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Effect of bath and specimen temperature on the thermal stress resistance of brittle ceramics subjected to thermal quenching

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The effect of specimen and bath temperature on the failure of brittle ceramics in a thermal quench experiment was studied by quenching glass and alumina rods in water and silicon oil baths at different temperatures. The results were discussed in terms of the variation of heat transfer coefficient of the quenching media and the change in material properties as a function of temperature. It was found that the usual assumption of constant heat transfer coefficient and material properties may lead to considerable errors in the quantitative interpretation of the results of thermal quench experiments. Effective values for the film coefficient of heat transfer for water and oil baths were estimated as a function of film temperature from thermal quench data. Recommendations were made for the selection of quenching media and for the procedure to be followed in reporting the results.

1. Introduction

Because of their favourable properties at high temperature, as well as in corrosive and erosive environments, ceramic materials are finding ever wider applications in industrial and many other fields. Because of their inherent brittleness and combination of other pertinent properties, ceramic materials, however, exhibit poor resistance to catastrophic failure under thermal conditions which generate thermal stresses. Therefore, for the purpose of reliable engineering design, it is important that the variables which control thermal stress failure of brittle ceramics are well understood. For a given thermal environment, the selection of materials with optimum thermal stress resistance can be based on "thermal stress resistance parameters" obtained from theory [1–6] or on comparative testing.

One such method of testing thermal stress resistance of ceramic materials consists of quenching appropriate specimens from high temperature into a fluid medium at a lower temperature [7–9]. Typical specimens are rods with circular or square cross-section. Quenching media can include water, oils or fluidized beds. Although, this test method enjoys considerable popularity because of its relative simplicity, the experimental results obtained frequently do not agree well with predictions based on theory [8–10]. In part, such discrepancies possibly can be attributed to differences in the susceptibility of ceramic materials to subcritical crack growth which was shown to be significant in the water quenching of a glass [11].

Other possible variables to which a discrepancy between theory and experiment can be attributed, can be ascertained by the theoretical expression for the critical temperature difference (ΔT_c) required to induce thermal stress failure in a ceramic specimen subjected to a thermal quench

$$\Delta T_c = \frac{AS_t(1-\nu)}{\alpha E} + \frac{BS_t(1-\nu)K}{\alpha Eah}, \quad (1)$$

where A and B are constants which depend on the geometry of the specimen, a is the characteristic dimension (i.e. radius of a circular cylinder), S_t is the tensile strength, E is Young's modulus, ν is Poisson's ratio, α is the coefficient of thermal

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expansion, K is the thermal conductivity and h is the heat transfer coefficient.

Equation 1 indicates that ΔT_c depends on a number of material properties, the dimensions of the specimen and the heat transfer coefficient, h. The properties such as the coefficient of thermal expansion, Young's modulus, Poisson's ratio and thermal conductivity as well as their temperature dependence are readily measured. An assessment of the tensile strength is more difficult due to the statistical nature of brittle fracture. This latter phenomenon poses the need to establish fracture probabilities based on stress distributions, specimen dimensions, etc. by such theories advanced by Weibull [12] or others. The need for such an approach was established in a previous study [13] of the thermal fatigue of a glass. The constants A and B are readily determined by analytical or numerical methods [14–16]. Specimen dimensions can be obtained to high precision. This leaves the heat transfer coefficient as the last remaining significant variable, which could be responsible for the discrepancy between theory and experiment. Generally, in the comparison and interpretation of quenching data, the heat transfer characteristics of the fluid medium are assumed to be invariant.

To establish the validity of this latter assumption, it should be noted that the transfer of heat between a solid and fluid is governed by a number of variables. Firstly, the heat transfer coefficient depends on the fluid properties such as the coefficient of volume thermal expansion, the density, the specific heat, the thermal conductivity as well as the viscosity. For purposes of the objectives of this study, it is important to note that these fluid properties, especially the viscosity, can show a strong temperature dependence. Secondly, the rate of heat transfer depends on the geometry and dimensions of the solid and the difference in temperature between the solid surface and fluid [17]. Thirdly, for a specimen falling through the fluid at its terminal velocity,* the rate of heat transfer also is a function of the relative difference in density of the fluid and solid [5]. For a water bath [17], the heat transfer is also affected very strongly by the effect of nucleate boiling which enhances the heat transfer coefficient and the effect of film formation at very high temperature levels which tends to suppress the transfer of heat.

Calculations of heat transfer coefficients generally are based on the properties of the fluid corresponding to the "film" temperature taken as the average of the temperature of the fluid and the surface of the solid. It should be noted then that ceramic specimens with differences in values of the critical quenching temperature (ΔT_c) will show differences in the film temperature even for identical bath temperatures. In view of the temperature dependence of the fluid properties, especially the viscosity, differences in values of ΔT_c will lead to differences in the values of the heat transfer coefficient. At least in part, then, the aforementioned discrepancies between predicted and measured thermal stress resistance in quenching experiments occur because the heat transfer coefficient is a function of temperature rather than a constant as is usually assumed.

The validity of this hypothesis can be verified by measuring ΔT_c for a range of film temperatures which can be achieved by varying the bath temperature. For a given material and bath temperature, the film temperature can be varied by changing the size of the specimen. The purpose of the present paper is to report the results of such a study.

2. Experimental procedure

2.1. Materials and quenching media

Specimens consisted of circular rods 6.25 cm long and diameter 0.53 cm of a soda-lime–silica glass† also used in previous studies [11, 13] and a polycrystalline aluminium oxide‡ with a range of diameters. The values for the material properties of the glass are presented in Table I. Similarly Table II lists the flexural strength of the aluminium oxide as a function of diameter size and other relevant properties.

The quenching media consisted of water and two silicon oils§ with nominal viscosities of 5 and 100×10^{-6} m^2 sec^{-1} at 25° C. The alumina specimens were tested in both the water and silicon oils. The glass rods were tested in the silicon oils only, in view of the considerable stress corrosion effects encountered in water [11, 13].

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*Terminal velocity is the constant velocity attained by the specimen during its fall through the fluid bath when the downward force of its weight is balanced by the upward buoyancy and shear forces.

†R-6, Owens Corning Fiberglass Corp., Toledo, Ohio, USA.

‡998 Alumina, McDanel Refractory Porcelain Co., Beaver Falls, PA, USA.

§Type 200, Dow-Corning Corp., Midland, Michigan, USA.

TABLE I Properties of Na_2O–CaO–SiO_2 glass [11]

Property	Value
Flexural strength at liquid nitrogen temperature (MPa)	216 (± 22)
Young's modulus of elasticity (GPa)	69
Coefficient of thermal expansion ($^\circ C^{-1}$)	9.3×10^{-6}
Poisson's ratio	0.25
Thermal conductivity ($J m^{-1} {}^\circ C^{-1} sec^{-1}$)	1.0465

2.2. Quenching procedure

The quenching media were contained in a stainless steel beaker with diameter 18 cm and depth 20 cm. The steel beaker was held in a thermostatically controlled glass tank 30 cm in diameter by 30 cm deep. Prior to quenching, the ends of the glass rods were wrapped with glass insulating tape to prevent fracture due to unknown stresses at the rod ends. Because of the very high density of cracks in the alumina, this procedure was not required for these specimens.

Sets of five specimens were heated slowly to a preselected temperature in a small electrically heated laboratory furnace and held for 10 to 15 min to assure thermal equilibrium, after which they were dropped into the quenching bath. This procedure was repeated for a range of quenching temperature differences (ΔT), defined as the difference in initial temperature of the specimen and the bath temperature. For the glass, the critical quenching temperature difference (ΔT_c) was defined as the value of ΔT at which three or more specimens had visible cracks. For the alumina specimens in which the cracks were not easily detectable, the critical temperature difference was obtained by measurements of the strength in four-point bending with loading and support spans of 1.6 and 5 cm, respectively, and cross-head speed of 0.05 cm min^{-1}. Prior to the strength test, all specimens quenched in water were dried at ~ 110° C for 2 to 3 h. The critical temperature difference was reported in terms of a temperature range. The lower limit of this range corresponded to the value of ΔT at which one specimen in five exhibited a significant strength loss (strength reduction of ~ 45% or more with respect to the initial value). The upper limit corresponded to the value of ΔT at which four or more specimens exhibited a strength loss.

3. Results and discussion

Fig. 1a and b show the value of ΔT_c as a function of bath temperature for the glass specimens quenched into the silicon oils with viscosity of 100 and 5×10^{-6} m^2 sec^{-1} (at 25° C).

The decrease in ΔT_c with increasing bath temperature for the oil with viscosity 100×10^{-6} m^2 sec^{-1} above 30° C is thought to be due primarily to the decrease in viscosity with increasing temperature. Such a decrease in viscosity increases the heat transfer coefficient which in turn decreases ΔT_c. On comparison of Fig. 1a and b, this conclusion is supported by the fact that for a given bath temperature the oil with the higher viscosity

TABLE II Properties of polycrystalline alumina*

Parameter	Flexural strength (MPa)	Strength relative to diameter of 0.318 cm
Diameter (cm)		
0.318	327 (± 25)	1.0
0.475	311 (± 64)	0.95
0.635	271 (± 51)	0.83
0.953	251 (± 20)	0.77
Young's modulus of elasticity (GPa)		393
Coefficient of thermal expansion ($^\circ C^{-1}$)		7.4×10^{-6}
Poisson's ratio		0.265
Thermal conductivity ($J m^{-1} {}^\circ C^{-1} sec^{-1}$)		
200° C		18.2922
300° C		13.0599
350° C		12.1808

*The data on flexural strength and the thermal conductivity were obtained in our laboratory and the other material properties were supplied by the manufacturer.

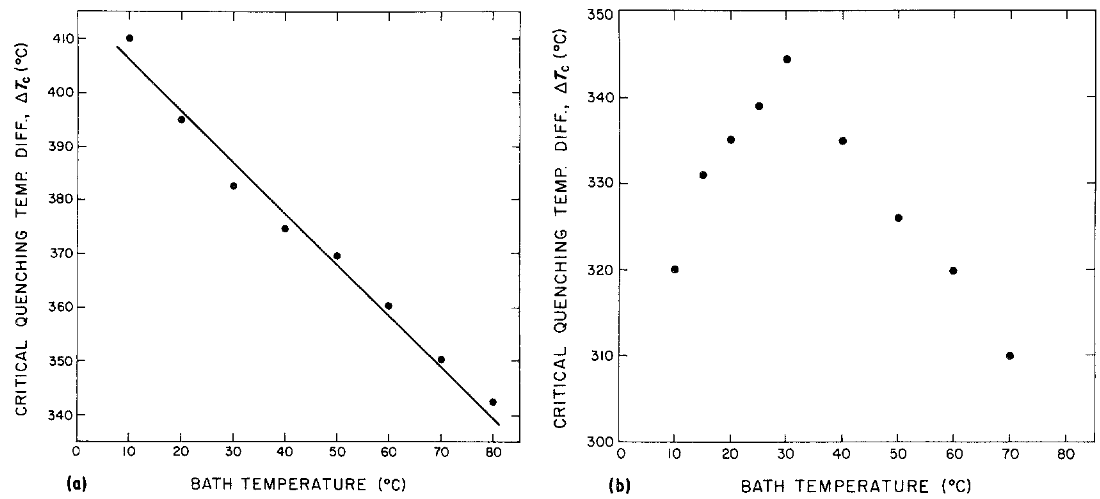

Figure 1 Effect of bath temperature on ΔT_c for glass rods quenched in silicon oil: (a) 100×10^{-6} m² sec⁻¹ (b) 5×10^{-6} m² sec⁻¹.

also yields the higher value of ΔT_c. At least for the $100 \times 10^{-6}\,\text{m}^2\,\text{sec}^{-1}$ oil, the variation of ΔT_c with temperature cannot be attributed to the temperature dependence of the relevant material properties of the glass, since the initial specimen temperature (i.e. bath temperature + ΔT_c) is constant over the total range of bath temperature.

Of interest in Fig. 1b is the decrease in ΔT_c with decreasing bath temperature below 30° C. This behaviour is directly opposite to the effect noted for temperatures in excess of 30° C. It is thought that this apparent anomaly can be attributed to at least two effects. Firstly, a calculation of the heat transfer coefficient from the fluid properties near room temperature indicates that the temperature dependence of the heat transfer coefficient decreases with decreasing fluid temperature. For this reason, the effect of bath temperature on ΔT_c is expected to be less pronounced at the lower than at the higher bath temperatures. Secondly, it should be noted that with decreasing bath temperature the initial specimen temperature also decreases. This implies that the temperature dependence of the properties of the glass should be taken into account. In this respect, the rather high temperature dependence [18] of Young's modulus and strength of a soda-lime–silica glass over this range of initial specimen temperature, tends to reduce ΔT_c with decreasing bath temperature. For a range of bath temperatures of 10 to 30° C, the strength increases due to increasing initial specimen temperature prior to quenching beyond which the strength remains constant due to approximately constant initial specimen temperature. The anomaly in ΔT_c for the low bath temperature shown in Fig. 1b, results from the combined effects of the temperature dependence of the heat transfer coefficient, Young's modulus and failure stress, with the temperature dependence of these latter two properties, contributing the major effect. In view of the relatively low temperature dependence of the coefficient of thermal expansion and the thermal conductivity, these two properties are thought to have little influence on the anomaly.

Support for this latter conclusion is given by the experimental data shown in Fig. 2 for ΔT_c for the alumina rods with diameter 0.63 cm quenched in the same silicon oil ($5 \times 10^{-6}\,\text{m}^2\,\text{sec}^{-1}$) as the glass data shown in Fig. 1b. For these alumina specimens the anomaly at the low value of bath temperature is absent. The value of ΔT_c decreases monotonically with increasing bath temperature probably due primarily to the decrease in viscosity and corresponding increase in heat transfer coefficient with increasing bath temperature. At the initial specimen temperature of 450° C the aluminium oxide is not expected to exhibit a significant change in fracture stress during the 15 min anneal. This suggests that indeed the anomaly shown in Fig. 1b is due to the temperature dependence of the strength and Young's modulus of the glass over the range of initial specimen temperatures involved.

Fig. 3 shows the effective heat transfer coefficient for the two silicon oils as a function of the film temperature calculated by means of

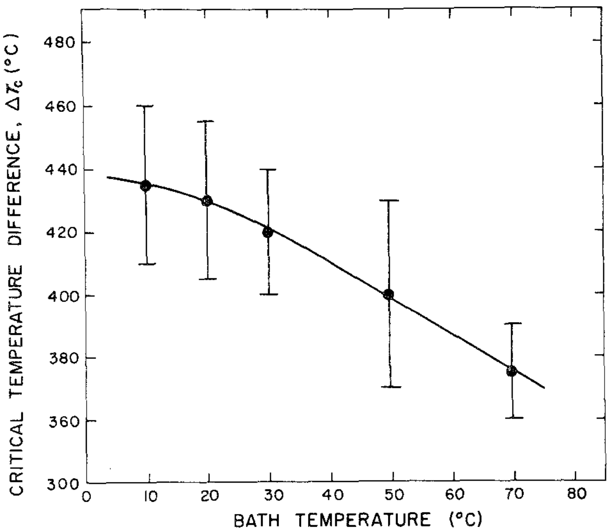

Figure 2 Effect of bath temperature on ΔT_c for alumina rods ($d = 0.63$ cm) quenched in silicon oil (5×10^{-6} m^2 sec^{-1}).

Equation 1 from the experimental data for ΔT_c and the relevant material properties. In these calculations the strength at liquid nitrogen temperature ($-196°$ C) was taken to be 194 MPa as measured by Badaliance *et al.* [11] and its temperature dependence was assumed to be similar to that given by Shand [18]. The strong temperature dependence is evident. Clearly these data are relevant only to the present quenching experiments and probably are not appropriate to other heat transfer conditions. Nevertheless, they may serve as a guide for the design of quenching experiments of other materials.

For water as the quenching medium, the experimental data for ΔT_c differed significantly from those obtained for silicon oil. Fig. 4 shows the dependence of ΔT_c on the water bath temperature for the alumina rods with diameter 0.318 cm. In contrast to the data obtained for the silicon oil, ΔT_c for the water bath shows an upward curvature with a minimum near 60° C. At a water temperature of 90° C, only a small fraction of the specimens could be fractured by the thermal stress generated at quenching temperature differences from 225 to 425° C. Fig. 5 shows the distribution of the percentage of specimens fractured during the quench as a function of ΔT. No specimens could be fractured for values of $\Delta T > 425°$ C. For these reasons, for the bath temperature of 90° C, a value for ΔT_c could not be defined. Comparison of the data in Figs 2 and 4 indicates that ΔT_c for the silicon oil exceeds ΔT_c for the water bath, due to large differences in heat transfer coefficients, as will be shown later.

Fig. 6a shows the size dependence of ΔT_c for the alumina specimens quenched into the water bath at 25 and 90° C. For the purpose of interpretation of the data, it is convenient to eliminate

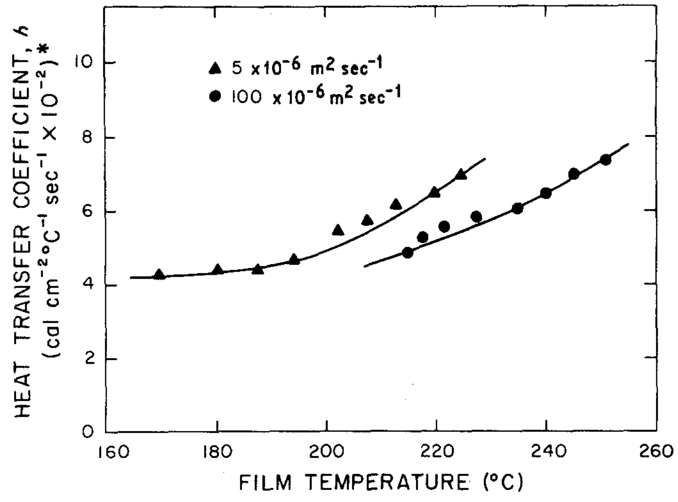

Figure 3 Effective heat transfer coefficient for silicon oil baths inferred from the thermal fracture data of Fig. 1a and b
*1 cal = 4.1855 J.

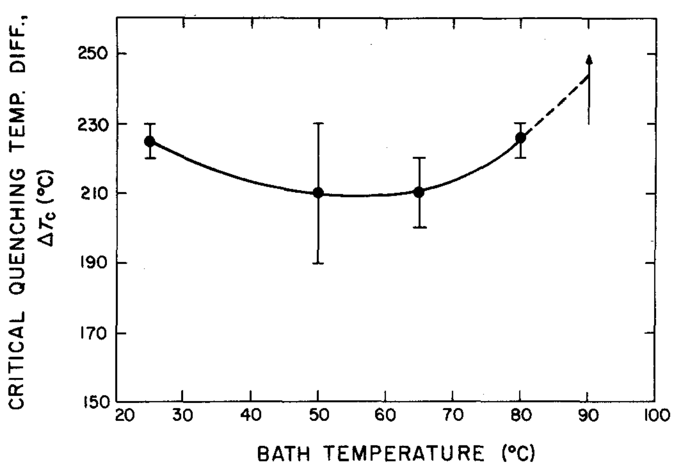

Figure 4 Effect of bath temperature on ΔT_c for alumina rods ($d = 0.318$ cm) quenched in water.

the effect of rod diameter on the flexural strength. This was accomplished as shown in Fig. 6b by dividing the observed values of ΔT_c by the ratio of the flexural strength relative to the strength for the rod diameter of 0.318 cm, listed in Table II. For this reason, ΔT_c in Fig. 6b is referred to as "normalized" critical quenching temperature difference, $\Delta T_c'$. It is interesting to note that $\Delta T_c'$ decreases with an increasing value of $1/d$, in contrast to an increase predicted by Equation 1. Since the derivation of Equation 1 was based on theory, this discrepancy cannot be attributed to a lack of fundamental understanding of thermomechanical principles.

At least a qualitative explanation for the data shown in Figs 4 and 6a can be based on the general temperature dependence of the heat transfer coefficient for a hot surface in water. This is shown schematically in Fig. 7 as a function of the difference in temperature of the hot surface and the water temperature. At low temperature differences, the heat transfer coefficient increases with increasing temperature as a direct result of the nature of convective heat transfer coupled with nucleate boiling [17]. At the higher temperature differences, the rate of heat transfer is limited by the formation of steam-film on the hot surface. Because of the existence of such a film, the value of the heat transfer coefficient will go through a maximum followed by a decrease with further increases in temperature difference. At sufficiently high temperatures, where radiation from the hot surface becomes significant, an increase in "effective" heat transfer coefficient can be observed. Increases in water bath temperature towards boiling for a given hot surface temperature will enhance the formation of the steam-film accompanied by a corresponding decrease in heat transfer coefficient.

Examination of Fig. 4 in view of the results of

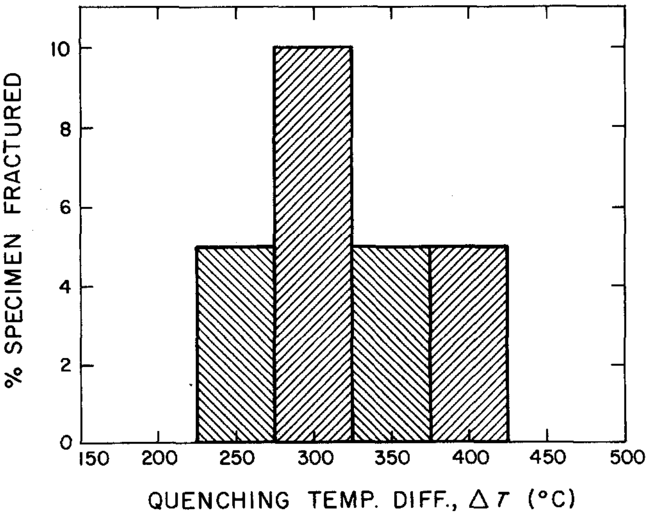

Figure 5 Probability of failure of alumina rods ($d = 0.318$ cm) quenched in water at 90° C.

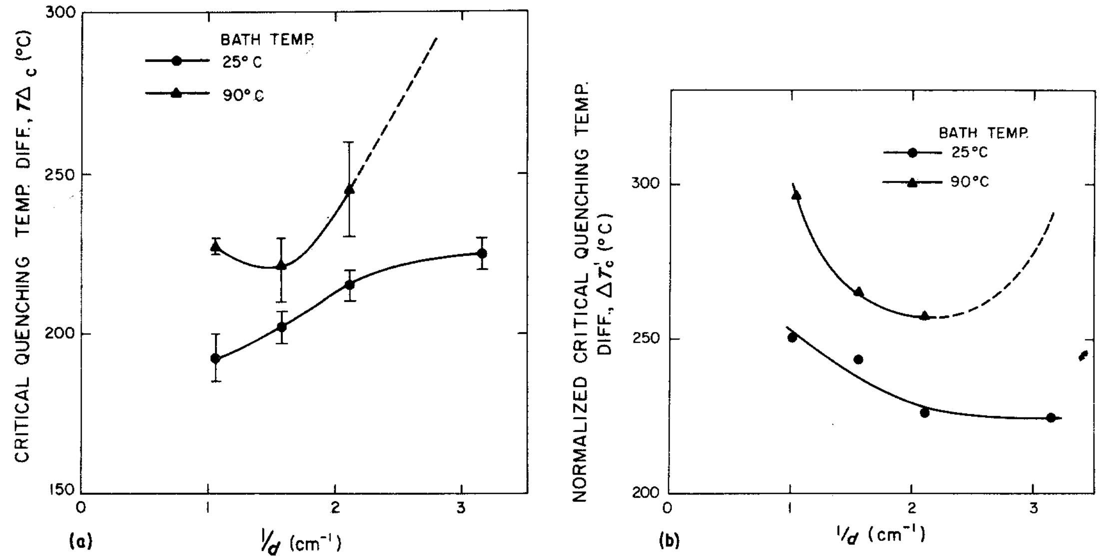

Figure 6 (a) Effect of specimen size on ΔT_c for alumina rods quenched in water at 25 and 90° C, (b) normalized $\Delta T_c'$ for alumina rods quenched in water at 25 and 90° C.

Fig. 7 suggests that the minimum in the curve for ΔT_c against water bath temperature corresponds to the maximum in the curve of heat transfer coefficient as a function temperature difference. The data in Fig. 6a for 25° C can be explained in terms of the rapid rise in heat transfer coefficient with temperature difference in the range of nucleate boiling. An increase in ΔT_c automatically increases the heat transfer coefficient which in turn limits the increase in ΔT_c which can be achieved. In fact, the plot in Fig. 6b suggests that the relative rate of increase in heat transfer coefficient exceeds the rate at which ΔT_c should increase with decreasing specimen size. In spite of these effects, ΔT_c increases with decreasing specimen size because of the corresponding increase in strength with decreasing specimen size. A similar explanation can be given for the data with a bath temperature of 90° C if it is assumed that the heat transfer coefficient corresponds to the low values near the minimum between the regions of steam-film formation and radiation indicated in Fig. 7.

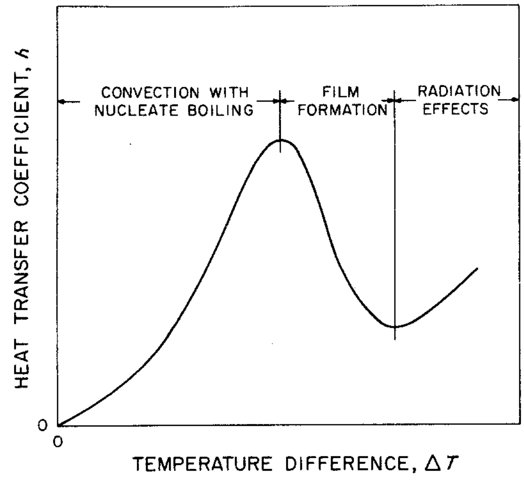

Figure 7 Schematic diagram for the variation of the heat transfer coefficient of water as affected by the solid surface temperature.

Support for the above explanations is found in the form of the "effective" heat transfer coefficient for the water bath calculated from the observed values of ΔT_c (Fig 4 and 6a) with the exception of those for 0.318 cm diameter rods at 90° C and the property data for the alumina given in Table II. These calculated values are shown in Fig. 8 as a function of film temperature defined earlier. These calculated data show the same general trends as those shown schematically in Fig. 7. It should be noted that these calculated data vary over an order of magnitude. Again, it is cautioned that these data are valid only for the particular testing conditions and materials of the present study. Unless verified, their use for other test conditions could lead to misleading results.

The data for the effective heat transfer coefficient for the water bath shown in Fig. 8 permit a reasonable explanation for the absence of thermal stress failure in the majority of specimens with a diameter of 0.318 cm and quenched into water at 90° C. Because of their small size, as

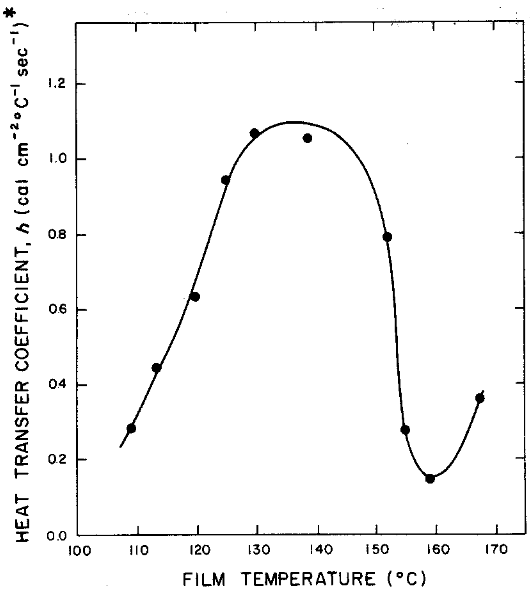

Figure 8 Effective heat transfer coefficient for water inferred from the experimental ΔT_c values in Figs 4 and 6a. *1 cal = 4.1855 J.

indicated by Equation 1, these specimens require high values of ΔT to induce thermal stress failure. Such high values of ΔT imply high film temperature with corresponding low values of the heat transfer coefficients. The few specimens in which failure could be induced represented the weakest specimens of the strength distribution for this size of specimen. It is likely that if the strength values for these specimens had been only slightly higher, failure would not have occurred for any of the specimens. On the other hand, slight reduction in the strength would have lowered ΔT_c with a corresponding large increase in heat transfer coefficient so that all the specimens would have failed.

Manson and Smith [15] found for disc specimens peripherally quenched in water, that the thermal stresses were nearly constant with ΔT, exhibiting only a slight concavity with a maximum near $\Delta T_c \simeq 120^\circ$ C. At least qualitatively this suggests that the heat transfer coefficients for those experiments corresponded to the range of film temperature shown in Fig. 8 which includes the maximum and the regime over which h decreases rapidly with increasing film temperature.

Becher *et al.* [19] calculated the effective heat transfer coefficient from ΔT_c for a number of ceramic materials. A plot of these values against ΔT resulted in a curve similar to the one shown in Fig. 8. However, if the values of h calculated from the present data were plotted in a similar manner, the h values for a 90° C bath temperature would fall to the left of the data for the lower bath temperatures. This, then, no longer would result in the expected inverted V-shaped curve. For this reason, its appears preferable to use the concept of film temperature (or perhaps initial specimen temperature) in order to properly reflect changes in heat transfer coefficient due to differences in bath temperature.

Davidge and Tappin [7] found that on increasing the water bath temperature from 20 to 95° C resulted in a decrease in ΔT_c from 168 to 88° C for alumina and from 95 to 45° C for polycrystalline magnesium oxide. These observations appear to contradict the present findings. However, as judged by the relatively low values of ΔT_c for the samples of Davidge and Tappin [7] (presumably due to low values of tensile strength), the corresponding film temperatures for bath temperatures of 20 as well as 95° C, fall in the region of nucleate boiling. In this region, the heat transfer coefficient increases rapidly with increasing film temperature so that a decrease in ΔT_c on increasing water bath temperature is reasonable. For the alumina of this study, however, ΔT_c is sufficiently high that the heat transfer coefficient decreases with increasing ΔT. Consequently, ΔT_c increases with increasing bath temperature.

4. General

The data presented in this paper are illustrative of the difficulties encountered in establishing the relative thermal stress resistance of ceramic materials by the quenching method. The interpretation of experimental data obtained by this method requires *a priori* quantitative data for the heat transfer characteristics of the fluid media used. Such data are not easily obtained. Furthermore, since the specimen continuously changes temperature, the heat transfer coefficient is expected to vary for the duration of the quench. Under these conditions a calculation of the thermal stresses, which takes into account time-dependent film temperatures and corresponding heat transfer coefficients, may have to rely on numerical methods. Spatial variations of the heat transfer coefficients around the specimens introduced an additional complexity.

For the above reasons, the heat transfer data shown in Figs 3 and 8 can only be described as "effective" since they were inferred from the experimental data. Although these data show the anticipated dependence on film temperature, their general applicability to other materials must be treated with caution. This is true, in particular, for the water bath, in which the heat transfer coefficient as affected by nucleate boiling and steam formation is expected to be a function of the surface condition (roughness, etc.) of the specimen. It is even conceivable that differences in thermal stress resistance of different materials (i.e. values of ΔT_c) can be found entirely due to differences in surface condition which modifies the heat transfer characteristics.

Even for materials with surface conditions which yield identical heat transfer coefficients for a given film temperature, the assessment of the relative thermal stress resistance based on data for ΔT_c is not simple. Consider two different materials with relatively low thermal stress resistance so that at ΔT_c, the corresponding film temperature for a water quench is in the nucleate boiling range. The material with the higher thermal stress resistance will require a higher value of ΔT_c. This however will involve a higher film temperature and corresponding higher value of the heat transfer coefficient. This, in turn, will suppress the value of ΔT_c. For this reason, differences in relative thermal stress resistance of two materials subjected to a water quench involving nucleate boiling may be difficult to assess.

The opposite effect occurs for two materials for which ΔT_c corresponds to values of film temperatures which involve steam-film formation. In this range even small differences in ΔT will involve large differences in heat transfer coefficient and corresponding large differences in ΔT_c. This situation, for instance could arise in comparisons of a polycrystalline aluminium oxide and a highly thermal shock resistant material such as high-strength hot-pressed silicon nitride. It seems reasonable to point out that water, despite its convenience, appears less useful for measurement of thermal stress resistance by the quenching method.

Silicon oils, on the other hand, appear more suitable in view of their monotonic increase of the heat transfer coefficient with increasing film temperature. For these oils the relatively low value of effective heat transfer coefficient may prevent the use of very small specimens which could be inconvenient especially for newly developed materials for which larger specimens may not be available. It is anticipated, however, that oils can be found or developed with heat transfer characteristics higher than those used in the present study.

A final recommendation is in order for purposes of facilitating quantitative comparison of results of different investigators. In reporting such results, the conditions of the quenching tests should be stated. This includes the bath temperature as well as the range of quenching temperature differences, in order to estimate the appropriate film temperature. In view of the rapid variation of the heat transfer coefficient with film temperature, especially for water, the actual temperature of the bath should be stated. Baths at nominal room temperature do not appear adequate, in view of large regional as well as seasonal variations of this quantity.

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