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Numerical Determination of Inert Gas Permeability Parameters of High- Temperature Gas-Cooled Reactor (HTGR) Fuel-Particles

J. S. Tolliver

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UNION CARBIDE CORPORATION
FOR THE UNITED STATES
DEPARTMENT OF ENERGY

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Computer Sciences Division

**NUMERICAL DETERMINATION OF INERT GAS PERMEABILITY PARAMETERS
OF HIGH-TEMPERATURE GAS-COOLED REACTOR (HTGR) FUEL-PARTICLES**

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for the
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ABSTRACT

The high temperature diffusion of inert gases through an outer layer of dense carbon into the ThO_2 core of biso-coated High-Temperature Gas-Cooled Reactor (HTGR) fuel-particles is studied numerically. A mathematical model of diffusion through a dense spherical shell into a spherical core is used to numerically calculate the theoretical gas content of the core. This theoretical calculation, in tandem with an optimizing computer code and experimental data, is used to determine the diffusion coefficient of the shell and the porosities of the shell and inner core. The activation energy is also determined for use in an Arrhenius relationship between the diffusion coefficients and absolute temperature.

I. INTRODUCTION

Biso-coated fuel-particles, consisting of an inner core of ThO_2 fuel, a buffer layer of porous carbon, and an outer layer of dense carbon, are being developed at Oak Ridge National Laboratory (ORNL) for use in High-Temperature Gas-Cooled Reactors (HTGRs). The dense outer coating serves as a pressure vessel to retain gaseous fission products and to provide mechanical strength. The high-temperature inert gas permeability of the fuel-particles is of interest and has been studied experimentally by C. S. Morgan et al. of the Metal and Ceramics Division of ORNL and G. L. Powell of the Oak Ridge Y-12 Plant Development Division.²⁻⁵ The mathematical aspects of the problem have been reported by Reeves and Tolliver.⁶ A computer code which calculates the theoretical gas content of the inner core is combined with a data-fitting program to obtain the best theoretical fit to experimental measurements of the gas content of HTGR fuel-particles. The present paper provides a brief discussion of the computer program and the results obtained from it.

II. THEORETICAL RESULTS AND DATA-FITTING PROCEDURE

The theoretical expression obtained by Reeves and Tolliver⁶ for the gas content of the spherical core of a dense spherical shell is given below. Here, and in the equations to follow, a and b respectively denote the inner and outer radii of the spherical shell, s and n respectively denote the porosities of the inner core and outer shell, c_0 is the constant concentration of gas surrounding the shell, and D is the diffusion coefficient of the shell. Then the gas content or uptake U as a function of time is given by

$$U(t) = \gamma \bar{U}(t) \quad (1)$$

where

$$\gamma = \frac{4}{3} \pi a^2 b c_0 s \quad (2a)$$

$$\bar{U}(t) = \frac{a}{b} - \sum_{j=1}^{\infty} g(\alpha_j, 0) e^{-D \alpha_j^2 t} \quad (2b)$$

$$g(\alpha, 0) = \frac{2a}{\alpha a(2q+1) \sin \alpha l + (\alpha^2 q a l - b) \cos \alpha l} \quad (2c)$$

$$l = b - a \quad (2d)$$

$$q = 8a/3n \quad (2e)$$

and the α 's are the positive solutions to

$$\tan \alpha l = \frac{\alpha a}{\alpha^2 q a - 1} \quad (2f)$$

For constant geometry the α 's depend only on the ratio q so the uptake U can be uniquely specified by the three parameters D , γ , and q

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plus, of course, the radii a and b and the outside gas concentration c_0 . The porosities n and s are easily obtained from the parameters γ and q as can be seen from the definitions in Eqs. (2a) and (2e).

A computer program has been developed to calculate the theoretical uptake as a function of time from Eqs. (1) and (2) when given values for the three parameters D , γ , and q and the geometric constants a and b along with the outside gas concentration c_0 . In practice, the outside pressure P is more easily known and maintained than the concentration so the relation

$$c_0 = KP/T \quad (3a)$$

is used to calculate c_0 in moles/m³ from the pressure P measured in kilopascals and the absolute temperature T . The conversion factor is

$$K = 1.203 \times 10^2 \text{ mole-}^\circ\text{Kelvin/m}^3/\text{kilopascal} \quad (3b)$$

The uptake calculation has been incorporated into an optimization program which varies the parameters D , γ , and q to minimize the chi-squared function for the uptake. Chi-squared is the function

$$\chi^2 = \sum_{n=1}^N \left[\frac{U_n - \hat{U}_n}{\delta_n} \right]^2, \quad (4)$$

where U_n , $\hat{U}_n$, and δ_n are, respectively, the theoretical uptake, experimentally measured uptake, and the experimental error for each of N data points. The program returns the values of the parameters D , γ , and q which yield the minimum chi-squared. From γ and q , the porosities n and s are determined.

III. RESULTS

Experimental measurements by Powell² of the helium content of the inner core of HTG² fuel-particle batch OR2261HT have been used as input to the data-fitting procedure. Figure 1a shows the experimental data and the theoretical fit along with experimental error bars for the OR2261HT particles annealed in helium at 850° C at a pressure of 98 KPa. Evidently, the theoretical fit is very good, falling well within the error bars at all times. Figure 1b shows the behavior of the same data at small times. Figure 2 shows the results for a helium anneal at the same pressure but at a temperature of only 450° C. The diffusion here is much slower; the uptake did not reach its asymptotic value even after more than 40 days of diffusion. The theoretical fit for this case is not as good as for the 850° case for at least two reasons. First, the physical model used to characterize the diffusion may not adequately explain all of the details of the diffusion process at high and low extremes of temperature where the diffusion is either very fast or very slow. More likely, however, the problems involved with maintaining such an experiment for more than 40 days and the problems of accurately measuring the small quantities of gas present at small times contribute to less than ideal data. Since our model differs from a first-order approximation primarily at small times (see Ref. 6), it is evident that the small time regime is of critical importance to the determination of the theoretical fit. Table I shows the results, that is, the diffusivities D and the porosities n and σ , for a range of temperatures extending from 450° to 1425° C for the OR2261HT fuel-particles. The rather large variability in n is of some concern and will be discussed later.

Fig. 1a. Experimental Uptake Data and Fitted Curve for OR2261HT Fuel-Particles Annealed in Helium at 850°C.

Fig. 1b. Small Time Behavior of OR2261HT Helium Uptake
Data and Theory at 850° C.

Fig. 2. Experimental Data and Fitted Curve for OR2261HT Fuel-Particles Annealed in Helium at 450° C.

Table 1. Fitted Fuel-Particle Parameters D , n , and s for OR2261HT Particles Annealed in Helium at 98 KPa

$T(^{\circ}\text{C})$	$D(\text{m}^2/\text{s})$	$n(\text{m}^3/\text{m}^3)$	$s(\text{m}^3/\text{m}^3)$
450	2.162×10^{-14}	0.00781	0.1942
550	6.086×10^{-14}	0.0329	0.1947
650	2.750×10^{-13}	0.0493	0.1947
750	1.300×10^{-12}	0.0463	0.1946
850	4.055×10^{-12}	0.0471	0.1948
1000	1.922×10^{-11}	0.0455	0.1872
1200	7.181×10^{-10}	0.0611	0.1905
1425	9.412×10^{-10}	0.332	0.1950

If one assumes that the diffusivities obey an Arrhenius relationship

$$D = D_0 e^{-Q/RT} \quad (5)$$

with the activation energy Q and absolute temperature T , then the points on $\ln D$ vs. $1/RT$ should fall on a straight line. A least squares fit made to the data produces the results shown in Fig. 3. Evidently, the Arrhenius relationship is obeyed except at the high and low temperatures. The data at the low end, 450°C , is already suspect as mentioned earlier, and the high temperature data, at 1425°C , consisted of only two data points so the results there are not entirely meaningful. Accordingly, the first and last data points, indicated by $\bullet$ are rejected from the least squares fit and all further considerations. The fit yields a standard deviation of 0.108 and the constants $\ln D_0 = -14.228 \text{ m}^3/\text{sec}$, $\sigma_{\ln D_0} = 0.236$ and $Q = 111.66$ kilojoules/mole- $^{\circ}\text{Kelvin}$, $\sigma_Q = 2.06$.

Other measurements by Powell⁴ include the neon uptake of OR2261HT fuel-particles and the helium uptake of OR2261HT particles that had received an extra 48-hour vacuum anneal at 2000°C to remove process argon

Fig. 3. Plot of $\ln D$ vs. $1/RT$ with Least Squares Line Fit. Showing the Correctness of the Arrhenius Relationship for OR22611T Data.

(see Ref. 4). This data, along with the uptake data for six other fuel-particle batches (OR2263HT, OR2269HT, J596, J598, J605, and J606),^{5,7} is analyzed with the data-fitting procedure described above. The process variables and geometrical parameters of these six batches are reported in Ref. 7. Of the six, the four J prefixed batch numbers were originally labeled by A prefixes. The J numbers, which are used in Ref. 5, were assigned after the original A numbered batches were annealed for 30 minutes at 1800° C. The correspondence between the A numbers and J numbers is as follows: A770 becomes J596; A772 becomes J598; A786 becomes J605; and A787 becomes J606. The results are reproduced in Table 2. The ID column labels the fuel-particles batches as identified in Ref. 5 with OR2261/XX indicating the particles which received the extra 2000° C vacuum anneal and OR2261/Ne indicating the neon permeation data. Hereafter, the OR2261HT helium permeation data discussed above will be referred to as OR2261/He. All data presented here, with the exception of OR2261/Ne, is in regard to the helium permeation of the fuel-particles; all uptake measurements are the anneals at a pressure of 98KPa. An Arrhenius relationship is assumed, and the diffusivities of Table 2 are fitted by a least squares method as described above yielding the values of $\ln D_0$ and Q shown in Table 3. Also shown in Table 3 are the average values of n and s along with the OR2261/He results for convenience.

Figures 4a and 4b show the data and theoretical fits for two of the fuel-particle batches of Ref. 5. Figure 4a is for fuel-particle batch J596 annealed at 550° C and Figure 4b is for J598, also at 550° C. Note that J596 has a much lower diffusion rate than J598; the time axis in Figure 4a is longer, but yet the curve rises much more slowly than in Figure 4b. Also note that the error bars are larger and that the data is

Table 2. Fitted Diffusivities and Porosities for Additional Experimental Measurements of Refs. 4 and 5. (See Text)

ID	T(°C)	D(m ² /s)	τ	ε
OR2261/Ne	850	6.672×10^{-15}	0.0471	0.195
	1000	7.197×10^{-15}	0.0428	0.194
	1200	6.863×10^{-13}	0.0394	0.195
OR2261/XX	550	1.301×10^{-14}	0.0888	0.195
	1000	8.141×10^{-12}	0.0610	0.183
J556	550	1.133×10^{-14}	0.00929	0.104
	750	2.606×10^{-13}	0.0103	0.101
	1000	5.041×10^{-12}	0.00874	0.104
	1200	1.005×10^{-11}	0.0337	0.0970
J598	450	4.040×10^{-14}	0.0256	0.112
	550	3.438×10^{-13}	0.00943	0.115
	750	6.186×10^{-12}	0.0164	0.119
	1000	1.132×10^{-10}	0.0135	0.118
J605	550	1.385×10^{-13}	0.00924	0.107
	750	3.529×10^{-13}	0.241	0.101
	1000	1.375×10^{-10}	0.00380	0.105
J606	550	1.577×10^{-14}	0.0905	0.109
	750	2.613×10^{-13}	0.172	0.103
	1000	1.405×10^{-11}	0.0261	0.103
OR2263	550	4.710×10^{-14}	0.0293	0.196
	750	3.182×10^{-13}	0.181	0.185
	1000	2.653×10^{-11}	0.0156	0.186
OR2269	450	2.381×10^{-13}	0.00452	0.202
	550	1.003×10^{-12}	0.00957	0.202
	750	2.581×10^{-11}	0.00798	0.215
	1000	5.841×10^{-11}	0.0512	0.214

Table 3. Arrhenius Coefficients, Activation Energies, and Averaged Porosities

ID	Standard Deviation of Data	$\ln D_0 (\text{m}^2/\text{s})$	$\sigma_{\ln D_0}$	$Q \left(\frac{\text{Kjoules}}{\text{mol} \cdot ^\circ\text{K}} \right)$	σ_Q	$\bar{\alpha}$	$\sigma_{\bar{\alpha}}$	$\bar{n}$	$\sigma_{\bar{n}}$
OR2261/He	0.108	-14.228	0.236	111.66	2.06	0.1928	0.0032	0.0470	0.0090
OR2261/Ne	0.0658	-13.115	0.347	182.06	3.66	0.1945	0.0005	0.0431	0.0039
OR2261/XX	*	-13.769	*	124.53	*	0.19	0.0082	0.0749	0.020
J596	0.361	-16.109	0.830	109.10	7.37	0.1014	0.0031	0.0155	0.012
J598	0.562	-13.097	1.376	104.78	10.27	0.1161	0.0031	0.0162	0.0069
J605	2.348	-11.517	7.780	129.64	64.16	0.1043	0.0032	0.0846	0.13
J606	0.773	-13.037	2.562	130.19	21.13	0.1047	0.0037	0.0960	0.073
OR2263	1.298	-13.559	4.300	120.43	35.46	0.1891	0.0064	0.0753	0.092
OR2269	0.578	-15.482	1.416	81.422	10.569	0.2082	0.0071	0.0183	0.022

*Data at only two temperatures was available thus yielding a "perfect" least squares fit having zero standard deviation.

Fig. 4a. Experimental Data and Fitted Curve for J596
Fuel-Particles Annealed in Helium at 550° C.

Fig. 4b. Experimental Data and Fitted Curve for J598
Fuel-Particles Annealed in Helium at 550° C.

generally more scattered than the OR2261/He data of Figure 1. Having been measured at more temperature isotherms, and having more data points along each isotherm and smaller error bars for each data point, the OR2261/He data is better than the rest of the data of Refs. 4 and 5, and therefore the OR2261/He fits are the most reliable.

The final step in our fitting procedure is to force the diffusivities D to lie on a line obeying the Arrhenius relationship and to force the inner porosity ϵ , which is related to γ , to be constant for each fuel-particle batch. Thus, using the Arrhenius coefficients and averaged inner porosities of Table 3, each set of data in the ID column of Table 3 is fitted again with the theoretical uptake model allowing only the parameter q to vary. With s known, the outer porosity n is proportional to q so all the variability falls on n . The fitted outer porosities are shown in Table 4. The porosity should be constant among particles of the same batch so these n 's are averaged with the results appearing in Table 5. For convenience Table 5 also reproduces the Arrhenius coefficients and the inner porosities shown in Table 3.

It must be noted that the values given for $\ln D_0$ and Q in Tables 3 and 5 are not the true values but only estimates determined from the linear least squares fit. In the process of linearizing Eq. (5) by taking its logarithm, it is necessary to assume that the statistical errors are multiplicative rather than additive. Then, in applying a linear fit to the linearized equation,

$$\ln D = \ln D_0 - Q/RT \quad ,$$

it is implicitly assumed that $\ln D_0$ and Q are normally distributed random variables with variances $\sigma^2_{\ln D_0}$ and σ^2_Q respectively. Since Q is normally distributed, the best estimate for the activation energy is simply the Q shown in the tables. However, the normal distribution of $\ln D_0$ means

**Table 4. Fitted Outer Porosity n While Holding
all Other Parameters Constant**

ID	T(°C)	n
OR2261/He	450	0.0384
	550	0.0384
	650	0.0406
	750	0.0457
	850	0.0447
	1000	0.0515
	1200	0.0599
	1425	0.0853
OR2261/Ne	850	0.0455
	1000	0.0459
	1200	0.0380
OR2261/XX	550	0.0892
	1000	0.0587
J596	550	0.00864
	750	0.00981
	1000	0.0144
	1200	0.0216
J598	450	0.0176
	550	0.0192
	750	0.0109
	1000	0.0151
J605	550	0.0266
	750	0.00937
	1000	0.0120
J606	550	0.168
	750	0.0509
	1000	0.0424
OR2263	550	0.0571
	750	0.0277
	1000	0.0322
OR2269	450	0.00431
	550	0.00723
	750	0.0162
	1000	0.0337

Table 5. Arrhenius Coefficients, Activation Energies, and Averaged Porosities After the Final Fit

ID	$\ln D_0 (\text{m}^2/\text{s})$	$\sigma_{\ln D_0}$	$Q \left(\frac{\text{Kjoules}}{\text{mol} \cdot ^\circ\text{K}} \right)$	σ_Q	$\bar{p}$	σ_p	π	σ_π
OR2261/He	-14.228	0.236	111.66	2.06	0.1928	0.0032	0.0468	0.0078
OR2261/Ne	-13.115	0.347	182.06	3.66	0.1945	0.00005	0.0431	0.0044
OR2261/XX	-13.769	*	124.53	*	0.1889	0.0082	0.0740	0.0216
J536	-16.109	0.830	109.10	7.37	0.1014	0.0031	0.0136	0.0059
J598	-13.097	1.376	104.78	10.27	0.1161	0.0031	0.0157	0.0036
J605	-11.517	7.780	129.64	64.16	0.1043	0.0032	0.0160	0.0093
J606	-13.037	2.562	130.19	21.13	0.1047	0.0037	0.0870	0.0699
OR2263	-13.559	4.300	120.43	35.46	0.1891	0.0064	0.0390	0.0158
OR2269	-15.482	1.416	81.422	10.569	0.2082	0.0072	0.0154	0.0132

*See note at end of Table 3.

that D_0 follows what is called a log normal distribution. From statistical considerations of the log normal distribution, a simple application of the exponential function to the estimate $\ln D_0$ yields an estimate for D_0 which is biased by the factor $\exp\{\sigma^2_{\ln D_0}/2\}$. When the standard error in $\ln D_0$ is large, the biasing factor is quite significant and must be accounted for by the formula

$$\hat{D}_0 = \exp\{\ln D_0\} \times \exp\{-\sigma^2_{\ln D_0}/2\} \quad (6)$$

Here, the symbol " $\hat{}$ " over D_0 is used to indicate "the best estimate of." In like manner the best estimate for the standard error in $\hat{D}_0$ is given by

$$\hat{\sigma}_{\hat{D}_0} = \exp\{\ln D_0\} \times \sqrt{\exp\{\sigma^2_{\ln D_0}\} - 1} \quad (7)$$

When the standard error in $\ln D_0$ is large, the standard error in $\hat{D}_0$ is also large. The standard errors for $\ln D_0$ tabulated in Table 5 are significant for particle batches other than OR2261, and this fact, along with Eqs. (6) and (7), must be noted by the reader.

The large standard errors for $\ln D_0$ and the high variability of n for all except particle batch OR2261 are speculated to arise from two sources. The primary source of error is the real macroscopic particle-to-particle variation in the geometric dimensions of the particles.^{1,2} This variability is relatively small for the OR2261/HT particles and is reflected in the good fits for OR2261/He and OR2261/Ne. The good fit for OR2261/He also results from the relatively large number of isotherms at which data were measured for this particle batch. For the other particle batches the real particle-to-particle variability is greater, the number of isotherms is less, and the data available for each isotherm

are fewer and have larger errors. All these factors combine to result in poorer fits for particle batches other than OR2261. These particle-to-particle deviations arise from nonuniform coating thicknesses for particles in the same batch and a lack of sphericity of the particles. Cracks in the outer coating of the particles would also contribute to large errors in the data since these cracks would allow vastly higher diffusion rates. However, the particles were carefully manufactured in order to prevent cracks, and the fraction of particles with cracked coatings is known to be quite low. The manufacture, geometry, and uniformity of the fuel-particles are discussed in Refs. 1 and 7

IV. CONCLUSION

The theoretical expression derived by Reeves and Tolliver⁶ for the gas content of the spherical core of a dense spherical shell after the diffusion of gas through the shell has been used to make a numerical calculation of the gas content of the core of HTGR biso-coated fuel particles. The numerical calculation has been combined with a data-fitting program and experimental measurements of the gas content of HTGR fuel-particles to determine the values of the physical parameters D , n , and s of the fuel-particles for several different temperatures. The results have been reported and sample plots of the experimental data and fitted curves have been shown. An Arrhenius relationship between the absolute temperature and the diffusivity D has been assumed and the activation energy Q reported for each batch. The values of $\ln D_0$ have been reported along with instructions for obtaining the best statistical estimate for the coefficient D_0 and its standard error from the quantities $\ln D_0$ and $\sigma_{\ln D_0}$. The theoretical fit for OR2261/He is quite good, having standard errors in the activation energy and $\ln D_0$ of less than 2%. However, for fuel-particle batches other than OR2261, the standard errors in $\ln D_0$ are large, leading to wide confidence limits on the value of D_0 . These large standard errors are primarily due to wide confidence limits on the data itself due to the particle-to-particle geometric variability. To a lesser extent these large uncertainties are contributed to by the lack of data since only a few isotherms with only a few points per isotherm were measured for these particle batches. A better fit and more reliable results would be obtained by using an optimizing computer code with the absolute temperature as well as the time as

independent variables in the expression for the gas content of a fuel-particle. By explicitly including the temperature dependence, all of the data for each fuel-particle batch could be used, and the Arrhenius coefficients D_0 and Q , as well as quantities γ and q , could be self-consistently determined for all of the isotherms for each batch. By coupling all of the data for each isotherm, the uncertainties due to fewer isotherms and fewer points for all batches except OR2261/He would be reduced but not eliminated. More data at more isotherms would obviously be desirable. Of course this procedure would not eliminate the major source of errors -- that due to the particle-to-particle geometric variability. These errors can only be reduced by the careful manufacture of uniform particle batches.

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