Observations on the characteristics of a fluidized bed for the thermal shock testing of brittle ceramics

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The heat transfer characteristics of a fluidized bed used as a quenching medium for the thermal stress testing of brittle ceramics, were determined by measurements of the thermal shock behaviour of rods of a soda-lime—silica glass. The heat transfer coefficient was found to be strongly dependent on the mean particle size of the powder and air flow rate, and was relatively independent of the position within the bed. The results indicate that the heat transfer coefficient during thermal shock fracture may have a value lower than that obtained under heat transfer conditions which more closely resemble steady state. The heat transfer data inferred from the quenching experiments with the glass gave excellent agreement between calculated and measured values for the thermal shock behaviour for rods of a polycrystalline aluminium oxide. It is concluded that fluidized beds are excellent inert quenching media with variable heat transfer coefficient controlled by particle size and flow rate.

1. Introduction

Technical ceramics, because of their combination of high melting point and chemical stability are excellent candidate materials for engineering applications involving extreme thermal and chemical environments. However, due to an unfavourable combination of values for such properties as thermal conductivity, Young's modulus, coefficient of thermal expansion and tensile strength, many technical ceramics exhibit low resistance to failure by thermal stresses, frequently encountered during transient or steady-state heat flow [1]. Due to the inherent brittleness of ceramic materials such failure can be highly catastrophic. For reliable engineering design and optimum material selection it is imperative that the thermal stress behaviour of technical ceramics be well understood. This can be accomplished on the basis of theoretical principles or by actual testing. One popular test method, due to its simplicity, consists of quenching specimens in the form of circular or square cylinders from a preselected higher temperature into a fluid medium at lower

temperature. Such a fluid medium can consist of either silicone oil [2], a eutectic salt mixture [3, 4], water [5], liquid metals [6] or fluidized powders [7]. For purposes of the quantitative evaluation of the test results, the heat transfer characteristics of these fluid media must be well understood. In this respect, a number of experimental studies [8-11] have shown that water as a quenching medium can be highly variable in its heat transfer characteristics, primarily due to nucleate boiling and/or steam film formation which are dependent not only on the temperature but also on the surface condition of the material. The decrease of the viscosity of silicone oils with increasing temperature causes a corresponding increase in the heat transfer coefficient [11]. Increased turbulence of eutectic salts with increasing temperature tends to create a corresponding increase in the rate of heat transfer [4]. A recent analysis showed that the results of quenching experiments also depend on whether the heat transfer occurs by natural or forced convection [12]. Water has the disadvantage of promoting

stress corrosion [13]. Oils are subject to decomposition which can lead to the formation of strongly adhering coatings on the specimen surface and subsequent changes in mechanical or other properties [14].

Fluidized beds which use ceramic powders as the fluid medium offer the advantage of chemical inertness over a wide range of temperature. Literature data [7, 15] show considerable differences in the heat transfer characteristics of fluidized beds used for thermal shock testing. These differences, as indicated by the experimental data of Callahan [15] for the heat transfer coefficient of fluidized beds obtained by a direct method, may be attributable to differences in the way these fluidized beds were being operated. In this respect, then, the primary objective of this study was to investigate the effect of gas flow rate and powder particle size on the heat transfer characteristics of fluidized beds used for the thermal shock testing of brittle ceramics. The secondary objective of this study was to compare the values of the heat transfer coefficient inferred indirectly from the thermal shock data with those measured by direct means.

2. Experimental procedure

2.1. Test materials

A soda-lime-silica glass^{*} and a polycrystalline alumina[†] were selected as materials appropriate for testing of the fluidized bed. Both these materials were used in a number of previous studies [11, 13] which facilitated comparison of the experimental data. The materials were supplied

TABLE	I	Properties	of test	materials

by the manufacturers in the form of circular rods. Their property values and diameter are given in Table I. Because of the ease of crack detection in the glass by visual means, most tests were conducted with this material. The aluminium oxide specimens were used for the purpose of illustrating the use of data obtained.

2.2. Equipment and test procedures

The fluidized bed of commercial design[‡], suitable for operation at elevated temperatures, if desired, consisted of a vertical stainless steel hollow cylinder with an inner diameter of approximately 0.1 m and a height of approximately 0.15 m. In order to extend the range of the rates of air flow and to minimize the loss of the fluidizing powder, a silica tube approximately 0.5 m high and 0.09 m inner diameter was placed inside the steel cylinder. Air was used as the fluidizing agent. The rate of air flow was controlled by a flow meter. To ensure a uniform flow of air across the width of the fluidizing bed, the perforated bottom was covered with a fibreglass cloth pad, which also prevented the loss of finer powders through the bed bottom between experiments. For all tests the fluidized bed was maintained at room temperature.

An aluminium oxide powder f was selected as an appropriate fluidizing medium. By screening, the powder was divided into a number of size fractions corresponding to screen sizes of 60 to 65, 100 to 115, 200 to 270 and 325 to 400 mesh, to give average particle sizes of 231, 138, 69 and 38 μ m.

Prior to quenching, the specimens were pre-

Property	Soda-lime glass*	Alumina†
Young's modulus, E (GPa)	69	393
Coefficient of thermal expansion, α 10 ⁻⁶ (° C ⁻¹)	9.3	9 (725° C)
Poisson's ratio, ν	0.25	0.265
Thermal conductivity, k 10 ⁻³ (cal cm ^{-1°} C ⁻¹ sec ⁻¹)	2.5	21.9 (725° C)
Flexural strength (MPa)	194	271
	(Liquid N_2 temperature)	(Room temperature)
Diameter, $d (10^{-3} \text{ m})$	5.3	6.35
Weibull parameter, m	8	16

*Singh et al. [11] and Hasselman et al. [13].

[†]Young's modulus value was supplied by the manufacturers, Poisson's ratio and the coefficient of thermal expansion were obtained from [16], and other properties values were obtained in the writers' laboratory.

*R-6, Owens-Corning Fiberglass Corp., Toledo, Ohio.

[†]998 Alumina, McDaniel Refractory Procelain Co., Beaver Falls, PA.

[‡]TECAM Fluidized Bath Model FBS, Techne Incorp., 661 Brunswick Pike, Princeton, N.J.

[∮]Calcined alumina powder (- 230 mesh), Aluminium Corporation of America.

heated in an electrically heated, thermostatically controlled ($\pm 0.5^{\circ}$ C) tube furnace with an internal diameter of $\simeq 0.05 \,\mathrm{m}$, mounted vertically above the fluidized bed. The specimens in the form of a circular cylinder of length $\simeq 0.06$ m were attached to the lower end of a quartz rod mounted vertically along the centre line of the furnace tube. In the upper position of the rod, during preheating, the specimens were located in the axial and radial centre of the hot zone of the furnace. In the lower position of the rod, the specimen was located within the fluidized bed. This lower position was adjustable to permit measurement of the heat transfer characteristics as a function of the vertical position within the bed. Caps mounted over the ends of the furnace tube assured temperature uniformity and eliminated possible specimen cooling due to the upward flow of air through the tube furnace. Thermal equilibrium immediately prior to quenching was obtained by holding the specimens for at least 15 min at the level of preselected temperature. Quenching of the specimens was accomplished by dropping the rod from the upper to lower position under the influence of gravity without any mechanical constraint. Following quenching, the specimens were held in the fluidized bed for approximately 5 min after which they were removed and examined. All specimens were tested singly, in order to ensure that the heat transfer was not affected by the presence of other specimens. However, in order to take account of possible specimen-to-specimen variation, a total of five specimens were tested for any given temperature difference and operating condition of the fluidized bed. To eliminate any fatigue effects on the results obtained each specimen was tested only once.

The critical temperature difference, ΔT_c , required to induce thermal stress fracture in the specimens for a given air flow rate and alumina particle size was established by varying the temperature difference between the tube furnace and the fluidized bed. To reflect the variation from specimen to specimen, ΔT_c for the glass was reported in terms of the temperature range required to fracture one and four of the five specimens tested. For the alumina specimens in which the cracks were not easily detected by visual or other means, ΔT_c was determined by measuring the fracture strength of the quenched specimens at room temperature in four-point bending with an inner and outer span of 0.016 and 0.05 m, respectively. For these specimens, ΔT_c was defined as the value of quenching temperature difference which caused a 50% or greater decrease in strength compared to the non-quenched specimens.

The heat transfer characteristics of the fluidized bed were determined by indirect as well as direct means. Indirectly, the heat transfer coefficient was determined from the experimental data for $\Delta T_{\rm e}$ for the glass specimens with the aid of thermoelastic theory. Thermal stress failure in brittle materials generally occurs under the influence of the tensile stresses. For the quenching conditions of the present study, the maximum tensile thermal stresses occur in the specimen surface. For a solid circular cylinder and 0 < Rh/k < 10, from the solutions of Jaeger [17] the magnitude of these stresses to a very good approximation can be described by:

$$\sigma_{\max}^{-1}(r=R) = \frac{1.45(1-\nu)}{\alpha E} \quad 1 + \frac{3.41\,k}{Rh}$$
(1)

where α , E, k and v are defined in Table I, r is the radial distance from the centre of the rod, R is the radius of the cylinder and h is the heat transfer coefficient. At $\Delta T = \Delta T_c$, the maximum value of tensile thermal stress equals the tensile fracture stress. Following the approach of Becher et al. [10] and Singh et al. [11], the heat transfer coefficient was calculated by substitution of the values for the material properties, R and ΔT_{e} into Equation 1. In the calculations, the relative temperature dependence of the strength of the glass specimen was assumed to be identical to the strength of Shand [18]. Using the strength of 194 MPa at liquid nitrogen temperature [13], an estimate of the strength of the present glass specimens at different temperatures was easily made.

The direct measurement of the heat transfer coefficient relied on the method of Callahan [15] and Trantina [14], which consisted of measuring the transient temperature response of a small, preheated specimen quenched in the fluidized bed. For the present study the specimen consisted of an alumina sphere of diameter 1.25 cm, with a thermocouple in a hole drilled to the exact centre of the sphere. For the alumina, the thermal conductivity and diffusivity were established independently by the laser—flash method. A plot of the non-dimensional transient temperature at the centre of the sphere, on comparison with the charts of Schneider [19], yields the appropriate value of Rh/k from which the value of h can be calculated.

The value of h obtained from the data for ΔT_c may possibly reflect the highly transient nature (if any) of the heat transfer during the quench. The value of h obtained by the immersed-sphere method requires a time period in excess of 40 sec well in excess of the time period of < 0.1 sec of insertion of the specimen in the fluidized bed or the time period of about 0.3 sec [20] for the tensile thermal stress to reach its maximum value. For this reason, the value of h obtained by the latter method more closely corresponds to the steady-state heat transfer conditions.

3. Experimental results and discussion

Fig. 1 shows the values of ΔT_c for the glass specimens as a function of air flow rate for four values of mean particle size of the aluminium oxide powder. At approximately 0.8 dm³ min⁻¹ cm⁻² the air flow rate was sufficiently high that some of the powder with the lowest mean particle size value was carried out of the bed. The data indicate that ΔT_c depends strongly on the air flow rate as well as particle size. Such variation can only be attributed to the strong dependence of the heat

transfer coefficient, h, on flow rate and particle size.

The validity of this latter conclusion is substantiated by the values of the heat transfer coefficient shown in Fig. 2 evaluated from the data for ΔT_{c} of Fig. 1. These values of h vary by nearly as much as a factor of three. At the lower range of flow rate, the increasing value of h with increasing flow rate is most probably the result of the corresponding increasing degree of fluidization of the bed. The decrease in h with increasing flow rate at the higher values of flow rate probably results from the decrease in net density of the bed, which as judged by the observed increase in relative bed height for the highest flow rate could amount to as much as 30%. The relative dependence of heat transfer coefficient on flow rate as shown in Fig. 2, is similar to the findings of Callahan [15].

Fig. 3 shows the dependence of the heat transfer coefficient inferred from corresponding data for ΔT_c for the glass specimens as a function of the depth of the bed for the specific value of air flow rate of 0.47 dm³ min⁻¹ cm⁻² and average alumina particle size of 138 μ m. For convenience, the depth of the bed was defined as the distance





Figure 1 Effect of air flow rate and alumina particle size on the critical quenching temperature difference (ΔT_c) for soda-lime-glass rods (d = 0.53 cm) quenched in a fluidized bed.

Figure 2 Effect of air flow rate and alumina particle size on the heat transfer coefficient h for a fluidized bed as inferred from the results for ΔT_c in Fig. 1.



Figure 3 Heat transfer coefficient h as a function of specimen position in the fluidized bed for air flow rate of 0.47 dm³ min⁻¹ cm⁻² and mean alumina particle size of 138 μ m inferred from values of ΔT_c for soda-lime-glass rods.

from the bottom of the bed to the centre of the vertical specimens of length $\sim 6 \,\mathrm{cm}$. Experimentally, it was observed that the specimen did not undergo fracture at a preferred location along its length. This observation combined with the relative independence of the heat transfer coefficient on depth of the bed suggests that the heat transfer appears to be relatively uniform along the length of the specimen. Practically the position of the results obtained. Of course, insertion of the specimens close to or in direct contact with the side walls should be avoided.

Fig. 4 shows the experimental data for the heat transfer coefficients obtained by the sphere immersion method for three of the identical batches of powders used to establish the data shown in Figs 1 and 2. Comparison of Figs 2 and 4 shows that the values of the heat transfer coefficient obtained by both methods show the same relative dependence on the air flow rate. Quantitatively, however, the maximum values of h inferred from the data for ΔT_{c} significantly exceed the corresponding values obtained by the sphere immersion method. In explaining this discrepancy at least two major effects must be considered. The first effect results from the differences in the geometry of the test specimens. As presented by Gebhart [21], the forced convection heat transfer coefficients, h_{c} and h_{s} for a cyclinder and sphere, respectively, can be expressed by:

$$h_{\rm c} = 0.69(Re)^{0.466}(Pr)^{1/3}k_{\rm f}/d_{\rm c}$$

for 40 < Re < 4000 (2)



Figure 4 Heat transfer coefficient h for the fluidized bed obtained by the sphere-immersion method as a function of air flow rate and alumina particle size.

$$h_{\rm s} = (2.0 + 0.6(Re)^{0.5}(Pr)^{1/3})k_{\rm f}/d_{\rm s}$$
 (3)
for $1 < Re < 70\,000$

where $Re = Vd/\eta$ is the Reynolds' number, V is the fluid velocity, η is the fluid viscosity and d is the specimen diameter. Pr is the Prandtl number, $K_{\rm f}$ is the thermal conductivity of the fluid, and $d_{\rm c}$ and $d_{\rm s}$ are the diameters of the cylinder and the sphere, respectively.

Taking the case that $2 \ll 0.6 (Re)^{0.5} (Pr)^{1/3}$ assuming no significant difference between the exponents of 0.466 and 0.5 for the Reynolds' number in Equations 2 and 3, yields the ratio

$$h_{\rm c}/h_{\rm s} \simeq 1.15 \, (d_{\rm s}/d_{\rm c})^{1/2}$$
 (4)

which for the present specimens with $d_s/d_c \simeq 2.29$ yields:

$$h_{\rm c}/h_{\rm s} \simeq 1.74. \tag{5}$$

This result suggests that an upward adjustment of the data of Fig. 4 for the spherical geometry by a factor 1.74 yields the appropriate values for the heat transfer coefficient for the vertical glass circular cylinders. For example, the value of $0.061 \text{ J cm}^{-2} \text{ C}^{-1} \sec^{-1}$ (0.0145 cal cm⁻² ° C⁻¹ sec⁻¹) (Fig. 4) inferred from immersed sphere data for fluidized beds with particle sizes of $69 \,\mu\text{m}$ and an air flow rate of 0.47 dm³ min⁻¹ cm⁻² must be multiplied by 1.74 in order to account for a geometry effect. This brings the peak value to about 0.1046 J cm⁻² ° C⁻¹ sec⁻¹ (0.025 cal cm⁻² ° C⁻¹ sec⁻¹) which is still only about 71% of the corresponding value for *h* determined from ΔT_c (Fig. 2).

The second effect to which the remaining discrepancy between the data of Fig. 2 and the adjusted data of Fig. 4, can be attributed is based on the statistical nature of brittle fracture, which states that the strength of a brittle solid is a function of the volume of the stressed material as well as the stress distribution. Theoretical analyses of this effect have been presented by Weibull [22] and others. As a result of this statistical effect, the tensile fracture stress of the glass specimens under conditions of thermal shock with a uniform biaxial stress distribution in the specimen surface is expected to differ from the strength under conditions of four-point bending on which the data of Fig. 2 were based.

In a previous study [13] of the thermal fatigue behaviour of the identical glass specimens of the present study, it was shown that the ratio of the tensile fracture stress (σ_{ts}) during thermal shock to the corresponding fracture stress in four-point bending (σ_b) for a population of surface flaws, by means of the Weibull theory, can be calculated to be:

$$\sigma_{\rm ts}/\sigma_{\rm b} \simeq 0.59. \tag{6}$$

This result suggests that multiplication of the data shown in Fig. 2 by the factor 0.59, will yield values of the heat transfer coefficient which

correspond more closely to the magnitude of the tensile failure stress encountered during quenching in the fluidized bed. The peak value for the heat transfer coefficient h determined from $\Delta T_{\rm e}$ for the glass specimens then becomes approximately $8.786 \times 10^{-2} \text{ J cm}^{-2}^{\circ} \text{ C}^{-1} \text{ sec}^{-1}$ $(2.1 \times 10^{-2} \text{ cal})$ $cm^{-2} \circ C^{-1} sec^{-1}$). The corresponding h value obtained by the correction for geometry of the results in Fig. 4 evaluated from the sphereimmersion data is $10.46 \times 10^{-2} \, \text{J cm}^{-1} \,^{\circ} \, \text{C}^{-1} \, \text{sec}^{-1}$ $(2.5 \times 10^{-2} \text{ cal cm}^{-2} \circ \text{C}^{-1} \text{ sec}^{-1}$, which is higher than the corrected value inferred from ΔT_c . In view of the total uncertainties in establishing both values, possibly no major significance should be attached to this discrepancy. Nevertheless, the lower value of h inferred from $\Delta T_{\rm c}$ may possibly reflect the highly transient nature of the heat transfer in the very brief time period required for fracture to occur. Possibly, in the brief period, the transfer of heat from the specimen cannot benefit from the motion of the fluidized powder. A detailed analysis of this effect if beyond the scope of the present discussion.

Fig. 5 shows the results for the strength behaviour of the alumina specimens quenched into the fluidized bed with a mean particle size of $138 \,\mu\text{m}$ and an air flow rate of $0.47 \,\text{dm}^3 \,\text{min}^{-1} \,\text{cm}^{-2}$. The dependence of strength on temperature difference indicates a value of $\Delta T_c \simeq 800^\circ \text{ C}$. From the data of Figs 2 and 4, the values appropriately adjusted for the statistical and geometry effects as discussed earlier are approximately 8.368 and $10.46 \times 10^{-2} \,\text{J cm}^{-2^\circ} \,\text{C sec}^{-1}$ (2.0 and $2.5 \times 10^{-2} \,\text{cal cm}^{-2^\circ} \,\text{C sec}^{-1}$), respectively. Using these latter values, the data in Table I and the ratio



Figure 5 Strength as a function of quenching temperature difference for alumina rods (d = 0.63 cm) quenched in a fluidized bed with an air flow rate of 0.47 dm³ min⁻¹ cm⁻² and mean alumina particle size of 138 μ m.

of strength under thermal shock to the strength in bending, $\sigma_{\rm ts}/\sigma_{\rm b} \simeq 0.79$ inferred from the Weibull parameter for a distribution of surface flaws, the values for ΔT_c can be calculated to be 830 and 675° C. The value of 725° C for the property values listed in Table I, corresponds to the mean specimen temperature at the instant of fracture for $\Delta T_{\rm c} \simeq 800^{\circ}$ C. The much better agreement between this latter value and 830° C predicted from the h value inferred from ΔT_{c} for the glass specimen than that predicted from the value obtained from the sphere-immersion method, provides confirmation for the earlier suggestion that the heat transfer in a fluidized bed during a thermal quench is less severe than under conditions which more closely resemble steadystate conditions.

A comparison of data shows that the peak value of the heat transfer coefficient of approximately 8.7864 to $10.46 \times 10^{-2} \text{ J cm}^{-2^{\circ}} \text{ C sec}^{-1}$ (2.1 to $2.5 \times 10^{-2} \text{ cal cm}^{-2^{\circ}} \text{ C sec}^{-1}$) found in the present study, agrees quite well with the value of approximately $8.368 \times 10^{-2} \text{ J cm}^{-2^{\circ}} \text{ C sec}^{-1}$ ($2.0 \times 10^{-2} \text{ cal cm}^{-2^{\circ}} \text{ C sec}^{-1}$) reported by Ammann *et al.* [23] and Callahan [15]. The much lower value of about $3.347 \times 10^{-2} \text{ J cm}^{-2^{\circ}} \text{ C sec}^{-1}$ ($8 \times 10^{-3} \text{ cal cm}^{-2^{\circ}} \text{ C sec}^{-1}$) found by Trantina [14] probably can be attributed to differences in experimental conditions.

Comparison of the values found in the present study with those for other quenching media, shows that even the peak values for the heat transfer coefficient for the fluidized bed is less than the corresponding values for the effective heat transfer coefficients for silicone oils or water obtained in a previous study [11]. This statement holds even if these latter data are adjusted downward by the factor 0.59 to account for the statistical nature of brittle fracture. It appears then that fluidized beds for purposes of thermal shock testing of brittle ceramics extend the range of heat transfer coefficients available to the researcher. As indicated by the results of the present study as well as those of Callahan [15] a particular advantage of fluidized beds is that they offer considerable flexibility in adjusting the heat transfer coefficient by modification of gas flow rate and particle size.

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