JOURNAL OF MATERIALS SCIENCE 26 (1991) 3862–3868

Open pore description of mechanical properties of ceramics

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A dependence of Young's modulus of elasticity on open porosity in ceramics is derived from an open-porosity model, which in the literature, is applied to salinity conductivity and fluid permeability in rocks. A random distribution of grain and pore size is assumed. The relation developed, $E(p) = E_0(1 - p)^m$, where E is the modulus of elasticity of the porous ceramic, E_0 is the theoretical elastic modulus, p is the porosity and m is an exponent dependent on the tortuosity of the structure of the ceramic, adequately describes the dependence of the modulus of elasticity on porosity. The model is applied to the experimental data from several ceramics such as alumina, silicon nitride, silicon carbide, uranium oxide, rare-earth oxides, and $YBa_2Cu_3O_{7-\delta}$ superconductor, and the value of m is obtained for each case. We have shown that m has a value of nearly 2 for sintered ceramics, unless sintering aids or hot pressing have been used during fabrication of the ceramic. Such additional procedures approximately double the magnitude of m.

1. Introduction and review of previous work

The dependence of the mechanical properties of ceramics on type, size and distribution of pores is an old subject of investigation. In recent years, it lost its importance due to an overall interest in high-strength, and thus, dense ceramics for both structural and electronics applications. A number of modern applications, however, require knowledge of the effect of porosity on several physical properties of structural ceramics. Gas sensors [1], heat exchangers [2], and particulate filters [3] are such applications. Even the traditional ceramics, such as construction material (bricks, tiles, cement, concrete blocks), are porous and are under scrutiny in radon emanation studies [4], because the radon emanation rate depends on the porosity of such materials. In addition, the ion-exchange capacity of concrete matrix used for the containment of radioactive waste [5], and adsorption/desorption of sulphur compounds in regenerative scrubbers used in desulphurization of coal gas [6], depend directly on the surface area of open-pore channels. At the same time, the mechanical strength of these devices, and hence their durability, will also depend on the shape and size of the pores. As a result, the dependence of various physical parameters on porosity is crucial in the performance of these materials.

Several formulations relating modulus of elasticity and total porosity have been reported in the literature. The semi-empirical formulae of Duckworth [7], Spriggs [8], and McKenzie [9], for example, have been used widely to fit the measured data of Young's modulus, E, of various ceramics. These equations are

$$E(p) = E_0 \exp(-bp) \tag{1}$$

$$E(p) = E_0\{1 + Ap/[1 - (A + 1)p]\} \tag{2}$$

$$E(p) = E_0(1 - f_1 p + f_2 p^2) \tag{3}$$

where, E_0 and p are Young's modulus of the pore free material and volume fraction of porosity, respectively. All other notations are adjustable parameters. At low porosity, these three relations lead to

$$E(p) = E_0(1 - f_1 p) \tag{4}$$

Several investigations have reported agreement of these formulations with experimental results [10]. The empirical nature of these formulations, however, does not allow correlation between the microstructure of the material and its mechanical properties; hence, the constants have no physical significance.

A second set of equations is proposed on the basis of the theory of elasticity of a continuum, in which pores are a second phase. Phani and Niyogi [11] derived an equation for the porosity dependence of the modulus of elasticity by assuming that the physical processes such as stress distribution and elongation are dependent only on total porosity; thus, the modulus of elasticity depends only on the total porosity. The equation proposed by them is

$$E(p) = E_0(1 - ap)^n \tag{5}$$

where a is related to the packing density and is the inverse of the corresponding critical porosity, p_c [10] where the modulus of elasticity vanishes. This definition of a suggests that it is always greater than 1.

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0022-2461/91 $03.00 + .12

However, fittings of curves of experimental data over a wide range of porosity invariably gives $a = 1$ [11, 12]. Thus, Equation 5 does not explain the experimental data accurately. In addition, because the approach taken by Phani and Niyogi [11] does not involve microstructure of the material, such as the random shapes of the pores and their random size distribution, the theory cannot be extended to more general applications in fracture mechanics such as the dependence of fracture strength on grain and pore size.

A similar difficulty arises when one considers pores as geometrically regular shapes embedded in a continuum matrix. The method followed by Ishai and Cohen [13] is representative of this approach. They assume a cubic pore and derive the relation

$$E(p) = E_0(1 - p^{2/3}) \qquad (6)$$

Bert [14] critically reviewed this type of approach and concluded that the equations derived by taking regular shapes for the pores have limited application.

The difficulties encountered in the approaches summarized above reveal a need for a model that will explain the porosity dependence of mechanical and fracture properties of ceramics, and predict microstructure–property relationships by retaining the random structure of ceramics. Such a model should be based on microscopic details of the structure of ceramics, i.e. the irregular shapes and sizes of the pores and their random distribution. Such a model is proposed in this paper. It is a modification of an earlier model proposed by Wong *et al.* [15] to explain charge and mass flow through pores in rocks. In its first application, it is applied to study the porosity dependence of the modulus of elasticity. In subsequent publications, its applications will be extended to the study of the fracture and thermophysical properties of ceramics.

To develop such a model, certain observations are necessary. The agreement of Equation 5 with the experimental data for $a = 1$ suggests that the modulus of elasticity does not vanish unless $p = 1$, or porosity is 100%. This means it is possible to fabricate ceramics with a continuous material network with a very high porosity, e.g. cellular ceramics, in which porosity is as high as 93% [16]. Similar to this continuous material network, it is found that the pores are also connected in continuous channels, even at low porosity. This has been observed in the study of electrolyte conductivity through open-pore channels of ceramics, where conductivity is directly proportional to the square of the porosity of the material (Archie's law [17]). In a related study, fluid permeability through the open-pore channels is found to be proportional to the cube of the porosity (Kozeny Equation [18]). Both of these observations imply that conductivity and permeability are zero when the porosity is zero. Brouers and Ramsamugh [19] confirmed this in the case of alumina ceramics by studying both Archie's law and the Kozeny equation over a wide range of porosity. Drory and Glaeser [20], Yen and Coble [21], and Gupta [22] attempted studies of the kinetics of pore closure, especially in the cylindrical shape, and they show that pore closure results only in the final stage of sintering. This implies that the open pores exist until the final stages of sintering; beyond that the open-pore fraction will depend on the extent of sintering. The data on UO_2 [23], magnesia and zirconia [24] reveal open pores even at a very low porosity. Thus, it is justified to assume that open pores exist over a wide range of porosity in ceramics, unless they are fabricated with special techniques, such as the use of pore formers. In addition, these studies also find that the fraction of the closed-pore volume is very small compared to the open-pore volume, except at very low porosity. The model proposed here is based on these observations. We assume that open pores exist in ceramics even at low porosity. We also assume that pore distribution, size, and shape are random. The simulation of the model based on these observations is presented and porosity dependence of the modulus of elasticity is derived. Subsequently, the model is applied to the experimental data and conclusions are drawn on its applicability.

2. Open-pore model of ceramic structures

As stated earlier, this model is based on an earlier one proposed by Wong *et al.* [15] to explain the electrolyte conductivity and fluid permeability through pore channels in rocks. In rocks, it is found that both electrolyte conductivity and fluid permeability exist at low porosity because open-pore channels are present at that porosity. The origin of the open pores responsible for the flow of fluids is attributed to the mode of formation of rocks at high pressure [15]. When rocks are formed, the packing of particles (grains) starts with a high initial porosity. In the initial and intermediate stages of consolidation, the cross section of a pore channel is reduced by the external pressure. Wong *et al.* claim that the probability of a channel becoming completely blocked by the pressure is small, because as the consolidation of the wall of the channel under pressure reduces local stress, it strengthens the wall, and resists further deformation. These arguments may very well apply to ceramics too, because the process of development of a ceramic material is very similar to that of rock formation. Therefore, the model proposed by Wong *et al.* [15] is adopted in this work.

We assume that a ceramic structure is a three-dimensional, intertwined, continuous network of material chains and open-pore channels. The chains are formed by connecting grains, each grain representing a nearly crystalline structure. Closed pores, if present, are few in proportion to the total porosity; hence, their effects are negligible. We also assume a random distribution of the grains and the pore channels and retain this random structure, unlike the treatments presented in the literature [11, 25]. To evaluate the effect of porosity on the modulus of elasticity, we propose to simulate a structure with random cross sections of material chains and pore channels. To achieve this, we begin with a cubic structure of packed cylinders of negligible initial porosity. The cross-sectional radius of each cylinder is equal to the maximum grain size in sintered ceramics.

In a given direction, the cylinders are connected in series to form a material chain. A one-dimensional analogue of such a model is illustrated in Fig. 1a. We also assume in the first approximation that each cylinder (grain) is crystalline, so that a basic lattice-dynamical model of crystalline structure can be used to calculate mechanical and thermal properties. This means that each cylinder consists of a regular array of atoms connected by elastic springs representing bonds between the atoms. The number of springs parallel to the length of an individual cylinder is proportional to the area of the cylinder cross-section. Thus, when a cylinder is stretched, each spring is elongated and the net extension of the cylinder is a collective effect of the extension of these parallel springs.

To simulate the random distribution of the material with non-uniform cross-section of material chains, cylinders are chosen at random and their cross-sections are reduced randomly. Repeated random reductions will simulate a material chain of random cross-section. At the same time, it will also simulate connected pore structures between two material chains. A one dimensional illustration of three successive reductions is presented in Fig. 1b–d. As seen in Fig. 1d, the simulation also leads to coalescence of the channels in the transverse direction. In three dimensions, such a model provides material chains connected intricately in all directions. In fact, the material chain model is an exact replica of the open-pore channel of Wong *et al.* [15].

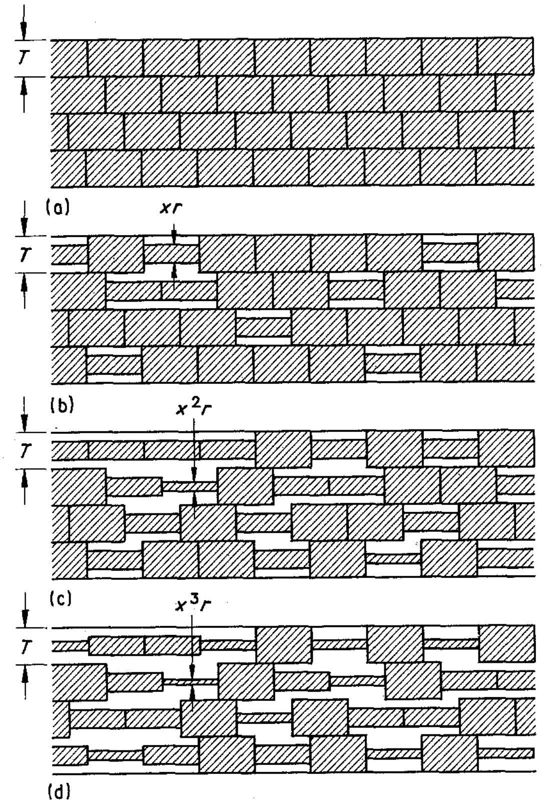

Figure 1 One dimensional simulation of material channels. (a) Uncontracted structures, (b) radii of the cylinders are reduced by a factor of x, (c) the reduction is repeated choosing cylinders again at random. Note that same cylinder may be chosen and in that case the radius is reduced by a factor x^2, and (d) third simulation presents a more realistic picture of ceramic structures.

For simplicity, let us assume that there are N cylinders, all of them initially identical, as in Fig. 1a. An ith cylinder has a radius r_i and consists of n_i parallel springs with a net spring constant of the cylinder being k_i, which is the sum of spring constants of individual springs. We reduce the radius by a fixed factor x, where $x < 1$. Thus, r_i changes to xr_i; hence n_i reduces to $x^2 n_i$. This yields

$$k_i \propto x^2 r_i^2 \tag{10}$$

This shrinking procedure can be repeated to achieve desired porosity by reducing the radius of the cylinders, in turn, reducing the number of springs (n_i) and consequently k_i. Because the modulus of elasticity E_i of the ith spring is proportional to k_i, this procedure will also reduce the modulus proportionately. Thus, with n reductions, the modulus of elasticity, E_i, is reduced to $x^{2n}E_i$.

The probability $\Phi(n)$ of any particular cylinder shrinking n times is given by the binomial distribution

$$\Phi(n) = [M!/(M-n)!n!](1/N)^n[(N-1)/N]^{M-n} \tag{11}$$

where M is the total number of reductions performed. The average spring constant of the cylinder after M reductions will be

$$\begin{aligned} k_{\mathrm{av}} &= \langle k_i \rangle \\ &= k_0 \sum_{n=0}^{M} x^{2n}\Phi(n) \\ &= k_0[(N + x^2 - 1)/N]^M \end{aligned} \tag{12}$$

where $\langle \ldots \rangle$ denotes the average, and k_0 is the spring constant of the material without pores.

Although the problem requires computer simulations in three dimensions, it has an exact analytical solution in one dimension, which also gives us some physical insight into the problem. Therefore, we obtain an analytical solution in one dimension first. Because the cylinders are in series, the true spring constant, k, is different from its average and is given by

$$\begin{aligned} k^{-1} &= N\langle k_i^{-1} \rangle \\ &= N k_0^{-1} \sum_{n=0}^{M} x^{-2n}\Phi(n) \\ &= N k_0^{-1}[(N + x^{-2} - 1)/N]^M \end{aligned} \tag{13}$$

From Equations 12 and 13, one can write

$$\frac{\ln(k^{-1}/N k_0^{-1})}{\ln(k_{\mathrm{av}}/k_0)} = \frac{\ln[1 + (x^{-2} - 1)/N]}{\ln[1 + (x^2 - 1)/N]}$$

Because N is a large number, we have

$$\begin{aligned} \frac{\ln[1 + (x^{-2} - 1)/N]}{\ln[1 + (x^2 - 1)/N]} &= (x^{-2} - 1)/(x^2 - 1) \\ &\approx -(x^{-2}) = -m \end{aligned} \tag{14}$$

Thus

$$\ln(k^{-1}/N k_0^{-1}) = \ln(k_{\mathrm{av}}/k_0)^{-m} \tag{15a}$$

or

$$(k^{-1}/N k_0^{-1}) = (k_{\mathrm{av}}/k_0)^{-m} \tag{15b}$$

or

$$k = (1/N)k_0(k_{\mathrm{av}}/k_0)^m \tag{15c}$$

Because k_0 is a material property independent of porosity, one can write

$$k \propto (k_{\text{av}})^m \tag{16}$$

We also note that the average volume $\langle V \rangle$ of a cylinder of length, l, is

$$\begin{aligned} \langle V \rangle &= \pi l \langle r_i^2 \rangle \\ &= \pi l r_0^2 \sum_{n=0} x^{2n} \Phi(n) \\ &= V_0[(N + x^2 - 1)/N]^M \end{aligned} \tag{17}$$

where V_0 is the initial volume of a cylinder or the maximum volume of a grain in the final ceramic structure. Also

$$\begin{aligned} N\langle V \rangle &= \text{volume of the material} \\ &= NV_0(1 - p) \end{aligned} \tag{18}$$

From Equations 17 and 18 we have

$$[(N + x^2 - 1)/N]^M \propto (1 - p) \tag{19}$$

Therefore, from Equation 13, we have

$$k_{\text{av}} \propto (1 - p) \tag{20}$$

Combining Equations 16 and 20, we have

$$k \propto (1 - p)^m \tag{21}$$

Because the modulus of elasticity is proportional to k, we have

$$E(p) = E_0(1 - p)^m \tag{22}$$

where E_0 is the modulus of elasticity of the pore-free material. Equation 22 gives the porosity dependence of the modulus of elasticity for ceramics in one dimension. This model can be generalized to higher dimensions only numerically, and, in our case, may simply be adopted from Wong *et al.* [15]. The value of m in a three-dimensional model, however, differs from that in Equation 14 and is derived exactly in the thermodynamic limit in the Appendix. The only difference between the model of Wong *et al.* [15] and the present model is that they applied it to pore channels and our model is applied to material chains. Thus, we obtain our porosity dependence by replacing p by $(1 - p)$ in their model, as is evident in the one-dimensional description given above. They tabulated the sample results for various values of x in their article; hence, we have not reproduced them here. Extensive numerical calculations lead to the following inferences.

1. x is a measure of the tortuosity (skewness) in the material. The smaller x leads to wider distribution of the cross-sections in the chains; thus, the disorder in the size distribution is generated by a proper choice of x.
2. As x decreases, m increases. This implies that skewness, as a measure of the disorder, is higher for higher values of m.
3. The properties of the entire structure are simply related to the statistical distribution of the individual cylinders, regardless of how they are connected in detail.

In the next section, we shall apply this model to various ceramics and use the inferences presented above to obtain an insight into the effect of fabrication method on microstructure.

3. Applications of the model

Table I is a summary of the exponents m obtained from various fittings of Equation 22 with experimental results by linear regression analysis. The exponents from these fittings are quoted in the table with appropriate references. For fittings done for the first time in the present work, the results are also shown in Figs 2–4. It was observed that the value of the exponent is approximately 2 for materials fabricated without sintering aids, or hot pressed. In other cases, much higher values are observed.

The exponent m for α alumina [26], fabricated without any sintering aid, with data in the high-porosity region (0.22–0.43) is 2.14. Rare-earth oxide ceramics of yttrium, holmium, dysprosium and erbium [27–29] have been studied at very low porosity. Again no sintering aids were added during fabrication. In Fig. 2, the values of the modulus have been plotted as a function of porosity. The least square straight line fits of Equation 22 to the data results in slopes of $m \approx 2$. It has been shown [29] that there was very little difference between open and total porosity, implying a negligibly small fraction of closed pores.

TABLE I Exponents m obtained by fitting Equation 22 with various experimental results by linear regression analysis

Material	Porosity range	m	References		Comments
			Data	Fitting	
α-alumina	0.22–0.43	2.14	[26]	11	consistent with conductivity data
β-alumina	0.02–0.41	4.12	[34]	11	Sodium aluminate as a glassy phase
Y_2O_3	0.03–0.19	2.02	[29]	this work	pure oxide
Er_2O_3	0.11–0.3	2.14	[29]	this work	pure oxide
Dy_2O_3	0.07–0.38	2.47	[29]	this work	pure oxide
Ho_2O_3	0.01–0.18	2.4	[29]	this work	pure oxide
SiC	0.02–0.16	3.8	[32]	this work	hot pressed
Si_3N_4	0.2–0.2	2.58	[40–43]	11	reaction bonded
Si_3N_4 + MgO	0.08–0.45	5.48	[33]	this work	Hot pressed, sintering aid used
Si_3N_4 + CeO_2	0.02–0.38	4.2	[33]	this work	sintering aid used
Si_3N_4 + Y_2O_3 + SiO_2	0–0.12	3.81	[33]		sintering aid used
UO_2	0.02–0.06	2.27	[31]	31	pure oxide
$YBa_2Cu_3O_{7-\delta}$	0.07–0.301	2.05	[30]	this work	oxide mixture

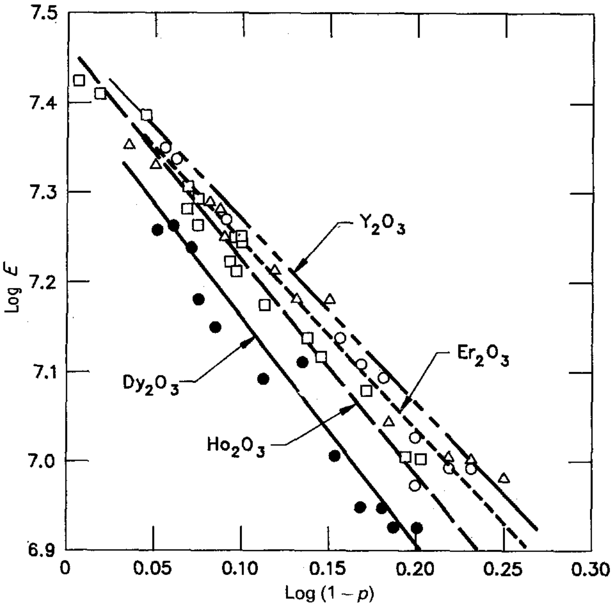

Figure 2 Comparison of Young's moduli of rare-earth oxide ceramics with Equation 21.

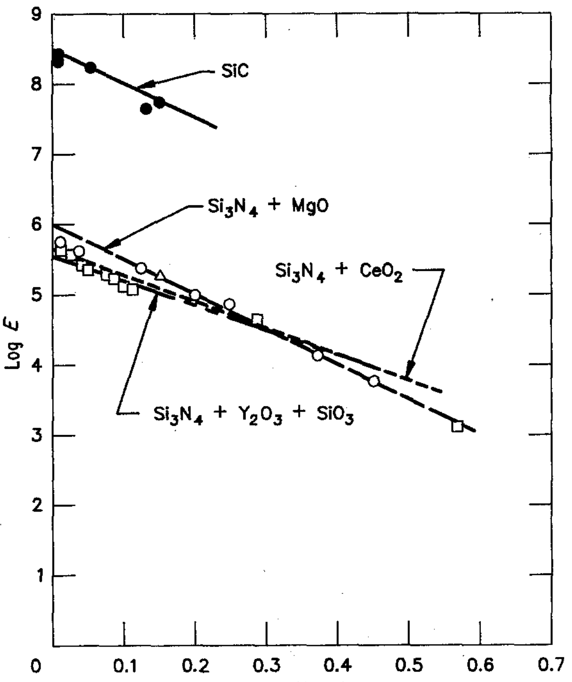

Figure 3 Porosity dependence of elastic moduli of hot-pressed silicon carbide and silicon nitride specimens.

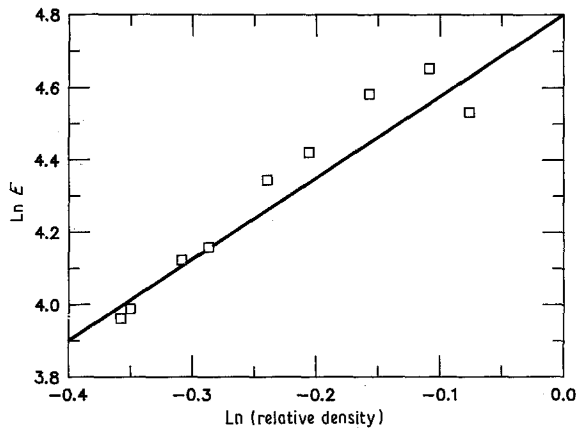

Figure 4 Logarithms of relative per cent density and Young's modulus for the superconductor. Theoretical density = 6.3 g cm^{-3} and E_0 = 110.8 GPa.

This justifies our use of an open-porosity model in this case, even at low porosity.

The case is similar when one compares values from Equation 22 with the data of Singh *et al.* [30] on the superconductor $YBa_2Cu_3O_{7-\delta}$. Fig. 4 shows the agreement of Equation 22 with the experimental results. The exponent is 2.43. This shows that the behaviour of the superconductor is similar to the other ceramics presented above in its porosity dependence of the modulus of elasticity. The same is the case with the data on UO_2 measured by Forlano *et al.* [31]. They measured density using Archimedes principle, which gives only open porosity. The corresponding exponent is found to be 2.27 (again close to 2), consistent with the behaviour of other materials cited above.

The situation is quite different when the material is hot-pressed. In this case, external pressure leads to accelerated densification, implying a lower value of x in our model and consequently, a higher value of m. Silicon carbide [32] is a good example of this. The value of m in the case of hot-pressed silicon carbide is found to be 3.38, considerably higher than the exponent found in the materials discussed above. Similarly during fabrication, the use of sintering aids leads to accelerated densification. This is evident from the data on silicon nitride doped with MgO, CeO_2 and Y_2O_3 + SiO_2 [33]. Here, the exponents are 5.48, 4.2 and 3.81, respectively. These values of m are much higher than the 2.58 obtained in the case of undoped, reaction-bonded silicon nitride [34–37]. The agreement of Equation 22 with the data on these materials with higher values of m is shown in Fig. 3.

Finally, we consider the case of β alumina. Evans *et al.* [38], who measured its modulus of elasticity, and Phani and Niyogi [11], who fitted the data using empirical Equation 6, obtained a value of 4.12 for m, which is considerably higher than that obtained for alumina. The likely reason for this high value of m is a glassy phase formed during the sintering process. Evans *et al.* observed that during sintering of β alumina, sodium aluminate was formed at the intermediate stage, and β alumina was a result of reaction between α alumina and this phase. It is possible that sodium aluminate also acted as a glassy phase and helped densification; this is reflected in a larger value of m, as discussed. Thus, in this respect, β alumina behaves like doped silicon nitride and one obtains a higher value of m.

There is a similarity between Equation 22 for the modulus of elasticity and Archie's law of electrolyte conductivity. Equation 22 implies that the modulus of elasticity in the material chains is a power law of the volume fraction of the material in a porous material, whereas Wong *et al.* [15] show that the conductivity, σ, in the pore channels is a power law of the volume fraction of pores given by the relation

$$\sigma = \sigma_0 p^s \quad (23)$$

where σ_0 is the conductivity of the electrolyte. In most of the materials, s is found to be equal to 2; hence, Equation 23 is a mathematical presentation of Archie's law. Brouers and Ramsamugh [19] tested this law in α alumina and found that $s = 1.91$, again nearly equal to 2. The fact that the present model applied to material chains and that used by Wong *et al.* [15] for pore channels are identical and yield approximately the same exponent, implies that the overall microstructure and tortuosity of material chains and the pore channels are similar in ceramics fabricated without sintering aids. It would be interesting to see how Archie's law (Equation 23) behaves in materials such as β alumina, where the glassy phase influences the tortuosity.

4. Discussion and conclusions

The observations in the previous section allow us to draw some general conclusions.

Porosity dependence of the modulus of elasticity of ceramics is a simple power law given by $E(p) = E_0(1-p)^m$. For ceramics fabricated without hot-pressing or sintering aids, m is approximately 2, which is consistent with Archie's law of electrolyte conductivity through porous media. As the porosity is reduced by sintering aids or external pressure, its value increases, implying an accelerated densification.

The behaviour reflected in these conclusions is not limited to ceramics, but is applicable to porous materials in general. Phani and Mukerjee [39] showed that $m = 1.85$ in the case of porous epoxy resin, whereas a similar study by Hasselman and Fulrath [40] on glass with spherical pores yielded an exponent of 2. Several investigators have studied cellular solids. The most comprehensive discussion is given by Ashby [16], who derived an exact relation for the modulus of elasticity as a power law of the density of geometrically regular cellular solids such as natural polymers, ceramic, and glass foams; he determined the exponent to be 2. This was further verified by Dam [41] and Hagiwara [42] for cellular ceramics. Theoretically, Spinner *et al.* [43] showed that, at zero porosity

$$-\mathrm{d}/\mathrm{d}p(E/E_0)|_{p\to 0} = [(1+\mu)(39-45\mu)/6 \times (7-5\mu) + 1] \qquad (24)$$

where μ is Poisson's ratio. From Equation 22, we obtain

$$m = -\mathrm{d}/\mathrm{d}p(E/E_0)|_{p\to 0} \qquad (25)$$

For a typical Poisson's ratio, μ, between 0.2 and 0.3, Equation 24 gives $m \approx 2$, confirming the findings of this work. In spite of this general behaviour of an exponent being approximately equal to 2, porous ceramics can behave differently, with a higher value of m, when a glassy phase is present or external pressure is applied during fabrication. In that respect, behaviour of porous ceramics is very similar to that of rocks, where the exponent is generally 2 but may differ if skewness varies due to their formation under the pressure of overburden [25].

Appendix: Exact value of the exponent *m* in the thermodynamic limit in three dimensions

The shrinkage probability $\Phi(n)$ given by Equation 11 becomes a Gaussian distribution in the thermodynamic limit, i.e. as M, N, and $M/N \to \infty$ [15]. This distribution is centred at $\beta = (M/N)$ with a half-width of $\partial\beta = \beta^{\frac{1}{2}}$. β here is the most probable value of n. The most probable (mp) value of the force constant k is given by

$$k_{\mathrm{mp}} = x^{2\beta} k_0 \qquad (\mathrm{A1})$$

This k_{mp} clearly differs from the average value k_{av} for finite M and N. However, in the thermodynamic limit, β becomes negligible and one may write

$$k_{\mathrm{mp}}/k_0 \propto k/k_0 \qquad (\mathrm{A2})$$

Using Equations A1, A2 and 12, we obtain

$$\begin{aligned} \ln(k_{\mathrm{mp}}/k_0)/\ln(k_{\mathrm{av}}/k_0) &= \ln(x^{2M/N}) \\ &\quad \div \ln[1 + (x^2-1)/N]^M \\ &\approx (\ln x^2)/(x^2-1) \\ &= \ln x^2/(x^2-1) \\ &= m \qquad (\mathrm{A3}) \end{aligned}$$

Therefore, $k_{\mathrm{mp}} \propto k_{\mathrm{av}}^m$; hence $k \propto k_{\mathrm{av}}^m$
or

$$k = k_0(1-p)^m \qquad (\mathrm{A4})$$

and

$$m = \ln x^2/(x^2-1) \qquad (\mathrm{A5})$$

Acknowledgement

This work was supported by the US Department of Energy, Fossil Energy Program, Morgantown Energy Technology Center, under Westinghouse Electric Corporation ACK 85208, "Materials Qualification Technology for Cross-Flow Filter".

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Received 11 April
and accepted 4 September 1990