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Effect of spherical pores on the strength of a polycrystalline ceramic

Ö. VARDAR and I. FINNIE

Department of Mechanical Engineering, University of California, Berkeley, California 94720, USA

D. R. BISWAS and R. M. FULRATH

Department of Materials Science and Engineering and the Materials and Molecular Research Division, Lawrence Berkeley Laboratory, University of California, Berkeley, California 94720, USA

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ABSTRACT
Bending tests on a ceramic material, lead zirconate-titanate, with varying amounts of porosity, show that the decrease in strength due to spherical pores is much less than would be expected from the stress concentration factor. Using the Weibull probabilistic approach to brittle strength, it is shown that reasonable predictions may be made for the median strength for porosity up to about ten percent.

1. Introduction

The effect of stress concentrations on the average strength of brittle solids, subjected to tensile states of stress, is invariably less than would be expected from the "theoretical" elastic stress concentration factor. This observation can be explained qualitatively by noting that brittle solids contain a distribution of inherent flaws of varying severity. The probability of finding the most severe flaw in the specimen in the localized stress field around a stress concentration is very low. For this reason, the flaws responsible for fracture with stress concentrations present will be less severe, generally, than those in smooth specimens where large regions are exposed to uniform stresses.

A quantitative explanation of the effect of stress concentrations is possible in some cases by using the Weibull probabilistic treatment of brittle strength [1]. The assumptions involved in applying the Weibull treatment to multiaxial stresses and details of the formulation are given in a recent paper [2] and will not be repeated. Applications of Weibull's approach to predict the role of stress concentrations in brittle solids have been limited. This may be due in part to the complexity of the numerical computations involved. However, with high speed computers this is no longer a problem.

The case we shall consider is that of the strength of polycrystalline ceramics containing spherical holes. After obtaining some general results we will show that predictions are in good agreement with experiments on a ferroelectric ceramic, lead zirconate-titanate (PZT).

2. The state of stress in a porous specimen

We consider the configuration shown in Fig. 1 in which a specimen of total volume V is loaded by a nominal stress S. The specimen contains N spherical pores, each of volume V_0. We assume that the pores are far enough apart so that the regions of high

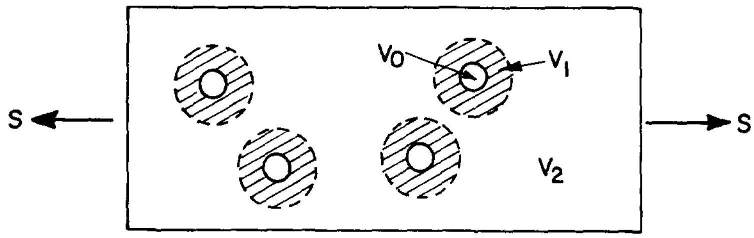

Figure 1. Specimen of total volume V containing N spherical pores and subjected to uniaxial tensile stress S.

stress do not interact. Taking the highly stressed region around a pore to have a volume V_1, which we will specify later, the remaining material of volume V_2 is assumed to be subjected only to a uniform uniaxial stress.

Because of the decrease in cross-sectional area due to the pores, the stress in volume V_2 will have a value τ greater than the nominal stress S. Since the area fraction of a second phase is equal to its volume fraction, $\tau = S \div (1-p)$.

For an isolated spherical hole, as shown in Fig. 1, the stress state for elastic behavior was given by Goodier [3]. We reproduce his results, using the coordinate system shown in Fig. 2.

$$\sigma_r = \frac{S}{2}(1-\cos 2\theta) + \frac{S}{2(7-5\nu)}\left(\frac{a}{r}\right)^3 \left\{(5\nu-13) + 6\left(\frac{a}{r}\right)^2 + \left[5(5-\nu) - 18\left(\frac{a}{r}\right)^2\right]\cos 2\theta\right\}$$

$$\sigma_\theta = \frac{S}{2}(1+\cos 2\theta) + \frac{S}{4(7-5\nu)}\left(\frac{a}{r}\right)^3 \left\{(13-20\nu) - 3\left(\frac{a}{r}\right)^2 - \left[5(1-2\nu) - 21\left(\frac{a}{r}\right)^2\right]\cos 2\theta\right\}$$

$$\sigma_\alpha = \frac{3S}{4(7-5\nu)}\left(\frac{a}{r}\right)^3 \left\{1 - 3\left(\frac{a}{r}\right)^2 - 5\left[(1-2\nu) - \left(\frac{a}{r}\right)^2\right]\cos 2\theta\right\}$$

$$\tau_{r\theta} = \frac{S}{2(7-5\nu)}\left(\frac{a}{r}\right)^3 \left[-5(1+\nu) + 12\left(\frac{a}{r}\right)^2\right]\sin 2\theta - \frac{S}{2}\sin 2\theta.$$

The ratios of the stress components, or of the principal stresses σ_1, σ_2, σ_3, to the nominal stress S are functions only of r/a and θ for a given Poisson's ratio, ν. The maximum stress concentration $\sigma_\theta(r=a,\ \theta=0) \div S$ is $(27-15\nu)/(14-10\nu)$. For the range of Poisson's ratio we will be considering this varies from a value of 2 for $\nu = 0.2$ to 2.045 for $\nu = 0.3$. When many non-interacting spherical holes are present, the previous stresses must be modified by replacing S by τ.

One approach to predicting the strength of a porous ceramic would be to equate the maximum local stress at the surface of the pore to the strength of material with zero porosity.

However, this approach greatly underestimates the strength of the porous material we tested and sheds no light on the observed variation of strength with

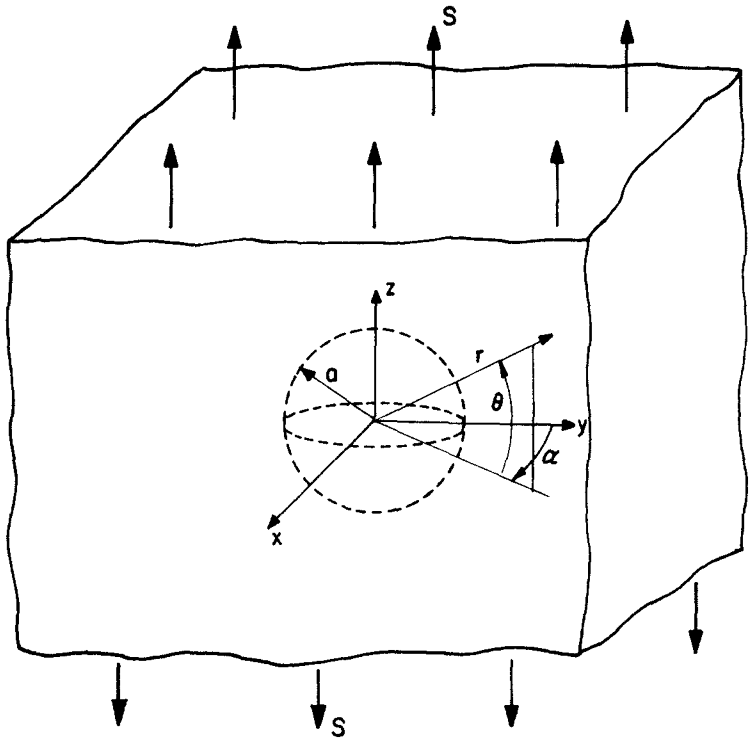

Figure 2. Coordinate system used to describe stress state around a spherical hole.

porosity. For this reason we turn to the Weibull probabilistic approach to brittle strength which allows us to consider the effect of the stressed volume, and the stress distribution on the probability of failure.

3. The Weibull approach

Since the Weibull approach for multiaxial stress states was described in detail in an earlier paper [2], our treatment here will be concise.

For uniaxial stress states, Weibull [1] took the probability of failure as

$$F(\sigma) = 1 - \exp - \left[\int (\sigma/\sigma_0)^m \, dV \right] \tag{1}$$

with the integral being taken over the region stressed in tension. The parameters m and σ_0 which are assumed to characterize a given material may be obtained, for example, from bending test data.

For multiaxial stress states such as shown in Fig. 3 (taken from [2]), Weibull wrote the cumulative distribution of failure probability as:

$$F(\sigma) = 1 - \exp[-B] = 1 - \exp\left[- \int \left(K \int_{A \atop \text{unit sphere}} \sigma_n^m \, dA \right) dV \right] \tag{2}$$

where the normal stress at each point on the unit sphere is

$$\sigma_n = \cos^2 \phi(\sigma_1 \cos^2 \Psi + \sigma_2 \sin^2 \Psi) + \sigma_3 \sin^2 \phi; \qquad dA = \cos \phi \, d\Psi \, d\phi$$

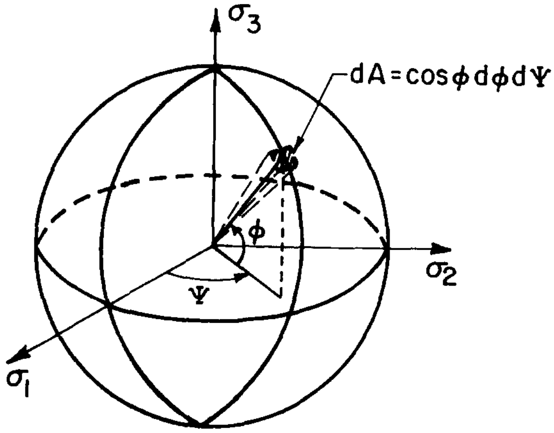

Figure 3. Geometrical variables used to describe location on a unit sphere.

and the integration is limited to the range of angles for which σ_n is tensile. Thus, in the present problem we have to carry out the integration over unit spheres, throughout the volumes V_1 and V_2. The quantity B in Eqn. (2) is then

$$B = \int_{V_2} \left[K \int_{\substack{A \\ \text{unit} \\ \text{sphere}}} (\sigma_3 \sin^2 \phi)^m \cos \phi \, d\Psi \, d\phi \right] dV$$

$$+ N \int_{V_1} K \int_{\substack{A \\ \text{unit} \\ \text{sphere}}} G(r, \theta, \phi, \Psi) \, d\phi \, d\Psi \, d\theta \, d\alpha \, dr$$

where G is a function to be defined later.

The first integral is for the region of uniaxial stress and presents no problem. Putting $\sigma_3 = \tau$ and using the uniaxial formulation it is merely $V_2(\tau/\sigma_0)^m$. The second integral can be written in detail as:

$$Na^3K \int_{r/a} \int_\theta \int_\alpha \left\{ \int_\Psi \int_\phi [\sigma_1^2 \cos^2 \Psi \cos^2 \phi + \sigma_2^2 \sin^2 \Psi \cos^2 \phi + \sigma_3 \sin^2 \phi]^m \right.$$

$$\left. \times \cos \phi \, d\phi \, d\Psi \right\} (r/a)^2 \cos \theta \, d\theta \, d\alpha \, d(r/a)$$

where a is the pore radius and the upper limit of integration on r/a corresponding to volume V_1 has to be specified. The stresses σ_1, σ_2, σ_3, vary with location θ and r/a but for a given Poisson's ratio depend only on θ and r/a. Thus for a given upper limit of integration on r/a

$$B = V_2 \left(\frac{\tau}{\sigma_0} \right)^m + Na^3K\tau^m H(m).$$

Letting λ denote the upper limit of r/a, i.e. λa is the radius of region V_1, we have $V_2 = V - NV_1 - NV_0 = V(1 - p\lambda^3)$. Then,

$$B = V(1-p\lambda^3)\left[\frac{S(1-p)^{-1}}{\sigma_0}\right]^m + \frac{3}{4\pi}Kp[S(1-p)^{-1}]^m H(m)$$

where $H(m)$ will depend on the choice of λ. Comparing with the uniaxial formulation*

$$K = \frac{2(2m+1)}{\pi\sigma_0^m}.$$

So

$$B = \left(\frac{S}{\sigma_0}\right)^m V\left\{\frac{1}{(1-p)^m}\left[(1-p\lambda^3) + \frac{3(2m+1)}{2\pi^2}pH(m)\right]\right\} = \left(\frac{S}{\sigma_0}\right)^m V\{A\}. \tag{3}$$

In practice, we prefer to test brittle solids in 3 or 4 point bending rather than uniaxial tension. There is no problem in extending the previous equation to these cases if we assume the pores are very small compared to the dimensions of the beam and large in number. Taking a span L between outer supports and a span of $L-2l$ between inner supports ($L-2l=0$ for 3 point bending) we may use the Weibull uniaxial formulation, treat the problem as if pores were not present, and then multiply by the quantity between parentheses $\{A\}$ in Eqn. (3). Thus if S is taken as the maximum, outer fiber, tensile stress in bending we obtain an expression for B by multiplying the preceding equation by

$$\left[\left(\frac{m+1}{2}\right) - \frac{ml}{L}\right] \Big/ (m+1)^2.$$

To minimize computation it is more convenient to work with the median strength in a bending test rather than the mean. In this case $B = 0.693$ and

$$\frac{S\text{ median}}{\sigma_0} V^{1/m} = \left(\frac{0.693}{\{A\}}\right)^{1/m} \left(\frac{(m+1)^2}{\left(\left(\frac{m+1}{2}\right) - \frac{ml}{L}\right)}\right)^{1/m}. \tag{4}$$

All that needs be decided before computations can be carried out in the upper value λ for r/a corresponding to volume V_1. Too small a value for λ will underestimate the effect of the pores in reducing the strength while too large a value requires excessive computation and for the larger porosities will lead to an overlap of the regions V_1. Guided by the fact that σ_θ at $\theta = 0$ is within about 5% of the nominal stress at $r/a = 2$ we have evaluated $\{A\}$ and $\{A\}^{1/m}$ for $\nu = 0.3$, $m = 8$ and the values of λ and porosity shown below. Angular increments of 3° and increments in radius of 0.03 a were used in the numerical integration.

Convergence of $\{A\}^{1/m}$, the value of interest for strength predictions, should be more rapid than shown in Table 1 for $m > 8$ and less rapid for $m < 8$. Based on the results given in this table, a value of $\lambda = 2.02$ was selected for subsequent calculations. This choice limits the analysis based on the assumption of isolated spherical voids to a porosity of about ten percent. Having selected a value for λ, the numerical evaluation of Eqn. (4) can be presented in the concise form shown in Fig. 4. Since the

* This differs by a factor 4 from the value given on p. 497 of Ref. [1] because here we have taken advantage of symmetry to integrate only over one quarter of the unit sphere.

TABLE 1
Influence of λ and p on {A} and $\{A\}^{1/m}$ (in parentheses).

p \ λ	1.75	2.02	2.50	2.90
2%	1.606	1.612	1.603	1.582
	(1.061)	(1.061)	(1.060)	(1.059)
4%	2.265	2.262	2.198	2.104
	(1.107)	(1.107)	(1.103)	(1.097)
6%	2.980	2.942	2.767	—
	(1.146)	(1.144)	(1.135)	—
8%	3.749	3.646		
	(1.179)	(1.175)	—	—
10%	4.572	4.358	—	—
	(1.209)	(1.202)	—	—

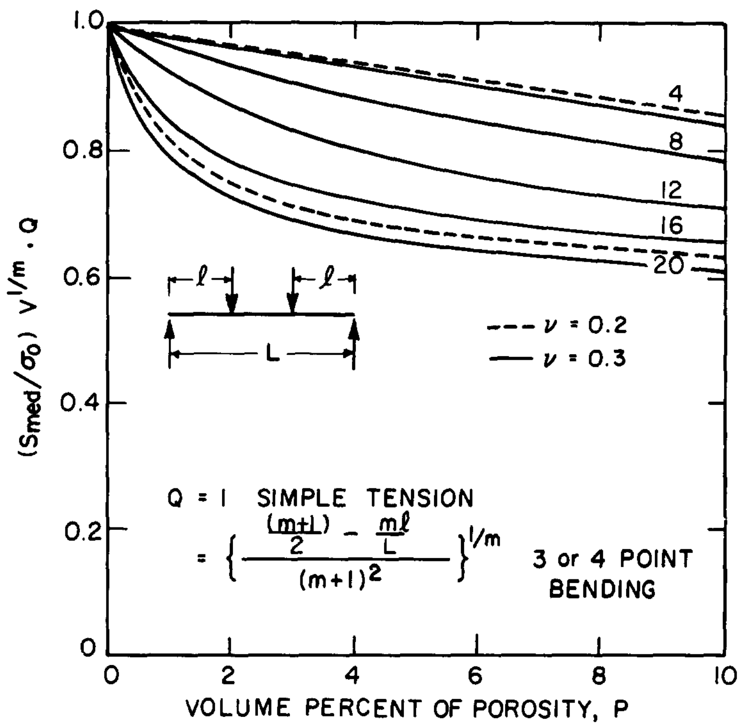

Figure 4. Predicted variation of median strength as a function of porosity for values of the Weibull parameter *m* shown on curves.

effect of Poisson's ratio is fairly insignificant, it was evaluated only for $m = 4$ and $m = 20$.

4. Experimental results

The material used in this study was lead zirconate-titanate (PZT). The samples were fine grained (2–3μ) polycrystalline ceramics, in which the grain size is controlled by doping with 2 mole percent $NbO_{2.5}$. The advantage of using this material is that specimens are easy to fabricate with good reproducibility and control of grain size from batch to batch. It was found that the addition of excess PbO (5.5 weight percent) enhances the ease of fabricating this material by sintering and densities of over 99% of the theoretical value may be obtained without difficulty.

To produce pores, spherical organic powders were introduced in the pressed powder compacts prior to sintering. In the present work, three ranges of spherical pore sizes were used: 25–40μ, 60–75μ and 110–150μ. The percentage of porosity was controlled by weighing the powders before compaction and confirmed by subsequent density measurements on the test specimens. Size ranges for the spherical powders were obtained by screening with sieves. A typical photograph of the microstructure is shown in Fig. 5.

Specimens of nominal dimensions 0.05 inch thickness and 0.3 inch width were cut from the sintered discs using a diamond saw. They were tested in 4 point bending with a distance between supports of 0.75 inch and a distance between loading points of 0.25 inch. Actual dimensions varied slightly; exact values were used in calculating stresses.

From a set of nine tests on fully dense specimens a median strength of 12,390 psi was obtained and values of $m = 20$ and $\sigma_0 = 7750$ were deduced from the distribution of strength values. No correction was made for friction at the supports since the median bending strength is going to be compared with that obtained on porous specimens tested in the same configuration. However, if comparison were to be made with other types of tests, the values of median strength and σ_0 would have to be decreased by about 7%.

Similar series of bending tests were made for each of the twelve combinations of porosity and pore size. Seven series involved 9 specimens, four involved 8 and one

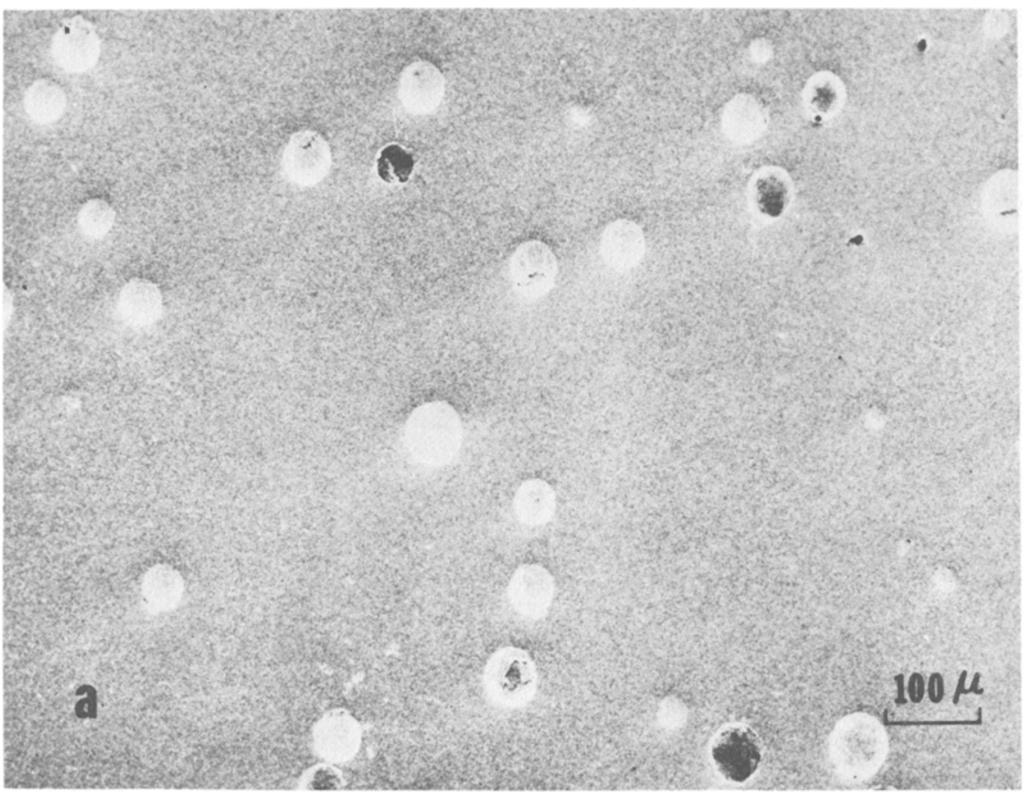

Figure 5. Photograph of specimens containing 4.65% volume fraction of spherical pores 60–75μ.

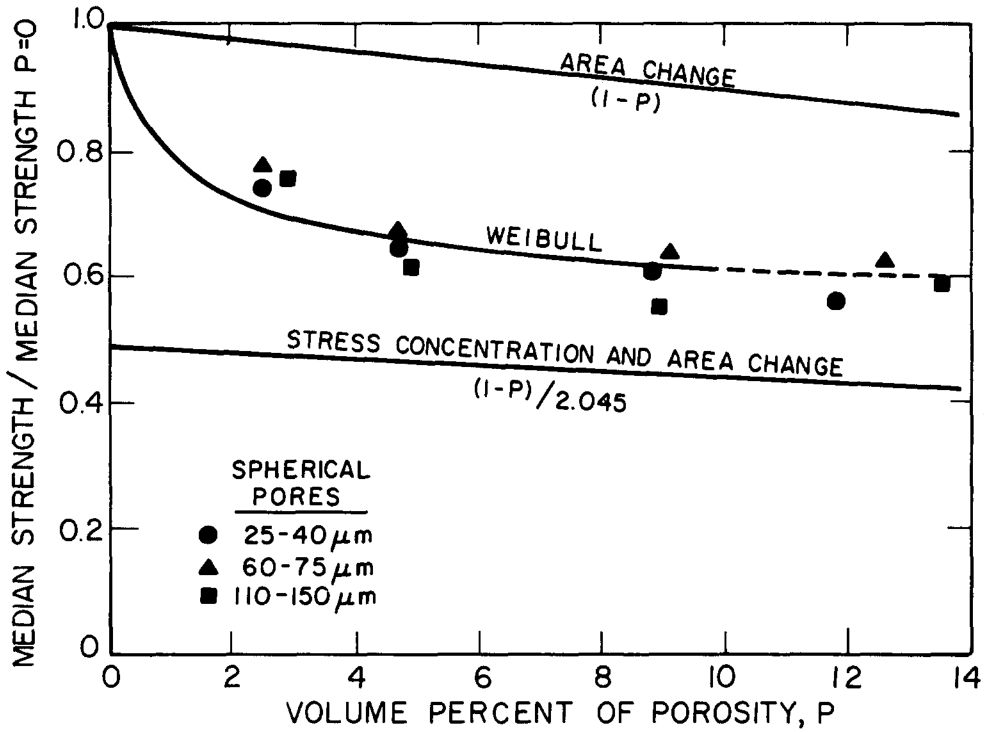

Figure 6. Predicted and observed ratio of median strength with porosity to median strength with zero porosity. The prediction of Fig. 4 is extrapolated for values over $p = 10\%$.

had only 7 specimens. Since the specimens have the same volume and the value of Q in Fig. 4 is unchanged, the ordinate in Fig. 4 may be used directly to estimate the effect of porosity on median strength. Figure 6 shows the experimental results and the prediction based on the Weibull approach. Tests (4) on this material led to an estimate of Poisson's ratio of $\nu = 0.29$. From Fig. 4 it is seen that this value will lead to predictions almost coincident with those for $\nu = 0.3$. For this reason the curve for $m = 20$ and $\nu = 0.3$ is shown in Fig. 6. Also shown in Fig. 6 are the predictions for relative strength obtained by considering only the decrease in cross-sectional area and that obtained by combining the stress concentration factor for $\nu = 0.3$ with the decrease in cross-sectional area due to pores.

There is always uncertainty in estimating parameters of a probability distribution such as m from a limited number of tests. It has been pointed out (5) that various methods of estimating m lead to a normal distribution of m values with the ratio (standard deviation $\div$ mean) $\simeq N^{\frac{1}{2}}$ where N is the sample size. Fortunately, in the present case, it can be seen from Fig. 4 that this uncertainty in m will not have a serious effect on the predictions. The twelve sets of data for porous material whose median values are shown in Fig. 6 should show the same value of m as the fully dense material if our derivation is correct. In fact, the mean value of m from the porous specimens was about ten percent less than that deduced from the solid material with the ratio (standard deviation $\div$ mean) of the m values agreeing well with the "rule of thumb" just quoted. Thus, it could be argued that a value of $m = 18$ would be more appropriate for predictions than $m = 20$. However, the difference is small and since our objective is to predict the behavior of porous ceramics from tests on fully dense material we feel it more consistent to use $m = 20$, while pointing out the uncertainty in estimating parameters from small samples.

5. Conclusions

The effect of porosity on bending strength is predicted quite well by the Weibull approach. Other approaches which might be considered based on stress concentration factors or effective cross-sectional area are completely inadequate.

An interesting feature of the Weibull analysis is the prediction that only the total porosity, and not the pore diameter, will control the strength. This is borne out by the experimental results.

The Weibull analysis is based on the assumption that a large number of inherent flaws exist throughout the stressed volume. The present material with its very fine grain size relative to the pore size is probably very close to this ideal. In other cases, with larger grain sizes relative to the pore size, predictions might not be as successful.

Acknowledgement

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RÉSUMÉ

Les essais de flexion sur un matériau céramique: zirconate-titanate de plomb, avec des quantités variables de porosités, montrent que la diminution de la résistance due à la porosité est plus faible que celle donnée par le facteur de concentration de contraintes. En utilisant la méthode statistique de Weibull pour la résistance fragile, il est montré que des prévisions raisonables peuvent être faites pour déterminer la résistance médiane pour une porosité jusqu'aux environs de dix pourcents.