CONTROLLED FRACTURE TESTS OF BRITTLE CERAMICS

C. Baudín[1], A. García[1] , J. Hernández[2], M. López[2]

[1] Instituto de Cerámica y Vidrio, CSIC
CSIC-Campus de Cantoblanco, Kelsen 5, 28049 Madrid, Spain
e-mail: cbaudin@icv.csic.es

[2] Microtest S.A.
Valle de Tobalina 10, 2801 Madrid, Spain
e-mail: mla@microtest-sa.com

ABSTRACT

Controlled fracture tests are required for the accurate determination of the toughness parameters of materials in order to assure the full conversion of the supplied energy into crack surface energy. From the three parameters involved in the test, load, displacement of the load frame and crack mouth opening displacement (CMOD), this latter is the only one that continuously increases as fracture proceeds. Therefore, the CMOD has been proposed as control variable for the stable fracture tests. In this work, a new equipment to perform stable fracture tests of ceramics controlled by the CMOD is presented. The developed equipment allows performing stable fracture tests of extremely brittle ceramics such as fine grained magnesium-aluminium spinel. The developed equipment is presented together with results obtained for fine grained ceramic using different experimental conditions.

KEY WORDS: ceramics, stable fracture, toughness

1. INTRODUCTION

Stable crack growth is necessary to get reliable and accurate fracture toughness data. If the fracture toughness values are determined from test configurations that do not allow stable crack growth, then the measurement have to be solely related to crack initiation [1]. In such cases, the calculated value of the fracture toughness may be over-evaluated. In addition to the conventional fracture toughness, stable fracture tests allow the determination of fracture energy and crack-growth resistance curves.

Controlled fracture tests for brittle materials as most ceramics are difficult to accomplish, therefore, they are not usually performed. The chevron notched geometry allows reaching stable fracture in brittle specimens using the displacement of the loading frame as control parameter [2] but specimens are difficult to fabricate especially for fine grained materials. For other geometries such as straight notch beams in flexure and load frame displacement controlled tests, stable fracture is only reached for materials with some extent of R curve behaviour [3-4]. The load frame displacement should decrease after the peak load for stable testing of brittle materials.

The crack mouth opening displacement (CMOD) is the only parameter which increases throughout the whole test. CMOD controlled stable fracture tests have been reported for advanced ceramics with R-curve behaviour such as yttria-partially-stabilized zirconia [5] but not for extremely brittle ceramics such as fine grained magnesium-aluminium spinel. Such kind of tests is performed using specific experimental laboratory set ups.

In this work, a new equipment to perform stable fracture tests of ceramics controlled by the crack mouth opening displacement (CMOD) is presented.

2. EQUIPMENT

2.1 Loading device

The MICROTEST EM1/50 (Figure 1) is a single screw, dual column, servo-controlled electromechanical test machine. Electromechanical or universal testing machines are most commonly used for static testing in a tensile or compression mode within a single frame. The control is performed by an electronic system that generates the control signal to make the actuator move the crosshead in an upward or downward direction via a drive system. The different test set ups are placed between the rigid frame (stiffness < 2.10^8 N/m) and the moving crosshead.

The maximum load range is 50kN, both tension and compression. The displacement range is 0-100mm and the maximum speed is 100mm/min. The actual applied loads are measured by extensometric load cells mounted in the line of force application. The displacement of the moving crosshead can be controlled and measured by means of an optical encoder placed in the motor axis.

The SCM3000 electronic controller includes load and position channels as well as position auxiliary ones with the option to add additional strain channels for extensometers. In this case, the signal from the contactless optical measurement system for CMOD determination is directed to one of these auxiliary channels. Therefore, the control parameters can be not only force and displacement but also CMOD.

Figure 1. Test machine MICROTEST EM1/50 with the experimental set up.

The test specimen is placed between the rigid frame and the moving crosshead in a stainless steel three points bending test fixture with a span of 40 mm (Figs. 1-2) with a load cell of 5KN. This load cell was selected to assure high stiffness of the loading setup. The stiffness of the machine, load cell, and supports arrangement was determined experimentally using an uncracked alumina bar (4mm x 6mm x 50mm); the obtained values were $1.6x10^6$ N/m up to 20N and $1.7x10^7$ N/m up to 150N.

2.2 Crack mouth opening displacement measurement unit.

For the CMOD measurement and control, a high precision optical micrometer KEYENCE LS7010 is used (Figures 1 and 2). This optical system provides a measurement accuracy of ± 0.5µm and repeatability of ± 0.06µm. The measurement range of this optical system goes from 0.04mm to 6mm. The equipment carries out a continuous measurement, averaging up to 2400 samples/s, and so very stable readings are obtained.

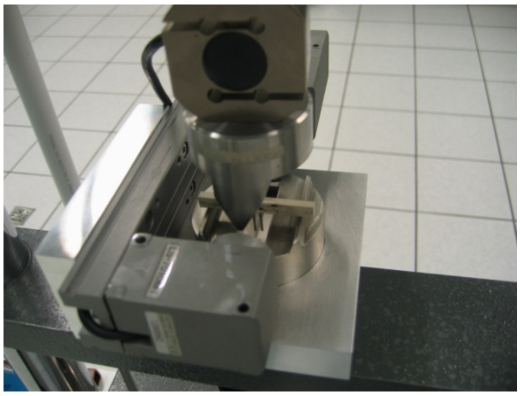

Figure 2. Detail of the three point bending device with the optical micrometer.

The principle of measurement of the optical system is as follows (Figure 3). A high-intensity GaN green LED radiates light, which is changed into uniform parallel light through the special diffusion unit and collimator lens and emitted to the target in the measuring range. This parallel beam “illuminates” measurement area. Then the shadow image of the target appears on the HL-CCD (high-speed linear CCD) through the telecentric optical system. With the telecentric system of lenses the size of the image on the CCD does not change even if it moves, thus, the same precision all along is maintained

The output incident signal of the HL-CCD (charge coupled device) is processed by the DE processor (by means of a digital edge-detection algorithm) in the controller and CPU. The detection threshold is an adjustable parameter. As final result, it is possible to obtain the separation between two pins such as those used, as described below.

The measuring head incorporates a CMOS (complementary metal oxide semiconductor) camera to capture real-time image of the target.

The controller of the optical system incorporates a function of elimination of abnormal values, to improve the precision of the measurement. This function ignores the abnormal values exceeding a preset value to prevent malfunctions caused by dust or other irrelevant factors. Figure 3 shows other interfaces not used in the present setup (RS-232C input/output, 2 channel BCD output, 2 I/O channels, and 2 analog output channels).

2.3 Setup

The optical micrometer is attached to the lower loading support; in this way the mechanical interferences are avoided and a correct orientation of the light beam with respect to the axis of load and the bending fixture is assured.

Given the small opening displacement of the notch, in order to be able to detect and measure its width during the test (the size of detectable minimum object by the system is of 0.04mm), pins of 1.5mm in diameter and 12mm length are adhered to both sides of the notch so that they are perpendicular to the light beam. In this way the equipment detects the edges of these elements giving the straightforward values of CMOD variations.

The analog output signal from the optical device is connected to the control and measurement system SCM3000 of the electromechanical MICROTEST testing machine, so the CMOD can serve as control variable during the test. This allows performing fracture tests at constant CMOD rate.

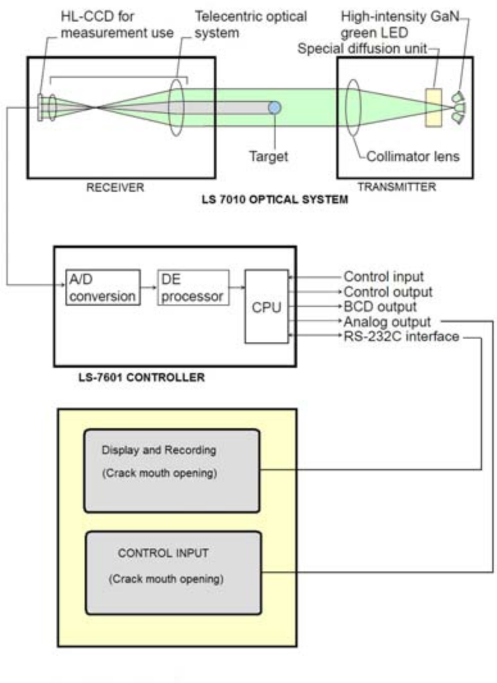

Figure 3. SCM 3000 system

The analog channel in the SCM3000 electronics receives the CMOD. The 16 bits A/D converter in the SM3000 system converts the continuous CMOD analog signal to discrete digital numbers proportional to the magnitude of the voltage of this signal. The whole output analog range corresponds with the measurement range of the optical micrometer (6mm) and this is converted in 2^{16} levels (65536 values). That means that a theoretical resolution of 0.1μm is obtained for this maximum range. In order to reach the highest accuracy the tests were performed at 20 ºC, with a separation between edges of pins bonded to the sides of the crack of about 1mm. The repeating accuracy of the optical micrometer for this separation was checked to be ± 0.06μm with 1.0 mm-diameter round bar located in the centre of the measuring area.

The output analog range of the optical micrometer selected by this test is ±50μm that corresponds with the scaling value of 5 μm/V. That means that a theoretical resolution of about 0.02μm is obtained. This converted signal is used to control the CMOD rate by the microprocessor in the SCM3000 system. The process makes use of a PID algorithm with adjusted parameters to perform and maintain the desired CMOD speed during the test. The equipment carries out a continuous measurement of the value average of 512 samples/s, and so a very stable reading of the separation of the edges of the crack mouth is obtained.

The motor driver uses the control signal from the SCM3000 system to generate the suitable current that operates over the motor to adjust the CMOD rate. The measurement of the CMOD by the LS7010 allows the control loop to be closed (Fig. 3).

3. SPECIMEN PREPARATION

A fine grained magnesium-aluminium spinel labelled SP, with density of 3.46 ± 0.03g/cm^3 and Young's Modulus of 251 ± 13GPa, was prepared from a commercial spinel $MgO.Al_2O_3$ powder and sintered at 1750ºC for 2h [6].

Single Edge V-Notch Beams (SEVNB) of 4mm x 6mm x 50mm were diamond machined. The notch was initially cut with a 300 μm wide diamond wheel. Using this pre-notch as a guide, the remaining part of the notch was done with a 150 μm wide razor blade sprinkled with diamond pastes of successively 15 and 6μm. Tip radii of about 25 μm were obtained (Fig. 4). The relative notch length, a/D (a=notch length, D=width of the specimen), was 0.64.

One cylindrical steel pin was glued at each side of the notch. The diameter of the pins was 1.5 mm and the length was 12 mm.

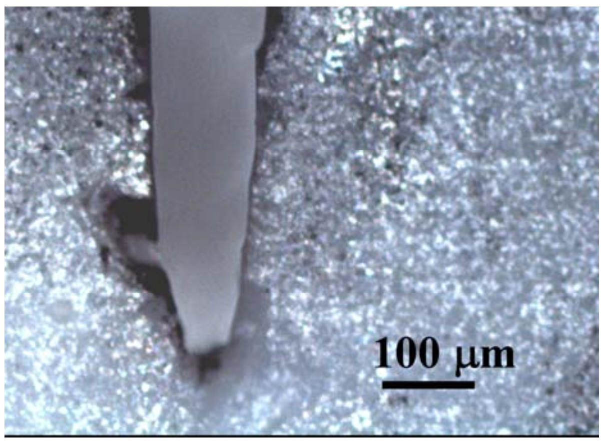

Figure 4. Notch for SP specimens. The black coloration of the tip corresponds to the diamond used.

4. TESTS AND DATA ANALYSIS

Figures 5 and 6 show the CMOD-time plots corresponding to tests performed at different constant CMOD rates.

The programmed CMOD rates were reached in all cases. Variations of less than about, 0.02 μm were obtained in all cases, demonstrating the very stable reading of the separation of the CMOD obtained.

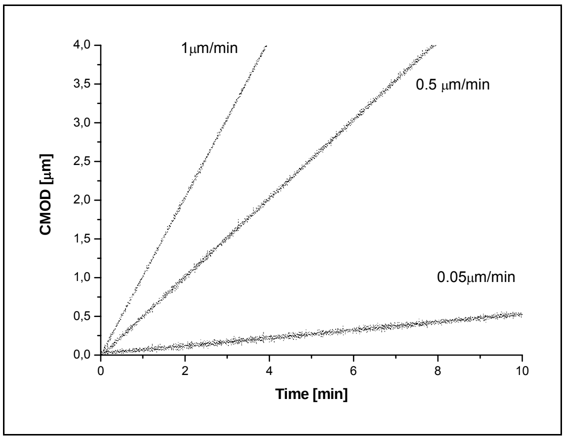

Figure 5. CMOD-time for tests performed at three different CMOD rates.

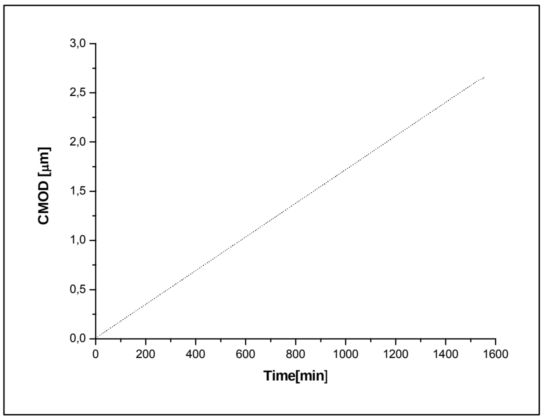

Figure 6. CMOD-time curve for SP tested at 0.1 μm/min.

Figure 7 is characteristic of a setup leading to stable tests in CMOD control. It increases throughout of the whole test.

Figure 8 shows the load-displacement curve corresponding to the tests of figures 6 and 7. From the maximum load, decreases in the displacements are needed to reach stable tests.

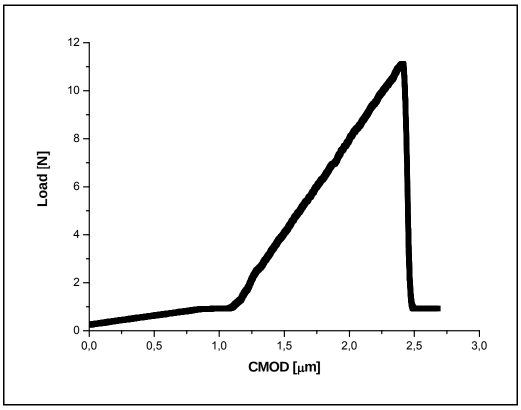

Figure 7: Load-CMOD plot.

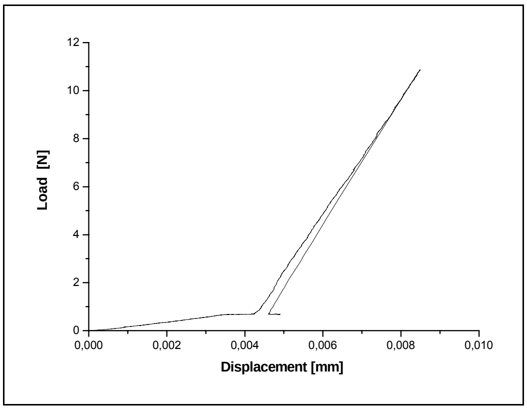

Figure 8: Load-displacement plot.

Using the stiffness values determined for maximum loads lower than 20N, the properties of the material and the analysis in ref [7], stable fracture tests could not be obtained under displacement control for this spinel. On the contrary, stable fracture tests have been possible for specimens with relative notches of about 0.64 using CMOD control.

The CMOD for a general three point bend specimen, can be derived by superposing the solution for a reference beam with fixed L/D ratio (=4) and the solution for pure bending in adequate proportions so as to give the same law of bending moments over the central part [8]. General expressions have been determined for the CMOD and its inverse function, giving the crack length as a function of CMOD, equations (1), (2) and (3). These expressions are valid for any crack length and for span-to-depth ratios larger than 2.5.

$$\alpha = \frac{\gamma^{3/3} + m_1(\beta)\gamma}{[\gamma^2 + m_2(\beta)\gamma^{3/2} + m_3(\beta)\gamma + m_4(\beta)]^{3/4}}$$
(1)

where γ is given by

$$\gamma = \frac{w_M BE'}{6P} \tag{2}$$

and

$$\begin{aligned}
m_1(\beta) &= \beta(0.25 - 0.0505\beta^{1/2} + 0.0033\beta) \\
m_2(\beta) &= \beta^{1/2}(1.155 - 0.215\beta^{1/2} + 0.0278\beta) \\
m_3(\beta) &= -1.38 + 1.75\beta \\
m_4(\beta) &= 0.506 - 1.057\beta + 0.888\beta^2
\end{aligned} \tag{3}$$

For three-point-bend beams, the values of K_{IC} can be determined from the notch depths and the maximum loads reached in the tests according to a general expression, equation (4), for the stress intensity factor , valid for any value of the crack-to-depth ratio ($0 \leq \alpha \leq 1$) and span-to-depth ratios larger than 2.5 ($2.5 \leq \beta \leq 16$):

$$\begin{aligned}
K_I \big/ K_0 (\alpha,\beta) &= \frac{p_\infty(\alpha) + 4/\beta[p_4(\alpha) - p_\infty(\alpha)]}{(1-\alpha)^{3/2}(1+3\alpha)\sqrt{\pi}} \\
K_0 &= \frac{3LP}{2BD^2}\sqrt{\pi a}
\end{aligned} \tag{4}$$

where w_M is the crack opening displacement, L is the span, P is the maximum load, B and D are the width and the depth of the bars, , a is the crack length, α is the crack-to-depth ratio ($\alpha = a/D$), β is the span-to-depth ratio ($\beta = L/D$).

The $p_4(\alpha)$ and $p_\infty(\alpha)$ given by equations (5) and (6) are cubic polynomial for $\beta = 4$ and $\beta = \infty$ (formally equivalent to pure bending).

$$p_4(\alpha) = 1.9 + 0.41\alpha + 0.51\alpha^2 - 0.17\alpha^3 \tag{5}$$

$$p_\infty(\alpha) = 1.99 + 0.83\alpha - 0.31\alpha^2 + 0.14\alpha^3 \tag{6}$$

The fracture toughness value, K_{IC}, was calculated using the general expression of the stress intensity factor (eq. 4) and the value of the maximum load attained during the test. The onset of crack propagation was considered in the peak load. The value of K_{IC} obtained was 1.4 MPa $m^{1/2}$. It was 50% lower than the value determined in unstable tests (≈3 MPa$m^{1/2}$ [6]).

From K_{IC} and Young`s modulus, G_{IC}, can be calculated according to the analysis of Irwin that relates the stress-derived fracture toughness (K_{IC}) and the energy-derived fracture toughness (G_{IC}) for plane strain conditions [4]:

$$G_{IC} = \frac{K^2{}_{IC}}{E'} \tag{7}$$

where $E' = E/(1-\nu^2)$ is the generalized Young´s modulus for plane strain (E is the Young´s modulus and ν is the Poisson´s ratio). The value of G_{IC} obtained was 7.5 J/m^2

6. CONCLUSIONS

An experimental device to perform stable fracture tests of ceramics by controlling the CMOD has been developed. Stable fracture tests for extremely brittle ceramics such as a fine grained magnesium-aluminium spinel have been performed using this device. A toughness value is about 50% lower than ref. [6].

ACKNOWLEDGEMENTS

Projects: MEC MAT2006-13480 C02 and MCI-PET 2008-0113

REFERENCES

[1] B.F. Sorensen , A. Horsewell, O. Jorgensen and A.N. Kumar, "Fracture Resistance Measurement Method for in situ Observation of Crack Mechanisms", *J. Am. Ceram. Soc.*, 1998, 81, [3], 661-69.

[2] A. Ghosh, M.G. Jenkins, K.W. White, A.S. Kobayashi and R.C. Bradt, "Elevated-Temperature Fracture Resistance of a Sintered α-Silicon Carbide", *J. Am.Ceram. Soc.,* 1989, 7, [2], 242-247.

[3] H. Wieninger, K. Kromp and R.F. Pabst, "Crack resistance curves of alumina and zirconia at room temperature", *J. Mat. Sci.,* 1986, 21, 411-418.

[4] S. Bueno, M.H. Berger, R. Moreno and C. Baudín, "Fracture behaviour of microcrack-free alumina-aluminium titanate ceramics with second phase nanoparticles at alumina grain boundaries", *J. Eur. Ceram. Soc*, 2008, 28, 1961-1971.

[5] J.Y. Pastor, J. Planas and M. Elices, "Ambient and High-Temperature Stable Fracture Tests in Ceramics: Applications to Yttria-Partially-Stabilized Zirconia", *J. Am. Ceram. Soc*, 1993, 76 [11], 2927-29.

[6] C. Baudín, R. Martinez and P. Pena, "High-Temperature Mechanical Behaviour of Stechiometric Magnesium Spinel", *J .Am. Ceram. Soci*.,1995, 78 [7], 1857-62.

[7] I. Bar-On, F. I. Baratta and K. Cho, "Crack Stability and Its Effect on Fracture Toughness of Hot-Pressed Silicon Nitride Beam Specimens", *J. Am. Ceram. Soc.*, 1996, 79 [9], 2300-308.

[8] G.V. Guinea, J.Y. Pastor, J. Planas and M. Elices, " Stress intensity factor, compliance and CMOD for a general three-point-bend beam", *Int. J. Fract.*, 1998, 89, 103-116.