Elastic properties of porous oxide ceramics prepared using starch as a pore-forming agent

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Abstract

The elastic properties, in particular the tensile modulus (Young's modulus) and Poisson ratio, of porous alumina, zirconia, and alumina–zirconia composite ceramics are studied using the resonance frequency method and the results compared with theoretical predictions. Starch is used as a pore-forming agent, so that the resulting microstructure is essentially of the matrix-inclusion type (with large bulk pores, connected by small throats when a percolation threshold is exceeded). It is found that for this type of microstructure the porosity dependence of the Young's modulus is significantly below the upper Hashin–Shtrikman bound and the power-law prediction; it corresponds well, however, to a recently proposed exponential relation and to an empirical volume-weighted average of the upper and lower Hashin–Shtrikman bounds. Results for all three types of ceramics indicate that – in the porosity range considered, i.e. up to approximately 50% – the Poisson ratio depends only slightly on porosity.
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1. Introduction

Porous ceramics are widely used for a variety of applications, ranging from filters, membrane supports and catalyst carriers to thermal insulation, lightweight structural components, biomedical implants and bone tissue engineering scaffolds.[1–4] The introduction of porosity into materials reduces the weight of components and may thus significantly improve their specific properties (i.e. the properties per unit mass).[5] In particular, porous ceramics generally exhibit lower elastic moduli and lower thermal conductivity than their fully dense counterparts (i.e. the solid phase itself), since the modulus or conductivity of the pore phase can in many cases be neglected in comparison to the corresponding solid phase property. Thus, porous ceramics are more compliant (i.e. less rigid) and more efficient insulators (i.e. their thermal resistivity is enhanced). Powerful relations have been recently proposed which can predict the porosity dependence of some of these properties, in particular the Young's modulus, for common microstructures more

or less quantitatively.[6–9] On the other hand, for some properties – among them the Poisson ratio – simple predictive relations are not available up to now, and even to the question whether the effective Poisson ratio decreases, increases or remains constant with increasing porosity, there is no universally acknowledged answer so far. These difficulties arise because the porosity (volume fraction of pores) is but one of the many microstructural descriptors and because higher order microstructural information[10] is usually not accessible without an immense effort (e.g. using image analysis or tomographical methods), far exceeding that of directly measuring the property in question. In the general case of porous materials with arbitrary microstructures the only predictions that can be made are in the form of very wide bounds, restricting the domain of admissible effective property values (e.g. the Voigt bound[11] or the Hashin–Shtrikman upper bound[12] for the elastic moduli and the thermodynamic stability bounds for the Poisson ratio[13]). In spite of this unpleasant situation, for common microstructures (e.g. materials made with pore-forming agents) simple predictions are available for the Young's modulus, and the various theoretical predictions for the Poisson ratio are relatively close as long as the porosities are not too high. It is the purpose of this paper to demonstrate the capability of different relations

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to describe the Young's-modulus-vs.-porosity and the Poisson-ratio-vs.-porosity dependence by comparing the predictions with experimentally measured values. We investigate porous alumina, zirconia and alumina–zirconia composite ceramics prepared with starch as a pore-forming agent (including alumina prepared by starch consolidation casting) and measured by the resonant frequency method.

2. Theoretical

The effective Young's modulus of porous ceramics of arbitrary microstructure is bounded from above by the Voigt bound,[10,11]

$$E_V = E_0 (1 - \phi), \tag{1}$$

where ϕ is the porosity (volume fraction of pores), E_0 the Young's modulus of the solid phase and the subscript "V" denotes the Voigt bound. If the microstructure is isotropic, the effective Young's modulus is bounded by the upper Hashin–Shtrikman bound[12] (subscript "HS", superscript "+"), which is approximately[6–9]

$$E_{HS}^+ = E_0 \left(\frac{1 - \phi}{1 + \phi} \right). \tag{2}$$

Lower Young's modulus bounds do not exist for porous materials (more precisely, the Reuss bound and the lower Hashin–Shtrikman bound degenerate to zero for all finite porosities, i.e. $\phi > 0$).[6–9] Moreover, no comparable bounds exist for the Poisson ratio. For porous materials (with infinite phase contrast, i.e. an infinite ratio of the solid and pore phase bulk and shear moduli) there are no bounds in addition to the universal Poisson ratio bounds

$$-1 < \nu < 0.5, \tag{3}$$

which follow from thermodynamic stability arguments or, in other words, from the required non-negativity of elastic moduli.[13,14]

In the absence of general lower bounds it is useful to dispose at least of special relations which may be used to approximately predict (estimate) the porosity dependence of the elastic moduli for certain types of microstructures. Physically meaningful relations of this kind should reduce to the exact solution (linear approximation) in the dilute limit (i.e. for very small porosities) and must not violate the Voigt bound (or the upper Hashin–Shtrikman bound if the microstructure is isotropic) at any porosity value. For realistic solid phase Poisson ratios in the range 0.1–0.4 and isometric pore shape the dilute limit approximation for the effective Young's modulus is[6–8,10]

$$E = E_0 (1 - 2\phi). \tag{4}$$

We note in passing that this linear relation is at the same time identical to the self-consistent prediction[15,16] when a perfectly complaint pore phase is assumed (as usual). These self-consistent schemes have been criticized from a fundamental theoretical standpoint by Torquato.[10] In particular, since this

linear relation (and *eo ipso* also the self-consistent models) predict a spurious percolation threshold (where E becomes zero) at a porosity of 50% ($\phi = 0.5$),[10] which would erroneously imply the non-existence of solid porous materials at higher porosities, it is clear that non-linear relations are required to describe real material behavior, because in real materials the percolation threshold–if present at all–depends on the details of the microstructure (beyond porosity). Now there are – apart from the upper Hashin–Shtrikman bound, Equation (2) – two meaningful non-linear relations that reduce to the linear approximation, Equation (4), in the dilute limit and at the same time do not violate the upper bounds. The first is the power-law relation[6–9]

$$E = E_0(1 - \phi)^2, \tag{5}$$

the second is the exponential relation[6–9]

$$E = E_0 \exp\left(\frac{-2\phi}{1 - \phi}\right). \tag{6}$$

Both can be derived using a functional equation approach.[17,18] The latter predicts significantly lower elastic moduli, and its conductivity counterpart (where the numerical coefficient is 3/2 insetad of 2) has turned out to provide a good prediction for the porosity dependence of the thermal conductivity for porous alumina ceramics prepared with pore-forming agents.[19]

Apart from these two theoretically relatively well founded relations, another, more empirical, relation may be devised, based on the fact that the effective properties of many two-phase composites tend to be close to the upper bound when the high-value property phase is prevailing, and vice versa. With increasing content of the second phase (i.e. the low-value-property phase) the effective property exhibits a decrease with an inflection, until for prevailing low-value-property phase content the lower bound (which may be zero in the case of porous materials) is asymptotically approached. Based on this argumentation, which is very similar to that leading to the Bruggeman–Landauer relation[20,21] or the third-order Miller bounds[22] for the conductivity of symmetric-cell materials, we may apply simple volume-fraction weighting to obtain an "empirical" average of the upper and lower bound, i.e.

$$E = \phi_1 E^+ + \phi_2 E^-, \tag{7}$$

where ϕ_1 and ϕ_2 are the volume fractions of the high- and low-value-property phase, respectively (with $\phi_1 = 1 - \phi_2$ and $\phi_2 = \phi$ in the case of porous materials), and E^+ and E^- are the upper bound (volume-weighted arithmetic average) and lower bound (volume-weighted harmonic average), respectively (the latter being zero for porous materials). Taking the Voigt bound as the upper bound, we regain the power-law relation, Equation (5). However, inserting for E^+ the upper Hashin–Shtrikman bound we obtain the "empirical" average relation ("symmetric-cell average")

$$E_{emp.av.} = E_0 \left[\frac{(1 - \phi)^2}{1 + \phi} \right]. \tag{8}$$
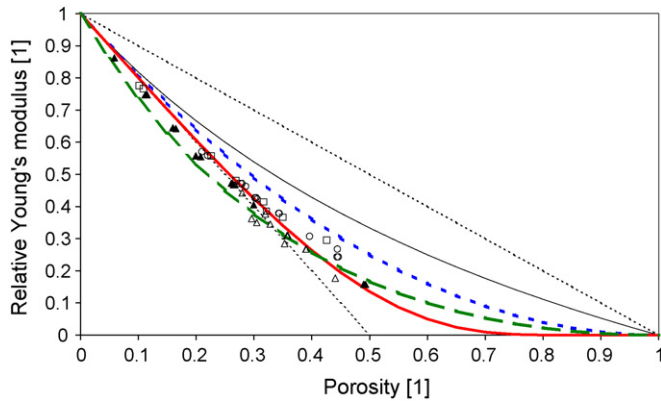
Fig. 1. Relative Young's moduli calculated from resonance frequency results for porous alumina (prepared by TSC—full triangles or SCC—empty triangles), ATZ composite ceramics (SCC—empty circles) and zirconia ceramics (SCC—empty squares), and theoretical predictions of the porosity dependence of the relative Young's modulus: upper bounds (dotted thin top line: Voigt, full thin curve: upper Hashin–Shtrikman), dilute approximation (dashed thin bottom line) and other predictions (dotted bold: power-law, full bold: exponential, dashed bold: symmetric-cell average).

To the best of our knowledge this relation is new and has not been used in the elasticity literature before. Fig. 1 compares the porosity dependences of the relative (or reduced) Young's modulus $E_r = E/E_0$, as predicted via Equations (1), (2), (4–6) and (8). In concluding this paragraph we would like to emphasize that more sophisticated predictions are available in the literature, in particular those based on the Eshelby solution for general ellipsoidal inclusions.[23] All these models, however, require an independent quantitative knowledge of the pore shape (e.g. in the form of an aspect ratio), which–even in those lucky cases where a pore shape can be defined–is not readily accessible in most cases. Of course, in those few cases where it is, the approaches of Wu[24] or Ondracek-Boccaccini[25] may be applied to predict the Young's modulus of materials with ellipsoidal or spheroidal pores. However, whenever explicit pore shape information is lacking, the assumption of isometric pores seems the only feasible choice. Therefore we believe that the benchmark relations given above [Equations (4–6)], which owe their numerical parameter value of 2 to the spherical pore solution (i.e. a special case of the Eshelby solution), will retain their significance (not to say their superiority) for most practical purposes.

The effective Poisson ratio of porous materials is difficult to predict. However, many authors seem to agree upon the fact that the effective Poisson ratio of the porous material decreases with increasing porosity when the Poisson ratio of the solid phase is above a certain value, and increase otherwise.[26,27] The rationale behind this agreement is the assumption that the same type of porosity dependence (though generally with different numerical coefficients) is exhibited by two elastic moduli. That means, it is assumed that a porous material for which the bulk modulus depends on porosity according to the Hashin–Shtrikman upper bound will exhibit the same porosity dependence also for the shear modulus (model materials with this feature may be called "Hashin–Shtrikman materials"). Alternatively, when the porosity dependence of the Young's modulus is described by a power-law relation also the porosity dependence of the

shear modulus is expected to be of the power-law type ("power-law materials") and in a completely analogous manner we may define "exponential materials". The resulting expressions for the effective Poisson ratios are

$$\nu_{HS} = \frac{(1 + \nu_0)(1 + B\phi) - (1 - 2\nu_0)(1 + A\phi)}{2(1 + \nu_0)(1 + B\phi) + (1 - 2\nu_0)(1 + A\phi)} \quad (9)$$

$$\nu_{power} = (1 + \nu_0)(1 - \phi)^{[E]-[G]} - 1 \quad (10)$$

$$\nu_{exponential} = (1 + \nu_0)\exp\left(\frac{([G] - [E]\phi)}{1 - \phi}\right) - 1 \quad (11)$$

for Hashin–Shtrikman, power-law and exponential materials, respectively, where

$$A = \frac{1 + \nu_0}{2(1 - 2\nu_0)}, \quad (12)$$

$$B = \frac{2(4 - 5\nu_0)}{(7 - 5\nu_0)}, \quad (13)$$

$$[G] = \frac{15(1 - \nu_0)}{7 - 5\nu_0}, \quad (14)$$

$$[E] = \frac{3(1 - \nu_0)(9 + 5\nu_0)}{2(7 - 5\nu_0)}, \quad (15)$$

see Refs.[6,8,28] The dilute limit approximation for the porosity dependence of the Poisson ratio is[10]

$$\nu = \nu_0 + \frac{3(1 - \nu_0^2)(1 - 5\nu_0)}{2(7 - 5\nu_0)} \cdot \phi. \quad (16)$$

All these relations predict that the effective Poisson ratio of a porous material should be independent of porosity when the solid Poisson ratio is 0.2, in agreement with the differential scheme approach prediction (Zimmermann approximation[29]) and other relations,[28] while for solid Poisson ratios above 0.2 the effective Poisson ratio of the porous material exhibits a decreasing trend with porosity (and vice versa). Note that–in contrast to the elastic moduli–it makes no sense to define a relative (or reduced) Poisson ratio. In the case of two-phase composites where the phase elastic moduli (subscript 1 and 2, respectively) are of the same order of magnitude, the Poisson ratio of the fully dense (pore-free) composite may be calculated in good approximation via the mixture rule[6]

$$\nu = \phi_1\nu_1 + \phi_2\nu_2. \quad (17)$$

3. Experimental

Porous ceramics samples have been produced by two variants of slip casting, traditional slip casting (TSC, into porous plaster molds; in this method starch is used only as a pore-forming agent) and starch consolidation casting (SCC, into non-porous brass molds; in this method starch is used as a combined pore-forming and body-forming agent: body formation occurs by starch swelling and gelatinization during heating to approximately 80 °C which is accompanied by water absorption from the suspension). The ceramic powders used in this work were submicron alumina powder (CT 3000 SG, Almatis,

Germany, purity approximately 99.8% α-Al$_2$O$_3$, median grain size approximately 0.7–0.8 µm), submicron tetragonal zirconia powder stabilized by 3 mol.%, i.e. 5 wt.%, Y$_2$O$_3$ (TZ-3Y-E, Tosoh, Japan, containing >99.7 wt.% ZrO$_2$ with a crystallite size of order tens of nanometers and 0.25 ± 0.1 wt.% Al$_2$O$_3$) and alumina-containing tetragonal zirconia composite powder (ATZ-80, Daiichi, Japan, a powder mix containing 80 wt.% ZrO$_2$ and 20 wt.% Al$_2$O$_3$, corresponding to a zirconia volume fraction of 0.726). "Waxy" corn starch with a median granule size of approximately 14 µm (Amioca Powder TF, National Starch & Chemical, UK) was used as a pore-forming agent (and a body-forming in SCC).

In both cases aqueous suspensions were prepared with 70–80 wt.% ceramic powder, 1 wt.% deflocculant (Dolapix CE64, Zschimmer & Schwarz, Germany) and starch concentrations ranging from 5 to 50 vol.% (related to ceramic powder) in TSC and 10–50 vol.% in SCC. Details of the starch granule size (distributions) and shape as well as suspension preparation, casting techniques (TSC and SCC) and resulting microstructures have been published in several papers, to which the reader may refer.[30–32] After demolding, the sample bodies (cylinders of approximately 7 mm diameter and 80 mm length for SCC and cylinders of approximately 5 mm diameter and 60 mm length for TSC) were dried for 24 h at room temperature, followed by drying at 105 °C to constant mass. Subsequently, the bodies were fired according to a standard schedule (heating rate 2 °C/min, 2 h dwell at maximum temperature) at 1570 °C, 1520 °C and 1490 °C for alumina, alumina-containing tetragonal zirconia composite (ATZ) and zirconia ceramics, respectively. Starch burnout takes place during the heating period in the temperature range 300–600 °C.[32] Finally, the as-fired cylindrical bodies were saw-cut into samples with aspect ratio approximately 10 for the elastic property measurements. The bulk density ρ was determined both from the mass and the geometrical dimensions of the samples as well as by the Archimedes method. Total porosites ϕ were determined using the relation

$$\phi = 1 - \frac{\rho}{\rho_0}, \tag{18}$$

where ρ_0 is the theoretical density of the fully dense, i.e. pore-free ceramics (4.0 g/cm^3, 5.52 g/cm^3 and 6.1 g/cm^3 for alumina, ATZ and zirconia, respectively).

The (adiabatic) elastic constants (Young's modulus E and Poisson ratio ν) were measured using a dynamic technique, the resonant frequency method.[33] The measurements were performed with a resonant frequency tester (Erudite, CNS Electronics Ltd., London, UK) in the frequency range 10 kHz–1 MHz, using a unit based on electrostatic excitation and sensing of mechanical longitudinal vibrations. This electrostatic variant of the resonant frequency method is especially suitable for measuring small specimens because of the minimum number of constructional elements which might contribute to the measured spectrum with their own frequencies.

Due to the cylindrical shape of the samples, it is possible to determine the Young's modulus and Poisson ratio from longitudinal vibrations, evaluated on the basis of Pochhammer's theory of the propagation of stress waves in cylindrical bodies.[34,35] This approach enables an exact analytical solution to be obtained and ensures a rather precise description of the geometrical dispersion of stress waves for materials with negligible attenuation (i.e. purely elastic materials). For materials with Poisson ratios between 0.15 and 0.5 and specimens with appropriate geometry, i.e. d/λ_n ratios in the range 0–0.6 (d = specimen diameter, λ_n = wavelength of the n-th resonant frequency mode, i.e. fundamental mode $n = 1$, first overtone $n = 2$, second overtone $n = 3$ etc.) the Young's modulus can be experimentally obtained from an approximation to the exact analytical solution (approximation to the first branch of Pochhammer frequency equation),[36] which can be written as

$$E = \rho c^2 \left[1 + \left(\frac{d}{2\lambda_n} \right)^2 \right]^{-2\left[P\nu + Q + (R\nu + S)(d/2\lambda_n) \right]}. \tag{19}$$

In this relation ρ is the density (in the case of porous materials the bulk density of the specimens), c the wave velocity (longitudinal phase velocity). For the case of longitudinal resonant frequencies the wave velocity can be determined by the relation[33]

$$c = \frac{2 L f_n}{n}, \tag{20}$$

where L is the specimen length and f_n the resonant frequency of the n-th longitudinal vibration mode. Further, the wavelength corresponding to the longitudinal resonant frequency is related to the specimen length via the relation $\lambda_n = 2L/n$, the numerical parameters are $p = -6.35$, $Q = 1.34$, $R = 4.0$ and $S = -4.5$, and the Poisson ratio can be determined from two different resonant frequencies (modes denoted by indices A and B) via the relation[36]

$$\nu = \frac{\ln(c_A/c_B) + (Q + S(d/2\lambda_B)) \ln\left(1 + (d/2\lambda_B)^2\right) - (Q + S(d/2\lambda_A)) \ln\left(1 + (d/2\lambda_A)^2\right)}{(P + R(d/2\lambda_A)) \ln\left(1 + (d/2\lambda_A)^2\right) - (P + R(d/2\lambda_B)) \ln\left(1 + (d/2\lambda_B)^2\right)}. \tag{21}$$

For maximum precision the two frequencies (A and B) have to be maximally apart, of course without exceeding the range of validity of Equation (1), i.e. $d/\lambda_n < 0.6$ and measured ν values in the range $0.15 \leq \nu < 0.5$. The error in the Young's modulus is usually estimated to be <1%,[37] while the error in the Poisson ratio can attain several percent.[38,39]

4. Results and discussion

Fig. 1 shows relative Young's moduli $E_r = E/E_0$, calculated from the measured effective Young's moduli E (determined by the resonant frequency method) and the E_0 values based on literature data (400, 251 and 210 GPa for alumina, ATZ and zirconia, respectively[40]) for porous alumina (prepared via TSC or SCC, respectively), ATZ composite and zirconia ceramics (both prepared via SCC only). All E_r values are clearly below the power-law prediction and, therefore, far below the upper Hashin–Shtrikman bound. There is of course no doubt that the

dependence is nonlinear. Irrespective of the slight tendency to higher values for the zirconia-based ceramics as compared to the pure alumina ceramics (see below) it can be said that the exponential relation [Equation (6)] and the "empirical" average relation [Equation (8)] provide the best parameter-free predictions of the observed porosity dependence. It has to be remembered that Equation (6) can be derived via a functional equation approach,[17,18] and, very recently, the form of Equation (6) has been attributed to a high degree of disorder in the pore space.[41] On the other hand, for Equation (8) no rigorous theoretical foundations are known. This might be an argument in favor of Equation (6), even in cases where Equation (8) seems to describe the porosity dependence equally well or better. For higher porosities it seems that the E_r values are slightly higher for the zirconia-based ceramics than for the alumina. Of course, this finding should not be overinterpreted, because it may be due to an accidental sample-to-sample variation or slightly underestimated E_0 values (and would certainly require verification with a broader data base, before definitive conclusions should be made). We note, however, that a similar tendency has been observed for the relative thermal conductivity of alumina.[19] The difference between TSC values (full triangles) and SCC values (empty triangles) seems to be insignificant, as evidenced for alumina in Fig. 1, indicating that the important (i.e. property-determining) microstructural features resulting from the two processes are very similar.

Fig. 2 shows the porosity dependence of the Poisson ratio for porous alumina ceramics. The errors are relatively high, as expected, but the results can be interpreted in the sense that the Poisson ratio does not change substantially with porosity (up to a porosity value of 50%). This is the expected result predicted by all models considered, since the Poisson ratio of the solid phase ($\nu_0 = 0.23$ for pure alumina when $\phi = 0$) is very close to 0.2.[40] For the zirconia-based ceramics (see Figs. 3 and 4), which have been prepared with porosities of up to approximately 45%, the solid Poisson ratio is higher (0.29 and 0.31 for ATZ and zirconia, respectively)[40] and a decreasing trend is observed. A similar trend has been observed by Puchegger et

Fig. 3. Poisson ratio of porous ATZ composite ceramics (prepared by SCC—empty symbols); measured values and predictions for Hashin–Shtrikman materials (dashed thin line), linear materials (dotted thin line), power-law materials (dotted bold curve) and exponential materials (dashed bold curve), respectively.

al. for an alumina–zirconia composite with 82.5 vol.% Al_2O_3 and 17.5% ZrO_2, where the measurements have been made by a similar dynamic method (resonant beam technique).[42] While for the ATZ composite it is not easy to decide which predictive model is to be preferred, for zirconia (highest ν_0) the prediction for exponential materials, Equation (11), seems to be the best. That means, when explicit measurement results are not available, Equation (11) seems to be a useful relation for estimating the Poisson ratio of porous alumina- and zirconia-based ceramics with porosities up to approximately 50%. It has to be emphasized, however, that the porosity dependence up to 50% porosity is relatively weak for all these ceramics, wheras extrapolations to porosities significantly higher than 50% are questionable and would require further investigation. In particular, although there are papers reporting slightly negative Poisson ratios for cellular ceramics,[43,44] the fundamental question as to the conditions of a possible occurrence of auxetic behavior at high porosities is still unsolved.[28] The only definitive statement that can be made today is that the Poisson ratio is not a function of porosity alone. Or more precisely (and less trivial), it is sure that even to the
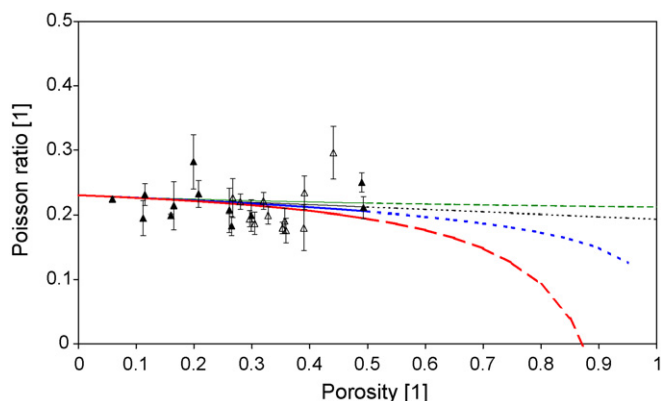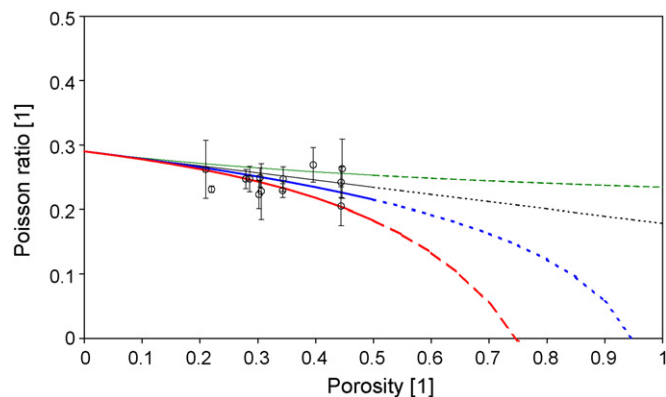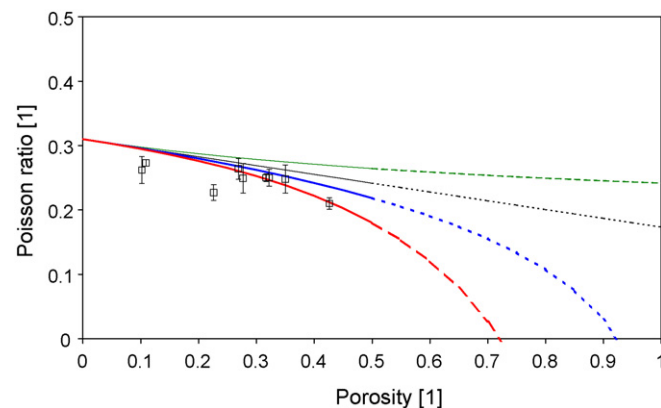

Fig. 2. Poisson ratio of porous alumina ceramics (prepared by TSC–full symbols or SCC–empty symbols); measured values and predictions for Hashin–Shtrikman materials (dashed thin line), linear materials (dotted thin line), power-law materials (dotted bold curve) and exponential materials (dashed bold curve), respectively.

Fig. 4. Poisson ratio of porous zirconia ceramics (prepared by SCC–empty symbols); measured values and predictions for Hashin–Shtrikman materials (dashed thin line), linear materials (dotted thin line), power-law materials (dotted bold curve) and exponential materials (dashed bold curve), respectively.

degree of approximation to which the other elastic constants are functions of porosity alone the Poisson ratio is not. However, the main influential microstructural parameter in addition to porosity is yet to be identified.

5. Summary and conclusion

Well-known theoretical relations for the prediction of the porosity dependence of the Young's modulus have been briefly summarized, a new relation has been proposed (the "empirical" symmetric-cell average) and recently derived predictive relations for the Poisson ratio have been recalled. Young's moduli and Poisson ratios have been measured for porous alumina and zirconia-based ceramics (prepared with corn starch as a pore-forming agent) via the resonant frequency technique, using an approximation to the Pochhammer relation for data evaluation. It has been found that the resulting Young's moduli are far below the upper Hashin–Shtrikman bound and also clearly below the power-law relation. Both the exponential relation and the newly proposed symmetric-cell average provide a reasonable prediction of the porosity dependence of the Young's modulus (the former with the advantage of being theoretically rather well founded). Systematic differences in the relative Young's moduli between alumina ceramics prepared via traditional slip casting (TSC) and starch consolidation casting (SCC), respectively, could not be confirmed (indicating that the property-determining microstructural features are essentially the same) and the differences in the relative Young's moduli of alumina ceramics and zirconia-based ceramics are too small to be conclusive. Up to porosities of approximately 50% the porosity dependence of the Poisson ratio is rather weak, but the decreasing trend for the zirconia-based ceramics can be well described by the relation assuming exponential material behavior.

Acknowledgements

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