

## The effect of porosity in thermal shock

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**Abstract** The effects of porosity on cracking during thermal shock have been studied by directly observing the cracks that formed after quenching heated porous alumina bars into water. The porosity was introduced by adding different volume fractions of fugitive inclusions and the behaviour compared with that obtained by partial sintering of a powder compact. Where fugitive inclusions had been used, there was little effect of either pore size or pore volume fraction over the ranges studied. The extent of cracking was always slightly less than that of a monolithic, dense alumina and gave reasonable agreement with predictions using experimentally measured data. However, cracks grew much further in the partially sintered material. This discrepancy became greater as the temperature change increased, inconsistent with existing analyses. It is suggested that this difference in behaviour arises predominantly because of the greater measured fracture energy of the alumina made using fugitive inclusions compared with that made by partial sintering.

### Introduction

Why pores appear to improve the resistance to thermal shock is an old problem. It is generally thought this arises because porosity decreases the elastic modulus of the material and hence reduces the driving force for cracking. However, the evidence for such an improvement is by no means clear. Undoubtedly, the situation is complicated as the thermal shock resistance is dependent both upon the environment in which the material must operate, in particular on whether the material is being heated or cooled, and also on the application for which the material is being used.

In applications such as heat engines, it is critical that there is no, or only a minimal, decrease in the strength after thermal shock. In this case, it is the criteria which determine the onset of cracking that are important, which, in the case of a very rapid temperature change, will occur if the temperature change is greater than some critical value,  $\Delta T_c$ , given by

$$\Delta T_c = \left[ \frac{1}{\beta} \right] \left[ \frac{R}{Ec} \right]^{1/2} \quad (1)$$

where  $R$  is the fracture energy of the material,  $E$  the Young modulus,  $c$  the flaw-size and  $\beta$  a numerical constant. Generally, this situation appears to be reasonably well understood provided that account is also taken of the need to transfer heat across an interface with a finite thermal conductance. Experiments in porous materials show that it is only in the case of very high porosities, such as occur in foams, that improvements have been observed and this has been attributed to penetration of the foam structure by the quenching medium [1]. Lower porosities cause a reduction in the thermal shock resistance expressed in terms of  $\Delta T_c$  [2, 3].

Using Eq. 1, this would suggest that either the critical defect size is increased by the presence of the pores or that

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the fracture energy decreases with increasing porosity more rapidly than does the elastic modulus. There have been many measurements of the change in elastic modulus and of fracture energy with porosity and although it is claimed that they change at the same rate [4] the observations overall do not support this view [5, 6].

In certain applications, such as the handling of liquid metals and glasses, some cracking can be tolerated and it is more important to limit the extent of cracking. This has normally been determined from measurements of the residual strength after the heated body has been quenched, rather than by direct observation of the cracks that have formed. In this case, there is much less consistency between the data. In some cases, it is found that the reduction that takes place, as a fraction of the original strength, becomes greater as the porosity increases [3], whilst in others the reduction becomes less with increasing porosity [7, 8]. It is also often observed that the absolute magnitude of the residual strength is similar for different porosities [7, 8].

Although analyses exist for the case where the temperature through the body remains uniform during the temperature change [9], it is more common, both in the laboratory and in service, for cracking to be caused by gradients of temperature within the body. In this case, the extent of cracking may only be determined by more complex calculations, where the change in temperature and hence the crack driving force, throughout the body with time is calculated [10, 11]. These analyses show that, at a given time after the temperature change has occurred, the crack driving force initially increases with crack length and then decreases. For a given length of crack, the crack driving force also initially increases with time as the thermal front moves into the material and then decreases as the temperature in the body becomes more uniform. Using this approach, a defect of a given length is assumed to start growing when the crack driving force (for a defect of that length) becomes equal to the fracture energy and that this will be followed by a burst of crack growth to the point where, again the crack driving force is equal to the fracture energy. The crack will then grow more slowly into the sample as the thermal stresses change within the body, until some maximum crack length is reached which is the furthest point in the body where the crack driving force becomes equal to the fracture energy [11, 12].

In this article, the effect of porosity on the resistance to thermal shock, in terms of the extent of crack growth, has been studied, where the extent of cracking that occurs on quenching a heated body into water at room temperature has been characterised by observation, thus allowing direct comparison with quantitative predictions.

## Experimental procedures

### Sample fabrication

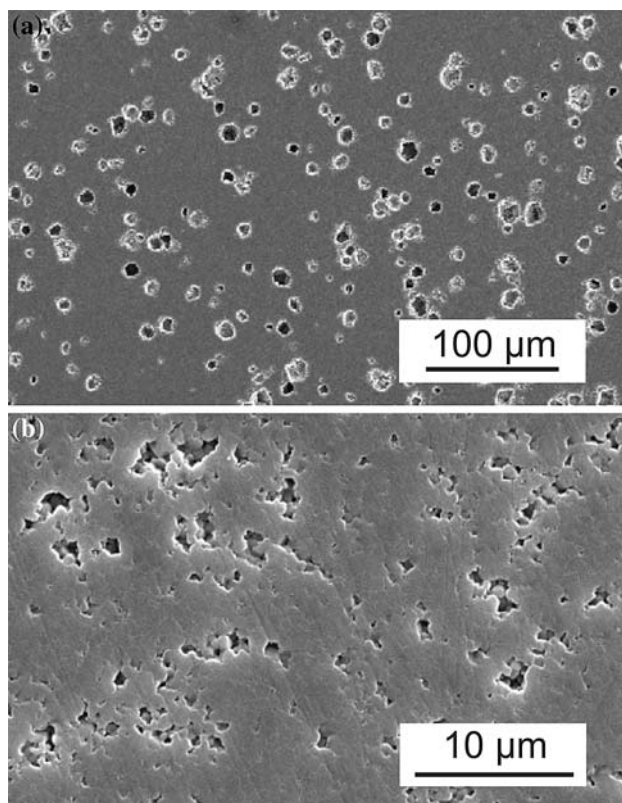
Samples were made by slip casting. Suitable slurries were made by mixing alumina powder (Sumitomo, AKP-30) with distilled water using polyacrylic acid (Rohm and Haas, D3021) as a dispersing agent at a concentration of 0.3% by weight of the solids, followed by ball milling for 24 h in a polyethylene jar using  $\text{Si}_3\text{N}_4$  milling media. The resulting slurries were poured into metal moulds on a disc of Plaster of Paris to remove the water. The discs were then dried at 80 °C for 12 h. Fully dense samples were made by heating the alumina compacts at a rate of 5 °C per minute to 1,550 °C and holding them at this temperature for 1 h.

Porosity was introduced by adding the appropriate volume amount of particles of starch (Sigma, UK) to the slurry shortly before the slurry was poured, to give a volume fraction of pores in the final body corresponding to 0.1, 0.2 and 0.3. Most of the experiments were carried out using corn starch with an average diameter of 10  $\mu\text{m}$ . To study the influence of pore size, some experiments were also performed using potato starch, with an average size of 40  $\mu\text{m}$  and rice starch, with an average size of 5  $\mu\text{m}$ . The resulting samples were then heated at a rate of 1 °C per minute to 500 °C to burn away the starch and then heated at 5 °C per minute to 1,550 °C for 1 h, giving a matrix material with a grain size of 3  $\mu\text{m}$  containing a random distribution of pores due to the added starch particles, as shown in Fig. 1a.

Some porous materials were also made by sintering the alumina powder compacts (containing no starch particles) at lower temperatures to give bodies which were not sintered to full density. The resulting structure of a body sintered at 1,350 °C, to give a porosity of 0.1 is shown in Fig. 1b. It can be seen that the pores are less regularly shaped and also appear to be less uniformly distributed.

### Thermal shock testing

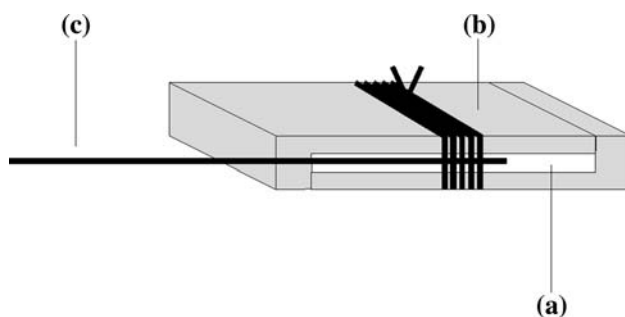
Thermal shock testing was carried out using a technique originally developed by Davidge and Tappin [13] and modified by Bahr et al. [11] in which samples were thermally insulated using a pyrophilite material on all sides except one, as shown in Fig. 2. The dimensions of the plates were approximately 40 mm  $\times$  10 mm  $\times$  2 mm. The sample, encased in thermal insulation, was slowly heated in a tube furnace in air to a pre-set temperature and held at that temperature for 20 min to ensure that the temperature was uniform throughout the sample. It was then quenched in water at room temperature. The temperature difference through which the sample was quenched,  $\Delta T$ , was



**Fig. 1** Typical structures for materials containing porosity introduced by either (a) adding starch particles to the slip or (b) partial sintering at 1,350 °C. Both samples contain a volume fraction of porosity of 0.1

determined by measuring the initial temperature of the sample with a thermocouple close to the sample, and the initial temperature of the water with a standard thermometer. In this test, cracks are initiated at the cooled face and grow into the interior of the sample. The cracking patterns that form during the thermal shock can then be observed directly by removing the insulation.

To study the cracks that had formed during thermal shock more clearly, the surfaces of the samples were impregnated



**Fig. 2** A schematic representation of a sample prepared for testing. The sample, (a), is surrounded by insulation, (b), before preheating and quenching into water. The thermocouple, (c), is used to measure the surface temperature of the sample

with a penetrating dye (Johnson & Allen Ltd., UK, Neopen JAP penetrant). The surfaces were then digitally scanned to give a permanent record of the sample surface. Measurements of the number of cracks that had grown and the maximum extent of crack growth were made by observing the surface traces of the cracks using both optical and scanning electron microscopy.

#### Estimating the extent of crack growth

To see whether these observations were what might be expected, estimates were made of the extent of crack growth. First, the temperature distribution within the body at any given time was calculated using a finite difference method. The stress distribution within the body was then estimated from the temperature distribution. The resulting crack driving force can then be calculated using a Green's function, as explained by Evans and Charles [10] and Fett and Munz [14]. It was assumed that the presence of the cracks that had formed did not influence the temperature distribution in the body. Further growth of the crack into the sample was assumed to occur if the crack driving force for the overall penetrating crack was greater than the measured fracture energy. The details are given elsewhere [15]. This approach has given a good description of cracking in a range of materials including silicon nitrides [16] and layered structures of various types [15].

#### Thermal and mechanical properties

The mechanical and thermal properties of the different porous materials were measured to provide the material data for making the estimates of the extent of crack growth. The Young modulus,  $E$ , was determined using an impulse excitation technique (J. W. Lemmens, Grindosonic Mk. 5). The fracture toughness,  $K_{1C}$ , was determined using single edge notch bend samples, using samples with a span of 40 mm and a cross-section of  $3 \times 3$  mm and an initial crack length of approximately one quarter of the overall sample thickness.

The thermal diffusivity,  $\lambda$ , was measured using a laser flash technique using polished discs with a diameter of 10 mm and a thickness of 2 mm. The surface of the discs was polished to a finish of  $0.25 \mu\text{m}$  and coated with colloidal graphite in order to give more complete absorption of the laser light. All the measurements were made under vacuum.

The thermal diffusivity of all samples was first measured at room temperature. High temperature measurements up to 1,000 °C were also carried out on the fully sintered samples without starch and with 30% corn starch. The high

temperature results have been corrected for thermal expansion using an expansion coefficient of  $8 \times 10^{-6} \text{ K}^{-1}$ . The thermal conductivity,  $\kappa$ , was obtained from the thermal diffusivity,  $\lambda$ , using the expression

$$\kappa(T) = \rho(T) c_p(T) \lambda(T)$$

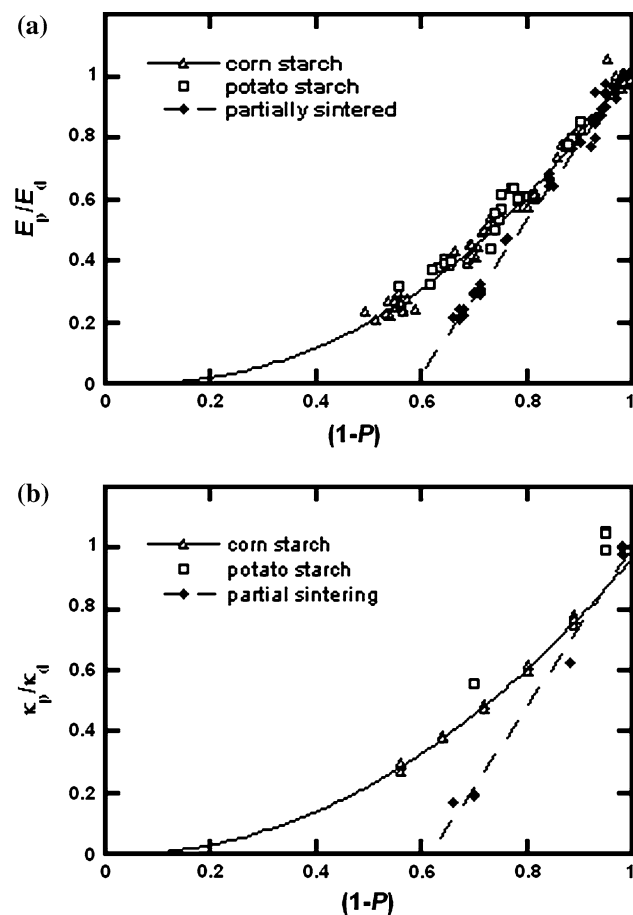
where  $\rho$  is the density and  $c_p$  is the specific heat.

The interfacial conductance was taken as  $20 \text{ kW m}^{-2}$ , as measured by Lee et al. [17], and was assumed to remain unchanged with increasing porosity.

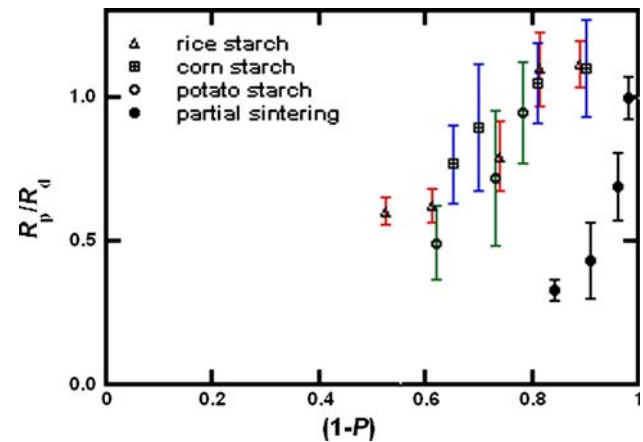
## Results

### Thermal and mechanical behaviour

The variation in the Young modulus and thermal conductivity is shown in Fig. 3. These are similar to those observed elsewhere and will not be discussed in detail here. It is worth noting that there is a slight difference at higher

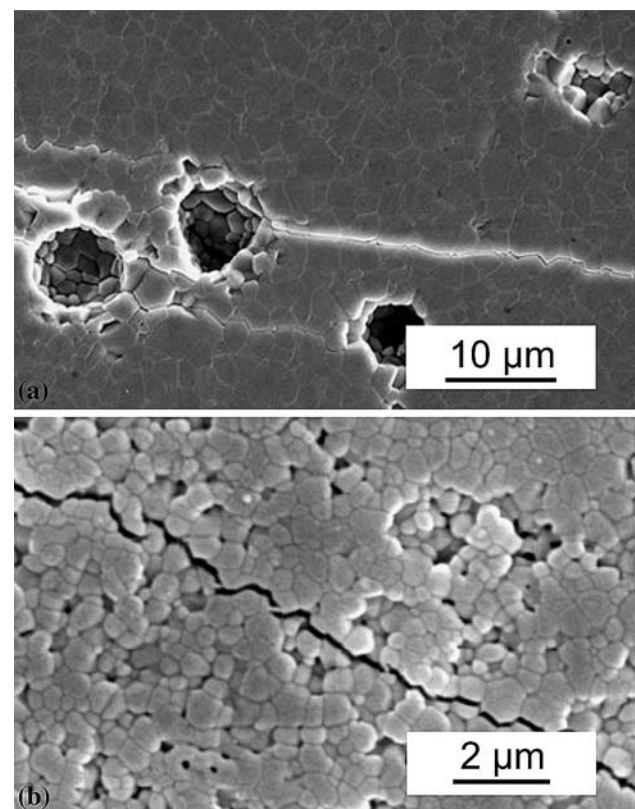


**Fig. 3** The effect of the volume fraction of pores,  $P$ , on the (a) Young modulus,  $E_p$ , normalised by the Young modulus of dense alumina,  $E_d$ , and on (b) the thermal conductivity,  $\kappa_p$ , normalised by the thermal conductivity of dense alumina,  $\kappa_d$ , for both types of porosity



**Fig. 4** The effect of the pore volume fraction,  $P$ , on the ratio of the fracture energy of the porous material,  $R_p$ , and the fracture energy of dense alumina,  $R_d$ , for both types of porosity. Note that the fracture energy of the partially sintered material decreases in a manner similar to the Young modulus in the previous figure, whereas the fracture energy for the material made by adding starch particles is significantly greater

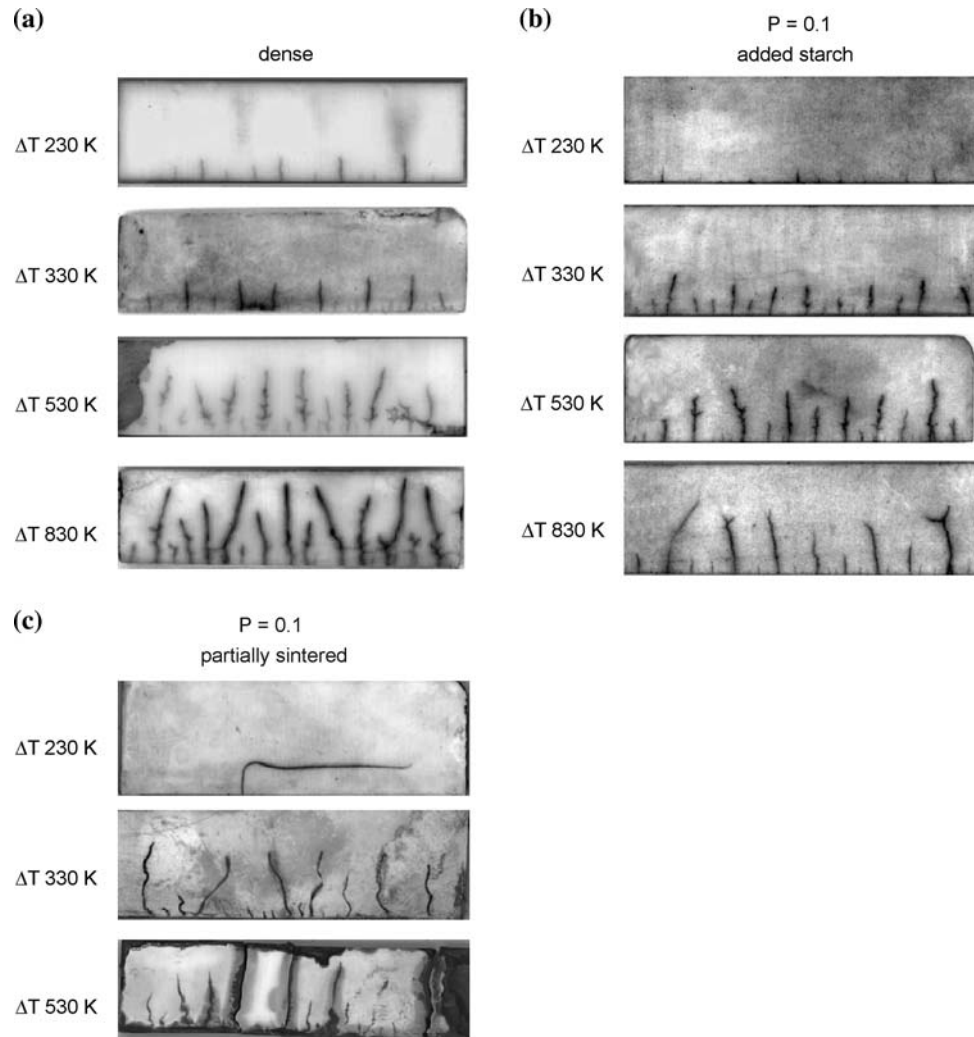
porosities between the porous materials made by adding starch and those made by partial sintering, consistent with earlier studies. This is thought to arise due to the presence



**Fig. 5** Showing the cracking behaviour during the mechanical loading of the material made by (a) adding starch particles and (b) partial sintering. Both samples contain a volume fraction of pores of 0.1. Note the crack branching that occurs in the material made by adding starch particles, which does not occur in the partially sintered materials



**Fig. 6** The cracking patterns observed after thermal shock through 230, 330, 530 and 830 K, in (a) dense alumina, in (b) alumina made by adding starch particles to the slip and in (c) alumina made by partial sintering. All the porous materials contain a volume fraction of pores of 0.1



of necked regions between the alumina particles in the partially sintered materials, as opposed to the structure of separate pores embedded in a matrix where starch particles are used to introduce the porosity [18].

However, there are significant differences in the measured fracture energies between the two structures, as shown in Fig. 4. The partially sintered material shows a decrease in fracture energy similar to that observed in the elastic modulus, although at a slightly greater rate, consistent with work in similar systems [19]. In the material where the pores were incorporated by adding starch, however, it is found that the fracture energy rises slightly as the porosity increases reaching a maximum slightly greater than that of the fully dense material at a porosity of approximately 0.2. The fracture energy then decreases, but is always significantly greater than that of the partially sintered material at the same porosity. Similar observations have been made previously and have been associated with microcracking ahead of the main crack [6]. No evidence for the formation of microcracks was found in this

material, although there was some evidence of crack branching not observed in the partially sintered materials, as shown in Fig. 5.

#### Thermal shock behaviour

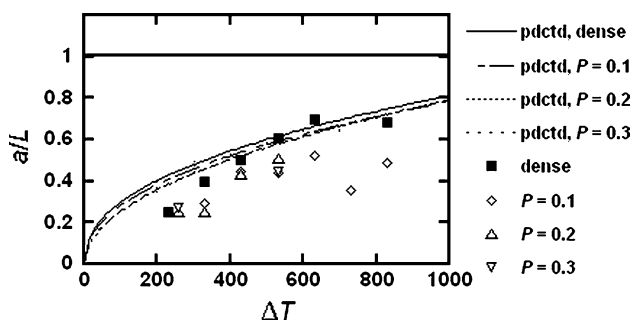
The cracking patterns that formed in the different materials are shown in Fig. 6 for different types of the porosity and for increasing values of temperature change,  $\Delta T$ , comparing materials which are either dense or had a pore volume fraction of 0.1. In the fully dense material, cracking was observed when  $\Delta T$  became greater than 230 K. This was also the case in material containing a volume fraction of pores of 0.1 but increased slightly to 240 and 260 K for porosities of 0.2 and 0.3, respectively. At the lower temperatures relatively few cracks were formed. In the partially sintered material, where only one crack had initiated, this arrests and then grows laterally. As  $\Delta T$  is increased more cracks grow. However, at the highest

values of  $\Delta T$ , there was also a tendency for lateral growth of cracks to occur. Whilst some distinct lateral cracking can be observed, it was more common for this type of behaviour to occur either in the form of a crack oscillating on either side of some direction or as branching, giving rise to a ‘fir-tree’ effect, where smaller cracks appeared to have grown laterally off the main crack. Both effects can be seen in Fig. 6. The cracks also tended to grow deeper as  $\Delta T$  increases.

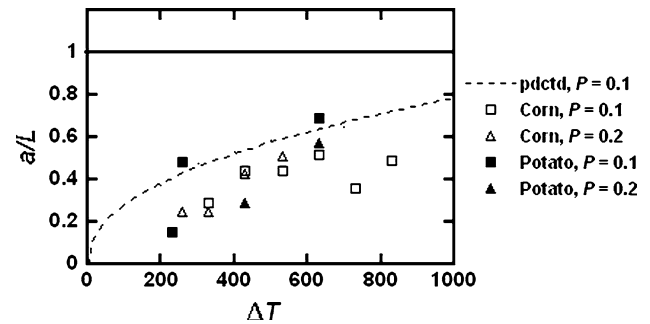
Another feature that can be clearly seen is that the cracks do not all have similar lengths, rather there appear to be some very short cracks and some much longer ones. This effect is widely observed in thermal shock and is caused by competition for the available driving force between the cracks [20]. This leads to a surprisingly well-defined spacing between the longer cracks which does not appear to be substantially influenced by the quenching temperature. The crack spacing observed in the partially sintered material is somewhat greater, although the cracks do not appear to be as evenly spaced.

The residual properties are likely to be determined by the longer cracks and it is the change in the average length of these longer cracks with increasing  $\Delta T$ , rather than the overall average, that has been plotted in Fig. 7 for materials containing different volume fractions of pores. These measurements show that different levels of porosity, introduced by adding starch particles to the slip, have relatively little effect on the extent of cracking. The crack patterns were also similar.

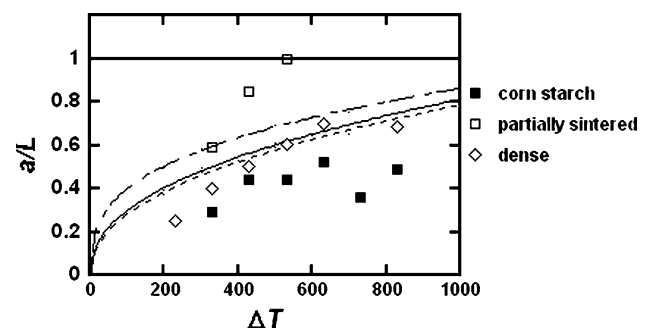
Varying the pore size between 10  $\mu\text{m}$  up to 40  $\mu\text{m}$  also appeared to have relatively little effect, as can be seen in Fig. 8, which shows the average length of the longer cracks in samples where the pore size was either 10 or 40  $\mu\text{m}$  at pore volume fractions of 0.1 or 0.2. There do appear to be some differences, in particular the material made with the potato starch, that is with 40  $\mu\text{m}$  pores at a porosity of 0.1, was less thermally shock resistant than the other materials, although the same pore size at a higher porosity



**Fig. 7** The ratio of final crack length,  $a$ , over sample height,  $L$ , observed in alumina containing different volume fractions of pores, made by adding starch particles as a function of the quench temperature difference,  $\Delta T$ , and comparison with the predictions



**Fig. 8** The ratio of final crack length,  $a$ , over sample height,  $L$ , as a function of the quench temperature difference,  $\Delta T$  observed in alumina containing pores of either 10  $\mu\text{m}$  (corn starch) or 40  $\mu\text{m}$  (potato starch), at porosities of 0.1 and 0.2. The dashed line shows the predicted crack lengths for a pore volume fraction of 0.1



**Fig. 9** The effect of the quench temperature difference,  $\Delta T$ , on the average length of the longer cracks observed in the different samples compared with predicted values. The solid line represents the prediction for the dense material, the line slightly below it, that for the starch containing materials and that above for the partially sintered materials. The volume fraction of porosity is 0.1

was much more consistent with the overall results and all showed the same general trends.

Much greater differences were found between the different types of porosity, see Fig. 9. In the partially sintered material containing a porosity of 0.1, cracking was observed to begin at a  $\Delta T$  of 235 K, similar to the materials above containing lower volume fractions of porosity. However, as  $\Delta T$  was increased, it became clear that the material was much less resistant to cracking than for the case of separated pores in a matrix at the same porosity. Indeed once the  $\Delta T$  reached 530 K the specimen had completely broken. In the case of the dense material, the crack had penetrated approximately 0.6 of the way through the body, but only 0.4 of the section was fractured for the materials made using starch particles.

## Discussion

The extent of crack growth in the different materials was estimated using the measured property data given in

Table 1 and are shown as lines in Figs. 7–9. The measured properties were not found to depend on pore size. Hence, the calculations predict the same maximum crack penetration depth irrespective of pore size. As shown in Fig. 8, this is essentially consistent with the experimental observations. It is noticeable that in all the porous materials made using starch, that the predicted crack lengths are somewhat greater than those observed, with the possible exception of the material made using potato starch and containing a porosity of 0.1. This might be expected as the analysis used considers only a single crack, whereas it is known that the available driving force is diminished in the case of an array of cracks [20], although the effect is not as great as might be expected due to the gradual reduction in the numbers of cracks as they grow into the sample. This view is consistent also with the observation that the rate of change of crack length with  $\Delta T$  is similar to that predicted, see Figs. 7–9.

It can be seen from Fig. 7 that for the starch materials the extent of cracking experimentally measured was only weakly dependent on the volume fraction of pores, at least up to a volume fraction of 0.3. Again this is consistent with the predictions using measured properties, which also show a negligible influence of pore volume fraction.

However, cracks grow much further in the partially sintered material compared to the starch material, see Fig. 9, even though at a pore volume fraction of 0.1, both have the same Young modulus and thermal conductivity, see Fig 3. The predominant material variable must therefore be the measured fracture energy.

These differences are clearly shown in Figs. 3 and 4. In the partially sintered material, the measured fracture energy falls rapidly with pore volume fraction, at least as rapidly as the Young modulus. In the starch materials, however, the measured fracture energy remains remarkably constant, at least up to pore volume fractions of 0.3. The differences in overall thermal shock behaviour must therefore be caused by these differences in fracture energy, or at least the ratio of the fracture energy to the Young

modulus, consistent with the observation by Rice [5] that many thermally shock resistant materials had pore volume fractions of approximately 0.1.

Work elsewhere [21] has shown that the measured fracture energy of materials containing pores was greater than might be expected from that given by the amount of material that had fractured. Indeed for some pore volume fractions the measured values of the fracture energy were slightly greater than the fully dense material. Following direct observations of cracks growing around pores it has been shown that this effect can arise because cracks must spread out around pores and do not simply cut through them as might be expected [21]. The effect of bowing around the pores is to cause the crack front to curve so that the work required to drive the crack through a porous material is increased.

It has been suggested elsewhere that fracture in partially sintered materials occurs by the fracture of interparticle contacts and that analyses of such behaviour would give rise to the behaviour that is observed in the partially sintered materials here [18], although observations in sintered silicon nitride powders [22] show that this cannot be universally true. Moreover, the situation in partially sintered materials can be complicated by the effects of grain size as grain growth and densification occur together, especially in the later stages of sintering [23]. As yet the important structural factors are not understood. However, the differences in thermal shock behaviour are entirely consistent with the differences in measured fracture energy, confirming that these differences in measured fracture energy are both real and significant.

However, this does not explain why the cracks in the partially sintered materials grow further than predicted, see Fig. 9. It should be noted that the analysis used here has been successfully used in a wide range of materials, including layered structures, with and without internal stresses [15], and silicon nitrides with varying pore and grain structures [16]. It has been suggested that kinetic effects might explain this behaviour [11]. However, such

**Table 1** The values of mechanical and thermal properties used in calculating the extent of cracking

		Dense	Partially sintered	Porous alumina made by adding starch		
Pore volume fraction	$P$	0.02	0.1	0.1	0.2	0.3
Density	$\rho$ , kg m <sup>-3</sup>	3900	3600	3600	3190	2850
Thermal diffusivity	$\lambda$ , W m <sup>-1</sup> K <sup>-1</sup>	32.3	25.3	25.3	19.9	15.8
Heat capacity	$c_p$ , J kg <sup>-1</sup> K <sup>-1</sup>	777	777	777	777	777
Interfacial conductance	$H$ , W m <sup>-2</sup> K <sup>-1</sup>	20000	20000	20000	20000	20000
Coefficient of thermal expansion	$\alpha \times 10^{-6}$ K <sup>-1</sup>	8.0	8.0	8.0	8.0	8.0
Young modulus	$E$ , GPa	396	325	325	243	180
Poisson ratio	$\nu$	0.2	0.2	0.2	0.2	0.2
Fracture energy	$R$ (J m <sup>-2</sup> )	39	17	43	41	35

analyses predict that such effects should be greatest at a temperature just above that required for crack growth. This is the reverse of what is seen here where the discrepancy increases as the temperature change,  $\Delta T$ , is increased.

An alternative view is that this is caused by the cooler fluid penetrating into the growing crack. The effect of cooling the crack surfaces acts in itself as a driving force for crack growth and this additional driving force can be expected to increase with  $\Delta T$  as the localised cooling induced by the fluid will increase. This only affects cracking in the material with the lowest fracture energy, suggesting that the additional driving force is relatively small.

Although the extent of cracking in the starch materials is only weakly dependent on the volume fraction of pores (over the range of volume fractions tested here of 0–0.3), the onset of cracking is influenced by the ratio of  $R/E$  as given in Eq. 1. One might therefore expect materials with higher pore volume fractions to show a greater resistance to the onset of cracking, consistent with the increase in the value of  $\Delta T_c$  with increasing pore volume fraction. However, increasing the porosity is often associated with an increase in the defect size [16] and this may be why the increase is not as great as might be expected from Eq. 1. There is no need for this to occur, although it often does in practice. The reasons for the confusion in the literature are therefore due, not only to differences in the morphology of the pores, but also in whether one is considering the onset or the extent of cracking, as might be expected.

## Conclusions

The aim of this article was to investigate how porosity influences cracking in thermal shock. To do this experiments have been carried out in which the extent of crack growth after thermal shock could be directly observed in porous aluminas. The pores were introduced either by adding starch particles to an aqueous dispersion of alumina or by partial sintering of a powder compact. The former enabled a range of pore volume fractions from 0 to 0.3 as well as a range of different sizes, from 10 to 40  $\mu\text{m}$  to be studied. Partial sintering was used to obtain a pore volume fraction of 0.1.

It was found that the extent of cracking in the porous aluminas made using starch particles were consistent with predictions, were independent of pore size and somewhat

better than the dense monolithic alumina. Compared with the dense material, cracks extended much further in the partially sintered alumina. The Young modulus and thermal conductivities of both the starch and partially sintered materials were similar at the same pore volume fractions. However, the measured fracture energy was much greater in the starch material than it was in the partially sintered material, showing that the differences in fracture energy are real and are not associated with measurement difficulties. Although explanations exist for the interactions between cracks and pores that give rise to the measured fracture energy being greater than expected, the significant differences between the two types of porosity are still unclear.

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