two samples was found to be ~0.1 and 0.05, corresponding to a resistance of ~800 $\text{M}\Omega$ and 5 $\text{G}\Omega$. For each sample, readings from two sensors were found to match well. The results show that new circuit is capable to accurately measure high resistances in the range of $\text{G}\Omega$ in wood samples. For MC below FSP, log of the resistance appears to linearly change with true MC. A best linear fit is determined for MC data below FSP as shown in Figs. 12 and 13.

Conclusions

The electrical resistance-based method is the most commonly used methods to measure MC under field conditions because of the simple and inexpensive design.

The method involves using a simple potential divider circuit for resistance measurement across a pair of electrical sensors. Traditional potential divider circuit tends to significantly lose accuracy at higher MC corresponding to $M\Omega$ – $G\Omega$ range resistances. In this study, we first investigate a traditional two-reference resistor-based potential divider circuit to understand how different combinations of these two references affect the measured resistance range. It is found that to have a good output voltage sensitivity, the first reference needs to be lower in magnitude than the second reference across which load resistance is applied. A combination of these two references can be designed to tune the resistance measurement range. Three combinations were used in the study: (1 $M\Omega$, 1 $M\Omega$), (1 $M\Omega$, 10 $M\Omega$), and (10 $M\Omega$, 100 $M\Omega$). The measurements showed that all three combinations provided good accuracy in the $k\Omega$ to $M\Omega$ range, corresponding to a higher MC regime. However (1 $M\Omega$, 1 $M\Omega$) and (1 $M\Omega$, 10 $M\Omega$) began to show significant errors for resistances above 1 and 10 $M\Omega$, respectively. A (10 $M\Omega$, 100 $M\Omega$) combination produced the best results with very good accuracy until 100 $M\Omega$.

A new circuit has been developed to be able to measure the resistances in the $M\Omega$ – $G\Omega$ range. In this circuit, the load resistance was put in series with a reference resistance across which voltage was measured. The voltage sensitivity analysis showed that peak of the sensitivity occurs at a load resistance equivalent to the reference resistance. This allows a simple way of tuning the desired dynamic resistance range by adjusting the reference resistance. The magnitude of the voltage sensitivity for the new circuit was found to be almost 4 times higher than the old circuit when both circuits were designed to have peak sensitivity at 1 $M\Omega$. Three references of 59 $k\Omega$, 150 $k\Omega$, and 1 $M\Omega$ were tested with the new circuits. For all the three circuits, there was less than 1 % and 9 % errors at 100 $M\Omega$ and 1 $G\Omega$, respectively, showing the usefulness of the new circuit for low MC measurements.

Finally, two different species of wood were calibrated using the new circuit. Two metallic screw sensors were inserted into each of the sample. The samples were allowed to dry in ambient from an MC level of ~ 0.7 down to EMC. The new circuit allowed the measurement of low MC of as low as 0.05 corresponding to 5 $G\Omega$ resistances. The data from the two sensors were found to be very close to one another. A calibration relation between the resistance and the true MC is determined by fitting the data below FSP.

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Aaron Seitz,¹ Kaushik Biswas,² Kenneth Childs,²
Lawrence Carbary,¹ and Roland Serino,³

High-Performance External Insulation and Finish System Incorporating Vacuum Insulation Panels—Foam Panel Composite and Hot Box Testing

Reference

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ABSTRACT

An exterior building facade system that provides high thermal resistance in a relatively thin profile is proposed. The design concept incorporates insulation modules constructed from vacuum insulation panels (VIPs) within protective thermoplastic foam. These modules can be substituted in place of standard expanded polystyrene panels used in existing exterior insulation and finish systems (EIFSs). In addition to providing some additional thermal resistance, the foam serves to protect the vacuum panels during construction and to provide a surface appropriate for an adhesive joint on both sides of the foam-VIP unit. The Oak Ridge National Laboratory helped optimize the process by measuring the thermal transmission of prototype VIP/foam composites and testing wall configurations in a guarded hot-box apparatus. Two concepts in construction were tested in full-scale hot-box testing in an Oak Ridge National Laboratory

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¹Dow Corning Corporation, P.O. Box 994, Midland, MI 48686-0994, United States of America.

²Oak Ridge National Laboratory, P.O. Box 2008, Oak Ridge, TN 37831, United States of America.

³Dryvitt Systems, Inc., West Warwick, RI 02893, United States of America.

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facility. The first concept was one of maximum flexibility and VIP protection, and the second was one of minimum necessary protection flexibility but maximum practical performance. The major conclusions of the project can be summarized as follows: (1) It may be possible to develop a wall with an overall performance of R30 or greater with multiple VIP arrangements as shown by heat flow meter and heat flux mapping data. (2) The configurations with the highest thermal resistance are those that maximize VIP coverage. (3) A VIP encapsulated in a foam block, of material similar to what is in use today, could be used as a substitute in an EIFS facade system to gain high thermal efficiency in a thin profile. (4) Infrared imaging shows that fumed silica core VIP panels have a higher thermal resistance than foam insulation only, even in punctured sections.

Keywords

EIFS, VIP, heat flow meter, hot box

Introduction

An exterior building facade system that provides high thermal resistance in a relatively thin profile is proposed. The design concept incorporates insulation modules constructed from vacuum insulation panels (VIPs) within protective thermoplastic foam. These modules can be substituted in place of standard expanded polystyrene (EPS) panels used in existing exterior insulation and finish systems (EIFSs). In addition to providing some additional thermal resistance, the foam serves to protect the vacuum panels during construction and to provide a surface appropriate for an adhesive joint on both sides of the foam-VIP unit. Multiple configurations of the composite structure were considered and experimental measurements were sought to provide more information in the final selection.

The Oak Ridge National Laboratory (ORNL) contributed to the process by measuring the thermal transmission of complete wall sections in a guarded hot box and in a natural exposure test facility in Charleston, SC. These tests are rather large in scale and cost, so it was not practical to perform separate wall-scale tests for each of the foam-VIP arrangements under consideration. Therefore, before test wall sections were built, smaller subsections of each candidate arrangement were prepared and tested in a special-purpose heat flux meter (HFM) apparatus. The focus of these tests was to explore the effects of module configuration options such as VIP size, foam type, edge protection size, adhesive impact, and thickness.

Because even a very versatile HFM still cannot account for an entire array of panels and their associated edge effects, a new procedure was developed to map the HFM measurement results onto full-scale wall designs to predict the system's thermal performance.

All of the vacuum panels were prepared using commercial equipment. The barrier material was a laminate of three metalized polyester films and a linear, low-density polyethylene heat-seal layer. The filler material was a powdered form of fumed silica with opacifiers added to reduce radiative heat transfer.

Additionally, two concepts in construction were tested in full-scale hot-box testing in an ORNL facility. The first one was designed with the goal of being most flexible in construction, so it was constructed of multiple small VIP panels and with extra edge protection. This configuration was meant to demonstrate a concept that was robust in terms of protection on the job site, could offer the most size flexibility, and would provide a panel that could be edge trimmed if needed. The second concept was designed with the goal of maximum performance, so it was constructed of the largest practical panels with minimal edge protection that would not be as robust in handling or installation but would offer maximum thermal performance. Other simple tests were conducted (e.g., with and without batt insulation in the cavity), and the effect of a punctured panel on the wall performance was investigated.

Heat Flow Meter Measurement

The heat flow meter apparatus is a secondary measurement method often used to evaluate the thermal conductivity of homogeneous materials. Its typical construction and use are described in ASTM C518 [1]. The two plates, shown in Fig. 1, are carefully controlled at fixed temperatures. As the heat flows from the hot plate and through the test specimen into the cold plate, a small temperature difference across the transducers generates an electrical signal that is proportional to the heat flux through the transducer. For homogeneous specimens with uniform plate temperatures, the heat flux is unidirectional and can therefore be used with Fourier's law to calculate the thermal conductivity of the material.

Most heat flow meter apparatuses use a single transducer on each of the temperature-controlled plates. A special-purpose apparatus has been designed to facilitate the characterization of nonhomogeneous specimens. This apparatus has an array of transducers on both the top and the bottom plates, so that nonuniform heat flux patterns can be measured. The design of this array is shown in Fig. 2. The test produces a map of heat transfer over this collection of 30 (15 top and 15 bottom) 7.5 cm by 7.5 cm (3 in. by 3 in.) transducers. Even with this extensive measurement scheme, it is typically necessary to merge the test measurements with a mathematical model to fully characterize complex specimens. This apparatus has been used to examine VIPs as a part of a refrigerator design project [2–4].

FIG. 1 Conceptual cross-section of a heat flux meter apparatus.

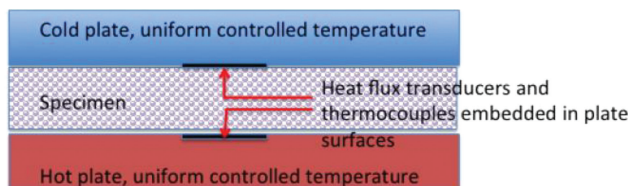
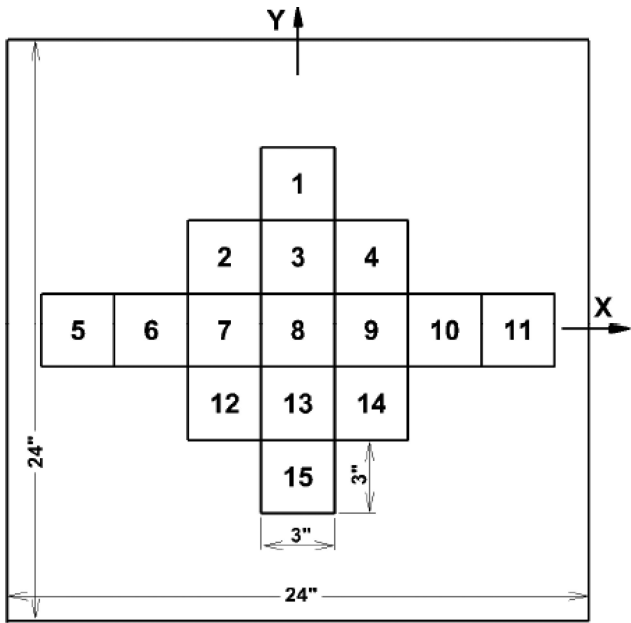


FIG. 2 Heat flux transducer array.

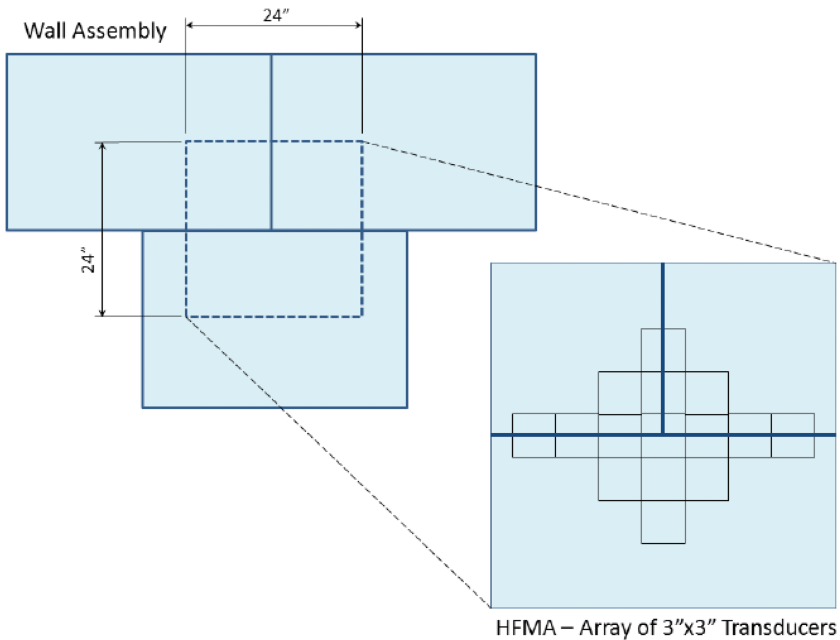


A nonstandard calibration method, similar to that described in ASTM C1667 [5], was used for this project to more accurately measure very small heat flux values. Based on this calibration process, the heat flux values reported here have an extended combined uncertainty between 5 % and 10 % at a 95 % confidence level, with the greater uncertainty corresponding to the lower heat flux values found over the center of a VIP [6].

Description of Heat Flow Meter Apparatus Specimens

In order to use VIPs in a building, it is necessary to protect them from puncture and abrasion during the construction process. In the proposed designs, this is accomplished by encasing the vacuum panels within polystyrene foam insulation. Within this general design framework, there are a large number of possible permutations with regard to the arrangement of panel seams relative to joints where panels are adjacent to each other, the type of foam insulation, the thickness of the foam separation between VIPs, and other possible protective materials. Ten test specimens were prepared to place these variations within the monitored area of the HFM apparatus. Figure 3 demonstrates how these test specimens were representative of panel intersections within a full wall. The joints between VIPs in the test

FIG. 3 Test specimen representing panel joints on a wall.



specimens were located along the lines of central transducers. All of the assemblies covered an area of slightly less than 60 cm by 60 cm (24 in. by 24 in.) and consisted of a layer containing VIPs 2.5 cm (1 in.) thick sandwiched between layers of rigid foam insulation, either EPS or extruded polystyrene (XPS).

The assemblies differed in (1) the number and size of VIPs they contained (one large panel, two half-panels, or one half-panel with two quarter-panels), (2) the type and thickness of the rigid foam insulation layers, and (3) the material adjacent to the edges of the VIPs (Table 1). An example of an assembly construction is illustrated in the exploded view of design option 1 shown in Fig. 4.

Test Data

In the series of tests performed on the 10 VIP assemblies, the bottom plate temperature was set to 35°C and the top plate temperature was set to 12.8°C. Figure 5 shows the measured fluxes from a representative test specimen. The top figure gives the measured flux (W/m^2) for each heat flux transducer (HFT) (transducer) location. The top number for each location is the flux on the upper surface, and the bottom number is the flux on the lower surface. Note that HFT 12 on the bottom plate was inoperable. To aid in the interpretation of the fluxes, the location of each

TABLE 1 Test specimen construction details.

Design Option	Number of VIPs	Material Surrounding VIPs		Layers (From Top to Bottom)			
		Assembly Edges	Between VIPs	1	2	3	4
1	3	0.5 in. XPS	1 in. XPS	1 in. XPS	1 in. VIP	1 in. EPS	–
2	3	0.5 in. XPS	1 in. XPS	1 in. EPS	0.5 in. XPS	1 in. VIP	0.5 in. EPS
3	3	0.5 in. XPS	1 in. XPS	1 in. EPS	0.5 in. XPS	1 in. VIP	0.5 in. XPS
4	3	0.04 in. PVC ^a	0.08 in. PVC ^a	1 in. EPS	1 in. VIP	1 in. EPS	–
5	1	0.5 in. XPS	1 in. XPS	1 in. EPS	1 in. VIP	1 in. XPS	–
6	3	0.5 in. XPS	1 in. XPS	1 in. XPS	1 in. VIP	1 in. XPS	–
8	3	0.5 in. EPS	1 in. XPS	1 in. EPS	1 in. VIP	1 in. EPS	–
10	2	0.5 in. XPS	Butt joint, edge to edge (not flap to flap)	1 in. XPS	1 in. VIP	1 in. XPS	–
11	3	Silicone sealant + gap	Butt joint, edge to edge (not flap to flap)	1 in. EPS	1 in. VIP	1 in. EPS	–
12	3	?	?	1 in. EPS	1 in. VIP	1 in. XPS	–

^aPVC was approximately 50 mm (2 in.) high, covering the side of the VIP and the side of the bottom foam, so that it touched the bottom plate of the apparatus but not the top plate.

VIP in the assembly is also outlined in the figure. The white space represents the material surrounding the VIP(s). Figure 6 contains plots of the heat fluxes along the two centerlines of the transducer array. Uncertainty bounds of 7.5 % are shown on these plots.

FIG. 4 Exploded view of design option 1.

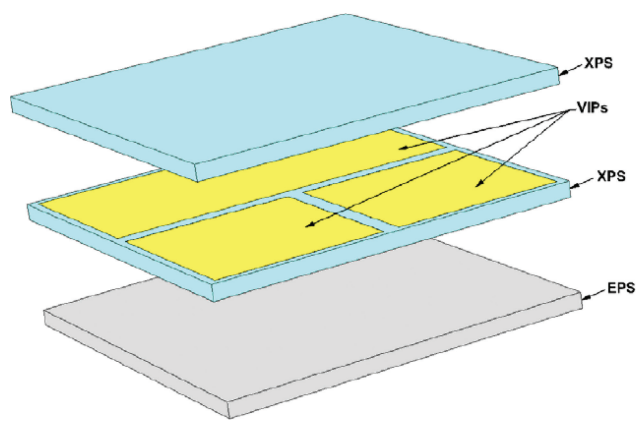
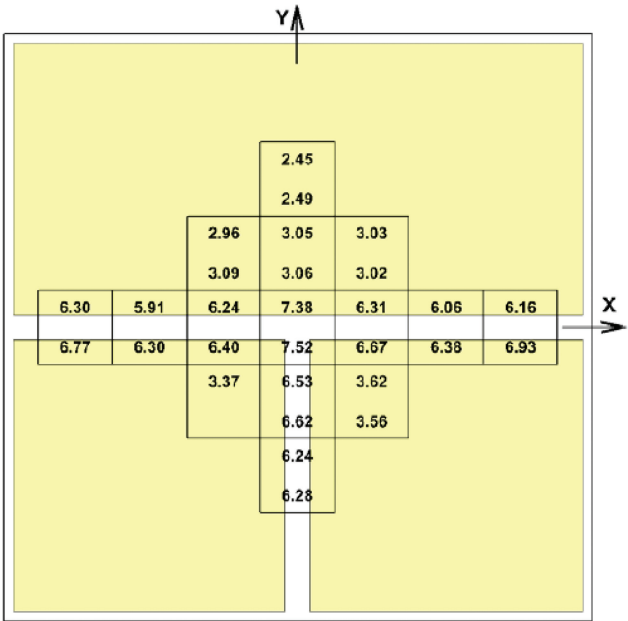


FIG. 5 Design option 1 configuration.



Figures 5 and 6 are illustrative examples of the effect of edge protection on the heat flux through the unit. Figures 7 and 8 show the best-case thermal performance of all the units tested. The full effect of the design configuration is shown in more detail in Refs 7 and 8.

FIG. 6 Design option 1 heat fluxes.

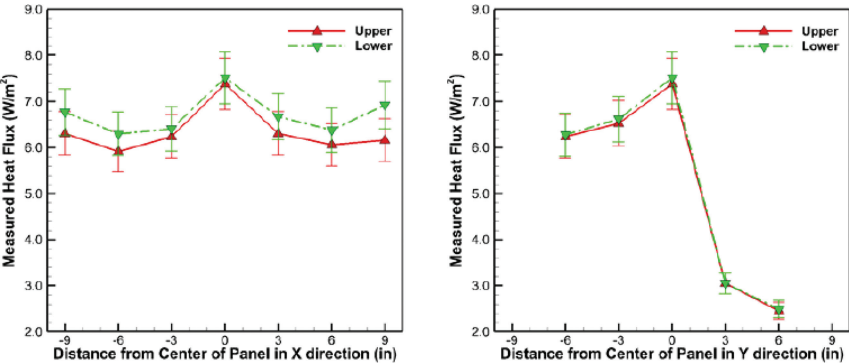
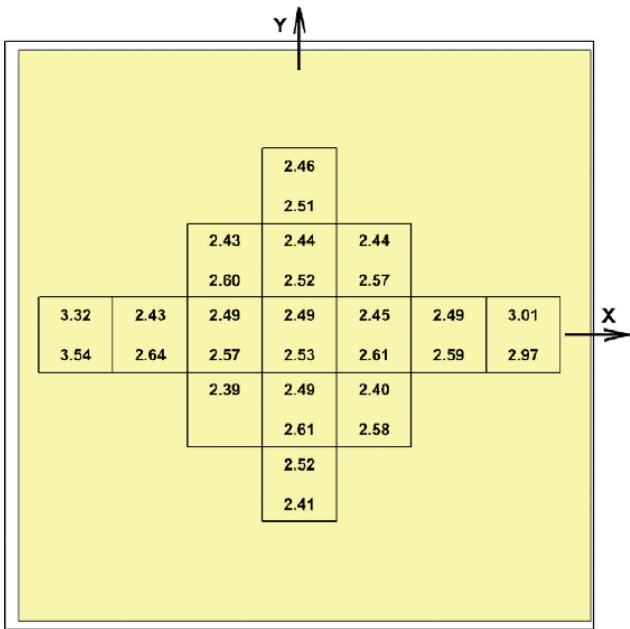


FIG. 7 Design option 5 configuration.



Mapping Heat Flux Tranducer Readings to Full Vacuum Insulation Panel Assemblies

The test specimens were designed so that the junctions between VIPs were placed directly within the metered areas, and specifically with the junctions centered along

FIG. 8 Design option 5 heat fluxes.

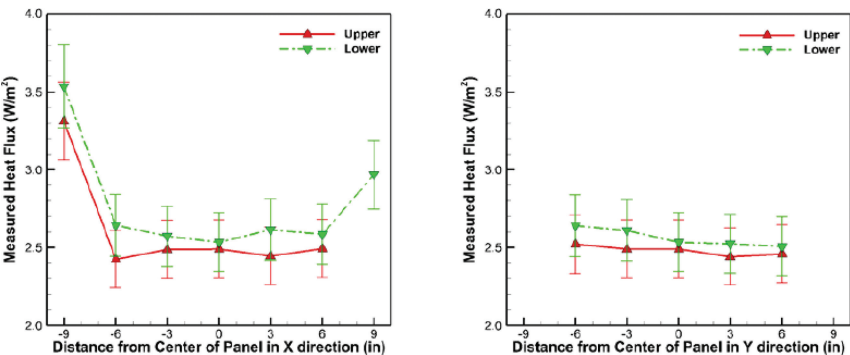
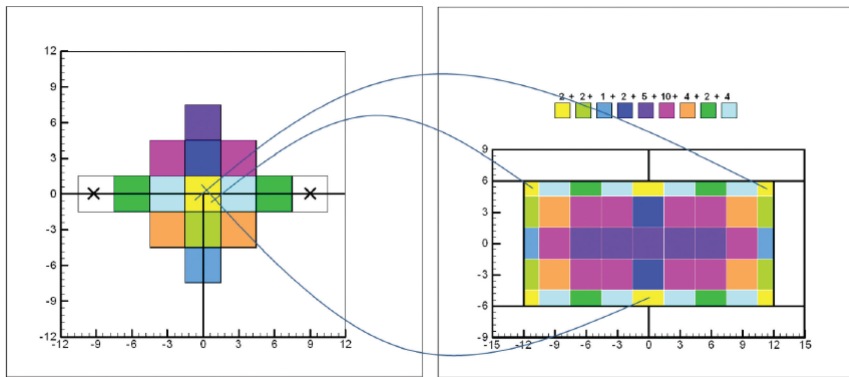


FIG. 9 Mapping heat flux meter data to a staggered array of 12 in. by 24 in. (30 cm by 60 cm) VIP assemblies.



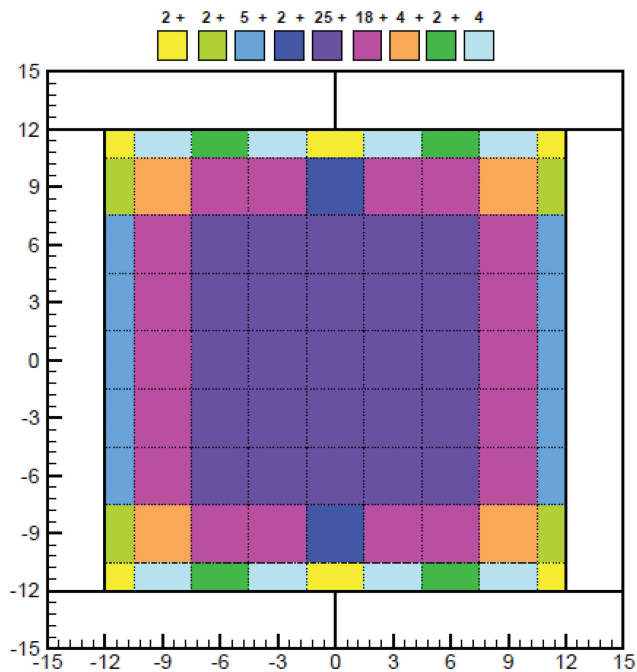
a centerline of the transducers. Each transducer reported the average heat flux over an area of 75 mm by 75 mm (3 in. by 3 in.). The collection of test specimens therefore produced information typical of heat transfer through the center of a panel (purple in the left half of Fig. 9), near the corner of a panel (tan), along a straight junction of panels (blues and greens), and at a junction where two corners come together beside the continuous edge of a third panel (yellow). There is no information for a four-corner junction, but such an arrangement is not anticipated in a wall where the units would be staggered to reduce edge losses. The data from the HFM tests are therefore sufficient to estimate the effective R -value of a VIP-foam panel applied in a staggered array of panels to form a wall. Figures 9 and 10 demonstrate how the HFM readings can be mapped to a 30 cm by 60 cm (12 in. by 24 in.) or a 60 cm by 60 cm (24 in. by 24 in.) panel.

Table 2 gives an estimate of the effective R -value for one of the best-performing configurations, design 4. The heat flux listed is the average of the top and bottom fluxes for the transducer locations listed.

Guarded Hot Box Measurement

ORNL operates and maintains a guarded hot box that is used to measure the thermal resistance (R -value) and thermal transmittance (U -factor) of full-size wall and window assemblies. The box operates under the requirements of ASTM C1363 [9]. A photograph of the RGHB is shown in Fig. 11. Test assemblies are installed in a specimen frame mounted on a moveable dolly. The specimen frame has an aperture 4 m (13 ft) wide by 3 m (10 ft) high. The specimen frame/test assembly is inserted between two “clam-shell” chambers of identical cross-section. The placement of the test wall assembly between the chambers allows the chamber temperatures to be

FIG. 10 Mapping heat flux meter data to a staggered array of 24 in. by 24 in. (60 cm by 60 cm) assemblies.



independently controlled, thus creating a temperature difference across the specimen. The chambers are designated as the climate (cold) and metering/guard (hot) chambers. Figure 12 illustrates a typical wall specimen installed in the test frame. The central 2.4 m by 2.4 m (8 ft by 8 ft) wall section, which aligns with the metering chamber boundary, is used for the actual test.

The surface-to-surface thermal resistance of the wall assembly R_{wall} is calculated as

$$R_{\text{wall}} = A_{\text{wall}} \times (T_{\text{ms}} - T_{\text{cs}}) / Q_{\text{wall}}$$

where:

- Q_{wall} = total energy flow rate through the wall assembly, W,
- R_{wall} = surface-to-surface thermal resistance of the wall assembly, $\text{m}^2 \cdot \text{K}/\text{W}$,
- A_{wall} = area of wall, m^2 ,
- T_{ms} = average metering-side surface temperature, $^{\circ}\text{C}$, and
- T_{cs} = average climate-side surface temperature, $^{\circ}\text{C}$.

The meter-side and climate-side air film coefficients R_{ms} and R_{cs} are calculated as

TABLE 2 Design option 4 mapped heat flux.

Transducer Color Code	Transducer (s)	Average Flux, W/m ²	12 in. × 24 in. Panel Assembly		24 in. × 24 in. Panel Assembly	
			Area Factor	Product	Area Factor	Product
Yellow	8	7.06	2	14.12	2	14.12
Light green	3	6.24	2	12.47	2	12.47
Medium blue	1	5.74	1	5.74	5	28.72
Dark blue	13	2.72	2	5.45	2	5.45
Purple	15	2.39	5	11.95	25	59.77
Magenta	12, 14	2.57	10	25.73	18	46.32
Orange	2, 4	3.23	4	12.91	4	12.91
Medium green	6, 10	5.76	2	11.53	2	11.53
Light blue	7, 9	6.01	4	24.03	4	24.03
Area-weighted average flux, W/m ²				3.87		3.36
Assembly R-value, m ² · K/W				5.74		6.60
Assembly R-value, h ft ² °F/Btu				32.6		37.5

$$R_{msair} = A_{wall} \times (T_{ma} - T_{ms})/Q_{wall}$$
$$R_{csair} = A_{wall} \times (T_{cs} - T_{ca})/Q_{wall}$$

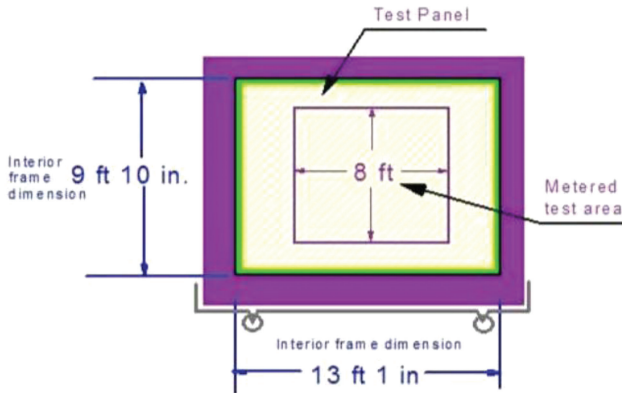
where:

- $R_{ms\ air}$ = thermal resistance of the meter-side air film, m² · K/W,
- $R_{cs\ air}$ = thermal resistance of the climate side air film, m² · K/W,
- T_{ma} = average meter-side air temperature, °C, and

FIG. 11 ASTM C1363 hot box apparatus.



FIG. 12 Schematic of a typical test wall within the hot-box test frame.



T_{ca} = average climate-side air temperature, °C.

The overall air-to-air thermal resistance of the wall assembly R_{uwall} is given by

$$R_{uwall} = R_{wall} + R_{ms\ air} + R_{Cs\ air}$$

Description of Test Walls

Four wall configurations using foam-encapsulated VIPs in a commercially available EIFS system were tested. Supports for all EIFS test walls were constructed from metal studs at 40.6 cm (16 in.) on center, exterior 12.7-mm (0.5-in.) gypsum board, and interior 12.7-mm (0.5-in.) gypsum board. The EIFS system consisted of an air and water barrier troweled onto the exterior gypsum, an adhesive applied in vertical strips to provide a drainage plane and adhere the encapsulated VIP panels, a base-coat and fiberglass mesh applied to the panels, and a finish coat. The exterior insulation consisted of EPS foam blocks 7.6 cm (3 in.) thick and containing vacuum insulated panels 25 mm (1 in.) thick. The walls were allowed to condition for over 28 days at ambient conditions after construction to allow drying of the wall assembly.

In configuration 1 [10], 2 ft by 4 ft (60 cm by 120 cm) foam panels were constructed using 12 in. (30 cm) square VIPs, Type I EPS foam, and silicone adhesive and leaving 1 in. of XPS border protection so that the panels would be edge trimmable. The completed modules were 3 in. (7.5 cm) thick with 1 in. (2.5 cm) comprising the center VIP section. Figure 13 shows the assembly and the configuration of the panels. The net VIP coverage over the wall area was 78 %.

Configuration 2 was the same wall tested in configuration 1, with two VIP panels intentionally vented to measure the effect on the assembly's thermal resistance. After completion of the first test, four puncture holes were drilled in the wall on the

FIG. 13 Assembly of panels used in configurations 1 and 2.



side containing the EPS foam to compromise some of the vacuum panels. Thus, 4 VIPs out of 64 in the test area, or 6.25 %, were compromised. The holes were drilled to be at least 6.35 cm (2.5 in.) deep using a 0.25-in. drill bit. Prior to the test, the tops of the holes were filled with caulk. See Fig. 14 for a diagram of the vented panel locations.

FIG. 14 Test wall diagram for configurations 1 and 2. In configuration 2, the highlighted VIPs were intentionally vented.

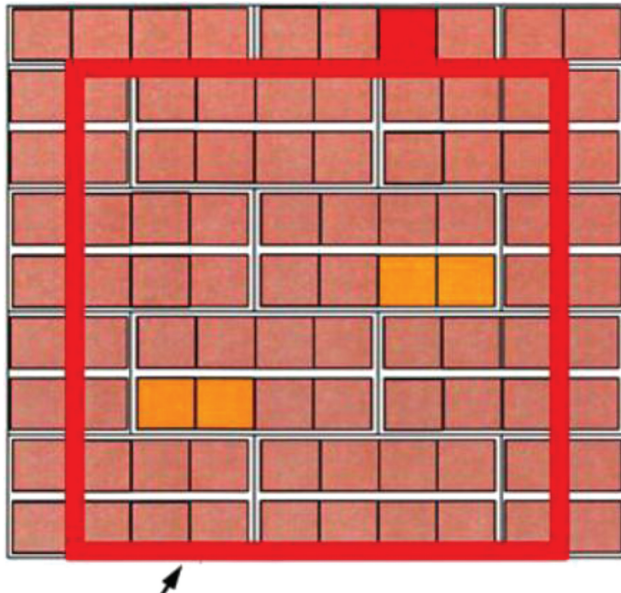
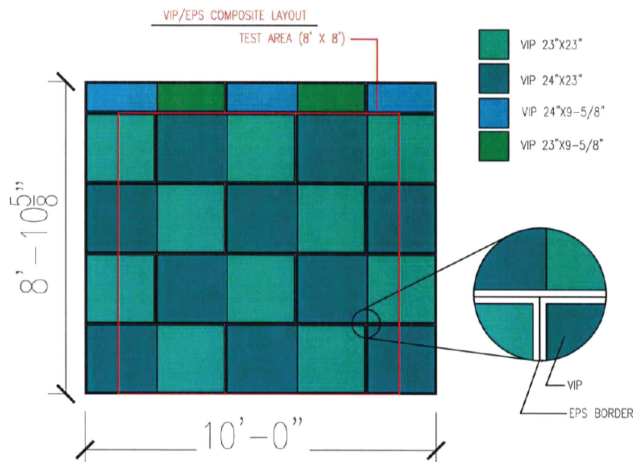


FIG. 15 Test wall layout for configurations 3 and 4.



In configuration 3 [11], 2 ft by 4 ft (60 cm by 120 cm) foam panels were constructed using nominal 24 in. by 24 in. (60 cm by 60 cm) vacuum panels, Type II EPS, and silicone adhesive and leaving 0.5 in. (12.7 cm) of edge protection. The panels were 3 in. (7.5 cm) thick with 1 in. (2.5 cm) comprising the center VIP section. The net VIP area coverage was 94 % of the wall area. Figure 15 diagrams the test wall layout.

Configuration 4 was the same wall tested in configuration 3, but with the wall cavity filled with R-11 fiberglass insulation.

Prior to testing, extruded foam insulation and R-11 fiberglass batt insulation were used to block and insulate the space outside the central metering area used for testing to reduce lateral heat losses. EIFS installation and the finished “exterior” and “interior” sides of the wall are shown in Figs. 16, 17, 18, and 19. The interior stud side was covered with 1.27-cm (0.5-in.) gypsum board and taped prior to testing.

Test Conditions

The temperature conditions for these tests were 37.8°C (100°F) and 10.0°C (50°F) in the metering and climate chambers, respectively. The exterior (VIP/EPS) side of the wall was facing the climate chamber, and the interior (stud and gypsum board) side of the wall was facing the metering chamber. Thermocouple arrays were installed on both hot and cold sides of the test walls to monitor temperatures over the wall cavities, studs, and top and bottom tracks. The temperatures of the hot and cold wall surfaces were determined via area-weighted averaging of the thermocouples attached to the individual components. Figure 19 shows the sealed and instrumented interior and exterior sides of the test wall.

FIG. 16 Installation of gypsum, air water barrier, and encapsulated VIP panels.

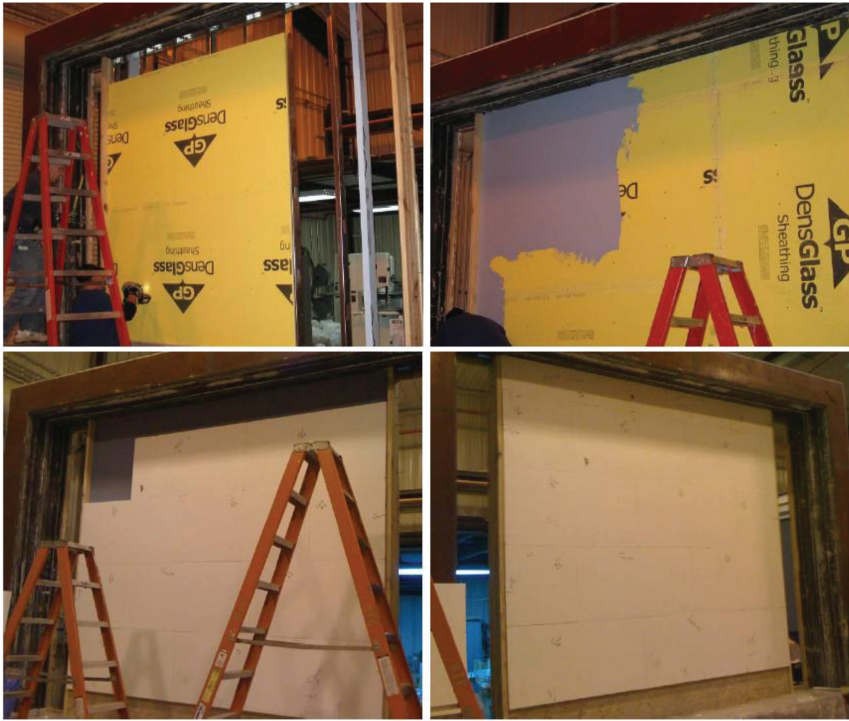


FIG. 17 Installation of basecoat and mesh over encapsulated VIP panels.



FIG. 18 Finished exterior and interior wall elevations.



Once the tests were started, it took between 40 and 60 h to reach stable temperature and heat flow conditions. The tests ran for a total of at least 150 to 180 h, and data from the final 15 to 40 h were used for analysis.

Test Results

Temperatures, heat flows, and R -values are presented in Table 3.

Configuration 1, with small VIPs and more edge protection, resulted in a surface-to-surface R -value of $3.7 \text{ m}^2 \cdot \text{K/W}$ ($20.8 \text{ h ft}^2\text{°F/Btu}$).

Configuration 2 (i.e., configuration 1 with some VIPs vented) resulted in a $\sim 3 \%$ reduction in R -value relative to configuration 1.

Configuration 4, with large VIPs, resulted in a $\sim 24 \%$ increase in R -value relative to configuration 1.

FIG. 19 Instrumented test walls.



TABLE 3 Hot box results.

	Configuration			
	1	2	3	4
	12 in. VIP	12 in. VIP, Four Vented	24 in. VIP + Cavity Insulation	24 in. VIP Only
T_{cs} , °C (°F)	10.0 (50)	10.0 (49.9)	10.0 (50.0)	10.0 (50.0)
T_{ms} , °C (°F)	37.9 (100.2)	37.8 (100.0)	37.8 (100.0)	37.7 (99.9)
T_{ma} , °C (°F)	38.6 (101.5)	38.5 (101.3)	38.1 (100.5)	38.3 (100.9)
T_{ca} , °C (°F)	9.6 (49.2)	9.5 (49.1)	9.6 (49.3)	9.6 (49.2)
ΔT , °C (°F)	27.9 (50.2)	27.8 (50.1)	27.8 (50.0)	27.8 (50.0)
T_{mean} , °C (°F)	23.9 (75.1)	23.9 (75.0)	23.9 (75.0)	23.9 (74.9)
Q_{wall} , W (Btu/h)	45.2 (154.2)	46.6 (158.9)	30.0 (102.3)	36.4 (124.2)
R_{wall} , m ² C/W (h ft ² °F/Btu)	3.7 (20.8)	3.6 (20.2)	5.5 (31.3)	4.5 (25.7)
$R_{U\ wall}$, m ² C/W (h ft ² °F/Btu)	3.8 (21.7)	3.7 (21.1)	5.6 (32.0)	4.7 (26.6)
$R_{ms\ air}$, m ² C/W (h ft ² °F/Btu)	0.1 (0.6)	0.1 (0.5)	0.05 (0.3)	0.08 (0.5)
$R_{cs\ air}$, m ² C/W (h ft ² °F/Btu)	0.05 (0.3)	0.06 (0.4)	0.07 (0.4)	0.07 (0.4)

Configuration 3, configuration 4 with cavity insulation, resulted in a ~50 % increase in R -value relative to configuration 1.

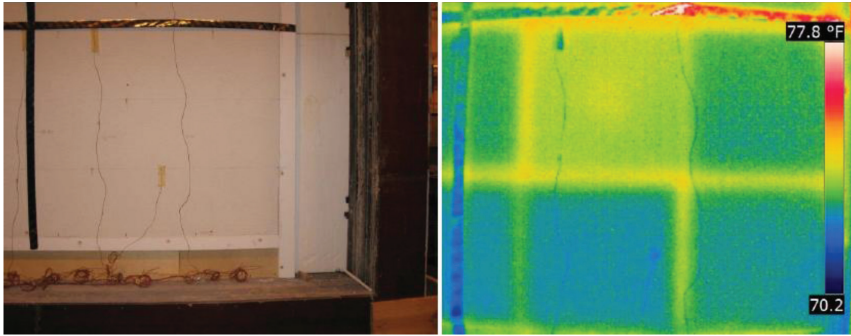
The difference between the hot-box testing result ($R = 25.7$) and the model ($R = 37.5$) was – 31 %.

Infrared Thermography

In addition to the whole-wall R -value tests, infrared (IR) thermographic images of a test wall were taken for qualitative assessment of the heat flow through the different wall components. For IR imaging, the test wall was clamped to the meter/guard chamber and the meter chamber heaters were turned on. The steel-framed side was facing the meter chamber, and the IR images were taken from VIP/EPS side.

Figure 20 shows the visual and IR images of the lower right quadrant. For identification, the wall sections were divided into four quadrants using reflective aluminum tape. Some heat flow characteristics are easily identifiable from the apparent temperature map. The center of the VIP panels shows uniform lower temperatures, with some thermal bridging occurring at the foam borders of the VIP/EPS composites (indicated by the relatively higher apparent surface temperatures). Further, there is some thermal bridging at the interface of the two VIPs within each composite, but it is less severe than through the foam edges. Figure 20 also shows the visual and IR images with one VIP punctured. The failed VIP is clearly indicated in the IR image; however, because the core consisted largely of microporous fumed silica, it still appeared to allow less heat flow than the foam borders.

FIG. 20 Infrared photography of VIP wall with vented panel.



Summary

A proposed wall system that incorporates vacuum insulation panels (VIPs) protected within polystyrene foam and finished with a commercially available EIFS system was evaluated via heat flow meter methods and guarded hot box methods. In addition to adding some thermal resistance, the foam serves to protect the vacuum panels during construction and to provide a surface appropriate for an adhesive joint on both sides of the foam-VIP unit. Multiple configurations of a composite VIP-foam insulation structure were evaluated using small subsections that could be placed within an HFM apparatus. Through careful test-specimen construction, all component joints were located within the range of an array of HFTs installed within the upper and lower plates of that apparatus. Special calibration procedures were used to ensure accurate heat flux measurements for these specimens with very low thermal conductivity. The resulting measurements were combined with modeling efforts to investigate the effects of vacuum panel size, the type of foam used to encase the vacuum panels, the thickness and shape of the foam sections between panels, and adhesives. A new procedure was developed to map the HFM measurement results onto full-scale wall designs to predict the system's thermal performance. This new method for mapping transducer measurements onto an array of larger panels shows promise, and was used to quantify the expected performance of several candidate construction arrangements. Further, four configurations of test walls were constructed and tested via ASTM C1363 hot-box testing. The configurations reflected one that was of maximum constructability for a VIP and another that was of maximum performance but still contained some degree of constructability.

The major conclusions of the project can be summarized as follows:

- It may be possible to develop a wall with an overall performance of R30 or greater with multiple VIP arrangements as shown by guarded hot plate and heat flux mapping data.

- The configurations with the greatest thermal resistance are those that maximize VIP coverage.
- A VIP encapsulated in a foam block, of material similar to what is in use today, could be used as a substitute in an EIFS facade system to gain high thermal efficiency in a thin profile.
- IR imaging showed that fumed silica core VIP panels have a higher thermal resistance than foam insulation only, even in punctured sections.

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Young Cheol Kwon,¹ Yang O Kim,² and Gil Yong Lee³

An Innovative Low-Emissivity Insulation Developed in Korea

Reference

Kwon, Young Cheol, Kim, Yang O, and Lee, Gil Yong, "An Innovative Low-Emissivity Insulation Developed in Korea," *Next-Generation Thermal Insulation Challenges and Opportunities*, STP 1574, Therese K. Stovall and Thomas Whitaker, Eds., pp. 101-118, doi:10.1520/STP157420130082, ASTM International, West Conshohocken, PA 2014⁴

ABSTRACT

A type of low-emissivity insulation (LEI), which is a type of reflective insulation assembly, has been developed in Korea. The critical difference between LEI and conventional reflective insulations is the presence of a honey-comb structure formed from polyethylene, which serves as the core material. The thermal resistivity for LEI is almost twice as much as that of expanded polystyrene foam board and many conventional reflective insulation assemblies. As a result, the thickness of LEI required to meet building codes is less than that of many competing insulations. Low-emissivity insulation has enclosed reflective air spaces between aluminum foils which have very low emissivity. The reflective air spaces are composed of optimum-sized air cells, which are enclosed by polyethylene foam. The product development and the test results for U value will be discussed in this paper. The LEI product development involved a large number of small-scale hot-box tests. The use of LEI in building envelopes in Korea will be described. LEI is proposed as a contribution to the next generation of building insulations needed to improve building energy efficiency.

Keywords

low-emissivity insulation, honey-comb structure, building energy, efficiency, hot-box test

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¹Dept. of Architecture, Halla Univ., Wonju, Gangwon, 220-712, South Korea.

²Research Institute, Ilsin Company, Gyung-san, Gyungbuk, 712-861, South Korea.

³Fire Insurers Laboratories of Korea, Yeosu, Gyunggi, 469-881, South Korea.

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Nomenclature

- A = area of specimen, m^2
- Q = supplied heat, W
- R = overall thermal resistance, m^2K/W
- T_{Hn} = environmental temperature of hot box, $^{\circ}C$
- T_{Cn} = environmental temperature of cold box, $^{\circ}C$
- U = overall thermal transmittance, W/m^2K

Introduction

LEI is a multilayer product consisting of layers of aluminum foil and a honey-comb structure produced from polyethylene foam. The resulting structure creates reflective airspaces within the cavity that reduce radiant heat transfer and heat flow by convection. The use of aluminum foil ensures a minimum 95 % reduction in radiation across the enclosed regions.

Figure 1 shows a 20-mm-thick, low-emissivity insulation structure.

LEI has foil surface emissivity of 0.04 with enclosed air cells between foils. Its core material is 35 times foamed polyethylene foam, which is expanded to make air cells. The enclosed air cells with foil reduce convective and radiative heat transfer.

This paper discusses the evolution of innovative LEI from conventional reflective insulation and its possible application to the building envelope to satisfy the increasing requirements for exterior wall insulation. This configuration indicates the thickness of LEI is less than that of many existing mass insulations. The critical difference between LEI and existing reflective insulations is the presence of a honey-comb core structure that reduces or eliminates convective heat transfer.

Investigation of Affecting Factors on Insulating Performance of Reflective Insulation

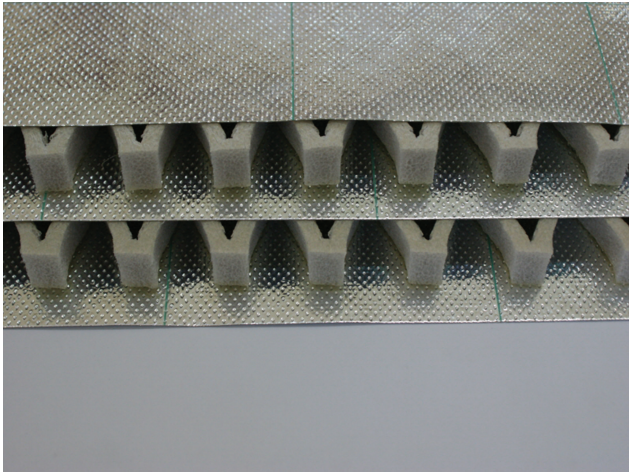
SURFACE EMISSIVITY

The emissivity of foils used to produce LEI is determined in accordance with ASTM C1371 [1] using the instrument shown in Fig. 2.

Unprotected aluminum foil, which is usually used as a reflective insulation surface, is susceptible to damage when contacted with an alkaline substance like concrete. Because concrete is a common construction material in Korea, aluminum foil must be coated to prevent damage. Table 1 shows the surface emissivity of four aluminum foil products.

The test results indicate that the emissivity of non-treated aluminum foil is the lowest. However, considering the reaction with alkaline, the 0.5- μm -thick coating on aluminum was used as the material for LEI. Aluminized surface or PET film

FIG. 1 20-mm-thick, low-emissivity insulation.



over aluminum is not recommended as material for LEI because of their high emissivity, even though they look almost the same as non-treated aluminum foil.

APPARENT THERMAL CONDUCTIVITY OF CORE MATERIALS

To maximize the insulating property of reflective insulation, apparent thermal conductivity of core materials should be as low as possible. They are used between

FIG. 2 Surface emissivity tester.

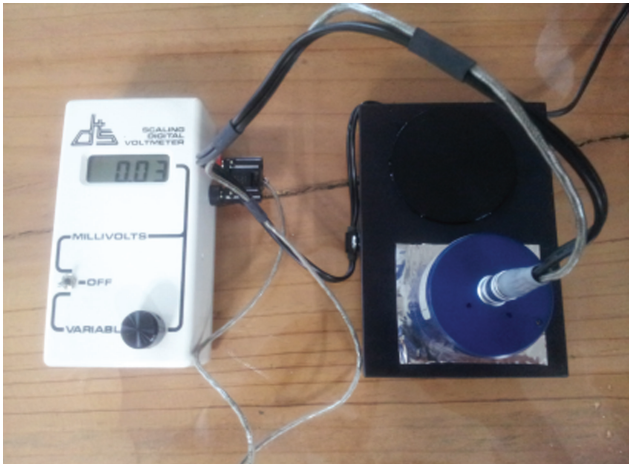


TABLE 1 Emissivity of four aluminum foil materials.

Material	Emissivity
Non-treated aluminum foil	0.03
0.5 - μ m-thick coating on aluminum foil	0.04
Aluminized surface	0.48
PET film over aluminum	0.55

aluminum foils to reinforce the tensile strength and reduce convective heat transfer. The thickness of reflective insulation material is determined by the core material. Multi-layered reflective insulation in Korea, for example, is typically 6–13 mm thick [2]. Figure 3 shows an existing reflective insulation whose core materials are non-woven fabric and polyethylene foam.

The apparent thermal conductivity of major core materials was measured in accordance with ISO 8301 (Thermal Insulation-Determination of Steady-State Thermal Resistance and Related Properties). ISO 8301 is similar to ASTM C518 [3]. The measurements were obtained in a laboratory maintained at $23^{\circ}\text{C} \pm 1^{\circ}\text{C}$ with relative humidity $50\% \pm 5\%$. The mean test temperature was $20^{\circ}\text{C} \pm 1^{\circ}\text{C}$ and the temperature difference across the test specimens was 26°C . The experimental uncertainty of the apparatus was $\pm 2\% \sim 5\%$. Figure 4 contains a diagram of the heat flow meter apparatus that was used to measure apparent thermal conductivity [4].

Test results for the core materials for LEI are shown in Table 2.

FIG. 3 Existing reflective insulation in Korea.

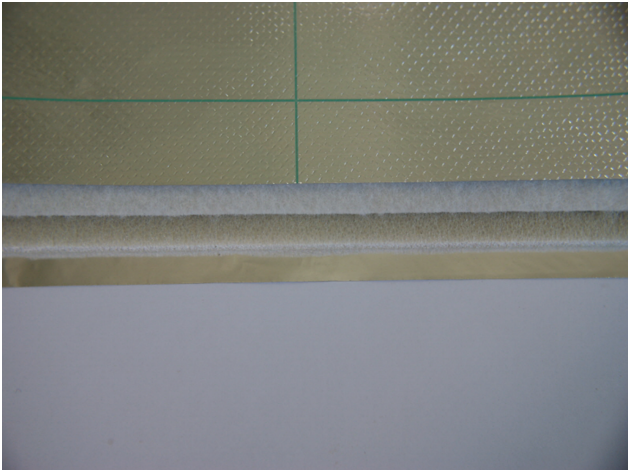


FIG. 4 Diagram of heat flow meter apparatus.

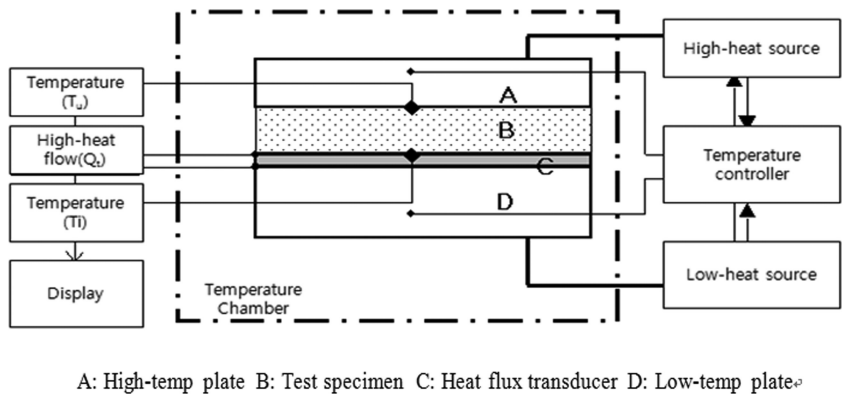


Table 2 shows that spunbonded nonwoven fabric (120 g/m²) has the lowest value of 0.031 W/mK. In case of polyethylene foam, 35 times the foamed one has lower value than 40 times the foamed one.

OPTIMUM SIZE OF AIR CELLS SEPARATED BY POLYETHYLENE FOAM

Polyethylene foam as a core material can be used to produce a honey-comb structure like that shown in Fig. 5.

The structure turns out to be an important factor for optimizing thermal performance. To determine the optimum size of air cells, overall thermal transmittance tests were conducted for LEI with various air cell sizes and horizontal heat flow. The overall thermal transmittance was measured in accordance with ISO 8990 (Thermal Insulation-Determination of Steady-State Thermal Transmission Properties-Calibrated and Guarded Hot Box), a test method that is like ASTM C1363 [5]. The hot-box apparatus is shown in Fig. 6.

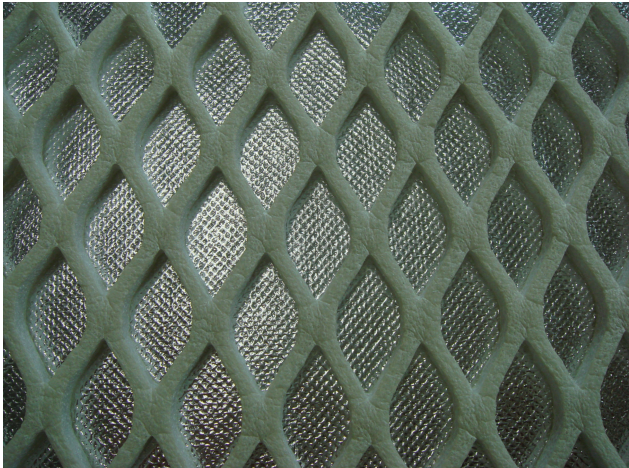
The hot-box facility was calibrated using polystyrene foam board whose thermal conductivity had been acquired from a heat flow meter apparatus test at an

TABLE 2 Apparent thermal conductivities of four core materials.

Major Core Materials	Apparent Thermal Conductivity (W/mK)
Spunbonded nonwoven fabric (120 kg/m ³)	0.031
Recycled nonwoven fabric (140 kg/m ³)	0.034
Polyethylene foam (35 times foaming) ^a	0.043
Polyethylene foam (40 times foaming)	0.048

^aPolyethylene foam whose volume is increased 35 times after foaming.

FIG. 5 Honeycomb structure provided by punctured PE foam.



accredited laboratory designated by KOLAS (Korea Laboratory Accreditation Scheme).

Tests were conducted with a hot region temperature of 20°C and a cold region temperature 0°C. The heat flow rate from hot region to the cold region is determined from energy input to the hot region as in the case with ASTM C1363. The overall thermal transmittance was calculated after steady-state conditions have been

FIG. 6 Overall thermal transmittance test apparatus.



achieved. Data were recorded three times every hour and averaged. The overall thermal transmittance (U) can be obtained from Eq 1 [6].

$$U = 1/R = Q/[(T_h - T_c) * A] \tag{1}$$

- where:
- U = overall thermal transmittance ($\text{W}/\text{m}^2 \text{ K}$),
 - R = overall thermal resistance ($\text{m}^2\text{K}/\text{W}$),
 - Q = supplied heat (W),
 - T_h = temperature of hot region ($^{\circ}\text{C}$),
 - T_c = temperature of cold region ($^{\circ}\text{C}$), and
 - A = area of specimen (m^2).

The thickness of polyethylene foam was fixed at 10 mm by production limitations. The base wall configuration for the comparison tests consisted of 130 mm of concrete, reflective specimen, 40 mm of enclosed reflective air space, and 30 mm of granite. The test structure is shown in Fig. 7.

To find out the optimum size of separated reflective air space, the thickness of PE foam was selected as 6, 10, and 20 mm, and 70 % and 80 % air cell in the PE foam were compared as shown at Table 3.

From the combination of above variables, six assembly variations result. In addition to the six assembly variations, three more cases for no insulation and normal 6 mm and 20 mm PE foam were added to compare the relative insulating performance of the specimens. The nine assemblies are identified in Table 4. The % air (void space) indicates the volume of foam used to produce the honeycomb structure.

To compare the relative insulating performance of the above specimens, a base wall configuration that has no foam was evaluated (Case 1 in Table 4). Table 5 provides additional details about the assemblies that were evaluated.

FIG. 7 Section of wall assembly containing test specimen.

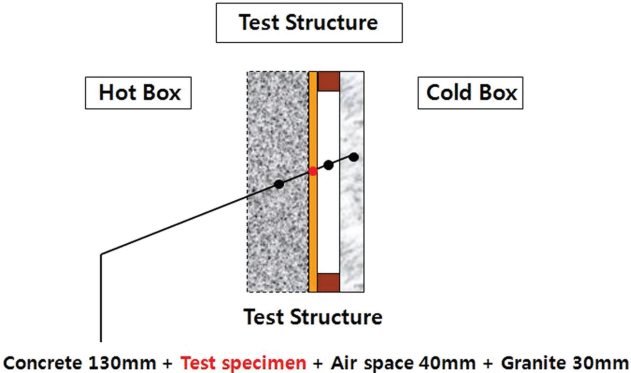


TABLE 3 Variables for optimum design of specimen.

Variables	Values	
Thickness of PE foam (mm)	6, 10, 20 ^a	
Percentage of air cell in the PE foam (%)	70 ^b	80 ^c

^aAL + PE foam 10 mm + AL + PE foam 10 mm + AL.
^bIn case the width of PE foam is 5 mm, the size of separated air cell is about 25 by 25 mm, and the ratio of air volume in punctured PE foam is 70 %.
^cIn case the width of PE foam is 4 mm, the size of separated air cell is about 35 by 35 mm, and the ratio of air volume in punctured PE foam is 80 %.

The overall thermal transmittance test for the wall configurations in Table 5 was determined in accordance with ISO 8990. The average value was calculated from three days of testing after steady state was attained. The test results are contained in Tables 6, 7, and 8.

The overall thermal transmittance of Case 1 wall configuration, which is composed of 130 mm concrete + 40 mm air space + 30 mm granite, was 2.97 W/m²K. Thanks to adding a reflective insulation of 6 mm and 20 mm, the overall thermal transmittance was lowered to 0.57 and 0.52 W/m²K. These results show that the thickness of normal PE foam is not the critical factor for increasing the insulating performance of a reflective insulation.

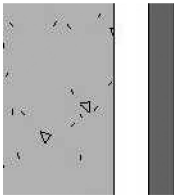
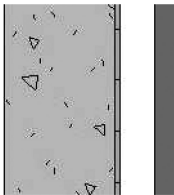
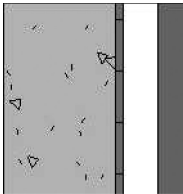
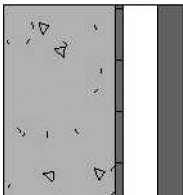
Case 7–Case 9, which has 80 % air volume in punctured PE foam, shows about 12 % higher insulating performance than Case 4–Case 6, which has 70 % air volume in punctured PE foam. Case 7 and Case 9 indicate that they have, respectively, 9 % and 24 % higher insulating performance than Case 2 and Case

TABLE 4 Various types of test specimen.

Type of Test Specimen	Thickness of PE Foam (mm)	Percentage of Air Volume in the PE Foam (% Void Space)
Case 1	0	0
Case 2	6	0
Case 3	20	0
Case 4	6	70
Case 5	10	70
Case 6	20 ^a	70
Case 7	6	80
Case 8	10	80
Case 9	20 ^a	80

^aAL + PE foam 10 mm + AL + PE foam 10 mm + AL.

TABLE 5 Wall configuration containing test specimens.

Type	Wall Configuration	Note
Case 1		No LEI specimen
	Concrete 130 + air space 40 + granite 30	
Case 2 Case 3		Normal PE foam was installed as thick as 6 mm (case 2) and 20 mm (case 3)
	Concrete 130 + PE foam 6 (20) + air space 40 + granite 30	
Case 4 Case 5 Case 6		Punctured PE foam was installed as thick as 6 mm (case 7), 10 mm (case 8), and 20 mm (case 9).
	Concrete 130 + punctured PE foam 6 (10,20) ^a + air space 40 + granite 30	70 % Air volume in punctured PE foam
Case 7 Case 8 Case 9		Punctured PE foam was installed as thick as 6 mm (case 7), 10 mm (case 8), and 20 mm (case 9).
	Concrete 130 + punctured PE foam 6 (10,20) ^a + air space 40 + granite 30	- 80 % Air volume in punctured PE foam

^aPunctured PE foam 20 is composed of AL + PE foam 10 mm + AL + PE foam 10 mm + AL.

3, where the specimens have normal PE foam. Table 9 shows that 80 % air volume in punctured PE foam has the best thermal performance.

After considering all the above test results, we can reach the following conclusions: punctured PE foam has better insulating performance, 10-mm-thick punctured PE foam is better than 6 mm, and 80 % air volume in punctured PE foam has higher insulating performance than 70 %.

TABLE 6 Overall thermal transmittance.

Type	Insulation	Thickness of PE Foam (mm)	Overall Thermal Transmittance (W/m ² K)
Case 1	None	–	2.97
Case 2	Normal PE foam	6	0.57
Case 3	Normal PE foam	20	0.52

TABLE 7 Overall thermal transmittance for honeycomb structures with 70 % void space.

Type	Ratio of Air Volume in Punctured PE Foam (%)	Thickness of Punctured PE Foam (mm)	Overall Thermal Transmittance (W/m ² K)
Case 4	70	6	0.58
Case 5	70	10	0.53
Case 6	70	20	0.47

TABLE 8 Overall thermal transmittance for honey-comb structures with 80 % void space.

Type	Ratio of Air Volume in Punctured PE Foam (%)	Thickness of Punctured PE Foam (mm)	Overall Thermal Transmittance (W/m ² K)
Case 7	80	6	0.52
Case 8	80	10	0.47
Case 9	80	20	0.42

TABLE 9 Overall thermal transmittance.

Type	Ratio of Air Volume in Punctured PE Foam (%)	Thickness of PE Foam (mm)	Overall Thermal Transmittance (W/m ² · K)
Case 3	0	20	0.52
Case 6	70	20	0.47
Case 9	80	20	0.42

TABLE 10 Optimum design of LEI.

Variables	Optimum Design
Thickness of punctured PE foam (mm)	10
Width of the foam (mm)	4
Size of separated air cell (mm ²)	35 × 35
Ratio of air volume in punctured PE foam (%)	80

FIG. 8 LEI with 80 % void space provided by punctured PE foam.

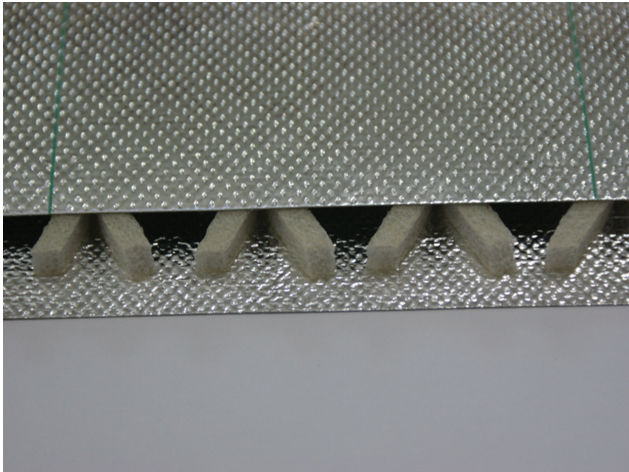


FIG. 9 Drawing of the test specimen.

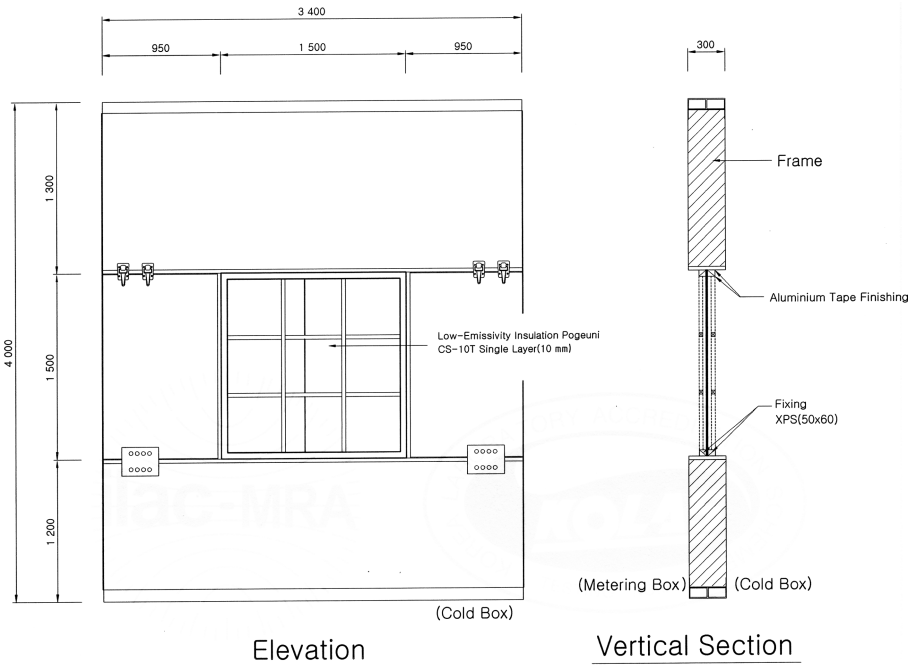
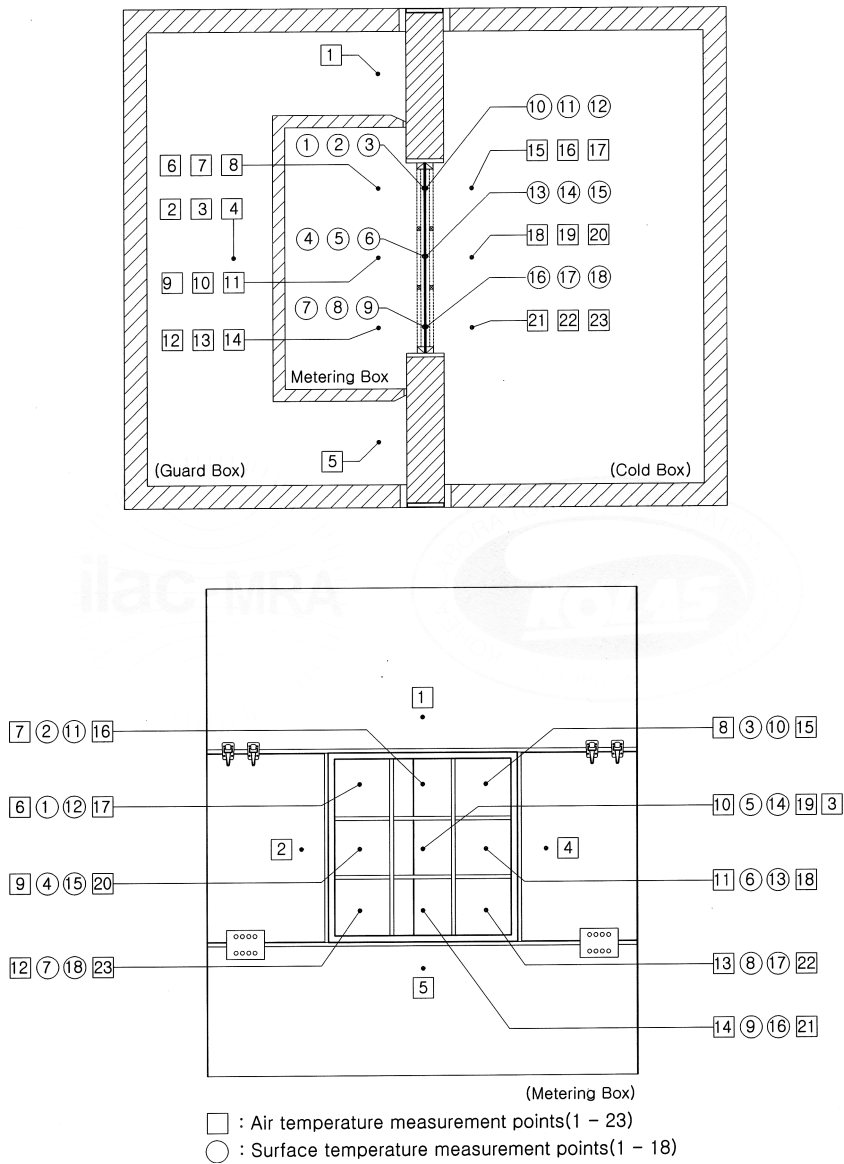


FIG. 10 Location of temperature sensors.



Based on the various comparative experiments, the optimum design for the LEI is summarized in Table 10.

Based on the various comparative experiments, the optimum design for the core material is as follows: thickness of punctured PE foam of 10 mm, width of the foam of 4 mm, size of separated air cell of 35 by 35 mm², and air volume in

punctured PE foam of 80 % (80 % void space). Figure 8 shows a 10-mm-thick LEI product resulted from optimum design factors in Table 10.

Insulating Performance of LEI

Based on this research, a high-efficiency low-emissivity insulation (LEI) was developed.

To verify the high efficiency of low-emissivity insulation, it has been tested by an accredited laboratory designated by the Korean government. The overall thermal transmittance tests were conducted for the low-emissivity insulation of various thickness specimens. The thicknesses of the specimens were 10, 20, 30, 40, 50, 60, 80, and 100 mm. The overall thermal transmittance test was conducted in accordance with ISO 8990 (Thermal Insulation-Determination of Steady-State Thermal Transmission Properties-Calibrated and Guarded Hot Box). Figure 9 shows a drawing of the test specimen.

The test specimen size is 1500 mm². Figure 10 shows the locations of temperature sensors.

To get more accurate measurement results, three temperature sensors were located at each point. The temperature at each point was determined by averaging the three measured values. Test results are contained in Table 11.

Table 12 contains normal wall configurations showing applications of LEI in Korea and the test results of their overall thermal transmittance by an accredited laboratory.

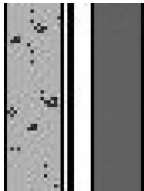
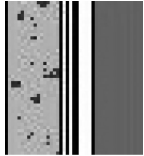
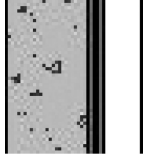
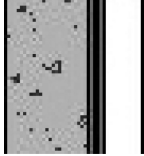
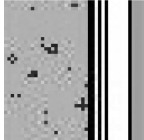
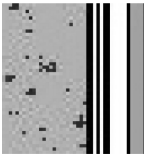
LEI Thickness to Meet the Required U-Values by Korean Building Insulation Code

Table 13 shows required *U* values for central region by Korean building insulation codes [7,8].

TABLE 11 Overall thermal transmittance of low-emissivity insulation.

Thickness of Low-Emissivity Insulation (mm)	Overall Thermal Transmittance (W/m ² K)
10	1.12
20	0.55
30	0.45
40	0.32
50	0.27
60	0.22
80	0.19
100	0.14

TABLE 12 Overall thermal transmittance for walls with LEI.

Type	Wall Configuration	Overall Thermal Transmittance (W/m ² K)
Case 1		0.41
	Concrete 100 + LEI 20 + air space 30 + red brick 90	
Case 2		0.34
	Concrete 100 + LEI 30 + air space 30 + red brick 90	
Case 3		0.35
	Concrete 150 + LEI 20 + air space 70 + AL panel 4	
Case 4		0.32
	Concrete 150 + LEI 30 + air space 70 + AL panel 4	
Case 5		0.36
	Concrete 150 + LEI 30 + air space 40 + granite 30	
Case 6		0.31
	Concrete 150 + LEI 40 + air space 30 + granite 30	

Note: Table contains normal wall configurations showing applications of LEI in Korea and the test results of their overall thermal transmittance by an accredited laboratory.

TABLE 13 Required U values for the central region of Korea.

	Building Envelope		Required U-Value (W/m ² · K)
Exterior wall	Adjacent to outside directly		Less than 0.36
	Adjacent to outside indirectly		Less than 0.49
Roof	Adjacent to outside directly		Less than 0.20
	Adjacent to outside indirectly		Less than 0.29
Floor of the lowest floor	Adjacent to outside directly	Floor heating	Less than 0.30
		No floor heating	Less than 0.41
	Adjacent to outside indirectly	Floor heating	Less than 0.43
		No floor heating	Less than 0.58
	Sidewall of apartment building		Less than 0.27
Floor between levels	Floor heating		Less than 0.81
	No floor heating		Less than 1.16
Fenestrations and doors	Adjacent to outside directly	Apartment	Less than 2.10
		Others	Less than 2.40
	Adjacent to outside indirectly	Apartment	Less than 2.80
		Others	Less than 3.20

Table 13 indicates that required *U* value for exterior wall adjacent to outside directly should be less than 0.36 W/m²K. For the designer’s convenience, the building insulation codes provide the required insulation thickness according to each

TABLE 14 Required thickness of insulation for central region.

Building Envelope	Required Insulation Thickness According to Grade		Thickness (mm)			
			A	B	C	D
Exterior wall	Adjacent to outside directly		85	100	115	130
	Adjacent to outside indirectly		60	70	80	90
Floor of the lowest floor	Adjacent to outside directly	Floor heating	105	125	140	160
		No floor heating	75	90	100	115
	Adjacent to outside indirectly	Floor heating	70	80	90	105
		No floor heating	50	55	65	70
Roof	Adjacent to outside directly		160	190	215	245
	Adjacent to outside indirectly		105	125	145	160
	Sidewall of apartment building		120	140	160	175
Floor between levels	Floor heating		30	35	45	50
	Other		20	25	25	30

The insulation grade is given as A, B, C, and D according to thermal conductivity of insulation. Table 15 shows how the grade is determined.

insulation grade. Table 14 shows the required thickness of insulation to obtain the required *U* values.

Table 15 indicates; for example, that XPS, Neopor, PU, and high-density glass wool belong to Grade “A.”

According to Tables 14 and 15, existing insulations should be used as thick as 85 mm or 100 mm for an exterior wall, which is directly adjacent to the outside for the central region in Korea.

On the bases of the test results in Table 12, 30 mm or 40 mm LEI can meet the required *U* value for the same wall. This means that the thickness of LEI required to meet building codes is less than that of many competing insulations.

Results and Discussion

0.5 μ m-thick coated aluminum surface with emissivity of 0.04 is recommended for the use in a cement–concrete structure. Aluminized surface and PET film over aluminum are not recommended as a surface of reflective insulation because of its high emissivity.

TABLE 15 Insulation classification according to thermal conductivity in Korea.

Grade	Thermal Conductivity		Insulation Type (note)
	W/mK	kcal/mh °C	
A	0.034 or lower	0.029 or lower	XPS (extruded polystyrene board) outstanding type, type 1, type 2, type 3 EPS (expanded polystyrene) class 2 (neopor) no. 1, no. 2, no. 3, no. 4 Rigid polyurethane foam, class 1 no. 1, no. 2, no. 3 and class 2 no. 1, no. 2, no. 3 Glass wool insulating board 48 K, 64 K, 80 K, 96 K, 120 K Thermal conductivity is 0.034 W/mK (0.029 kcal/mh °C) or lower as other insulations
B	0.035 ~ 0.040	0.030 ~ 0.034	EPS (expanded polystyrene) class 1 no. 1, no. 2, no. 3 Mineral wool no. 1, no. 2, no. 3 Glass wool 24 K, 32 K, 40 K Thermal conductivity is 0.035 ~ 0.040 W/mK (0.030 ~ 0.034 kcal/mh °C) as other insulations
C	0.041 ~ 0.046	0.035 ~ 0.039	EPS (expandable polystyrene) class 1 no. 4 Thermal conductivity is 0.041 ~ 0.046 W/mK (0.035 ~ 0.039 kcal/mh °C) as other insulations
D	0.047 ~ 0.051	0.040 ~ 0.044	Thermal conductivity is 0.047 ~ 0.051 W/mK (0.040 ~ 0.044 kcal/mh °C) as other insulations

Low-*E* insulation is an innovative material, which was developed in Korea using the principles of reflective insulation. Its insulating efficiency is competitive with that of polystyrene foam board and glass wool. So the thickness of low-*E* insulation to meet the building insulation code is less than many competing systems.

Low-*E* insulation has enclosed reflective air spaces between aluminum foils, which have very low surface emissivity. The reflective air spaces are composed of optimum-sized air cells that are enclosed by polyethylene foam

Low-*E* insulation is believed as an innovative material that contributes to the next generation of building insulation to improve the building energy efficiency.

The results on this paper are totally based on experiments. Future research will focus on the insulating mechanisms of low-*E* insulation.

Conclusions

Based on the various comparative experiments, the optimum design for the core material is as follows: thickness of punctured PE foam of 10 mm, width of the foam of 4 mm, size of separated air cell of 35 by 35 mm², and air volume in punctured PE foam of 80 % (80 % void space).

The overall thermal transmittance of 40-mm- and 50-mm-thick low-*E* insulation was measured as 0.32 and 0.27, respectively.

The thickness of LEI required to meet building codes is less than that of many competing insulations.

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Roy E. Smith,¹ Jeffrey M. Alcott,¹ and Michael H. Mazor²

Design Considerations for Sustainable Extruded Polystyrene (XPS) Thermal Insulation

Reference

Smith, Roy E., Alcott, Jeffrey M., and Mazor, Michael H., "Design Considerations for Sustainable Extruded Polystyrene (XPS) Thermal Insulation," *Next-Generation Thermal Insulation Challenges and Opportunities*, STP 1574, Therese K. Stovall and Thomas Whitaker, Eds., pp. 119–130, doi:10.1520/STP157420130089, ASTM International, West Conshohocken, PA 2014.³

ABSTRACT

Extruded polystyrene (XPS) foam has been a useful and cost-effective thermal insulation for commercial and residential buildings for over 50 years. Producers have responded to a variety of factors, such as environmental regulations, building codes, and energy costs, to obtain effective alternative XPS products. This paper reviews XPS insulation formulations with blowing agents, HFC-134a and HFC-152a, that have long-term thermal performance to meet or exceed current codes. The developed XPS insulations have a zero ozone-depletion potential and provide a sustainable benefit to society.

Keywords

sustainable XPS foam, thermal insulation, greenhouse gas

Introduction

Extruded polystyrene (XPS) foam thermal insulation is used in residential and commercial buildings to save energy and valuable resources. The physical blowing

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¹Dow Chemical Company, Dow Building Solutions R&D, 2030 Dow Center, Midland, MI 48674, United States of America.

²Dow Chemical Company, Energy and Climate Change, 2030 Dow Center, Midland, MI 48674, United States of America.

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agents used to produce XPS foam have evolved over the past three decades primarily because of a changing regulatory landscape. Global regulations have been implemented to mitigate the effects that chlorine and fluorine containing carbon gases have on the atmosphere of the earth. Additional regional regulations impact the use of volatile organic compounds (VOCs), which are also frequently used as foam blowing agents. These regulations, along with the increasing desire for energy efficiency and environmental sustainability, have made it increasingly difficult and challenging to develop next-generation formulations for foam insulation. Moreover, the lack of clarity and specificity regarding insulation performance and exposure conditions have made it difficult for building designers/owners to decide with certainty which insulation to choose and how to properly insulate to maximize energy efficiency and resulting value.

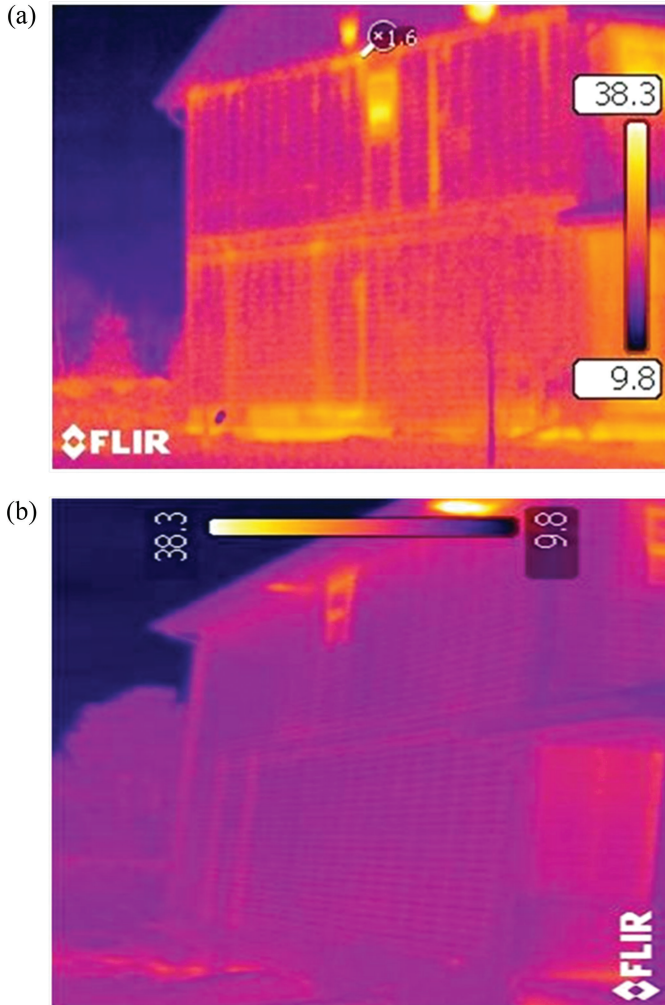
This paper will explore the issues faced by one industry as it embarked on the development of the next generation of XPS products. The emphasis will be placed on blowing-agent technology, because this plays a critical role in defining the long-term thermal performance element of a sustainable product design.

Sustainable XPS Product Evolution and Product-Design Considerations

Polystyrene foam has been in commercial existence for over 60 years. Initially, XPS plastic foam was made via a batch foaming process and used primarily as flotation foam, with the military as a large customer. As company scientists explored other uses for this unique foam, thermal insulation applications became an obvious choice. This is illustrated by the thermal images in Fig. 1(a) and 1(b) for the residential insulated sheathing application. There are several ways that an exterior building wall can be insulated and protected from the damaging effects of moisture and comply with building and energy codes. An exterior wall can be insulated with fiberglass insulation in the wall cavity and the wall can be covered with an oriented strand board (OSB) sheathing and a house wrap to manage moisture issues. The thermal shorts created by the uninsulated wood framing, which typically comprises about 20 % of the opaque wall area, are clearly evident in the thermal image shown in Fig. 1(a). An exterior wall can also be insulated with a continuous insulated foam sheathing that provides both thermal efficiency and moisture control. In conjunction with continuous insulated foam sheathing, fiberglass insulation can be added to wall cavity to provide for a very thermally efficient wall, as shown by the thermal image shown in Fig. 1(b). The smart use of insulating foam sheathing helps reduce heat loss or gain and saves energy.

Early candidates for blowing agents were generally compounds that were highly soluble in polystyrene, which facilitated good mixing and easy dissolution in the batch foaming process. However, these early blowing agents also tended to be highly flammable and their environmental health and safety impact was not well understood at the time.

FIG. 1 (a) Thermal image of home constructed with OSB, Batts, and housewrap, which meets 2012 ICC-ES building code [1] (courtesy of Gary Parsons), indicated that temperature range is in °F. (b) Thermal image of home constructed with R-5 continuous XPS and Batts, which meets 2012 ICC-ES building code [1] (courtesy of Gary Parsons), indicated that temperature range is in °F.



Through extensive company research in the late 1950s and early 1960s, it was determined that blowing agents, such as dichlorodifluoromethane (CFC-12), would not only function as excellent foaming agents, but because of the low thermal conductivity and extremely low permeability through polystyrene, they would also increase the insulation value by more than 20 %. Moreover, CFC-12 had the added

benefits of not being flammable, posing minimal exposure concerns, and was widely available. Therefore, it was deemed a nearly ideal blowing agent for polystyrene foam because it was relatively inexpensive, non-flammable, provided a significant benefit to the insulation value of the foam, and, at the time, was considered to be environmentally benign.

By the end of the 1950 s, the equipment for making XPS thermal insulation had been converted to a continuous extrusion process. Combining the improved process technology with the better insulating blowing agents allowed XPS foam manufacturers to develop low-cost methods for efficiently producing accurately dimensioned rigid insulating foam boards, which were used by builders to improve the energy efficiency of buildings. However, because of the low cost of energy during the 1960 s, XPS thermal insulation usage in buildings remained relatively small. XPS thermal insulation did not see significant growth as an insulation material until the oil embargoes of the early 1970 s, which made it apparent that helping homeowners save on heating and cooling costs presented a significant value proposition for builders.

During the 1980 s, the environmental science on the impact of chlorinated carbons on the ozone layer was developed and regulations were established in response to the Montreal Protocol [1,2] to eliminate the use of CFC-12. Company researchers developed and quickly implemented an alternative formulation, where CFC-12 was replaced with 1-chloro-1,1-difluoroethane (HCFC-142 b), that has 97 % less ozone-depletion potential (ODP) relative to CFC-12. Research continued in more sustainable foam plastic insulation technology during the early 2000 s ahead of the deadline for the phase-out of HCFC-142 b in the United States and Canada. Continuing research led to the development of XPS thermal insulation with zero ODP, which is sold today [3]. For this paper, the focus of sustainable plastic foam insulation technology development will be limited to blowing agents, because resin design considerations have been reviewed elsewhere [4].

The factors to be considered when selecting blowing agents include: reactivity, diffusivity, solubility, and permeability in the base plastic, flammability, toxicity, human-exposure concerns, thermal conductivity, water absorption, ozone-depletion and global warming potential (GWP), and VOC status (see Tables 1 and 2).

Whether direct or indirect, all of these variables will have an impact on sustainability as a building material, but yet no one can establish sustainability completely on its own. Some of these variables may be dependent on the application of environmental conditions. One example is thermal conductivity, which is a function of temperature. The measured R value of all insulation products will vary with mean-temperature test conditions as illustrated in Fig. 2. The prescriptive path of the ICC International Energy Conservation Code (IECC) [5,6] is based on insulation R values determined at 75°F mean temperature. For many insulation products, the measured R value will go up with decreasing mean temperature. A summary of R value/in. versus mean temperature for polystyrene-based foams is provided in Fig. 2.

TABLE 1 Ideal blowing agent properties.

Blowing Agent Property	Optimum Position
Solubility in base plastic	Low to moderate
Permeability in base plastic	Very low
Diffusivity in base plastic	Low
Reactivity	None
Flammability	None
Toxicity	None
Human exposure concerns	Minimal
Thermal conductivity	Less than air (O ₂ and N ₂)
Water absorption	Very low
Ozone-depletion potential	None
Global warming potential	Low
VOC status	Not a VOC ^a
Boiling point	Lower than general building construction application temperatures
Availability	Commercially available
Cost	Provides sustainable value

^aIn some situations, it may be sustainable to capture production emissions and destroy.

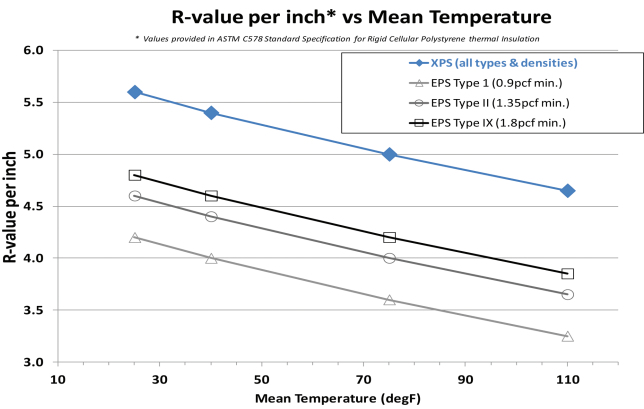
There is currently a question in the industry regarding the *R* value of polyisocyanurate foam at lower mean temperatures. Typically the *R* value will increase with decreasing mean temperature; however, data generated by the National Roofing Contractors Association (NRCA) [7] indicates that the *R* value of polyisocyanurate foam will decrease below a 75°F mean temperature (see Fig. 3). This data

TABLE 2 Sustainability attributes and properties of gases w/polystyrene.

Blowing Agent	VOC	ODP	GWP	Flammable	Permeability	Thermal Conductivity
CFC-12	No	1	10 900	No	<1	<0.1
HCFC-142 b	No	0.065	2310	Yes	<1	<0.1
HFC-134 a	No	0	1430	No	<1	<0.1
HFC-152 a	No	0	124	Yes	8.4	<0.1
Pentane	Yes	0		Yes	3.5	<0.1
CO ₂	No	0	1	No	>1000	0.11
Nitrogen	No	0			57	0.18
Oxygen	No	0			343	0.18

Note: VOC, volatile organic compound as defined by the U.S. EPA; ODP, ozone-depletion potential; GWP, global warming potential per IPCC 4th Assessment Report. Permeability units are cm³-mil/day-100 in. ²-atm at 25°C. Thermal conductivity units are BTU-in./h-ft ²-°F at 24°C.

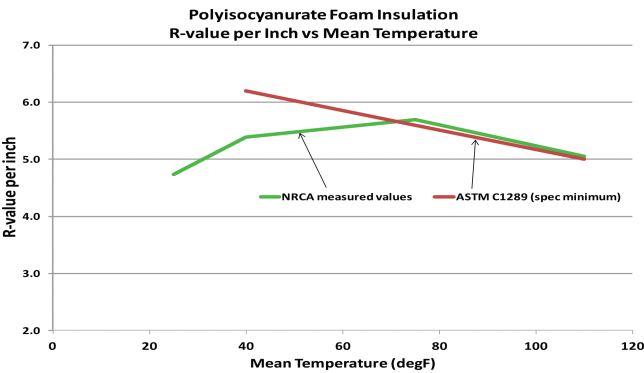
FIG. 2 R value per in. versus mean temperature of XPS versus EPS [6].



suggests that the foam-blowing agent, pentane, may be condensing at or near its boiling point of around 40°F. This may affect applications where cold-temperature conditions exist, such as coolers and freezers.

Many of these variables also impact long-term product performance, which is, unfortunately, overlooked in some sustainability assessments. Combining the sustainability of raw materials used in the manufacture of a product along with the long-term product performance would enable the buyer/user/builder to make more informed decisions regarding which insulation product would best meet their overall needs.

FIG. 3 R value per in. versus mean temperature of polyisocyanurate foam insulation [7,8].



XPS Life-Cycle Performance and Comparisons

Before one can begin the process of designing the next generation of sustainable XPS foam insulation products, it is critical to understand the perceived meaning of the term “sustainable,” through consideration of the relative weighting of: (a) the initial environmental footprint, and (b) the long-term thermal performance of the insulation over the lifetime of the building. It is our opinion that the definitions for these two terms are rather nebulous, and consequently some insulation suppliers may selectively advertise those points that favorably highlight their product and deemphasize any that may suggest a negative attribute when compared to other insulation products.

To illustrate this point, the following questions can be asked:

- Which definition of a sustainable insulation product is most appropriate?
- product A, which uses no VOCs, but contains a greenhouse gas (GHG) and has a higher insulation value, or
- product B, which uses VOCs, but contains no GHG and has a lower insulation value.
- How can the buyer/user/builder make an appropriate determination?
- What criteria should one use, VOC content, GHG content, or long-term thermal insulation value?
- Is potential ground level smog and pollution more significant than GWP considerations, or does the overall energy savings imparted by the product outweigh both?

With Tables 1 and 2 as guides, it is now possible to begin the selection of an acceptable blowing agent, by comparing different sustainability aspects for a variety of insulation products. Recent blowing agent replacement research efforts were directed in two primary areas: (a) options that minimize atmospheric impact (CO_2 -based foams), and (b) options that provide higher insulating values, which are based on the use of GHGs. Each approach carried its own set of technical challenges; however, both have been successfully implemented globally depending on government regulations, building code requirements, and local buildings practices.

The greenhouse-gas footprint arising from the materials, production, distribution, fugitive emissions, and end-of-life was estimated for both EPS and XPS on a board foot basis (12 in. $\times$ 12 in. $\times$ 1 in.). This allows for a comparison of the carbon footprint of a unit volume of foam. Because the thermal performance is not identical, the amount of foam required to meet the application specifications or industry energy conservation codes are not the same. The impact of this can be estimated by the following analysis in Table 3 for an application for foundation insulation in northern climates (zones 4–6).

In this calculation, the 2012 IECC residential energy conservation code [5] was used to establish the thickness of XPS products (R -5.0/in.) and the thickness of molded bead EPS product (R -4.0/in.). To meet energy-code requirements for a given application, more EPS is required to meet the same thermal performance, so, on a volume (board foot) basis, the EPS foam saves less energy and associated GHG

TABLE 3 Greenhouse gas reductions for foundation insulation in climate zones 4, 5, and 6 [9].

Basis: Concrete Foundation Wall Meeting the 2012 International Energy Code ^a	GHG Savings Difference XPS Versus EPS for Heating Over 50 years (pounds CO ₂ /BDF)	GHG Footprint Difference Comparing XPS Versus EPS (pounds CO ₂ /BDF)
Zone 4: R10 (Baltimore)	+59	−19
Zone 5: R15 (Chicago)	+59	−19
Zone 6: R15 (Burlington)	+79	−19

Note: 1 board foot (BDF) = 1 inch × 1 foot × 1 foot of foam.
^aCode required continuous insulation *R* value per climate zone for foundations.

emissions. In foundation insulation applications in northern climates, each board foot of XPS foam avoids 3 to 4 times more the greenhouse gases over lifetime use than its GHG footprint when compared to a board foot of EPS used to meet the same thermal requirements [8–10].

The comparison becomes even more difficult if one also includes fiberglass, mineral wool, and polyisocyanurate foams, each of which also possesses a specific and different environmental footprint. It is evident that improved definitions, standards, and specific criteria are needed to more clearly define product sustainability in a way that would be meaningful and useful to a potential user of insulation materials.

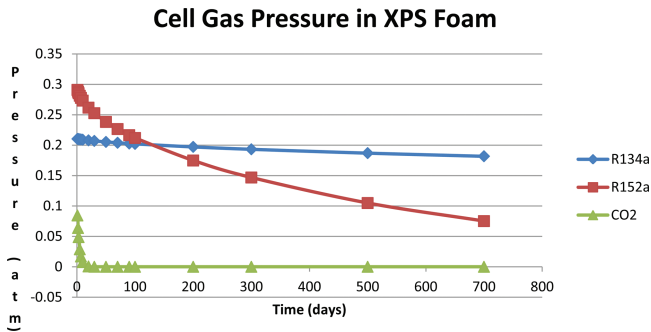
All insulation products have thermal performance claims (“*R* value,” “*k* factor,” or “lambda”), yet these may not completely reflect the insulation efficiency of the material. Thermal performance of insulation materials is dependent upon many factors including product age, specific application, and exposure conditions, including temperature, moisture, and installation details. It complicates the task of the buyer/user/builder in choosing an insulation material to address long-term insulation requirements. Therefore, it is necessary to clarify what long-term thermal performance means in terms of how insulation materials improve energy savings, so that the vast majority of buyers/users/builders think beyond “the higher the *R* value, the greater the insulating power.”

Long-Term Thermal Performance

In plastic foams, the blowing agents do not remain in the product forever and air is typically not used as a blowing agent. The rate at which air diffuses into foam and the rate a blowing agent leaves XPS foam is highly dependent upon the permeability of air and the blowing agents through the base polymer, with permeability being a product of the solubility and diffusivity. Figure 4 illustrates the effect permeability has on the amount of blowing agent remaining in a polystyrene foam with certain characteristics (e.g., density, cell size) as a function of time.

Most foam plastics have a moderate to high permeability to air. Because air typically has a higher thermal conductivity than many blowing agents, the *R* value of

FIG. 4 Evolution of partial pressure in XPS foam cells for three blowing agents with different permeability rates (units are $\text{cm}^3\text{-mil/day-100 in.}^2\text{-atm}$ at 25°C) ($\text{CO}_2 > \text{HFC-152a} > \text{HFC-134a}$).

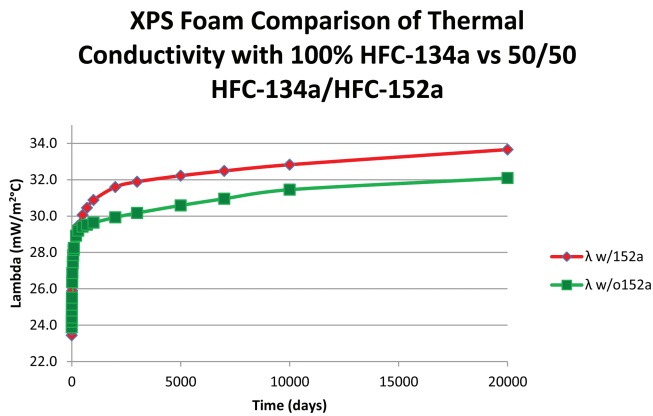


foam will decrease until the diffusion of air into the foam has stabilized. The rate of diffusion of CO_2 is extremely fast, and conventional foams do not benefit from its low thermal conductivity beyond a few days. Hydrofluorocarbon gases (HFCs) with zero ODP also greatly differ in their permeability characteristics. HFC-152 a (1,1-difluoroethane) diffuses out of the foam in the first few years, whereas HFC-134a's (1,1,1,2-tetrafluoroethane) lower permeability allows for most of it to remain in the foam for an extended period of time (100+ years), depending on polymer choices. Thus, the energy-saving benefit of using a lower GWP blowing agent, like HFC-152 a, is decreased because of its rapid release in the atmosphere, whereas the use of HFC-134 a will significantly enhance the insulation value of the foam and provide energy savings well beyond 50 years.

As a result, if the blowing agent has a thermal conductivity less than air, then the insulation value of the foam will also change as a function of time. This change in R value over time will be more rapid for more fugitive blowing agents, such as CO_2 , butanes, pentanes, and HFC-152 a. Knowing the permeability of the blowing agents and air in the polymer and the density and cell structure of the foam is often sufficient to estimate the change in R value over time. Using a more advanced proprietary model, this effect is illustrated for various examples in Figs. 5 and 6.

Figure 5 shows the benefit of reducing the level of fugitive blowing agent (HFC-152 a) on the long-term lambda value of 134 a-containing XPS foams. Both foams have an R value of $R\text{-5.0/in.}$ at 75°F mean temperature at 180 days, but the foam with only HFC-134a retains a higher R value over the useful life of the product. Moreover, if one considers the time it takes for insulation foam to be purchased, shipped, and installed, and then for a new home/building to be occupied, 180 days would be a reasonable starting point for calculating future energy savings/benefits. Integrating the area under the curves for the two cases in Fig. 5 yields an

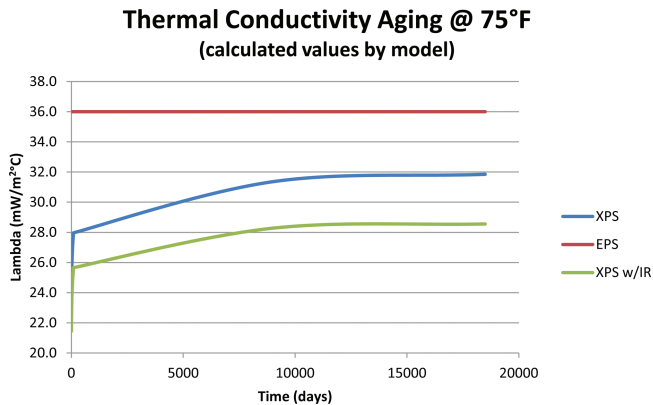
FIG. 5 Comparison of thermal conductivity change over time for two XPS foams of equivalent density, 100 % HFC-134a versus 50/50 HFC-134a/HFC-152a (calculated values by model).



improvement of 6 % for the HFC-134 a-only scenario versus the 50/50 blend of HFC-134a and HFC-152a over a period of 50 years. Yet, this difference is not apparent to the buyer/builder/building owner.

Figure 6 show another comparison, this time between a generic molded bead EPS (R-4.0/in.) that a builder might purchase at the local big box store or lumberyard and XPS foam using HFC-134 a as the only insulating blowing agent. Again, integrating the area under the curves and using 180 days as the starting point, the

FIG. 6 Thermal conductivity aging of various polystyrene foams at 75°F mean temperature.



XPS foam yields an improvement of 20 % versus the EPS foam over 25 years. This difference increases to 30 % if an infrared attenuating agent, like carbon black or graphite, is added to the XPS foam.

So how do all these data comparisons help the buyer/user/builder? According to the U.S. EPA [11], the average home spends \$1300 per year for heating/cooling and emits over 26|000 pounds of CO₂. A 20 % reduction in energy expenses yields a savings of \$260 per year and a proportional reduction in CO₂ emissions. So it is incumbent upon the buyer/user/builder to consider the overall impact of the insulation, *not* just the initial purchase cost [12,13].

Summary

Selection of blowing agents for XPS thermal insulation does include consideration of overall environmental impact, and this continually evolves as the environmental science improves and regulatory landscape changes. The net energy savings derived over the lifetime of a building through the use of thermal insulation needs to be weighed against the environmental footprint of the specific insulation product. Objective measures should be developed and implemented, e.g., by ASTM, universities, and/or governmental agencies, along with industry associations, to help define appropriate methodologies for these assessments.

The authors have focused their research effort for XPS foam to provide a long-term thermal performance solution that goes over and beyond short term *R*-value performance, which is required to meet current codes and standards. Many factors impact long-term thermal performance, including formulation, product quality, service environment, product age, and specific construction practices. The authors believe the use of blowing agents, such as HFC-134a, are sustainable and provide an overall benefit to society.

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Phalguni Mukhopadhyaya,¹ Minh-Tan Ton-That,²
Tri-Dung Ngo,² Nathalie Legros,² J.-F. Masson,³
Sladana Bundalo-Perc,³ and David van Reenen³

An Investigation on Bio-Based Polyurethane Foam Insulation for Building Construction

Reference

Mukhopadhyaya, Phalguni, Ton-That, Minh-Tan, Ngo, Tri-Dung, Legros, Nathalie, Masson, J.-F., Bundalo-Perc, Sladana, and van Reenen, David, "An Investigation on Bio-Based Polyurethane Foam Insulation for Building Construction," *Next-Generation Thermal Insulation Challenges and Opportunities*, STP 1574, Therese K. Stovall and Thomas Whitaker, Eds., pp. 131–141, doi:10.1520/STP157420130107, ASTM International, West Conshohocken, PA 2014.⁴

ABSTRACT

Bio-based renewable construction material is an old concept. Wood, straw, and other products of nature have been used for millennia around the world. However, in modern construction, the ratio of bio-based to non-renewable building materials is very low. This is primarily due to performance requirements. Purely bio-based construction materials sometimes have performance levels not quite equal to modern construction materials. The biggest challenge for the development of bio-based construction materials is to bring environmental friendliness and high engineering performance together in a single material. This paper presents results from a laboratory screening study on the development and the assessment of rigid partially bio-based polyurethane (PU) foams (seven different formulations) that contain lignin-based polyols, up to 20 % of polyol weight. The formulation strategy, morphology, and hygrothermal performance of rigid bio-based PU foams are presented and compared with the traditional

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¹National Research Council (NRC) Canada, Ottawa, ON, K1A 0R6, Canada (Corresponding author), e-mail: phalguni.mukhopadhyaya@nrc-cnrc.gc.ca

²National Research Council (NRC) Canada, Boucherville, QC, J4B 6Y4, Canada.

³National Research Council (NRC) Canada, Ottawa, ON, K1A 0R6, Canada.

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petroleum-based reference PU foam. This study demonstrates that partially bio-based rigid PU foam with appropriate formulation can have characteristics that may be suitable for the construction industry applications. For market acceptance, further investigation is needed on long-term performance and durability.

Keywords

thermal insulation, polyurethane foam, bio-based building materials, bio-foam, hygrothermal performance

Introduction

Growing concerns about climate change and rapid depletion of non-renewable resources provide an impetus to use renewable materials in the construction industry. The National Research Council (NRC) of Canada has a strong R&D program on the development of bio-based raw materials and the use of bio-based products for industrial applications. The primary focus of this program is on construction and automotive applications.

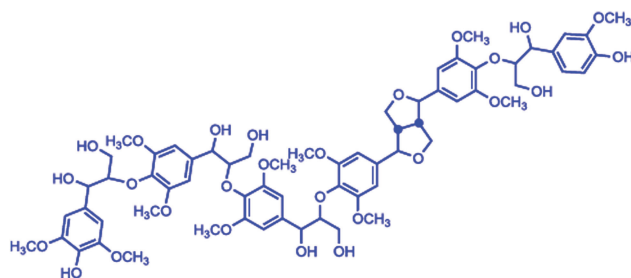
The use of bio-based renewable construction materials is not a new concept for the construction industry as these materials have been widely used as various building components around the world for ages. However, in modern construction, the ratio of bio-based to non-renewable building materials is very low. This is primarily due to the performance requirements. Purely bio-based construction materials have performance levels not quite equal to modern construction materials. The biggest challenge for the development of biobased construction materials is to bring environmental friendliness and performance together in a single material. Berge [1] has recently produced an extensive review of the opportunities for ecomaterials for construction.

Research Background

Lignin is an important constitutive component of plant, wood and some algae. Lignin is an amorphous, complex, and highly branched polyalcohol-phenolic macromolecule (Fig. 1). Lignin is available in large quantities as a byproduct of the paper-pulp and textile fiber industries. Lignin is the second most naturally abundant biopolymeric substance after cellulose. During industrial processing, lignins undergo significant structural changes and byproduct lignins are no longer identical to their original native structures [2–5]. Due to its complex nature and undefined chemical structure, the industrial applications of lignin are rather limited. Presently, lignin is utilized almost exclusively as fuel to power the evaporators of the chemical recovery processes and liquor concentration system of pulp mills [6].

The ultimate goal of this research initiative is to find value-added applications for lignin, and more specifically to produce bio-based polyurethane (PU) foam that

FIG. 1. Representative macromolecular structures in lignin, a natural polyol and polyphenol.



can be used as thermal insulation in buildings. At present, research is being conducted to partially replace petroleum-based polyol in PU foam with the ligno-polyol developed from commercially available non-food grade lignins. However, the impact of the presence of bio-based ligno-polyol in PU foam on its short and long-term performance, particularly for the application in buildings, is a matter of concern for building designers and construction professionals.

Formulation Strategy

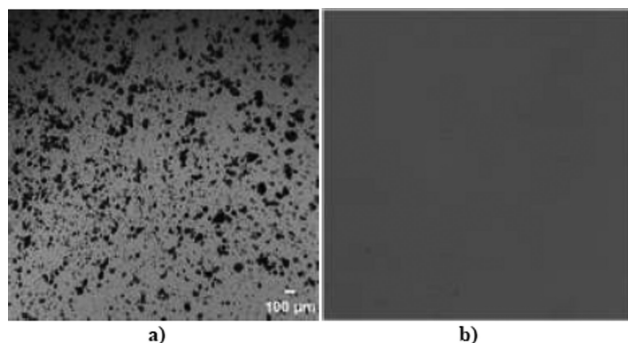
Kraft lignin from a commercial source was used in this study. It was dried at 60°C under vacuum for at least 4 h prior to use. A proprietary polyol blend was prepared from a mixture of commercial polyester and polyether polyols. Lignin was dispersed into polyol by different means with low, medium, and high shear mixers [7–9]. The obtained lignin-polyol mixture was mixed with catalyst additives and then with isocyanate to form cellular PU foams.

Foam Morphology

An optical microscope (OM) was used to observe the dispersion of lignin into the polyol. The viscosity of the polyol-lignin was measured with a viscometer. A scanning electronic microscope (SEM) was used at a low voltage of 5 kV to view the morphology of the PU foams after their surface had been covered with a thin conductive gold/palladium coating. The opening cell content and density of the foams were measured according to ASTM D6226 [10].

Figure 2(a) shows discrete lignin particles after low shear mixing with the liquid polyols. By increasing the shear, the large lignin particles essentially disappeared (Fig. 2(b)); however, it remains unclear if they really dissolved in the polyol or if the particle sizes are too fine to be detected by OM. It is possible that with the aid of high shear force lignin particles could be easily broken into smaller particles

FIG. 2. Optical microscope image of lignin-polyol right after mixing at (a) low shear and (b) high shear.



and even can dissolve into the polyol if the thermodynamics of the system is favorable.

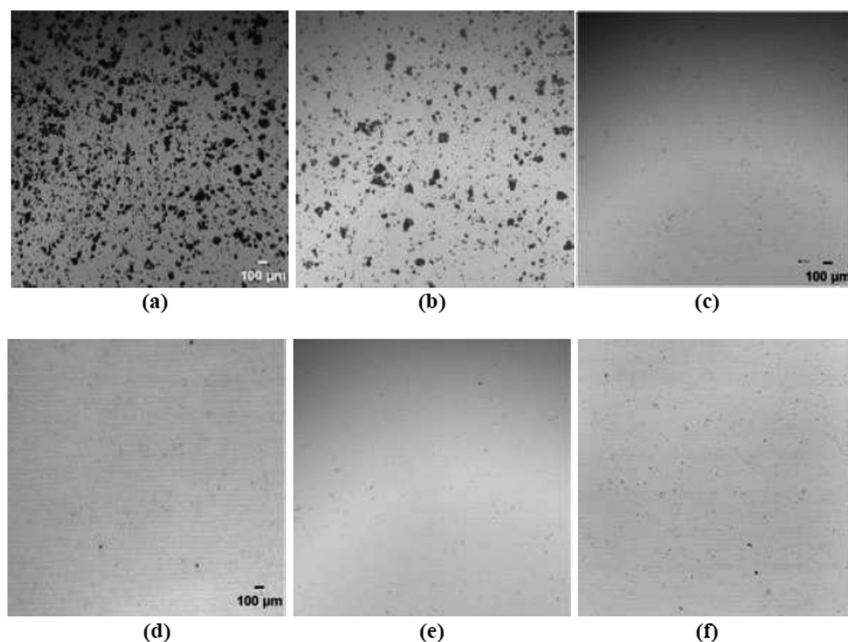
The long-term performance of the ligno-polyol PU foams will greatly depend on the lignin solubility in the polyol with time. The evolution of the lignin dispersion in polyol prepared at low shear during storage is shown in Fig. 3. As it can be seen, a number of lignin particles disappeared while the other continued to swell up (Fig. 3(b)) and eventually dispersed further in the polyol even without any external shear force or elevated temperature. This demonstrates that the proprietary polyol had good compatibility with the selected lignin, thus facilitating the dispersion of lignin into polyol during storage.

The SEM images of the foams are shown in Fig. 4, illustrating the cellular morphology, in the direction of foam rise, at different magnifications. In general, the cellular structure of the foam is identical in both cases, with and without lignin, which consists of regular round and closed cells. There is no significant change in the cellular size and shape with the presence of lignin. In addition, no evidence of cellular collapse with the presence of lignin can be observed. This indicates the presence of lignin in the appropriate formulation does not alter the formation of the foam cells. In addition, no lignin particles could be found in the bio-foams. One could speculate that lignin has been assimilated into the PU network structure.

Hygrothermal Performance

The performance of foam insulation to be used in building envelope construction is critically dependent on its hygrothermal (i.e., moisture/water and heat) performance. A series of investigations have been carried out to assess the hygrothermal

FIG. 3. Optical microscope image of lignin-polyol prepared at low shear: (a) right after mixing, and after a number of days: (b) 2 days, (c) 7 days, (d) 14 days, (e) 21 days, and (f) 28 days.



performance of the newly developed bio-based PU foam insulation. These investigations were carried out to determine water vapor permeability, thermal conductivity, and sorption/desorption properties of laboratory made small-scale foam specimens.

Eight foam samples were fabricated in paper cups (Fig. 5) for this study with five different types of lignin at two different lignin concentrations 10 and 20 % (weight) of the polyol. One of them was reference petroleum-based PU foam and the other seven were bio or ligno-polyol based PU foams. After fabrication, foam samples were stored for about 60 days in controlled laboratory environment (50 ± 5 % RH; $22 \pm 1^\circ\text{C}$ temperature). Table 1 lists respective tested samples and related physical properties/descriptions. After removal of the foam from the cup, each cup-shaped foam had top and bottom diameters of 105 mm and 90 mm, respectively, with a height of 170 mm (Fig. 6).

Six rectangular (40 mm by 40 mm) specimens of 12 mm thickness from each foam sample were used for the determination of sorption isotherms. Three circular (70 mm diameter) specimens of 12 mm thickness from each sample were used for

FIG. 4. SEM image of the foams: (a), (b) without and (c), (d) with lignin at two different magnifications.

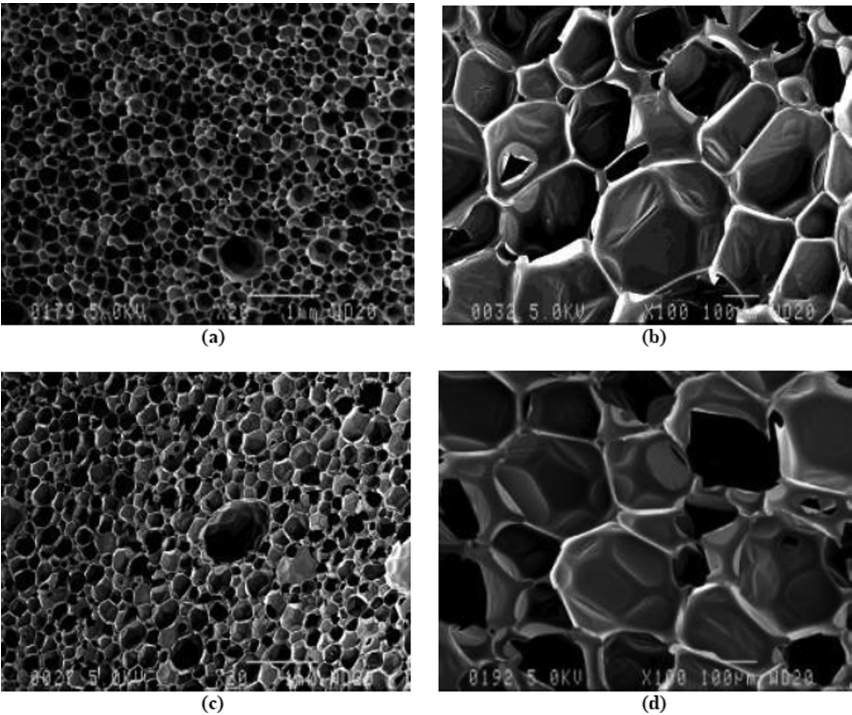


FIG. 5. Typical PU foam samples in a paper cup.



TABLE 1. Bio-foam insulation samples received.

Sample Name	Density (kg/m ³)				Lignin Type*	Lignin Concentration (% of polyol weight)
	Overall	WVP Specimens	Thermal Conductivity Specimens	Sorption Specimens		
Reference Foam	36.5	32.97	32.24	31.70	–	–
Bio-foam 1	36.1	32.84	32.54	31.92	Lignin 1	10 %
Bio-foam 2	39.0	36.06	33.94	33.85	Lignin 1	20 %
Bio-foam 3	39.1	35.57	34.39	33.81	Lignin 2	10 %
Bio-foam 4	38.3	34.29	34.14	32.85	Lignin 3	10 %
Bio-foam 5	41.0	38.34	37.14	36.46	Lignin 3	20 %
Bio-foam 6	40.0	37.38	36.18	34.53	Lignin 4	10 %
Bio-foam 7	38.6	35.50	33.82	34.29	Lignin 5	10 %

the determination of water vapour permeability. For thermal conductivity measurement, one rectangular (70 mm by 70 mm) specimen of 12 mm thickness was cut from each foam sample. These specimens were taken from various regions of the foam sample as shown in Fig. 7 and densities of these specimens are shown in Table 1. While specimen sizes, for sorption and water vapour permeability, conform to the requirements of corresponding standards (ASTM E96 [11] and ASTM C1498 [12]), the size of the foam specimen for thermal conductivity measurement is far smaller than the minimum required (300 mm by 300 mm). A new test method was developed, reported in an earlier publication [13], where traditional

FIG. 6. Typical dimensions PU foam samples.

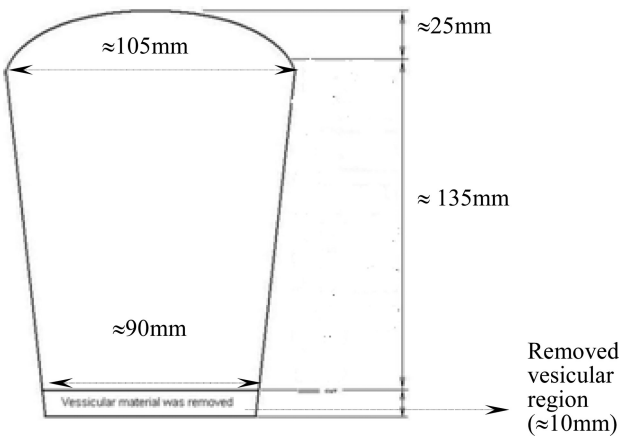
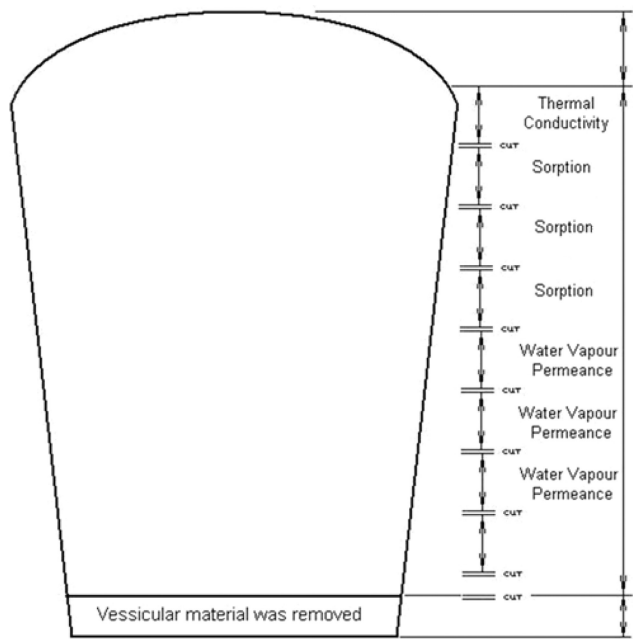


FIG. 7. Locations of test specimens.



heat heat-flow-meter (300 mm by 300 mm with a metering area of 150 mm by 150 mm) was used to measure thermal conductivity of 70 mm by 70 mm (12 mm thickness) specimens.

Hygrothermal responses of bio-foams (i.e., seven ligno-polyol based PU foams) as characterized by thermal conductivity and water vapor permeability are shown in Table 2, and sorption isotherm up to 95 % RH (relative humidity) data points are shown in Fig. 8.

The dry cup water vapor permeability values, measured at four different chamber RHs, of seven bio-foam specimens and one reference petroleum-based PU foam specimen are found to be of same order of magnitude. The water vapor permeability values for all of them also increase with increasing chamber RH. In general, the water vapor permeability values of bio-foams are not significantly different from the values obtained for petroleum-based PU foam.

The thermal conductivity values of seven bio-foams and reference petroleum-based PU foam compared in Table 2 indicate very little thermal conductivity difference due to the presence of bio-based ligno-polyol in PU foam. However, in general (except in one case), there is a trend of slightly increasing thermal conductivity due to the addition of ligno-polyol in PU foam. With the

Conclusions and Scopes for Future Investigations

In general, the results and their critical interpretations from a series of studies on formulation strategy, morphology, and hygrothermal performance of ligno-polyol or bio-based PU foam insulation presented in this paper have demonstrated that well formulated bio-based foam insulation, with up to 20 % ligno polyol (% of polyol weight), can have a similar foaming characteristics and satisfactory initial performance as the conventional PU foam insulation fabricated under the same conditions.

Notwithstanding, further investigations are needed on long-term performance, bio-degradability and durability of ligno-polyol based PU foam insulation to move these materials closer to market place.

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Nitin Shukla,¹ Peng Cao,¹ Ramin Abhari,² and Jan Kosny¹

Lab-Scale Dynamic Thermal Testing of PCM-Enhanced Building Materials

Reference

Shukla, Nitin, Cao, Peng, Abhari, Ramin, and Kosny, Jan, "Lab-Scale Dynamic Thermal Testing of PCM-Enhanced Building Materials," *Next-Generation Thermal Insulation Challenges and Opportunities*, STP 1574, Therese K. Stovall and Thomas Whitaker, Eds., pp. 142–154, doi:10.1520/STP157420130090, ASTM International, West Conshohocken, PA 2014.³

ABSTRACT

Previous research studies have shown that incorporation of the phase-change material (PCM) in a building envelope material/component may bring significant reduction in the building energy consumption. A detailed knowledge of the key phase-transition (dynamic) properties, such as latent heat, sub-cooling, hysteresis during melting and freezing, etc., of the PCM-enhanced building materials is required to perform the whole building energy simulations and code work. In addition, the dynamic test data is critical in optimizing the distribution and location of the PCM within a building to maximize the energy savings. Until recently, the differential scanning calorimeter (DSC) has been the only available method to determine the dynamic properties of a PCM. Unfortunately, the DSC method is valid for small and homogeneous specimens, and is incapable of capturing the complexities observed in large-scale building components. Materials with non-uniform temperature distribution and non-homogeneity caused by the presence of additives, such as fire retardants, conduction inhibitors, and adhesives, cannot be analyzed by the DSC testing method. Dynamic heat-flow meter apparatus (DHFMA) is a recently developed method for dynamic property measurement of system-scale PCM and other building construction products. Although the DHFMA method is gaining acceptance among the scientific and research community, it is

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¹Fraunhofer Center for Sustainable Energy Systems, Boston, MA 02130, United States of America.

²Syntroleum Corporation, Tulsa, OK 74135, United States of America.

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still under development. In this study, we focus on advancing the development, and conducting the validation of the DHFMA method. A detailed description of the DHFMA method is presented to highlight the difference with the conventional HFMA method. Next, a large-scale bio-based shape-stabilized PCM (ss-PCM) sample was tested using both DHFMA and DSC test methods. Specific heat as a function of temperature data measured by DHFMA method was found to be in very good agreement with slowest ramp and step data. This is the first direct verification of the HFMA method with the DSC method for PCMs.

Keywords

phase change materials, building materials, heat flow meter apparatus, heat capacity

Introduction

Phase change is a process where a material transforms from one phase into another. The process is accompanied by absorption or release of large amounts of latent heat at a constant temperature defined as the phase-change temperature. A material that uses its phase-changing ability for the purpose of heating, cooling, or temperature stabilization is defined as a phase-change material (PCM). PCMs have found applications in several areas such as thermal energy storage, building energy efficiency, food products storage, spacecraft thermal systems, solar power plants, microelectronics thermal protection, and waste heat recovery [1–6]. In buildings, introduction of PCMs in the building enclosure has been shown to reduce the building energy consumption as PCM helps maintain and regulate the interior temperature [7–12]. Field studies conducted across various locations in the United States have demonstrated that application of PCM in a building envelope causes reductions in the peak-hour loads and shifting of the peak demand time. Cooling energy savings of up to 25 % have been demonstrated with the application of PCM in a building [8].

Currently, several types of PCMs are available in the market for building applications with different dynamic or phase-change properties such as phase-change temperature, latent heat, sub-cooling, and hysteresis. In the majority of building applications, PCM in its pure form is integrated with a building component such as insulations or wall boards. The integration is achieved mainly in two ways: (1) uniformly or randomly dispersing small-sized PCM elements (microcapsules, millimeter-sized pellets) into the building element, or (2) using concentrated large-sized PCM elements (cm-thick layers, cm-sized pockets) into the building element. At the system scale, dynamic thermal properties of the PCM-integrated component is dependent on several, often unknown, factors such as mass fraction of the PCM, heat capacity and thermal conductivity of different parts of the component, and presence of additives (fire retardants, conduction inhibitors, adhesives). In addition, the dynamic properties of PCM itself may change because of surrounding materials and introduction of foreign materials. Therefore, dynamic properties of PCM-integrated components may be significantly different than the one derived using pure PCM dynamic properties.

An accurate knowledge of the dynamic properties of PCM-integrated components is critical in predicting the energy-saving potential of the PCM technology on a whole-building scale [13,14]. Conventionally, a differential scanning calorimeter (DSC) has been used to measure the dynamic properties of a PCM. However, the DSC method is applicable for specimens with typical sizes of millimeter length scale and mass on the order of milligrams. The DSC method also requires that specimen is relatively homogeneous in composition. System-scale building components are significantly larger in both the length and mass scales, and are often too inhomogeneous in composition to be used as a test specimen in DSC. In addition, the DSC system may become unreliable and inaccurate for slow measurements imitating the temperature profile experienced by the building (about $0.1^{\circ}\text{C}/\text{min}$) [15,16]. Data obtained by the DSC is often used in computer models to analyze PCM materials, leading to significant inaccuracies in the performance evaluation.

A heat-flow meter apparatus (HFMA)-based method, commonly referred to as dynamic HFMA or DHFMA, has been recently developed to measure the dynamic properties of large-scale PCM-integrated components and products [17–19]. The DHFMA method is suitable only for solid-to-liquid phase transitions. Conventional HFMA systems use heat-flow transducers (sensors) to measure heat-flow rates at steady state, allowing determination of the steady-state heat-transmission characteristics of building materials as described in ASTM C518 [20]. Conventional HFMA systems may use one or more heat-flux sensor and specimen as specified in ASTM C518. On the other hand, the DHFMA method is applicable for HFMA systems that have at least one heat-flow sensor on each of the isothermal plates. DHFMA is a variation of the HFMA method as it uses the same heat-flow sensor in rather a different manner. In DHFMA, heat-flow sensors record heat-flow rate as the sample is exposed to a temperature step. The heat-flow data is integrated over time as equilibrium is achieved, providing enthalpy change during the temperature step. In principle, this method may be applied for specific heat measurement of any material including PCM and products. Although DHFMA has been used in several research laboratories across North America to investigate PCM-integrated building components, the method is still under development and undergoing continuous modification [18,19,21]. To date, there has been no study performed to determine the accuracy of this method in part because of a lack of availability of a well-established enthalpy measurement method for large-sized specimens. In addition, there is need to investigate complex PCMs that exhibit multiple peaks or significant sub-cooling to be able to determine the limitations and improve the measurement capability of the method. These improvements will help advance the development of this method, allowing scientific and engineering community access to a reliable and accurate measurement tool for PCMs and PCM-enhanced products.

In this study, we focus on development of DHFMA methods to improve the accuracy and resolution of the measurements and to verify the DHFMA with the DSC method. For the purpose of this study, we investigated a shape-stabilized PCM

(ss-PCM) because it can be synthesized in a large-sized system-scale sample with an almost similar composition as in a small-sized material-scale sample. This allowed us to directly compare the DHFMA method with the DSC data obtained on the small-scale sample. To our best knowledge, this is the first study to validate the accuracy of DHFMA method with a well-established DSC method. The studied ss-PCM is paraffin derived from abundant bio-renewable feeds such as vegetable oils and animal lard.

Differential Scanning Calorimeter (DSC)

DSC measures heat flow in and out of a sample as a function of time and temperature during a material transition relative to a reference sample. This information is used to calculate dynamic properties and heat capacity of a PCM as the material undergoes phase transition. DSC uses small mass samples on the order of milligrams. A reference is tested under the same conditions to subtract the effects of a sample holder from the heat-flow signal. This leads to higher precision in the determination of the heat flow into the sample [22]. According to the first law of thermodynamics, during any incremental process:

$$(1) \quad dH = dQ + Vdp$$

where:

dH = the change in the enthalpy of the system,

dQ = heat supplied to the system,

V = the volume of the system, and

dp = the incremental change in the pressure of the system.

For constant pressure:

$$(2) \quad dH = dQ$$

Therefore, the phase-change enthalpy can be determined by measuring the heat flow to the system.

Furthermore, heat capacity can be determined by taking the derivative of enthalpy with temperature T :

$$(3) \quad C_p = dH/dT = dQ/dT$$

Both the sample and the reference are placed in the same testing chamber with constant pressure. Usually the weighted sample is put in a small pan, whereas the reference is in an identical empty pan. The thermocouples located under the sample and reference measure the corresponding temperatures. The temperature difference developed across sample and reference is proportional to the heat flux caused by enthalpy or heat-capacity changes. The signals are recorded, and the difference in the heat flux between the sample and reference is analyzed and transferred to the data-acquisition system so that any heat flux not related to the sample can be

cancelled. The heating process is usually achieved by an internal furnace or electric resistance, whereas the cooling process is usually achieved by an external cooling device such as a refrigerator or a chillier.

DSC is usually run in scanning or ramp mode where the sample is subjected to a constant heating or cooling rate, and heat flow through the sample is measured. The heat capacity is then calculated by dividing heat-flow rate by ramp rate, i.e., dividing both the numerator and the denominator terms in Eq 3 by time interval dt :

$$(4) \qquad \qquad \qquad C_p = (dQ/dt)/(dT/dt)$$

The accuracy of dynamic mode is significantly affected by the ramp rate. It is important to note that as a result of finite thermal mass of the sample, thermal response of the DSC may lag behind the ramp input, resulting in ramp-rate-dependent dynamic property data. A high ramp rate will give a very inaccurate result. A general drawback of the ramp method is that during phase transition, the sample is not isothermal. This may lead to a shift of several degrees Kelvin of the indicated heat-storage capacity.

To study phase-transition processes, DSC may be run in isothermal step mode. The temperature of the sample is changed in increments or steps, and heat flow during the step change is measured. Heat capacity may then be calculated using Eq 3. The step method gives very accurate measurements of PCM enthalpy as a function of temperature, provided that sufficient time is given during each temperature step change to ensure negligible heat flow at the end of the of each measurement. Usually a step test generates a curve of specific heat versus temperature or time. The integration of heat flux with respect to time gives the enthalpy of the phase-change process.

The DHFMA Principle

In principle, the DHFMA test method is similar to the step-mode DSC method, but is applicable for large-scale samples such as building components. DHFMA utilizes temperature and heat-flux information from a conventional HFMA to determine the dynamic thermal properties of PCM-enhanced components. The conventional HFMA method is based on the specification described in ASTM C518 [20]. The DHFMA method is an upgrade to the previously developed rapid temperature ramp methodology based on HFMA that was developed to test PCM-enhanced fiber insulations [17], and allowed for more accurate test data analysis. The upgrades improved the accuracy of the results with minimal modification to the existing equipment and requiring no costly hardware upgrades.

In principle, a phase-transformation event in a PCM-enhanced material system can be studied using the heat balance equation for the material system with the consideration of temperature-dependent specific heat [23,24]. The one-dimensional heat-transport equation for such a case is:

$$(5) \quad \frac{\partial}{\partial t}(\rho h) = \frac{\partial}{\partial x} \left[\lambda \frac{\partial T}{\partial x} \right]$$

where:

ρ and λ = the material density and thermal conductivity, respectively, and
 T and h are temperature and enthalpy per unit mass, respectively.

Considering a constant pressure during the thermal event, which is a valid assumption for a solid–liquid phase-change process, the effective heat capacity, c_{eff} , is defined as the derivative of the enthalpy (including latent and sensible heats) with respect to the temperature:

$$(6) \quad c_{\text{eff}} = \frac{\partial h}{\partial T}$$

For most PCMs, an enthalpy profile with temperature is dependent on the direction of the phase-change process, and oftentimes, enthalpy profile during melting is different than that during solidification. Therefore, it is important to consider separate temperature-dependent specific heat functions for melting and solidification in the thermal design of the PCM-enhanced material. Effective heat capacity for a material that is a blend of a PCM, and its carrier material may be expressed as:

$$(7) \quad C_{\text{eff}} = (1 - \alpha)C_{\text{carrier}} + \alpha C_{\text{effPCM}}$$

where:

α = the percentage of PCM,
 C_{carrier} = the specific heat of the carrier without PCM, and
 C_{effPCM} = the effective heat capacity of PCM.

In the liquid state, the effective heat capacity of PCM does not show temperature dependence; it may be thus represented as the sum of two terms:

$$(8) \quad C_{\text{effPCM}}(T) = C_l + (C_{\text{effPCM}}(T) - C_l)$$

where:

C_l = the temperature-independent specific heat in the liquid state.

As mentioned previously, a conventional HFMA is used to measure steady-state thermal properties following ASTM C518. In general, an HFMA consists of two isothermal plate assemblies with one or more heat-flux transducers bonded to each plate. The plate temperatures are controlled using equipment such as thermoelectric elements and water chillers. In the DHFMA method, top and bottom plates are set to the same temperature, unlike the conventional HFMA method (following ASTM C518) where top and bottom plates are set to different temperatures to impose a temperature gradient on the specimen. Plate temperature and plate heat-flow rates of the top and bottom plate, QT and QB , are recorded at time interval, τ , by thermocouples and heat-flow transducers, respectively, for each plate. Each temperature step is allowed to continue until the thermal equilibrium condition is reached. Considering a constant pressure, enthalpy H (in terms of heat per unit square of surface area), is determined by integrating heat-flow rates over time:

$$(9) \quad H = \sum [H_i + [(QT_i - QT_{\text{final}})S_T + (QB_i - QB_{\text{final}})S_B]\tau]$$

where:

QT_{final} and QB_{final} = the residual heat-flow signals from the upper and lower plates, respectively, at equilibrium that are subtracted from the signal of interest to eliminate drift caused by small edge heat losses, and

S_T and S_B = the calibration factor for top and bottom plates, respectively.

Effective volumetric heat capacity of the specimen, C_{eff} (in terms of heat per unit volume per unit temperature change), is defined as a derivative of the enthalpy with respect to the temperature. In other words, effective volumetric heat capacity of the PCM-enhanced building component can be determined by taking the slope of the enthalpy-temperature curve as follows:

$$(10) \quad C_{\text{eff}} = \frac{1}{L} \frac{dH}{dT} \approx \frac{1(H_{i+1} - H_i)}{L(T_{i+1} - T_i)}$$

where:

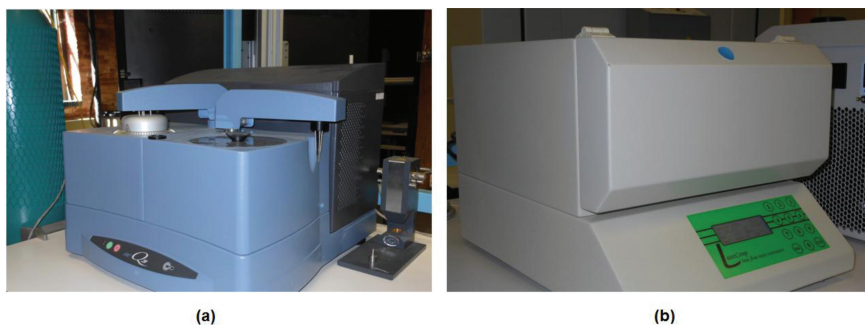
L = the thickness of the specimen.

Results

The ss-PCM selected in the study was a paraffinic composition synthesized from biological wastes, and further stabilized in a polymer matrix. A TA Q20 DSC system (see Fig. 1(a)) was used to study small mm-sized pellets of ss-PCM (see Fig. 2(b)). The pellets were cut into small pieces of 20–30 mg to be able to fit into sample holder. First, the product is tested in ramp mode with temperature rates of 10, 5, 1, and 0.2°C/min. Temperature ramps of 0.1°C/min and below compromise the accuracy of the data; hence, are not considered in this study. Figures 3 and 4 show the specific heat of the sample as a function of temperature during the melting and the solidification phase-change process. It is observed that the specific heat curve shifts to left and right for melting and solidification processes, respectively, and the phase-change temperature span narrows down with slower ramps. These observations are in agreement with previous experimental studies performed on other configuration of PCMs [22,25,26]. The enthalpy change during the phase change is defined as the heat capacity data integrated over a phase-change temperature span. It is found that although curves shift with ramp rate, the enthalpy change remains the same. In essence, distribution of the latent heat absorption or release is governed by the ramp rate.

Next, the ss-PCM sample is tested in the step DSC mode, and the results are shown in Figs. 3 and 4. Enthalpy changes during melting and solidification are found to be 120 and 95 J/g, respectively, agreeing very well with the ramp results. Melting and solidification data for the slowest ramp used in the study, i.e., 0.2°C/min matches closely with the corresponding step data. This suggests that 0.2°C/min temperature ramp is slow enough to ensure uniform temperature distribution

FIG. 1 Experimental apparatus employed to determine dynamic thermal properties of PCM-enhanced materials: (a) differential scanning calorimeter (DSC), and (b) LaserComp FOX200.



within the sample. For the melting process, the peaks of slowest ramp and step mode curves are offset by $\sim 0.5^{\circ}\text{C}$, which is within the temperature resolution of 1°C used for the step mode during melting. On the other hand, for the solidification process, the slowest ramp exhibits multiple peaks in the enthalpy curve with the first and the highest peaks occurring at $\sim 24.5^{\circ}\text{C}$ and 23°C , respectively. The step mode shows three peaks in the enthalpy profile with both the first and the highest peaks occurring at $\sim 25^{\circ}\text{C}$.

Next, we used the LaserComp FOX200 HFMA instrument (see Fig. 1(b)) to investigate the dynamic properties of the same composition ss-PCM as was used in the DSC experiments, but available as sheet rolls of 1 mm thickness (see Fig. 2(b)). To be able to fit into an HFMA instrument, $20\text{ cm} \times 20\text{ cm}$ samples were cut from the sheet roll. To improve signal-to-noise, four such samples were stacked to create

FIG. 2 The ss-PCM samples used in the study: (a) pellets, and (b) roll of sheet.

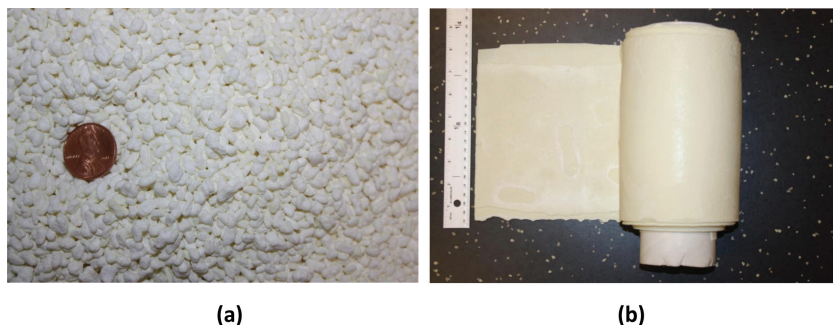
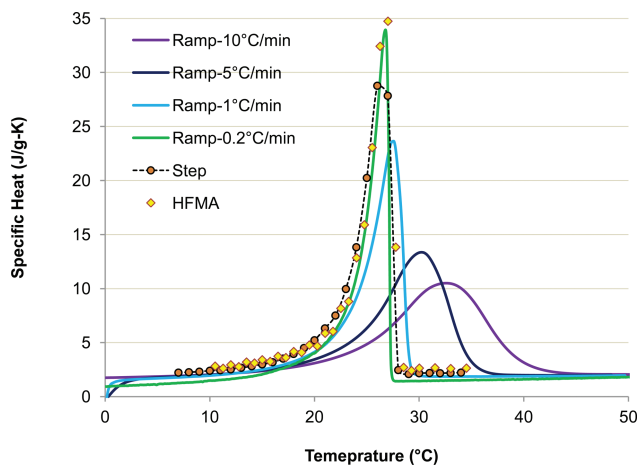
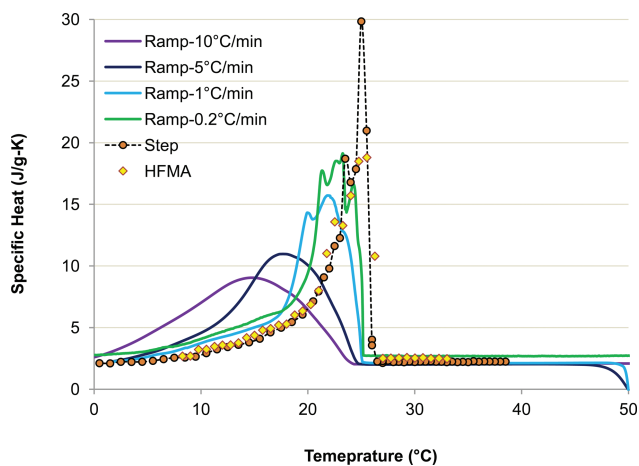


FIG. 3 Specific heat as a function of temperature during melting for ss-PCM in pellet and sheet forms. Pellets were measured using the DSC method in both the ramp and step modes, while sheet was measured using the DHFMA method.



a 4-mm-thick test sample. It is evident from the DSC experiments that the temperature resolution of less than 1°C is required to capture the contours of the specific heat curve. Because the accuracy of heat-flow sensors used in a typical HFMA

FIG. 4 Specific heat as a function of temperature during the solidification process for ss-PCM in pellet and sheet forms. Pellets were measured using the DSC method in both the ramp and step modes, while sheet was measured using the DHFMA method.



instrument becomes questionable for temperature steps smaller than 1°C , it is not possible to use temperature steps of less than 1°C in the HFMA method. To improve the temperature resolution below 1°C , two separate tests covering the phase-change span were conducted. Although both tests used temperature steps of 1.5°C , there was a difference of 0.75°C between the first set point of the two tests. The specific heat data of the two tests were then combined to obtain an overall temperature resolution of 0.75°C . Figures 3 and 4 include the HFMA results for melting and solidification, respectively. A close match between DHFMA and DSC data is observed, demonstrating the accuracy of the DHFMA method. The slight difference observed between DHFMA and DSC curves is well within the range of temperature resolution adopted for these two tests.

A closer look at the melting data reveals that DHFMA provides more precise determination of the peak temperatures compared to the step DSC method. This is attributed to the fact that HFMA tests used higher temperature resolution of 0.75°C compared to 1°C used for step DSC. On the other hand, for solidification, step data shows much higher and much clearer temperature peaks than the HFMA because temperature resolution is 0.5°C near the peak phase-change regime as compared to 0.75°C for HFMA. These results demonstrate that, for the studied ss-PCM, the HFMA method is as accurate as the DSC method, which is a very well-established method.

Conclusions

Previous numerical and experimental studies have shown that inclusion of PCM in the building envelope may result in improved building energy performance. However, a detailed knowledge of dynamic properties of the PCM is required to perform the whole building energy simulations. In addition, the dynamic test data is critical in optimizing the distribution and location of the PCM within a building to maximize the energy savings. Until recently, DSC has been the only available method to determine the dynamic properties of a PCM. Unfortunately, the DSC method is valid for small and homogeneous specimens, and is incapable of capturing the complexities observed in large-scale building components. Materials with non-uniform temperature distribution and non-homogeneity, because of the presence of additives such as fire retardants, conduction inhibitors, and adhesives, cannot be analyzed by the DSC testing method. DHFMA is a recently developed method for dynamic property measurement of system-scale PCM and products. Although the DHFMA method is gaining acceptance in the scientific and research community, it is still under development. In this study, we focus on further development and validation of the DHFMA method. A description of the DHFMA method is described. In principle, heat-flow signals from heat-flow sensors are integrated over time to determine enthalpy change during a temperature step change.

A bio-based ss-PCM is investigated because it can be synthesized in both small pellets and large-scale sheet forms that can be tested using DSC and DHFMA

methods. The flexibility of the bio-based ss-PCM allows a direct comparison between these two methods. First, a series of DSC tests in ramp and step modes is performed to evaluate the dynamic properties of ss-PCM. For the melting process, the peak of the specific heat data as a function of temperature shifted to lower temperatures with slower temperature ramps, whereas for solidification it shifted to higher temperatures. This is because of the fact that the sample has finite thermal mass, causing the heat-flow signal to lag behind temperature input. Slower ramps allow more time for the specimen to reach equilibrium, representing a true dynamic response from the PCM. This is verified by comparing data obtained using the slowest ramp ($0.2^{\circ}\text{C}/\text{min}$) with the step data. Next, dynamic properties of a 20 cm long, 20 cm wide, and 0.4 cm thick ss-PCM sample was investigated using a FOX200 HFMA system.

To improve the temperature resolution of the measurements to 0.75°C , two separate tests covering the phase-change regime were performed for both melting and solidification processes. Each of these tests used the same temperature steps of 1.5°C , whereas a difference of 0.75°C was kept between the first set point of the two tests. For the investigated ss-PCM, the specific heat as a function of temperature data measured by the DHFMA method was found to be in very good agreement with the slowest ramp and step data. This is the first direct verification of the HFMA method with DSC method for PCMs.

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Charles Petty¹

Presentation at ASTM C16 Symposium on Next-Generation Thermal Insulation Challenges and Opportunities

Reference

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ABSTRACT

Vapor retarder materials of extremely low permeance are specified and used in many insulation applications. For mechanical systems operating at below ambient conditions, such materials are required to minimize intrusion of water vapor into the insulation; the lower the operating temperature, the more critical the vapor retarder function becomes. Some of these materials exhibit water vapor permeance of under 0.01 perm, and some are literally impermeable. The question of how reliable test method E96 is for testing such materials is often asked. In 2010, the task group under Committee C16 for thermal insulation that is responsible for E96, "Standard Test Methods for Water Vapor Transmission of Materials," undertook an Inter-laboratory Study (ILS), or "round robin," in which four materials of extremely low permeance were tested. The objective of this ILS was to develop a precision and bias (P&B) statement to characterize the robustness of the test for evaluating such materials, and to see what problems the labs experienced in the course of testing at this low level of water vapor transmission. The data obtained by the six participating labs and the statistical analysis of it would suggest that good precision can be obtained and it can serve the need for testing at these low levels. If performed with careful sample

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¹Lamtec Corporation, Mount Bethel, PA 18343, United States of America.

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preparation, good control of conditions and close attention paid to data generated during the test, reliable results can be obtained. However, erroneously high results are not uncommon, and a critical take-away is the need for recognition of those outliers and the cause of them. As a related matter, within ASTM and the insulation industry there has been discussion of what defines, and how to classify, so-called “zero perm” vapor barriers. An overview of this topic is presented herein.

Keywords

vapor retarder, vapor barrier, zero perm, dry cup, permeance, water vapor transmission, below ambient, partial vapor pressure

Introduction

Thermal insulation serves the critical function of assuring and enhancing heating and cooling efficiency and conserving energy. It is one the most, if not *the* most, cost-effective means of saving energy that can be employed in both inhabited structures and in industrial and commercial mechanical systems. The “bang for the buck” provided by thermal insulation is enormous. This is probably preaching to the choir for the audience of this paper.

Providing for the proper insulation configuration to obtain the desired balance of initial cost, pay-back, and performance is the primary objective of the insulation specifier. However, just as important is the consideration of what is required to assure that the insulation performs as intended for the length of its service life.

Ideally, the insulation should be expected to perform consistently for the life of the structure or system that is being insulated. Especially in the case of insulation on mechanical systems, however, physical abuse, weathering, and moisture vapor intrusion on below-ambient systems cannot be avoided, and is expected to limit the useful life of the insulation. This discussion concerns the last-noted aspect of life-limiting causes.

In the case of a conditioned structure, a vapor retarder can be employed on the outside of the insulation or the inside, or not at all. Varying geographical locations, climate conditions, interior conditions, and structural design determine how and where a vapor retarder is used and located in the structure. The criticality of the use of a vapor retarder, level of performance, and installation technique varies according to the combination of the above factors.

In building structures—in most situations—use of an extremely high barrier (extremely low permeance) material for the vapor retarder is not necessary, although one may be employed when special conditions demand it or economics allow it. On the other hand, mechanical systems or industrial processes which operate continuously at below ambient temperatures present very challenging requirements for vapor retarder materials. Such systems range from chilled water for air conditioning in the range of 36°F–40°F to cryogenic industrial applications at minus 300°F or lower. Here is where “vapor-proof” or nearly vapor-proof materials and systems must be employed.

In the below-ambient systems, vapor pressure differential between the ambient air and that adjacent to the insulated pipe or equipment will create a driving force that constantly pushes water vapor toward the cold surface. Said differently, the cold surface is a permanent magnet, if you will, for water vapor. Furthermore, this magnet provides a surface on which the water vapor will condense.

Condensation of water vapor on the cold insulated surface means that liquid water will begin to accumulate there and, in most cases, will continue to accumulate unabated for the duration of time that an entry for water vapor through the vapor retarder and insulation is available. Depending on the type of insulation being employed, this water will have varying degrees of detrimental effects on the thermal insulating properties. In a worst case, the insulation will become virtually useless in conserving energy in the system. This is to say nothing of the physical damage that the insulated pipe or equipment is likely to suffer and which can lead to a very expensive, if not catastrophic, failure.

The above creates the backdrop for the need to test materials that allow extremely low transmission of water vapor through them and, in some cases, must be totally impermeable to vapor transmission. ASTM E96 [1], *Standard Test Methods for Water Vapor Transmission of Materials*, falls under the stewardship of Committee C16 for Thermal Insulation, and has been in existence since 1953. In essence, it is a simple test wherein a specimen is mounted with exposure to higher relative humidity on one side, and lower relative humidity on the other, in a chamber or room at a set temperature. The specimen is mounted such that the differing RH conditions are totally isolated to one side or the other. As a result, there is a higher partial vapor pressure on one side of the sample versus the other, resulting in a unidirectional transfer of water vapor through the specimen to the lower partial vapor pressure side. The specimen in its test “dish” is weighed regularly to determine the rate of gain or loss of water weight.

The two basic methods employed are “dry cup” and “wet cup.” The former utilizes a theoretical 0 % RH on one side of the specimen (achieved with a mass of desiccant in a small volume area) and a higher RH—usually 50 % or greater—in the conditioned chamber or room. The latter employs a theoretical 100 % RH on one side (achieved with a mass of water in a small vessel) and a lower RH, usually 50 %, in the chamber or conditioned room. Both methods can be run at the temperature of choice, generally between 73°F and 100°F. Procedures A through E of ASTM E96 [1], calling out specific chamber conditions, are considered standard, but the user may run the test at the conditions of his choice.

In the dry cup method, vapor is driven through the test specimen from the environmental chamber into the desiccant underneath the specimen. This causes the test dish to gain weight over time. In the wet cup test, partial vapor pressure pushes the moisture vapor through the specimen into the environmental chamber or room, causing the test dish to lose weight continuously. Obviously, weight gain or loss will only occur if the material being tested is not totally impermeable.

In both tests, sealing of the specimen to the test dish to totally isolate vapor transmission to the test area of the specimen is most commonly done with molten wax mixtures. Gasket or mechanical sealing is not acceptable for testing very low permeance materials.

There are no limits as to the types of materials that can be tested except that, within current limitations of the test, specimens must be flat and generally not excessively thick, although provisions can be made to test relatively thick materials, such as insulation of two inches or more.

ASTM E96 [1] is used to obtain three primary measures:

- Water Vapor Transmission Rate (WVTR): the rate of water vapor flow through a material or structure under specific conditions.
- Water Vapor Permeance (WVP): commonly referred to simply as permeance; WVTR through a material or structure induced (divided) by the vapor pressure differential between sides.
- Permeability: permeance of a homogeneous material per unit thickness.

In the arena of vapor retarders (or vapor barriers) and materials used to make vapor retarders, the question of how adequate and precise ASTM E96 is for testing extremely low WVTR has been raised in the producer, user, and testing communities. Further discussion has centered on whether or not the performance of a truly impermeable material can be measured. In the case of some applications as mentioned above, products claimed to be water vapor impermeable—so-called “zero perm” materials—are being used.

In 2010, the task group under Committee C16 that is responsible for ASTM E96, undertook an Inter-laboratory Study (ILS), or “round robin,” in which four materials of extremely low permeance were tested. The objective of this ILS was to develop a precision and bias (P&B) statement to characterize the robustness of the test for evaluating such materials, and to see what problems the labs experienced in the course of testing at this low level of water vapor transmission [2]. The Research Report tied to the ILS provides details on the study and statistical analysis used with the data obtained.

This paper attempts to give an overview of that study, provide practical interpretation of the data and results, and delve into the discussions and questions noted earlier. It is not presented as a highly technical statistical discourse, but rather a pragmatic one based on inferences drawn from the ILS and experience of the author.

In the ILS, four materials were tested, with the *highest* expected permeance of the four being 0.01 perm. For comparison, a typical asphalt coated kraft residential facing/vapor retarder has a permeance of about 1.0 perm, or 100 times the level of the ILS materials. A perm is defined as one grain of moisture passing through one square foot of area per hour, as induced by one inch of Hg vapor pressure differential from side to side (grain/h/ft²/in. Hg).

With conditions of 73°F/50 %RH on one side, and 73°F/0 %RH on the other, a one perm material would allow about 5 gallons of water through a 4 ft by 8 ft panel in a year. A 0.01 perm material would allow about 6.5 fluid oz of water to pass. If a 0.01 perm material was tested per ASTM E96 [1] in a typically used 6 in. dish under

the above conditions for one month, only about 0.03 gs of water weight would be gained, or 0.001 gs (1 mg) per day.

Six test labs participated in the ILS, all with experience in testing materials with extremely low permeance performance. They were instructed to follow ASTM E96 Procedure A, which is the dry cup method at chamber or room conditions of 73°F and 50 % relative humidity. The RH under the specimen is assumed to be 0 % in this test, as produced by a mass of dry desiccant in the sealed space.

Table 1 shows the permeance results for all materials and labs [2].

The result for each material represents the average of three specimens.

Sample material D should provide evidence that the reproducibility (between lab) variation seen is due to factors in the testing, not material variation. This is because sample D was a lamination that included a 1 mil thick aluminum foil, pre-checked for pinholes. This material should not have allowed any passage of water vapor through it, as it is impermeable. Two labs obtained results with zeroes to three or four decimals with this material, and one recorded well under 0.005 perms, all well within rounding rules (to be discussed later) for recording zero, or nil, permeance. Results from the other three all round to 0.01 perm. While this may sound like a trivial disparity, it is significant for a material that is known to be of zero permeance.

The highlighted values throughout the table indicate results that are significantly out of line with the others, and could, or should, be considered outliers. The expected perm result is per the product manufacturer.

Table 2 shows the data from one lab for material B [2].

As can be seen, one result of three was many times higher than the other two, and many times higher than expected. Given the material is a homogeneous film of measurably consistent weight and thickness, it is highly unlikely that this result indicates true

TABLE 1 Permeance results, all labs, all materials.

AVERAGE PERMEANCE OF TEST SET, PERMS, ILS Number 512				
	TEST MATERIAL			
	A	B	C	D
EXPECTED PERM→	0.01	0.005	0.006	0.00
LAB				
1	0.0076	0.0131	0.0165	−0.0002
2	0.0057	0.0040	0.0360	0.0027
3	0.0064	0.0034	0.0130	−0.00007
4	0.0142	0.0086	0.0155	0.0066
5	0.0089	0.0161	0.0255	0.0135
6	0.0099	0.0053	0.0150	0.0115
AVG	0.0088	0.0084	0.0203	0.0068
HIGH	0.0142	0.0160	0.0360	0.0135
LOW	0.0057	0.0034	0.0130	−0.00007
STD. DEV.	0.0031	0.0052	0.0089	0.0058

specimen performance; rather it is likely due to a problem in preparation of the test dish or cup, which involves careful sealing with a low melt point wax mixture.

The high value was included in the calculation of the average, yielding a result much higher than that expected, and most likely erroneous, based on all indications from the study.

Table 3 shows all individual specimen results from one lab for all materials [2].

The expected perm result was provided to the labs along with the material samples. In Table 3 we see that, except for material A, there are what appear to be outlying results (highlighted) in each test set, based either on expected results or the disparity in individual results. Although this lab would not have known it during the tests, results from other labs support an indication of erroneous values with assignable causes for these outliers.

Again, due to the consistent, homogeneous nature or the structural constituents of these materials, it should be expected that results would fall in a tight range. Materials produced with inherent holes, internal voids, inconsistent density, thickness, or internal structure can be expected to yield highly variable results with attendant high standard deviation, but the types of materials tested here should not.

The following is the precision and bias statement that was generated from the study [3].

9 Precision and Bias Statement

9.1 The precision of this test method is based on an interlaboratory study of ASTM E96, Standard Test Methods for Water Vapor Transmission of Materials, conducted in 2010, as well as studies conducted previously. Six laboratories participated in this study, analyzing four different materials. Each “test result” reported represents an individual determination and all participants reported three replicate test results for every material. Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report No. C16-1040.ⁱ

9.1.1 *Repeatability limit (r)*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the “ r ” value for that material; “ r ” is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

9.1.1.1 Repeatability limits are listed in Table 4 below.

9.1.2 *Reproducibility limit (R)*—Two test results shall be judged not equivalent if they differ by more than the “ R ” value for that material; “ R ” is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

9.1.2.1 Reproducibility limits are listed in Table 4 below.

9.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E 177.

9.1.4 Any judgment in accordance with statements 9.1.1 and 9.1.2 would have an approximate 95 % probability of being correct.

The statistics table would indicate that this test is not very reliable for evaluating the types of materials tested in the study. This writer believes that an assessment

TABLE 2 Permeance data from one lab for one material.

WEIGHING AND COMPUTATION OF PERMEANCE-ONE TEST SET

Hours	Material B		Cup Weights			Hours	Adjusted Weights		
	Weight	Gain	Cup 1	Cup 2	Cup 3		Cup 1	Cup 2	Cup 3
0	105.228	0.000	157.385	160.526	159.050	0	157.385	160.526	159.050
24	105.228	0.000	157.389	160.529	159.050	24	157.389	160.529	159.050
48	105.227	-0.001	157.385	160.529	159.050	48	157.386	160.530	159.051
115	105.227	-0.001	157.386	160.535	159.050	115	157.387	160.536	159.051
139	105.227	-0.001	157.386	160.536	159.050	139	157.387	160.537	159.051
163	105.227	-0.001	157.387	160.541	159.050	163	157.388	160.542	159.051
187	105.227	-0.001	157.386	160.543	159.050	187	157.387	160.544	159.051
212	105.227	-0.001	157.387	160.545	159.049	212	157.388	160.546	159.050
290	105.227	-0.001	157.388	160.555	159.055	290	157.389	160.556	159.056
338	105.226	-0.002	157.391	160.557	159.053	338	157.393	160.559	159.055
383	105.227	-0.001	157.388	160.563	159.054	383	157.389	160.564	159.055
504	105.228	0.000	157.390	160.577	159.055	504	157.390	160.577	159.055
553	105.227	-0.001	157.391	160.580	159.055	553	157.392	160.581	159.056
619	105.226	-0.002	157.391	160.590	159.056	619	157.393	160.592	159.058
667	105.226	-0.002	157.392	160.592	159.057	667	157.394	160.594	159.059
789	105.225	-0.003	157.396	160.607	159.065	789	157.399	160.610	159.068
835	105.228	0.000	157.391	160.614	159.062	835	157.391	160.614	159.062
888	105.225	-0.003	157.397	160.620	159.063	888	157.400	160.623	159.066
956	105.228	0.000	157.398	160.624	159.066	956	157.398	160.624	159.066

TABLE 2. Continued

WEIGHING AND COMPUTATION OF PERMEANCE-ONE TEST SET										
Hours	Material B		Cup Weights			Hours	Adjusted Weights			Cup 3
	Weight	Gain	Cup 1	Cup 2	Cup 3		Cup 1	Cup 2	Cup 3	
1008	105.225	−0.003	157.399	160.628	159.066	1008	157.402	160.631	159.069	
1058	105.224	−0.004	157.398	160.634	159.066	1058	157.402	160.638	159.070	
1128	105.224	−0.004	157.399	160.638	159.068	1128	157.403	160.642	159.072	
1153	105.224	−0.004	157.399	160.642	159.070	1153	157.403	160.646	159.074	
1176	105.224	−0.004	157.399	160.646	159.070	1176	157.403	160.650	159.074	
1199	105.224	−0.004	157.398	160.648	159.071	1199	157.402	160.652	159.075	
1223	105.225	−0.003	157.401	160.653	159.070	1223	157.404	160.656	159.073	
		Grams/h	0.00001	0.00010	0.00002	Grams/h	0.00001	0.00011	0.00002	
		Grains/h/ft ²	0.00019	0.00160	0.00028	Grains/h/ft ²	0.00023	0.00164	0.00032	
		Area, ft ²	0.1364	0.1364	0.1364	Area, ft ²	0.1364	0.1364	0.1364	
		gn/h/ft ²	0.001379	0.011753	0.002067	gn/h/ft ²	0.001669	0.012043	0.002357	
		RH	0.499	0.499	0.499	RH	0.499	0.499	0.499	
		Sat Pressure	0.8213	0.8213	0.8213	Sat Pressure	0.8213	0.8213	0.8213	
		Delta P	0.409829	0.409829	0.409829	Delta P	0.409829	0.409829	0.409829	
		Perms	0.003	0.029	0.005	Perms	0.0041	0.0294	0.0058	
							AVG.	0.0131		
							Std. dev.	0.014154		
							Cf. Var.	1.08		

TABLE 3 Permeance results from one lab, all specimens.

ASTM E 96 INTERLABORATORY STUDY #512				
SAMPLE SET DATA, ALL MATERIALS, LAB 5				
EXPECTED PERM SAMPLE A:		0.01		
DISH NUMBER	PERM	STD. DEV.	COEFF. VAR.	
1	0.0108			
2	0.0070			
3	0.0090			
AVG.	0.0089	0.001900877	0.21	
EXPECTED PERM SAMPLE B:		0.005		
DISH NUMBER	PERM	STD. DEV.	COEFF. VAR.	
1	0.0415			
2	0.0034			
3	0.0035			
AVG.	0.0161	0.0220	1.37	
EXPECTED PERM SAMPLE C:		0.006		
DISH NUMBER	PERM	STD. DEV.	COEFF. VAR.	
1	0.0177			
2	0.0444			
3	0.0145			
AVG.	0.0255	0.0164	0.64	
EXPECTED PERM SAMPLE D:		0.00		
DISH NUMBER	PERM	STD. DEV.	COEFF. VAR.	
1	0.0108			
2	0.0002			
3	0.0295			
AVG.	0.0135	0.0148	1.10	

of E96 for its suitability in testing extremely low permeance materials should look beyond the pure statistics that were produced in this study.

Table 5 shows all individual specimen results from a second lab for all materials [2].

If we look at the data obtained and statistics generated by lab 3, it is apparent that a very low level of variation can be observed in testing such materials. If certain

TABLE 4 Water vapor transmission (perms). For a copy of the draft Research Report, please contact Caitlin Farrell at cfarrell@astm.org.

Material	Average ⁱ	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	$\bar{x}$	s_r	s_R	r	R
A	0.00877	0.00166	0.00336	0.00465	0.00940
B	0.00843	0.01067	0.01067	0.02988	0.02988
C	0.02028	0.01698	0.01698	0.04756	0.04756
D	0.00567	0.00938	0.00967	0.02626	0.02706

ⁱThe average of the laboratories' calculated averages.

sample sets are isolated in the other labs, we also see good repeatability in given tests. If we assume that the materials here are very consistent in structure—and the evidence is there to support this—it points to the outliers being generated by assignable causes in how the test was conducted.

It seems that the appearance of outlying data was not attended to properly in most, if not all, cases in this study. In some cases, this is due to the fact that some labs did not generate permeance results until the raw data was all collected, which would make it difficult to pick out results that were trending out of line with others.

A second reason for not responding to an apparent outlier would simply be not recognizing it as such. When testing at such low levels of water vapor transmission, seemingly benign variation is not necessarily so.

Another reason could be recognizing a possible problem through the data, but not being able to find a cause and continuing the test.

Significant data input errors can of course also create an outlier, and these should be easily recognized.

When testing at these low WVTR or permeance levels, outliers appear invariably as high results, not low. It is not logical for a given specimen of such materials to magically produce a result much lower than other specimens of the same material. Results that are significantly higher than others being obtained, or notably higher than expected, should be very suspect as erroneous.

The most common causes for outliers are leaking seals or physical damage to the specimen. These should be considered erroneous results or invalid tests, and not included in the data set. Close examination of the seal or specimen should show one of these problems, although it can be very difficult to see seal leaks, which can occur at the dish or sample interface.

When an apparent outlying test dish shows up, the seal should be closely examined. If a crack or gap is seen at the interface with the dish wall, it can usually be resealed with a soldering iron. If it is seen at the specimen interface, the dish should be taken out of the test. If a seal failure is not detected, the specimen should be examined visually for physical damage (hole). Some polymeric materials have low

TABLE 5 Permeance results from one lab for all materials.

ASTM E96 INTERLABORATORY STUDY Number 512			
SAMPLE SET DATA, ALL MATERIALS, LAB 3			
EXPECTED PERM SAMPLE A:		0.01	
DISH		STD.	COEFF.
NUMBER	PERM	DEV.	VAR.
1	0.0063		
2	0.0062		
3	0.0068		
AVG.	0.0064	0.0003	0.05
EXPECTED PERM SAMPLE B:		0.005	
DISH		STD.	COEFF.
NUMBER	PERM	DEV.	VAR.
1	0.0036		
2	0.0032		
3	0.0034		
AVG.	0.0034	0.0002	0.06
EXPECTED PERM SAMPLE C:		0.006	
DISH		STD.	COEFF.
NUMBER	PERM	DEV.	VAR.
1	0.0134		
2	0.0131		
3	0.0131		
AVG.	0.0132	0.0002	0.01
EXPECTED PERM SAMPLE D:		0.00	
DISH		STD.	COEFF.
NUMBER	PERM	DEV.	VAR.
1	-0.00013		
2	-0.00013		
3	0.00000		
AVG.	-0.00009	0.0001	-0.87

surface energy and can be difficult to seal. With such materials, it is especially important to be cognizant of possible seal failures and leaks.

The problem becomes when to call a result an outlier. Obviously, we cannot throw out results simply because they are high (i.e., higher than we would like to see). However, logically, it does not make sense—especially for thin, dense structures or materials—that a high level of variation would be seen between specimens. It is suggested that, if a cause is not found for the outlier, the test needs to be repeated one or more times with a new set of specimens.

How to deal with apparent low outliers is the next obvious question. There is very little likelihood of low outliers being produced when testing these types of materials in this range of permeance. Because they are so thin and dense, it does

not seem reasonable that a given piece of material would be significantly less permeable than other specimens.

Incorrect test conditions producing low vapor drive would affect all specimens, not just some, so such malfunctions should not be an assignable cause for an individual outlier. Data input errors could cause a low result, or outlier, in a given specimen. This would be readily detected, since it would typically be limited to one data point input, and would have to be greatly out of line with all others.

In the case of dish 2 of sample D in Table 3, the result would appear to be an outlier, when in fact it is an expected result and the other two dish results are much higher than expected. Since this material was known to be impermeable, the higher results should have been questioned and investigated.

If the operator did not know what the expected result was, the obvious choice for the cause of a low outlier would be data input error, which could have easily been checked. The point is that outlying low results should not be expected, so rather than a low result, or results being wrong, the higher results should be suspect.

Given the high probability that the cause for outliers (i.e., high results) is in the test and not the material, the greater danger, in the author’s opinion, is in assigning erroneously poor performance results to a material versus the appearance of manipulating results or inappropriately creating a desired result.

Table 6 shows results for all labs and all materials, calculated after obvious individual outliers in sample sets have been removed [2].

Overall, a total of seven results were removed from 72 in this exercise. As is to be expected, the results tighten up significantly and, with one exception, come in close to expected values. Again, the assumption here is that the cause for outliers is most likely leaking seals, and the point is that the test would appear much more

TABLE 6 Permeance results from all labs for all materials, outliers removed.

AVERAGE PERMEANCE OF SET, PERMS				
	TEST MATERIAL			
	A	B	C	D
EXPECTED PERM→	0.01	0.005	0.006	0.00
LAB				
1	0.0076	0.0050	0.0165	−0.0002
2	0.0057	0.0040	0.0140	0.0027
3	0.0064	0.0034	0.0130	−0.00007
4	0.0050	0.0086	0.0155	0.0066
5	0.0089	0.0035	0.0161	0.0002
6	0.0099	0.0053	0.0150	0.0015
AVG	0.0073	0.0050	0.0150	0.0018
HIGH	0.0099	0.0086	0.0165	0.0066
LOW	0.0050	0.0034	0.0130	−0.00007
STD. DEV.	0.0019	0.0019	0.0013	0.0026

robust if more careful attention was paid to the presence of outliers and addressing the problems that created them.

Material C requires a different consideration. Here, the results obtained across the board were all notably higher than expected. In this case, the high results are not really due to assignable cause outliers; this is evident because the results (16 of 18 individual specimens) were running at this higher level. The two apparent outliers were much higher. Even though the results are consistent, they are higher than expected, so as with any potential outlying result, all specimens must first be checked carefully for failed seals. If none are detected, it is advisable to test a new sample set if there is any question about the performance results.

Obviously, there will be cases where the operator does not know what results are to be expected. Consistency in results, such as those seen in Table 5 for Material C, does indicate that there are no individual specimen problems, and that the results probably are accurate for the material.

It is always good practice to check seals early on in the test regardless of the type of material being tested.

Table 7 lists correlation coefficients for all tested materials in all labs [2].

The values used to calculate these are those for elapsed time in hours on the *x* axis, and weight of the test cup on the *y* axis. The value shown is the average of the individual correlation coefficients for three test cups. The correlation coefficient shown for each material is an average of that of each specimen in the sample set. This statistic is a measure of the relationship of weight gain versus time. Perfect

TABLE 7 Correlation coefficients from all labs for all materials.

ASTM E96 INTERLABORATORY STUDY Number 512					
CORRELATION COEFFICIENT ALL LABS, ALL MATERIALS					
SET AVERAGE CORRELATION COEFFICIENT					
Correlation of weight of test dish versus elapsed time					
LAB NUMBER	TEST MATERIAL (OVERALL AVERAGE PERM)				
	A(0.009)	B(0.008)	C(0.020)	D(0.007)	
1	0.978	0.975	0.997	−0.125	
2	0.980	0.970	0.973	0.770	
3	0.985	0.965	0.997	0.427	
4	0.973	0.943	0.973	0.750	
5	0.947	0.998	0.998	0.635	
6	0.994	0.994	0.999	0.738	
AVG	0.976	0.974	0.988	0.533	0.868
HIGH	0.994	0.998	0.999	0.770	0.999
LOW	0.947	0.943	0.973	−0.125	−0.125
STD. DEV.	0.0160	0.0202	0.0128	0.3464	0.09883
COEFF. VAR.	0.02	0.02	0.01	0.65	0.18

correlation between multiple x and y data coordinates equals 1. What constitutes “good” or “minimum acceptable” correlation apparently is not set in stone in the statistics community, although 0.95 might be at the lower end of such a discussion.

The correlation coefficient generally tracks with the WVTR or permeance result; that is, it goes down as the result gets lower. The author’s experience has shown that good correlation is generally seen at 0.005 perm and above. Below that, it begins to drop off to the point where, at zero or virtually zero permeance, there is no correlation between weight gain and time. This is because the test dish is not gaining measurable water and the weight data points bounce above and below what is a horizontal (no slope) line due to slight variations in test conditions.

For the theoretically zero permeance sample D, results from those labs which obtained values closest to zero had by far the worst correlation, although all were quite poor. All other materials in all labs came in well above 0.90, with only two slightly below 0.95. It is important to note that poor correlation for extremely low WVTR or permeance measurements is to be expected. As a guide, from the author’s experience, if poor correlation is seen on results above 0.005 perms, most likely there is an error in data entry, or significant control issues in the test environment.

Very high correlation should serve as an indication that steady state weight gain has been achieved. Again, above a certain point this is useful, but not so for extremely low permeance materials.

ASTM E96 [1] makes provision for corrections in dish weights to account for barometric pressure fluctuation, which would serve to produce better correlation in extremely low measurements. However, application of the correction factor is very complex and seems to be rarely employed. It likely has negligible effect on the final result; one that surely would be lost in rounding.

Note in Table 1 that labs 1 and 3 obtained negative values for sample D. Application of a correction calculation could result in lower likelihood of obtaining a negative value, which can occur when testing zero permeance materials. However, the appearance of a negative perm value after two weeks or so is strong reinforcement that the material is indeed impermeable, or zero permeance.

Sample D of the study used a one mil thick aluminum foil sandwiched between two layers of 0.5 mil polyester film. The product was pre-screened by checking for pinholes. Again, this material should be totally impermeable to moisture vapor.

Table 8 shows all individual test specimen results from all labs, with and without obvious outliers removed [2].

If one considers the results of labs 1, 2, 3, and 6 only, and removes obvious outliers (3 of those 48 results), those results appear to fall in a reasonable range within and between labs. Lab 4 showed consistent values with no apparent outliers, but results for materials A, B, and D are out of line with the above labs, probably indicating sealing issues with certain types of materials, which could be related to the seal wax formulation or temperature. Lab 5 had a number of outliers, indicating significant seal leaks.

TABLE 8 Permeance results from all labs with and without outliers removed.

ALL INDIVIDUAL SPECIMEN RESULTS, ALL LABS				
LAB	SAMPLE A	SAMPLE B	SAMPLE C	SAMPLE D
1	0.0076	0.0041	0.0170	−0.0005
	0.0072	0.0294	0.0163	−0.0003
	0.0079	0.0058	0.0161	0.0000
2	0.0055	0.0045	0.0126	0.0032
	0.0054	0.0035	0.0800	0.00082
	0.0061	0.0041	0.0151	0.0041
3	0.0063	0.0036	0.0134	−0.00013
	0.0062	0.0032	0.0131	−0.00013
	0.0068	0.0034	0.0131	0.00000
4	0.0169	0.0085	0.0165	0.0075
	0.0132	0.0083	0.0151	0.0063
	0.0124	0.0090	0.0149	0.0059
5	0.0108	0.0415	0.0177	0.0108
	0.0070	0.0034	0.0444	0.0002
	0.0090	0.0035	0.0145	0.0295
6	0.0129	0.0056	0.0154	0.0016
	0.0081	0.0049	0.0148	0.0014
	0.0087	0.0055	0.0150	0.0317
Overall average	0.0088	0.0084	0.0203	0.0057
Rounded o.a. average	0.01	0.01	0.02	0.01
Average -outliers	0.0088	0.0050	0.0151	0.0020
Rounded avg. -outliers	0.01	0.01	0.02	0.00
Expected perm	0.01	0.005	0.006	0.00

Note: Values in bold were considered outliers. Highlighted values were considered questionable, but left in.

The above observations are obviously based on a non-statistical, or at least an unsophisticated, assessment. A practical view of the data in Table 8 would say that the test is reliable for confirming the performance of zero permeance products, especially since, per the method, results are to be reported to two significant figures, or 0.00 perm. The author believes that results could be rounded to three significant figures at 0.005 perm to say, 0.05 perms. Below 0.005 perm, good correlation is lost, and we cannot be comfortable with a reported measurement to three decimal places, although application of correction factors should mitigate this. An exception to this would be when extremely poor correlation is seen and negative permeance results appear during the course of the test. In such cases, it is a safe assumption that the material is truly water vapor impermeable.

It cannot be stressed enough that it is critical for test operators to be vigilant and to recognize outliers during the course of the test, and to investigate possible seal or specimen damage problems. Permeance must be calculated as the weights are taken, not after all weighing data is collected.

Very thin foils (not tested in the ILS)—less than one mil thickness—will have pinholes. These materials are used as components of vapor retarders and introduce some interesting considerations. In the case of such materials, true permeation through the molecular structure of material is not the mechanism of vapor transfer, but rather mass transfer through open passageways is the predominant route (laminations can provide secondary vapor blocking with adhesives and other substrates). Hence, for example, one could see a specimen of one material yielding zero or nil permeance, and another at 0.03 perms. This should not necessarily raise the suspicion of the latter specimen being an outlier, because the material is known to have inherent holes.

It is possible that with products relying on very thin foil as the vapor retarding component, that significant variation can be seen between specimens, variation that with other materials would represent questionable if not unacceptable data. This reinforces the need to be aware of what type of material is being tested, and how the results being generated compare to the expected range.

The “Zero Perm” Vapor Retarder, or Vapor Barrier

As a point of interest, ASTM C168 [4], Standard Terminology Related to Thermal Insulation, shows the same definition applying to water vapor retarder and water vapor barrier: *a material or system that significantly impedes the transmission of water vapor under specified conditions*

Some would argue that anything other than a zero permeance material should be called a vapor *retarder*, and not a *vapor barrier*. This definition would say that should not be the case, and evidently considers a barrier to not always be absolute.

The questions are asked: “Do impermeable vapor retarders really exist?” and “How do we know something is truly a zero permeance material or product?” The answers are “Yes, they do,” and “Test using ASTM E96.”

Certainly, pinhole-free aluminum foil is impermeable to water vapor. It is generally accepted in that foil one mil (0.001 in.) or thicker will be pinhole-free, or contain pinholes so sporadic that they will have no measurable effect on a vapor retarder system (likewise, laminations containing two layers of a thinner foil have been shown to yield zero permeance, with pinholes in one layer being blocked by solid foil in the other).

Looking at the one mil foil lamination tested in this study, results confirm it is impermeable. It is critical for vapor barrier products, however, to include other layers that serve to protect the foil from handling damage through the final installation. Usually, these products will be covered with a protective jacket to protect

them from physical abuse in service. ASTM C1136 recently added new classifications for zero perm materials, and specifies that the foil be laminated to another substrate to provide structural/protective support as noted above. Section 5 of the most recent edition of this specification outlines the requirements and is shown below.

5. Materials and Manufacture

5.1 Vapor retarders, with the exception of Type IX, are constructed of various films, metallic foils, fabrics, papers and reinforcements, alone or in combination as a lamination.

5.2 *Metallic foils, the most commonly used being aluminum of a minimum gauge, are the only materials expected to provide the impermeable component in a vapor retarder required for Type IX.*

5.2.1 *Type IX vapor retarders must contain at a minimum the following components in the structure:*

5.2.2 *One layer of metallic foil with a minimum thickness of 0.00095 in. (24.1 μ) or*

5.2.3 *Multiple layers of metallic foil with a cumulative minimum thickness of 0.0012 in. (30.5 μ).*

5.2.4 *A fully laminated, continuous protective substrate on at least one side of the foil layers [5].*

Only products utilizing metallic foil are allowed for the zero permance category because, to this point, no other products have been commercially marketed that claim zero permance. Although a plastic film theoretically will never reach true impermeability, as seen in the study, an adequate thickness of film can provide results that will round to 0.00 perm.

In the real world, the availability of truly impermeable or zero perm vapor barriers is only part of the equation for assuring that a below-ambient insulation system is protected against water vapor intrusion. On insulation and vapor barriers on pipe, equipment, or enclosures, there will be the following possible problem areas: joints between insulation sections, fittings, penetrations, breaches from other components, as well as holes caused by physical damage. All are present as potential compromises to the system if not properly sealed in the field.

The use of low permeability foam insulation (with attendant joint sealing compounds) does not necessarily preclude or eliminate the need for a separate vapor barrier. In high vapor drive applications, a second layer of defense, or a redundant vapor barrier is preferred, if not necessary.

Presently, some studies are being undertaken or proposed that would attempt to measure the intrusion through the joints and seals that make up a vapor retarder system. The two major factors in how such joints and seals perform are materials and workmanship. It is fully logical that the weak link in the chain of the vapor retarder system would be the joints and seals; however, there seems to be no data

available in the public domain to quantify how they work as a system with the base vapor retarder. Initial studies will likely have joints and seals made in a controlled benchtop setting. The next step might be trying to replicate actual field installation conditions.

In below-ambient systems, selection of the appropriate materials, along with high-quality workmanship in sealing joints and penetrations is critical. While zero perm products are not always necessary with these insulations, they are available if desired. Whether a system that uses a zero perm product actually performs measurably better overall than a system using, for example, a 0.02 perm vapor retarder, will not be known until detailed studies determine this.

Summary

ASTM E96 [1] should be considered a fully acceptable method for confirming performance of zero permeance or water vapor impermeable materials and products, as well as for those near zero or of extremely low permeance. One can have confidence in such results, as long as it is demonstrated that careful attention has been paid to the critical aspects of specimen preparation and inspection and data monitoring as discussed herein.

The performance of “Zero Perm” Vapor retarder or vapor barrier products being totally impervious to water vapor can be confirmed by this test.

While we can be assured that zero perm products do in fact provide absolute or virtual impermeability to water vapor intrusion, it must be kept in mind that the system of which they are part will, in all likelihood, not perform at that level. How these systems actually perform will be the subject of future investigation.

References

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Therese Stovall¹

Evaluation of Homogeneity Qualification Criteria in the Accelerated Aging of Closed-Cell Foam Insulation, Results after Five Years of Full-Thickness Aging

Reference

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ABSTRACT

The thermal conductivity of many closed-cell foam insulation products changes over time as production gases diffuse out of the cell matrix and atmospheric gases diffuse into the cells. Thin slicing has been shown to be effective in accelerating this process in such a way as to produce meaningful results. One of the challenges for this test method is determining whether a foam product is sufficiently homogenous so that the accelerated prediction accurately reflects the aging of the full-thickness product. A related question is whether or not thin slices extracted from one product thickness can accurately predict the aged thermal conductivity for the same product in a different thickness. Qualification criteria were developed for the prescriptive version of the ASTM C1303 standard test method to ensure that predicted thermal conductivity results would be acceptably accurate. A ruggedness test (a "test the test" process) was initiated to examine multiple test parameters, and to determine whether these

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¹Oak Ridge National Laboratory, Oak Ridge, TN 37831, United States of America.

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qualification criteria were adequate. This test program included the aging of full size insulation specimens for time periods of 5 years for direct comparison to the predicted results. The test program has been completed, and this report discusses the accuracy of the 5-year thermal conductivity prediction for various levels of the homogeneity and alternate product thickness qualification criteria.

Keywords

accelerated aging, foam insulation, homogeneity

Introduction

Closed-cell foam insulation is used in many applications with lifetimes ranging from 8 to over 40 years, and users are, therefore, interested in the long-term thermal resistance values, as opposed to the thermal resistance of new foam. Heat transfer through gaseous conduction in closed-cell foam insulation is partially determined by the composition of the gas mixture within the foam cells. Many closed-cell foam insulation products are produced using a blowing agent with a lower thermal conductivity than that of air. Over time, the cell contents change as atmospheric gases diffuse into the cells and the blowing agent gases diffuse out into the surrounding environment. For insulation sheets where the thickness is small relative to the width and length, this diffusion process has been shown to follow Fick's Law for one-dimensional diffusion through homogenous materials. The gaseous diffusion is governed by the diffusion coefficient for each gas for the foam, the foam thickness, and time [1–12]. Most importantly, for the purpose of accelerating the aging process, Fick's Law shows that the aging is directly proportional to the time divided by the square of the diffusion thickness. So, for example, if a 1-cm (0.4-in.) slice is taken from a 5-cm (2-in.) product, the slice will age at 25 times the rate of the full-thickness product. Five years of aging can thus be accelerated via thin slicing to $73 (= 5 \times 365/25)$ days. This normalized time is used throughout this analysis, usually in units of days/cm². Multiple accelerated-aging test methods have been developed and validated based upon this approach [13–15].

A ruggedness test is used to “test the test method.” Two previous ruggedness tests addressed questions within ASTM C1303, Standard Test Method for Predicting Long-Term Thermal Resistance of Closed-Cell Foam Insulation, regarding the cutting methods used to prepare the foam thin slices and the thickness of the destroyed surface layer that results from the cutting process [16–18]. The ruggedness test reported here was initiated to support the development of the prescriptive option within ASTM C1303. The work has taken place over a 6-year time span and multiple papers have been published describing the test protocol and results. The first paper describes the test protocol and initial results from the extensive slicing operations [19]; the second compares predictions to measurements after 2 years of aging [20]; and the third is a full research report covering the study goals and results [21]. (Another paper will be published summarizing the accuracy implications for 5-year predictions at a conference in December of 2013 [22].)

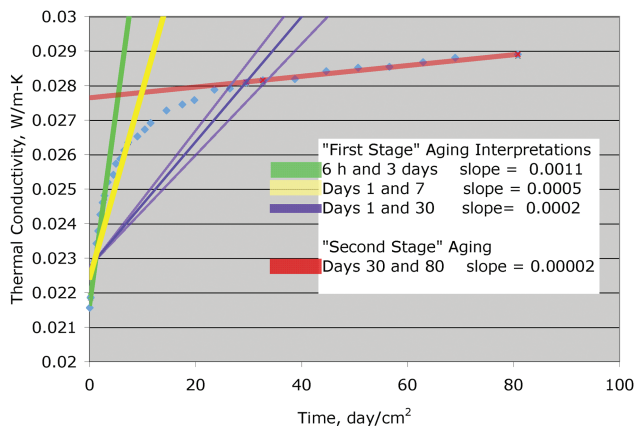
Fick's Law holds true for perfectly homogenous products, but real foam insulation materials are not perfectly homogenous. Slices taken from different locations throughout the foam's thickness may exhibit different thermal conductivity and there may be differences in cell morphology, such as cell size or structure, which affect the gas diffusion and, therefore, impact the aging rate. There may be adhered or intrinsic facings at the outer surfaces that may be more or less resistant to gas diffusion. Recognizing these complexities, there have been questions regarding the degree to which portions of the foam cross section must be similar for the age acceleration process to produce an acceptably accurate prediction of the full-thickness aged thermal conductivity. Questions were also raised regarding the applicability of accelerated aged performance values derived from thin slices taken from 50 mm (2 in.) products to products of other thicknesses. Within C1303 and in this paper, this is called an "alternate thickness" or "alternate product thickness" prediction.

Although the ruggedness test program encompassed many factors, this paper is focused on the influence of screening criteria upon the test accuracy, where the accuracy is determined by directly comparing the accelerated-aging prediction to the full-thickness aged thermal conductivity after 5 years.

History

An early version of ASTM C1303 defined a foam product as sufficiently homogenous if the slope of the thermal conductivity versus the normalized time during the first stage of aging did not vary more than 10 % between multiple specimens taken from the core and surface regions. As shown in Fig. 1, this criterion left the definition of the "first stage" of aging to the user [19]. Also, the earliest version of ASTM C1303 did not explicitly address whether a set of thin slices taken from

FIG. 1 Homogeneity criteria from ASTM C1303 (2000); 10 % acceptable bounds are shown for the aging slope based upon data from days 1 and 30 [19].



one product thickness could be used to predict the thermal conductivity for products of other thicknesses.

Qualification tests were more precisely defined in the 2007 version of ASTM C1303 for both product homogeneity and the use of thin slice data from alternate product thicknesses. The method of examining the first stage of aging was altered into a more prescriptive “age equivalence” qualification criteria, shown in Eqs 1 and 2, based upon the ‘aging factor’ ratio approach from CAN/ULC S770 [14]. In this qualification test, the change in thermal conductivity over a period of time of approximately 1 month for surface slices is compared to the corresponding change for core slices over the same normalized time period (similar to the line shown with a slope of 0.0002 in Fig. 1). One of the objectives of the ruggedness test is to determine an appropriate “passing grade” for these criteria. The criteria were arbitrarily set at a broad level in 2007 pending the results from this study.

Figure 1 shows homogeneity criteria from ASTM C1303 (2000); 10 % acceptable bounds are shown for the aging slope based upon data from days 1 and 30 [19].

$$\text{Aging Rate} = k1/k30 \tag{1}$$

$$H = 1 - \frac{(\text{Aging Rate}_{\text{Core}} - \text{Aging Rate}_{\text{Surface}})}{\text{Average Aging Rate}} \tag{2}$$

where:

- $k1$ = thermal conductivity after 24 h/cm², W/m·K,
- $k30$ = thermal conductivity after 30 days/cm², W/m·K, and
- H = homogeneity aging equivalence.

The 2007 version of ASTM C1303 has two qualification requirements for alternate thickness predictions. First the core and surface stacks from each product thickness must age at similar rates over the first 30 d/cm², as shown in Eqs 3 and 4. Second, the core and surface stacks from each product thickness must have similar thermal conductivities at the end of the first 30 days/cm², as shown in Eqs 5 and 6. Because of the comparative nature of the alternate product thickness qualification test, it requires a set of eight ASTM C518 test results for each application of results from one product thickness to another [15]. All four of these comparisons were required to fall between 92 % and 108 % to satisfy the 2007 ASTM C1303 qualification requirements for an alternate thickness prediction:

$$\text{Age Equivalence}_{\text{Core}} = 1 - \frac{\Delta \text{Aging Rate}_{\text{Core}}}{\text{Average Aging Rate}_{\text{Core}}} \tag{3}$$

$$\text{Age Equivalence}_{\text{Surface}} = 1 - \frac{\Delta \text{Aging Rate}_{\text{Surface}}}{\text{Average Aging Rate}_{\text{Surface}}} \tag{4}$$

$$K \text{ Equivalence}_{\text{Core}} = 1 - \frac{\Delta k30_{\text{Core}}}{\text{Average } k30_{\text{Core}}} \tag{5}$$

$$K \text{ Equivalence}_{\text{Surface}} = 1 - \frac{\Delta k_{30_{\text{Surface}}}}{\text{Average } k_{30_{\text{Surface}}}} \quad (6)$$

where:

Δ refers to the difference between the values for the two product thicknesses under evaluation.

Methodology

The ruggedness study evaluated several possible combinations of surface and core slices, called stacks. These various arrangements were all in use by one test laboratory or another when the ruggedness study began, and there were questions regarding which stack would produce the most accurate prediction of the aged thermal conductivity for the full-thickness product. Many of the results shown here are for the “Math” stack, because that arrangement gave reasonable results for both XPS and PIR, whether or not the source product was the same thickness as the product under evaluation [21]. The math stack type is a mathematical derivation, using a series resistance expression to weight the measured values from stacks comprised solely of core slices and solely of surface slices. The intent of this expression is to determine the effective thermal conductivity of the overall product structure, as shown in Eqs 7 and 8, by considering the fraction of the total product thickness best represented by the slices included in the surface stack and the fraction of the cross section best represented by the stack of core slices.

$$R_{\text{total}} = \frac{L_{\text{product}}}{k_{\text{effective}}} = \Sigma R = \Sigma \frac{\Delta x}{k} = \left(\frac{2L_{\text{surface}}}{k_{\text{surface}}} \right) + \left(\frac{L_{\text{product}} - 2L_{\text{surface}}}{k_{\text{core}}} \right) \quad (7)$$

$$\text{for } F_{\text{surface}} = \frac{2L_{\text{surface}}}{L_{\text{product}}}, \text{ then } \frac{1}{k_{\text{effective}}} = \left(\frac{F_{\text{surface}}}{k_{\text{surface}}} \right) + \left(\frac{1 - F_{\text{surface}}}{k_{\text{core}}} \right) \quad (8)$$

where:

R = thermal resistance, K/W ,

L = thickness, m ,

k = thermal conductivity, $W/m \cdot K$, and

F = fraction.

This ruggedness test included specimens from 2.5 to 10 cm (1 to 4 in.) products of PIR with two different facings and XPS of two different densities from multiple manufacturers. The full experimental design involved the production of ~ 250 thin slices and ~ 1000 ASTM C518 thermal conductivity measurements [15,21]. Uncut full-thickness specimens of each insulation product were maintained in a conditioned space. After a 5-year period, the thermal conductivity of these full-thickness specimens was measured for comparison to the thin-slice predictions.

The error, or difference between the predicted value and the measured value as a fraction of the full-thickness thermal conductivity, was calculated for each prediction. Using this definition:

- A positive error indicates the predicted thermal conductivity was too high—and, therefore, the predicted thermal resistance was too low.
- A negative error indicates the predicted thermal conductivity was too low—and, therefore, the predicted thermal resistance was too high.

General linear models were used to evaluate the impact of a wide range of single-factor and multiple-factor sets on the error within this extensive dataset. The population marginal means values correct for the unbalanced nature of the dataset and were calculated for those factors that showed statistical significance. These analysis tools are described in the full project report [21]. Again, this paper is focused on the results for the homogeneity and alternate thickness screening criteria. Many of the other test factors, especially stack type, were found to have much more impact on the prediction accuracy.

Analysis

HOMOGENEITY

The homogeneity qualification test depends on a set of four ASTM C518 test results to compare the aging behavior, over the first 30 days, of sets of slices taken from the surface and core of the material. The intent is to determine whether the foam is homogenous enough throughout its thickness so that a subset of that thickness, in the form of thin slices, can be used to adequately represent the aged thermal conductivity of the whole. For the materials included in this ruggedness study, the qualification test values all fell in a narrow range of 92 % to 99 % [17,19]. Considering the narrow range of these results, an alternative definition for homogeneity was explored, as shown in Eq 9. This simpler approach compares the absolute values of thermal conductivity of the core and surface slices after 30 d/cm² instead of the aging rates. Based on the results of this ruggedness test, these two forms are closely related as shown in Fig. 2, but the simpler form produces a broader range of values than the one based on the aging rates (Eq 2).

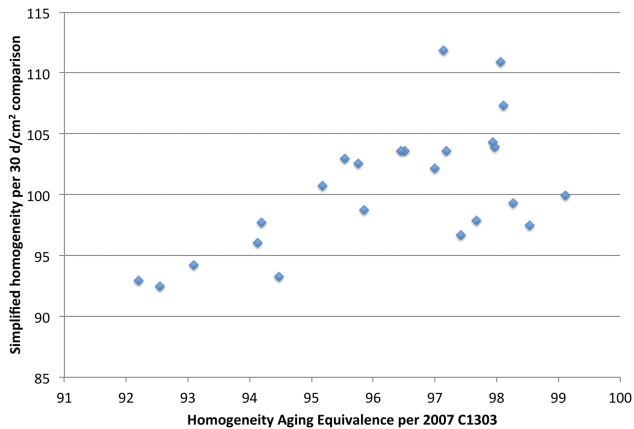
$$H_{\text{simplified}} = 100\% \left\{ 1 - \frac{(k_{\text{core}} - k_{\text{surface}})}{k_{\text{surface}}} \right\} \quad (9)$$

Both of these homogeneity definitions were examined to determine whether there was a relationship between the homogeneity value and the error. The homogeneity values were treated as both continuous variables and as class variables, where the analysis examines whether the error is statistically different between classes. This approach was used to examine multiple possible pass/fail criteria for the homogeneity criteria as shown in Table 1.

ALTERNATE PRODUCT THICKNESS

The alternate product thickness criteria tests shown in Eq 3 are used to compare the 30-day aging performance of core samples from one product thickness to core samples from another product thickness. For example, the aging performance of

FIG. 2 Comparing the 2007 C1303 homogeneity calculation to a proposed simplified version.



core samples taken from a 50 mm (2 in.) product would be compared to the aging performance of core samples taken from a 25 mm (1 in.) product. A similar comparison is made for the surface slices as shown in Eq 4. The core and surface values for these specimens range from 96 % to 104 %, so they are all within the 2007 ASTM C1303 required range of 92 % to 108 % [17,19].

In addition to comparing the aging rates, the absolute thermal conductivities after 30 days of aging are compared for both core and surface sets from each

TABLE 1 Summary of screening criteria tested for statistical significance with regard to effect on the error of the predicted thermal conductivity.

Homogeneity		Alternate Thickness (Eqs 3–6)	
(Eq 2)	Simplified (Eq 9)	Maximum and Minimum Values	Average Values
92 % to 108 %; 93 % to 107 %; 94 % to 106 %; 95 % to 105 %	94 % to 106 %	Aging only (Eqs 3–4)	k30 only (Eqs 5–6)
		92 % to 108 %; 96 % to 104 %; 97 % to 103 %	95 % to 105 %; 98 % to 102 %
		k30 (Eqs 5–6) only: 92 % to 108 %; 95 % to 105 %	
		Combination: Aging 97 % to 103 % and k30 95 % to 105 %	Combination: Aging rates 98 % to 102 % and k30 95 % to 105 %
		Combination: Aging 96 % to 104 % and k30 92 % to 108 %	
		Combination: All 92 % to 108 % (per 2007 C1303)	

product thickness (see Eqs 5 and 6). The values for the core and surface thermal conductivity comparisons for these specimens ranged from 89 % to 111 %, so some of them were outside the allowed range from 92 % to 108 %.

Other approaches to the alternate product thickness qualification were explored as shown in Table 1. For some cases, the average of the core and surface *k*-equivalence values was used. By using the average, the maximum or minimum value is no longer used for the pass/fail test. Therefore, tighter limits were explored.

Results and Discussion

For both PIR and XPS, the ruggedness test showed that it is important to restrict the stack type and to limit the product thickness used to produce the thin slices to produce more accurate predictions of the aged thermal conductivity [21]. The raw data are summarized in Fig. 3, showing that the bulk of the predictions are within $\pm 5\%$ of the full-thickness values, but there are also some outliers. In Table 2 and Fig. 4, the dataset has been reduced to only those stack types and product thicknesses recommended in the final project report. Note that these restrictions are quite effective and reduce the errors from a range of -9% to $+15\%$ to a range of -4% to $+6\%$. The mean and standard deviations summarized in Table 2, as well as the graphic distributions in Fig. 4, show that most of the errors are within -2% to $+2\%$.

This paper looks at whether the homogeneity and alternate product thickness screening criteria can further improve the accuracy of the test method.

The 2010 C1303 criteria limited the application of the test method to products with homogeneity between 90 % and 110 %. In fact, the values were all within 92 %

FIG. 3 Unscreened dataset, range of values for PIR and XPS for alternate thickness comparisons and same thickness comparisons.

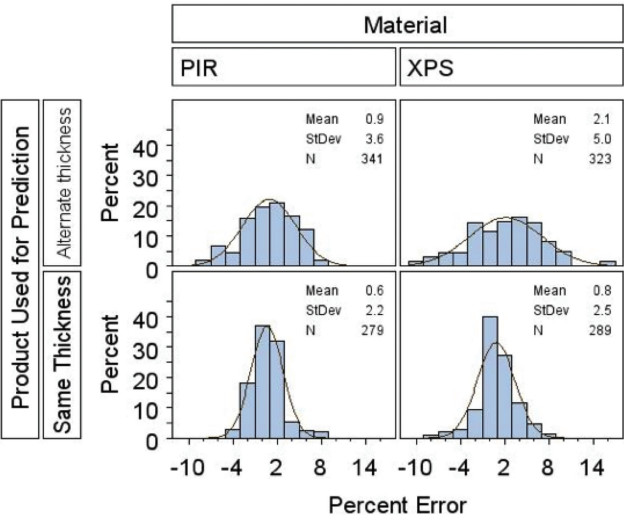


TABLE 2 Errors in predicted aged thermal conductivity for selected combinations of stack type and product origin thickness.

Material	Comparison Type	Stack Type	Mean	Standard Deviation	N
PIR	Alternate thickness	Core	−0.5	1.6	47
		Math	1.4	2.7	47
	Same thickness	Core	−0.7	2.6	51
		Profile	0.6	1.0	51
		Math	0.1	1.5	51
XPS	Alternate thickness	Math	1.3	2.2	33
	Same thickness	Profile	0.7	1.1	59
		Math	1.2	1.2	53

Note: See Figure 4 for error distribution profiles.

FIG. 4 Distribution of errors (%) in predicted aged thermal conductivity for selected stack types and original product thicknesses. Error magnitude, grouped in bins of 2 %, shown on horizontal axes; % of results for each bin shown on vertical axes.

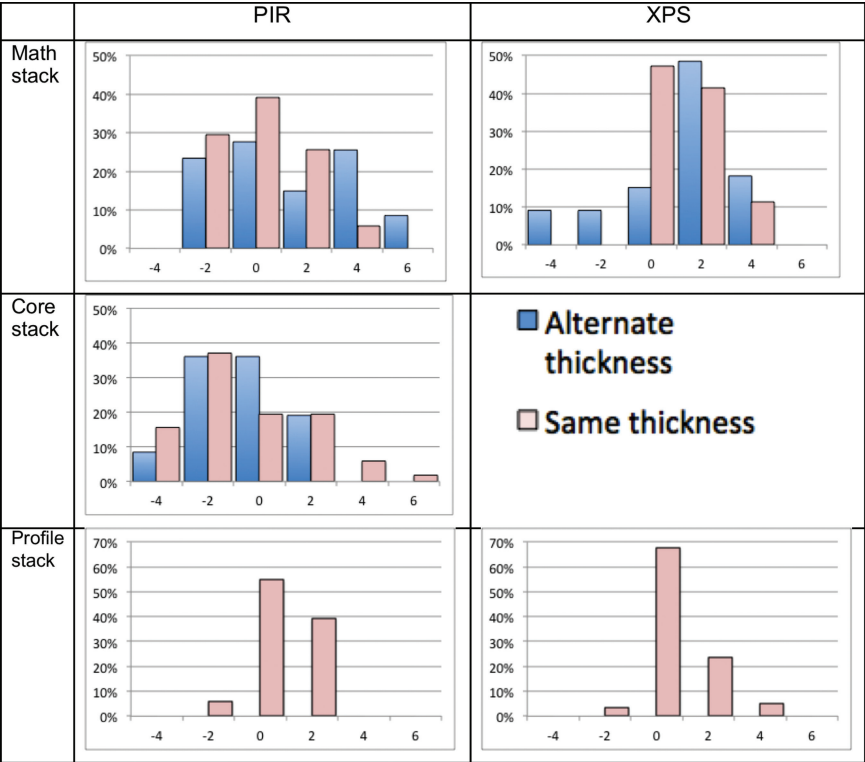
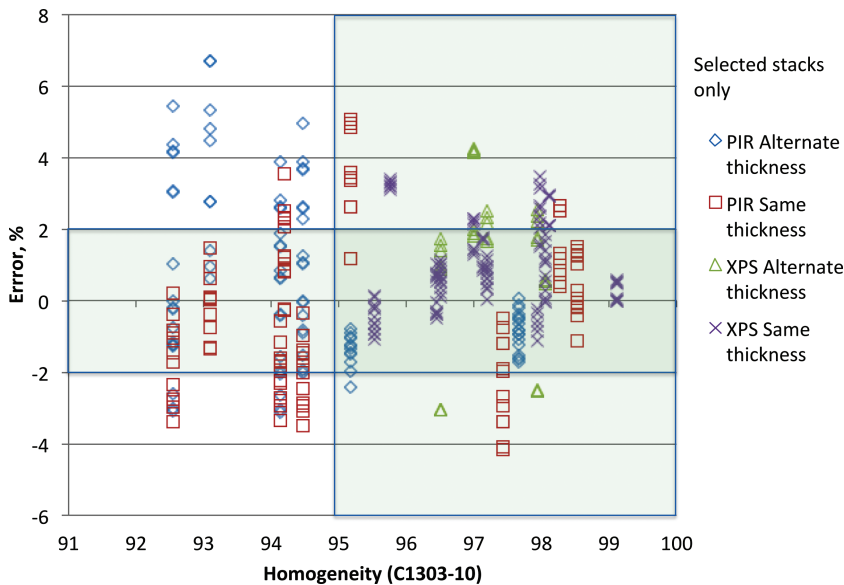


FIG. 5 Data representing recommended stack types only, looking at homogeneity based on aging as defined in the 2010 version of ASTM C1303. Shaded area from 95 % to 100 % represents “better” homogeneity. Shaded area from -2 % to 2 % represents “better” error.

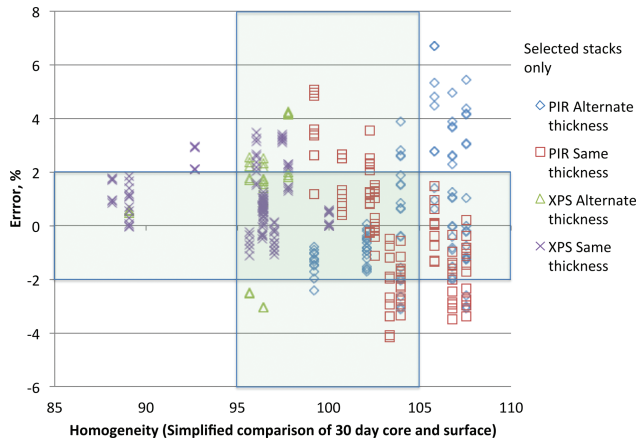


to 100 % for the XPS and PIR products tested. Figure 5 shows that tightening up that requirement (see the vertical shaded area with homogeneity between 95 % and 100 %) would not be an effective way to improve the standard. (A horizontal area has been shaded to highlight the areas of better accuracy, with errors ~ -2 % to 2 %.) Fig. 5 also shows that the homogeneity criteria from Eq 2 is not correlated with the accuracy of the aged thermal conductivity prediction.

An alternative form of the homogeneity criteria was developed during this study. This simplified version uses the 30-day thermal conductivity data alone, and would therefore simplify both the calculations and the test execution. The simplified homogeneity measure spread the values out over a broader spectrum, from ~ 85 % to 110 % for this dataset. As shown in Fig. 6, there was also no relationship between this measure and the accuracy of the predictions for the products tested here. However, if some form of homogeneity qualification is maintained in the C1303 test methodology to screen out products with extreme variations in homogeneity, this simpler measure may be useful.

Based upon the general linear model analyses, none of the proposed screening criteria listed in Table 1 were significant for the XPS products tested. For the PIR products tested, the only screening criteria found to be statistically significant were the simplified homogeneity from 94 % to 106 % and the alternate product average k_{30} from 95 % to 105 %. As Table 3 shows, every alternate product thickness case

FIG. 6 Dataset for selected stacks, looking at proposed “simplified” homogeneity measure based on a comparison of core and surface thermal conductivity at 30 days/cm². Shaded area from 95 to 105 represents “better” homogeneity. Shaded area from -2 % to 2 % represents “better” error.



that was disallowed under the 2007 C1303 alternate thickness criteria is also disallowed with the proposed criteria averaged criteria, along with a few additional cases. For both criteria, all comparisons between 25 and 100 mm (1 and 4 in.) products failed the thermal conductivity comparison tests.

The Population Marginal Means values, shown in Table 4, correct for the unbalanced nature of the dataset. These values indicate the criteria, while qualifying as statistically significant at the 95 % confidence level, are of less importance for the purpose of reducing the error in the predicted thermal conductivity than the stack type limitations. These population marginal mean values for PIR indicate that the homogeneity and alternate thickness thermal conductivity screening criteria produce results that are either insignificantly different, or actually contrary to the desired goal (that is, materials that show “better” criteria produced greater errors).

Tables 5 and 6 show the means and standard deviations for the actual test values where the dataset has been limited to recommended stack types and product source thicknesses [21]. These data show a small advantage gained by applying the modified form of the alternate thickness criteria screened at 95 % to 105 %.

Discussion and Recommendations

The broader scope of the ruggedness test showed clearly that the best way to improve the accuracy of the results is to limit the type of thin-slice stacks used and to limit the product thickness used to produce the thin slices. This portion of the work examined whether it would be possible to further improve the accuracy by applying screening criteria based on product homogeneity and similarities between products of alternate thicknesses.

TABLE 3 Alternate product thickness qualification screening criteria.

Material	Original Product Thickness mm (in.)		Product Thickness mm (in.)	No. Comparisons	2007 C1303, Showing Maximum and Minimum from the Core and Surface Stacks (92-108 % Allowed)		Average of Core and Surface (95-105 % Allowed) ^a
					Age Equivalence	k Equivalence	
PIR	25 (1)	Class 1	50 (2)	1	99-101	104-109 F ^b	107 F
			100 (4)	2	96-99	88-98 F	91-97 F
		Class 2	50 (2)	2	96-101	95-106	97-103
	50 (2)	Class 1	25 (1)	4	99-102	91-110 F	93-105 F
			100 (4)	4	97-103	102-105	102-104
		Class 2	25 (1)	2	99-104	94-105	97-103
	100 (4)	Class 1	25 (1)	2	101-104	102-112 F	103-109 F
			50 (2)	2	99-103	95-97	96-97
XPS	25 (1)	Standard Density	50 (2)	2	97-99	101-104	101-103
			100 (4)	2	96-99	88-90 F	89 F
		Standard Density	25 (1)	4	101-103	96-101	97-101
			100 (4)	4	98-102	102-106	102-104
	50 (2)	High Density	75 (3)	2	97-100	100-107	101-107 F
		Standard Density	25 (1)	2	101-104	110-112 F	111 F
			50 (2)	2	100-102	94-98	96-98 F
	75-100 (3-4)	High Density	50 (2)	2	100-103	93-100	93-99 F

^aProposed criteria.
^bF indicates that the test results exceed the allowed limits.

For the high- and low-density XPS products from two manufacturers included in this study, none of the screening criteria had a statistically significant effect on the test method accuracy.

The current homogeneity test appears to be of limited value. However, this may well reflect the relatively good degree of homogeneity in these factory-manufactured XPS and PIR foam insulation board stock products. Every specimen tested had a homogeneity value between 92 % and 100 % using the 2010 ASTM C1303 definition.

Although this qualification requirement seems unneeded for the products included in the ruggedness test, it is still desirable to maintain some form of homogeneity qualification in the standard because the theoretical foundation for

TABLE 4 Multi-factor analysis results from the full dataset: Population marginal means of “error.”

Simple 30-day Homogeneity Screen (Eq 9)	k30 Screen at 95 % to 105 % (Average of Eqs 5 and 6)	Error (Standard Deviation of Error), %	
		Alternate Thickness	Same Thickness
Pass			1.7 (0.2)
Fail			−2.3 (0.5)
Fail	Fail	0.8 (0.3)	
Fail	Pass	1.8 (0.2)	
Pass	Fail	−2.7 (0.4)	
Pass	Pass	1.3 (0.1)	

Note: Greater than 99 % confidence level (CL) except where indicated otherwise.

TABLE 5 PIR dataset single-effect results for error (%) for test methodology and screening criteria classes, limited to recommended stack type and product sources.

	Alternate Thickness			Same Thickness		
	Mean	Standard Deviation	No. of Comparisons	Mean	Standard Deviation	No. of Comparisons
k30 screen at 95 % to 105 % (average of Eq 5 and Eq 6)						
Fail	1.9	2.4	12			
Pass	0.2	2.3	83			
Simple 30-day homogeneity screen (Eq 9)						
Fail	0.8	2.5	31	−1.0	1.6	26
Pass	0.1	2.3	58	0.8	1.9	116

TABLE 6 PIR dataset multiple-effect results for error (%) for test methodology and screening criteria classes, math stack only.

Original Product Thickness, mm (in.)	k30 Screen at 95 % to 105 % (Average of Eqs 5 and 6)	Alternate Thickness			Same Thickness		
		Error	Standard Deviation	No. of Comparisons	Mean	Standard Deviation	No. of Comparisons
25 (1)	Pass	−0.3	2.6	10	0.5	1.3	23
	Fail	−6.6	0.5	9			
50 (2)	Pass	1.0	2.6	41	−0.2	1.6	28
	Fail	4.0	0.9	6			

the test method is based upon the assumption of product homogeneity. For that purpose, a simpler measurement of homogeneity is proposed that would be simpler, and therefore less costly, to execute. The simplified proposal would no longer require a test measurement at 24 h/cm², instead basing the criteria solely on comparisons of thermal conductivity taken 30 days/cm² after the slicing is complete. Neither the simpler nor the current homogeneity criteria showed statistical significance for XPS, and even for PIR, the values were contrary to the expected result. That is, the predictions for “more homogenous” materials, as defined by the 2010 version of ASTM C1303, provided the same or less accurate predictions for 5-year thermal conductivity. If the simpler homogeneity measure is adopted, broader acceptance criteria should be used based upon the results from this study, because the simpler approach spreads out the calculated values for “homogeneity” from 85 % to 110 %, so that reasonable criteria could be from 85 % to 115 %. This would simplify the test procedure and, based on the experience gained during this ruggedness test, accomplish the same goals.

For alternate product thickness applications, that is, where thin slices produced using one product thickness are used to predict the 5-year thermal conductivity for products of another thickness, the 2010 C1303 requires two separate qualification tests. Both of these tests use the measured thermal conductivity of stacks of core and surface slices. The first compares the aging rates of the two products during the period from 1 to 30 days/cm². The second compares the thermal conductivities of the two products after 30 days/cm². The aging rate comparisons showed no statistical significance whatsoever in any of the analyses for any of the products. The thermal conductivity comparisons at 30 days/cm², however, were useful in screening out some of the greatest errors for PIR products. Also, rather than requiring the surface and core *k*-equivalence be independently met, it is proposed that the average of those two values be required to be between 95 % and 105 %. This was found to serve as a more effective screen based upon the errors in the 5-year predictions than the 2007 ASTM C1303 criteria.

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