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PHYSICAL REVIEW B VOLUME 4, NUMBER 8 15 OCTOBER 1971

Diffusion of Helium Isotopes in Vitreous Silica*

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The diffusion of He^3 and He^4 in vitreous silica was experimentally measured in the temperature range 100–760 °C. The ratio of the diffusion coefficients is given by $D(He^3)/D(He^4) = (1.205 \pm 0.003) \exp[-(131 \pm 7 \text{ cal/g-at.})/RT]$. These results cannot be explained by classical diffusion theory, which does not predict an isotope effect in the activation energy for diffusion. The quantum corrections proposed by LeClaire for the special case of low-atomic-mass isotope diffusion satisfactorily explain the observed results.

I. INTRODUCTION

There have been several studies of the diffusion of He^4 in vitreous silica,[1–5] but only one determination of the relative diffusivities of He^3 and He^4 in vitreous silica has been reported.[6] Jones[6] calculated the isotopic diffusivity ratio from the corresponding ratios for permeability and solubility. However, no direct determination of the diffusivity of He^3 in glass has been previously reported.

The magnitude of the experimentally observed isotope effect, when compared with theoretically predicted values, should allow a distinction between various possible diffusion mechanisms. Classical diffusion theory predicts that the ratio of the isotopic diffusion coefficients should vary as the reciprocal of the square root of the mass of the diffusing atoms.[7] LeClaire[8] has suggested that a quantum correction be applied to classical theory when the diffusing species are of low atomic mass. He has applied these corrections to hydrogen isotope diffusion in metals with some success. However, this analysis has not been tested on helium isotope diffusion because of a lack of experimental data. The relatively large values of the He^4 diffusivity in vitreous silica[1–5] would suggest that a helium isotope diffusion study in this material might provide a significant test of LeClaire's the-

TABLE I. Thickness and chemical analysis of diffusion specimens. (ND means not detectable.)

Material	Thickness (mm)	Concentration in ppm Al	Na	Ca	Fe	OH
Spectrosil	1.56 ± 0.03	ND	3	ND	ND	1280
Infrasil	1.63 ± 0.01	64	21	ND	11	11

ory. This work reports the results of such a study.

II. EXPERIMENTAL PROCEDURE

A. Materials and Preparation

The vitreous silica diffusion specimens used in this study were disks of Spectrosil A (Thermal Syndicate, Ltd.) and Infrasil (Amersil, Inc.). The former is produced by flame hydrolysis of $SiCl_4$, whereas the latter is produced by fusion of powdered crystalline quartz. Results of an atomic absorption and infrared chemical analysis of each specimen are given in Table I, as is the specimen thickness. Each specimen was optically ground and polished by the supplier and had a nominal diameter of 3.8 cm. Each disk was sealed into a vitreous silica tube in such a manner that it formed a boundary between two chambers (Fig. 1). The flame used in sealing was applied to the other surface of the vitreous silica tube and at no time was in direct contact with the specimen.

B. Diffusion Measurements

The experimental procedure was similar to that described previously by Swets, Lee, and Frank.[1] The apparatus is shown schematically in Fig. 2. One chamber of the specimen tube was connected to the mass spectrometer system and the other chamber to a gas manifold whereby the desired gas could be introduced to the specimen. Three series of measurements were made on the Spectrosil specimen: the first using He^4, the second He^3, and finally using an approximately equimolar mixture of these two helium isotopes. Measurements of the first and third type were also made using the Infrasil specimen. The specimen was brought to equilibrium at the desired temperature with a pressure of 2×10^{-7} Torr on the high-vacuum surface and a pressure of 1×10^{-3} Torr on the surface to be exposed to the gas. The appropriate isotope or isotopic mixture was then admitted to the system. The mass spectrometer continuously monitored the permeation of the helium through the specimen. After a constant permeation rate was attained, indicating steady-state flow, the helium was evacuated from the higher-pressure system and the process of helium evolution from the specimen recorded. Measurements were made at temperatures between 100 and 760 °C, with a total of 37 points for He^4, 28 for He^3, and 20 for the isotopic mixture for the Spectrosil sample, and 23 points for He^4 with 15 for the isotopic mixture for the Infrasil sample. Sample temperature as indicated by a thermocouple located within 0.3 cm of the specimen surface was maintained within ± 1 °C throughout each measurement.

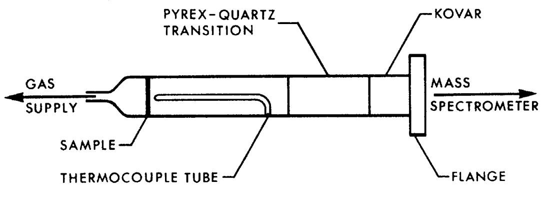

FIG. 1. Sample configuration for measurement of helium diffusion in vitreous silica.

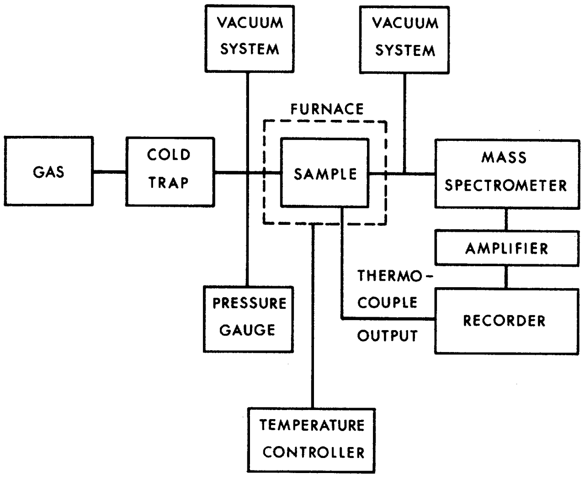

FIG. 2. Mass spectrometer for diffusion and permeation measurements.

A thorough discussion of the mathematics utilized to calculate the diffusion coefficient from the helium evolution curve is readily available in the literature.[1] It can be shown that, although a complete mathematical description of the evolution process involves a converging series of exponentials, the rapid convergence of this series allows the major portion of the evolution curve to be described by a single exponential. Thus the diffusion coefficient can be determined from the equation

$$D=\frac{L^2\ln(h_1/h_2)}{\pi^2(t_2-t_1)}\,, \tag{1}$$

where D is the diffusion coefficient, L is the specimen thickness, and h_1 and h_2 are the relative permeation rates at times t_1 and t_2, respectively.

TABLE II. Diffusion coefficients of He^3 and He^4 in vitreous silica (Spectrosil).

Temperature (°C)	$D(He^3)$ (10^{-6} cm^2/sec)	$D(He^4)$ (10^{-6} cm^2/sec)	$D(He^3)/D(He^4)$
286	2.26	2.11	1.070
306	2.88	2.67	1.079
329	3.62	3.36	1.080
348	4.31	3.99	1.081
368	4.89	4.48	1.089
374	5.07	4.67	1.086
397	5.97	5.46	1.093
415	6.73	6.14	1.097
424	6.99	6.43	1.087
438	8.09	7.40	1.094
456	8.89	8.03	1.106
474	9.95	9.06	1.099
498	11.3	10.2	1.107
513	12.5	11.3	1.104
524	12.9	11.7	1.107
546	14.9	13.3	1.120
565	16.5	14.8	1.116
582	17.5	15.6	1.120
595	18.8	16.8	1.116
602	19.1	17.1	1.120

III. RESULTS

A. Diffusion Measurements

Diffusion coefficients of He^3 and He^4 in vitreous silica were measured at random temperatures from 100 to 760 °C. The technique involved both simultaneous measurements utilizing an isotopic mixture (method I) and sequential measurements utilizing isotopically pure gases (method II). Although no isotopic interaction effects were expected, this procedure was adopted to ascertain the independence of the measurements. No substantial difference was detected in the two methods. The results of the method-I measurements on the Spectrosil specimen are given in Table II as well as graphically in Figs. 3 and 4. The data for the Infrasil sample are essentially the same and are not included for brevity.

Data such as those in Table II are frequently expressed mathematically by utilizing a least-mean-squares fit to the equation[7]

$$D = D_0 e^{-Q/RT}, \tag{2}$$

where D_0 is a constant pre-exponential factor, Q is the activation energy in cal/g-at., R is the gas constant, and T is the absolute temperature. However, it has been recently demonstrated that for the case of helium diffusion in vitreous silica, a more realistic representation of the data is obtained if the usual expression is modified by inclusion of a temperature-dependent pre-exponential term,[5] i.e.,

$$D = D_0 T e^{-Q/RT}, \tag{3}$$

where D_0 is now the constant portion of the pre-exponential term and both D_0 and Q have values different from those obtained from Eq. (2). The values of D_0 and Q obtained from both methods I and II are given in Table III. There is no evidence that the results are affected by the choice of experimental technique.

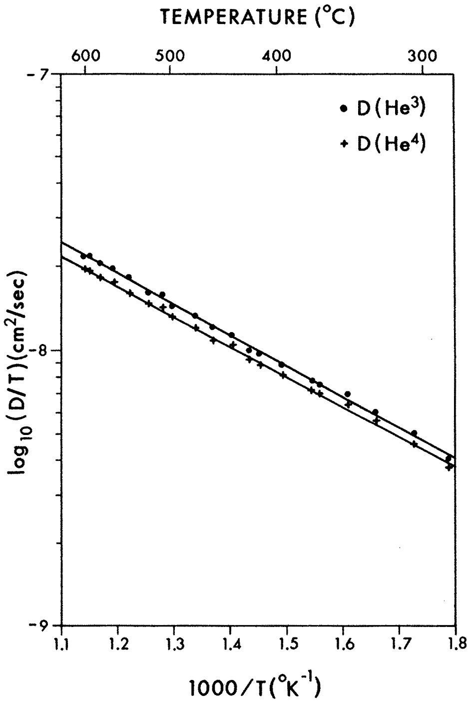

FIG. 3. Temperature dependence of helium isotope diffusion coefficients in vitreous silica (Spectrosil).

The temperature dependence of the isotopic dif-

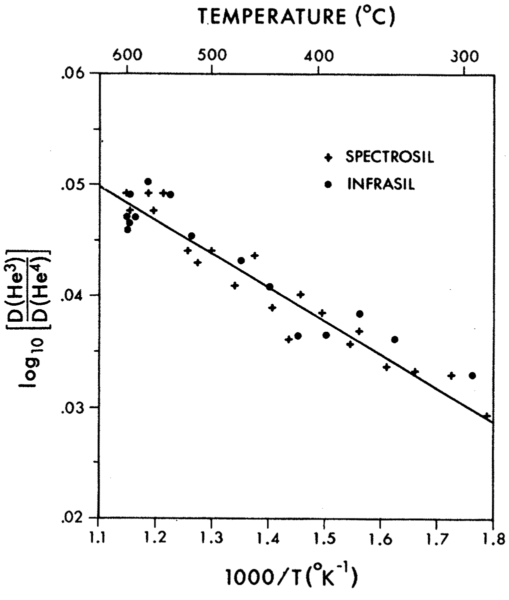

FIG. 4. Temperature dependence of the isotope diffusion coefficient ratio for helium in vitreous silica.

TABLE III. Activation energy and constant portion of pre-exponential term for He^3 and He^4 diffusion in vitreous silica.

Experimental material and method		$D_0(He^3)$ (10^{-7} $cm^2/sec\,deg$)	$D_0(He^4)$ (10^{-7} $cm^2/sec\,deg$)	$Q(He^3)$ (cal/g-at.)	$Q(He^4)$ (cal/g-at.)
Spectrosil:	method I	4.088 ± 0.020	3.378 ± 0.020	5082 ± 48	4945 ± 48
	method II	4.091 ± 0.020	3.313 ± 0.019	5048 ± 43	4946 ± 24
Infrasil:	method I	3.884 ± 0.008	3.241 ± 0.007	5018 ± 42	4898 ± 40
	method II	• • •	3.013 ± 0.007	• • •	4844 ± 35

fusion coefficient was determined by a least-mean-squares fit of the values of $D(He^3)/D(He^4)$ given in Table II versus reciprocal temperature. These results are expressed by

$$D(He^3)/D(He^4) = (1.210 \pm 0.003)\exp[-(137 \pm 6\ \text{cal/g-at.})/RT] \quad (4)$$

for the Spectrosil specimen,

$$D(He^3)/D(He^4) = (1.198 \pm 0.004)\exp[-(121 \pm 12\ \text{cal/g-at.})/RT] \quad (5)$$

for the Infrasil specimen, and

$$D(He^3)/D(He^4) = (1.205 \pm 0.003)\exp[-(131 \pm 7\ \text{cal/g-at.})/RT] \quad (6)$$

for the combined Spectrosil and Infrasil data.

In a previous study, Jones[6] measured the permeability ratio and the solubility ratio of He^3 to He^4 in vitreous silica. The permeability K is equal to the rate of diffusion of individual atoms D multiplied by the number of diffusing atoms S, i.e., $K = DS$. Jones utilized this expression to calculate $D(He^3)/D(He^4)$, obtaining the relationship

$$D(He^3)/D(He^4) = (1.167 \pm 0.005)\exp[-(51 \pm 3)/RT]. \quad (7)$$

There appears to be a discrepancy between Jones's results and those of the present study. It should be noted, however, that the results of the present study were obtained from direct measurement of the He^3 and He^4 diffusion coefficients, whereas those of Jones are derived indirectly from permeation and solubility measurement. In addition, the agreement between the values obtained in the present study for two vitreous silicas of considerably different types, coupled with the large number of data points used in this study as compared to the seven points measured by Jones, increases confidence in the values reported in this paper.

B. Error Analysis

Two types of errors must be considered in the determination of any experimental quantity: the reproducibility of the data (the precision) and the correctness of the absolute magnitude of the quantity (the accuracy). An excellent discussion of the analysis of these errors in isotopic diffusion studies is found in the work of Frank, Swets, and Lee,[9] whose techniques have been applied to the error analysis of the present study.

Since the errors in the activation energies and in the ratio of the D_0's are dependent only upon the relative values of the various diffusion coefficients, they are derived entirely from the precision of the experiment. The standard deviation of the activation energy is calculated from the expression

$$\sigma_Q = R\left(\frac{1-r^2}{N-2}\right)^{1/2}\frac{\sigma_y}{\sigma_x}, \quad (8)$$

where r is the correlation coefficient, N is the number of data points, σ_y is the standard deviation in $\ln D$, and σ_x is the standard deviation in the reciprocal temperature. All quoted limits on Q are 1 standard deviation.

The standard deviation in the D_0's is a function of both the scatter in the data points and the uncertainty of the activation energy. The expression

$$\sigma_0 = \sigma_y\left[\frac{N-1}{N-2}(1-r^2)\left(\frac{N^{-1}+\bar{x}^2}{(N-1)\sigma_x^2}\right)\right], \quad (9)$$

where $\bar{x}$ is the mean value of the reciprocal temperature, can be used to calculate σ_0, the standard deviation in D_0. The only additional error contributing to the absolute accuracy of the D_0's is that in measuring the specimen thickness as shown in Table I. This error is combined with the precision of the D_0's by the usual procedure of using the square root of the sum of the squares of the fractional errors as the over-all error. The errors expressed previously in the pre-exponential terms for He^3 and for He^4 diffusion are of this type. However, since the thickness error affects both the He^3 and He^4 diffusion coefficients equally, only the standard deviation, as calculated from Eq. (9), must be included in calculating the error in the value of $D_0(He^3)/D_0(He^4)$.

IV. DISCUSSION

A. Classical Diffusion Theory

According to classical rate theory,[7] the vibrational frequencies, and hence the jump frequencies, of two isotopes of the same element at the same temperature are inversely proportional to the square root of their masses. In the absence of correlation effects, the ratio of the diffusion coefficients for the two isotopes is equal to the ratio of their jump frequencies and hence to the reciprocal of the square root of the mass ratio. If the correlation factor is not equal to 1, i.e., the jump probability depends on the direction of the previous jump, the effective jump frequency is reduced.[7] In this case, the ratio of the diffusion coefficients is given by

$$D_i/D_j - 1 = f_i\,[(M_j/M_i)^{1/2} - 1]\,, \tag{10}$$

where D_i and D_j are the diffusion coefficients of isotopes of mass M_i and M_j, respectively, and f_i is the correlation factor for isotope i. [In general, $f_i \approx f_j$, so Eq. (10) is usually written without the subscript on f.] In reality, the masses used in Eq. (10) should not be the actual masses of the diffusing species, but rather should be "reduced masses."[10] The use of such a reduced mass is required by the effect of the coupling of the vibrations of the diffusing atom with those of the surrounding lattice atoms. In this case,

$$D_i/D_j - 1 = f\Delta k\,[(M_j/M_i)^{1/2} - 1]\,, \tag{11}$$

where Δk is a constant $\leq$ unity and defined as the "fraction of the translational kinetic energy which is possessed by the solute atom as it crosses the saddle point."[10] Since both f and Δk are always positive and always $\leq$ unity, the product $f\,\Delta k$ is always $\leq$ unity.

If Eq. (11) is applied to the special case of the diffusion of He^3 and He^4 in vitreous silica, the prediction that $D(He^3)/D(He^4) \leq 1.155$ at all temperatures is made. However, the present study yields a value greater than 1.155 when extrapolated to infinite temperature. In addition, the activation energy for the diffusion of He^3 is approximately 130 cal/g-at. greater than that for the diffusion of He^4, whereas classical diffusion theory assumes that the activation energies for the diffusion of the two isotopes are identical. Thus classical diffusion theory cannot adequately explain these results. It appears that adequate explanation is found in the quantum corrections proposed by LeClaire.[8]

TABLE IV. Expected values of $Q(He^3) - Q(He^4)$ and $D_0(He^3)/D_0(He^4)$ associated with experimentally determined values of $D(He^3)/D(He^4)$.

Temperature (°C)	$D(He^3)/D(He^4)$	$Q(He^3) - Q(He^4)$ (cal/g-at.)	$D_0(He^3)/D_0(He^4)$
286	1.070	163	1.240
306	1.079	151	1.231
329	1.080	155	1.230
348	1.081	158	1.229
368	1.089	146	1.221
374	1.086	154	1.224
397	1.093	143	1.217
415	1.097	137	1.213
424	1.087	163	1.223
438	1.094	149	1.216
456	1.106	123	1.204
474	1.099	144	1.211
498	1.107	127	1.203
513	1.104	138	1.206
524	1.107	132	1.203
546	1.120	99	1.190
565	1.116	112	1.194
582	1.120	103	1.190
595	1.116	116	1.194
602	1.120	105	1.190

B. Quantum Corrections to Classical Diffusion Theory

A thorough discussion of quantum effects in the diffusion of low-atomic-mass isotopes is found in the work of LeClaire.[8] Although classical diffusion theory predicts an isotope effect only on the pre-exponential factor, LeClaire's results require that both the pre-exponential factor and the activation energy for diffusion be mass dependent. LeClaire's analysis results in the following expressions for the isotopic effect on the activation energy and pre-exponential factor:

$$\frac{D_i}{D_j} = \left(\frac{M_j}{M_i}\right)^{1/2}\left(1 - \frac{Q_i - Q_j}{2RT}\right)\,, \tag{12}$$

where Q_i and Q_j are the activation energies for isotopes of mass M_i and M_j, respectively, and

$$\frac{D_i}{D_j} = \left(\frac{M_j}{M_i}\right)^{1/2}\left[2 - \left(\frac{M_i}{M_j}\right)^{1/2}\frac{D_{0i}}{D_{0j}}\right]\,, \tag{13}$$

where D_{0i} and D_{0j} are the pre-exponential factors for isotopes of mass M_i and M_j, respectively. These equations allow a determination of the values of $Q_i - Q_j$ and D_{0i}/D_{0j} which might be expected to be associated with the experimentally determined val-

TABLE V. Comparison between theoretical and experimental values of the isotope effect on activation energy and pre-exponential factor.

	Experimental results		Theoretical results	
Material	$Q(He^3) - Q(He^4)$ (cal/g-at.)	$D_0(He^3)/D_0(He^4)$	$Q_3 - Q_4$ (cal/g-at.)	D_{03}/D_{04}
Spectrosil	137 ± 6	1.210 ± 0.003	136 ± 19	1.211 ± 0.015
Infrasil	121 ± 12	1.198 ± 0.004	128 ± 19	1.205 ± 0.014

ues of D_i/D_j (see Table IV).

If one now considers the case of He^3 and He^4 diffusion, Eq. (12) can be solved for $Q_3 - Q_4$ (the subscripts $i = 3$ for He^3 and $j = 4$ for He^4 are used) to yield

$$Q_3 - Q_4 = 2RT\left(1 - \frac{D_3/D_4}{1.155}\right). \tag{14}$$

Thus, unless $D_3/D_4 = 1.155$ at all temperatures (which has been shown experimentally not to be the case), there must be a difference in the activation energy for the diffusion of these two helium isotopes. In addition, unless the variations in D_3/D_4 with temperature exactly offset the $2RT$ term, the value of $Q_3 - Q_4$ will be temperature dependent and an Arrhenius plot of $\ln (D_3/D_4)$ vs $1/T$ will be curved. Similarly, Eq. (13) can be solved for D_{03}/D_{04} to yield

$$D_{03}/D_{04} = 2(1.155) - D_3/D_4 , \tag{15}$$

which shows that D_{03}/D_{04} also varies as a function of D_3/D_4 (which, of course, it must if $Q_3 - Q_4$ varies). Unfortunately, the scatter in the data of the present study, coupled with the small variations expected in $Q_3 - Q_4$ and D_{03}/D_{04}, prevents such a detailed analysis of the results. However, the data have been analyzed by a linear least-mean-squares technique, which effectively averages the slope and intercept values. Thus, the experimental values for $Q(He^3) - Q(He^4)$ and $D_0(He^3)/D_0(He^4)$ as shown in Eqs. (4) and (5) can be compared with the mean values of $Q_3 - Q_4$ and D_{03}/D_{04} as calculated from Eqs. (14) and (15), respectively. These values are given in Table V. In view of the various approximations made and the accuracy of the experimental data, there appears to be excellent agreement between the theoretical and experimental values. These results strongly support the validity of the quantum corrections proposed by LeClaire.

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Calculation of the Optical Properties of Amorphous SiO_x Materials

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The electronic structure and optical behavior of materials of the amorphous series SiO_x with $0 \leq x \leq 2$ are calculated using a quantum chemical cluster approach. These materials are both compositionally and structurally disordered, and exhibit energy gaps ranging from ~1 eV for Si to ~ 9 eV for SiO_2. Each composition, i.e., value of x, is represented here by a number of topologically distinct clusters each containing 8 silicon atoms and $8x$ oxygen atoms. Each silicon is tetrahedrally coordinated with y oxygens and $4-y$ silicons with $0 \leq y \leq 4$ and $\langle y\rangle = 2x$. We saturate peripheral bonds by a generalization of the periodic boundary conditions appropriate for a regular array. A simple molecular-orbital scheme, the extended Hückel theory, is applied to obtain electronic energy levels for each cluster. Using the indirect constant-matrix-element approximation and taking a weighted average over the various configurations, we obtain the imaginary part of the dielectric constant for a given composition x. The calculated results are in rather good semiquantitative agreement with the experimental results of Philipp, including the variation of the energy gap with composition and the general shape of the ϵ_2-vs-frequency curves.

I. INTRODUCTION

Silicon and oxygen may be combined to form amorphous materials of composition SiO_x with x varying from 0 to 2. The optical properties of amorphous Si,[1] SiO,[2] $SiO_{1.5}$,[2] and SiO_2[3] have been studied by many investigators. Recently, Philipp[4] reported systematic measurements of the absorption coefficient and reflectance of SiO, $SiO_{x\sim1.5}$, and SiO_2. These materials exhibit a wide spectrum of