Improved method for severe thermal shocks testing of ceramics by water quenching

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Abstract

The thermal shock testing of ceramics by water quenching in a bath of water at room temperature is a very popular one because of its simplicity. However it presents some disadvantages, the most critical of them being the water phase changes in the sample vicinity which strongly affect heat exchanges between sample and water. This study proposes a new experimental technique which minimizes the previous phase changes in order to perform severe thermal shocks. The sample, uniformly heated at a temperature higher than the boiling water temperature, is suddenly cooled in periphery by a system of water jets at room temperature. The water flow is sufficiently high to insure a permanent contact of the sample periphery with water in liquid state. So, the surface heat exchanges are significantly greater than those developed during a thermal shock by immersion and thus make it possible to approach the conditions of very severe thermal shock.
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Keywords: Resistance; Testing; Thermal shock

1. Introduction

1.1. Theoretical considerations

Two theoretical approaches are generally used to analyze the effects of a thermal shock: the thermoelastic analysis suggested by Kingery[1] and energetic analysis suggested by Hasselman.[2] In spite of their weaknesses, they have been applied by numerous authors to multiple concrete cases of thermal shocks.[3–6]

The thermal shock resistance parameters introduced by the thermoelastic analysis (R and R') make it possible to rank materials with respect to their cracking resistance when subjected to thermal stresses. The R parameter which deals to the case of hard thermal shocks allows to estimate the value of the critical drop in temperature (ΔTc) by means both of the stress reduction coefficient Ψ ($\Delta Tc = \Psi^{-1} R$) and the Biot's number β: $\beta = ah/\lambda$ a: typical sample dimension h: superficial heat exchange coefficient λ: thermal conductivity of material.

However, the practical use of this relationship knock against the ambiguous parameter a and especially against the thermal parameter h, non directly measurable, and capable of considerable variations according to the quenching bath temperature.[7]

One reproaches this analysis to be purely thermoelastic and to disregard the characteristic pre-cracking of ceramic materials.

The energetic approach takes into account these pre-existent defects and studies their stability and kinetics during the shock. It introduces thermal shock parameters allowing the ranking of materials with respect to their damage resistance to thermal shock. Because it rests on too simplifying assumptions, it cannot give rise to numerical practical applications.

Thus, the thermal shock phenomenon is not easily describable by universal laws, usable to perform reliable analysis of experimental results according to the experimental process.

1.2. Experimental considerations

Because of its simplicity, the water quench is the most used experimental test. However, the critical temperature differences measured (ΔTc) depend on many

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experimental parameters (bath temperature, geometry of the coin, orientation of the coin compared to the bath...) and are thus comparable for identical experimental conditions only.

One of the most critical parameters is the thermal shock severity. Its influence is easily shown by tests for which the nature of the quenching bath varies (water,[8] molten salt,[9] silicon oil,[10] fluidized beds[11,12]) or by tests using a given quenching bath (water) at various temperatures.[13] The thermoelastic theory accounts for this influence by means of the Biot's number β but, on the one hand its formulation is too approximate and, on the other hand, the numerical data dealing with the heat exchange coefficient are too rare. So, this analysis does not appear as a reliable tool for prediction and analysis.

In the case of water quench, the phase changes of the quenching bath in the vicinity of the sample (located vaporization, nucleation boiling, calefaction) are responsible, for a given sample temperature, to the considerable variations of the heat exchange coefficient h underlined by Becher et al.,[14] when the bath temperature varies.

The aim of this work is to propose an original technique of water quench, minimizing the phase changes of the cooling liquid of and thus reducing the influence of the heat exchange coefficient variations on the results of a thermal shock test.

2. Experimentation

2.1. Experimental device

The bench of thermal shock consists of mechanical, thermal and pneumatic subsets (see Fig. 1).

The mechanical part consists of a frame used as support for the sample supports, the heating furnace and the quenching bath.
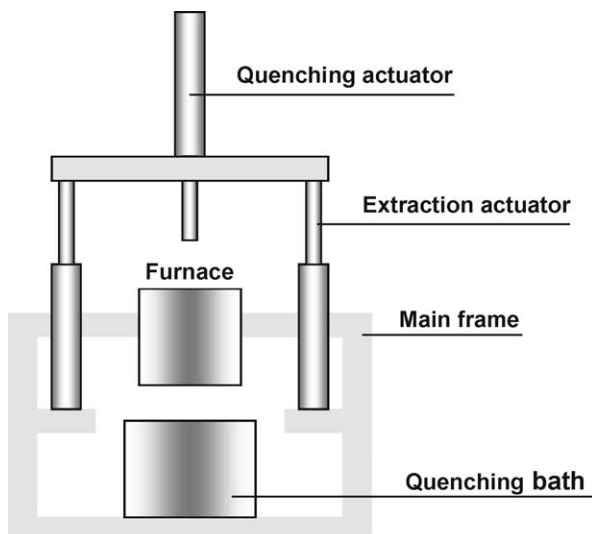
The pneumatic subset comprises three actuators. On one hand, a double effect actuator called quenching actuator allows the transfer of the sample from the furnace towards the quenching bath and vice versa. On the other hand, a set of two double effect actuators supplied in parallel and called extraction actuators. They support a cross-piece on which the quenching actuator is fixed and make it possible to position the previous set in test position or in loading position (samples positioning).

The thermal subset consists of an electric furnace and a cooling system. The heating furnace is of a wound type ($T_{max} \approx 1000$ °C). A regulating circuit allows to control of its heating rate and to maintain its temperature at a given level. A calibration was carried out in order to know the difference between the programmed temperature and the real temperature reached by the sample inside the furnace.

The water bath used for tests by immersion, is replaced by an original device of a shower type illustrated in Fig. 2. The sample is located in the center, and along the axis, of a system composed of eight tubes in copper (50 mm length), each pierced of 20 holes of 1 mm in diameter along a generating line. By these holes water jets at high pressure (5 bars) flow out.

They are all directed, radially, towards the axis of the sample. These jets run out when the sample, coming from the furnace, arrives in test position in the shower. The sample length is lower than the height of the shower system in order to ensure a complete cooling of the peripheral surface of the samples during quenching.

The purpose is to carry out more severe shock conditions than those induced during a thermal shock by immersion. Indeed, if the water flow is sufficiently high (high kinetic jets energy), the "skin" of the sample is continuously in contact with water in liquid state and the heat exchange developed is not decreased by the development of a gaseous phase.
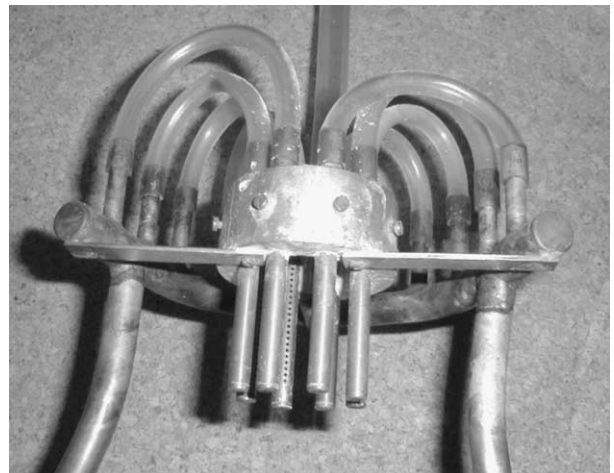

Fig. 1. Principle of the termal shock bench.

Fig. 2. View of the shower thermal shocks device.

2.2. Experimental procedure

The samples are brought up to temperatures varying from 170 to 280 °C by step of 10 °C. After being maintained at 15 mn in the furnace they are quenched during 12 s by immersion or by shower in water at 20 °C (150 °C < Δ T < 260 °C). For each initial temperature value, four samples are tested.

2.3. Material and samples

A set of 60 bars (4×4×38 mm) of alumina of commercial quality (Degussa Al23) was used. A half was subjected to thermal shocks by immersion and the other half was tested according to the original shower technique. Before tests, sample edges were beveled by polishing with a diamond paste in order to decrease the risks of accidental cracking due to edge effects.

2.4. Damage control

The critical temperature difference ΔT_c has been estimated, after thermal shock, by a nondestructive dye penetrant method on samples impregnated of penetrating product during 15 min by immersion then washed and dried during 10 min at ambient temperature.

The values thus obtained were validated by strength measurements in 4 point bending of the same bars.

3. Results and discussion

The Fig. 3 shows the results obtained for the two series of tests. For each of them, the graphs $\sigma = F_{(\Delta T)}$ exhibit the typical pattern forecasted by the energetic theory. Indeed, they present a strength loss characteristic of the catastrophic damage induced by the critical thermal shock, followed by a stage then by a new σ_R decrease.

Whereas ΔT_c is about 220 K for the tests by immersion, it does not exceed 160 K for the tests using the shower system.

As expected, the shower technique imposes a more severe thermal shock than the technique of immersion. It prevents the stable formation of a phase vapor in the vicinity of the sample periphery and thus maintains a high level of heat exchange between the sample and the cooling water.

The thermoelastic analysis allows an easy interpretation of these results by means of the first resistance parameter to thermal shocks, R, and Biot's number β.

$$R = \frac{\sigma_R}{E\alpha}(1 - \nu)$$

σ_R is the material strength, E the young's modulus, α the thermal expansion coefficient and ν the Poisson ratio.

Using the properties of alumina AL23 given by the manufacturer and by Glandis [8], and supposing that the normal stress distribution induced by the thermal shock is sufficiently close to the 4 point bending stress distribution, one obtains: $R \# 82$ K. Starting from: $\Delta T_C = \Psi^{-1} R$ and from (case of infinite bars):

$$\Psi^{-1} = 1.45 + \frac{4.95}{\beta} - 0.45 \, \exp\left(-\frac{16}{\beta}\right)$$

Ψ is the coefficient of stress reduction and $\beta = ah/\lambda$ the Biot's number one can write:

$$\Delta T_C \approx 119 + 406\frac{\lambda}{ah} - 37 \, \exp\left(-16\frac{\lambda}{ah}\right)$$

It is known that cracking occurs few time after the beginning of the thermal shock, that is to say when the sample temperature is about 180 °C for shower quenching and 240 °C for quenching by immersion. For these temperature levels, the thermal conductivity is roughly equal to 22.5 and 20 W/m/K. By supposing the
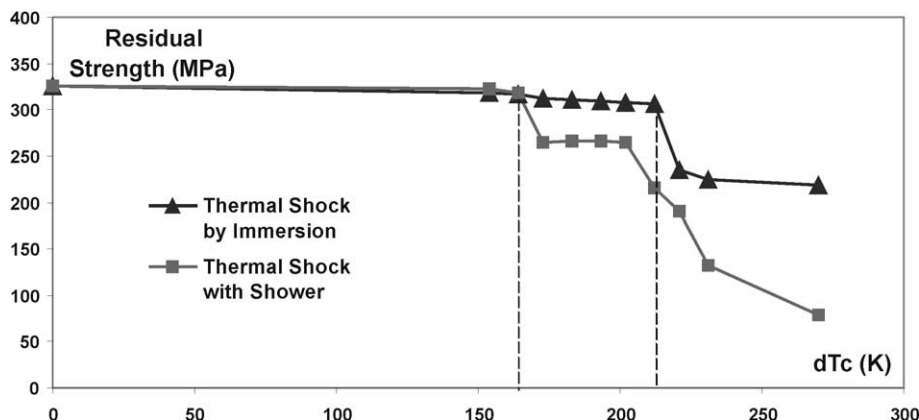

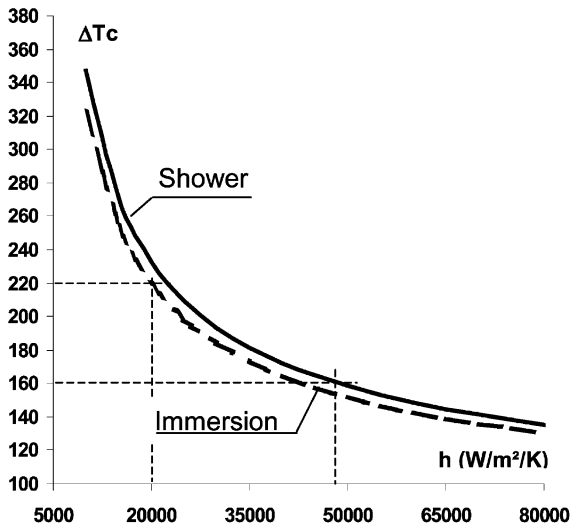Fig. 3. Experimental results: residual strength vs temperature difference.

Fig. 4. Critical temperature difference vs heat exchange coefficient.

typical dimension of the samples equal to their transversal dimension (4 mm), one obtains:

$$\Delta T_C \approx 119 + \frac{2.28 * 10^6}{h} - 37\ \exp\left(-\frac{9 * 10^4}{h}\right)$$

for the shower quenching and:

$$\Delta T_C \approx 119 + \frac{2.03 * 10^6}{h} - 37\ \exp\left(-\frac{8 * 10^4}{h}\right)$$

for the quenching by immersion.

This result is illustrated by Fig. 4, which represents the ΔT_c variations vs h. One notes that the shower technique allows to reach values of the heat exchange coefficient much higher than those involved by immersion: approximately 49 kW/m²/K instead of 20, that is to say an increase of about 150%.

Taking into account the weakness of the thermoelastic analysis which, moreover, considers the initial and final states of the system only, this result has an indicative significance only. Thus, it must be refined by means of more sophisticated methods such as, for example, the F.E.M. Such a modeling leads to greater h values but does not modify significantly the relative increase in this parameter: indeed, one finds that h varies from 27 to 65 kW/m²/K, that is to say an increase of 140%, fully comparable with the increase given by the rough thermoelastic analysis.

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