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Crack Deflection in Ceramic Laminates Using Porous Interlayers

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Abstract

Ceramic laminates have been made from alternating layers of silicon carbide and silicon carbide containing a fugitive polymer, which can be pyrolysed to produce porous interlayers. It is shown that such interlayers can be used to deflect cracks and that the volume fraction of porosity, due to the added polymer, that is required to cause crack deflection is approximately 0·4. A simple model has been developed which describes the fracture behaviour of porous solids and also predicts the volume fraction of porosity required to give crack deflection in the laminate and which is in good agreement with experiment. © 1998 Elsevier Science Limited. All rights reserved

1 Introduction

Previous work has demonstrated the benefits of laminating either strong ceramic layers[1] or fibres[2] with crack deflecting interlayers to produce ceramic components with enhanced resistance in applications where there are high thermal loads.[3,4] Because the laminates are produced using ceramic powders, the production method could potentially be used for many systems. However, to date the approach has only been demonstrated for a limited range of systems due to the difficulty in finding suitable interfacial materials.[1,5–8]

To be useful, such an interfacial material must be chemically compatible with the laminae, so that it can be both co-fired and later used at an elevated temperature, and it must also reliably deflect cracks. The requirement of chemical compatibility suggests that both lamina and interlayer should ideally be made from the same material. This would have the added advantage that any internal stresses due to differences in thermal expansion coefficients between the lamina and interlayer materials, which can cause delamination in these systems,[8] could be avoided.

The possibility of using porous or intermittent interlayers to deflect cracks was first investigated by Atkins for use in fibrous polymer composites.[9,10] The approach has also been employed in polymer laminates[11] and metal matrix composites.[12] More recently the possibility of using such an interface in ceramic matrix composites has been investigated, but it does not appear to be possible to produce an interface that will have both the ability to deflect a crack and to have a sufficiently low shear strength to enable fibre pull-out.[13,14] In systems made by laminating either tapes or fibres separated by some crack deflecting interface, the only source of toughening is crack deflection so that it is no longer necessary to obtain a low shear strength. That porous interlayers can deflect cracks in laminate type ceramic structures has already been demonstrated.[1,7] The aim of this work, therefore, is to establish what levels of porosity are required to ensure crack deflection, at least for the situation where no residual stresses are present.

2 Experimental

Slurries suitable for tape casting were made by mixing the silicon carbide powder (Superior Graphite, HSC-059 s) with distilled water and a dispersing agent (Lignotech, Sweden, grade Wargonin Extra) at a concentration of 0·15% by weight of the silicon carbide and then ball milling for 24 h in a polyethylene jar using zirconia milling media. Before milling, the pH of the slurry was adjusted to 9 using ammonia solution. To produce handleable ceramic tapes, an acrylic styrene latex binder (Hoechst Perstorp, grade Mowilith DM 765S) was

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added to the slurry and stirred for 1 h. The amount of latex polymer used was 0·3 by volume of the total solids, that is the silicon carbide and the latex.[15,16]

To make the pore-containing materials, polytetrafluoroethylene (P.T.F.E.) particles with a diameter between 5 and 9 μm (Goodfellow) were added to the silicon carbide dispersion and gently mixed. The same concentrations of dispersant and latex as above were used except that the P.T.F.E. was included as one of the solids. The volume fractions of P.T.F.E. quoted here are based on the volume of P.T.F.E. as a fraction of the volume of the P.T.F.E. and the silicon carbide together, V_{st}, and ranged between 0·35 and 0·65, using the densities of P.T.F.E. and silicon carbide as 2·20 and 3·21 $\mathrm{Mg\,m^{-3}}$. The resulting slurries were then sieved through a mesh with a fibre spacing of 31 μm to remove air bubbles.

The slurries were tape cast using a continuous feed tape casting machine (Wallace Technical Ceramics Inc, model TC 155) onto a polypropylene carrier tape moving at 20 $\mathrm{mm\,s^{-1}}$. This gave a total drying time of 186 s. The tapes were of a uniform thickness, had smooth surfaces and were free of visible pinholes. The gap between the doctor blade and the carrier tape was 200 μm and gave tapes with a thickness of approximately 100 μm. The sheets were then cut into squares 50×50 mm. Squares of the P.T.F.E.-containing and P.T.F.E.-free sheets were stacked alternately and pressed together at room temperature at a pressure of 40 MPa. Bonding at room temperature was possible because the, latex binder was above its glass transition temperature of −16°C. The resulting plates were then heated at 0·1°C per min to 650°C for 30 min under nitrogen to pyrolyse, the polymer and then heated under argon to 2050°C for 30 min. For material containing no added P.T.F.E. the final density was 0·95.

The volume fraction of pores in the green body that are caused by the added P.T.F.E. particles, V_g, is given by Slamovich and Lange [17] as

$$V_g = \frac{V_{st}\rho_g}{1 - V_{st} + V_{st}\rho_g}$$

where ρ_g is the volume fraction of the matrix material, in this case taken to be equal to that of the silicon carbide green body to which no P.T.F.E. has been added. If these pores are sufficiently large that they do not themselves sinter, then they will shrink by the same amount as the surrounding matrix so that the volume fraction of pores in the body will remain constant during sintering. The fracture surface of a porous layer after sintering, containing a volume fraction of pores due to the added P.T.F.E. of 0·44 is shown in Fig. 1.

To investigate whether cracks could be deflected at the porous interlayers, beams were cut from the sintered plates approximately 0·5 mm thick, 2 mm in breadth and 35 mm long, using a diamond impregnated saw. These were then tested in three point flexure using a loading span of 30 mm.

3 Results and discussion

It was found that interlayers which contained a volume fraction of porosity of 0·34 or less, corresponding to a volume fraction of P.T.F.E. particles of 0·45, did not reliably deflect cracks, as shown in Fig. 2(a). It can be seen that the crack does change direction somewhat, although this produces only a negligible increase in the resistance to crack growth. However, increasing the volume fraction of porosity due to the P.T.F.E. particles to 0·44, that is a volume fraction of P.T.F.E. on the silicon carbide of 0·55, caused the fracture behaviour to change such that extensive crack deflection occurred, as shown in Fig. 2(b).

The most straightforward approach to considering crack deflection at a porous interlayer is to simply treat the porous interlayer as a continuum whose fracture and elastic properties are equal to the values obtained from bulk samples, allowing existing models for crack deflection to be used- Most of these consider that crack detflection occurs when the driving force for the growth of the interfacial crack equals the fracture energy of the interface at a lower load than that at which the driving force of the penetrating crack is equal to the fracture energy of the matrix.[18,19]

However, it is clear that there are significant differences between the way in which the crack is observed to deflect and the situation that has been

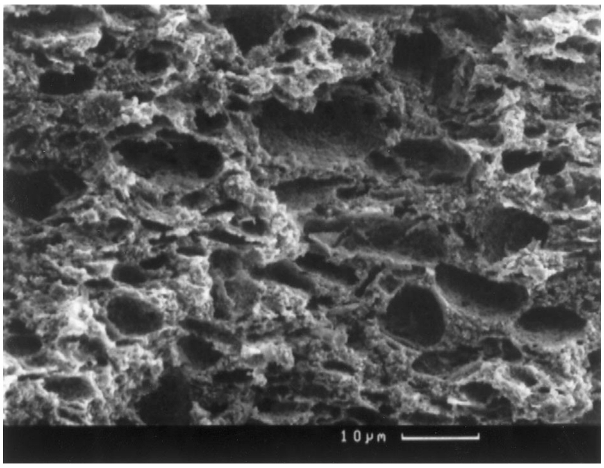


Fig. 1. Showing the fracture surface of a porous silicon carbide in which a volume fraction of porosity of 0·44 has been incorporated by the addition into the tape casting slurry of a volume fraction of P.T.F.E. particles on the silicon carbide of 0·55.

modelled. In particular, it is known from experimental observations, supported by theoretical calculations, that defects in an interface ahead of a growing crack significantly affect the interfacial properties required for crack deflection.[20,21] In the present case such defects exist in large numbers.

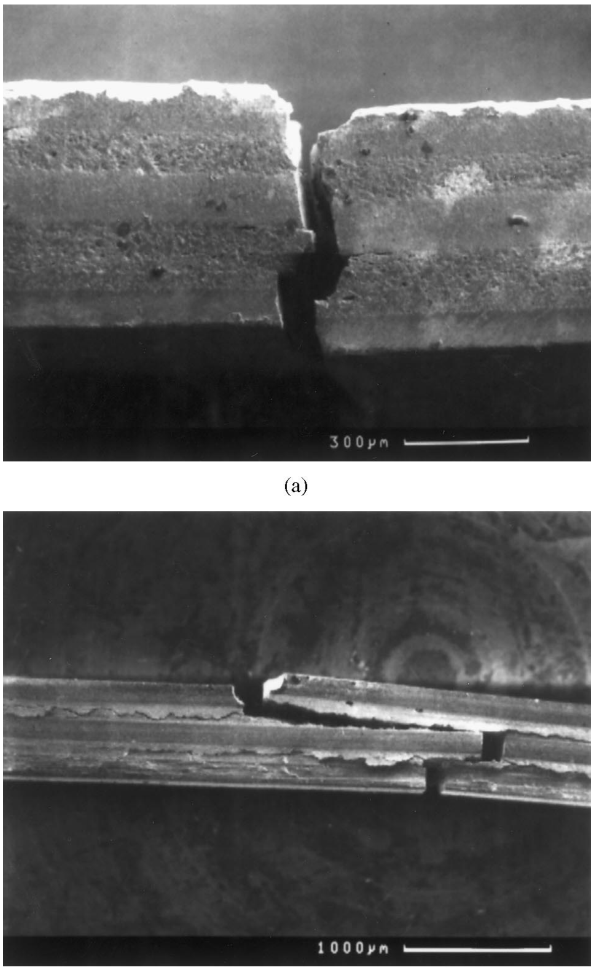
For a homogeneous interface with a fracture energy R_i, it has been shown that where a defect lies in an interface ahead of a crack growing through the lamina, the condition for continued crack deflection is[22]

$$\frac{R_i}{R_o} < 0{\cdot}57$$

where R_o is the fracture energy of the lamina. Here, the defect sits in an otherwise uniform interface whose fracture energy is lower than that of the matrix, In the experimental situation considered here, the defects sit in an interlayer, whose fracture energy, at least on the scale of the crack tip stress field, is equal to that of the matrix. One might therefore expect the crack to kink out of the interface immediately, as observed in the intertayer containing the lower volume fraction of pores. In this case ensuring that the crack travels in the interface means that the ligament of material between the, crack tip and the pore ahead of it in the interface must fracture, in other words

$$\frac{R_{lig}}{R_o} < 0{\cdot}57 \tag{1}$$

where R_{lig} is the fracture energy of the ligament of ceramic between the crack and the pore ahead of it, as shown in Fig. 3. If this is correct it is clear that the presence of the pores must somehow modify the fracture energy of the ligament. To examine this idea further, the fracture behaviour of porous solids, in monolithic form, is considered first.



(a)

(b)

Fig. 2. Showing the fracture path through a laminate made of silicon carbide laminae separated by porous silicon carbide layers containing a volume fraction of added porosity of (a) 0·34 and (b) 0·44. Note that although there is some change to the direction of the crack, there is no substantial crack deflection in the former whilst there is extensive crack deflection in the latter.

3.1 Fracture of porous solids

Clearly, increasing the porosity reduces the amount of material to be broken and hence the amount of new surface to be created, so that, if only a single crack is growing, one might expect the fracture energy to vary according to the area fraction of ceramic material in the crack plane, as given by

$$R_p = R_o(1 - A) \tag{2}$$

where R_p and R_o, are the fracture energies of the porous and dense material respectively and A is the area fraction of porosity in the crack plane. If the crack grows in some random plane then the area fraction of pores is equal to the volume fraction of pores, P, giving

$$R_p = R_o(1 - P) \tag{3}$$

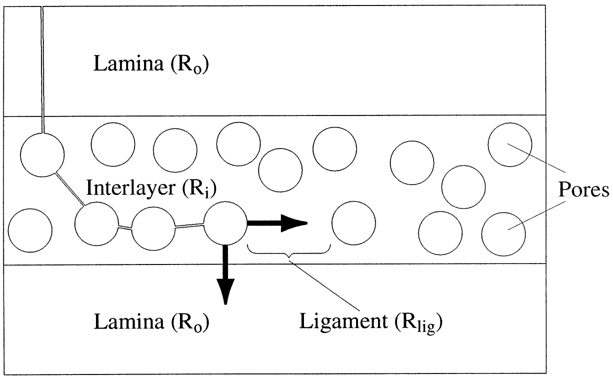


Fig. 3. Showing a schematic of a deflected crack lying in a porous interlayer.

An expression for the toughness, T, can be obtained from this using the expression

$$ER = T^2$$

and an appropriate expression for the Young modulus of the porous body, such as

$$E_{\mathrm{p}} = E_{\mathrm{o}}(1 - P)^2$$

where E_{p} and E_{o} are the Young moduli of the porous and dense materials respectively. This latter expression was chosen because it was found to describe well the Young moduli of the porous silicon carbide in this investigation. This gives the toughness of the porous body as

$$T_{\mathrm{p}} = T_{\mathrm{o}}(1 - P)^{3/2} \qquad (4)$$

where T_{p} and T_{o} are the fracture toughnesses of the porous and the ligament materials respectively, where the toughness of the ligament is set equal to that of the bulk material, T_{o}. An expression of the same form has also been derived for foams.[23]

However, the fracture energy decreases more rapidly with increasing porosity than is predicted assuming a random crack plane, as shown in Fig. 4, which shows data obtained from partially sintered alumina powders made from powder compacts having different green densities.[24] The squares represent values for samples whose green density was 0·50, whereas the circles represent samples which had a green density of 0·62. It is worth noting that the data for these samples with different green densities fall onto the same line, suggesting that the green density is unimportant, in contradiction with expressions that have been used elsewhere.[24]

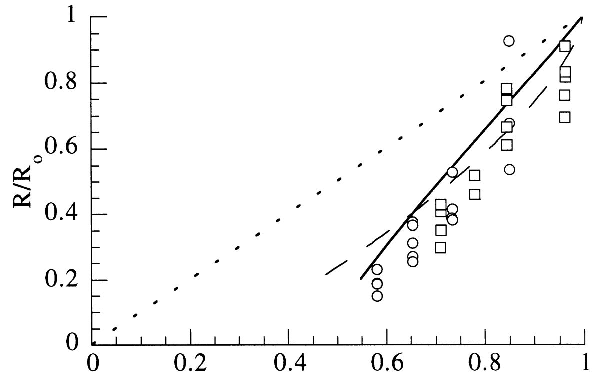


Fig. 4. Showing the relative fracture energy for samples of alumina made by partial sintering of green bodies with relative densities of 0·5 (open circles) and 0·62 (open squares).[24] Note that the rate at which the fracture energy decreases with porosity is greater than that given by eqn (3), which is represented by a dotted line. Also shown are the predicted variations in the fracture energy with porosity estimated by considering either the area of fracture (shown by a dashed line) or the increase in the stress intensity factor at the crack tip due to the presence of nearby pores (shown by a continuous line) from eqn (5) and eqn (7) respectively.

Rice[25] has suggested that the reason why the fracture energy tends to be lower than would be predicted by considering the area of fracture of a random crack plane through the sample, is that the crack seeks out pores, resulting in a fracture surface that contains a higher area fraction of pores. For an ordered array of pores, there is a simply quantified minimum in the solid area of material through which the crack may grow. It is known that the Young modulus can be related to this minimum solid area and it would therefore be expected that the fracture energy would have the same dependence on porosity. This minimum solid area and its variation with porosity has been calculated for various different ordered arrays. Except at densities very close to the green density, it is found that the minimum solid area can be related to the volume fraction of pores, P, by an expression of the form[25]

$$\frac{A}{A_{\mathrm{o}}} = \exp -bP$$

where A/A_{o}, is the fraction of the solid area compared with that when the body is fully dense and b is a constant which depends on the ordered array that is chosen. This expression breaks down close to the green density, where there is a very rapid change in the minimum solid area although there is only a minimal change in the relative density and it is for this reason that there is no apparent effect of the green density.

The porous materials being studied here consist of essentially equiaxed pores in an otherwise dense matrix. Following the approach described above, the situation could be represented by ordering the equiaxed (in this case spherical) pores and allowing the crack to grow through the plane containing the highest area fraction of pores, as shown in Fig. 5, which shows a planar crack growing through a simple cubic array of pores.

In this case, the fraction of porosity in the crack plane, A, is equal to the area of a pore divided by the area of the base of the pore unit cell (the shaded square in Fig. 5). This is the minimum solid area of the material,[26,27] and is given by

$$A = \frac{\pi c^2}{D^2}$$

where c is the pore radius and D is the spacing between pores. The porosity, P, of the material is simply equal to the volume of a pore divided by the volume of the unit cell in Fig. 5.

The relative density of the material, $(1 - P)$, can therefore be written as

$$(1 - P) = \left(1 - \frac{4\pi c^3}{3D^3}\right)$$

and gives the fracture energy of the porous body as

$$R_p = R_o\left(1 - \pi\left\{\frac{3P}{4\pi}\right\}^{2/3}\right) \tag{5}$$

This is plotted onto Fig. 4, and it can be seen that, despite the crudity of the approach, the agreement with the experimental data is not unreasonable.

However, once the pore radius exceeds 0·5 D the pores begin to overlap. The relative density at this point is 0·48. At densities lower than this, eqn (5) is no longer valid because the volumes where the pores overlap are not taken into account. However, inspection of Fig. 5 shows that the area fraction of material in the crack plane will not reach zero until the porosity is extremely high, that is, when c becomes equal to $D/\sqrt{2}$.

An alternative approach is to consider the effect of the pores on the driving force of a crack growing through the porous solid. A similar problem has been addressed quantitatively by Gong and Horii,[28] who calculated the increase in stress intensity factor at the tip of a crack as a microcrack was moved closer to it, as shown in Fig. 6(a). They showed that the ratio of the stress intensity factor at the crack tip, if the flaw were not present, K_o to that in the presence of the microcrack, K_{MA}, varies with the ratio of the distance of the flaw from the crack to the half-size of the flaw, d/c, as shown in Fig. 7. It can be seen that putting a flaw just in front of the crack tip approximately doubles the stress intensity factor at the crack tip, K_{MA}. This would be seen as a reduction in the toughness, and hence the fracture energy, of the ligament material. The toughness of the ligament material is now given by the expression

$$T_{lig} = T_o\left(\frac{K_o}{K_{MA}}\right) \tag{6}$$

The crack-flaw interaction can therefore be regarded as providing an extra driving force for the crack to propagate toward the flaw. This reduction in the toughness occurs even though the ligament is made of the same material as the dense matrix.

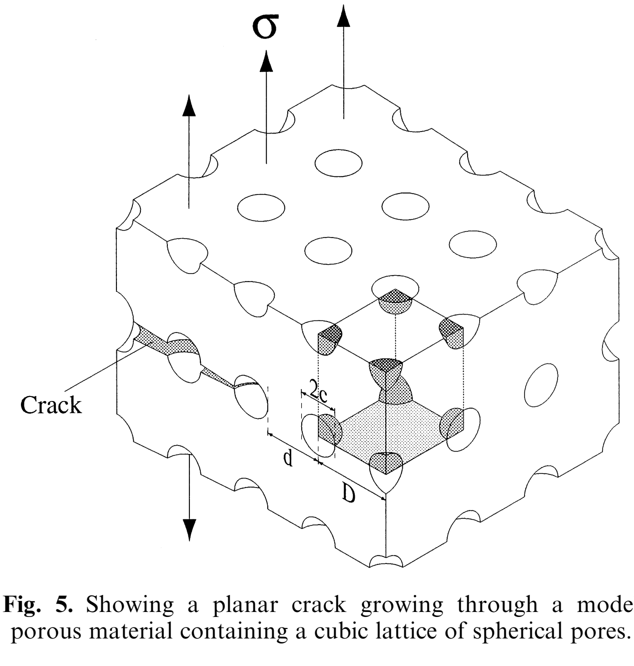


Fig. 5. Showing a planar crack growing through a model porous material containing a cubic lattice of spherical pores.

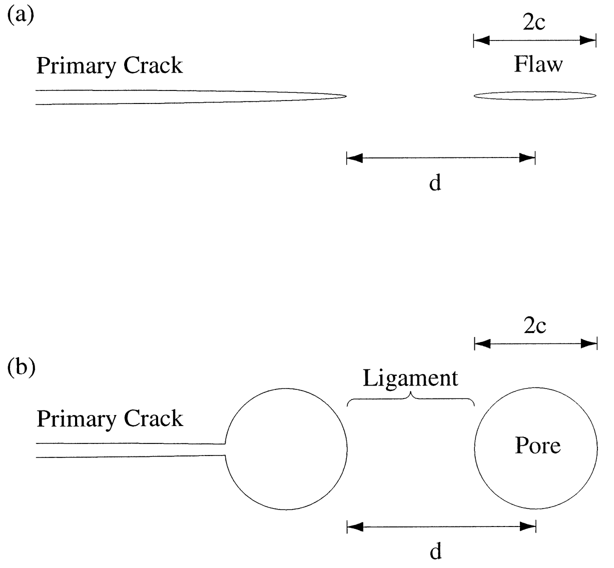


Fig. 6. (a) A short flaw lying ahead of a primary crack as considered by Gong and Horii.[28] When the flaw is close to the crack tip, that is d/c is close to unity, the presence of the flaw significantly increases the stress intensity factor at the tip of the primary crack. (b) The modification to the situation considered above, where the tip of the primary crack lies at a pore and another pore lies ahead.

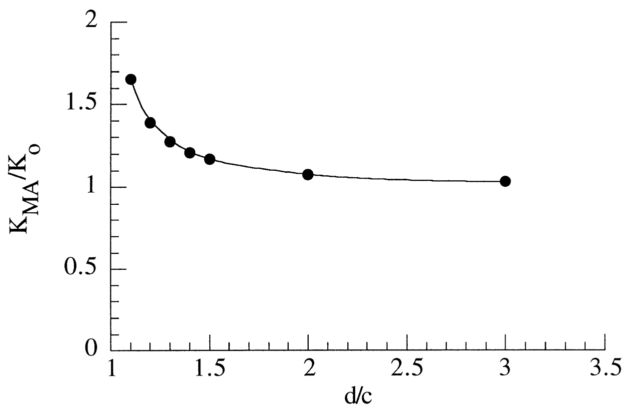


Fig. 7. Showing how the normalised stress-intensity factor at the main crack tip (K_{MA}/K_O) depends on the relative distance of the flaw ahead of the crack tip (d/c). The points plotted are obtained from Gong and Horii.[28]

This approach can be adapted if we consider the crack to grow from pore to pore as has been observed in porous silicon nitride, so that there is a pore at the end of the crack and another a distance d ahead of it, as shown in Fig. 6(b). The sharp crack tip and the microcrack are, therefore replaced by pores. Whilst this situation may appear to be very different to that considered by Gong and Horii. Alford *et al.*[29] have shown that the strengths of alumina and titania materials containing essentially pore-shaped flaws were accurately predicted by assuming that the pores were sharp penny-shaped cracks.

In eqn (4), which relates the toughness of the ligaments to the fracture toughness of the porous body, it was assumed that the appropriate value for the ligament toughness was that of the dense matrix material. That is where there is no interaction between the crack and the pores. However, as the pores become closer to the tip of the main crack the apparent toughness of the ligament is reduced as explained above, giving the toughness of the body as

$$T_\mathrm{p} = T_\mathrm{o}\left(\frac{K_\mathrm{o}}{K_\mathrm{MA}}\right)(1-P)^{3/2}$$

where $K_\mathrm{o}/K_\mathrm{MA}$ depends on the ratio of pore spacing to pore radius (d/c), as defined in Fig. 6(b). This gives the fracture energy of a porous body as

$$R_\mathrm{p} = R_\mathrm{o}\left(\frac{K_\mathrm{o}}{K_\mathrm{MA}}\right)^2(1-P) \tag{7}$$

The crack–pore interaction therefore reduces the fracture energy of the material, as might be expected. As the body with the pores arranged in a simple cubic array gave good agreement with the fracture energy, it is therefore assumed that this gives a good approximation to the interpore spacings seen by the crack as it fluctuates through the array of pores.

For the cubic array of spherical pores shown in Fig. 5 the volume fraction of pores, P, is related to the spacing and size of the pores by the expression

$$\frac{d}{c} = \left(\frac{4\pi}{3P}\right)^{1/3} - 1$$

This allows, the appropriate value of d/c for a given value of P to be estimated, which can in turn be used to determine the relevant values of $K_\mathrm{o}/K_\mathrm{MA}$ using Fig. 7. Substituting P and the appropriate value of $K_\mathrm{o}/K_\mathrm{MA}$ allows the fracture energy of the porous body, R_p, to be determined. A comparison of this approach with the other values is shown in Fig. 4, where it can be seen to give good agreement not only with the previous approach but also with the experimental values.

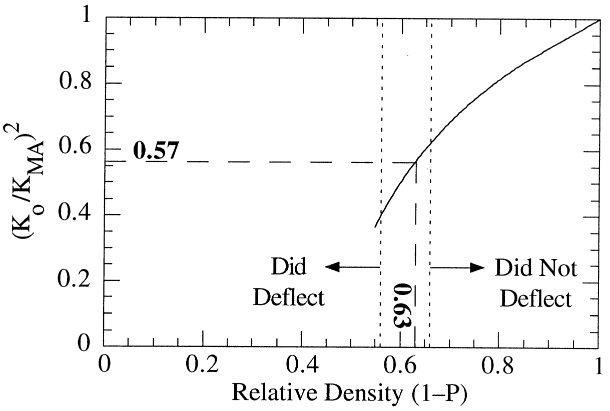


Fig. 8. Showing how the value of (K_{MA}/K_O varies with the relative density,ρ of the body, that is ($1-P$). Crack deflection is predicted to occur where $(K_O/K_{MA})^2$ is less than 0·57, equivalent to a porosity of 0·37, or a relative density of 0·63. Also shown is the relative density below which no crack deflection was observed (corresponding to $P = 0{\cdot}34$) and above which extensive deflection was obtained (corresponding to $P = 0{\cdot}44$). It can be seen that there is good agreement between the observations and the predictions.

3.2 Crack deflection at porous interlayers

For a crack to continue growing in the interface, as shown in Fig. 3, then there must some interaction between the growing (interfacial) crack and the pores ahead of it, which lowers the toughness, and hence the fracture energy of the ligament between the crack and the nearest interfacial pore.

The toughness of the ligament, T_lig, is given in eqn (6) and assuming that the modulus of the ligament material is not dependent on the direction in which the crack grows, the ratio of the fracture energies is given by

$$\frac{R_\mathrm{lig}}{R_\mathrm{o}} = \left(\frac{K_\mathrm{o}}{K_\mathrm{MA}}\right)^2$$

so, from eqn (1), crack deflection will occur if

$$\left(\frac{K_\mathrm{o}}{K_\mathrm{MA}}\right) < 0{\cdot}57$$

The corresponding value of d/c (expressed in terms of the volume of porosity, P) which will just allow deflection, can be obtained from Fig. 8. It is predicted that crack deflection will occur provided the porosity is greater than 0·37. This is within the range observed experimentally.

4 Conclusions

A simple model has been presented which predicts the fracture behaviour of porous solids. It is shown that this gives good agreement with materials which behave in a purely brittle manner. Furthermore it allows an understanding of why cracks are deflected at porous interfaces, quantitatively predicting the volume fraction of porosity required for deflection in reasonable agreement with experiment.

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