Influence of the Size Factor on the Thermal Shock Resistance of Ceramic Samples

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The cracking of brittle samples having suffered thermal shocks may be calculated by using the thermoelastic theory. This theory leads to a size effect, which is not always verified by experiments. The use of acoustic methods of characterization, for samples of various shapes and sizes, shows the size effect, but the experimental ΔT_c values are greater than the calculated ones. This discrepancy must be due both to an overestimation of the A coefficient and an underestimation of the R parameter.

1. INTRODUCTION

Because of their high thermomechanical properties, "structural ceramics" can be used in thermal engines to improve their efficiency. But such applications require a rigorous choice between various materials, and call for an accurate characterization of their properties. The pieces work in an atmosphere which may be corrosive, and suffer a high level of thermome-chanical stresses. Consequently, structural ceramics must be able to resist corrosion (O_2, CO, CO_2, H_2O . . .), their strength at high temperatures must be high, creep sensitivity must be low, and thermal shock resistance must be good. It is hoped that ceramics will be substituted for metallic alloys in Diesel engines and gas turbines [1].

The thermal shock resistance is an important parameter, because operation of thermal engines leads to variations in temperature (between room temperature and the operating temperature). Heat exchanges are usually due

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to gaseous convection, which induces rather low temperature variations and causes mild thermal shocks. However, such mild thermal shocks are not easily reproducible, and studies on the thermal shock resistance of materials are generally performed by using severe thermal shocks (mainly water quenching) [2]. For samples heated at a temperature T_1, then cooled in a liquid bath at a temperature T_2, the measurement of the strength σ of the shocked samples shows a curve $\sigma = f(\Delta T)$, which gives informations about the critical temperature difference ΔT_c (and thus about the resistance to cracking) and about the σ'/σ'' ratio of the strength before and after the shock. This ratio characterizes the resistance to damage [3,4].

Resistance to damage is the main parameter for refractories, whereas the first requirement for structural ceramics is the resistance to cracking. This is why it is important to determine the maximum temperature difference a piece can stand. This requires taking into consideration the size effect which states that the critical temperature decreases as the volume of the sample increases [5]. It is important to know the influence of the size effect for the following reason: the results obtained from laboratory experiments performed on small samples must be applicable to and used for industrial pieces, which may be much larger. The aim of the present study is to evaluate, for a given material, the critical temperature difference for samples of various shapes and sizes; the results are then discussed in the frame of the thermoelastic theory of thermal shocks [6].

2. MATERIALS, SAMPLES, EXPERIMENTAL PROCEDURE

2.1. Materials

All the samples used were made of 99.5% pure alpha alumina (Degussit Al 23), almost perfectly dense.

2.2. Samples

Samples of four different shapes have been used: prismatic bars, long cylinders, discs, and short cylinders. The prismatic bars (4 × 4 × 25 mm) were obtained by sawing rectangular plates 25 × 50 mm. The samples were machined, then diamond ground. The long cylinders (6 mm diam, 80 mm long) were used in the as received condition. The discs (30 mm diam, 2.5 mm thick; and 30 mm diam, 4.5 mm thick) were machined from discs of greater size (32 mm diam, 5 mm thick). The parallel faces were diamond ground. The short cylinders (20 mm diam, 20 mm long) were prepared by sawing a cylindrical bar 20 mm diam, 500 mm long. The parallel faces were machined up to a parallelism of better than 1 μm.

2.3. Experimental Procedure

2.3.1. Thermal Shocks

The samples were heated for 1 hr in a furnace at a temperature T_1, then quenched and stirred in water at 20°C ($\Delta T = (T_1 - 20°C)$), then dried for 15 min at 105°C.

2.3.2. Destructive Measurements of ΔT_c

Strength after thermal shocks was measured by three-point bending (21 mm span) on the prismatic bars, by three-point bending (60 mm span) on the long cylinders, and by biaxial flexure on the discs [7]. For the prismatic bars, the corners of the face in tension had been worn out.

2.3.3. Nondestructive Measurements of ΔT_c

Two dynamical methods, at medium and high frequencies, have been used [8] to determine ΔT_c by a nondestructive method. For the medium frequency method (~10 kHz), the resonant frequencies and the damping capacity of vibrating cylinders or vibrating discs were measured [9, 10]. These parameters were modified when $\Delta T > \Delta T_c$, and it has been pointed out [11] that the modifications are due to cracking of the sample. For the high frequency method (~10 MHz), the velocities of the shear and longitudinal waves and the acoustic attenuation were measured using either phase comparison or pulse echo overlap [12]. The velocities were changed only slightly by the shocks, contrary to the acoustic attentution, which dramatically increases at $\Delta T = \Delta T_c$, because of the scattering of acoustic waves due to the cracks. Since all the measurements are nondestructive, it is possible to operate by either cumulative or noncumulative shocks; there seems to be no thermal fatigue when there are a few shocks only [13].

2.3.4. Dye Penetrant Method

Because the Al 23 alumina is dense, a dye penetrant method permits observation of the extent of cracking.

3. RESULTS

For the prismatic beams, Fig. 1 shows the variations of strength versus the temperature difference, and Fig. 2 gives the variations of standard deviation: it shows that the scattering is maximum when $\Delta T = \Delta T_c$ [14] and that another peak is located at ΔT_c, which corresponds to the quasi-static propagation of cracks [15]. For the long cylinders broken by bending, the
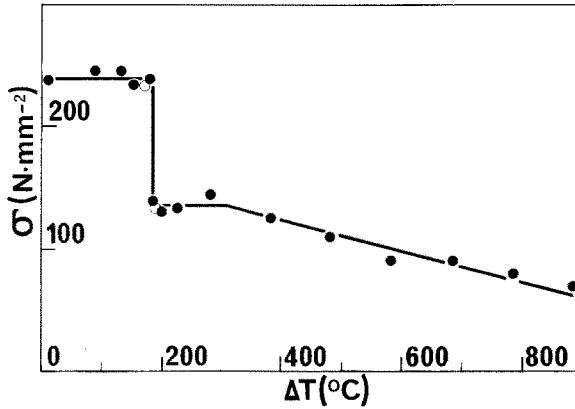
Fig. 1. Strength versus temperature difference for prismatic
bars.

results are similar. Figure 3 shows the variations of the damping capacity for
noncumulative shocks. Figures 4 and 5 show the variations of resonance
frequencies of the 30 mm diam, 2.5 mm thick discs, for cumulative and
noncumulative shocks. They show that ΔT_c is the same in both cases. For 30
mm diam, 4.5 mm thick discs, the curve is similar, but ΔT_c is different.

For the biaxial flexure of discs, the variations of strength are not
important, which makes it difficult to determine ΔT_c accurately [16]. In the
case of diamond ground discs, Fig. 6 shows that $\sigma''/\sigma' > 0.85$; in the case of
coarsely ground discs, Fig. 7 shows that σ remains constant, equal to 200 N ·
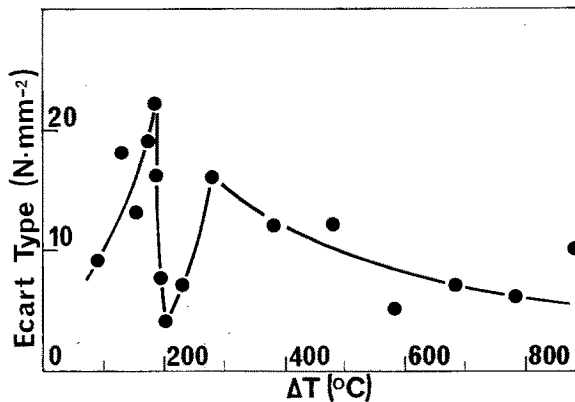mm^{-2} (instead of 250 N · mm^{-2} for the diamond ground discs). This lowering

Fig. 2. Standard deviation of strength versus temperature
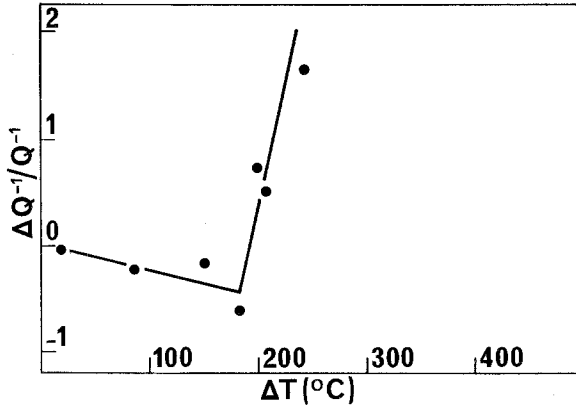difference for prismatic bars.

Fig. 3. Damping capacity of vibrating 6 (diam) × 80 mm bars versus temperature difference, for *noncumulative* shocks.

of the strength is due to machining flaws, which appear to be more critical than the thermal cracks. In such cases, the destructive measurements of strength are not suitable to study the resistance to cracking, but the nondestructive measurements of elastic constants always give accurate data (Fig. 8 shows unambiguously the value of ΔT_c).

Figure 9 shows the variations of the damping capacity for small cylinders. Such samples would require compression tests [14], which are not accurate, to determine their strength; as a result, the acoustic method is preferable. The dye penetrant test shows cracking of the samples (Figs. 10–12). A summary of the results is given in Table I.
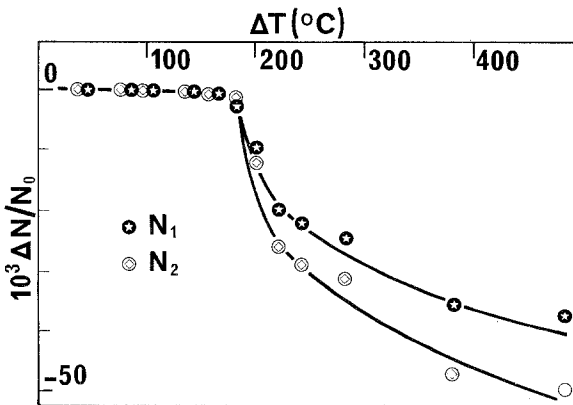

Fig. 4. Damping capacity of vibrating 30 (diam) × 2.5 mm discs versus temperature difference, for *cumulative* shocks (N_1 and N_2: first and second overtones).
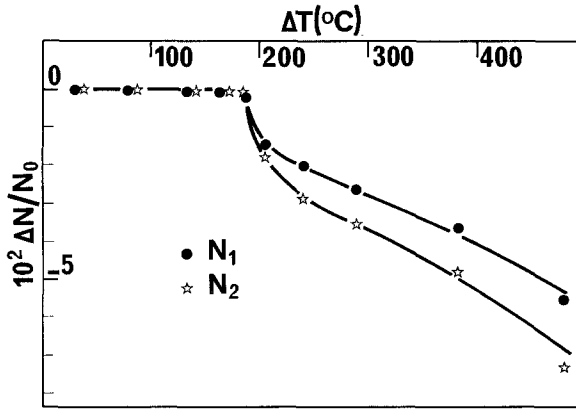
$$\Delta T(^{\circ}C)$$

Fig. 5. Damping capacity of vibrating 30 (diam) × 2.5 mm discs versus temperature difference, for *noncumulative* shocks (N_1 and N_2: first and second overtones).

4. DISCUSSION

The results show that ΔT_c decreases as the volume of the sample increases, but only in the case of the most voluminous samples (short cylinders). ΔT_c is about 180°C for small specimens (independent of their shape); the thick discs give $\Delta T_c \sim 165$°C; the massive cylinders give $\Delta T_c \sim 105$°C. The above results may be compared with the data in the literature concerning the thermal shock behavior of polycrystalline alumina. For samples comparable with the bars used in the present study, ΔT_c is about 180–190°C [16–18], and it is noted that the size effect is obvious in the
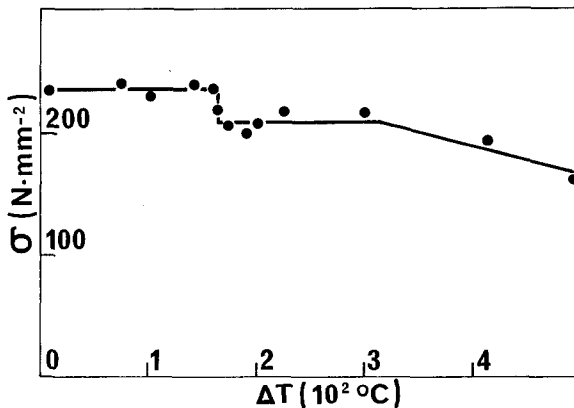

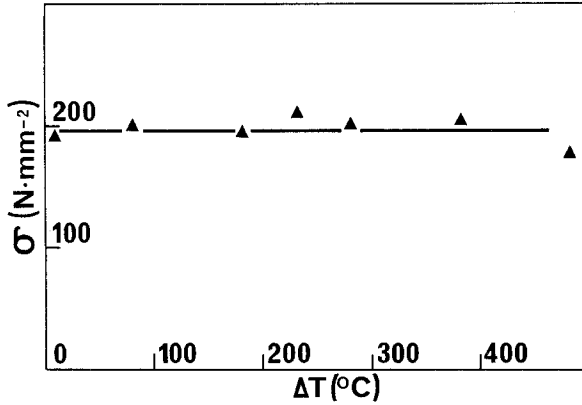**Fig. 6.** Strength versus temperature difference, for polished 30 (diam) × 5 mm discs.

Fig. 7. Strength versus temperature difference, for roughly ground 30 (diam) × 4.5 mm discs.

extreme cases only [4, 19], or may even disappear [14]. Becher et al. [20] give ΔT_c from 210 up to 280°C for bars and discs of thicknesses from 3 down to 1.5 mm.

The advantage of using different methods appears clearly in the present work. It is possible to obtain a wide variation of ΔT_c (ΔT_c of long cylinders $\sim 1.7\ \Delta T_c$ of voluminous cylinders). The most voluminous samples are kept small enough to avoid experimental difficulties such as inhomogeneities of quenching.

The critical temperature ΔT_c may be theoretically derived by taking into account the mechanical and thermal properties of the material, the shape and size of the sample, and the conditions of heat exchanges. The thermoelastic
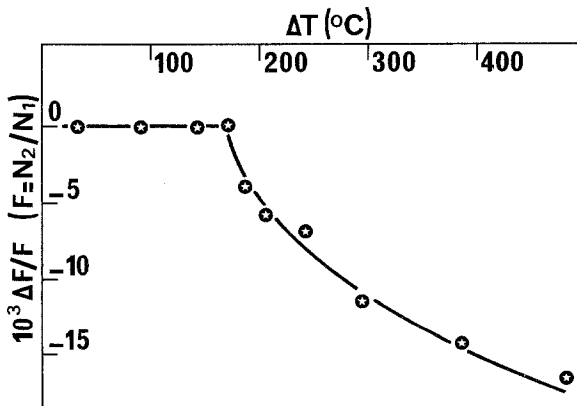

Fig. 8. Ratio of the frequencies of the two first overtones of vibrating 30 (diam) × 4.5 mm discs versus temperature differ-ence for *noncumulative* shocks.
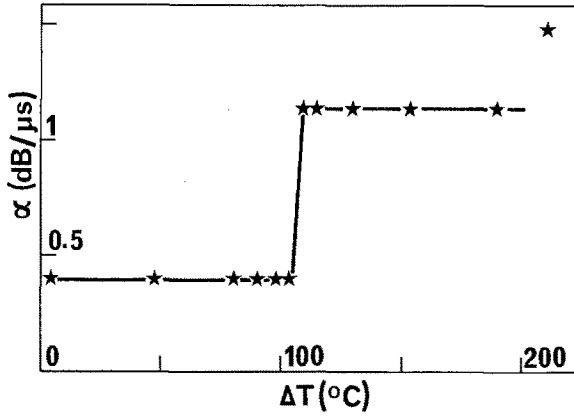
Fig. 9.   Acoustic attenuation versus temperature difference for
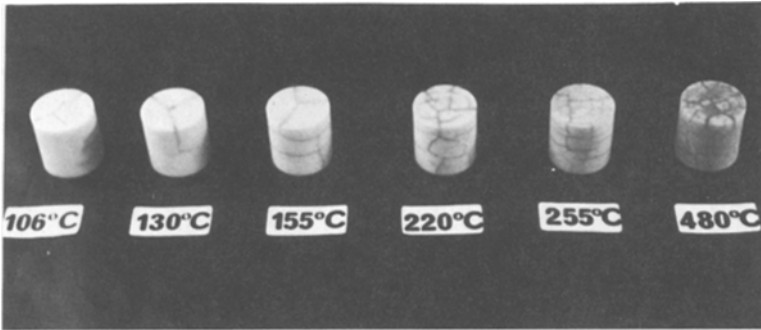short cylinders, for *cumulative* shocks.

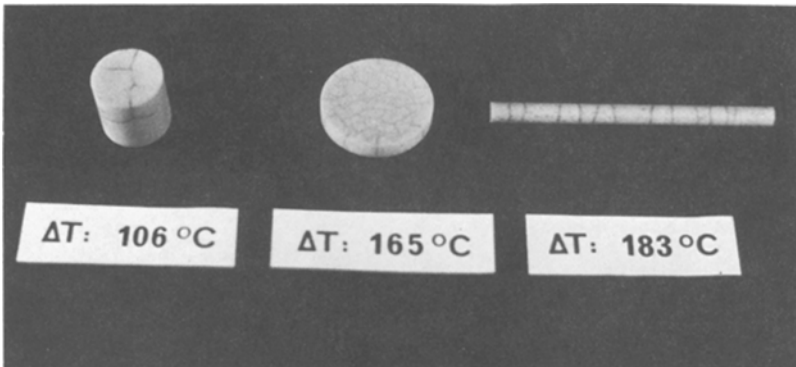Fig. 10.   Cracking of short cylinders, for increasing temperature difference.

Fig. 11.   Cracking of various samples, for $\Delta T = \Delta T_c + \epsilon$.
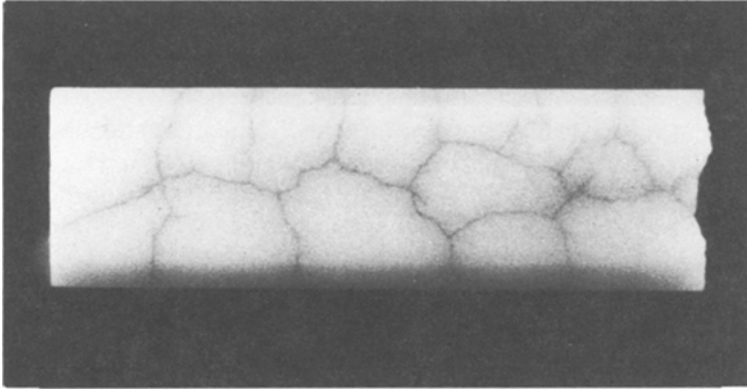
Fig. 12.   Cracking of prismatic beam, for $\Delta T > \Delta T_c$.

theory [6] and the energetic theory [15] are based on a different hypothesis. The thermoelastic theory says that the cracking occurs when the maximum value of the thermal stresses reaches the fracture strength of the material, σ_f. Thus

$$\Delta T_{th} = \frac{\sigma_f f(\nu)}{A E \alpha} \quad \text{or} \quad \Delta T_{th} = R/A \tag{1}$$

where R is the parameter of resistance to thermal cracking, E is Young's modulus, α is the linear thermal expansion coefficient, $f(\nu)$ is a function of Poisson's ratio ν and sample shape, and A is a dimensionless coefficient that varies with the experimental procedures [21]. A is related to Biot's number, $\beta = ah/k$, where a is the "mean size" of the sample, usually taken as the ratio of specimen volume to surface area, or a multiple of this length; h is the heat exchange coefficient; and k is the thermal conductivity of the material.

The energetic theory says that the preexisting defects (for instance, internal flaws or notches due to the machining) propagate when the lowering of the elastic energy is greater than the increase of surface energy on the

Table I. Critical Temperature for Different Samples

Sample	Size (mm)	Volume (mm³)	ΔT_c (°C)
Prismatic beam	4 × 4 × 25	400	D, 180
Long cylinder	6 (diam) × 80	2,260	D, 180 ND
Thin Disc	30 (diam) × 2.5	1,770	ND, 180
Thick Disc	30 (diam) × 4.5	3,180	ND, 170; D, 165
Short cylinder	20 (diam) × 20	6,280	ND, 105

crack boundaries. Consequently, the ΔT_c value depends upon the density of the internal defects. The main result of this theory is that two opposite behaviors are expected: the flaws propagate kinetically if they are small, while they propagate quasi-statically if they are large.

The energetic theory is known to be well suited to materials having a great number of rather large defects (for instance, porous refractories) and gives an excellent frame to study the resistance to damage. In the present case of dense alumina, it seems better to discuss the results in the frame of the thermoelastic analysis, which is better suited to study the resistance to cracking.

For this theory, ΔT_c may vary with the shape and the size of the sample. Indeed, $f(\nu)$ is a function of the specimen shape: $f(\nu) = (1 - 2\nu)/1 - \nu)$ for the discs, and $f(\nu) = (1 - \nu)$ for the rods; the fracture strength σ_f is a function of the specimen size (Weibull effect); and A depends upon the severity of the shock, and consequently, upon the mean size a, which influences Biot's number β.

Concerning the σ_f value, it is not clear what an appropriate value should be. Becher et al. [20] think that σ_f might be related to the lower extreme of the strength distribution, not the mean strength. However, the ΔT_c values are very reproducible, contrary to the strength as determined by a three-point bend test: for Al 23 alumina, the mean flexural strength is $\sigma_f \sim 260$ N · mm^{-2}, and Weibull's modulus $m = 13$. Thus we have preferred to choose $\sigma_f = \sigma_b$. Other measurements have given $E \sim 35 \times 10^4$ N · mm^{-2}, $\nu \sim 0.25$, and $\alpha \sim 8 \times 10^{-6}$ K^{-1}. Thus $R \sim 70°C$ for bars or cylinders, and $\sim 60°C$ for discs. It must be noted that when $\nu \sim 0.25$, $f(\nu)$ is almost the same for rods and discs, allowing that $R \neq \sigma_f(1 - \nu)/E\alpha$ in both cases.

The main problem is to evaluate β. The mean size a is not precisely defined, and this term may introduce an error, the sign of which is unknown. By taking $a = 2V/S$, where V is the volume and S is the area, one obtains $a = 1/2$ radius for cylinders and $a = $ thickness for discs (but some authors take $a = 1/2$ thickness for plates). The thermal conductivity is not accurately measured, and varies with the temperature. For dense alumina, $k \sim 5$ W · m^{-1} · K^{-1}. However, the principal uncertainty arises from the heat exchange coefficient h. Singh et al. [2] and Becher et al. [20] have pointed out the difficulties in determining h. This coefficient is a function of the nature of the quenching media, the velocity of the sample in the fluid, and the size, the shape, the nature, and the smoothness of the specimen, as well as the instantaneous temperature difference between the specimen surface and the fluid. For water quenching of small specimens, Singh et al. give a very low value of $\beta < 0.5$ (natural convection). For a specimen moving in water, Becher et al. [20] have chosen $h \sim 3$–4×10^4 W · m^{-2} · K^{-1}; they have thought that $h \sim 10^5$ W · m^{-2} · K^{-1} is a high value for alumina. In the

present work, the samples were vigorously stirred in water; however, they were smooth (which reduces h). Moreover, the ΔT_c values were rather low, which leads us to take into account a rather low value of h (when alumina is quenched in room temperature water, h increases rapidly in the 150–250°C range) [20]. Due to these contradictory reasons, a value of $h \sim 3 \times 10^4$ W $\cdot$ m$^{-2} \cdot$ K^{-1} seems to be acceptable: the corresponding β values are given in Table II.

For the limiting case of $\beta \rightarrow 0$, $\Delta T_{th} = R$. As a result, the theoretical and the experimental values of ΔT_c do not seem to agree with each other: such a discrepancy always leads to $\Delta T_c > \Delta T_{th}$. This point agrees with the conclusion of Becher et al. [20] that ΔT_{th} must take into account β, even in the case of large specimens suffering severe thermal shocks. However, it must be noted that for the short cylinders, $\Delta T_c/R$ is 1.5, whereas for the long bars, $\Delta T_c/R$ is 2.6. This point clearly shows the size effect, and most voluminous sample corresponding to $\beta = 60$ and the bar to $\beta \sim 15$.

The previous results show that β is small enough to lead to $A < 1$, and consequently, $\Delta T_{th} > R$, in Eq. (1). To calculate $\Delta T_{th} = R/A$, the A parameter must be known. Becher et al. [20] use the equation

$$\Delta T_{th} = R\,(B + C/\beta) \quad \text{for large } \beta \; (\beta > 100) \tag{2}$$

with $C = 3.25$ for the plate and 4.67 for the rod.

In the case of transient cooling of an infinitely long circular cylinder, Singh et al. [2] and Ziegler et al. [22] use the equation

$$\Delta T_{th} = 1.451\,R\,(1 + 3.41/\beta) \tag{3}$$

By taking the A values for an infinite circular cylinder [21] and an infinite flat plate [6], it is possible to obtain ΔT_{th} (See Table II). The A values are very similar to the ones obtained by Eqs. (2) or (3). It must be noted that Kingery [6] takes $a = 1/2$ thickness for the plate, whereas $a =$ thickness is taken in this work.

The functions $A(\beta)$ that have been chosen are only a rough estimate: the

Table II. Comparison of Thermal Shock Data for Different Samples

Sample	β	A	ΔT_{th}	ΔT_c	$\Delta T_c/\Delta T_{th}$
Prismatic beam	12	0.5	140	180	1.29
Long cylinder	18	0.6	116	180	1.55
Thin disc	15	0.55	109	180	1.65
Thick disc	27	0.65	92	165	1.79
Short cylinder	60	0.85	82	105	1.28

discs are assimilated to an infinite plate, the other shapes to an infinite cylinder. Consequently, it seems difficult to say if the shape effect is significant by considering the $\Delta T_c / \Delta T_{th}$ ratios, due to the poor accuracy of A. However, it must be noted that thermal stress theory would predict a lower ΔT_c for the disc than for the bar, which corresponds to experiments of the present work, whereas the experiments by Becher et al. [20] indicate the opposite.

5. CONCLUSIONS

The use of both destructive and nondestructive experimental methods has allowed an accurate determination of the maximum temperature difference that alumina samples will accept. These different methods have used thin and massive samples: in particular, the high frequency acoustic techniques allow one to measure low values of ΔT_c, by using samples that are massive but not very voluminous. Consequently, a size effect has been clearly shown.

It is difficult to know if the shape effect is significant, due to the rough approximation of the A parameter. However, it seems that the $A(\beta)$ functions are better suited for the bars than for the discs. It has been verified that the thermoelastic theory does not help in calculating the true value of ΔT_c for a given sample and given quenching conditions: the experimental value of ΔT_c is always significantly greater than the calculated value of ΔT_{th}.

The main reason for such discrepancies must be due to an overestimation of the h coefficient, and consequently of the A parameter, but this overestimation of A does not account for the full extent of the discrepancy. In the case of massive samples, $\beta \rightarrow \infty$, which means that $A \rightarrow 1$ whatever the exact expression of $A(\beta)$ is, and $\Delta T_{th} \rightarrow R$. This last observation suggests the necessity of modifying the R parameter, which seems to be underestimated.

The question is about the fracture strength σ_f: contrary to the usual claim for taking σ_f equal to the Weibull lower limiting tensile strength, it would appear that a rather high value of σ_f must be chosen. This point is perhaps related to the fact that the thermal stresses result from a triaxial state of deformation, which is not the case of the tensile test or the bend test.

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