

TEMPERATURE DEPENDENCE OF HELIUM PERMITTIVITY
THROUGH FREELY Poured QUARTZ GLASS POWDER

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The temperature dependence of relative helium flow through a free poured powder of amorphous SiO_2 held in a quartz glass cell is determined experimentally.

The motion of gases in porous media is widely used in chemical technology and is often found in natural phenomena. Therefore gas transport in dispersed systems, in rigid porous structures, and in fluidized layers has been studied in many works [1-6]. However, the literature has insufficiently illuminated questions involving the temperature dependence of gas permeability through powders, in particular, questions involving possible changes in the structure of freely poured powders under loading.

In order to investigate the effect of possible changes in structure on gas permeability through freely poured powders (the specimens) upon their heating the apparatus shown schematically in Fig. 1 was constructed.

At the beginning of an experiment the apparatus was filled with helium. The gas pressure was monitored with a reference manometer 3 of accuracy class 0.15 or with optical manometer 2. The specimen to be studied was placed in quartz cell 5: inner diameter of the cell was 3.36 mm, with wall thickness of 0.5 mm. After the specimen was installed, to eliminate air the system was evacuated with forevacuum pump 7 and washed with helium from source 1 for some time.

In the first step of the experiment the helium flow rate through the specimen at room temperature T_r was measured, after which flow rate was measured during a continuous heating of the specimen to fixed temperature values. After a temperature of $T = 1173^\circ\text{K}$ was reached the specimen was cooled and the flow rate at T_r was measured again.

During the course of the experiments the gas pressure at the input to the cell was maintained constant, with the gas continually escaping through the specimen into the atmosphere or a vacuum. The pressure range studied comprised 0.007-0.130 MPa.

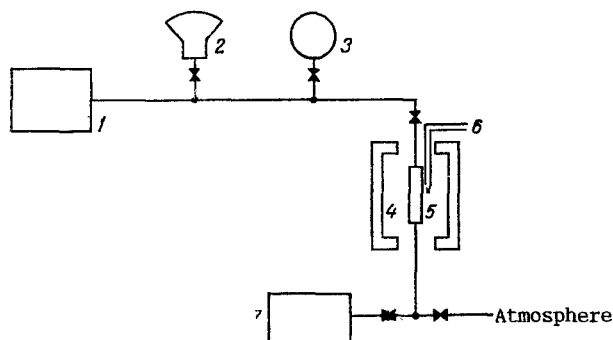


Fig. 1. Schematic diagram of experimental apparatus.

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The specimen was heated by furnace 4, the heating element of which was a nichrome wire 0.8 mm in diameter. Temperature measurements were made with a Chromel-Alumel thermocouple 6 to an accuracy of 0.1°C. With a specimen height of 100 mm the temperature difference between its ends did not exceed 1% of the maximum temperature recorded in the center of the specimen.

Helium mass flow rates were determined by the nonsteady state flow method. During the course of the experiments a Ch3-34 electronic clock was used to measure the time required for the manometer indicator to traverse one and the same section of its scale to an accuracy of 0.01 sec. The pressure change at the cell input during the measurement period did not exceed 5% of the absolute pressure. Flow rates were measured over the range 0.3-0.003 cm³/sec.

The specimens used were powders of porous and nonporous quartz glass. The nonporous specimens were obtained by grinding a quartz tube prepared in the city of Gus'-Khrustal'nyi. In order to study the effect of powder dispersion on its behavior at various temperatures standard sieves were used to prepare two fractions of nonporous quartz glass, which when analyzed with a "5000 M" sedigraph indicated mean grain diameters of 21 and 9 μm. The porous specimens were prepared by the chemistry department of Sverdlovsk Polytechnic Institute. Mean grain diameter for those specimens was 36 μm. Pore diameter within the grains was 50-100 nm. Specific surface of the porous specimens was 20 m²/g.

The model of identical straight capillaries [7] can be used to describe gas motion in a porous medium. In accordance with this model the gas motion in the porous medium can be described with the aid of Poiseuille's and Knudsen's laws for the viscous and free-molecular gas flow regimes in a straight channel:

$$Q^b = \frac{n\pi R_c^4}{8L} \frac{\bar{P}}{k} \frac{\Delta P}{\eta T} \quad (1)$$

and

$$Q^m = \frac{2}{3} \frac{n\pi R_c^3}{L} \sqrt{\frac{8R}{\pi M}} \frac{\Delta P}{k} \frac{1}{\sqrt{T}} \quad (2)$$

When interested in the temperature dependence of gas permeability, it is convenient to utilize the ratio of the gas flow through the porous medium at a specified temperature to the flow rate corresponding to room or some other predefined temperature T₀. In this case, using Eqs. (1), (2), it is simple to obtain the dependence of relative flow rate in the viscous Q_r^v and free molecular Q_r^m gas flow regimes on temperature:

$$Q_r^v = \frac{\eta_0 T_0}{\eta T}, \quad (3)$$

$$Q_r^m = \sqrt{\frac{T_0}{T}}. \quad (4)$$

In the intermediate gas flow regime the flow rate Qⁱ can be determined with the expression

$$Q^i = \omega(Kn) Q^m. \quad (5)$$

Values of ω(Kn) are tabulated in [8] for the range Kn = 0.1-100. The number Kn which is realized in a porous medium for a given mean pressure and temperature can be determined from the expression

$$Kn = \frac{\sqrt{\pi}}{4} \left[\left(\frac{2RT}{M} \right)^{1/2} \eta \right] \frac{1}{\bar{P} R_p}. \quad (6)$$

The pore radius R_p in the porous medium can be determined from the mean grain diameter d_g from an expression presented in [9]

$$R_p = \frac{1}{2} \Pi^2 d_g, \quad (7)$$

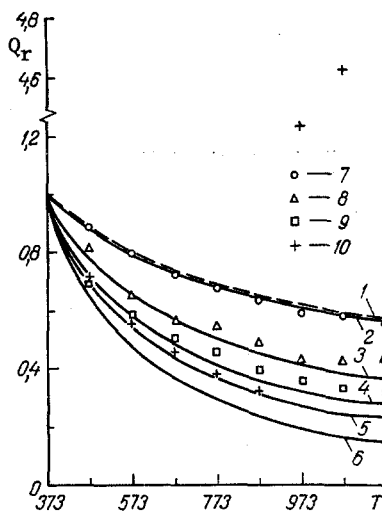


Fig. 2. Temperature dependence of relative helium flow rate through free poured powder of quartz glass (1-6, theory; 7-10, experiment): 1) $Q_r^m(T)$, $Kn = \infty$; 2) $Q_r^i(T)$, $Kn = 0.850-3.41$; 3) $Q_r^i(T)$, $Kn = 0.09-0.36$; 4) $Q_r^s(T)$, $Kn = 0.05-0.2$; 5) $Q_r^s(T)$, $Kn = 0.019-0.098$; 6) $Q_r^v(T)$, $Kn = 0$; 7) $P = 0.007$ MPa, $d_g = 21 \mu\text{m}$ (escape into vacuum); 8) 0.130 and 9; 9) 0.120 and 21; 10) 0.115 and 36 ((8-10), escape into atmosphere) (7-9, Q_r for specimens of nonporous quartz glass; 10, Q_r for porous quartz glass specimens). T , °K; Q_r , dimensionless.

where Π is the specimen porosity.

Using Eqs. (4), (5), we obtain the dependence of relative gas flow rate in the intermediate flow regime Q_r^i upon temperature

$$Q_r^i = \frac{\omega(Kn)}{\omega(Kn_0)} Q_r^m. \quad (8)$$

Here $\omega(Kn)$ and $\omega(Kn_0)$ are calculated for one mean pressure, but at different temperatures T and T_0 , respectively.

For numbers $Kn \ll 1$ a viscous flow regime with slippage is realized in the channels. If we take the coefficient of diffuse molecular reflection on the wall $\epsilon = 1$, then in the first approximation in Knudsen number Kn the gas flow rate Q^s will be determined [8] by the expression

$$Q^s = Q^v \left[1 + \frac{4(\pi+4)Kn}{\pi} \right]. \quad (9)$$

Then the temperature dependence of the relative gas flow rate in the viscous flow regime with slip Q_r^s will have the form

$$Q_r^s = \frac{4(\pi+4)Kn + \pi}{4(\pi+4)Kn_0 + \pi} Q_r^v. \quad (10)$$

Figure 2 shows experimental data for helium flow rate as a function of temperature in free poured powders of porous and nonporous quartz glasses. The temperature used for T_0 was $T_0 = 373^\circ\text{K}$. The same figure shows theoretical dependences of gas flow rate on temperature for $Kn = 0$, $Kn = \infty$, and Kn corresponding to the experimental data. For nonporous quartz glasses the difference between theoretical and experimental values does not exceed 3%. Thus it can be concluded that during heating of the nonporous quartz glass specimens from 300 to 1173°K in a continuous helium flow no changes in their geometric structure take place.

The experimental values of Q_r for the porous glass specimens show good agreement with the calculations only to $T = 873^\circ\text{K}$. For $T = 973$ and 1073°K the relative gas flow rates in experiment increase in comparison to the calculated values by factors of 3.5 and 17 times, respectively. It is obvious that changes have occurred in the structure of these specimens related to their heating in a medium of continuous helium flow. The irreversible character of these changes was confirmed by comparing helium flow rates through the specimen at T_r before and after specimen heating. In the given case the flow rate at T_r through the specimen after heating increased by a factor of ~ 20 times as compared to the flow rate at T_r before heating. Visual observation of the column of sintered glass formed as well as a decrease in porosity by a factor of 1.5 times confirm the changes which took place in the structure.

Consequently, the temperature dependence of relative gas flow rate determined in experiment permits detection of the beginning of the sintering process in the material studied.

NOTATION

Q^v , Q^m , Q^i , Q^s , flow rates for viscous, free-molecular, intermediate, and viscous slip regimes; Q_r^v , Q_r^m , Q_r^i , Q_r^s , Q_r , calculated relative gas flow rates for viscous, free-molecular, intermediate, and viscous slip regimes and experimental relative flow rate; n , number of channels in porous body; R_c , radius of single channel; \bar{P} , mean gas pressure; ΔP , pressure head across ends of cell; T , gas temperature; T_0 , specified gas temperature; T_r , room temperature; Kn , Knudsen number; Kn_0 , Knudsen number at $T = T_0$; $\omega(Kn)$, relative gas flow rate as function of Kn ; $\omega(Kn_0)$, relative gas flow rate at $Kn = Kn_0$; η , gas viscosity; η_0 , gas viscosity at $T = T_0$; M , molecular mass of gas; d_g , mean specimen grain diameter; R_p , specimen pore radius; R , universal gas constant; k , Boltzmann's constant.

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