

# Measurement of Dynamic Fracture Toughness of Ceramic Materials at Elevated Temperature by One-point-bend Impact Test

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## ABSTRACT

A new instrumentation system is developed for measurement of the dynamic fracture toughness of ceramic materials at temperatures up to 1200°C. The impact test involves only central loading with free ends. The specimen is of Charpy type made of silicon carbide, sialon, or PSZ and contains a crack or an 0.2 mm deep notch and is suspended in an infrared image furnace by two thin ceramic threads cemented onto the end surfaces of the specimen. Thus, the heat conduction that usually takes place through a support is kept to a minimum. The impact force is provided by a falling steel cylinder with a pair of semiconductor strain gauges cemented onto the cylindrical surface to measure the stress wave history and hence to determine the impact force. Since the thin threads are broken at the moment of impact, the specimen is assumed to fracture with both ends free from constraint. A simple formula is employed for determining the dynamic stress intensity factor from the measured impact force. A series of impact tests is performed with drop-height increased gradually and the dynamic fracture toughness is determined from the test data of the minimum drop-height that caused specimen fracture. The dynamic fracture toughness values obtained by the impact test are compared with the results of quasi-static tests.

## KEYWORDS

Ceramics; impact test; one-point-bend specimen; dynamic stress intensity factor; dynamic fracture toughness; high temperature fracture toughness.

## INTRODUCTION

The development of the hot-section of gas turbines, turbochargers or conventional engines with ceramic materials is

thought to enable a quantum jump improvement in durability, efficiency, response and multi-fuel capability. Many ceramic materials have high temperature strength as well as hot-corrosion and oxidation resistance much greater than those of metallic alloys. The problem stems from their brittle or nonductile nature and vulnerability to impact loading, so that the use of ceramics as hot-section components requires a knowledge of dynamic fracture toughness at elevated temperatures. However, few investigations have been published on the dynamic fracture toughness of ceramics especially at elevated temperatures. Measurement of dynamic fracture toughness is usually performed on three point-bend specimens at room temperature by means of instrumented Charpy tester or drop-weight tester (Kobayashi et al., 1987). Gonzy and Johnson (1987) carried out three point-bend impact fracture test on hot-pressed silicon nitride specimens at temperature up to 1400°C and found a 20% drop in  $K_{Ic}$  at 1400°C. Peuser (1983) has analyzed the dynamic behavior of three point-bend impact specimen. The impacted specimen first leaves the supports then strikes the supports before leaving for a second time. Thus the modelling of the supports as rigid hinges can lead to mistakes and the supports should be modelled by springs which only be subjected to compression and not to tension. The calculation of the dynamic stress intensity factor therefore involves many complications. To overcome these difficulties Kalthoff et al., (1983) and Giovanola (1986) have introduced one point-bend test in which the impact test involves only central loading with free ends. In the present investigation a new instrumentation system is developed on a basis of one point-bend impact test. A specimen of Charpy type is suspended in an infrared image furnace by two thin ceramic threads cemented onto the end surfaces of the specimen. The impact force is provided by a falling cylinder. Since the thin threads are broken at the moment of impact, the specimen is assumed to fracture with both ends free from constraint. In this arrangement, the heat conduction that usually takes place through supports can be avoided or kept to a minimum.

With ceramics it was difficult to generate a plane crack front of a certain depth. However, Nose and Fujii (1988) have developed a method named "bridge indentation" to introduce a through crack. In the present investigation through cracks were introduced following this method, although specimens with a deep notch cut by a diamond wheel were also prepared.

## MATERIALS AND SPECIMENS

### Materials

The materials tested are silicon carbide (SiC), sialon ( $Si_3N_4$ ) and partially stabilized zirconia (PSZ). The chemical compositions and mechanical properties given by a manufacturer as catalogue data are presented in Tables 1 and 2, respectively. The mechanical properties are measured at room temperature, unless noted otherwise.

Table 1 Chemical composition (wt. %)

SiC	$Si_3N_4$	PSZ
SiC>94%	$Si_{6-z}Al_zO_zN_{8-z}$	ZrO <sub>2</sub>
Al=2.7%	(z=0.4)	3mol%Y <sub>2</sub> O <sub>3</sub>
O=2.5%		

Table 2 Mechanical properties

	SiC	$Si_3N_4$	PSZ
Density(g/cm <sup>3</sup> )	3.08-3.12	3.26	6.08
Young's modulus(GN/m <sup>2</sup> )	382-412	294	209
Fracture toughness(MN/m <sup>3/2</sup> )	4.5	6.0	7.0
Bending strength(GN/m <sup>2</sup> )	R.T. 0.39-0.54 1200°C 0.38-0.43 1400°C 0.37-0.42	0.88	1.47
Poisson's ratio	0.14-0.16	0.30	0.31

### Specimens

The specimens tested are basically of Charpy type (6 mm width, 8 mm height and 50 mm length) with a edge crack or a 0.2 mm deep notch cut by a diamond wheel at the mid-span. The configuration of the specimen is shown in Fig. 1. Bar specimens cut into the specified size and polished on all sides were received from the manufacturer. A through specimen crack was introduced by the bridge indentation method developed by Nose and Fujii (1988) and crack length was measured by using penetration dye technique. The length and morphology of fractured crack surface were studied by scanning electron microscope and optical microscope using polarized and unpolarized light.

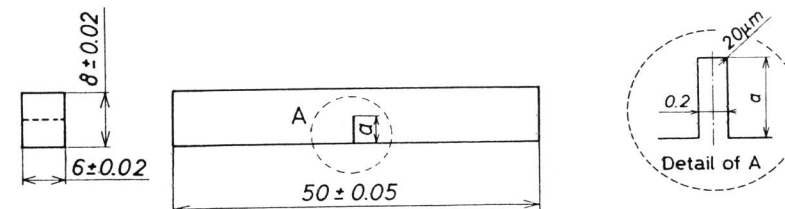


Fig. 1 Specimen configurations

## IMPACT TESTER AND INSTRUMENTATION SYSTEM

Figure 2 shows the general arrangement of the impact tester. The impact test involves only central loading with free ends. A specimen containing a crack or notch is suspended in an infrared image furnace by two thin ceramic threads cemented onto the end surfaces of the specimen and heated up to the specified temperature. By this arrangement the heat conduction that usually takes place through supports can be kept to a minimum. The specimen temperature is monitored by Pt-PtRh thermocouples and it has been confirmed by preliminary experiments that the specimen temperature is controllable within  $\pm 10^\circ\text{C}$  of the specified temperature.

The impact force is provided by a falling steel cylinder (6 mm dia and 1500 mm length) with a pair of semiconductor strain gauges cemented onto the cylindrical surface to measure the stress wave history and hence to determine the impact force. Since the thin threads are broken at the moment of impact, the specimen is assumed to fracture with both ends free from constraint. The falling cylinder is kept aligned with the specimen by two guides and the radius of the spherical impact surface is 100 mm. The length of the falling cylinder and the location where the strain gauges were cemented were designed so

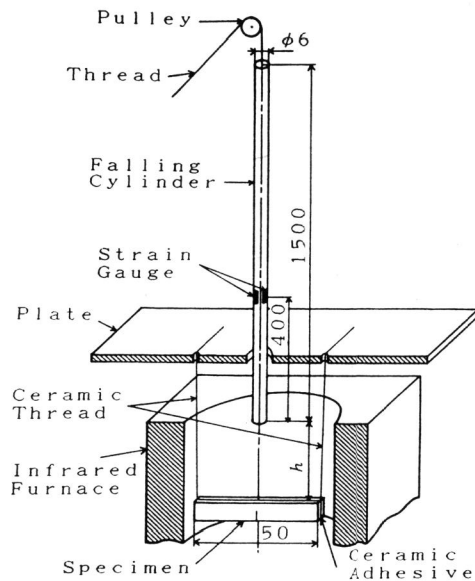


Fig. 2 General arrangement of impact tester

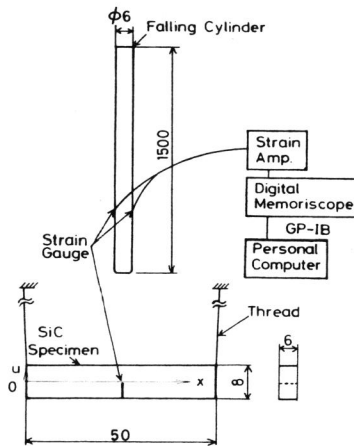


Fig. 3 Schematic representation of impact tester and instrumentation system

that the strain gauges remain out side of the furnace after the impact and the stress waves reflected at the upper surface of the cylinder will not arrive at the location of the strain gauges during the contact period of the cylinder and specimen. The instrumentation system employed is schematically represented in Fig. 3. The use of a microcomputer has enabled quick processing of the experimental data and a preliminary estimation of falling height  $h$  of the cylinder. The experimental results and comparison with the analytical predictions will be described later.

## DYNAMIC ANALYSIS FOR STRESS INTENSITY OF BEND SPECIMEN

### Simple Formula for Dynamic Stress Intensity Factor

Dynamic analysis has been presented for determining the stress intensity factor of impact specimen from a simple formula based on the Euler-Bernoulli beam theory (Nash, 1969; Kishimoto et al., 1980) so that a brief description is given here.

Consider a beam containing an edge notch and having a concentrated force  $F(t)$  as shown in Fig. 3. The equation of equilibrium is given by

$$\frac{1}{S^2} \frac{\partial^2 M}{\partial \xi^2} + \rho A \frac{\partial^2 u}{\partial t^2} = \frac{1}{S} F(t) \delta(\xi - \frac{1}{2}) \quad (1)$$

$$M = \frac{E}{S^2 \Phi} \frac{\partial^2 u}{\partial \xi^2} \quad (2)$$

$$\Phi = \frac{1}{I} + \frac{D}{S} \delta(\xi - \frac{1}{2}) \quad (3)$$

where  $S$  = span of the beam;  $\xi = x/S$ ;  $M$  = moment;  $u$  = transverse displacement;  $B$  = width,  $W$  = height,  $A = BW$  = area and  $I = BW^3/12$  = moment of inertia of cross-section;  $\rho$  = density,  $E$  = Young's modulus of the beam;  $a$  = crack length;  $\Phi$ ,  $D$  and  $V$  = functions representing the effect of the crack on beam deformation; and  $\delta$  = Dirac delta function. Here the following relation holds (Tada et al., 1973)

$$D = \frac{2(1-\nu^2)W}{I} V(a/W) \quad (4)$$

$$V(\alpha) = \left[ \frac{\alpha}{1-\alpha} \right]^2 (5.58 - 19.57\alpha + 36.82\alpha^2 - 34.94\alpha^3 + 12.77\alpha^4) \quad (5)$$

where  $\nu$  = Poisson's ratio and  $\alpha = a/W$ .

The boundary conditions for free edges are given by

$$\frac{\partial^2 u}{\partial \xi^2} \Big|_{\xi=0,1} = \frac{\partial^3 u}{\partial \xi^3} \Big|_{\xi=0,1} = 0 \quad (6)$$

Let the characteristic functions and characteristic values of the beam be denoted by  $Y_{Fm}(\xi)$  and  $\lambda_{Fm}$ , respectively, then the impulse response function, displacement and moment of the beam are respectively given as

$$h_{BF}(\xi, t) = \frac{t}{M_B} + \sum_{m=1}^{\infty} \frac{Y_{Fm}(\frac{1}{2}) Y_{Fm}(\xi)}{\omega_{Fm} W_{Fm} S} \sin \omega_{Fm} t \quad (7)$$

$$u(\xi, t) = \int_0^t F(\tau) h_{BF}(t-\tau) d\tau \quad (8)$$

$$M(\xi, t) = \frac{E}{S^2 \Phi} \int_0^t F(\tau) h_{BF}''(t-\tau) d\tau \quad (9)$$

where  $M_B$  = mass of the beam, prime ' = partial differentiation with respect to  $\xi$  and

$$Y_{Fm}(\xi) = (\cos \frac{\lambda_{Fm}}{2} - \cosh \frac{\lambda_{Fm}}{2})(\cos \lambda_{Fm} \xi + \cosh \lambda_{Fm} \xi) + (\sin \frac{\lambda_{Fm}}{2} + \sinh \frac{\lambda_{Fm}}{2})(\sin \lambda_{Fm} \xi + \sinh \lambda_{Fm} \xi) \quad (0 \leq \xi \leq \frac{1}{2}), \quad (m=1,2,3,\dots) \quad (10)$$

$$\omega_{Fm}^2 = \left[ \frac{\lambda_{Fm}}{S} \right]^4 \frac{E I}{\rho A} \quad (11)$$

$$W_{Fm} = 2 \rho A \int_0^{1/2} Y_{Fm}^2(\xi) d\xi \quad (12)$$

Here, the characteristic value  $\lambda_{Fm}$  is the  $m$ th root of the characteristic equation

$$\cos \frac{\lambda_{Fm}}{2} \sinh \frac{\lambda_{Fm}}{2} + \sin \frac{\lambda_{Fm}}{2} \cosh \frac{\lambda_{Fm}}{2} + \frac{D I}{2 S} \lambda_{Fm} (\cos \frac{\lambda_{Fm}}{2} \cosh \frac{\lambda_{Fm}}{2} - 1) = 0 \quad (13)$$

The dynamic stress intensity factor  $K_I(t)$  is assumed to be proportional to the bending moment at the mid-span and given by

$$K_I(t) = k_n \lim_{\epsilon \rightarrow 0} M(\frac{1}{2} - \epsilon, t) \quad (14)$$

where the constant  $k_n$  is assumed to be determined from the static solution. Substitution of Eqs.(7) and (9) leads to

$$K_I(t) = k_n \frac{E I}{S^2} \sum_{m=1}^{\infty} \frac{Y_{Fm}(\frac{1}{2}) Y_{Fm}''(\frac{1}{2})}{\omega_{Fm} W_{Fm} S} \int_0^t F(\tau) \sin \omega_{Fm}(t-\tau) d\tau \quad (15)$$

Since it is difficult to perform any static one point-bend experiment,  $k_n$  is determined in reference to the analytical relation between the stress intensity factor and the moment at the mid-span of 3 point bend specimen, and represented as

$$k_n = K_S / F(t) \frac{E I}{S^2} \sum_{m=1}^{\infty} \frac{Y_{Sm}(\frac{1}{2}) Y_{Sm}''(\frac{1}{2})}{\omega_{Sm}^2 W_{Sm} S} \quad (16)$$

where  $K_S$  denotes the stress intensity factor of 3 point-bend specimen subjected to static load and is given by (Tada et al., 1973)

$$K_S = \frac{F(t) S}{B W^{3/2}} f(\alpha) \quad (17)$$

$$f(\alpha) = \frac{3\alpha^{1/2} [1.99 - \alpha(1-\alpha)(2.15 - 3.93\alpha + 2.7\alpha^2)]}{2(1+2\alpha)(1-\alpha)^{3/2}}, \quad \alpha = a/W \quad (18)$$

The other quantities in Eq.(16) are given by

$$Y_{Sm}(\xi) = \cos \frac{\lambda_{Sm}}{2} \sinh \lambda_{Sm} \xi + \cosh \frac{\lambda_{Sm}}{2} \sin \lambda_{Sm} \xi \quad (0 \leq \xi \leq \frac{1}{2}), \quad (m=1,2,3,\dots) \quad (19)$$

$$\omega_{Sm}^2 = \left[ \frac{\lambda_{Sm}}{S} \right]^4 \frac{E I}{\rho A} \quad (20)$$

$$W_{Sm} = 2 \rho A \int_0^{1/2} Y_{Sm}^2(\xi) d\xi \quad (21)$$

where  $\lambda_{Sm}$  is the  $m$ th root of the characteristic equation of 3 point-bend specimen given by

$$\tan(\frac{\lambda_{Sm}}{2}) = \frac{2 S}{D I (\lambda_{Sm}/2)} + \tanh(\frac{\lambda_{Sm}}{2}) \quad (22)$$

The dynamic stress intensity factor  $K_I(t)$  of one point-bend specimen is calculated by Eq. (15) for given value of load  $F(t)$ .

Accuracy of Simple Formula

In order to demonstrate the accuracy of the simple formula, a notched specimen was made of steel and a strain gauge was cemented at the vicinity of the notch. The relation between the strain and the stress intensity factor was calibrated by a static 3 point-bend test. The results of a preliminary impact test are shown in Fig. 4, which represents a comparison of the time variation of the stress intensity factor determined from the output of the strain gauge and that of computation with the simple formula used. It is observed that both results are in good agreement at least during the initial stage after the impact.

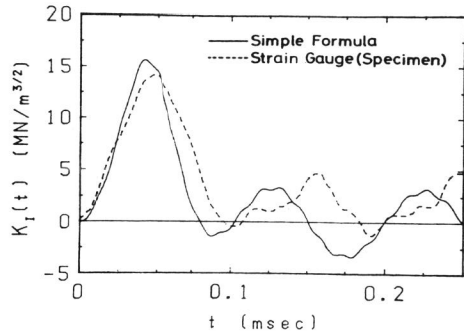


Fig. 4 Comparison of stress intensity factor obtained by simple formula and experiment

ONE POINT-BEND IMPACT TEST

The dynamic fracture toughness  $K_{I_d}$  is determined from the data of time variation of the dynamic stress intensity factor  $K_I(t)$ , if the point of crack initiation is given. However, it is difficult at elevated temperatures to use strain gauges to monitor crack initiation. In the present investigation, a series of impact tests is performed with drop-height increased gradually and the dynamic fracture toughness is determined from the test data of the minimum drop-height that caused specimen fracture. Some of the typical examples of time variation of the impact force and those of the stress intensity factor which were computed from the measured impact force data are shown in Figs. 5(a) and 5(b), respectively. The solid curves represent the results for fractured specimen while the dashed curves represent those for unfractured specimen. There is observed a difference between the curves for fractured and unfractured specimens. The dynamic fracture toughness,  $K_{I_d} = 6.3 \text{ MN/m}^{3/2}$ , is determined as the maximum stress intensity factor of the fractured specimen with drop-height  $h = 53 \text{ mm}$ .

The dependence of dynamic fracture toughness on loading rate  $\partial K_I / \partial t$  of SiC specimen with deep notch is shown in Fig. 6, and the dependence on temperature is presented in Fig. 7. The dynamic fracture toughness of cracked specimens of  $\text{Si}_3\text{N}_4$  and PSZ versus temperature is shown in Fig. 8.

It may be concluded that the system is satisfactory for measuring the dynamic fracture toughness of ceramic materials at elevated temperature.

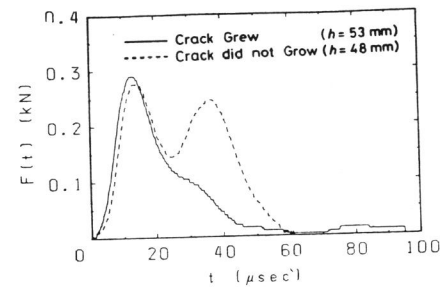


Fig. 5(a) Time variation of impact force

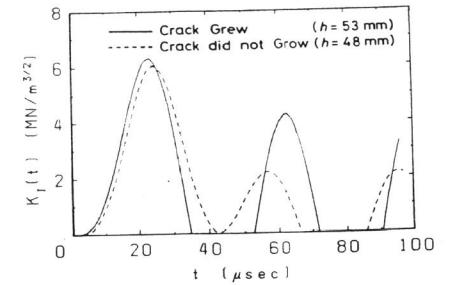


Fig. 5(b) Time variation of dynamic stress intensity factor

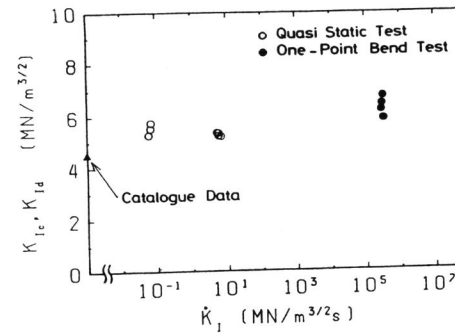


Fig. 6 Comparison of impact test results with quasi-static test results (SiC, notched specimen, room temp.)

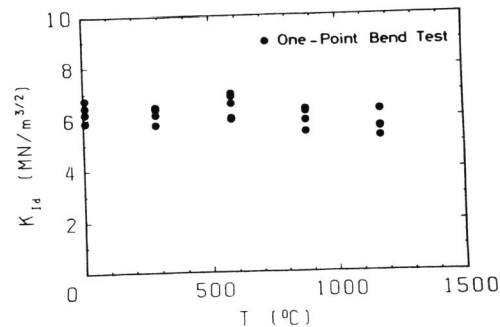


Fig. 7 Dynamic fracture toughness vs. temperature (SiC, notched specimen)

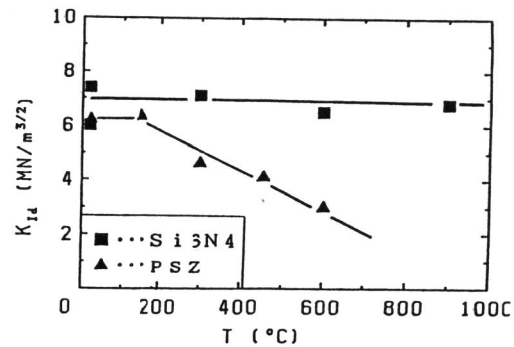


Fig. 8 Dynamic fracture toughness vs. temperature ( $\text{Si}_3\text{N}_4$  and PSZ, cracked specimen)

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