POINT CONTACT SURFACE FATIGUE IN SINGLE CRYSTAL AND POLYCRYSTALLINE MAGNESIUM OXIDE

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ABSTRACT

The effects of repeated point contact loading on the structural integrity of single crystal and polycrystalline magnesium oxide, MgO, have been investigated. Tests were conducted in air at room temperature using the soft impressor method (Guillou *et al.*, 1993, 1995; Brookes *et al.*, 1994; Henshall *et al.*, 1995; Maerky *et al.*, 1996). It was observed in the single crystal tests, on the (001) plane, that dislocations are initially generated and move on the $\{110\}_{45}$ slip planes. A relatively high density of these dislocations was achieved quite rapidly. This was followed by a more gradual, and at more variable rates, build up of dislocations on the $\{110\}_{90}$ slip planes. Subsequently, cracks were initiated at the edge of the contact zone between the flattened impressor and the substrate; these quickly propagated around the perimeter and then radially outwards. The polycrystalline MgO, which had approximately 4% porosity and 3% Y_2O_3 sintering aid, was considerably more resistant to the fatigue process. In the polycrystalline material, between 100 and 1000 times the number of cycles were required to initiate a crack, and the rate of crack extension was also much slower, than in the single crystal.

KEYWORDS

Surface fatigue, point contact, ceramic fatigue, MgO.

INTRODUCTION

Single crystal MgO has been the subject of much scientific study (*e.g.* Keh, 1960; Moriyoshi and Ikegami, 1984; Roberts, 1988) primarily because it is readily available, has a cubic structure, etch pits well, and is not too hard. However, the vast majority of the work has been performed under monotonic loading or indentation hardness test conditions, with relatively little attention paid to its fatigue behaviour (Brookes *et al.*, 1976, 1994; Guillou *et al.*, 1993).

Polycrystalline MgO is used primarily in refractory applications due to its high melting point and good resistance to attack by metals, fluxes and superconductor compounds. However, its hardness is relatively low for an engineering ceramic, ~ 6 GPa, which has probably limited its usage in load bearing applications. This degree of hardness should enable polycrystalline MgO to be fabricated without the difficulties associated with other ceramics, such as alumina and silicon nitride, whilst still providing reasonable service performance under some wear conditions.

The soft impressor technique (Guillou *et al.*, 1993, 1995; Brookes *et al.*, 1994; Henshall *et al.*, 1995; Maerky *et al.*, 1996) has been developed as a means of assessing the surface fatigue behaviour of ceramics. It involves pressing a softer, usually metallic, cone against a harder flat counterface, in this case MgO. The primary advantages of using this type of surface fatigue test are that (i) the plastic deformation of the metallic cone during the initial loading cycle results in good alignment of the two contacting surfaces and ensures a uniform pressure distribution, (ii) the softer impressor does not produce immediate gross plastic deformation of the ceramic unlike a harder indenter, and is thus more relevant to operational conditions, and (iii) the stress state has been characterised (unlike conventional indentation hardness testing).

EXPERIMENTAL PROCEDURES

The single crystal MgO, supplied by Spicer Ltd (London, UK), was one from a large batch which have been used for several previous studies of indentation and scratch hardness. The (001) surface was prepared by cleaving and chemically polishing for ~ 2 min in orthophosphoric acid at 110 °C, immediately prior to usage. The commercial, opaque, polycrystalline MgO (Aremco Super-heat 676) contained 3% yttrium oxide sintering additive, and had approximately 4% porosity. Figure 1 is a micrograph of the polished and etched surface showing the grain boundaries pinned by the porosity, and discrete islands of Y_2O_3 at triple points. The mean linear intercept grain size was 24 μm and the Knoop hardness (9.8 N applied load), H_K, was 5.4 GPa. With Vickers indentations, the cracking was predominantly transgranular, as previously observed by Cook and Liniger (1992).

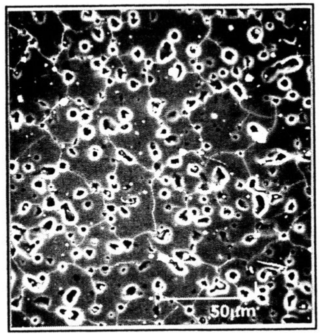


Fig. 1 Polished and etched surface of polycrystalline MgO.

The metallic impressors were initially machined from 12 mm diameter stainless steel bar, with the conical tips being ground to a sharp 120° apical angle. The original Knoop hardness (19.6 N applied load) of the impressor material was 3.8 GPa. The flattened tips after testing had a Knoop hardness of 5.2 GPa (0.25 N applied load).

The repeated point contact loading tests were performed on a purpose-built machine, shown schematically in Fig. 2. The extension of the spring was used to produce the applied loads, which were continuously monitored via a load cell connected to a PC. The same loading cycle was used in all cases, *i.e.* a sinusoidally varying waveform of magnitude 10.3 ± 9.3 N and frequency approximately 2 Hz. The softer conical impressor was flattened to a conforming circular cross-section on first application of the maximum load, and remained in contact with the ceramic substrate for the entire duration of the test. This test configuration was used to ensure a uniform pressure distribution across the contact zone. The resultant applied pressures were 1.05 ± 0.95 GPa. A new cone was used for each test. All tests were conducted at room temperature in air. The single crystal data were obtained under non-lubricated conditions, whereas the polycrystalline MgO was tested either unlubricated or with a grease ('Apiezon' vacuum grease) between the two surfaces, up to a maximum of 10^6 cycles.

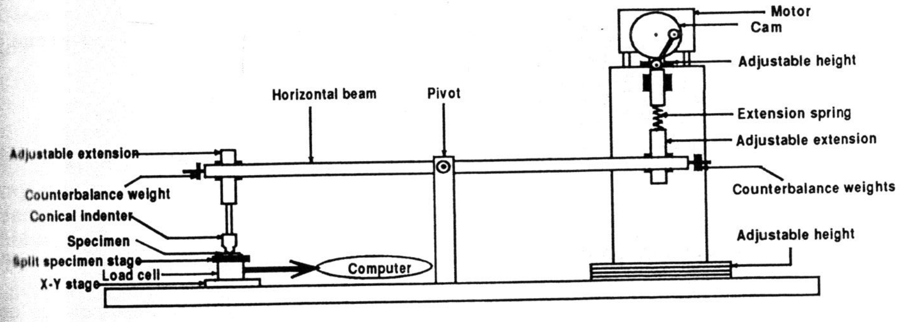


Fig. 2 A schematic illustration of the cyclic fatigue apparatus.

RESULTS AND DISCUSSION

Figures 3(a)-(d) are SEM micrographs of the tested (001) single crystal MgO surface, which has been etch pitted using the standard mixture (four parts of saturated ammonium chloride, one part of concentrated sulphuric acid and one part of distilled water), after testing between 10, Fig. 3(a), and 5000, Fig. 3(d), cycles. It is quite clear that considerable dislocation generation took place under these conditions and substantial cracking was produced after a relatively low number of cycles. The heavily dislocated central region, ~ 110 μm in diameter, developed within the contact area between the flattened impressor and the ceramic counterface. Over this fatigue range, the diameters of the flattened tips did not measurably change during a test.

The dislocation activity was initially primarily confined to the four oblique $\{110\}_{45}$ planes, which was followed by a more gradual increase in dislocation density on the two $\{110\}_{90}$ planes. In a sense this is the reverse of the behaviour in diamond pyramid indentations, whence the $\{110\}_{90}$ dislocations are formed initially during the formation of the impression, and the majority of the $\{110\}_{45}$ slip occurs on removal of the indenter. In the present tests, the maximum extent of dislocation activity, i.e. the radius of the 'plastic zone', did not appear to increase with increasing number of cycles. The traces of the $\{110\}_{45}$ slip lines were symmetrical about the centre line of the contact zone, whereas the $\{110\}_{90}$ slip traces were not. This is illustrated quite clearly in Fig. 3(b), where at least two or three $\{110\}_{90}$ slip traces were well developed on one side of the contact area, but there was virtually no corresponding trace on the opposite side. This would indicate that the structures of the dislocation loops were not the same. By analogy with the terminology of indentation cracks, from a consideration of the surface geometries, it would appear that the $\{110\}_{45}$ loops are 'median/half-penny' shaped and the $\{110\}_{90}$ loops are 'Palmqvist' type.

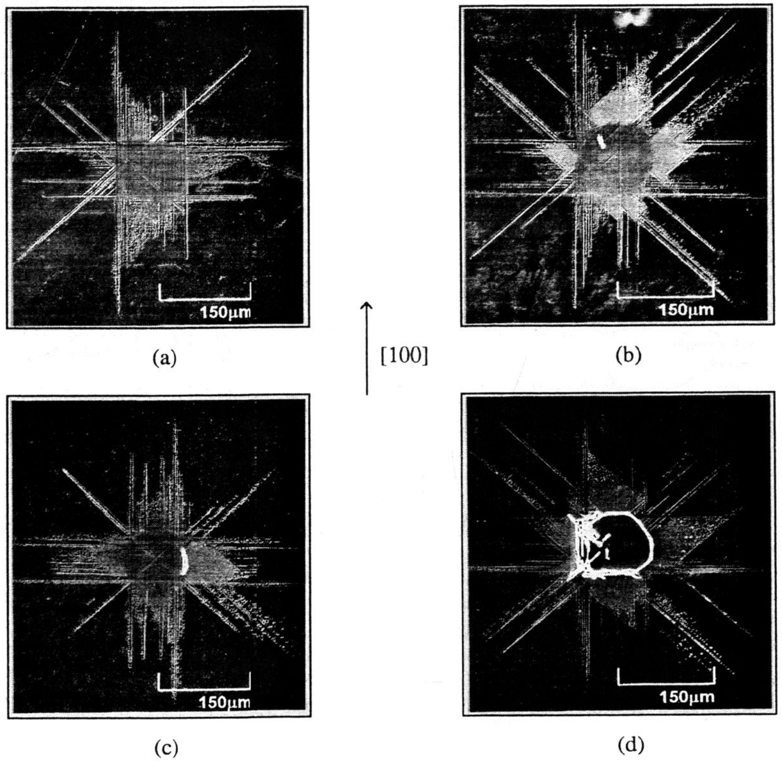


Fig. 3 SEM micrographs of the evolution of the fatigue induced dislocation activity and cracking in (001) single crystal MgO after (a) 10 cycles, showing primarily $\{110\}_{45}$ slip, (b) 100 cycles, where significant $\{110\}_{90}$ slip has also occurred, (c) 2000 cycles, with crack initiation occurring at the edge of the contact zone and (d) 5000 cycles, where the crack has propagated around the contact zone periphery and also radially along <110>.

Cracks are initiated at the edge of the contact zone after approximately 2000 cycles. These are crystallographic in nature, *cf.* Fig. 3(c). These rapidly propagate completely around the edge of the contact zone, followed by multiple cracking and extension in the radial directions. The cracks remain predominantly crystallographically oriented, as can be seen in Fig. 3(d). The majority of the cracking occurred on the $\{110\}$ planes, as would be anticipated by analogy with similar crystallographic diamond pyramid indentation induced fracture. The basis for the formation of these cracks was dislocation interactions of the form first described by Keh *et al.* (1959) in MgO, *e.g.* $a/2\,[0\bar{1}1]_{(011)} + a/2\,[10\bar{1}]_{(101)} \rightarrow a/2\,[1\bar{1}0]_{(112)}$.

The fatigue tests on the polycrystalline MgO were initially performed under non-lubricated conditions up to a maximum of 10^6 cycles. The main features observed after testing, as shown in Fig. 4(a), were : the formation of a very slight degree of plastic 'sinking-in' in the contact zone, metal-to-ceramic material transfer (which increased with increasing number of cycles), and some limited cracking at the edge of the impression. The main mechanism contributing to the transfer of metal was the stainless steel being pushed into the surface holes arising from the pre-existing porosity in the MgO, *cf.* Fig. 4(b). The surface cracking that was observed was associated with these 'in-filled' pores, and presumably arose from the extra fatigue loading imposed within the pore by the included metal.

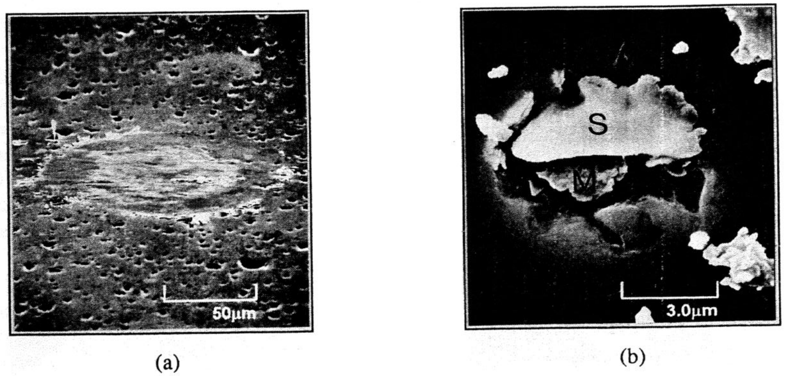
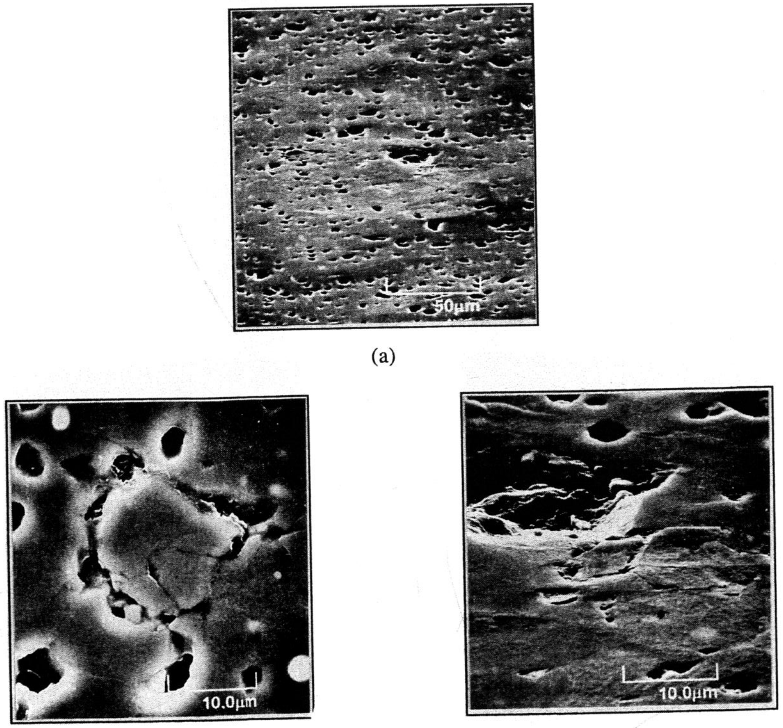


Fig. 4 Impression formed in fatigue tested polycrystalline MgO (10^6 cycles, no lubrication), (a) entire contact zone (70° tilt), and (b) pre-existing pore in MgO which has been partially in-filled by stainless steel (S), also showing fracture in the MgO (M).

It was found that if 'Apiezon' vacuum grease was used between the impressor and the flat specimen surface, then this effectively inhibited metal-to-ceramic transfer for otherwise identical test conditions, as shown in Fig. 5(a). A shallow impression, diameter ~ 110 µm, with a slight ridge at the edge of the contact zone was formed as a result of the cyclic loading. The delineation of the impression became clearer with increasing number of cycles in the range 10^3-10^6 cycles. It was not possible to etch pit this polycrystalline MgO to reveal the extent of dislocation activity. However, in some of the grains towards the edge of the contact zone, there were indications of surface steps arising from localised plastic deformation, presumably as a result of slip, Fig. 5(c). There was also a limited amount of surface cracking and grain spalling present at the edge of the contact zone, which became apparent only after 10^6 cycles, Fig 5(b)-(c). The cracking was predominantly intergranular, with cracks linking the pre-existing pores, although transgranular cracks were also observed, *cf.* Fig. 5(b).



(a)

(b) (c)

Fig. 5 Impression formed in fatigue tested polycrystalline MgO (10^6 cycles, grease lubricated), (a) entire contact zone (70° tilt), (b) surface cracking at the edge of the contact zone, and (c) deformation and also grain spalling.

The nature of the deformation and fracture observed in these tests appeared to be consistent with cyclically induced damage. However, to confirm this, static fatigue tests were performed on a polished polycrystalline MgO surface loaded by a flattened stainless steel cone with a 39.2 N load, giving a contact pressure of 2 GPa, for a period equivalent to 10^6 cycles, *i.e.* approximately 6 days. A slight 'sinking-in' of the substrate beneath the contact zone occurred, but no other deformation or cracking was observable. An unlubricated test showed no significant adhesion between the cone and the MgO. These results provide confirmation that

cyclic loading is required to induce the 'damage' observed in the MgO in the 'soft' impressor fatigue tests.

The substantial difference in the rate at which fatigue effects are observed in the single crystal and polycrystalline materials is surprising. The single crystal MgO exhibited greater deformation and fracture after 5×10^3 cycles than the polycrystalline material after 10^6 cycles. The diameters of the contact zones were all ~ 110 μm, which encompassed sufficient grains in the polycrystalline MgO to ensure that the apparent marked reduction in dislocation activity could not be simply due to an unfavourably oriented grain or grains. The maximum tensile stresses occur at the edges of the contact zones. Given the limited extent of the plasticity that occurred, it is not unreasonable to assume that the stresses here will be close to those given by elasticity theory, at least in the initial stages. The maximum tensile stresses at the edge of the contact zones are thus given by (Johnson, 1985) :

$$\sigma_m = (1-2\nu)P_m / 2\pi R^2$$
$$\Delta\sigma = (1-2\nu)\Delta P / 2\pi R^2$$

where σ_m is the mean of this maximum tensile stress, $\Delta\sigma$ is the corresponding cyclic stress amplitude, ν is Poisson's ratio (= 0.25), P_m and ΔP are the mean load and fluctuating load amplitude respectively, and R is the radius of the contact zone. Thus, for these test conditions the values of σ_m and $\Delta\sigma$ are approximately 260 and 240 MPa respectively. The local values of these stresses in the polycrystalline MgO will be increased as a result of the extra stress concentration around the pores which straddle the edge of the contact zone. Notwithstanding this additional stress increase, the polycrystalline MgO is more resistant to crack initiation. It would be expected that not all grains at the edge of the contact zone would be favourably oriented to produce slip on two intersecting slip bands as required to initiate cracks by the Keh mechanism. Nevertheless, given the symmetry of the cubic system, at least a small number of grains at the contact zone periphery should be favourably oriented, and thus this would result in {110} plane dislocation initiated cracking. However this is clearly not the case, *cf.* Figs. 4 and 5. Therefore it can be concluded that the initiation of cracks via the Keh mechanism was inhibited in the polycrystalline MgO.

The present work clearly demonstrated that under point contact surface fatigue conditions, polycrystalline MgO is more resistant to both crack initiation and propagation than (001) single crystal magnesia. This contrasts with the supposition of Cook and Liniger (1992), who assumed that for fracture under diamond pyramid indentation test conditions in single and polycrystalline MgO, the initiation phase is the same, but the propagation phase only is affected by the presence, and size, of the grains. An additional factor is that the polycrystalline MgO used by Cook and Liniger had slightly less sintering aid (2% vs 3% Y_2O_3) and also less porosity (2.5% vs 4%), than that used in this study. Further work is clearly required to determine if crack initiation in polycrystalline MgO under effectively monotonic loading conditions is more difficult than in single crystals, as it is in the present series of cyclic tests, and also the effects of grain size, impurity level and degree of porosity.

SUMMARY

This study provides additional experimental support for the observation that a loaded contact between a softer material, in this case metallic cone, and a harder material, *i.e.* flat polished ceramic surface, can produce plastic deformation in the harder body.

Cyclically loading a (001) surface of single crystal MgO under uniform applied pressures of 1.05 ± 0.95 GPa initially produced dislocation activity on the four $\{110\}_{45}$ slip planes, followed by a more gradual build up of slip on the $\{110\}_{90}$ planes. The $\{110\}_{45}$ dislocation loops appeared to be symmetrical about the appropriate centre line of the contact zone. In contrast, the $\{110\}_{90}$ loops appeared to be asymmetrical, having one end within the contact zone, with the other end lying outside the contact zone.

Fracture in the single crystal (001) MgO occurred initially at the edge of the contact zone after approximately 2000 cycles, and was predominantly crystallographic in form. The cracks were most likely to have been initiated as a result of dislocation interactions, akin to the Keh mechanism.

Polycrystalline MgO exhibited a much greater resistance to fatigue induced deformation and fracture than single crystal material. It proved necessary to use an interfacial grease film between the flattened cone and the polycrystalline MgO to prevent pressing of the stainless steel into the surface pores. The polycrystalline material proved not only more resistant to crack propagation than the single crystal, but was also substantially more resistant to crack initiation.

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