

Thermal shock resistance of yttria-stabilized zirconia with Palmqvist indentation cracks

G. Fargas, D. Casellas, L. Llanes, M. Anglada*

Departament de Ciència de Materials i Enginyeria Metallúrgica, Universitat Politècnica de Catalunya, E.T.S.E.I.B. Avda. Diagonal, 647, 08028 Barcelona, Spain

Received 20 September 2001; received in revised form 8 March 2002; accepted 24 March 2002

Abstract

Thermal shock behaviour of two tetragonal zirconia polycrystals stabilised with 2.5% molar yttria and with different fracture toughness have been investigated by analysing stable crack extension of indentation Palmqvist cracks in the quench indentation test. It is shown that the ratio between the thermal stress intensity factor and the fracture toughness can be easily obtained by measuring the stable crack extension. It is shown that deviations from the expected maximum stable crack extension during thermal shock can be accounted for by subcritical crack growth and by a reduction in the level of residual stresses. For small indentation loads, R-curve effects become important and must be considered to explain the experimental results. © 2002 Elsevier Science Ltd. All rights reserved.

Keywords: Indentation; Palmqvist cracks; Thermal shock resistance; ZrO₂

1. Introduction

Ceramic materials have excellent properties such as high hardness and resistance to oxidation, corrosion and creep at elevated temperatures. All these properties are needed in the development of devices for energy conversion and much progress has been made in these areas over the past two decades. However, the characteristic low toughness of monolithic ceramic materials has hampered their wide spread utilisation. One of the most effective methods to increase fracture toughness in monolithic ceramics is by transformation toughening.¹ Thus, the incorporation of metastable retained tetragonal zirconia in numerous compatible matrices, e.g. cubic zirconia in PSZ (partially stabilised zirconia) or alumina in ZTA (zirconia toughened alumina) leads to substantial enhancement in strength and toughness. The source of toughening lies on the stress induced transformation of tetragonal zirconia into the monoclinic polymorph that is accompanied by a volume increase of about 4%. Several factors, such as changes in free

energy, particle size and strain energy, affect the transformation of the constrained tetragonal particles.^{2,3}

Transformation toughening is also the main toughening mechanism in Y-TZP (yttria-stabilised tetragonal zirconia polycrystals), whose microstructure is formed by large volume fractions of very small metastable tetragonal grains as well as by relatively low proportions of cubic and monoclinic grains. In these materials, the exact fraction of each phase depends on the amount of oxide stabiliser, grain size, sintering temperature and time, cooling rate, etc.

Ceramics are also prone to fracture by thermal shock. If the surface of a ceramic material is subjected to a sudden decrease in temperature, the thermal stresses that are generated may be large enough to reach the fracture strength. For an infinite plate, the maximum stress reached at the surface, σ_{\max} , after a sudden change in temperature, ΔT , is given by,

$$\sigma_{\max} = \frac{E\alpha\Delta T}{1-\nu} F\left(\frac{hr}{k}\right) \quad (1)$$

where α is the thermal expansion coefficient, ν the Poisson coefficient, h the heat transfer coefficient, k the thermal conductivity, r is a specimen dimension and F is

* Corresponding author. Tel./fax: +34-93-401-6706.

a function of the Biot modulus, β , that is defined as hr/k . Some of the parameters related to quenching conditions such as, for instance, thermal gradient, heat transfer coefficient of the media, etc. are not often very well known and especially difficult to evaluate from the experiment. Therefore, the evaluation of thermal stresses from a theoretical standpoint is not a simple task.⁴

One of the most used parameters to evaluate thermal shock resistance of ceramics was introduced by Hasselman^{5,6} and it is defined as:

$$R''' = \frac{G_{Ic}E}{(1-\nu)\sigma_f^2} = \left(\frac{K_{Ic}}{\sigma_f}\right)^2 (1+\nu) \quad (2)$$

where G_{Ic} is the critical strain energy release rate, K_{Ic} the fracture toughness, and σ_f is the fracture strength of the material. However, in a quenching test fracture takes place by the extension of natural small cracks either by unstable catastrophic fracture or by a stable manner. In the latter, the material suffers some degradation of its load bearing capacity that is usually measured by means of the drop on σ_f in a bending test. This is usually plotted as a function of the temperature difference, ΔT , over which the sample is quenched and the critical temperature difference, ΔT_c , is defined as that at which the material shows a drastic drop in the remaining mechanical strength.

Because of the wide statistical distribution in dimensions and locations of natural cracks, there is a pronounced scatter in σ_f , so that a large number of specimens are needed to determine ΔT_c . In view of this, it has been found helpful to measure thermal shock in samples with relatively small cracks produced by indentation. Either because of the residual stress field produced by indentation or by the existence of microstructural barriers to crack extension, microcracks may increase in size during thermal shock and stop before reaching unstable conditions. Tancret and Osterstock⁷ and Tancret et al.⁸ used this indentation-quench technique on brittle materials and proposed a thermal shock parameter that is given by the ratio between K_{Ic} and thermal stress, σ_{th} :

$$R_m = \left(\frac{K_{Ic}}{\sigma_{th}}\right)^2 \quad (3)$$

The advantage is that for an indentation crack with indentation residual stress, this ratio can be calculated from the extension of radial cracks during thermal shock. In the limit, when the maximum thermal stress reaches σ_f , there is complete failure of the testing piece, and the value of $(1+\nu)R_m$ tends to R''' . More recently, Collin and Rowcliffe⁹ showed that this method could be used to explore susceptibility to thermal fracture in a range of brittle materials.

In this work, the thermal shock resistance of two zirconias stabilised with 2.5% molar yttria, but with a different microstructure and K_{Ic} is studied. The stable extension of Palmqvist cracks that takes place during the indentation quench test is discussed and related to the residual stresses and fracture toughness of each microstructure.

2. Thermal shock in specimens with Palmqvist cracks produced by a Vickers indenter

Some tough ceramic materials, such as cemented carbides and transformable zirconia ceramics may develop Palmqvist indentation cracks, whose geometry is shown in Fig. 1. The application of a load, P , by means of an indenter produces a displacement of matter at the contact of the specimen surface with the indenter and leaves residual stresses when the load is removed. These are responsible for the extension of the cracks during unloading. Their final length depends on the applied load, the K_{Ic} and other material characteristics such as hardness, H , and elastic modulus, E (see, for example, Lawn¹⁰). For semicircular Palmqvist surface cracks, Niihara et al.¹¹ proposed an expression for the stress intensity factor induced by this residual stress field, K_{res} , which can be written in terms of the semidiagonal of the imprint, d , and the crack diameter, l , as

$$K_{res} = \chi_0 \left(\frac{P}{d\sqrt{l}}\right) \quad (4)$$

where χ_0 is a material constant given by:

$$\chi_0 = C \left(\frac{E}{H}\right)^{2/5} \quad (5)$$

being C a calibration constant. If the indenter is applied under inert conditions, the length of the crack measured, l_{inert} , can be used in Eq. (4) for determining K_{Ic} , that is,

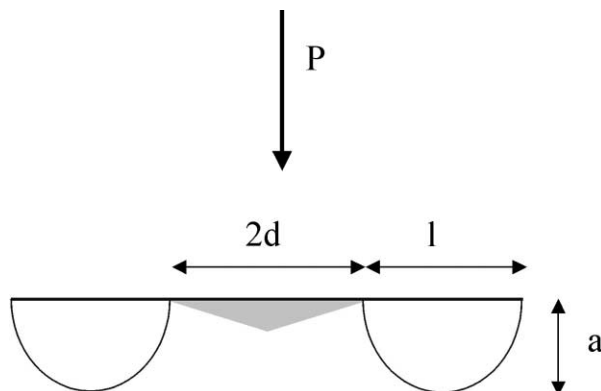


Fig. 1. Indentation Palmqvist cracks.

$$K_{Ic} = \chi_0 \left(\frac{P}{d\sqrt{l_{inert}}} \right) \quad (6)$$

The value of χ_0 can be obtained by comparing the value of K_{Ic} measured by this equation with those obtained by other well-established and reliable fracture mechanics methods. However, if indentation is carried out in air, crack growth might occur under the combined action of residual stresses and water molecules present in the air. Then the final length is dictated by the threshold value of K for subcritical crack growth, and it shall be referred as l_0 , with $l_0 > l_{inert}$.

For the stress distribution that results after quenching, Fett¹² have determined the stress intensity factor at the surface as well as at the deepest point of the crack. For a given time after thermal shock, the stress intensity factor may be written as:

$$K_{therm} = \sqrt{a\pi} \sum_{n=0}^2 \sigma_n F_n \quad (7)$$

where a is the crack depth, σ_n are the coefficients that result of representing the thermal stress in terms of exponential functions. F_n depends on both the crack shape and the point considered along the crack front. Here we make two simplifying assumptions: (a) the eccentricity of the crack does not change during its extension; and (b) crack extension starts at the surface and the stress intensity factor can be written as:

$$K_{therm} = Y\sigma_{max} \sqrt{\frac{l}{2}} \quad (8)$$

where Y is an “effective” geometrical factor and l is the total crack length of one crack (Fig. 1). By making this assumption we do not consider some stable crack extension that may take place at the surface before reaching critical conditions, even in the absence of residual stresses. The reason is that if the crack extends at the surface when the stress is still very low at its deepest point in the interior, crack extension at the surface may reduce its local stress intensity factor as a result of the corresponding increase in shape eccentricity. Then, it is still possible to have stable crack extension at the surface even without residual indentation stresses.¹²

The total stress intensity factor, K , is given by the sum of the thermal and residual stress intensity factors, given above in Eqs. (4) and (8):

$$K = \frac{\chi P}{d\sqrt{l}} + Y\sigma_{max} \sqrt{\frac{l}{2}} \quad (9)$$

Unstable fracture will occur when the two following conditions are simultaneously obeyed:

$$K = K_{Ic}, \quad \frac{\partial K}{\partial l} = \frac{\partial K_{Ic}}{\partial l} \quad (10)$$

Several factors may affect the level of residual stresses. One is the application of a tensile stress that may reduce the influence of residual stresses as shown by Fett.¹³ In addition, some relaxation of residual stresses could also take place during the exposure to high temperature before thermal shock. The two effects will be taken into account, in a first approximation, by assuming that χ in Eq. (9) is smaller than χ_0 . Then, by normalising Eq. (9) by K_{Ic} we have:

$$\frac{K}{K_{Ic}} = \frac{K_{res}}{K_{Ic}} + \frac{K_{therm}}{K_{Ic}} = \frac{\mu}{z} + R_0 z \quad (11)$$

where:

$$z = \sqrt{\frac{l}{l_0}} \quad (12)$$

$$\mu = \frac{\chi}{\chi_0} \sqrt{\frac{l_{inert}}{l_0}} \quad (13)$$

$$R_0 = \frac{Y\sigma_{max} \sqrt{\frac{l_0}{2}}}{K_{Ic}} \quad (14)$$

In a first approximation, it shall be assumed that K_{Ic} is independent of crack length, i.e., R curve either does not exist or is already completely developed if initial indentation cracks had reached plateau toughness. In Fig. 2 both terms of Eq. (11) are plotted in terms of z ,

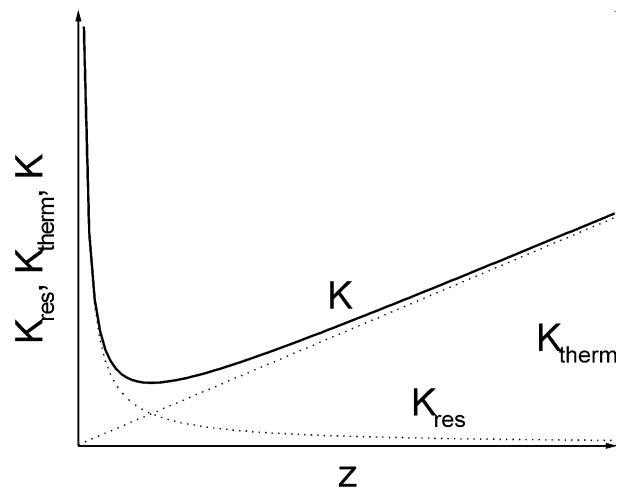


Fig. 2. Thermal, K_{ther} , residual, K_{res} , and total stress intensity factor in terms of z .

where it is observed that the total stress intensity factor shows a minimum. By imposing the first condition for fracture, i.e., $K/K_{Ic} = 1$, we have,

$$R_0 = \frac{z - \mu}{z^2} \quad (15)$$

The second critical condition for fracture, $dK/dl = 0$, gives the maximum stable crack growth that can be expected during thermal shock. By differentiating Eq. (11) with respect to the crack length, equating the result to zero, and using Eq. (15), the maximum crack extension is given by:

$$z_{cr} = 2\mu \quad (16)$$

and the critical value of R_0 for unstable fracture:

$$R_{0cr} = \frac{1}{4\mu} \quad (17)$$

Plotting R_0 in terms of z in Fig. 3 it may be seen that when μ is different from 1, stable crack growth is restricted to values of R_0 inside a small window, between $1 - \mu$ and $1/4\mu$, and no crack extension takes place if μ is 0.5. Moreover the critical stress for fracture is given by:

$$\sigma_{cr} = \frac{K_{Ic}}{4\mu Y \sqrt{\frac{l_0}{2}}} = \frac{\sigma_0}{4\mu} \quad (18)$$

where σ_0 is the critical stress for fracture under thermal shock in an inert environment and without residual stresses.

As defined in Eq. (14), R_0 represents the ratio between the initial stress intensity factor induced by thermal stresses and the fracture toughness. Then, R_0 may be used as a parameter for either classifying different materials with respect to their thermal shock resistance (the tougher the material, the smaller R_0) or measuring the severity of quenching for a given material with a fixed value of K_{Ic} .

It is interesting to note that when there is stable crack growth during thermal quenching, R_0 only depends on the ratio l/l_0 , see Eq. (14). This ratio can be easily measured without any knowledge of all parameters that influence the stress generated during thermal shock, which are often rather difficult to know accurately. Moreover, in contrast to the thermal shock parameter used by Tancret and Osterstock,⁷ R_0 does not depend on the absolute value of the initial crack length, unless small crack effects are present.

The fact that R'''' is related to the critical value of R_0 , by means of

$$R'''' = \frac{1}{2} l_0 \left(\frac{Y^2}{R_{0cr}^2} \right) (1 + \nu) = 8\mu^2 l_0 Y^2 (1 + \nu) \quad (19)$$

implies that this conventional thermal shock parameter may now also be determined without any knowledge of the σ_f . Eq. (19) allows to evaluate the effect of the indentation residual stresses on R'''' too. Thus, if the residual stresses are removed by heat treatment then $R_{0cr} = 1$, and $(R_{\mu=0})''''$ is now given by:

$$R''''_{\mu=0} = \frac{1}{2} l_0 Y^2 (1 + \nu) = \left(\frac{1}{16\mu^2} \right) R'''' \quad (20)$$

This indicates that residual stresses may have a very important effect in the evaluation of the thermal shock resistance.

3. Experimental procedure

3.1. Materials

Two microstructurally different zirconia ceramics, stabilised with a 2.5% molar of Y_2O_3 , were studied: (a) a fine-grained Y-TZP, which will be referred as AR, and (b) a zirconia material with a mixed cubic and tetragonal microstructure, named as 2H. The microstructure of the as received material (AR) has a very fine tetragonal microstructure, with an average grain size equal to of 0.3 μm , and shows high σ_f (1076 MPa) and moderate K_{Ic} (4.3 MPa $\text{m}^{1/2}$).¹⁴ Fracture resistance of this Y-TZP may be markedly increased by a suitable annealing at 1650 °C, as a result of the pronounced increase in transformation toughening efficiency, as has been shown elsewhere.¹⁴ In this work, the Y-TZP specimens have been annealed in air for 2 h at 1650 °C. The resulting material, which is referred as Y-TZP (2H), has a two-phase microstructure formed by large tetragonal grains together with cubic grains with very small precipitates. This Y-TZP, exhibits a non negligible R-curve, which reaches its peak or plateau value after crack extensions about 150 μm .

3.2. Determination of fracture toughness

K_{Ic} was determined by the surface crack on flexure (SCF) method, which is one of the more reliable procedures to measure fracture toughness.¹⁵ The observation of the crack profile by either tinting or sectional surface removal trough polishing confirmed that cracks were of Palmqvist type (Fig. 4a).¹⁶ That is, no transition to the halfpenny radial crack geometry was detected for loads smaller than 490 N. For Y-TZP(2H) the eccentricity of the crack shape, i.e., the ratio $2a/l$, ranges from 1 to 1.2 depending of the applied load. To assess if Eq. (6) for

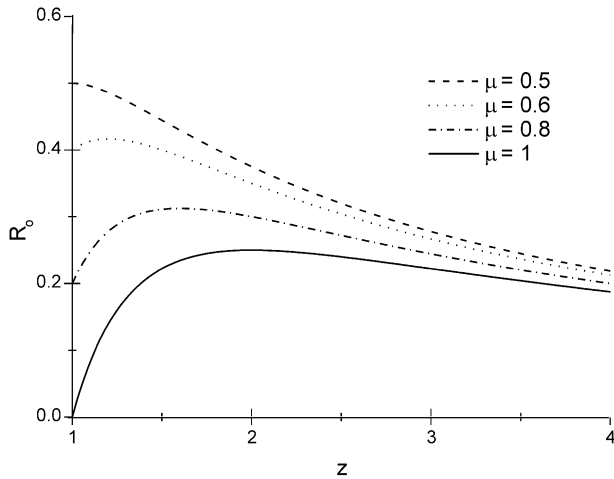


Fig. 3. Relative thermal stress intensity factor in terms of z .

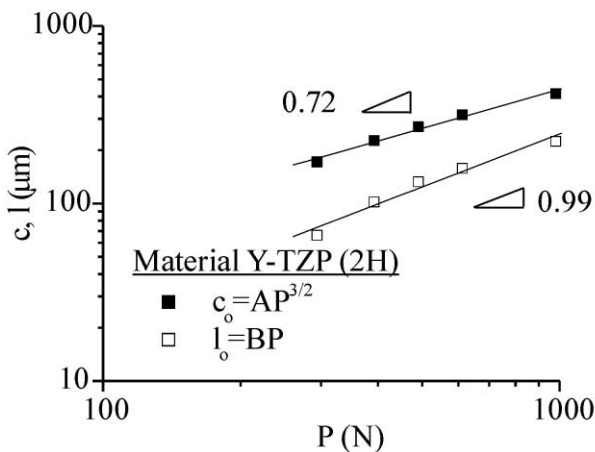
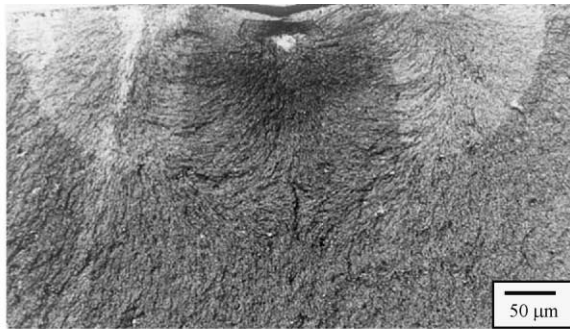


Fig. 4. (a) Palmqvist crack in Y-TZP (AR), $P = 294$ N. (b) $d + l = c$ in terms of indentation load for Y-TZP (2H).

Palmqvist cracks was suitable for describing the attained results, the crack length measured by applying different indentation loads was plotted in terms of crack length in a double logarithm scale (Fig. 4b). According to the value of the slope of the fitted straight-line (0.99), it is concluded that Eq. (6) properly describes the results. The other straight line corresponds to the half-penny radial crack. In this case, according to Anstis et al.¹⁷ the slope should be equal to 2/3, but in fact it is

slightly larger (about 0.72). Finally, Eq. (6) was calibrated so that acceptable values of fracture toughness could be estimated from indentation tests by using this equation.

R curves measured by the SCF method for Y-TZP and Y-TZP (2H) are shown in Fig. 5. It can be seen that Y-TZP (AR) hardly shows any R-curve behaviour, while for Y-TZP (2H) there is a clear increase in fracture resistance with crack extension. Table 1 gives the value of the plateau toughness that is reached after crack extensions of about 150 μm . It is then reasonable to expect that indentation cracks longer than this are fully shielded in most of the cases. The intrinsic fracture toughness of the studied materials is also given in Table 1. The value calibrated for the constant C of Eq. (5) is 0.0076 ± 0.0004 .

3.3. Thermal shock experiments

Thermal shock tests were conducted on small discs with 4 mm in thickness that were cut from cylindrical bars of 8 mm diameter. They were ground and polished with diamond pastes with 30 and 6 μm particle sizes. Final polishing was carried out with a diamond paste of 3 μm . The specimens were indented using a Vickers pyramidal diamond and the lengths of the cracks measured by means of an optical microscope and using Nomarski interference contrast. Samples were heated during 15 min in a furnace at different temperatures and quenched in silicone oil at room temperature. Crack extension after quenching was finally assessed through optical microscopy.

4. Results and discussion

Indentation loads and changes in temperature used in thermal shock experiments are shown in Table 2 together with the crack lengths measured before and after quenching.

The values of R_0 determined from indentation lengths are given in Fig. 3. It is interesting to point out that the critical value of R_0 only depends of μ . Hence, if both materials experience same reduction in residual stress as well as same behaviour with respect to subcritical crack growth, i.e., same value of μ , then a same critical value of R_0 follows independently of the material and the initial crack length. The lower is the indentation load and the initial crack length, the more severe may be the thermal shock that the material can withstand without breaking. However, the value of R_0 at which catastrophic fracture occurs is always the same. This is not the case with the R'''' parameter of Eq. (19) since it depends on the initial crack length.

The observation that crack extension does not occur until a relatively large temperature gradient is applied is

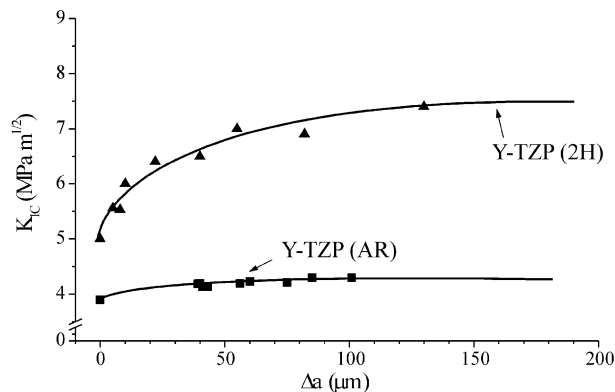


Fig. 5. R-curve for Y-TZP (AR) and Y-TZP (2H).

Table 1

Intrinsic fracture toughness (K_0), plateau fracture toughness ($K_{R, \max}$) and shielding (K_S)

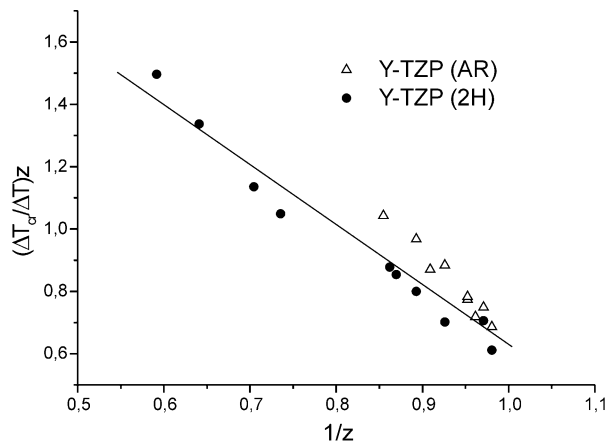
Material	K_0 (MPa \sqrt{m})	$K_{R, \max}$ (MPa \sqrt{m})	K_S (MPa \sqrt{m})
Y-TZP (AR)	3.9 ± 0.1	4.3 ± 0.2	0.4 ± 0.2
Y-TZP (2H)	5.0 ± 0.2	7.4 ± 0.2	2.4 ± 0.2

consistent with the assumptions made about subcritical crack growth and reduction of residual stresses. For example, no crack propagation is detected in Y-TZP until ΔT is close to 370 °C for an indentation load of 300 N. There are several reasons for such behaviour. First, the severity of the quench associated with the tempering bath used (silicone oil) is not as high as the conventionally referred, i.e. water. Second, there should be a reduction in the indentation residual stresses during the annealing and quenching process. It may be noticed that a similar effect is found in bending of bars with indentation cracks. Third, because of subcritical crack growth after indentation, a finite stress intensity factor must be applied for some length of time in order to restart crack extension from the threshold value.

From experiments it is possible to determine the value of μ if we assume that inside the relatively small range of temperatures for which stable crack growth occurs the maximum thermal stress developed is proportional to the change in temperature, i.e., $\sigma_{th} \propto \Delta T$. Under these conditions,

$$\frac{\Delta T}{\Delta T_{cr}} = \frac{R_0}{R_{crit}} = 4\mu \left(\frac{z - \mu}{z^2} \right) \quad (21)$$

and a straight line with a slope equal to $-4\mu^2$ should result when experimental values of $\frac{\Delta T}{\Delta T_{cr}} z$ are plotted in terms of $\frac{1}{z}$. From Fig. 6 it is clear the existence of a linear correlation for all experimental points of both zirconias investigated. From the measurement of the slope we obtain that $\mu \approx 0.68$.

Fig. 6. $\frac{\Delta T}{\Delta T_{cr}} z$ versus $1/z$ for the two zirconia materials studied.

It is worthwhile to recall that μ is defined as the product of two terms, both smaller than the unity. This is because l_{inert} is always smaller than l_0 and the other term, $\frac{z}{z_0}$, is also smaller than unity if there is a reduction in residual stresses. To analyse qualitatively the possible influence of subcritical crack growth on the measured value of μ , different indentation tests were carried out in silicone oil and the length compared with those obtained in air with a relative humidity of 62% (see Table 3). It is observed that the influence of $\sqrt{\frac{l_{inert}}{l_0}}$ on μ is only of about 10%. Hence, subcritical crack growth assisted by the environment during thermal shock can be neglected since the quenching medium used is silicone oil. Therefore, a total reproduction of the experimental results by the present analysis requires a reduction of about 25% in the level of residual stresses.

Such assumption may be experimentally supported through mechanical tests of indented samples of the materials studied after a heat treatment similar to that used for thermal shock testing (15 min at temperatures between 400 and 500 °C). The 3-point bending strength for the material with this heat treatment was found to increase by about 30%, a fact that completely sustains the hypothesis of reduced residual indentation stresses during the annealing and quenching experiences.

Stable crack extensions observed after thermal shock are generally small in comparison with the maximum stable crack extension expected. If no allowance for reduction of internal stresses were made, then $\mu = 1$ and z_{crit} would be equal to 2, so that, in principle, crack lengths close to 4 times the original length should be detected. However this is not the case. Considering $\mu = 0.68$, the critical value of the crack length is about twice the initial one, l_0 , and $z_{crit} \approx 1.4$. This is consistent with the values of z for Y-TZP as given in Table 2, where the larger crack measured after thermal shock corresponds to z equal to 1.36. Nevertheless, in general, the measured values of z are smaller than this critical value. On the other hand, for Y-TZP (2H) crack

Table 2
Experimental results for the studied materials

P (N)	l_0 (μm)	l_f (μm)	ΔT ($^{\circ}\text{C}$)	Z
<i>Y-TZP (AR)</i>				
300	170	175	370	1.02
300	162	177	380	1.04
300	171	181	400	1.03
300	164	179	405	1.05
300	166	183	410	1.05
300	170	204	435	1.10
200	133	156	450	1.08
200	135	170	475	1.12
200	133	183	490	1.17
200	135	183	510	1.36
<i>Y-TZP (2H)</i>				
625	271	282	420	1.02
625	309	360	455	1.08
625	292	312	480	1.03
625	286	306	480	1.03
625	257	323	500	1.12
625	276	367	520	1.15
625	280	374	530	1.16
300	148	273	540	1.36
300	146	298	560	1.42
200	100	245	600	1.56
200	98	280	620	1.69

Table 3
Indentation crack lengths in Y-TZP in air with 62% humidity and in silicone oil

Load (N)	l_0 in air (μm)	l_{inert} in silicone oil (μm)	$\sqrt{l_{\text{inert}}/l_0}$
300	173	151	0.93
400	225	179	0.89

extension is larger. In this case, because there is a significant R-curve behaviour, crack extension may take place under increasing fracture resistance, i.e. when saturation shielding due to the transformed zone around the crack may have not been reached, because its initial crack length is small. Therefore, it is convenient to consider the effect of a rising R-curve in the results.

In doing so, let us assume that K_{Ic} increases during crack extension, so that

$$K_{\text{Ic}} = K_0 z^n \quad (22)$$

where K_0 is the fracture toughness for the initial indentation crack when $z=1$ and n is a positive number that measures shielding. Thus, if the crack is already completely shielded, i.e., plateau toughness has been already achieved during indentation, n should be equal to zero. For partly shielded small cracks in high toughness materials, n will depend on the initial crack length.

Thus, n can be different for each indentation crack. The condition for stable crack growth can now be written as

$$R_0 z + \frac{\mu}{z} f = f z^n \quad (23)$$

where f denotes the ratio between K_0 and the plateau of K_{Ic} . By imposing the conditions for unstable crack growth, it is found that:

$$z_{\text{crit}} = \left(\frac{2\mu}{1-n} \right)^{\frac{1}{n+1}} \quad (24)$$

$$(R_0)_{\text{crit}} = \frac{1}{2} f (n+1) \left(\frac{1-n}{2\mu} \right)^{\frac{1-n}{n+1}} \quad (25)$$

Both expressions can be converted into those given in Eqs. (16) and (15), respectively, for $n=0$, since then $f=1$. The fact that under thermal shock there is more stable crack growth in Y-TZP (2H) than in Y-TZP is explained by the presence of an R-curve in the heat treated zirconia. When cracks are small in the tougher material, there is still some increase in shielding when they extend. But large cracks produced by high indentation loads are already completely shielded, and the amount of normalised crack extension is relatively shorter. The expressions given in Eqs. (24) and (25) with n about 0.5 could quantitatively account for the results attained in this investigation.

When no R-curve effects are present, the influence of indentation residual stresses are very important as can be seen from Eq. (22): the change in temperature that can be sustained for a material with indentation residual stresses $\Delta T_{\text{cr}}^{\mu}$ is reduced by a factor of 4μ in comparison with the same material with identical defects but without residual stresses, $\Delta T_{\text{cr}}^{\mu=0}$,

$$\Delta T_{\text{cr}}^{\mu} = \frac{\Delta T_{\text{cr}}^{\mu=0}}{4\mu} \quad (26)$$

Therefore, indentation residual stresses play an important role in thermal shock resistance.

5. Conclusions

The thermal shock behaviour of Y-TZP (AR) with 2.5% molar of yttria and of Y-TZP (2H), which is produced by heat treatment of the former material, has been studied by examining crack extension and fracture of Palmqvist indentation cracks. The main conclusions can be summarised as follows:

- The ratio, R , between the thermal stress intensity factor and the fracture toughness is a suitable

parameter to measure thermal shock behaviour. It can be obtained by measuring indentation crack lengths before and after thermal shock.

- (b) The maximum stable crack extension of indentation Palmqvist cracks and the thermal shock resistance can be accounted for in terms of sub-critical crack growth and reduction in the level of residual stresses.
- (c) For small indentation loads, there is more stable crack growth in Y-TZP (2H) because it has a non-negligible R-curve behaviour and therefore indentation cracks may be not completely shielded.

Acknowledgements

The authors gratefully acknowledge the Spanish Comisión Interministerial de Ciencia y Tecnología for its financial support under grant No. MAT94-0431 and to the Generalitat de Catalunya under grant No. 1999SGR00129.

References

1. Evans, A. G., Perspective on the development of high-toughness ceramics. *J. Am. Ceram. Soc.*, 1992, **73**, 187–206.
2. Hannink, R. H. J., Kelly, P. M. and Muddle, B. C., Transformation toughening in zirconia-containing ceramics. *J. Am. Ceram. Soc.*, 2000, **83**, 461–487.
3. Lange, F. F., Transformation toughening Part 1–4. *J. Mater. Sci.*, 1982, **17**, 225–254.
4. Faber, K. T., Huang, M. D. and Evans, A. G., Quantitatives studies of thermal shock in ceramics based on a novel test technique. *J. Am. Ceram. Soc.*, 1981, **64**, 296–301.
5. Hasselman, D. P. H., Elastic energy as design criteria for thermal shock. *J. Am. Ceram. Soc.*, 1963, **46**, 1535–1540.
6. Hasselman, D. P. H., Thermal stress resistance parameters for brittle refractory ceramics: A compendium. *Am. Ceram. Soc. Bull.*, 1970, **49**, 1033–1037.
7. Tancret, F. and Osterstock, F., The Vickers indentation technique used to evaluate thermal shock resistance of brittle materials. *Scripta Mater.*, 1997, **37**, 443–447.
8. Tancret, F., Monot, I. and Osterstock, F., Toughness and thermal shock resistance of $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ composite superconductors containing Y_2BaCuO_5 or Ag particles. *Mater. Sci. Eng.*, 2001, **A298**, 268–283.
9. Collin, M. and Rowcliffe, D., Analysis and prediction of thermal shock in brittle materials. *Acta Mater.*, 2000, **48**, 1655–1665.
10. Lawn, B., *Fracture Mechanics of Brittle Solids*. Cambridge University Press, UK, 1995.
11. Niihara, K., Morena, R. and Hasselman, D. P. H., *J. Mater. Sci. Lett.*, 1982, 13–16.
12. Fett, T., Failure due to semielliptical surface cracks under arbitrary stress distributions. *Fatigue Fract. Eng. Mater. Struct.*, 2000, **23**, 347–353.
13. Fett, T., An analysis of the residual intensity factor of Vickers indentation cracks. *Eng. Fract. Mech.*, 1995, **52**, 773–776.
14. Casellas, D., Llanes, L., Cumbreña, F., Sánchez-Bajo, F., Forsling, W. and Anglada, M., On the transformation toughening of Y-ZrO₂ ceramics with mixed TZP/PSZ microstructures. *J. Eur. Ceram. Soc.*, 2001, **21**, 147–159.
15. Quinn, G. D., Kübler, J. and Getting, R., *Fracture Toughness of Advanced Ceramics by the Surface Crack in Flexure (SCF) Method: A VAMAS Round Robin*. VAMAS Technical Working Area 3, Report No. 17, 1993.
16. Alcalá, J. and Anglada, M., Indentation pre-cracking of Y-TZP: implications to R-curves and strength. *Mater. Sci. Eng.*, 1998, **A245**, 267–276.
17. Anstis, G. R., Chantikul, P., Lawn, B. R. and Marshall, D. B., A critical evaluation of indentation techniques for measuring fracture toughness: I, direct crack measurements. *J. Am. Ceram. Soc.*, 1981, **64**, 533–538.