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Indentation crack profiles of cordierite materials under mechanical and thermal biaxial stresses

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Abstract

The indentation crack profiles under mechanical and thermal stresses of cordierite materials obtained from a commercial powder were studied. Disks were prepared by slip casting and sintering, and they were indented (Vickers; 44.1 N; 15 s) at the center of one of their surfaces to be subjected to flexion in the mechanical test or to a sudden temperature change in the thermal shock test. Indented specimens were fractured in biaxial flexure (BF) or thermal shock (TS) tests. Some indented disks were thermally treated following two different schedules that involve exposures at high temperatures without (T_1) and with a sudden cooling (T_2). These specimens were fractured in biaxial flexure to observe the fracture surfaces. In all specimens (BF, TS, T_1 and T_2), the evolution of the indentation crack profiles was fractographically analyzed by SEM. In order to study the crack shape and the profile evolution, both the length and the depth of the indentation cracks were measured.

Keywords: Fracture; Thermal shock resistance; Cordierite; Indentation

1. Introduction

Techniques based on the analysis of indentation cracks have been extensively used for the mechanical characterization of ceramics, especially to evaluate the fracture toughness. Less frequently, these techniques have also been used for the evaluation of thermal shock behaviour.[1,2]

In order to analyze the results of an indentation method, the consideration of the residual stresses generated around the contact site is essential because their presence complicates the data interpretation. This situation is more critical when high temperature tests are performed as regards that this condition could act assisting the subcritical growth of indentation cracks. The majority of the works on this matter have been carried out over transparent materials (glass), where the crack profile evolution is easily followed, but studies on polycrystalline materials are scarce.[3,4] Some remarkable facts were observed in those studies. The stable crack growth can occur even in the absence of the residual stress due to the radial/lateral crack interaction process.[5] It has been often observed that the indentation crack geometry evolves towards a different shape when stable crack growth takes place.[5–8] This fact influences lifetime predictions and it has been recognized that this variation in crack shape have to be taken account for accurate calculations.[7] In polycrystalline materials, subcritical crack extension with a change of crack geometry was observed in as-indented samples but not in annealed ones.[3,4]

In the present paper, the crack evolution of an indented cordierite material under mechanical and thermal stresses is studied. Changes in crack profiles were analyzed by fractography using SEM method that is unusual in this type of studies. Since cordierite ($2MgO{\cdot}2Al_2O_3{\cdot}5SiO_2$) is commonly used as a thermal shock resistant material, the understanding

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of its behaviour under this condition where the subcritical crack growth could occur results essential.

2. Experimental procedures

2.1. Specimen preparation and characterization

A commercial cordierite powder (CORCR Baikowski-France, 99.8 wt.%; average particle size, $D_{50} = 2.1$ μm; specific surface area, $S_S = 2.5\,m^2\,g^{-1}$; picnometric density, $\delta_{PIC} = 2.6\,g\,cm^{-3}$)[9] was used as starting material. Disks (diameter: 35.00 ± 0.02 mm, height: 2.67 ± 0.01 mm) were fabricated by slip casting of an aqueous suspension with 65 wt.% solid loading and 0.5 wt.% deflocculant (Dolapix CE64, Zschimer-Schwarz, Germany). Green samples were fired at 750 °C for 1 h, ground with SiC papers (120, 320 and 600 grit) and sintered in an electrical furnace ($MoSi_2$ heating elements) at 1450 °C for 2 h at a heating rate of 1 °C min^{-1}.

Cordierite and mullite were identified as the crystalline phases in the sintered bodies (XRD and FTIR)[9]. An apparent density $\delta_S = 2.50\,g\,cm^{-3}$ was determined (Archimedes method in water) giving a densification degree (%δ_S/δ_{PIC}) of 97.27%.[1] Microstructures with mainly equiaxed cordierite and mullite grains of similar submicron size (mean size ≈ 0.5 μm) were observed by SEM.[1,10] In addition, only a few elongated grains (aspect ratio ≈ 1.9) attributed to mullite were observed. A glassy phase (63 wt.% SiO_2, 25 wt.% Al_2O_3, 12 wt.% MgO; $\delta_{PIC} = 2.51$ g/cm^3)[9] was identified at triple points but it could also be present at grain boundaries because of its ability to wet the cordierite and mullite grains. Intergranular pores of size <10 μm, homogeneously distributed, were observed. In addition, a few isolated pores up to 40 μm, unavoidable in a slip casting process, were observed. The total porosity calculated from the apparent density was ≈3%.

2.2. Indentation

The disks were indented at the center of one of their surfaces using a Vickers indenter (Tukon 300 Microhardness Tester) at a load of 44.1 N and at a loading time of 15 s.

After removal of the indenter, an acceptable crack pattern was observed, in general: a symmetrical indentation with four well developed cracks from its corners, with median/radial geometry. A mean value of the crack length c_0 of 152 ± 17 μm was measured on a SEM image of the polished indented surface (diamond pastes up to 1 μm).

2.3. Mechanical and thermal shock tests

The mean fracture strength (σ_F) of the indented disks (labelled BF) was evaluated in biaxial flexure, employing a loading ball (8.04 ± 0.02 mm in diameter) on discontinuous ring fixture (19.50 ± 0.1 mm in diameter, 12 balls in contact).[1,11–13] The tests were performed (Instron Testing Machine model 8501) in displacement control (0.05 mm min^{-1}).

Disks (labelled TS) were individually tested under thermal shock conditions using the apparatus described in previous works.[1,13,14] The specimen mounted horizontally on a refractory material with the indented upper surface, was heated in an electric furnace up to a predetermined temperature (T_i) and allowed to equilibrate for 90 min. Then, it was subjected to a sudden cooling using a high velocity air jet at room temperature (T_0 = 26–27 °C) channelled by a silica tube (inner diameter: 3.58 ± 0.02 mm, 90° and 3 mm above the surface) for 20 s. After quenching, the specimen was cooled up to room temperature and examined for crack extension from the original indentation crack. Consecutive thermal shocks increasing the temperature differential between the disk and the air jet ($\Delta T = T_i - T_0$) by 10 °C, were done on each disk until the indentation crack propagation was detected (critical temperature differential = ΔT_C). Values of T_i between 840 and 1140 °C were employed.

2.4. Thermal treatments

Two thermal treatments were carried out on indented specimens: (1) the specimen (labelled T_1) was heated up to 900 °C and soaked at this temperature for 10 h in the thermal shock device (without air jet impingement); (2) the specimen (labelled T_2) was subjected to sudden cooling (in the same experimental conditions of thermal shock test) up to temperatures from 840 to 900 °C; as in thermal shock test, the disk was cooled up to room temperature and the initial temperature was incremented by 10 °C. The absence of macroscopic indentation crack extension from its original length was verified after each cooling up to room temperature. Both sets of specimens (T_1 and T_2) were fractured in biaxial flexure, in the same experimental conditions of BF disks.

The experimental conditions of both treatments were selected considering those ones that could be supported by a disk having the experimental mean ΔT_C value in thermal shock tests.

These two thermal treatments (1 and 2) were performed in order to separate two possible factors, the successive thermal exposures and stresses in the thermal shock tests, considered as significative candidates that determine the crack profile evolution.

2.5. Crack profile

The fracture surfaces produced in the rupture of the specimens BF, TS, T_1 and T_2, were fractographically studied by SEM using a Philips 505 equipment. The procedure given in ASTM-C 1322/96[15] was followed. The geometric parameters of the indentation cracks which act as critical defect,[1] i.e. the length c and the depth a, were determined in order to analyze the profile evolution.

3. Results and discussion

3.1. Mechanical and thermal shock tests

The results of the mechanical and thermal shock tests were reported and analyzed in a previous work[1]. A biaxial flexure strength $\sigma_F = 49.5 \pm 13.8$ MPa and a Weibull modulus $m = 4.23$ were obtained. The value of the critical temperature differential was $\Delta T_C = 970\,°C \pm 91\,°C$. In both the mechanical and the thermal shock tests, the indentation at the disk center acted as the fracture origin and the subsequent crack propagation originated a radial pattern, in agreement with the equibiaxial stress distribution that both loadings produce. Even when the failure by mechanical and thermal stresses produced a low fragmentation of specimens, both the crack paths and the fracture surfaces exhibited several differences among them.[1] The fracture surface of TS samples observed by SEM exhibited a smoother appearance and the major difference with those mechanically broken (BF) was in the unstable propagation of the median-radial cracks. This fact was mainly attributed to the effect of the temperature on the glass phase under thermal shock conditions.[1] The identification of the fracture mode (inter- or intra-granular) in the studied cordierite material was difficult because of the very fine grains developed (~0.5 μm).

3.2. Crack profile evolution

3.2.1. Biaxial flexure (BF) and thermal shock (TS) specimens

The average of the major (c) and minor (a) semiaxes of crack profile (semi-elliptical zone beyond the indentation impression) of specimens BF and TS are reported in Table 1, together with the ratio c/a.

The major axis c for mechanical fracture was statistically the same than that measured on a polished indented surface (c_0)[1] while the mean value under thermal shock conditions was higher (≈36%). In addition, the geometric parameter a together with the ratio c/a were both larger in TS specimens than those of BF. This fact could be attributed to a stable extension of the indentation crack under thermal shock test prior to the unstable propagation, that was absent in biaxial flexure specimens ($c \approx c_0$). The enlargement of the major axis c was higher than that occurred in the minor axis a (48% with respect to 13%). The crack geometry change results evident from the comparison between c/a ratio and SEM images under mechanical and thermal stresses (Fig. 1a and b, respectively). The crack that is initially rather semicircular ($c/a \approx 1$) became semielliptical with the stable propagation ($c/a > 1$).

Table 1
Average major (c) and minor (a) semi-axes of crack profile, and c/a ratio

Specimens	c (μm)	a (μm)	c/a
BF	140 ± 21	135 ± 38	1.0 ± 0.2
TS	209 ± 70	153 ± 27	1.4 ± 0.3
T_1	196 ± 50	148 ± 30	1.3 ± 0.3
T_2	189 ± 65	166 ± 22	1.1 ± 0.4

$c_0 = 152 \pm 17$ μm.

The stable crack propagation under thermal shock conditions will tend to balance the residual stresses around the crack and the crack propagation resistance of the material (K_{IC}).[1,2,16] The residual stress field in the indented cordierite disks is actually modified in thermal shock conditions. This fact is the result of two factors at least: the successive exposures to high temperatures over prolonged periods and the imposed stresses by the air-jet impinging (before T_C). In order to determine if any of above-mentioned factors prevails, the treatments 1 and 2 were performed.

3.2.2. Thermal treatments (1 and 2)

The crack geometric parameters, c, a and c/a ratio are also reported in Table 1 and SEM images are shown in Figs. 2 and 3, respectively.

For both treatments (1 and 2) the crack parameters are like those measured for TS specimens. However, the aspect of the fracture surfaces is in general similar to those of BF disks (Fig. 1a), in particular in the unstable crack propagation region, as would be expected since the fracture is produced in biaxial flexure. The cracks shown stable growth under both thermal treatments since c and a were larger than the values obtained for BF specimens. The geometry also changed

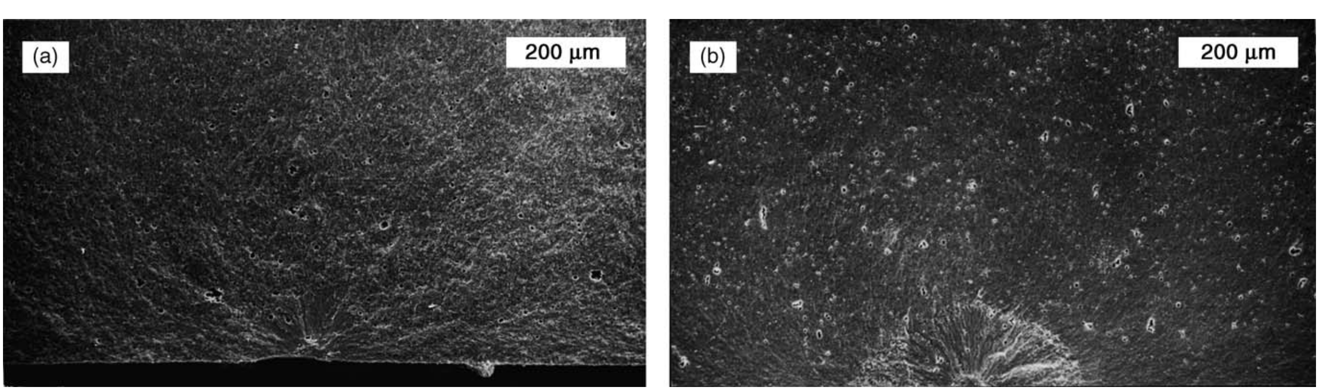


Fig. 1. Fracture surface of indented cordierite disks subjected to biaxial flexure (a) or thermal shock (b) tests. 100 μm scale bars.

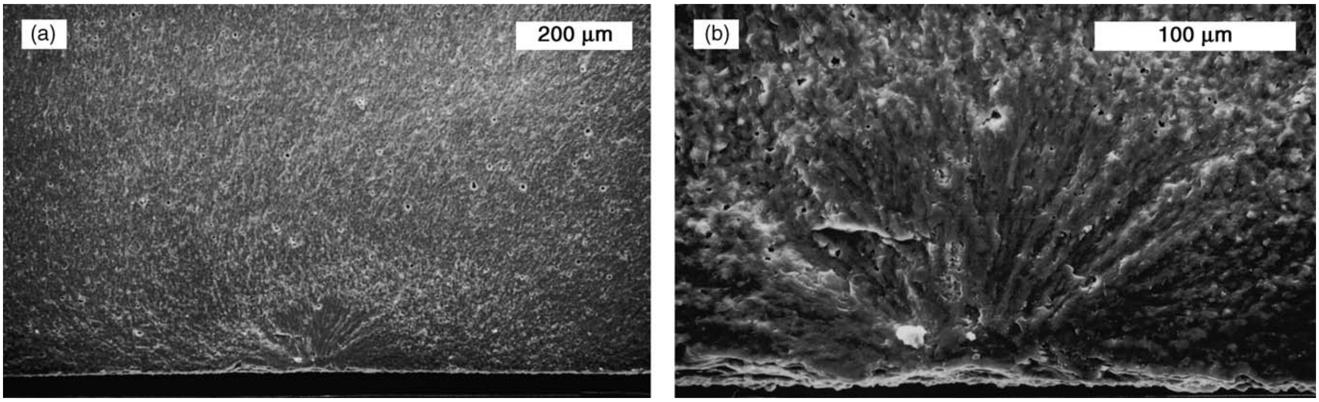


Fig. 2. Fracture surface of indented cordierite disks thermally treated according T_1 thermal treatment and then fractured in biaxial flexure. 100 μm scale bars.

during the stable propagation toward a more elliptical crack which can be inferred from the comparison of *c*/*a* ratio and SEM images with biaxial flexure ones. Similar behaviour was reported in the literature.[2,6,7]

The presence of compressive residual stresses around the median/radial cracks has been reported.[4,17] Also, the superposition of a residual stress field originated during the sintering of the disks cannot be discarded. Both treatments (1 and 2), as much as the thermal shock test (TS), involve the permanence of the specimen at a relative high temperature during a long period of time. So, these treatments could produce an annealing effect: a relieve of the compressive residual field around the indentation. This relaxation would free the constrain at the crack front and lead to change in K_I level. The crack will grow until a new balance with K_{IC} is achieved. These facts account for the stable propagation of indentation crack exhibited in T_1, T_2 and TS disks, that is absent in BF ones since the last specimens were tested at room temperature without previous thermal treatment.

The other factor that could be responsible for the stable crack growth of indentation cracks in the cordierite disks is the imposed thermal stress, in particular due to its temporal evolution.[1,18,19] However, no significant differences were registered in the crack geometric parameters of T_1 with respect to those of T_2, the last also involving the imposition of thermal stresses by the air jet impingement. So, it is considered that the main factor determining the stable propagation is the temperature in successive thermal exposures.

When a surface crack is growing it may experience a varying intensity factor along its front and consequently, the velocity of different points along the front will also be variable. As a result, a change in the crack shape will occur.[7,20] The presence of lateral indentation cracks together with the median/radial system can also alter the residual stress field around the last one and change the crack shape in its stable propagation. Nevertheless, the stable growth of the indentation cracks can occur in absence of a residual stress field as a result of radial/lateral crack interaction. These facts has been studied in transparent material as glasses in which the crack profile evolution is easily followed and a tendency to a more elliptical geometry with higher *c*/*a* ratio was reported.[5,6] A marked change in crack shape increasing *c*/*a* ratio was registered in both thermal treatments and in thermal shock tests, with respect to the original one (exhibited by BF specimens). From the inspection of the indented surface, the presence of lateral cracks was not evident. However, in several fracture surfaces of BF and TS disks cracks running parallel to the indented surface and normal to the median/radial crack plane were observed (Fig. 4a and b). Based on their fractures, these cracks can be considered as the lateral ones. The variation

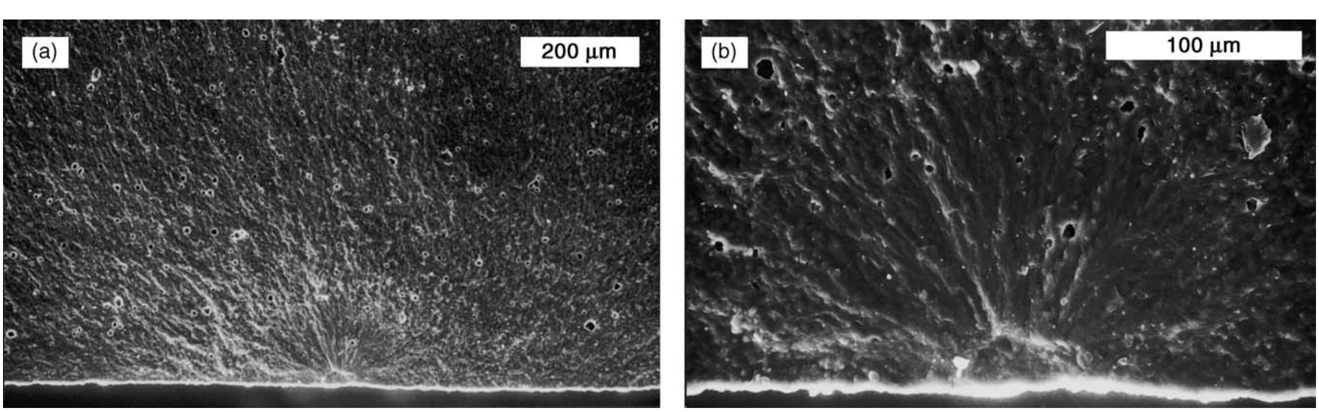
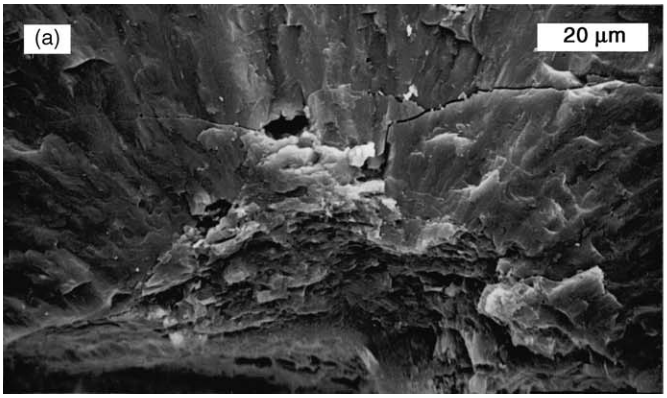


Fig. 3. Fracture surface of indented cordierite disks thermally treated according T_2 thermal treatment and then fractured in biaxial flexure. 100 μm scale bars.



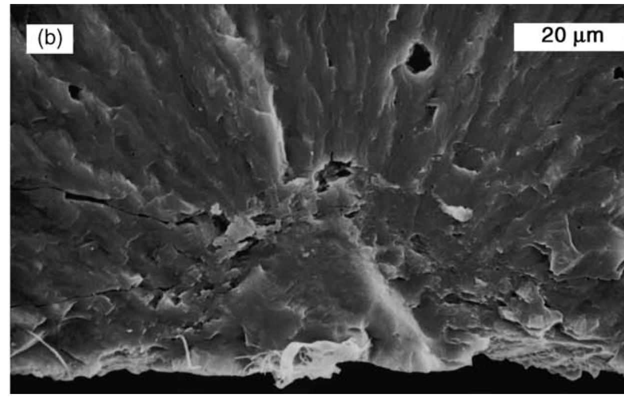


Fig. 4. Fracture surface of indented cordierite disks subjected to biaxial flexure (a) or thermal shock (b) tests, showing cracks running parallel to the surface. 10 μm scale bars.

of the crack shape toward a more elliptical geometry as the median/radial indentation cracks propagate subcritically by thermal effects is attributed to the interaction of these cracks with the lateral ones.

4. Conclusions

The evolution of the indentation crack profiles in a polycrystalline material as cordierite under mechanical and thermal stresses was successfully followed analyzing the fracture surfaces by SEM. In indented specimens without any thermal exposures (BF) only unstable propagation in median/radial cracks was detected. Conversely, in both the disks subjected to thermal shock conditions and broken by thermal stresses (TS) and those treated at high temperatures without and with sudden cooling and fractured in biaxial flexure (T_1 and T_2), stable crack propagation was also observed arising from the modification in the residual stress field around the indentation. The successive exposures at high temperatures were considered the main factor responsible of the stable propagation. The change in the crack shape that accompanies the stable crack growth was attributed to the presence of lateral cracks.

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