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Crack Propagation Behaviour in Mullite at High Temperatures by Double-Torsion Technique

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Abstract

The double-torsion technique has been used to study subcritical crack growth in mullite at room temperature (RT) and above. The crack velocity was evaluated by measuring a change in crack length during a prescribed time under a constant applied load. Threshold values of stress intensity factor, below which no propagation occurs, of 1·5 and 1·6 MPa $m^{1/2}$ were found at RT and high temperature, respectively. Considering a power law, measured stress exponents of 24, 35, 18 and 10 at RT, 1100, 1200 and 1300°C were obtained. A change of crack growth mechanisms is observed around 1200°C, with cleavage crack growth occurring below 1200°C and viscous intergranular crack growth dominating at higher temperatures. © 1996 Elsevier Science Limited.

1 Introduction

Recently, dense polycrystalline mullite with a little or nearly no glassy phase has become available by sintering of fine mullite powders.[1] This material shows the advantages of a relatively high flexural strength at high temperature and high creep resistance in addition to its low thermal expansion.[2,3] These characteristics makes mullite a promising material for high-temperature structural use. However, the crack growth behaviour of this material has not been extensively investigated.

Most mechanisms of slow crack growth (SCG) in ceramics, such as the environmentally controlled stress corrosion process, were examined at room temperature (RT) and only sparse SCG data are available at high temperatures.[4] Hot-pressed silicon nitride (HPSN), however, is an example of well-documented material that has largely been investigated at elevated temperatures.[5,6] By considering the power relation: $V = BK_I^m$, Evans and Wiederhorn[5] found that crack growth at low velocities is characterized by a constant slope $m = 10$ and that crack velocity increases with temperature. An activation energy value of about 920 kJ mol^{-1} was found. The mechanism of crack growth in this region was attributed to viscous flow. More recently, Lange[7] and Kingery *et al.*[8] investigated the crack growth mechanisms of HPSN in an inert atmosphere. They suggested that SCG in polycrystalline ceramics at high temperatures depends principally on grain boundary phase as well as on environment. On the other hand, Knight and Page[9] have studied SCG of porous ceramics such as CaO-stabilized ZrO_2 using the double-torsion technique at temperatures in the range 1010–1400°C. They observed three distinct propagation stages. At low K_I the behaviour can be described by $V = BK_I^m$, with m decreasing with increasing temperature. In the second stage the velocity is constant with increasing K_I, and finally for K_I near K_{IC}, V increases rapidly. The dominant mechanism of SCG appeared to be interparticle sliding (leading to pore rearrangement, pore linking and crack extension).

In this paper the results of crack propagation tests conducted on a mullite ceramic at several temperatures using a double-torsion technique are presented. The crack velocity has been measured as a function of the applied stress intensity factor and the results are discussed in terms of crack growth mechanisms.

2 Experimental Procedure

Several methods can be applied to measure the crack velocity via the double-torsion specimen.

The load relaxation method is the most frequently used because it is relatively rapid (one specimen is sufficient in principle). When associated with calibration of the compliance with crack length it presents the advantage of (at least theoretically) permitting a record of the full V–K_I curve with a single specimen, even in a closed furnace. But this method is sensitive to any temperature variation and it is therefore restricted to relatively high velocities ($>10^{-7}$ m s^{-1}). In addition, it cannot be applied when there is some plasticity. For these reasons the constant load method, which is very simple, less sensitive to temperature variation and therefore relevant for lower velocities, has been selected here. The main drawback is that the procedure for recording a full V–K_I diagram is time-consuming since one test gives only one point in the V–K_I diagram.

2.1 Material

The mullite used in this work was made by ITMA (Spain), from a Baikowski powder (CA 193 CR, France). Green compacts of mullite obtained by cold-isostatic pressing under 200 MPa were sintered for 2 h at 1750°C. A post heat treatment at 1450°C for 5 h was subsequently carried out to reduce the glassy phase content.

The density of the material measured by Archimedes' method is about 95·5%. The microstructure analysed by scanning electron microscopy (SEM) on polished and thermally etched surfaces (Fig. 1) revealed two distributions of grain sizes: equiaxed grains with size of 1–4 μm and elongated grains with long axis lengths of about 7 μm. Several pores at multiple grain junctions can also be observed. Transmission electron micrographs revealed the presence of glassy phase at three-grain junctions but since high resolution observations were not conducted, no intergranular glassy film has been observed.

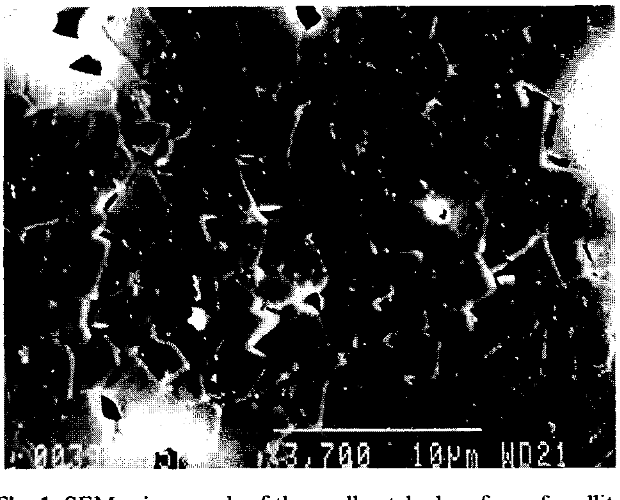


Fig. 1. SEM micrograph of thermally etched surface of mullite.

2.2 Preparation of specimens

The specimen dimensions were $20 \times 40 \times 2$ mm^3. A 10 mm long notch was sawn in the specimen after grinding, polishing and annealing. A pre-crack was initiated at RT from the notch tip by loading the specimen at very low speed, in order to obtain a sharp crack. It is important to note that no groove has been used to guide the crack.

A constant applied load on a load-controlled testing machine (Schenck Treble, RM-T 25) was maintained for prescribed times and the crack velocity $V = \Delta a/\Delta t$ was calculated by measuring the change in crack length by optical microscopy after each test. Crack growth measurements were conducted at RT, 1100, 1200 and 1300°C. The crack length was measured by optical microscopy and the resolution Δa^{min} was estimated as 10 μm. For relatively high crack velocities ($>10^{-7}$ m s^{-1}), the crack increments were in the range of about 100 μm to 1 mm, and the minimum prescribed times were a few minutes. The maximum error on the crack velocity measurement was therefore about 10%. For lower crack growth, the error might be slightly higher. For representation of the threshold, when no crack advance was observed, an upper velocity limit was calculated as $\Delta a^{min}/\Delta t$.

2.3 Analysis method

For this double-torsion geometry, determination of the stress intensity factor is based on the fact that the loading point displacement is proportional to the crack length and the applied load P:[5,10]

$$K_I = P\left[\frac{W_m}{e^2}\right]\sqrt{\frac{3(1+\nu)}{\psi(e/W)W}} \qquad (1)$$

where W is the width of the specimen, W_m is the moment arm and $\psi(e/W)$ is a calibrating factor.[11]

From eqn (1) it can be noted that the stress intensity factor K_I for the double-torsion configuration depends only on the applied load and not on the crack length.

3 Results and Discussion

3.1 Room temperature

A typical V–K_I diagram for mullite obtained at RT is shown in Fig. 2. Only the first propagation stage is observed in the crack velocity range investigated. It should be noted that this stage has been shown to be the most important for lifetime prediction.[12] In this region the crack velocity ranged from 10^{-11} to 10^{-5} m s^{-1} and the stress intensity factor K_I ranged from 1·5 to 2·3 MPa m$^{1/2}$.

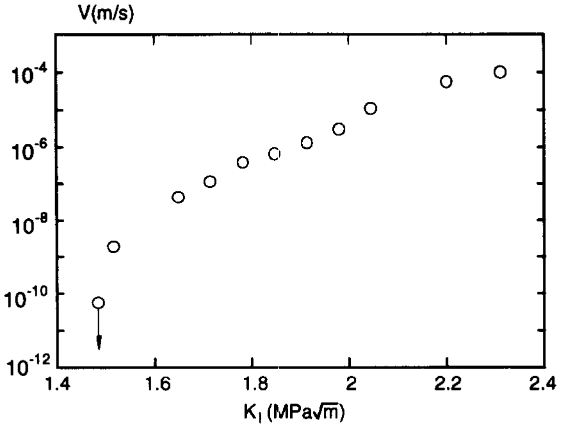


Fig. 2. V–K_I diagram for mullite at room temperature.

This diagram clearly shows a threshold stress intensity factor K_{IO} below which no crack propagation was observed. This is very important and suggests that, below this value, specimens of mullite should present unlimited lifetime at room temperature.

On the other hand, the V–K_I curve may be fitted by a power law: $V = BK_I^m$. The values of B, m and K_{IO} are given in Table 1.

3.2 High temperatures

Figure 3 shows the different measurements obtained at 1100°C represented on a single diagram. The procedure was such that several crack propagations were conducted on the same specimen (represented by the same symbol in Fig. 3). It can be observed that for most of the fourth, fifth and sixth tests on the same specimen (see Fig. 3), the crack length does not change after testing even when higher stress intensity factors are applied. This phenomenon has been attributed to crack healing, which increases the resistance to crack growth. This behaviour was also observed by Kubota *et al.*[13] in their study on the fatigue behaviour of mullite at 1200°C. They found that crack growth under an applied stress was interrupted by crack healing. In order to avoid this problem, a precrack at room temperature was made before each test at 1200 and 1300°C.

The different V–K_I curves measured at high temperatures are shown in Fig. 4. They exhibit similar characteristics to that obtained at RT with

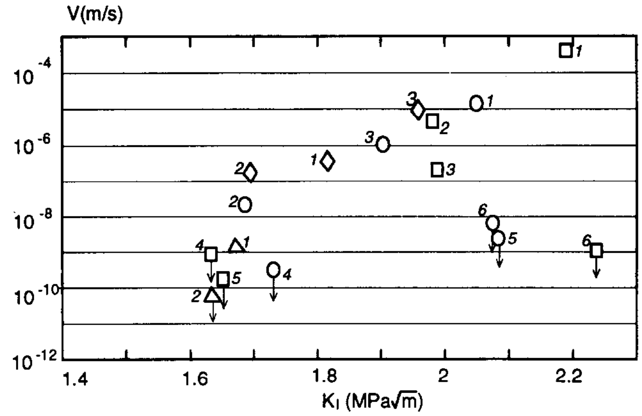


Fig. 3. V–K_I diagram for mullite at 1100°C. The different symbols represent different specimens. The number near the symbol represents the *i*th test conducted on a given specimen without an RT precrack. Arrows represent upper crack velocity limit when no crack advance was observed.

a threshold stress intensity factor shifted to higher values of K_I. It should be noted that the threshold values are the same for the different temperatures investigated. However, for 1200 and 1300°C one can observe the presence of a second region where the crack velocity increases very weakly with increasing K_I until K_{IC}, where fast failure occurred. Table 1 gives the SCG parameters for the different temperatures.

One can note that the SCG exponent m increases from 23·6 to 34·7 when the temperature increases from RT to 1100°C. However, the opposite tendency is observed for temperatures above 1100°C. Effectively, the exponent m falls from 34·7 to 18 at 1100 and 1200°C, respectively. This change in SCG behaviour suggests a change in the mechanism of crack growth around 1200°C. Fields and Fuller[14] have proposed a representation of crack velocity derived from 'creep maps', where constant velocity curves are plotted on a stress intensity factor–temperature diagram. This presents the advantage of outlining the propagation mechanisms. The results have been represented on such a plot where the toughness has also been included (Fig. 5). In the present case, it shows clearly that for a constant value of K_I the increase in temperature between 1100 and 1200°C induces a decrease of crack velocity, whilst above 1200°C the crack velocity increases with increasing temperature. This can be understood by analysing the

Table 1. SCG parameters at different temperatures

Temperature (°C)	K_{IO} *(MPa m$^{1/2}$)*	m_1	B_1	m_2	B_2
RT	1·51	23·6	$3{\cdot}4 \times 10^{-13}$		
1100	1·65	34·7	$1{\cdot}7 \times 10^{-16}$		
1200	1·66	18	$8{\cdot}6 \times 10^{-11}$	2·1	$7{\cdot}6 \times 10^{-7}$
1300	1·64	10·1	$5{\cdot}5 \times 10^{-9}$	2·2	$8{\cdot}8 \times 10^{-8}$

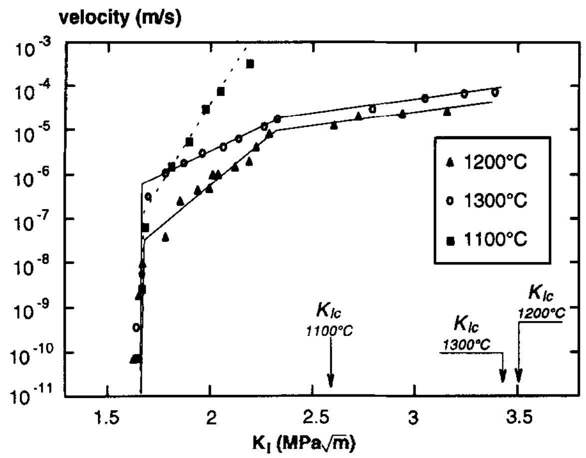


Fig. 4. V–K_I diagram for mullite at different temperatures.

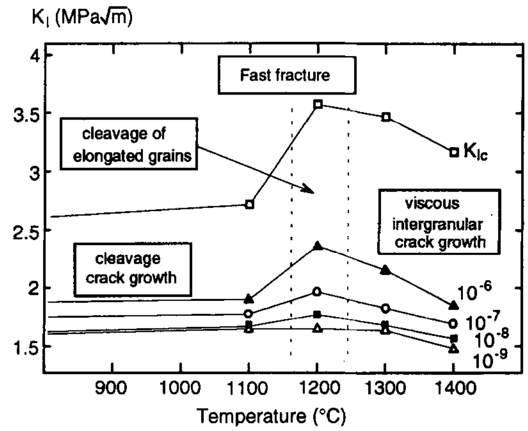
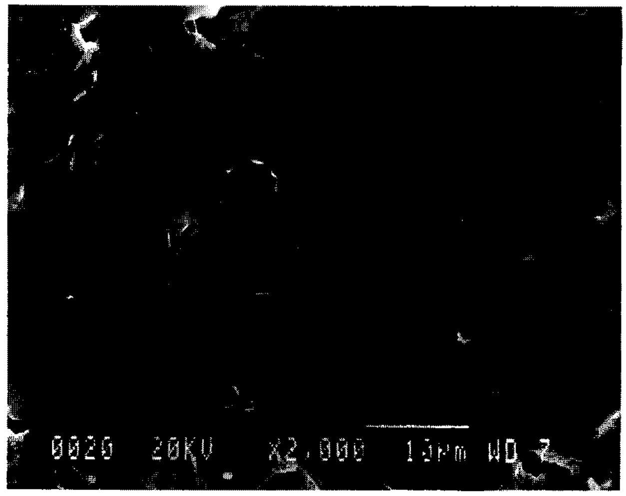


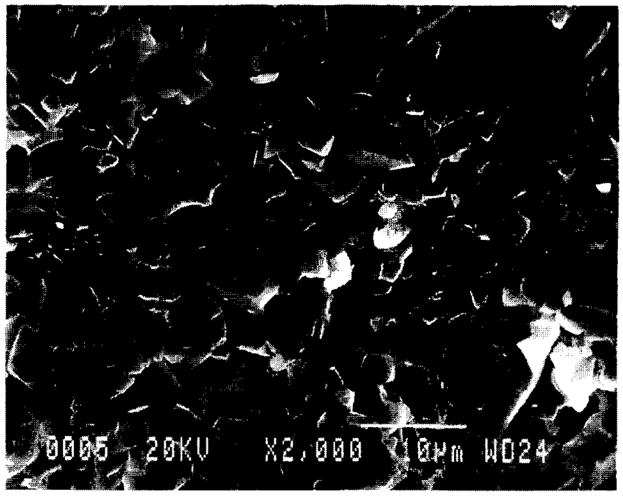
Fig. 5. K_I–T diagram for mullite at different crack velocities.

different fracture surfaces by SEM, as shown in Fig. 6. One can clearly note a change in SCG mechanisms; at 1100°C [Fig. 6(a)] only cleavage crack growth can be observed. At 1200°C, Fig. 6(b) shows two mechanisms of crack growth: cleavage of the elongated grains and intergranular mode near the equiaxed grains. On the other hand, at 1300°C we observe slow crack growth regions with a distinct appearance, characterized by bright regions. A detailed analysis of the SCG region shows that the nature and mode of fracture are completely intergranular [Fig. 6(c)]. This is attributed to the decrease of the intergranular glassy phase viscosity at high temperature. The propagation therefore arises by viscous intergranular crack growth.

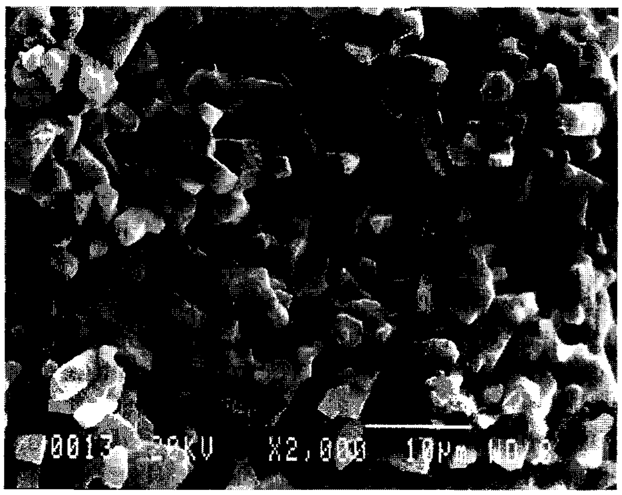
These double-torsion results measured on macrocracks have been compared with data obtained under static fatigue of bend bars without artificial flaws conducted on the same material, at 1200 and 1300°C (experimental details are given in Ref. 15). The results in terms of lifetime as a function of the applied stress are represented in Fig. 7. Some simulations of those static fatigue experiments



(a)



(b)



(c)

Fig. 6. SEM micrographs of the fracture surface of a double-torsion specimen tested at (a) 1100°C, (b) 1200°C and (c) 1300°C.

have been conducted by integration of the double-torsion V–K_I laws (taking into account the different stages). The results (Fig. 7) show that a fair agreement is obtained between the experiments

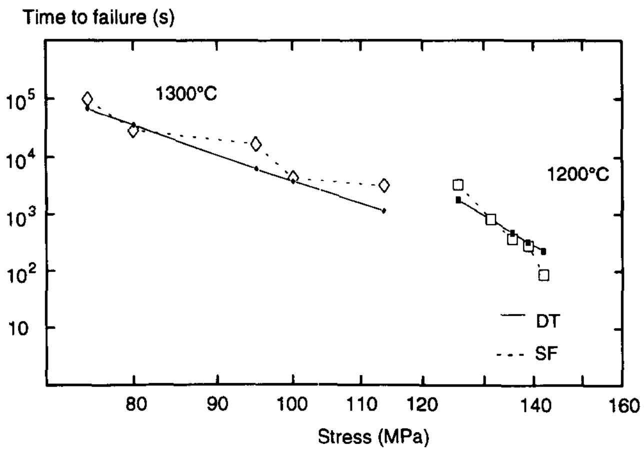


Fig. 7. Time to failure obtained by creep experiments and compared with the simulation.

and the simulation, suggesting that the double-torsion method allows crack growth behaviour to be predicted correctly.

4 Conclusion

The behaviour of slow crack propagation in mullite ceramic was investigated at room temperature, 1100, 1200 and 1300°C, using a double-torsion technique at constant load. A threshold value of stress intensity factor, below which no propagation occurs, was observed for all temperatures: it was about 1·5 and 1·6 MPa $m^{1/2}$ at room temperature and 1100°C respectively, and was approximately constant at higher temperatures. Considering a power law, the measured stress exponents of 24, 35, 18 and 10 at RT, 1100, 1200 and 1300°C were obtained. A change of crack growth mechanisms is observed around 1200°C: cleavage crack growth below 1200°C and viscous intergranular crack growth at higher temperatures.

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