NEW TECHNIQUE FOR MECHANICAL CHARACTERIZATION OF CERAMICS AT ROOM AND AT ELEVATED TEMPERATURE

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An original technique for performing stable fracture tests at room and at high temperature is reported here. This technique uses a laser metrology device to measure crack mouth opening displacement (CMOD) on single-edge-precracked-beam specimens (SEPB). The novelty at this technique is the use of an analog output of this measurement as a feedback signal to control a servo-hydraulic testing machine. Measurements of fracture toughness, specific fracture energy, and R-curve can be done from one single test. This technique was applied to measure fracture properties, at room and at high temperatures, of 3 mole per cent y-TZP.

INTRODUCTION

Because fracture tests in ceramic material are highly unstable, fracture toughness (K_{IC}), determined from indentation tests or from the maximum load in unstable tests, is the parameter widely used for fracture characterization of ceramic materials. Nevertheless, if stable fracture tests were performed, it could be possible to determine the K_{IC}, G_f (specific fracture energy), and R-curve (evolution of K_{IR} with crack growth), and a complete fracture characterization of the material could be obtained with no extra work. Moreover, the post-peak branch obtained in these tests provides useful information about the softening (damage) behaviour of the material.

The most common test geometries for ceramics have high stiffness in comparison with the commercial testing machines, and the parameters easily

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available for controlling the testing machine (load and crosshead displacement) decrease after peak load, and very often this leads to unstable tests.

This contribution presents a new technique using the single edge precracked beam specimens (SEPB) proposed by Nose and Fujii (1), and was applied to stable fracture tests in a zirconia base ceramic at 25, 300, and 600°C. The method employs a laser metrology device similar to that used for tensile creep tests by Carroll et al. (2). This apparatus was selected after a comparison of several extensometric techniques, Baxter (3), Geiger (4), Barlett et al. (5), Jenkins et al. (6), and Gyekenyesi and Bartolotta (7).

This apparatus measures the crack mouth opening displacement (CMOD), and sends the readings to the testing machine. Since the CMOD is monotonically increasing throughout the test, it is a convenient feed-back signal for servo control. Also, this equipment has been used to determine the dilatometric curve of zirconia based ceramic.

EXPERIMENTAL PROCEDURE

A commercial 3.0% mole yttria-tetragonal zirconia polycrystal (3Y-TZP) machined into rectangular bars (50x10x5 mm^3) was used for this research. The sintered material contains a 26 per cent volume fraction of monoclinic residual phase, and a 74 per cent volume fraction of tetragonal phase. Though most of the grains are submicronics, with an average grain size of 340 nm, ten per cent of the grains are larger than 6 μm.

Straight through cracks, with a length between 3 and 6 mm, were made in the specimen using the "bridge" indentation technique (1). In order to provide gauge marks for the measurement of the CMOD, two small alumina pins were fixed at each side of the crack (Figure 1) with a high temperature ceramic bond.

Tests were performed with an Instron 8501 servohydraulic testing machine that accepts analog inputs from external measuring devices. The loading system consists of a ceramic three point bending device with 40 mm loading span, which is placed inside a furnace. The load is applied to it by means of two alumina bars, connected respectively to the load cell and to the crosshead. Both ends of the alumina bars were water-cooled.

The non-contact laser extensometer selected is similar to that reported in (2). It consists of a He-Ne Laser emitter that scanned in a horizontal line 250 times per second. This laser scan is driven across the furnace through two silica windows (Figure 1). When an obstacle crosses the laser beam, it generates a shadow in the detector on the opposite side of the furnace. A 32-bit microcomputer, connected to the emitter and to the detector, determines the distance between the internal borders of the shadows projected by the two alumina pins (gauge marks). The variation of this distance is the CMOD. Also, the microcomputer supplies a 16-bit analog output that is used as a feedback signal to control the testing machine. The gain of this output was selected as 100 mV/μm.

The displacement of the cross-head of the machine was measured by means of a LVDT of ±2 μm accuracy, and a load cell of ±5 kN full scale measured the load.

ANALYSES OF THE CMOD FLUCTUATIONS

In order to control the testing machine with the CMOD signal coming from the laser extensometer, special care must be exercised in reducing the signal fluctuations. Although the resolution of this extensometer is very good, excessive fluctuations of the air density limit the optical accuracy of the equipment (2), so that, the thermal gradients inside the furnace chamber must be minimized. Our testing furnace has 150 mm thick insulating walls, and frictionless seals made of alumina tissue avoided the chimney effect.

Despite the good thermal isolation of the testing chamber, high frequency fluctuations appear in the readings. These fluctuations can be damped by averaging over a number of laser scans. However, every scan takes a small but finite amount of time (1/250 s), and since averaging is necessary to reduce excessive fluctuations, the feed-back signal is stepped rather than continuous. The output update interval Δt_u is given as the ratio between the number of averaged scans, N, and the scanning rate of 250 s^{-1}:

$$\Delta t_u = 4N\ 10^{-3}\ s$$

The effect of averaging over a certain number of scans is shown in Figure 2. A reading was taken every 3 10^{-3} s to record the very low period fluctuations. When the extensometer is not sending any reading, an electronic noise appears, with a standard deviation (RMS) of ±0.007 μm. This electronic noise threshold is unavoidable and appears in all the measurements. Now, if we measure a constant distance and average over a different number of scans, we can see that the RMS of the fluctuations decreases with the increasing number of averaged scans from 0.099 μm for 5 scans to 0.028 μm for 300 scans, while the period between two consecutive readings -slap period- rises from 0.09 to 1.2 seconds respectively.

Thus if averaging is performed over a short number of scans, the machine again runs out of control because of excessive fluctuation amplitude. On the other hand, if averaging is performed over a large number of scans, the fluctuations of the signal are reduced, but the output update interval is longer. If the delay is longer than about 1.5 seconds, the machine runs out of control.

Experimentally, averaging over 150 scans was found to be a good setting for our testing device. With this number of scans the fluctuations were kept within reasonable bounds, as shown in Figure 2, and it is possible to control the testing machine.

The influence of the temperature on the readings is shown in Figure 3, where a record was taken each second after averaging over 150 laser scans. It can be seen that the standard deviation of the readings in an unloaded specimen at 25 and 600 °C after ten minutes, are similar and better than ±0.2 μm. Therefore, the thermal insulation of our experimental set-up is good enough to avoid unwanted thermally induced fluctuations.

EXPERIMENTAL RESULTS AND DISCUSSION

Load-CMOD curves at 25, 300, and 600 °C, and the associated Load-Displacement curves, are shown in Figure 4. The snapback displayed in the Load-Displacement curves is characteristic of an experimental device leading to unstable tests in cross-head control. So, the CMOD control is absolutely necessary to perform stable fracture tests with this set-up.

Now, we can obtain the apparent fracture toughness, K_{IQ}, from the peak load and the initial crack length, using Tada's equation, Tada et al. (8). The specific fracture energy, G_f, can be calculated by dividing the work spent in completely fracturing the specimen (area under the Load-Displacement curve) by the surface of the initial uncracked ligament.

To determine the crack growth resistance curves, R-curves, the evolution of crack length must be determined. Since the experimental set up does not direct measurement of the crack length, the compliance method was used. In this method, the equivalent elastic crack length is found by writing that the experimental secant compliance is the one corresponding to a perfect linear elastic body. Using the known formulas for the compliance as a function of the crack length and equating them to the experimentally measured compliance, the equivalent crack length is determined (9). Then the stress intensity factor is computed from the load and crack length, and the crack growth resistance is found from Irwin´s relationship, $R = K_I^2/E$. The resulting R-curves are shown in Fig. 5.

The decrease of the fracture parameters as the temperature rises is shown in Table 1 (for toughness, fracture energy) and in Figure 5 (for the R-curve). To explain this behaviour, we have to consider that one of the main toughening mechanism in zirconia based ceramics is the martensitic transformation of tetragonal particles to monoclinic phase (t→m), Evans and Cannon (10). There is a temperature, A_s, at which the initial monoclinic phase (26%) starts to transform to tetragonal phase (m→t). Therefore, for temperatures higher than A_s, as more monoclinic phase is being transformed to tetragonal, it would be more difficult to induce the tough transformation, t→m, so the fracture properties of the material will decrease. Finally, there is a higher temperature, A_f, at which all the initial monoclinic phase has been transformed completely to tetragonal phase. At this temperature, A_f, the tough transformation t→m is virtually impossible and the material exhibits a toughness similar to the pure monoclinic material.

TABLE 1 — Mean Values of Toughness and Fracture Energy at 25, 300, and 600 °C.

T (°C)	K_{IQ} (MPa m$^{1/2}$)	G_f (N/m)
25	3.9	108.1
300	2.8	42
600	2.0	17

Measurement of A_s and A_f temperatures can be done with the same device. Since the martensitic transformation (m→t, or vice versa) implies a volume variation of about 4-6%, Heuer (11) , if it were possible to determine the dilatometric curve of the material, we could obtain these two temperatures. In order to determine the dilatometric curve of the 3Y-TZP, the laser extensometer was placed to perform a vertical scan inside the furnace. In this configuration the equipment measured the linear increment of the diagonal of a square section specimen. Figure 6 shows the dilatometric curve of this material, and the transition temperatures A_s = 290 °C, and A_f = 625 °C. These temperatures are in agreement with the variation of the fracture parameters obtained previously, which decrease as the temperature increases.

CONCLUSIONS

1. An experimental procedure to conduct stable fracture test on single-edge-precracked-beam ceramic specimens at room and at high temperature, was developed.

2. The method uses a CMOD analog signal, measured with a laser extensometer, to servo-control a hydraulic testing machine. For the control process, a very good thermal insulation of the furnace is essential to achieve low fluctuation measurements. Multiple scan averaging further reduces the fluctuations of the output. However, the averaging cannot be extended to a large number of scans because large output update intervals make the machine run out of control.

3. Stable fracture tests provide more information (fracture toughness, specific fracture energy and crack growth resistance curves) than the traditional unstable fracture tests based only on the measurement of the maximum load, or on indentation techniques.

4. The results for 3 mole percent Y-TZP show that toughness, fracture energy, and R-curve decrease quickly as the temperature rises.

5. With the proposed experimental device, it was also possible to measure the dilatometric curve of the 3Y-TZP, and justify the dependence on temperature of the fracture parameters.

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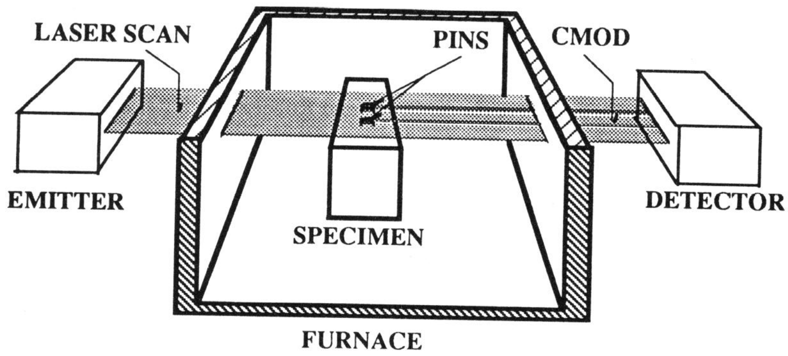

Figure 1 Simplified view of the CMOD measuring device

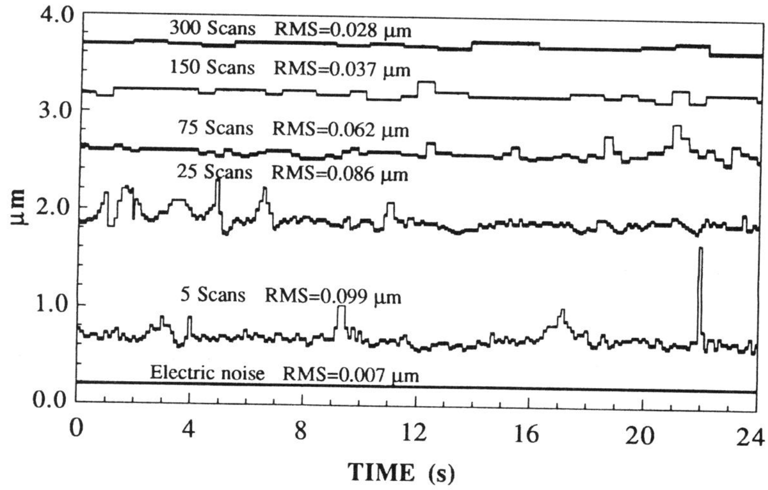

Figure 2 Effect on the CMOD of the increasing scan number average

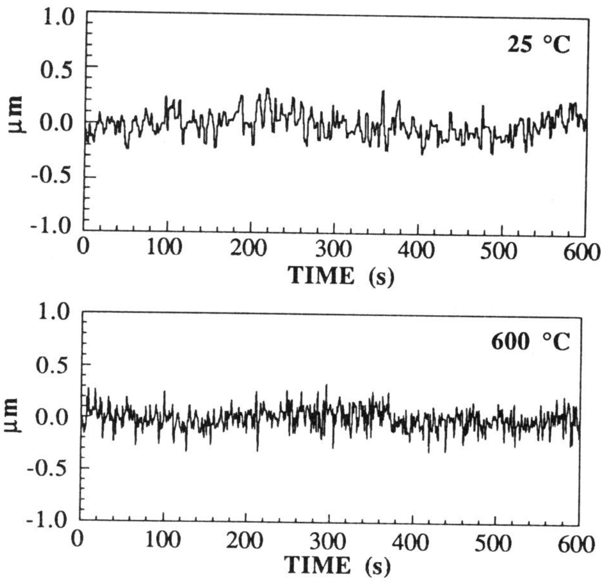

Figure 3 CMOD signal noise at 25 and 600 °C

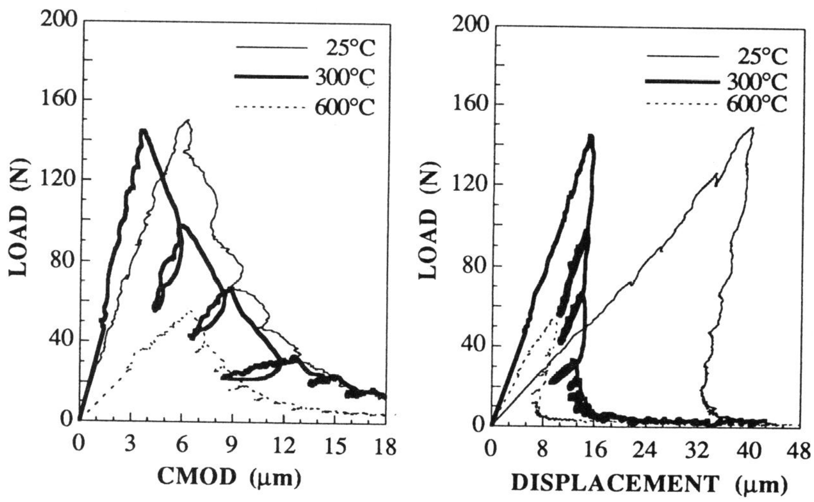

Figure 4 Load-CMOD and Load-Displacement curves

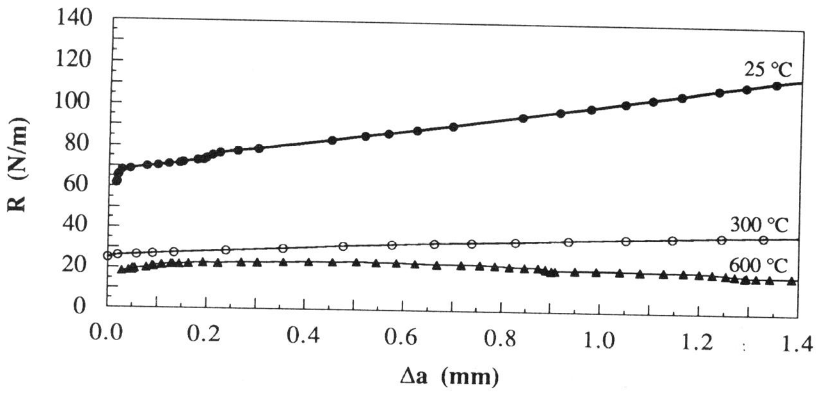

Figure 5 R-curves at 25, 300, and 600 °C

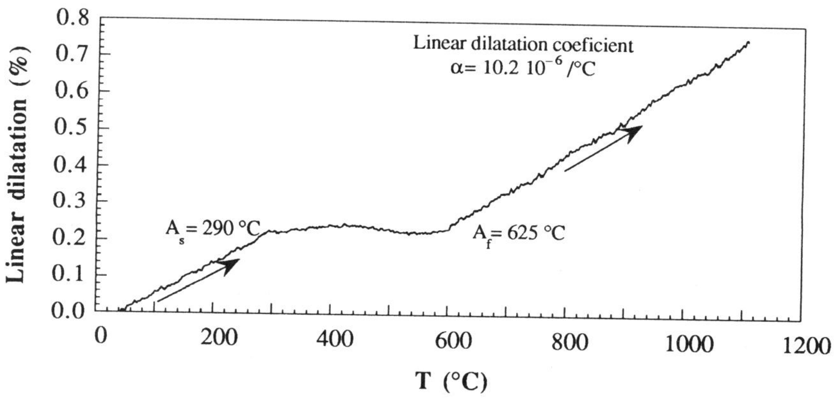

Figure 6 Dilatometric curve for 3Y-TZP