

THERMOPHYSICAL PROPERTIES OF THERMALLY INSULATING MATERIALS IN THE CRYOGENIC TEMPERATURE REGION

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Abstract—Experimental and theoretical methods are discussed for determination of thermal properties of capillary-porous multicomponent systems. Stress is laid on study of thermal properties of moist porous bodies with various porosity and moisture content over a wide temperature range (80–400 K).

The results are presented of experimental determination of thermal properties (thermal conductivity, thermal diffusivity, heat capacity) of moist sands, packings of metal and glass beads at temperatures 80–400 K.

A brief description is given of unsteady experimental procedure and apparatus.

NOMENCLATURE

| | |
|------------------------|---|
| q_w | heat flux; |
| R_1, R_2 | external and internal radii of a cylinder, respectively; |
| Δt | temperature difference; |
| $dt/d\tau$ | heating rate of a sample; |
| λ_{eff} | effective thermal conductivity; |
| a | thermal diffusivity; |
| c | specific heat capacity; |
| γ | density of material under test; |
| λ_{ik} | thermal conductivity of particle (solid skeleton); |
| Π | porosity; |
| λ_k | contact thermal conductivity; |
| λ_r | molecular and radiative thermal conductivity of a pore; |
| $\lambda_{r, 3}$ | molecular and radiative thermal conductivity of a microgap between particles; |
| \bar{L} | particle diameter; |
| K_m, K_k | empirical coefficients. |

to transport a liquid in weak gravitational fields due to capillary imbibition forces. In a number of processes not only hydrodynamics of motion of a liquid or gas in a porous body is of interest but also heat transfer in a body, in particular, effective thermophysical properties of porous materials. In order that interrelated heat and mass transfer processes in porous systems be studied, it is necessary to know the permeability of a porous material, as well as the coefficient of diffusion or filtration, thermal conductivity, thermal diffusivity and specific heat capacity both of the porous material and the liquid contained in the pores.

Usually for determination of thermal conductivity and thermal diffusivity of moist porous materials there is a tendency to find such a method of determination which would reduce the effect of mass transfer upon heat transfer to a minimum. This appears to be possible when unsteady-state methods are used and when the temperature gradient in a porous material is not high (3–4°C).

This method is based on the solution of the Fourier equation for a hollow infinite cylinder with a constant heat flux at the internal surface and with ideal thermal insulation at the external

LATELY, owing to exploration of outer space, considerable interest has been roused in heat- and mass-transfer processes in capillary-porous bodies since the latter have great possibilities

surface [1]

$$\frac{\partial T}{\partial \tau} = a \left(\frac{\partial^2 T}{\partial r^2} + \frac{1}{r} \frac{\partial T}{\partial r} \right). \quad (1)$$

The initial and boundary conditions are:

$$T(r, 0) = f(r) = T_0 = \text{const}$$

$$q(r, \tau) = 0; \quad q_2(r, \tau) = q_c = \text{const} \quad \text{at} \quad \tau \neq 0.$$

The solution of equation (1) has the form [1]:

$$\begin{aligned} T(r, \tau) - T_0 = & \frac{q}{\lambda} R_2 \left\{ \frac{R_2^2}{R_2^2 - R_1^2} \left[2F_0 - \frac{1}{4} \left(1 - 2 \frac{r^2}{R_2^2} \right) - \frac{R_1^2}{R_2^2} \left(\ln \frac{r}{R_1} + \frac{R_2^2}{R_2^2 - R_1^2} \ln \frac{R_1}{R_2} + \frac{3}{4} \right) \right] + \sum_{n=1}^{\infty} \frac{\pi}{\mu_n} \right. \\ & \times \frac{T_1 \left(\mu_n \frac{R_1}{R_2} \right) T_1(\mu_n)}{T_1^2 \left(\mu_n \frac{R_1}{R_2} \right) - T_1^2(\mu_n)} \left[Y_0 \left(\mu_n \frac{r}{R_2} \right) T_1 \left(\mu_1 \frac{R_1}{R_2} \right) - Y_0 \left(\mu_n \frac{r}{R_2} \right) T_1 \left(\mu_n \frac{R_1}{R_2} \right) \right] \\ & \left. \exp(-\mu_n^2 F_0) \right\}. \quad (2) \end{aligned}$$

However, for the quasi-stationary heating of a sample by an internal constant-power heat source, the solution of equation (2) may be presented thus:

$$\begin{aligned} T(r, \tau) - T_0 = & \frac{q}{\lambda} R_2 \left\{ \frac{R_2^2}{R_2^2 - R_1^2} R_1^2 \times \left[2F_0 - \frac{1}{4} \left(1 - 2 \frac{\tau^2}{R_2^2} \right) - \frac{R_1^2}{R_2^2} R_1^2 \right. \right. \\ & \left. \left. \times \left(\ln \frac{\tau}{R_1} + \frac{R_2^2}{R_2^2 - R_1^2} \ln \frac{R_1}{R_2} + \frac{3}{4} \right) \right] \right\}. \quad (3) \end{aligned}$$

By measuring temperature at two points of a sample and heating power of a heater, it is possible to obtain the predicted formulae for thermal conductivity, thermal diffusivity and

specific heat

$$\lambda = \frac{q_w R_1}{2(R_2^2 - R_1^2) \Delta t} \left\{ R_1^2 - R_2^2 - 2R_2^2 \ln \frac{R_2}{R_1} \right\} \quad (4)$$

$$a = \frac{1}{4\Delta t} \frac{dt}{d\tau} \left\{ R_1^2 - R_2^2 - 2R_2^2 \ln \frac{R_2}{R_1} \right\} \quad (5)$$

$$c = \frac{q_w R_1}{dt/d\tau (R_2^2 - R_1^2) \gamma}. \quad (6)$$

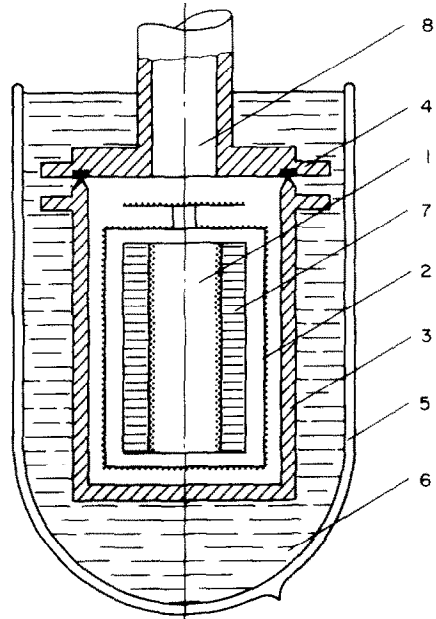


FIG. 1. Working chamber of the low-temperature device. 1—experimental volume; 2—adiabatic envelope; 3—vacuumtight glass; 4—cover; 5—Dewar flask; 6—liquid nitrogen; 7—substance under test; 8—pipe for wire output.

The experimental chamber (Fig. 1) consisted of two coaxial stainless steel cylinders 100 mm long, 40 mm and 30 mm dia., with wall thickness 0.07 mm and a welded bottom.

The material under investigation was placed in the gap between the cylinders (4–5 mm in width). The temperature gauges necessary for measuring a temperature difference and absolute temperature of a sample were pasted to the walls of the cylinders inside the gap. Copper-constantan thermocouples were used as the gauges over a temperature range of liquid nitrogen, and

platinum resistance thermometers, for helium temperatures. The readings of the thermometers were regularly made on the diagrammatic tape of the six-point recording potentiometer EPP-09. To ensure adiabatic heating conditions the experimental chamber was surrounded by an adiabatic envelope. The electric power supply for this envelope was automatically recorded by means of a differential thermocouple fixing a temperature difference between a wall of the sample and the envelope. To compensate for the heat flux, a guard heater is located inside the smaller-diameter cylinder. Thus, with the help of a system of controlled guard heaters the conditions of adiabatic heating of the material under test are ensured from liquid helium up to the room temperatures.

By means of the heater pasted to the internal surface of the cylinder with the smaller diameter the sample is monotonically heated, and the readings both of an absolute thermocouple for a temperature inside a sample and of a differential thermocouple for temperature difference between two sample points are recorded on the diagrammatic tape of the controlled electronic recording potentiometer EPP-09.

Determining Δt and dt/dt by means of the readings of the thermocouples on the diagrammatic tape, the thermophysical properties (a , λ , c) of a material may be calculated by equations (4)–(6).

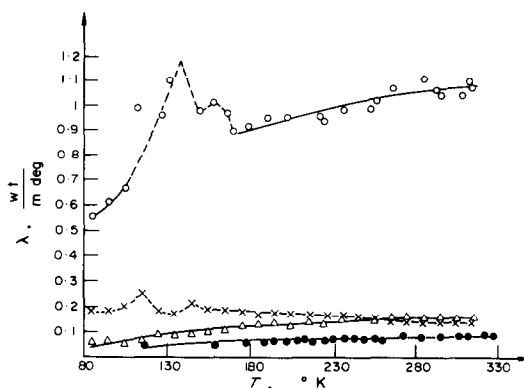


FIG. 2. Experimental relation of $\lambda(T)$ of dispersed materials. \bigcirc — Al_2O_3 powder-organosilicon liquid (in pores); \times —organosilicon liquid; \triangle — Al_2O_3 powder-air; \bullet —granulated Plexiglas-air.

The authors have experimentally found the thermophysical properties (a , λ , c) of the following multicomponent systems over a temperature range 80–300 K:

Granulated Plexiglass AKR-15-air, $\Pi = 40$ per cent; AKR-organosilicon liquid VKZh-94; glass beads ($d = 0.5$ mm, $\Pi = 35$ per cent)-air; glass beads ($d = 1.5$ mm, $\Pi = 40$ per cent)-air; glass beads ($d = 1.5$ mm, $\Pi = 40$ per cent)-organosilicon liquid VKZh-94; glass beads ($d = 1.5$ mm, $\Pi = 40$ per cent)-film coating of organosilicon liquid VKZh-air; Al_2O_3 powder-air; Al_2O_3 ($\Pi = 75$ per cent) powder-organosilicon liquid. The experimental data are presented in Figs. 2 and 3 and in Tables 1 and 2.

Table 1. Experimental data on thermal conductivity coefficients of some materials

| Material | T , K | 100° | 140° | 180° | 220° | 260° | 300° | 340° |
|---|------------------------|-------|-------|-------|-------|-------|-------|--------------|
| Granulated Plexiglas ($d = 0.5$ mm)- air | λ_{exp} | 0.070 | 0.072 | 0.073 | 0.075 | 0.078 | 0.083 | 0.039 |
| | λ_{tab} | 0.660 | 0.070 | 0.072 | 0.075 | 0.080 | 0.088 | 0.095 [3] |
| Glass beads ($d = 1.5$ mm)- air | λ_{exp} | 0.070 | 0.098 | 0.128 | 0.150 | 0.165 | 0.175 | 0.182 |
| | λ_{tab} | — | — | — | — | — | — | 0.180 [2] |
| Glass beads ($d = 0.5$ mm)- air | λ_{exp} | 0.080 | 0.110 | 0.145 | 0.170 | 0.190 | 0.209 | 0.218 |
| | λ_{tab} | — | — | — | — | — | — | 0.220 [2, 3] |
| Organosilicon liquid VKZh | λ_{exp} | 0.220 | 0.165 | 0.180 | 0.162 | 0.158 | 0.150 | 0.145 |
| | λ_{tab} | 0.205 | 0.162 | 0.158 | 0.158 | 0.156 | 0.150 | 0.142 [4, 5] |

$$P = 0.99 \times 10^5 \text{ N/m}^2$$

Table 2. Effective thermal conductivity of moist porous materials

| Material | Π (%) | λ of liquid | λ_{ck} | λ_{eff} | λ_{cal} |
|--|-----------|---------------------|----------------|-----------------|-----------------|
| Glass beads ($d = 0.5$ mm)–air | 35 | 0.028 | 1.2 | 0.22 | 0.25 |
| Glass beads ($d = 0.5$ mm)–water | 35 | 0.62 | 1.2 | 0.945 | 0.95 |
| Glass beads ($d = 0.5$ mm)–ethyl alcohol | 35 | 0.21 | 1.2 | 0.625 | 0.62 |
| Glass beads ($d = 0.5$ mm)–benzene | 35 | 0.14 | 1.2 | 0.5 | 0.52 |
| Quartz sand ($d = 0.6$ mm)–water | 32.4 | 0.62 | 5 | 2.33 | 2.2 |

$$T = 300 \text{ K}, P = 0.99 \times 10^5 \text{ N/m}^2$$

In Table 2 the experimental results are compared with the authors' predicted formula for the effective thermal conductivity of moist porous materials (two-phase system of solid particles and a liquid or a gas) [3]:

$$\frac{\lambda_{eff}}{\lambda_{ck}} = \frac{1}{[1/(h/L)^2] + A} + v_r(1 - h/L)^2 + \frac{2}{1 + h/l + [1/(v_r \cdot h/L)]}, \quad (7)$$

where

$$A = \frac{1}{\frac{\lambda_k}{\lambda_{ck}} + \frac{\pi}{16} \frac{v_{r.3}}{k_k k_m} \left(\frac{h}{L}\right)^2 \cdot 10^3};$$

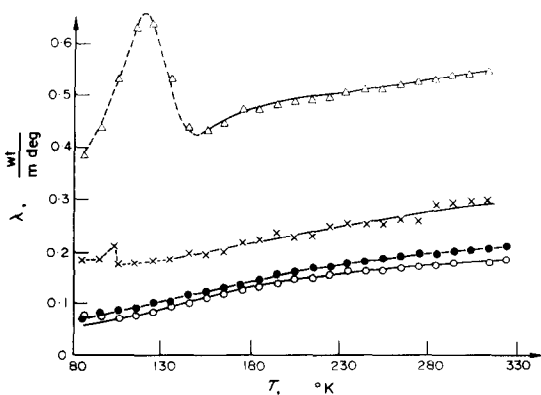


FIG. 3. Experimental relation of $\lambda(T)$ of dispersed materials. \circ —glass beads (1.5 mm dia.)–air; \bullet —glass beads (1.5 mm dia.)–air; \times —glass beads (1.5 mm dia.)–organosilicon liquid film; \triangle —glass beads (1.5 mm dia.)–organosilicon liquid (in pores).

$$L = l/h; \quad h/L = \frac{h/l}{1 + h/l}; \quad v_r = \frac{\lambda_r}{\lambda_{ck}}; \quad v_{r.3} = \frac{\lambda_{r.3}}{\lambda_{ck}}.$$

This formula takes into account a number of modes of thermal energy transfer in porous materials. The main difficulty in calculations is to allow for contact heat transfer between particles.

The comparison of the experimental data obtained with those of different authors has confirmed the applicability of the developed method of determination of thermophysical properties of granular materials and liquids over a wide temperature range.

This method is also promising for determination of the effective thermal conductivity of lattice-type inserts filled with a liquid used in heating pipes.

From Figs. 2 and 3 it is seen that the method proposed by the authors enables to register a phase transition zone of materials. So, for example, the first-kind phase transition in an organosilicon liquid over a temperature range of 100–140 K has been observed when the liquid goes over into amorphous ice. In this case two peaks of the thermal conductivity at temperatures of 110 K and 140 K are noticed.

These phase transitions are clearly seen when studying the thermal conductivity of an organosilicon liquid–powdered Al_2O_3 mixture since Al_2O_3 has high thermal conductivity of the solid skeleton.

Good agreement of the experimental data

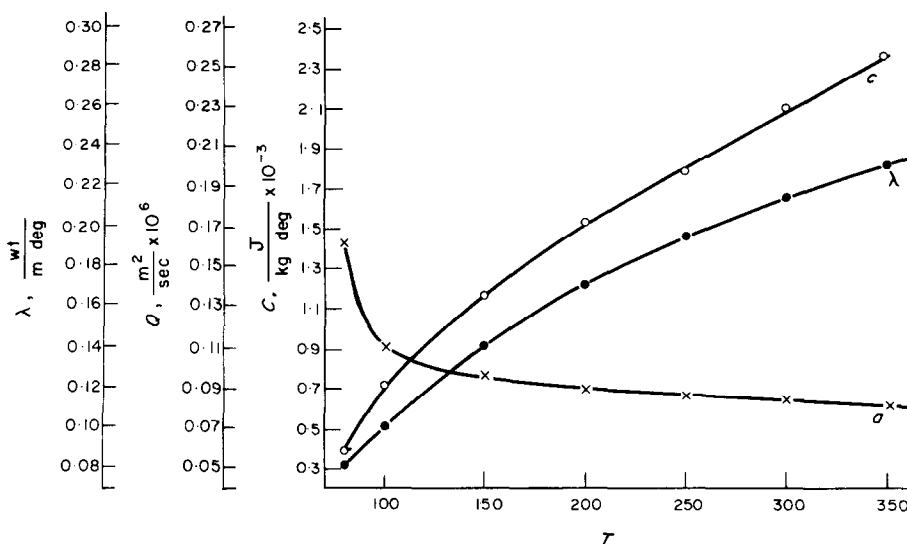


FIG. 4. Experimental dependence of thermophysical properties on temperature of crushed glass ($0.3 \text{ mm} < d < 0.6 \text{ mm}$, 43 per cent porosity). ●—thermal conductivity $\lambda(T)$; ○—heat capacity $C(T)$; ×—thermal diffusivity $a(T)$.

(Table 2) with the predicted values of λ_{eff} obtained by formula (6) shows that in most cases experimental determination of thermal conductivity of composite materials is not necessary. With the initial components known, it is possible to calculate effective thermal conductivity of heterogeneous systems.

Moreover, it is possible to indicate the way how to create composite materials with prescribed thermal properties.

Great possibilities of changing thermal conductivity of heterogeneous systems are facilitated by the complex mechanism of transfer heat. Absolute values and ratios of thermal conductivities of the components, their void fractions (porosity), diameters of particles and their pores, optical properties of substances, surface properties, shape of particles, packing, mechanical load on particles, their elastic properties, etc. are parameters which determine the effective thermal conductivity.

Thus, the present authors consider that study of thermal properties of pure substances and composite materials should follow the trends:

1. Experimental determination of thermal

properties of pure substances over wide ranges of temperature and pressure;

2. Development of analytical models of heterogeneous systems and calculation of composite material properties by appropriate equations (analytical or semiempirical) using experimental data on pure substances.

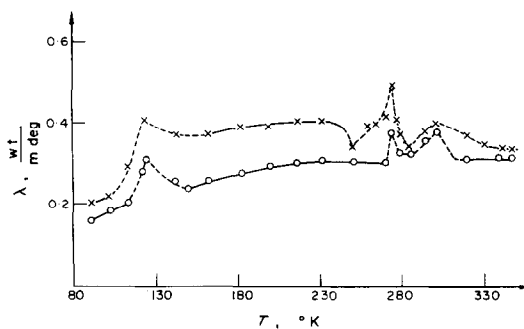


FIG. 5. Experimental relation of $\lambda(T)$ of moist quartz sand ($d = 0.5 \text{ mm}$, II porosity = 30.1 per cent). ×—moisture content 0.543 per cent.

Such an approach allows the demands of industry to be met, as it creates and uses more and more new materials.

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PROPRIETES THERMOPHYSIQUES DE MATERIAUX THERMIQUEMENT ISOLANTS DANS LA REGION DE TEMPERATURE CRYOGENIQUE

Résumé—On a discuté les méthodes expérimentales et pratiques de détermination des propriétés thermiques de systèmes microporeux à plusieurs composants. L'accent est porté sur l'étude des propriétés thermiques de corps poreux humides à porosité et humidité variables dans un large domaine de température (80–400° K).

On présente les résultats d'une détermination expérimentale des propriétés thermiques (conductivité thermique, diffusivité thermique, capacité calorifique) de sables humides, de lits à garnissage métallique ou en verre, à des températures comprises entre 80 et 400° K.

On a fait une brève description de l'installation et de la méthode expérimentale instationnaire.

WÄRMETECHNISCHE STOFFGRÖSSEN VON ISOLIERMATERIALIEN IM TIEFTEMPERATURBEREICH

Zusammenfassung—Experimentelle und theoretische Methoden zur Bestimmung thermischer Stoffgrößen von kapillar-porösen Vielkomponenten-Systemen werden besprochen. Hauptsächlich wurden die thermischen Stoffgrößen feuchter, poröser Körper von unterschiedlicher Porosität und Feuchtigkeit in einem grossen Temperaturbereich (80–400 K) untersucht.

Für feuchten Sand und Schüttungen von Metall- und Glaskügelchen sind die experimentellen Ergebnisse für die Wärmeleitfähigkeit, die Temperaturleitzahl und die Wärmekapazität im genannten Temperaturbereich angegeben.

Kurz beschrieben wird eine Versuchseinrichtung für instationäre Messungen.

ТЕПЛОФИЗИЧЕСКИЕ СВОЙСТВА ТЕПЛОИЗОЛЯЦИОННЫХ МАТЕРИАЛОВ В ОБЛАСТИ КРИОГЕННЫХ ТЕМПЕРАТУР

Аннотация—Рассматриваются экспериментальные и теоретические методы определения теплофизических характеристик капиллярнопористых многокомпонентных систем. Основное внимание уделяется исследованию тепловых свойств влажных пористых тел с различной пористостью и влагосодержанием в широком диапазоне температур (80–400°K).

Приводятся результаты экспериментального определения теплофизических характеристик (теплопроводность, температуропроводность, теплоемкость) влажных песков, засыпок из стеклянных и металлических шариков при температурах 80–400°K.

Кратко описывается нестационарная методика и установка для исследования материалов.