

**THERMAL
CONDUCTIVITY
20**

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THERMAL CONDUCTIVITY 20

Edited by
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PLENUM PRESS • NEW YORK AND LONDON

ISBN-13:978-1-4612-8069-9

e-ISBN-13:978-1-4613-0761-7

DOI: 10.1007/978-1-4613-0761-7

**Proceedings of the Twentieth International Thermal Conductivity
Conference, held October 19-21, 1987, in Blacksburg, Virginia**

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Softcover reprint of the hardcover 1st edition 1989**

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233 Spring Street, New York, N.Y. 10013**

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FOREWORD

The International Thermal Conductivity Conference was started in 1961 with the initiative of Mr. Charles F. Lucks and grew out of the needs of researchers in the field. The Conferences were held annually from 1961 to 1973 and have been held biennially since 1975 when our Center for Information and Numerical Data Analysis and Synthesis (CINDAS) of Purdue University became the Permanent Sponsor of the Conferences. These Conferences provide a broadly based forum for researchers actively working on the thermal conductivity and closely related properties to convene on a regular basis to exchange their ideas and experiences and report their findings and results.

The Conferences have been self-perpetuating and are an example of how a technical community with a common purpose can transcend the invisible, artificial barriers between disciplines and gather together in increasing numbers without the need of national publicity and continuing funding support, when they see something worthwhile going on. It is believed that this series of Conferences not only will grow stronger, but will set an example for researchers in other fields on how to jointly attack their own problem areas.

Of the first thirteen Conferences, only four published formal volumes of proceedings. However, effective with the Fourteenth Conference, a policy of publishing formal volumes of proceedings on a continuing and uniform basis has been established. Thus, including the present volume, the following formal volumes of proceedings have been published:

<u>Conference (Year)</u>	<u>Title of Volume</u>	<u>Publisher (Year)</u>
7th (1967)	THERMAL CONDUCTIVITY Proceedings of the Seventh Conference	U.S. Government Printing Office (1968)
8th (1968)	THERMAL CONDUCTIVITY Proceedings of the Eighth Conference	Plenum Press (1969)
9th (1969)	NINTH CONFERENCE ON THERMAL CONDUCTIVITY	U.S. Atomic Energy Commission (1970)
13th (1973)	ADVANCES IN THERMAL CONDUCTIVITY Papers Presented at XIII International Conference on Thermal Conductivity	University of Missouri, Rolla (1974)
14th (1975)	THERMAL CONDUCTIVITY 14	Plenum Press (1976)

15th (1977)	THERMAL CONDUCTIVITY 15	Plenum Press (1978)
16th (1979)	THERMAL CONDUCTIVITY 16	Plenum Press (1983)
17th (1981)	THERMAL CONDUCTIVITY 17	Plenum Press (1983)
18th (1983)	THERMAL CONDUCTIVITY 18	Plenum Press (1985)
19th (1985)	THERMAL CONDUCTIVITY 19	Plenum Press (1988)
20th (1987)	THERMAL CONDUCTIVITY 20	Plenum Press (1988)

Professors D. P. H. Hasselman and J. R. Thomas, Chairmen of the Twentieth Conference, are to be congratulated for their excellent leadership in conducting the Conference and for their painstaking efforts which made the present volume possible.

CINDAS looks forward to working with future host institutions and conference chairmen to ensure that future Conferences continue to produce high-quality volumes of proceedings in this important, specialized field.

West Lafayette, Indiana
July 1988

C. Y. Ho
Director
Center for Information and Numerical
Data Analysis and Synthesis
Purdue University

PREFACE

The 20th International Thermal Conductivity Conference (ITCC) was listed at the Virginia Polytechnic Institute and State University, Blacksburg, Virginia and sponsored by CINDAS of Purdue University, the EXXON Foundation and the Thermophysical Research Laboratory of the Department of Materials Engineering at VPI. The general chairmen of the conference were Professors D. P. H. Hasselman and J. R. Thomas. A listing of the previous ITTC is given in the subsequent pages.

The 20th ITCC was attended by 66 people, representing 12 different countries. Over forty papers were presented. Unfortunately, some of the contributors from abroad were unable to attend. Nevertheless, some of their papers are included in these proceedings. The content of the papers was presented under main subjects including: Insulation, Liquids, Metals, High-Temperature Materials, Other Materials and Effects, Methods and Composites.

At the banquet, the Thermal Conductivity Award was presented to Dr. A. Cezairliyan. Drs. H. J. Goldsmid, R. S. Graves and D. W. Yarbrough were made Fellows of the ITTC. Mr. L. F. Johnson, graduate student at VPI, was awarded the first Lucks Award.

The chairmen wish to acknowledge all those who have helped make the 20th ITTC a success. The 21st ITTC will be held at the University of Kentucky in Lexington in 1989.

Blacksburg, Virginia
October, 1987

D. P. H. Hasselman
J. R. Thomas
Co-Chairman, 20th ITTC

PREVIOUS THERMAL CONDUCTIVITY CONFERENCES

<u>Conf.</u>	<u>Year</u>	<u>Host Organization and Site</u>	<u>Chairman</u>
1	1961	Battelle Memorial Institute (Columbus, Ohio)	C. F. Lucks
2	1962	National Research Council (Canada) (Ottawa, Canada)	M. J. Laubitz
3	1963	Oak Ridge National Laboratory (Gatlinburg, Tennessee)	D. L. McElroy
4	1964	U. S. Naval Radiological Defence Lab (San Francisco, California)	R. L. Rudkin
5	1965	University of Denver (Denver, Colorado)	J. D. Plunkett
6	1966	Air Force Materials Laboratory (Dayton, Ohio)	M. L. Minges G. L. Denman
7	1967	National Bureau of Standards (Gaithersburg, Maryland)	D. R. Flynn B. A. Peavy
8	1968	Thermophysical Properties Research Center, Purdue University (West Lafayette, Indiana)	C. Y. Ho R. E. Taylor
9	1969	Ames Laboratory and Office of Naval Research (Ames, Iowa)	H. R. Shanks
10	1970	Arthur D. Little, Inc. and Dynatech R/D Co. (Boston, Massachusetts)	A. E. Wechsler R. P. Tye
11	1971	Sandia Laboratories, Los Alamos Scientific Laboratories and University of New Mexico (Albuquerque, New Mexico)	R. U. Acton R. Wagner A. V. Houghton, III
12	1972	Southern Research Institute and University of Alabama (Birmingham, Alabama)	W. T. Engelke S. G. Bapat M. Crawford
13	1973	University of Missouri - Rolla (Lake of the Ozarks, Missouri)	R. L. Reisbig H. J. Sauer, Jr.
14	1975	University of Connecticut (Storrs, Connecticut)	P. G. Klemens

15	1977	Dept. of Energy, Mines And Resources (Ottawa, Canada)	V. V. Mirkovich
16	1979	IIT Research Institute (Chicago, Illinois)	D. C. Larsen
17	1981	National Bureau of Standards (Gaithersburg, Maryland)	J. G. Hust
18	1983	South Dakota School of Mines and Technology (Rapid City, South Dakota)	T. Ashworth D. R. Smith
19	1985	Tennessee Technological University (Cookeville, Tennessee)	D. W. Yarbrough
20	1987	Virginia Polytechnic Institute and State University (Blacksburg, Virginia)	D. P. H. Hasselman J. R. Thomas

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SESSION 1

INSULATION

**LOAD-BEARING EVACUATED FIBROUS SUPERINSULATIONS -
IMPROVEMENTS WITH PEG-SUPPORT AND METAL-COATED FIBERS**

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ABSTRACT

Optimization of load-bearing evacuated thermal superinsulations requires a detailed investigation of two thermal loss channels: The solid conduction via contacting fibers, and the radiative heat transfer. In this paper we report two important findings which allow to further improve thermal superinsulations: (i) with metal-coated fibers we obtained extinction coefficients of several hundred m^2/kg ; this corresponds to a drastic reduction of radiative losses compared to the case of non-coated fibers; (ii) with peg-supported insulation systems the solid conductivity was reduced to about $0.5 \cdot 10^{-3} \text{ W}/(\text{m} \cdot \text{K})$ at 1.15 bar external pressure; this could lead to an improvement by more than a factor of three compared to not-segmented fibrous insulations. The measurements were performed with evacuable and load-controlled guarded hot plate devices and with a FTIR spectrometer.

INTRODUCTION

Load-bearing evacuated fiber systems have been shown to provide good thermal insulation at high temperatures. Best values (figure 1, curves b and c) for the total thermal conductivity are below $5 \cdot 10^{-3} \text{ W}/(\text{m} \cdot \text{K})$ at an external load of 1.3 bar and mean radiative temperatures of about 500 K (corresponding to boundary temperatures of approximately 650 K and 300 K, respectively) [1]. About 50 % of the thermal losses are caused by solid conduction via the contacting fibers, the other 50 % are from infrared (IR) radiative transport. Attenuation of radiative transport is provided by scattering and/or absorption. Typical specific extinction coefficients for plain fiber insulations are of the order of $50 \text{ m}^2/\text{kg}$.

Data for such insulations are measured with evacuable guarded hot plate devices under stationary conditions. Such systems have to be evacuated to pressures below 10^{-3} mbar in order to suppress thermal conduction by residual gas. In addition the load onto the insulating

specimen has to be adjusted for a controlled variation of the solid conductivity through the fibrous layer. IR transmission and reflection measurements are necessary in order to understand and quantify the radiative heat transfer. The spectral range to be investigated extends from about 2 μm to 50 μm .

The main topic in this report will be the question how and to which extent both solid conductivity, λ_s , and radiative conductivity, λ_r , can be reduced. Two measures pursued in this work are

- Use of a peg-supported system with a covered area fraction, $a \approx 0.18$, instead of a full-area load support.
- Application of metal-coated fibers in order to reduce the radiative heat transfer.

EXPERIMENTAL EQUIPMENT

All calorimetric data were retrieved using the two evacuable guarded hot plate machines LOLA I and LOLA II [1].

LOLA I has a circular metering section with $\phi \approx 480$ mm and two guard rings 84 and 15 mm wide. Two reference plates of size 780 x 780 mm² serve as cold boundaries and also as side walls of the vacuum chamber. The attachment of the reference plates to the frame of the vacuum chamber is flexible. This allows to exert an external pressure load p_{ext} between 0 and 1.3 bar onto the specimens. A large metering section is an absolute prerequisite for the investigation of peg-supported insulations.

LOLA II is much smaller with a metering section of $\phi \approx 120$ mm and two guard rings 18 and 20 mm wide. The external pressure load is provided by a hydraulic press, the piston of which penetrates the top part of the vacuum chamber.

Spectral IR reflection and transmission experiments were performed with a Perkin Elmer FTIR spectrometer 1700 within the wavelength range 2 to 45 μm .

MEASUREMENTS WITH PEG-SUPPORTED INSULATIONS

Measured total thermal conductivities λ and thermal loss coefficients k , respectively, generally are presented as a function of a radiative temperature $T_r = [(T_1^2 + T_2^2)(T_1 + T_2)/4]^{1/3}$. We use the diffusion model expression for the radiative heat flow

$$\dot{q}_r = 4 \cdot n^2 \cdot \sigma \cdot (T_1^4 - T_2^4) / (3 \cdot \tau_o) \quad (1)$$

n denotes the effective index of refraction of the porous medium, σ the Stefan Boltzmann constant, τ_o the optical thickness, and T_1 and T_2 are the absolute temperatures of hot and cold wall, respectively. With

$$\dot{q}_r = \lambda_r \cdot (T_1 - T_2) / d \quad (2)$$

a radiative conductivity follows (d is the thickness of the insulation):

$$\lambda_r = (16/3) \cdot n^2 \cdot \sigma \cdot T_r^3 / E_{eff} \quad (3)$$

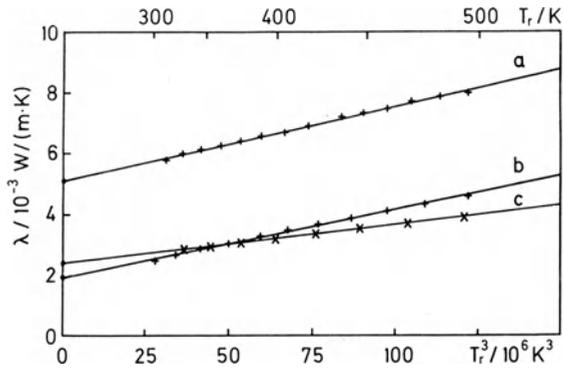


Figure 1: Thermal conductivities for several evacuated porous insulations under a pressure load of 1.3 bar as a function of T_r^3 ; curve a: fumed silica insulation, $\rho = 270 \text{ kg/m}^3$, opacified with Fe_3O_4 ; curve b: borosilicate glass fiber insulation, $\rho = 300 \text{ kg/m}^3$, the paper was thermally treated at 500°C at an external pressure load of about 1 bar; curve c: same type of glass fiber insulation opacified with Fe_3O_4 , $\rho = 330 \text{ kg/m}^3$.

Here $E_{\text{eff}} = \tau_{\text{eff}}/d$ denotes an effective value of the extinction coefficient. τ_{eff} is an effective optical thickness. In the following, λ_s describes the solid conduction part of the total thermal conductivity $\lambda = \lambda_s + \lambda_r$. The corresponding loss coefficients are k , k_s and k_r , with $k = \lambda/d$.

Figure 1, curve b, shows measured values of the total thermal conductivity λ as a function of T_r^3 of an evacuated, load-bearing glass fiber insulation (borosilicate glass, fiber $\phi \approx 1$ to $5 \mu\text{m}$, density of the insulation $\rho = 300 \text{ kg/m}^3$, porosity 0.885) for an external pressure load $p_{\text{ext}} \approx 1.3$ bar. The observed linear dependence of λ with T_r^3 allows the extraction of the specific extinction $e = E_{\text{eff}}/\rho \approx 50 \text{ m}^2/\text{kg}$ as well as an extrapolation of $\lambda(T_r)$ to $\lambda(T_r=0) \approx \lambda_s \approx 1.9 \cdot 10^{-3} \text{ W/(m}\cdot\text{K)}$. This value is considerably below the λ_s -value of an evacuated, load-bearing powder insulation (figure 1, curve a). Addition of an opacifier (Fe_3O_4) to the fiber insulation reduces the slope in the (λ, T_r^3) -plot (curve c) and thus the radiative conductivity.

Figure 2 shows the loss coefficient k and the thickness d for the same type of insulation as a function of the external pressure load p_{ext} at constant temperature. Upon compression, the loss coefficient increases. This increase can be attributed to solid conduction, which depends on the number of fiber contacts and their thermal resistance. The radiative loss coefficient is expected to be independent of p_{ext} , as long as the ir scattering processes are not altered upon compression. From $k(p_{\text{ext}})$ and $d(p_{\text{ext}})$ the total conductivity λ and its components, λ_r and λ_s , can be extracted (see figure 3). The most important result from this plot is the small increase of $\lambda_s < 2 \cdot 10^{-3} \text{ W/(m}\cdot\text{K)}$ at 1 bar to $\lambda_s < 4 \cdot 10^{-3} \text{ W/(m}\cdot\text{K)}$ at 5 bar. In other words: a five-fold increase of the load only doubles the solid conductivity.

This finding initiated a series of measurements with peg-supported insulations. The covered area fraction in these investigations was about ≈ 0.18 , thus $35 \times 35 \text{ mm}^2$ large glass fiber pegs were loaded with about 5.6 bar if the load on the total surface of the insulation was 1 bar. The number of pegs within the metering section of LOLA I was 29, thus proper spatial averaging of the thermal losses was guaranteed.

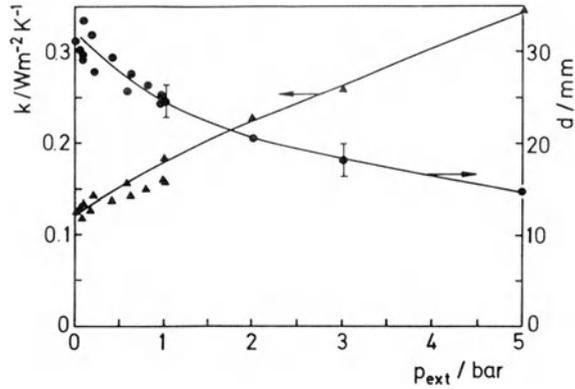


Figure 2: Total heat loss coefficient k (triangles) and thickness of samples d (circles) versus load p_{ext} at radiative temperature $T_r = 498$ K for glass fiber paper insulation.

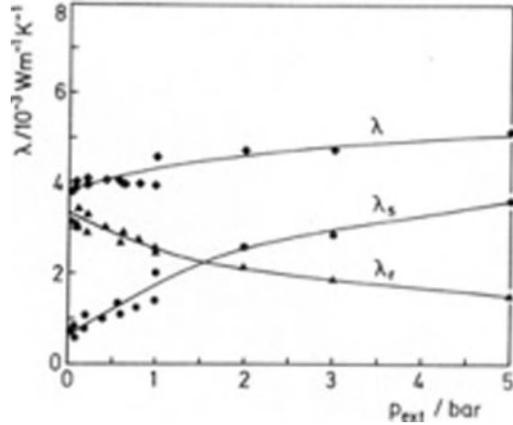


Figure 3: Total thermal conductivity λ (rhombi), and solid conductive (circles) and radiative (triangles) components λ_s and λ_r versus load p_{ext} at radiative temperature $T_r = 498$ K for glass fiber paper insulation.

First measurements were performed simply with glass fiber pegs installed in the gap between the hot plate and the reference plates, all of which had low emissivity surfaces ($\epsilon \approx 0.03$). No radiation shields were used to cover the area around the pegs. Due to the strong increase of radiative losses with temperature (see figure 4), this system would be superior to the non-segmented system (figures 2 and 3) only at $T_x < 200$ K. In order to suppress radiative losses, 10 aluminum foils were inserted into the spacing between the pegs (see figure 5). Thermal contact between the foils was prevented by separating them with thin layers of glass silk (mass per unit area and layer $m'' = 2.3 \cdot 10^{-2}$ kg/m²). As can be seen from figure 4 (curve c), firstly the losses due to solid conduction are extremely low ($\lambda_s \approx 0.5 \cdot 10^{-3}$ W/(m·K)), secondly the radiative transport is largely diminished, even with respect to the non-segmented case.

In a zero-order approximation we assume that the solid conduction is caused only by the glass fiber pegs. The thermal radiation proceeds via the fiber pegs and the remaining space. The total effective radiative conductivity then can be written approximately as

$$\lambda_x = 4 \cdot \sigma \cdot T_x^3 \cdot d \cdot \left\{ \frac{(1-a)}{(2/\epsilon_{eff}-1)(N+1)} + \frac{4 \cdot n^2 \cdot a}{3 \cdot \tau_o} \right\}. \quad (4)$$

With $N = 10$, $d = 0.015$ m, $a = 0.18$ and $\tau_o \approx 160$ (from figure 4, curve a) a value $\epsilon_{eff} \approx 0.07$ is derived if equation 4 is fitted to curve c.

As the surface emissivity for the foils is about 0.03, we have to

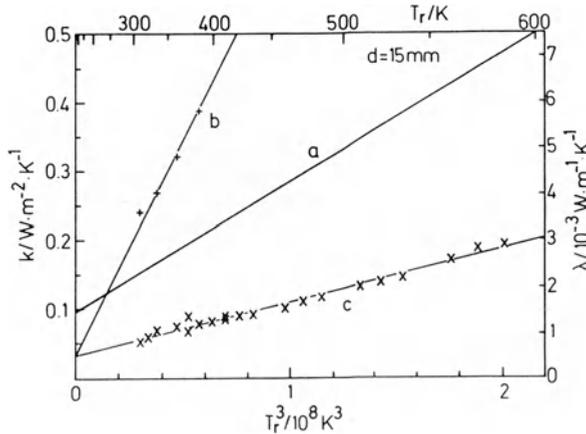


Figure 4: Loss coefficient k for various 15 mm thick insulation systems versus radiative temperature T_x at an external pressure load $p_{ext} \approx 1.15$ bar; curve a: non-segmented glass fiber paper system with $m'' = 3.46$ kg/m²; curve b: peg-supported glass fiber paper system, covered area fraction $a \approx 0.18$, peg size 35×35 mm², $m'' \approx 7.0$ kg/m² within the pegs, emissivity of boundary $\epsilon \approx 0.03$; curve c: same peg support plus additional 10 layers of aluminum foil ($\epsilon \approx 0.03$) as radiation shields.

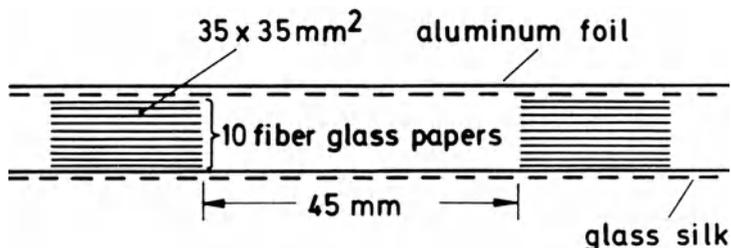


Figure 5: Segmented arrangement with glass fiber paper stacks and aluminum foils as radiation shields. The glass silk prevents direct thermal contact between adjacent Al-foils.

conclude that the glass silk is thermally coupled to the Al-foils. This leads to an increased effective emissivity of the layered structure. This effect has been discussed earlier in optically thin insulations like aerogels [2]. In order to diminish this effect, instead of the glass silk an insulating material with less mass per unit area has to be used. A second effect leading to an increased effective emissivity could be the absorption of radiation, transferred between the foils, by the peg surfaces. Which of these possibilities is the dominating one will have to be clarified by additional measurements.

In an earlier investigation [3] various peg-supported insulations have been studied. For the "IDLAS" and the "Multifoil" insulations, thermal conductivities of about 1.6 and $1.7 \cdot 10^{-3}$ W/(m·K), respectively, at $T_r \approx 500$ K are reported. These data, however, were derived using a rather small heat flux meter (5×5 cm²) covering not even 2 pegs. Furthermore gas conduction, solid conduction and radiative heat transport were treated as being independent of each other (which they are not). The radiative component was considered to be detectable from the slope of the conductivity versus temperature curve. Such a procedure is not in accordance with equation 3.

MEASUREMENTS WITH COATED FIBERS

Plain fibrous insulating materials have specific extinction coefficients of about 50 m²/kg. The extinction is caused by scattering (which is predominant in the forward direction) and by absorption. The wavelength dependence of the spectral specific extinction coefficient $e(\lambda)$ for a microglass board with fiber $\phi \approx 0.5$ to 1 μ m is depicted in figure 6. The extinction can be improved by adding suitable opacifiers, like Fe₃O₄. Though the radiative transport can be reduced (see figure 1), the added weight is non-negligible and the solid conductivity of the system usually increases.

Therefore we investigated the possibility to coat a microglass board with thin metal layers. It is known from theory that extremely thin metal fibers act like small antennae and effectively absorb thermal radiation [4]. Ni-fibers with $\phi \approx 0.3$ μ m in diam provide huge specific extinction coefficients $e \approx 1500$ m²/kg (see figure 7). The transmission measurements were performed with Ni-fibers embedded in KBr. For comparison another calculation using Mie theory for the extinction cross section was added. As such metal fibers are not readily available, we tried to produce metal-coated thin fibrous layers by using standard sputter technique instead. The coating process was applied to one side of microglass paper, and the metal penetrated into about $\frac{1}{4}$ of the paper. The specific spectral extinction derived from IR transmission measurements is shown in figure 6 (curve b). The reflectivity of the metal-coated side was about 15 %, and