The Vickers indentation technique applied to the evaluation of thermal transient stresses due to quenching of brittle materials

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Abstract

Use is made of the domain of stable extension of the Vickers indentation crack system in order to investigate both the thermal shock resistance materials and the thermal transient stresses which appear during quenching of brittle materials. The equivalency of thermally and mechanically induced stresses is assumed such as to describe the relative increase in length of the radial crack during quenching. This approach is checked in using two diffferent glass ceramics, various quenching conditions and sample sizes. Finally is the increase in length of Vickers indentation cracks measured or samples loaded in bending and a maximum of the thermal transient stress evaluated. This offers the mean to approach an effective heat transfer coefficient.

I Introduction

The brittle nature of materials like polycristalline ceramics and glass ceramics are characterized by a small strain at rupture and low values of toughness. This makes them very sensitive to rapid temperature variations which occur during quenching. Many attention has therefore been paid to that problem. Buesem (1) reviewed the physical parameters involved in heat transfer. Kingery (2) additionnally aimed to derive a critical stress level surface which would irreversibly damage the quenched part. However the maximum level of the transient thermal stress is still poorly known, because the heat transfer coefficients are also poorly known (3).

In this respect, have various thermal stressing techniques been put to work (4) (5) (6). They mainly use surface indentation cracks. They either anneal the residual stresses linked to such a crack system or remove them by polishing. However these residual stresses allow such cracks, when loaded, to extend first in a stable way before catastrophic propagation (7-10). This ability of stable extension is used in this work such as to evaluate the maximum of the thermal transient stress in a given case of sample size and quenching conditions.

II The median-radial crack system.

When indenting a brittle material with a Vickers diamond a symmetric crack pattern appears at the corners of the indentation and is defined as a median-radial crack system (9) (10). These cracks of length, c_o, are in equilibrium between the toughness of the material, Kc, and a residual central opening force. In the as-indented stage, and for a given indentation load, this writes:.

$$Kc = \chi_r.P.C_o^{-3/2} \qquad (1)$$

where χ_r is a proportionality factor depending on materials properties and indenter geometry. The parameters are given in figure 1.

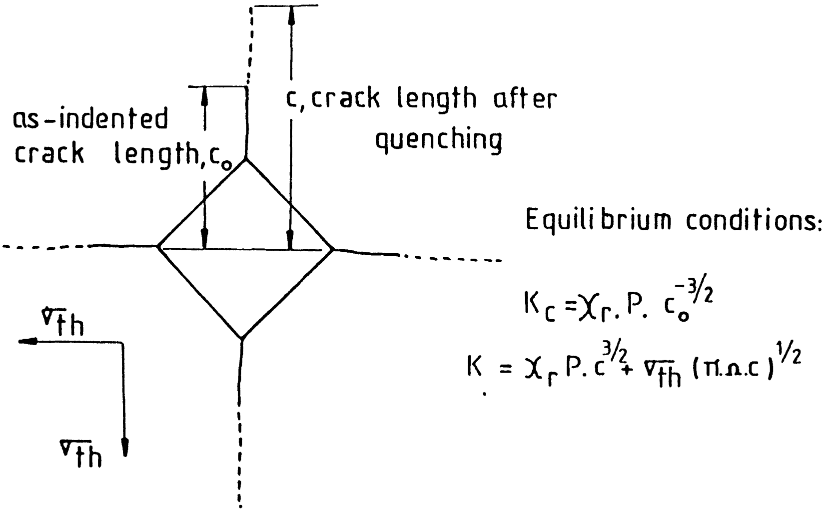

Fig. 1: Parameters of the Vickers indentation crack system.

If an external increasing stress, σ_a, is applied, the radial cracks increase in length, c, and the critical stress intensity factor, Kc, writes:

$$Kc = \chi_r.P.c^{-3/2} + \sigma_a (\Omega\pi c)^{1/2} \qquad (2)$$

where Ω is a geometrical factor of crack shape. From eq.(2), the functionality of applied stress, σ_a, versus actual crack length, c, can be deduced. This is shown in figure 2, for various indentation loads, P, and a given value of toughness, $Kc=4MPa\sqrt{m}$. The change of σ_a vs.c is described by first an increasing then decreasing part of a continuous curve. The increase in size, from the as-indenced value, c_o, to a value c_m (-given by $c_m = 4\chi_r P/Kc^{2/3}$), of the crack length describes the domain of stable crack extension during loading. At c_m the crack becomes unstable.

This stable crack extension, during the application of a thermally or mechanically induced stress will be used to determine the maximum of the transient thermal stress. It needs solely to compare the extension of cracks for different indentation loads after thermal and mechanical stressing.

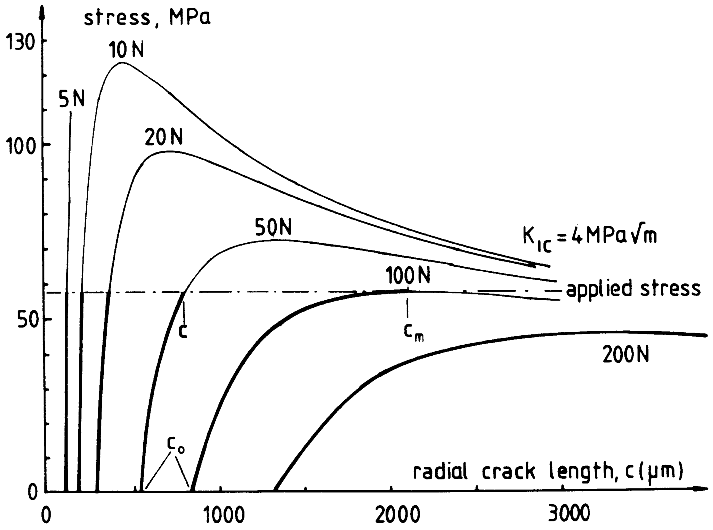

Fig. 2: Stable (increasing) and unstable (decreasing) branches of the extension of Vickers indentation radial cracks under the action of an applied stress. The value of the indentation load is given on the top of each curve. The extensions for a given level of applied stress are the thick part of the curve ($\chi_r = 1$ for simplicity).

A procedure can be used, in order to check experimentally the method, such as to range thermal shock resistances of given materials with different grain size (11)(12) or make evidence of various quenching conditions due to different sample sizes or quenching media. This can be deduced from figure 1. Consider the abscissa (actual crack length, c) of the intersect between a line of given stress with the σ_a vs. c curves (end of the thick part). If the relative increase in radial crack length, c/c_o, is plotted as a function of indentation load, P, curves, as shown in figure 3, are then obtained. For a given toughness these curves are the steeper the higher the thermal stress (more severe quenching conditions). Also should it be noticed that for a given thermal stress, increasing toughness (higher thermal shock resistance) produces flater curves.

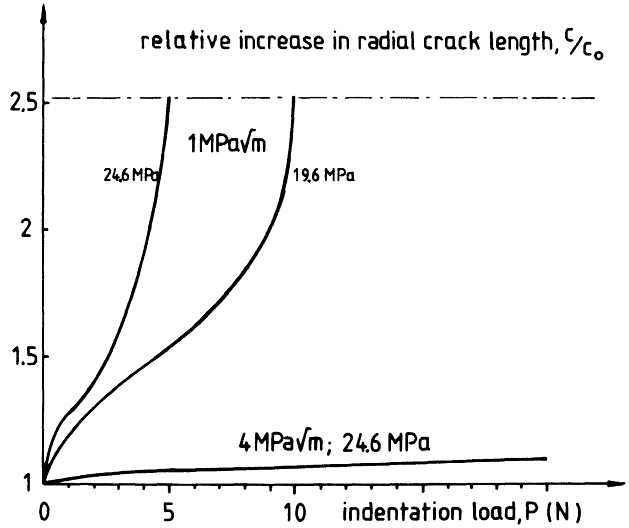

Fig. 3: Comparative effects of toughness and given applied stresses on the relative increase of the radial cracks from Vickers indentation, as a function of indentation load.

III Experimentals and results

Two materials have been used in this work; a glass-ceramic, ST3 (Kc = 1.3 MPa$\sqrt{m}$) and its precursor, VM-ST3 (Kc = 1MPa$\sqrt{m}$). The samples were small parallelepipeds (8 x 4 x 16 mm^3) and (10 x 4 x 60 mm^3), mirror polished on one face and then indented. These samples were then quenched either into liquid nitrogen (smooth thermal shock) or in oil at room temperature (hard thermal shock). Some results for the smaller are plotted in figure 4.

Now if largers samples are quenched in the same conditions it is expected that the amplitude of the thermal transient stress becomes higher. This is observed on the plot of figure 4. In the case of the ST3 material, is quenching large sample from RT into liquid N_2 more severe than quenching the smaller from 150°C into liquid nitrogen. The case of the VM-ST3 is worth being considered too. The open points describe mean values of the four cracks at the Vickers indentation. However the large samples should be considered as slabs in which an anisotropy of the thermal stresses exists. In particular are they highest in the direction perpendicular to the length of the slab, meaning thus that the cracks parallel to the length will increase more in length than the other. This is reproduced by the full points of figure 4.

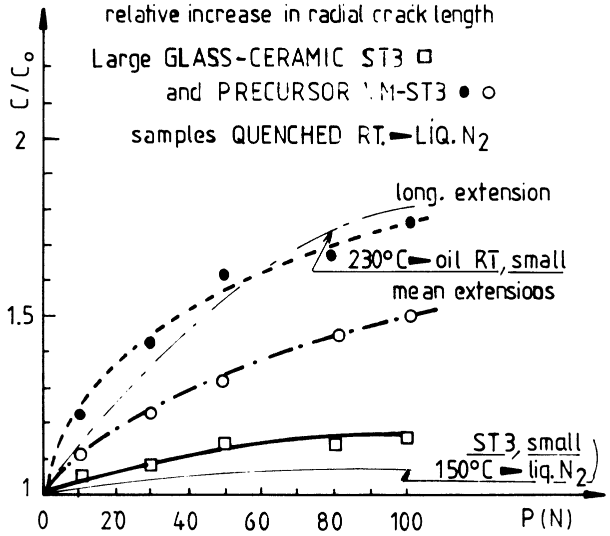

Fig. 4: Effects of sample size and shape and quenching conditions on the relative increase in radial crack length.

The maximum of the thermal transient stress can be obtained in comparing crack extension under thermally induced stresses with crack extension under mechanicall loading. In the present case have the large samples been also fitted with three indentation each and then stressed in four point bending. The procedure was to load to a given stress, to unload, measure the actual crack length, and then to reload to a higher stress, a.s.o. ..., such as to plot step by step the curves of figure 1 The results for VM-ST3 and ST3 are given in figures 5 and 6 respectively. It can be seen first, that the expected shape is reproduced as well as the shorter as-indented crack-lengths, c_o, for the tougher ST3.

Afterwards have the crack lengths after thermal shock been reperted. In the case of ST3 only small crack extension has occured during quenching, and the difference between the two orthogonal extension resp. principal stress directions is hardly discernible. This is not the case for the VM-ST3 and both the extensions in the length and lateral directions have been reported. From those diagrams maxima of the thermal transient stress can be read off.

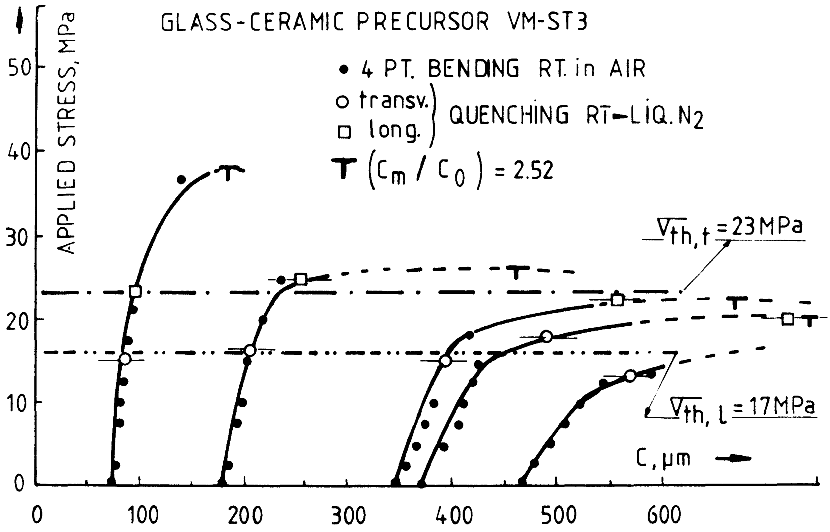

Fig. 5: Deduction of thermal stresses from a mechanical calibration (●) and the length of transverse (O) and longitudinal (□) cracks after quenching for the glass ceramic precursor.

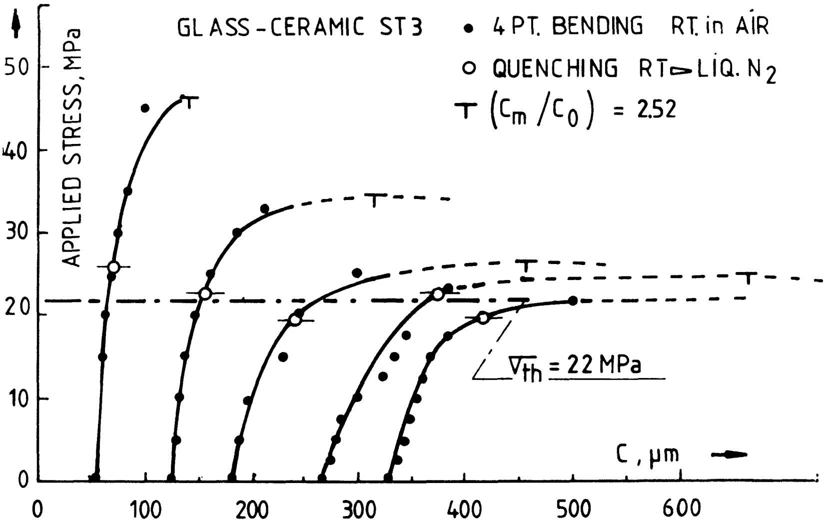

Fig. 6: Deduction of the thermal stress from a mechanical calibration (●) and the length of the cracks after quenching for the glass-ceramics. For given quenching conditions, do the cracks in the tougher glass-ceramic extend much less than in the precursor, although the thermal stress is of the same order of magnitude.

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IV Discussion and conclusion

The final experimental results show the reliability of the Vickers indentation technique in order to rank the severity of a thermal shock or the intrinsic thermal shock resistance of various materials. Furthermore has it proven able to take into account the size and geometry of the quenched samples such as to reveal the anisotropy of the thermal stress state as well as their respective values.

A number of continuum or numerical approaches destinated to calculate the transient thermal stresses have been made. They mostly start from known physical data, such as Young modulus and thermal properties. However, in addition to those concerning the variations of the thermal conductivity and the Biot number during temperature changes, more simplifying assumptions have been made on the value of the coefficient of heat transfer at the interface between the quenched body and the quenching medium.

The proposed methodology allows the maximum value of the thermal transient stress to be evaluated, and be used, in conjonction with other methods such as to calculate the heat transfer coefficient as a function of both quenching conditions and quenching temperature. This would allow the critical quenching temperature to be better defined as a function of sample geometry and the reliability of the rupture predictions be better assessed.

Litterature

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