

Thermal Cycling of Indented Ceramic Materials

Tomas Andersson* and David J. Rowcliffe

Materials Science and Engineering, Royal Institute of Technology, S-100 44 Stockholm, Sweden

(Received 4 June 1997; accepted 7 November 1997)

Abstract

A new technique, based on the limited propagation of small precracks, has been used to investigate the thermal shock and thermal cycling resistance of several ceramic materials quenched into boiling water. The ΔT_c 's of the materials are defined from the crack propagation and extension after a single quench. Statistically significant data are obtained by studying a large number of cracks in each specimen. Few specimens are thus required. The materials are ranked according to the individual values of ΔT_c . In order to estimate the effect of different quenching rates on the values of ΔT_c the results are compared with data from quenches into water at room temperature using the same technique. The experimentally determined values of ΔT_c are analysed in terms of R and R' parameters, specimen thickness and cooling rates. © 1998 Published by Elsevier Science Limited. All rights reserved

1 Introduction

Ceramic materials are sensitive to environments in which the temperature fluctuates rapidly.¹ The low thermal conductivity of ceramics leads to the build-up of thermal gradients. These thermal gradients create large thermal stresses, which may cause catastrophic failure. To determine the size and shape of a thermal gradient the thermal properties of the material as well as the heat transfer coefficient for the specific cooling case has to be known. The sizes of the thermal stresses arising from the thermal gradient are decided by the mechanical properties of the material.²

Buessem³ and Manson⁴ developed a semi-empirical formula, see eqn (1), which can be used to understand how the material properties decide

the thermal shock resistance of a plate subjected to cooling:

$$\Delta T_{\max} = 1.5R + 3.25 \frac{R'}{r_m h} - 0.5R e^{-16/\beta} \quad (1)$$

$$R' = R * k = \frac{\sigma_f [1 - \nu] k}{E \alpha} \quad (2)$$

$$\beta = \frac{r_m h}{k} \quad (3)$$

Here, ΔT_{\max} is the maximum temperature difference the material can stand, σ_f is the fracture strength of the material, ν is Poisson's ratio, E is Young's modulus, α is the thermal expansion coefficient, β is a dimensionless heat transfer number called Biot's modulus, r_m is a characteristic heat transfer length (half the thickness of the plate), k is the thermal conductivity of the material and h is the surface heat transfer coefficient acting between the plate and the cooling medium. The value of β decides the temperature distribution inside the plate. High values of h give high cooling rates. For rapid cooling conditions the R parameter will have the largest influence on the performance of the material.⁵ For this case, the mechanical properties of the material will decide the thermal shock resistance. With decreasing cooling rates the R' parameter and thus the thermal conductivity of the material will become more important for the performance of the material.⁵ An analytical ranking of materials with regard to their thermal shock resistance is thus only possible when the heat transfer conditions are well defined.

Equation (1) is valuable when the heat transfer conditions can be considered as fairly constant during the cooling process. Becher *et al.*⁶ have however shown that the value of the surface heat transfer coefficient may vary by as much as two orders of magnitude for quenches of alumina from temperatures in the range 200–300°C into water at room temperature. This phenomenon was attributed to the boiling conditions at the specimen surface,

*To whom correspondence should be addressed.

which decide the value of h , and these conditions are strongly dependent on the temperature of the specimen surface. Becher⁷ proposed that boiling water should give more uniform heat transfer conditions. Other quenching media such as fluidized beds,^{8,9} silicone oil¹⁰ and liquid nitrogen¹¹ have also been investigated previously. A general drawback with these is however that the value of h is so low during the quench that the thermal stresses are too low to induce thermal shock fracture in many high performance ceramics.¹² On the other hand, boiling water gives an intermediate value of h .⁷

Experimentally, thermal shock resistance is generally evaluated by measuring the residual strength after quenching^{13,14} or by measuring the effect of a quench on other material properties.^{15,16} These tests often require a specific geometry and size for the test specimen. Furthermore, many specimens have to be tested in order to assure statistical significance.^{16,17} This makes testing costly and time consuming. In a previous investigation a new method was developed for the determination of the thermal shock resistance of ceramics.¹⁸ The thermal shock resistance was measured by studying the propagation of small Vickers indentation precracks. The ΔT_c of the material was defined with reference to the number of propagating cracks and the amount of crack growth. Since a small number of specimens containing a relatively large number of indentations can be studied, the amount of material required is very small. Another attractive feature is that the method does not require any specific specimen geometry. The method can thus be applied directly to components.

In this paper the indentation/quench method¹⁸ for determining the thermal shock resistance of ceramic materials is used to study the effect of rapid cooling in boiling water for seven different ceramic materials. The thermal shock resistance of the materials is studied by measuring the extension of small Vickers indentations. The resistance to thermal cycling is also evaluated for two of the materials. The results from quenches into boiling water are compared to those determined from quenches into water at room temperature.

2 Experimental Approach

The thermal shock resistance, ΔT_c , was experimentally determined for seven different materials: (1) Al₂O₃-22.5 wt% TiN/7.5 wt% TiC, (2) Al₂O₃-11 wt% ZrO₂, (3) Al₂O₃-25 wt% SiC whiskers, (4) 99.7% pure Al₂O₃, (5) α -SiC, (6) 97.5% dense MgO containing 2 wt% Y₂O₃ and (7) fused silica. The samples of materials (1)–(3) were in the form of plates 12.7 mm square \times 4.0 mm thick, while

materials (4)–(7) were plates 10 mm square \times 4.0 mm thick. The specimens were polished with 3 μ m diamond in an automatic polishing machine (Prepamatic, Struers A/S, Denmark). This resulted in a well-defined surface finish, which allowed measurement of the precracks before and after quenching. The specimens were precracked through indentation with a Vickers diamond. The indentation load was chosen so that indentation crack sizes in the range 100 to 150 μ m were achieved for each material. This size of the precracks was chosen in order to facilitate comparisons with a previous study.¹⁸ Individual specimens were heated by inserting them into a preheated furnace (air atmosphere) and holding for 15 min before quenching by free fall into a bath of boiling water. The maximum achievable temperature of the furnace was 1300°C. The thermal shock resistance of the materials could thus be tested up to a temperature difference, ΔT , of 1200°C.

The indentation crack length was measured before and after thermal shock with an optical microscope. In those cases where oxidation occurred on the specimen surfaces, the oxide layer was polished away before measurement. The ΔT_c of the materials was defined as the lowest temperature difference where two conditions were simultaneously fulfilled: (a) the average extension of the cracks was more than 10% of the original length and (b) more than 25% of the indentation cracks propagated. This definition was postulated in a previous study.¹⁸ In short, criterion (a) ensures that the crack extension is larger than the measurement error of the optical microscope while criterion (b) eliminates chance growth of single cracks and provides a statistically significant number of growing cracks.

The approach was first to make a preliminary estimate of the ΔT_c value for each material, then to gather statistical data by quenching additional specimens at the nominal ΔT_c . In the third step the effect of 10 thermal cycles was determined for the Al₂O₃-TiN/TiC and the Al₂O₃-ZrO₂ materials. These two materials were chosen, since these had shown similar thermal cycling resistance in a previous study.¹⁸ The tests were concluded by heating the materials to a temperature of their respective $\Delta T_c + 100^\circ\text{C}$ and then quenching them into air at room temperature to verify that no thermally assisted crack propagation occurred during the holding time in the furnace.

3 Results and Discussion

The relevant properties of the materials and calculated values of R and R' [from eqn (2)] are listed in

Table 1. Table 2 shows the experimentally determined ΔT_c 's and the crack growth statistics from single quenches of the materials into boiling water. Before looking at these data it is important to notice that none of the materials showed crack propagation after a quench from a temperature of their $\Delta T_c + 100^\circ\text{C}$ to air at room temperature. It can thus be concluded that no crack growth occurred during the heat treatment of the specimens. Furthermore, for all the materials, the crack propagations and extensions were very small at ΔT 's that were 20°C lower than the experimentally determined ΔT_c values. This eliminates slow crack growth as the crack propagation mechanism. On the other hand, it indicates that the values of ΔT_c are significant to 20°C .

The experimental values of ΔT_c will be compared to the calculated values of the parameters R and R' , in order to evaluate the effect of the different quenching media. A short description of these parameters is therefore in order. Equation (1) shows that R represents the most severe case of thermal shock (infinite h). R is thus the lowest temperature difference at which the material will fracture. With decreasing values of h , the value of ΔT_c will gradually increase. How large this increase will be is decided by the value of R' . Valuable information about the cooling conditions can thus be gained by comparing the ranking of the materials from the analytical values of R and R' with the ranking from the respective experimental values of ΔT_c .

The materials can be divided into three classes with regard to their experimentally determined ΔT_c 's. The class with superior thermal shock resistance comprises $\text{Al}_2\text{O}_3/\text{SiC}$ whiskers, SiC and fused silica, the class with intermediate thermal shock resistance includes $\text{Al}_2\text{O}_3\text{-TiN/TiC}$, $\text{Al}_2\text{O}_3\text{-ZrO}_2$ and Al_2O_3 while the class with poor thermal shock resistance contains MgO. Fused silica is well known for having a low value of thermal expansion coefficient, α . The low value of α gives small thermal strains and hence low thermal stresses even in the case of quenches from large ΔT 's. This leads to a very good thermal shock resistance. Fused silica also has a very low value of the thermal conductivity and it is this which leads to the low value of R' , see Table 1. In this study, the highest ΔT (1200°C) at which fused silica was tested was lower than the value of R . This was reflected in the experiments in that no crack propagation could be seen, and thus $\Delta T_c > 1200^\circ\text{C}$. The effect of the R' parameter on the thermal shock resistance of fused silica could thus not be seen from these experiments.

As seen in Table 1, $\text{Al}_2\text{O}_3\text{-SiC}$ and SiC have fairly low values of R . The very high values of ΔT_c of the $\text{Al}_2\text{O}_3\text{-SiC}$ and SiC, compared to those of $\text{Al}_2\text{O}_3\text{-TiN/TiC}$ and $\text{Al}_2\text{O}_3\text{-ZrO}_2$ suggest a ranking relative to R' rather than to R , for quenches into boiling water. This has also been stressed by Becher *et al.*¹⁹ The superior thermal shock resistance of Al_2O_3 reinforced with SiC whiskers ($\Delta T_c = 1200^\circ\text{C}$) and $\alpha\text{-SiC}$ ($\Delta T_c > 1200^\circ\text{C}$) can

Table 1. Properties of materials and calculated thermal shock resistance parameters

Material	σ_f (MPa)	E (GPa)	α ($10^{-6} \text{ }^\circ\text{C}^{-1}$)	ν	k ($\text{Wm}^{-1} \text{ }^\circ\text{C}^{-1}$)	R ($^\circ\text{C}$)	R' (Wm^{-1})
$\text{Al}_2\text{O}_3\text{-TiN/TiC}$	550	398	8.6	0.21	24	127	3050
$\text{Al}_2\text{O}_3\text{-ZrO}_2$	675 ^a	380	8.7	0.25	12	153	1840
$\text{Al}_2\text{O}_3\text{-SiC}_w$	700 ^a	397	6.0	0.25	33	220	7270
Al_2O_3	300	370	7.8	0.25 ^b	26	78.0	2027
$\alpha\text{-SiC}$	612	427	4.5	0.14 ^b	110	274	30140
MgO	96 ^c	317 ^c	13 ^c	0.29 ^c	41 ^c	16.5	677
Fused silica	60 ^d	73 ^d	0.54	0.17 ^d	1.4 ^d	1324	1854

All data from manufacturer or as measured except: ^a from Ref. 19 for a similar material; ^b from Ref. 25; ^c from Ref. 26; ^d From Ref. 27.

Table 2. Experimentally determined ΔT_c values, total number of cracks, crack growth statistics and indentation loads for the seven materials

Material	ΔT_c ($^\circ\text{C}$)	Total number of cracks	Extension (%)	Propagation (%)	Indentation load (N)
$\text{Al}_2\text{O}_3\text{-TiN/TiC}$	120	16	25.3	62	70
$\text{Al}_2\text{O}_3\text{-ZrO}_2$	100	16	12	31	70
$\text{Al}_2\text{O}_3\text{-SiC}_w$	1200	32	10.7	31	70
Al_2O_3	100	32	39	41	35
$\alpha\text{-SiC}$	> 1200	36	7.2 ^a	14 ^a	70
MgO	60	32	22.8	50	30
Fused silica	> 1200	16	0 ^a	0 ^a	40

^aMeasured for $\Delta T = 1200^\circ\text{C}$

then be explained as arising from the effect of the high value of the thermal conductivity of SiC. Looked at from another point of view, quenching into boiling water is sufficiently slow that the thermal conductivity of the specimen is very important. Comparing a high and a low value of the thermal conductivity, the higher value means that the temperature distribution through the specimen stays more uniform during the cooling, which in turn leads to lower thermal stresses and a higher thermal shock resistance.

Another factor which has to be considered in these two materials is the oxidation of the SiC whiskers on the specimen surfaces. After a thermal cycle to 1300°C the specimens were covered with a thin oxide layer, which had to be polished away before measuring the crack extension and propagation. Hansson *et al.*²⁰ reported that the fracture toughness increases in the temperature region 1200–1300°C for a SiC whisker reinforced Al₂O₃ similar to that tested in this investigation. This effect was attributed to crack tip blunting, associated with the glass formation in the crack tip region. Crack tip blunting may thus contribute to the high thermal shock resistance of the materials containing SiC. In the present investigation cracking from the edges occurred in the SiC whisker reinforced Al₂O₃ at a ΔT of 1170°C, which is 30°C lower than the ΔT_c determined from the extension and the propagation of the precracks. For quenching into water at room temperature the same material showed cracking from the edges at a temperature which was approximately 40°C above the ΔT_c (=200°C) determined from the growth of the precracks. This difference could be accounted for if the surface is strengthened by oxidation.

Another effect of the oxide layer has been reported by Chiu,²¹ who investigated the effect of a heat treatment at 1450°C for 2 h and for 85 h on the thermal shock resistance of a similar material. A main conclusion in Chiu's study was that the oxide layer decreases the surface heat transfer coefficient, h , leading to lower thermal stresses and thus an increase in the ΔT_c of the material. Chiu's study was at temperatures well above those used in this investigation, but the presence of an oxide phase will probably have the same effect on the value of h also at lower temperatures.

If these two effects are significant, they will lead to the condition that, if the SiC materials can stand quenches from the lowest temperature, T_{ox} where the oxide layer covers the specimen surface, the materials will also be able to stand quenches from much higher temperatures.

In the category with intermediate thermal shock resistance materials, Al₂O₃-TiN/TiC showed the best performance with a ΔT_c value of 120°C, while

Al₂O₃-ZrO₂ and Al₂O₃ both had a ΔT_c of 100°C. Since the difference in ΔT_c was small and the three materials were polished to the same surface finish (the surface roughness has a large influence on the h value) it is reasonable to assume that the three materials experienced a quench at similar value of h . Considering the values of R and R' for Al₂O₃-TiN/TiC, Al₂O₃-ZrO₂ and Al₂O₃ in Table 1, a ranking from the value of R indicates that Al₂O₃-ZrO₂ should show slightly better performance than Al₂O₃-TiN/TiC, with monolithic Al₂O₃ in a clear third place, while a ranking from the value of R' would support Al₂O₃-TiN/TiC, with monolithic Al₂O₃ as second and Al₂O₃-ZrO₂ as third (the low value of R' for Al₂O₃-ZrO₂ is due to the low value of k , stemming from the low thermal conductivity of the ZrO₂ particles). The fact that Al₂O₃ and Al₂O₃-ZrO₂ get the same ranking in the experiments suggests, again, that the quenching conditions are so mild that the influence of the R' parameter (and thus the value of thermal conductivity) is substantial. This influence of the R' parameter would also explain why Al₂O₃-TiN/TiC is better than the other two materials.

Finally, MgO has the lowest value of R and R' of the seven materials, due principally to the combination of low strength and high thermal expansion coefficient. It is thus not surprising that the MgO material has the lowest value of ΔT_c in the experiments.

It is now of value to compare the values of ΔT_c of the Al₂O₃-TiN/TiC, Al₂O₃-ZrO₂ and Al₂O₃-SiC whiskers materials obtained from quenches into boiling water, with those determined from quenches into water at room temperature in a recent study, see Table 3. The results show that the difference between the ΔT_c 's of Al₂O₃-SiC whiskers and the other two materials have become much smaller. Furthermore, the ΔT_c of Al₂O₃-ZrO₂ has increased by 20°C, which results in an equal ranking for Al₂O₃-TiN/TiC and Al₂O₃-ZrO₂. The ranking order of the materials as well as the quantitative values of ΔT_c for the materials indicates that the R parameter has a large influence on the results from quenches into water at room

Table 3. The experimentally determined thermal shock resistances, ΔT_c , for quenching indented test specimens into boiling water. The results are compared with those obtained for quenching into water at room temperature on a previous study.¹⁸

Material	ΔT_c (°C)	ΔT_c (°C)
	Quench into water at 100°C	Quench into water at 20°C
Al ₂ O ₃ -TiN/TiC	120	120
Al ₂ O ₃ -ZrO ₂	100	120
Al ₂ O ₃ -SiC _w	1200	200

temperature. This would follow from a higher value of h . Assuming the same h value for all the three materials and temperature independent properties, a larger value of h should however also lead to a decrease in the ΔT_c 's for all the materials, which is not the case in the experimental results. There are two possible explanations for this discrepancy between theory and experiment: (1) The temperature dependence of the material properties and (2) The specimen size dependence of ΔT_c .

Consider first the temperature dependence of the properties. When comparing equal ΔT 's from quenches into boiling water and water at room temperature, it is important to notice that the initial specimen temperature is 80°C higher for quenches into boiling water. If any of the material properties of importance for thermal shock are temperature sensitive in the temperature range 20–240°C this will influence the comparison. Since the thermal conductivity, k , of Al₂O₃ is very dependent on the temperature,²² the decrease in k over the temperature range 20–240°C was calculated from literature values for Al₂O₃–TiN/TiC²³ and Al₂O₃–ZrO₂.²⁴ Although these data are not for the exact same materials, the calculations for Al₂O₃–TiN/TiC show a decrease in k of 23%, while the calculations for a material similar to the Al₂O₃–ZrO₂ show a decrease in k of 27%. This indicates that a lower value of k should be used for the quenches into boiling water. This, in turn, would explain why the ΔT_c is not increased for Al₂O₃–TiN/TiC and Al₂O₃–ZrO₂ when these are quenched into boiling water. The effects of a lower value of k will counteract the effects of the milder quenching conditions, which will lead to a ΔT_c of the same level as when quenching into water at room temperature.

The second explanation has also been provided by Becher and Warwick.¹⁹ They showed that Al₂O₃ and Al₂O₃–SiC can stand thermal shocks up to a ΔT of 1200°C when quenched into boiling water. This requires that the specimen thickness is below a certain critical thickness, t_c . Furthermore they showed that the ΔT_c values are approximately 200°C for Al₂O₃–SiC for thicknesses which are larger than t_c . This increase in ΔT_c of the material can qualitatively be explained from the size dependence of ΔT_c in eqn (1). At equal ΔT 's, thinner specimens will see smaller thermal stresses than thicker ones. Equation (1) also shows that this effect becomes more pronounced at slower cooling rates and for higher values of R' . The size effect can thus be expected to be large for Al₂O₃–SiC (high value of R'). The very sharp increase in ΔT_c for thicknesses below t_c (10 mm thickness: $\Delta T_c = 200^\circ\text{C}$, 6 mm thickness: $\Delta T_c = 1200^\circ\text{C}$)¹⁹ cannot be explained from eqn (1), unless the value of h is

assumed to decrease for thicknesses below t_c (eqn (1) predicts a more gradual increase of ΔT_c). The decrease in value of h can be explained as follows. In certain temperature ranges the value of h is strongly dependent on the temperature at the specimen surfaces.⁶ With decreasing specimen thicknesses the ΔT necessary to fracture the specimens will increase. A higher ΔT also means higher initial specimen temperatures. For thicknesses $< t_c$, the initial specimen temperature corresponds to a range of temperatures, where the value of h shows a drastic decrease with increasing temperature.

Comparing the experimental results for single thermal quenches in the present study with Becher *et al.*'s experimental result for a similar Al₂O₃–SiC material,¹⁹ the value of t_c for Al₂O₃–SiC was approximately 10 mm, which is much larger than the specimen size of 4 mm in the present study. For quenches into water at room temperature, Becher *et al.* reported ΔT_c values of approximately 200°C for 4 mm thick specimens. Becher *et al.*'s values of ΔT_c for Al₂O₃–SiC are thus similar to the results from the present investigation.

The final evaluation considered the resistance of Al₂O₃–TiN/TiC and Al₂O₃–ZrO₂ to 10 thermal quenches into boiling water at $\Delta T = 100^\circ\text{C}$. From the single quenches it may be remembered that this ΔT is 20°C below the experimentally determined ΔT_c of Al₂O₃–TiN/TiC, while it corresponds to the experimentally determined ΔT_c of Al₂O₃–ZrO₂. The results, which are shown in Table 4, revealed a large difference in resistance to thermal cycling of the two materials. Despite a lower ΔT_c , Al₂O₃–ZrO₂ had the best thermal cycling resistance. The percentage of cracks that had propagated in Al₂O₃–ZrO₂ increased from 31% to 81% when going from a single quench to 10 thermal cycles, while the average crack extension increased from 10.8% of the original length to 17%. These values indicate a good resistance to thermal cycling. Looking at the performance of Al₂O₃–TiN/TiC, the number of cracks, which had propagated after thermal cycling was the same (13 out of 16 cracks extended) as that for Al₂O₃–ZrO₂. The crack extension in Al₂O₃–TiN/TiC is, however, large. After thermal cycling, 3 of the 16 cracks had extended from approximately 0.14 mm to a length of more than 1 mm, one crack had extended to a

Table 4. Effect of a single quench (Q) and 10 repeated thermal quenches (10Q) in water at 100°C at a ΔT of 100°C. (Total number of cracks: 16)

Material	ΔT (°C)	% Propagation		% Extension	
		Q	10Q	Q	10Q
Al ₂ O ₃ –TiN/TiC	100	50	81	9.1	1000
Al ₂ O ₃ /ZrO ₂	100	31	81	10.8	17

size of approximately 0.5 mm, while the remaining seven showed extensions to 0.16–0.25 mm. Al₂O₃–TiN/TiC was thus more sensitive to thermal cycling than Al₂O₃–ZrO₂.

Discussing the reason for this, it is first of value to compare the results from thermal cycling in boiling water, in Table 4, with those from thermal cycling in water at room temperature. As seen in Table 5, the amount of crack extension and crack propagation after 10 thermal cycles in water at room temperature was smaller for both the materials. Furthermore, the ranking of the two materials was the same in the two quenching media. If the quenches into boiling water at a specific ΔT are considered as milder, then the thermal stresses will be lower, as compared to those from quenches into water at room temperature. Another effect is that the thermal stress duration will be longer. Considering the thermal stress duration, the different effects from the two quenching media and the difference in resistance to thermal cycling of the two materials can be explained with reference to the particulate reinforcements. The ZrO₂ phase in Al₂O₃–ZrO₂ is finely dispersed. A propagating crack in Al₂O₃–ZrO₂ will thus meet a rising toughness (from transformation toughening) no matter which way it takes through the Al₂O₃–ZrO₂ matrix. This will lead to little crack propagation in Al₂O₃–ZrO₂. Considering Al₂O₃–TiN/TiC, the particulates and the distance between the particulates are large, thus providing relatively free routes for cracks to propagate. The short duration of the thermal stress pulse from quenches into water at room temperature will cause the cracks to be retarded when they meet a particulate. The thermal stress decreases rapidly and the crack is trapped. In contrast, the longer time duration from quenches into boiling water will give the cracks time to choose the easiest way through the matrix and in this way they will bypass the particulates. This was seen for Al₂O₃–TiN/TiC, when investigating the crack path through thermal cycled specimens. These findings suggest that a well-dispersed second phase is more effective in resisting the propagation of cracks, when a ceramic is cycled close to its value on ΔT_c .

Before ending this discussion, some comments about the experimental technique are in order. In

Table 5. Effect of a single quench (Q) and 10 repeated thermal quenches (10Q) in water at 20°C at a ΔT of 100°C. (Total number of cracks: 32). Results from Ref. 18

Material	ΔT (°C)	% Propagation		% Extension	
		Q	10Q	Q	10Q
Al ₂ O ₃ –TiN/TiC	100	25	50	6.1	19.9
Al ₂ O ₃ /ZrO ₂	100	3	12	6.7	6.6

this investigation few test specimens were used for each material. In fact, three specimens were sufficient to determine the ΔT_c of each of the materials. The statistical significance was ensured by studying at least 16 cracks in each specimen. The validity of this was shown in a previous study.¹⁸ The size of the precracks was chosen so that comparisons with data from quenches into water at room temperature were possible. Comparisons with experimental data on similar materials containing only natural flaws do however show that the precracks do not change the relative ranking of the materials.¹⁸ Furthermore, the trends of the results are in good agreement with estimations from analytical expression, such as the R and R' parameters.

4 Conclusions

The new indentation quench technique has been used to determine the thermal shock resistance and the resistance to 10 thermal cycles for several ceramic materials. The quenching medium was boiling water. Since statistical significance was obtained by using many cracks in each specimen, few test specimens were needed. Another advantage of having precracks with a narrow size distribution was that the trends in the results are clearly defined. The ranking of the materials from their specific values of ΔT_c was furthermore shown to be in good agreement with existing theories of the initiation of thermal shock fracture.

A particular result worth noticing is the high value of ΔT_c from quenches into boiling water of the SiC whisker reinforced Al₂O₃ material. This should be compared to a ΔT_c value of 200°C for quenches into water at room temperature, using the same material, the same specimen thickness (4 mm) and the same indentation quench technique. The higher value of ΔT_c for quenches into boiling water was mainly attributed to the fact that the thermal conductivity becomes of importance for milder quenches, such as those into boiling water. A second factor that was considered was the effect of surface oxidation and glass formation at the high temperatures (1300°C) involved in the ΔT_c determination. This was indicated by the fact that the specimens showed macrocracks from the edges when quenched from ΔT 's just below the determined value of ΔT_c . For quenches into water at room temperature these macrocracks appeared approximately 40°C above that ΔT_c . This showed that surface strengthening effects are of importance at high temperatures.

The ranking of the materials in the thermal cycling experiments was the same for boiling water quenches as for quenches into water at room

temperature. The crack extensions were considered considerably larger for the quenches into boiling water, especially for $\text{Al}_2\text{O}_3\text{-TiN/TiC}$ which showed crack extensions of more than 1 mm. This can be compared to the less dramatic crack extensions of approximately $20\ \mu\text{m}$ of the $\text{Al}_2\text{O}_3\text{-ZrO}_2$. The larger crack extension after thermal cycling in boiling water is a consequence of the milder quenching conditions and longer duration times of the thermal stress peaks. This, in turn, gives the cracks longer times to advance through the matrix. The large difference between $\text{Al}_2\text{O}_3\text{-TiN/TiC}$ and $\text{Al}_2\text{O}_3\text{-ZrO}_2$ was attributed to the finer dispersion of particles in $\text{Al}_2\text{O}_3\text{-ZrO}_2$ compared with the large particulates in $\text{Al}_2\text{O}_3\text{-TiN/TiC}$. The relatively large distances between the TiN and TiC particulates makes it possible for the cracks to avoid them and to propagate long distances through alumina grains in the matrix. Further, the fairly long duration times of the stress peaks for quenches into boiling water gives the cracks time to choose the easiest way through the matrix.

Finally, this investigation shows that thermal shock resistance evaluation becomes more fruitful when the materials are evaluated for at least two different quenching rates. By comparing the ranking of the materials at different quenching rates much additional information can be gained regarding the role of the material properties at any specific quenching rate.

References

- Kingery, W. D., Factors affecting thermal stress resistance of ceramic materials. *J. Am. Ceram. Soc.*, 1955, **38**(1), 3–15.
- Hasselmann, D. P. H., Thermal stress resistance of engineering ceramics. *Mater. Sci. and Eng.*, 1985, **71**, 251–254.
- Buessem, W., Ring test and its application to thermal shock problems. O. A. R. Report, Wright-Patterson Air Force Base, Dayton, OH, 1950.
- Manson, S. S., Behaviour of materials under conditions of thermal stress. N.A.C.A. Technical Note 2933, Washington, DC, 1953.
- Manson, S. S., Thermal stresses, Part 1—Appraisal of brittle materials. *Mach. Des.*, 1958, **30**, 114–120.
- Becher, P. F., Lewis III, D., Carman, K. R. and Gonzalez, A. C., Thermal shock resistance of ceramics: size and geometry effects in quench tests. *Am. Ceram. Soc. Bull.*, 1980, **59**(5), 542–545, 548.
- Becher, P. F., Effect of the water temperature on the thermal shock of Al_2O_3 . *J. Am. Ceram. Soc.*, 1981, **64**(1), C17.
- Niihara, K., Singh, J. P. and Hasselmann, D. P. H., Observations on the characteristics of a fluidized bed for the thermal shock testing of brittle ceramics. *J. Mater. Sci.*, 1982, **17**, 2553–2559.
- Glenny, E., Northwood, J. E., Shaw, S. W. K. and Taylor, T. A., A technique for thermal shock and thermal fatigue testing based on the use of fluidized solids. *J. Inst. of Metals*, 1985, **87**, 294–302.
- Singh, J. P., Tree, Y. and Hasselmann, D. P. H., Effect of bath and specimen temperature on the thermal stress resistance of brittle ceramics subjected to thermal quenching. *J. Mater. Sci.*, 1981, **16**, 2109–2119.
- Lee, W. J., Kim, Y. and Case, E. D., The effect of quenching media on the heat transfer coefficient of polycrystalline alumina. *J. Mater. Sci.*, 1993, **28**, 2079–2083.
- Lewis III, D., Thermal shock and thermal shock fatigue testing of ceramics with the water quench test. In *Fracture Mechanics of Ceramics 6*, ed. R. C. Bradt, A. G. Evans, D. P. H. Hasselmann and F. F. Lange. Plenum Press, New York, 1983, pp. 487–496.
- Hasselmann, D. P. H., Strength behaviour of polycrystalline alumina subjected to thermal shock. *J. Am. Ceram. Soc.*, 1970, **53**(9), 490–495.
- Ashizuka, M., Easler, T. E. and Bradt, R. C., Statistical study of thermal shock damage of a borosilicate glass. *J. Am. Ceram. Soc.*, 1983, **66**(8), 542–550.
- Coppola, J. A. and Bradt, R. C., Thermal shock damage in SiC. *J. Am. Ceram. Soc.*, 1973, **56**(4), 214–218.
- Huang, H. Y. and Singh, R. N., Thermal shock behaviour of fiber reinforced composites. *Ceram. Eng. & Sci. Proc.*, 1994, **15**(4), 673–683.
- Rogers, W. P., Emery, A. F., Bradt, R. C. and Kobayashi, A. S., Statistical study of ceramic materials in the water quench test. *J. Am. Ceram. Soc.*, 1987, **70**(6), 406–412.
- Andersson, T. and Rowcliffe, David J., Indentation thermal shock test for ceramics. *J. Am. Ceram. Soc.*, 1996, **79**(6), 1509–1514.
- Becher, P. F. and Warwick, W. H., Factors influencing the thermal shock behaviour of ceramics. In *Thermal Shock and Thermal Fatigue Behaviour of Advanced Ceramics*, ed. G. A. Schneider and G. Petzow. Kluwer Academic Publishers, Netherlands, 1993, pp. 37–48.
- Hansson, T., Swan, A. H. and Warren, R., High temperature fracture of SiC-whisker reinforced alumina in air and vacuum. *J. Euro. Cer. Soc.*, 1994, **13**(5), 427–436.
- Chiu, C. C., Influence of surface oxidation on thermal shock resistance and flexural strength of SiC/ Al_2O_3 composites. *J. Mater. Sci.*, 1994, **29**, 2078–2082.
- Dörre, E. and Hübner, H., Alumina-processing, properties and applications. In *Materials Research and Engineering*, ed. B. Ilschner and N. J. Grant. Springer-Verlag, Heidelberg, 1984, pp. 14–15.
- Brandt, G., Thermal shock resistance of ceramic cutting tools. *Surf. Eng.*, 1986, **2**(2), 121–131.
- Greve, D., Claussen, N. E., Hasselmann, D. P. H. and Youngblood, G. E., Thermal diffusivity/conductivity of alumina with a zirconia dispersed phase. *Am. Ceram. Soc. Bull.*, 1977, **56**(5), 514–515.
- Kelly, A. and Macmillan, N. H., *Strong Solids*, 3rd edn. Clarendon Press, Oxford, 1986, p. 392.
- Brett, N. H., Magnesium and alkaline-earth oxides. In *Concise Encyclopaedia of Advanced Ceramic Materials*, ed. R. J. Brook. Pergamon Press, Oxford, 1991.
- Ceramics and Glasses, Engineered Materials Handbook*, Vol. 4. ASM International, USA, 1991, p. 1018.