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Discussion

Comments on "Thermal shock resistance of yttria-stabilized zirconia with Palmqvist indentation cracks" by G. Fargas, D. Casellas, L. Llanes, M. Anglada [J. Eur. Ceram. Soc. 23 (2003) 107–114]

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Abstract

In a recent paper, Fargas et al. [Fargas, G., Casellas, D., Llanes, L. and Anglada, M., Thermal shock resistance of yttria-stabilized zirconia with Palmqvist indentation cracks. *J. Eur. Ceram. Soc.*, 2003, **23**, 107–114] make use of the Vickers indentation technique to characterise the thermal shock resistance of brittle materials exhibiting the Palmqvist indentation crack system. They claim that their approach can provide a measurement of Hasselman's R'''' thermal shock resistance parameter. It is here demonstrated that the obtained parameter is in fact *different* from R''''. In parallel, it is shown that a previously developed approach, which is both consistent with the concepts of R'''' and of the critical quenching temperature difference, ΔT_C, can be used in the case of the Y-TZP ceramics of Fargas et al. This approach also allows an estimation of the maximum thermal stresses and of the coefficient of heat transfer at the fluid–solid interface as a function of the quenching temperature difference.

Keywords: Thermal shock resistance; Mechanical properties; Toughness; Indentation

1. Introduction

Thermal shock is a frequent problem for ceramic users, when sudden temperature changes are involved, for instance in refractories, superconductors, electronic components, fuel cells, filtration devices, thermostructural parts, etc. During thermal shocks, transient thermal stresses build up in the material and can become large enough to induce damage, such as microcracking or macrocracking. The nature and extent of thermal shock damage depend on the one hand on the ability of the ceramic to resist cracking, i.e. on its toughness and strength, and on the other hand on the severity of quenching, i.e. on the maximum value reached by transient thermal stresses during the process. If the former can easily be measured by conventional mechanical testing, the latter is rather difficult to evaluate since it is the result of complex transient mechanisms like heat transfer and conduction in the fluid, in the solid and at their interface. For this reason, many studies aim at the characterisation of the consequences of thermal shock on ceramic materials rather than the physics involved. In this respect, the investigation of the behaviour of Vickers indentation cracks under quenching conditions has raised interest in the past years.[1–9] In particular, an interesting work has been performed and published by Fargas et al.,[1] where the authors derive several quantities and parameters. The major interest of this work is that it proposes to take into account the effects of corrosion cracking, of a rising *R*-curve, and of the indentation residual stresses relaxation during crack extent.

In the present work, some aspects of the approach are analysed from a theoretical point of view, especially the proposed comparison of one of the derived parameters with Hasselman's R'''' thermal shock resistance parameter.[10] Then, a comparison is made with a previously proposed approach to characterise the thermal shock resistance of brittle materials exhibiting the Palmqvist indentation crack system.[4] Finally,

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the latter method is used to exploit and analyse the experimental results gained by Fargas et al. on Y-TZP ceramics.[1]

2. Theoretical background

2.1. Thermal shock and Hasselman's R'''' parameter

The present paper is voluntarily restricted to the quenching of brittle materials from high temperature into a colder fluid. In such a case, tensile transient thermal stresses appear near the surface of the quenched part, with a maximum value given by:[11]

$$\sigma_{\mathrm{th}} = \frac{E\alpha\Delta T}{1-\nu} f\left(\frac{r\bar{h}}{k}\right) \tag{1}$$

where E is the material's Young's modulus, α its coefficient of thermal expansion, ν its Poisson's ratio, k its thermal conductivity and r a characteristic dimension of the quenched part. $\bar{h}$ is the effective heat transfer coefficient at the fluid–solid interface, and f is a rising function, with values between 0 ("soft" quench) and 1 ("hard" quench). If the quenched material contains a natural flaw on which fracture can occur, with a characteristic size $a/2$, associated to a geometrical factor Y, its uniaxial fracture stress σ_{f} is governed by its toughness, K_{IC}, as:

$$K_{\mathrm{IC}} = \sigma_{\mathrm{f}} Y \sqrt{\frac{a}{2}} \tag{2}$$

Similarly, Hasselman has shown that, under thermal shock conditions, the critical natural flaw size which may cause fracture (if the thermal stress reaches the fracture stress) is proportional to a parameter called R'''':[10]

$$R'''' = \left(\frac{K_{\mathrm{IC}}}{\sigma_{\mathrm{f}}}\right)^2 (1+\nu) \tag{3}$$

R'''' can thus be understood as being proportional to the size of the natural surface flaw which may cause fracture during quenching.

2.2. The Vickers indentation method for Palmqvist cracks

When brittle materials are indented with a Vickers diamond, they may develop cracks around the indent, adopting one of the two observed systems, the median-radial or the Palmqvist crack system, the latter being the object of the present paper (Fig. 1). Cracks of initial length ℓ_0 form under an indentation load P following:[12]

$$K_{\mathrm{IC}} = B\left(\frac{H}{4}\frac{P}{\ell_0}\right)^{1/2} = \chi_{\mathrm{p}} P^{1/2} \ell_0^{-1/2} \tag{4}$$

where B is a geometrical constant, H the material's hardness, and χ_{p} is defined as $B(H/4)^{1/2}$. Under thermal shock conditions, thermal stresses superimpose to the indentation residual stresses, which provokes stable crack propagation (within certain limits) to a new length, ℓ. It has been previously shown that this writes:[4]

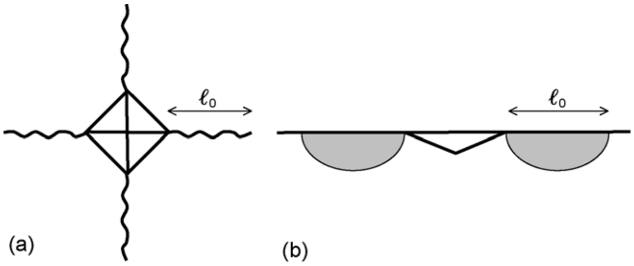

Fig. 1. Schematic representation of Palmqvist indentation cracks: (a) top view; (b) cross-section.

$$K_{\mathrm{IC}} = \chi_{\mathrm{p}} P^{1/2} \ell^{-1/2} + \sigma_{\mathrm{th}} \left(\pi \Omega \frac{\ell}{2}\right)^{1/2} \tag{5}$$

The right-hand part of this equation is the stress intensity factor induced by the thermal stresses, σ_{th}, at the tip of each semi-elliptical Palmqvist crack of length ℓ. The geometrical factor Ω is equal to $4/\pi^2$. The equation can also be written using a global geometrical factor Y, as proposed later by Fargas et al.[1] Using the present formalism, this gives:

$$K_{\mathrm{IC}} = \chi_{\mathrm{p}} P^{1/2} \ell^{-1/2} + \sigma_{\mathrm{th}} Y \left(\frac{\ell}{2}\right)^{1/2} \tag{6}$$

The combination of Eqs. (4) and (5) can be used to measure a number of quantities, such as the value of the maximum transient thermal stress, σ_{th}, and several thermal shock resistance parameters. Some of them will be presented and commented in the next section.

3. The Vickers indentation method and Hasselman's R'''' parameter

Fargas et al.[1] proposed to calculate a quantity which, if neither stress-corrosion cracking nor indentation residual stresses relaxation occurs (which corresponds to $\mu = 1$ in the Eq. (19) of the cited reference), reduces to:

$$R'''' = 8\ell_0 Y^2 (1+\nu) \tag{7}$$

This quantity is obtained by introducing, into the expression of Hasselman's R'''' parameter, the value of the fracture stress of an indented material (Eq. (18) of the cited reference):

$$\sigma_{\mathrm{cr}} = \frac{K_{\mathrm{IC}}}{4Y\sqrt{\ell_0/2}} \tag{8}$$

Fargas et al. claim that the parameter calculated in Eq. (7) is Hasselman's R'''' thermal shock parameter (they also obtain another parameter if contact residual stresses are removed, which is a 16th of the same quantity, if $\mu = 1$, but this does not change anything from a fundamental point of view). However, it is worth reminding that Hasselman's R'''' is proportional to the *natural* flaw size that may cause fracture. Indeed, combining Eqs. (2) and (3) gives the actual value of R'''':

$$R'''' = \frac{1}{2}aY^2(1+\nu) \tag{9}$$

It becomes obvious that the quantity calculated in Eq. (7) is not Hasselman's R'''', but is proportional to the *artificial* (indentation) crack size, ℓ_0, that will cause fracture. To avoid such a confusion between these two quantities, they should be given different names.

At the opposite, another thermal shock parameter had been proposed earlier.[3] This parameter reflects the fact that, to resist a given thermal shock, a material must exhibit a high resistance to cracking, i.e. a high toughness, and must be submitted to a low thermal stress. It writes:

$$R_{\mathrm{th}} = \left(\frac{K_{\mathrm{IC}}}{\sigma_{\mathrm{th}}}\right)^2 \tag{10}$$

It has been shown that, for Palmqvist indentation cracks, its value can be obtained by a simple measurement of the lengths of indentation cracks before and after quenching, as:[4]

$$R_{\mathrm{th}} = \frac{\pi\Omega\ell}{2[1-(\ell/\ell_0)^{-1/2}]^2} \tag{11}$$

It is important to note that, contrarily to the so-called R'''' parameter of Fargas et al. of Eq. (7), R_{th} does not depend on indentation conditions, such as initial crack length or load. Indeed, as defined in Eq. (10), R_{th} is only related to the material toughness and to the transient thermal stress during quenching. Only the *measurement* of R_{th} makes use of indentation cracks, but its value is always the same whatever the indentation conditions are. In fact, this particularity can be used to measure R_{th} with accuracy, for instance by introducing indents under different loads, giving different values of ℓ_0 and ℓ; R_{th} is then obtained from the slope of the ℓ versus $2[1-(\ell/\ell_0)^{-1/2}]^2$ line.[4]

When the quenching conditions become more severe, the maximum transient thermal stress σ_{th} increases. Fracture occurs in a non-indented material when σ_{th} reaches the material fracture stress σ_{f}, which corresponds to $R_{\mathrm{th}} = (K_{\mathrm{IC}}/\sigma_{\mathrm{f}})^2 = R''''/(1+\nu)$. In fact, when the quenching temperature difference ΔT increases, R_{th} tends towards[3] $R''''/(1+\nu)$.

4. Thermal shock of Y-TZP ceramics

The method to evaluate R_{th} in the case of Palmqvist indentation cracks had been exposed from a theoretical point of view, but could not be experimentally validated due to a lack of usable measurements.[4] In this respect, the data published by Fargas et al.[1] can be precious. These data concern the propagation of Palmqvist indentation cracks in discs (8 mm in diameter and 4 mm in thickness) of two different grades of yttria-stabilised zirconia, called Y-TZP (AR) and Y-TZP (2H), submitted to quenches from various temperatures into a silicon oil at room temperature. Fargas et al.[1] investigated the thermal shock behaviour of these ceramics using the stage of stable propagation of Palmqvist indentation cracks. Their results are presented in Table 1. It is important to note that both ceramics present a rising R-curve behaviour, especially the so-called Y-TZP (2H) grade. However, because all measured cracks have initial lengths correponding to toughness values close to the R-curve plateaus,[1] it can be approximated that, during subsequent crack propagation under thermal shock conditions, the toughness will remain nearly constant. Therefore, we will take $K_{\mathrm{IC}} = 4.3\,\mathrm{MPa\,m^{1/2}}$ for Y-TZP (AR) and $K_{\mathrm{IC}} = 7.4\,\mathrm{MPa\,m^{1/2}}$ for Y-TZP (2H).

Table 1
Palmqvist crack lengths before (ℓ_0) and after (ℓ) thermal shock for the two grades of Y-TZP quenched with various temperature differences, ΔT

P (N)	ΔT (K)	ℓ_0 (μm)	ℓ (μm)
Y-TZP (AR)			
300	370	170	175
300	380	162	177
300	400	171	181
300	405	164	179
300	410	166	183
300	435	170	204
200	450	133	156
200	475	135	170
200	490	133	183
200	510	135	183
Y-TZP (2H)			
625	420	271	282
625	455	309	360
625	480	289	309
625	500	257	323
625	520	276	367
625	530	280	374
300	540	148	273
300	560	146	298
200	600	100	245
200	620	98	280

Data from Fargas et al.[1].

4.1. Transient thermal stresses

The first interesting thing to do is to evaluate the maximum thermal stress in the ceramics, σ_{th}, and its evolution with the quenching temperature difference, ΔT. The former can be obtained by combining Eqs. (4) and (5), which gives:

$$\sigma_{\mathrm{th}} = K_{\mathrm{IC}}\left(\frac{2}{\pi\Omega}\frac{\ell_0}{\ell}\right)^{1/2}(\ell_0^{-1/2} - \ell^{-1/2}) \tag{12}$$

This quantity has been calculated from the values of Table 1, and plotted in Fig. 2 for both grades of Y-TZP as a function of ΔT. It is clear that, for a given ΔT, both materials experience similar stresses. This suggests that, because both ceramics exhibit almost identical fracture stresses (1076 MPa for Y-TZP (AR)[1] and 1058 MPa for Y-TZP (2H) [13]), their critical quenching temperature difference should be similar; this will be later evaluated by another method and discussed.

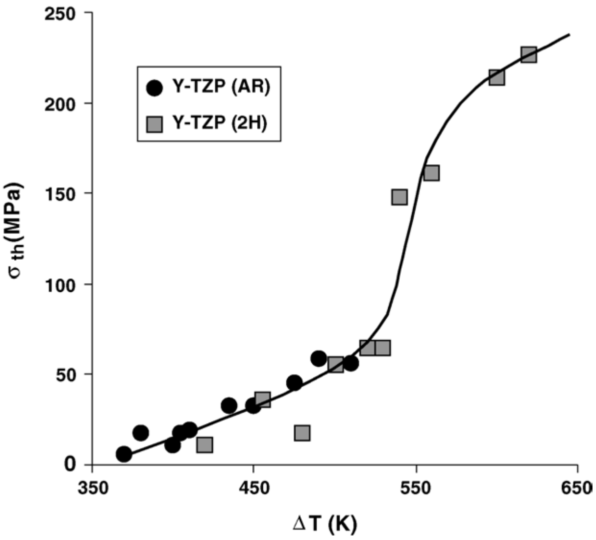

Fig. 2. Evolution of the thermal stress, σ_{th}, in Y-TZP ceramics as a function of the quenching temperature difference, ΔT.

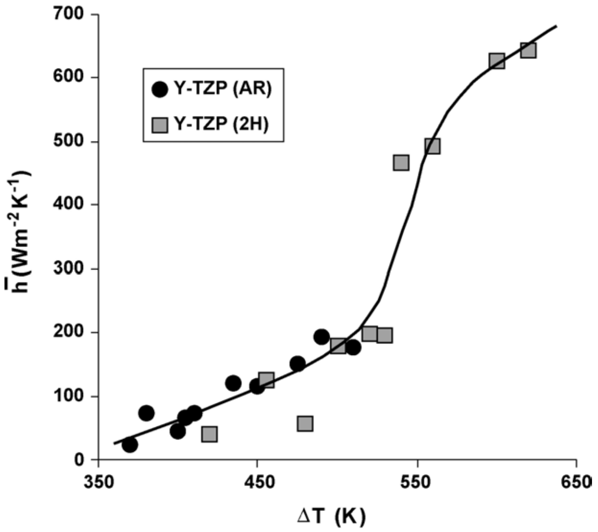

Fig. 3. Evolution of the heat transfer coefficient, $\bar{h}$, as a function of the temperature difference, ΔT.

4.2. Coefficient of heat transfer

Knowing the values of thermal stresses, it becomes possible to evaluate the coefficient of heat transfer at the fluid–solid interface, $\bar{h}$, which is an important parameter governing quenching processes. Indeed, Eq. (1) can be rewritten as:

$$f\left(\frac{r\bar{h}}{k}\right) = f(\beta) = \frac{\sigma_{\mathrm{th}}(1-\nu)}{E\alpha\Delta T} \tag{13}$$

The evolution of the reduced stress, $f(\beta)$, as a function of the Biot number, $\beta = r\bar{h}/k$, for this type of quenching geometry is well described by:[11]

$$f(\beta) = \frac{\beta}{\beta+4} \tag{14}$$

Combining Eqs. (13) and (14), and taking values of $r = 2$ mm (the discs half-thickness), $E = 200$ GPa and $\nu = 0.3$,[13] $\alpha \cong 10.10^{-6}\,\mathrm{K}^{-1}$[14] and $k \cong 2.2\,\mathrm{W\,m^{-1}\,K^{-1}}$,[15] it becomes possible to obtain a rough estimation of the evolution of $\bar{h}$ with ΔT (Fig. 3). The coefficient of heat transfer being characteristic of the heat that the fluid is able to take away from the interface per unit time, it is normal that the trend is the same for both grades of ceramics. The obtained values, of the order of several hundreds of $\mathrm{W\,m^{-2}\,K^{-1}}$, are typical of those obtained for silicon oils in this range of ΔT.[16]

4.3. Thermal shock resistance parameters

The measurement by Fargas et al.[1] of the propagation of Palmqvist cracks under quenching conditions provide an excellent opportunity to validate the previously proposed approach,[4] and the use of R_{th} as a meaningful thermal shock resistance parameter. In this aim, R_{th} was calculated with Eq. (11) from values of Table 1, for both grades of ceramics and for each temperature difference. Parallelly, the values of Hasselman's R'''' parameter and the quantities $R''''/(1+\nu)$ have been calculated for both materials with equation (3), taking toughnesses of 4.3 MPa m$^{1/2}$ and 7.4 MPa m$^{1/2}$, and fracture stresses of 1076 MPa and 1058 MPa for Y-TZP (AR) and Y-TZP (2H), respectively. For both ceramics, the obtained R_{th} parameters are plotted in Fig. 4 as a function of the temperature difference, and values of $R''''/(1+\nu)$ have been added to the graph.

First, the thermal shock resistance parameter R_{th} is higher for Y-TZP (2H) than for Y-TZP (AR), for all temperature differences. This can be explained by the higher toughness of the former. Indeed, it has been shown in the previous section that, due to similar thermophysical and elastic properties, both materials experience similar transient thermal stresses during quenching. Thus, differences in $R_{\mathrm{th}} = (K_{\mathrm{IC}}/\sigma_{\mathrm{th}})^2$ are governed by the sole toughness. Because R_{th} is proportional

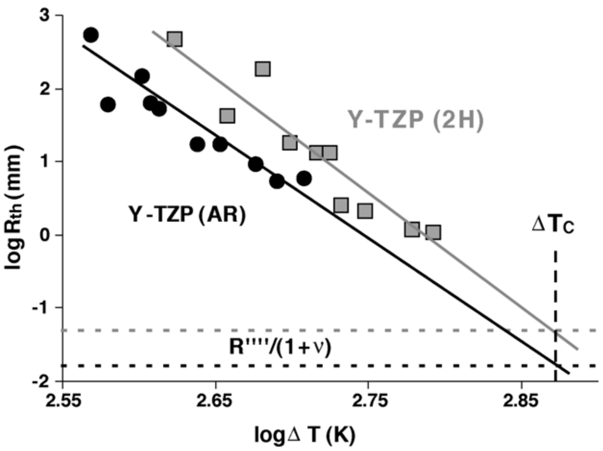

Fig. 4. Evolution of the thermal shock resistance parameter, R_{th}, in Y-TZP ceramics as a function of the quenching temperature difference, ΔT. As the latter increases, $R_{\mathrm{th}} = (K_{\mathrm{IC}}/\sigma_{\mathrm{th}})^2$ decreases and tends towards $R''''/(1+\nu)$. This allows to estimate the critical quenching temperature difference, ΔT_{C}.

to the size of the natural surface flaw that can be withstood by the material during quenching, this means that Y-TZP (2H) ceramics are more flaw-tolerant than the Y-TZP (AR) ones: under given thermal shock conditions, the former can resist cracking if they contain defects of a larger size than the latter. This is an extremely important issue, as ceramic fabrication always implies a statistical dispersion in flaw sizes, hence in fracture stresses. By using a more flaw-tolerant material, the user makes sure that a higher fraction of parts will resist a given thermal shock. Also, even in the case where both materials contain small initial flaws of comparable sizes, a higher R_{th} allows the user to tolerate the larger cracks which may form in service by thermal fatigue or by stress-corrosion slow crack growth.

Then, as expected, R_{th} decreases as ΔT increases, which is due to the increase of σ_{th}. Theoretically, R_{th} tends towards $R''''/(1+\nu)$ when ΔT tends towards ΔT_{C}. This makes possible, by extrapolating the evolution of R_{th} with ΔT towards $R''''/(1+\nu)$, to estimate the value of the critical quenching temperature difference, ΔT_{C}, which is the temperature difference that causes thermal shock fracture from natural flaws. This gives, for both grades of Y-TZP, critical temperature differences of around 740 K. Of course, due to the large extrapolations performed, these values are very approximate. It is interesting to note that, even if one ceramic is significantly superior to the other in terms of R_{th}, they have very similar ΔT_{C}, because their fracture stresses are close and they experience similar thermal stresses. This is interesting but, as already mentioned, ΔT_{C} also reflects the statistical dispersion in flaw sizes and fracture stresses. In this respect, R_{th} is a more deterministic parameter: it represents the intrinsic ability of the material to resist a given thermal shock, and not the fluctuations due to the fabrication procedure as ΔT_{C} does.

5. Conclusions

Several conclusions can be drawn from this critical analysis of the work of Fargas et al.[1] on the investigation of thermal shock behaviour of Y-TZP ceramics through the propagation of Palmqvist indentation cracks:

(i) The parameter proposed by the authors, which they named R'''', is proportional to the size of the *artificial* (indentation) crack on which fracture will initiate. It is therefore different from the actual Hasselman's R'''' thermal shock parameter, which is proportional to the size of the *natural* flaw on which fracture will initiate.

(ii) A previously proposed approach,[4] which describes the propagation of Palmqvist indentation cracks under thermal shock conditions, is validated with the use of data gained on Y-TZP ceramics and published in the cited reference.[1]

(iii) This method allows to estimate the maximum of the thermal transient stresses in the material, σ_{th}, as well as the effective heat transfer coefficient at the fluid–solid interface, $\bar{h}$, as a function of the quenching temperature difference, ΔT.

(iv) The thermal shock resistance parameter R_{th}[3,4] can be used to characterise Y-TZP ceramics quenched into a silicon oil. The extrapolation of R_{th} towards $R''''/(1+\nu)$ allows to evaluate the critical quenching temperature difference, ΔT_{C}, of the ceramics. The two investigated grades of Y-TZP exhibit a similar ΔT_{C} ($\cong 740\,\mathrm{K}$), due to similar thermoelastic properties and fracture stresses, but the thermal shock flaw tolerance (R_{th}) is higher for the material with a superior toughness.

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