Influence of surface preparation on the rotating flexural fatigue of Mg-PSZ

V. ZELIZKO, M. V. SWAIN
CSIRO Division of Materials Science and Technology, Locked Bag 33, Clayton, Victoria 3168, Australia

Specimens of Mg-PSZ were given three different surface preparations prior to testing in rotating flexural fatigue. The preparation consisted of precise grinding with relatively coarse (55 to 80 μm) diamond-impregnated wheels along axial and radial directions as well as by polishing. Only minor differences were noted in the strength–number of cycles to failure (S–N) data, with radial grinding being more deleterious. The data showed considerable scatter; however, from the results the stress corrosion exponents were typically between 65 and 92. Failure origins were often associated with the presence of flaws or pores near the surface.

1. Introduction

Magnesia-partially-stabilized-zirconia (Mg-PSZ) is one material in the growing list of transformation-toughened ceramics [1]. These materials exhibit high strengths and toughness because of a stress-induced phase transformation, and are beginning to attract considerable attention for a wide range of engineering applications [2]. The toughness of these materials is strongly temperature-sensitive and generally limited to applications below 800° C [3]. One of the most significant properties of these materials is their relative insensitivity to damage [4]. This arises partly because of the intrinsic high fracture toughness, making flaws difficult to initiate, and also because contact damage invariably initiates the volume-expanding phase transformation introducing a surrounding compressive stress state at the surface. Thus the damage introduced by grinding or grit-blasting of PSZ materials may enhance the flexural strength, whereas for most ceramic materials such damage significantly decreases the strength. In this note the influence of grinding and polishing of Mg-PSZ samples on rotating flexural fatigue behaviour is considered.

The rotating flexural fatigue method for evaluating the behaviour of metallic materials is well documented [5]. The simplicity of this technique, and its ready acceptance by the engineering design fraternity, make it an attractive test in comparison with other means for testing the fatigue sensitivity of materials, such as plane bending or push–pull compression–tension machines. The problems of edge effects in the former plus alignment and gripping of ceramic materials are severe handicaps for the latter tests. However, the application of the rotating flexural technique to ceramics is rather limited. Recently Ko [6, 7] has evaluated the fatigue behaviour of alumina and silicon nitride using this technique. In both materials, strength against number of cycles to failure (S–N curve) plots were made, with the hint of a fatigue limit in the region of 10^7 to 10^8 cycles. As discussed elsewhere [8], the stress corrosion exponent n may be calculated from the reciprocal of the slope of a log–log plot of the S–N data. Most studies, including those of Ko and a previous study of alumina [9], find that the stress corrosion exponent from such rotating flexural fatigue tests is significantly less than direct measurements from crack velocity tests.

2. Specimen preparation

The material evaluated in the present tests was an Mg-PSZ heat-treated by the manufacturer (Nilcra Ceramics, Melbourne) to the maximum-strength (MS) condition. This material was prepared by conventional pressureless sintering and contained tetragonal precipitates ($\sim 0.2\,\mu$m in length) distributed in a coarse-grained (50 μm) ceramic. Further details of the microstructure are presented elsewhere [10]. The flexural strength measured in four-point flexure testing was 715 ± 30 MPa for ground specimens.

The specimen geometry is shown schematically in Fig. 1. Specimen preparation was a two-step exercise. Initially the specimen was ground to a cylindrical rod, followed by grinding the waisted mid-section using specially prepared diamond-plated wheels. Two such wheels were prepared, one with a 25.5 mm radius to enable axial grinding and the other with a 150 mm radius and the geometry of the outer surface to provide an equivalent 25.5 mm radius for radial grinding. The grinding wheels were plated with 55 to 80 μm diamonds (Diamond Plating Co., Mentone, Victoria). The surface grinding speeds were 31 and 22 m sec^{-1} for axial and radial grinding conditions, respectively. In addition the ground specimens were polished with diamond-impregnated fabrics and pastes. This involved the removal of 30 to 40 μm with the fabric containing 30 to 40 μm diamonds (Dicofab, Kristalap Ltd, UK) followed by subsequent removal of 5 to 10 μm layers with pastes containing nominally 14 and 6 μm diamond grits. The surface finishes of the specimens were measured with a Talysurf axially

0022–2461/88 $03.00 + .12

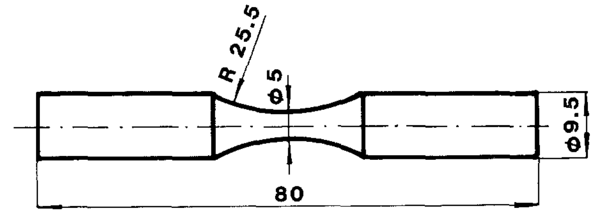


Figure 1 Specimen geometry of Mg-PSZ materials tested in cyclic flexural fatigue. Dimensions in millimetres.

along the waisted section. These results are summarized in Table I. Scanning electron micrographs of the various surfaces prior to testing are shown in Fig. 2.

3. Test results

The specimen geometry shown in Fig. 1 was clamped with a collet arrangement into the head of a spindle and an extension arm and weight on the other end. Spindle speeds of the tests were at 200 and 15.5 Hz. The bending stress was determined from the simple relationship $\sigma = (M/I)r$, where M is the bending moment, I the moment of inertia and r the radius of the minimum section thickness. The bending stress was varied such that lifetimes within the range 10^3 to 10^8 cycles were measured. Typically 10 to 15 specimens were broken for each surface condition. Tests were usually interrupted after 10^8 cycles, corresponding to a period of four days for the 200 Hz testing machine.

The *S-N* results of different surface conditions of the specimens tested at 200 Hz are shown in Fig. 3. Those rods that had not fractured after 10^7 to 10^8 cycles are marked with an arrow. The results have been plotted on log–log scales to enable the slope and the stress corrosion index to be determined. Fig. 4 shows the results for axially ground specimens tested at a frequency of 15.5 Hz.

The specimens tested at 200 Hz (Fig. 3) reveal only minor differences between the various surface preparations. The radially ground bars tended to have approximately 50 MPa ($\sim$ 10%) lower strengths than the axially ground specimens. The data, although limited, suggest that a fatigue limit of approximately $\pm$ 400 MPa, as specimens loaded at this stress did not fail after 10^8 cycles. The slightly lower strengths of the

TABLE I Specimen surface finishes

	Grinding speed ($m\,sec^{-1}$)	Roughness RMS (μm)
Axial ground	22	0.2
Radial ground	31	1.1
Polished	–	0.1

radially ground specimens are probably associated with the more favourable alignment of any flaws introduced during grinding in this direction. However, the reduction in strength between grinding directions parallel and perpendicular to the stressing direction is significantly less than for most ceramics. For instance Anderson and Bratton [11] found a 50% reduction in the strength of hot-pressed silicon nitride when ground perpendicular to the stressing direction in comparison with parallel-ground bars.

The decreasing strength of metallic materials with stressing cycles is generally associated with the initiation and propagation of surface cracks with increasing increments of crack growth with every stressing cycle. That is, localized plastic deformation at some stress concentrator or crack tip occurs during cycling plus any influence of the environment on bond rupture at the crack tip. For classic brittle materials such as glasses, strength degradation under static or cyclic stresses is considered to be a manifestation of moisture-assisted stress corrosion cracking. The rate of crack growth, V, has been found to fit a linear relationship of the form $V = AK^n$ where A is a constant, K the crack tip stress intensity factor and n the stress corrosion exponent. For cyclic stressing of such brittle materials it has been argued that the lifetime of the material is simply the integrated time under the tensile cycle [12]. However, even for polycrystalline alumina [9] the stress corrosion exponent calculated from cyclic stressing is significantly lower than that determined from static fatigue tests or direct crack velocity measurements.

For PSZ materials both reversible and irreversible transformations of the tetragonal to monoclinic phase of zirconia occur upon stressing [13]. The irreversible aspect of the transformation about a crack tip makes

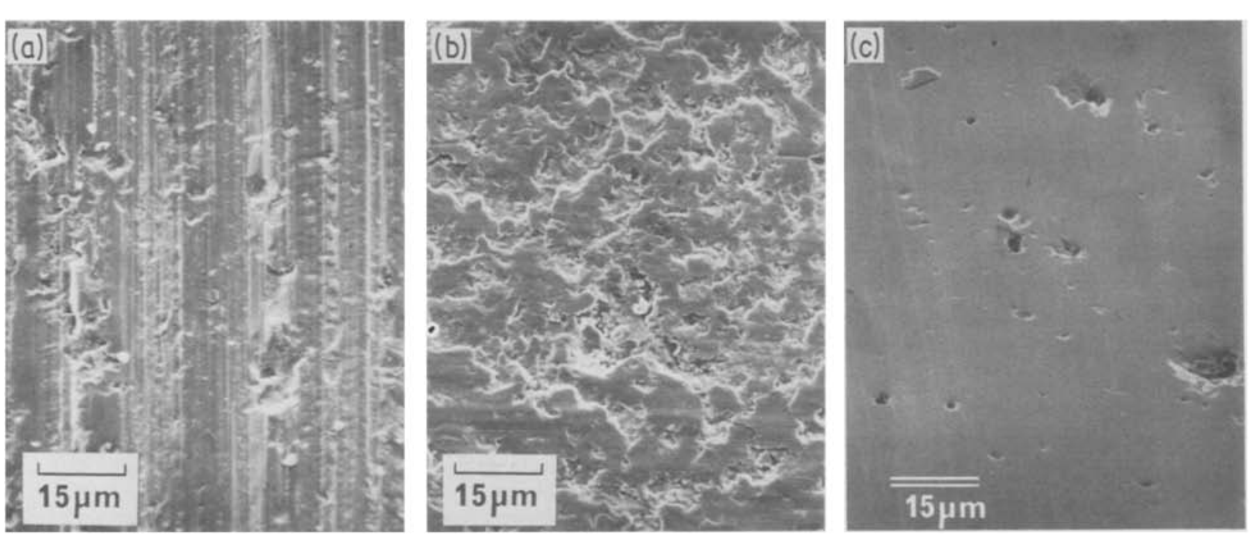


Figure 2 Scanning electron microscope observations of the surface finish of the specimens tested: (a) radial, (b) axial, (c) polished.

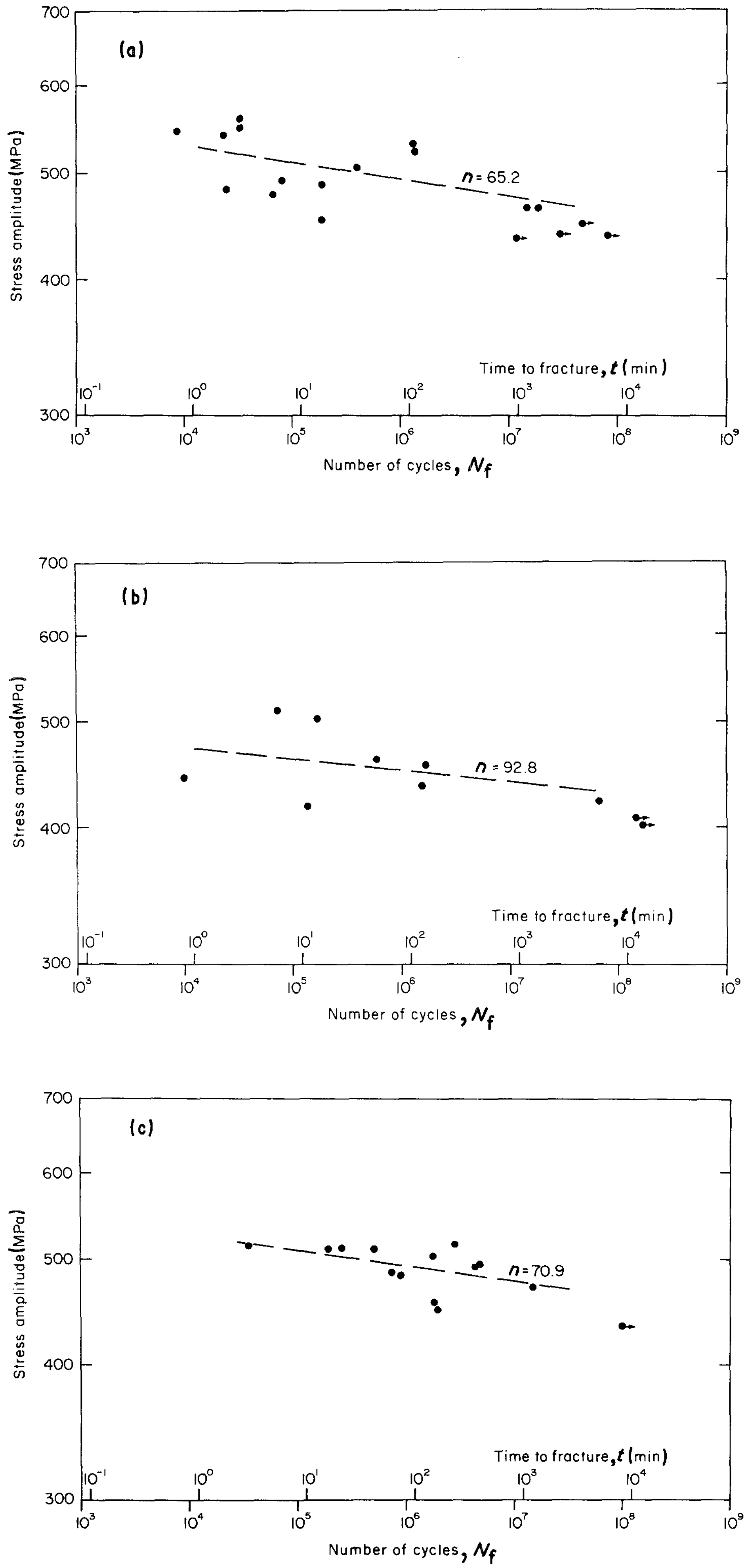


Figure 3 Observations of the strength–lifetime or cycles to failure of specimens (a) axially ground, (b) radially ground and (c) polished, all tested at 200 Hz in air.

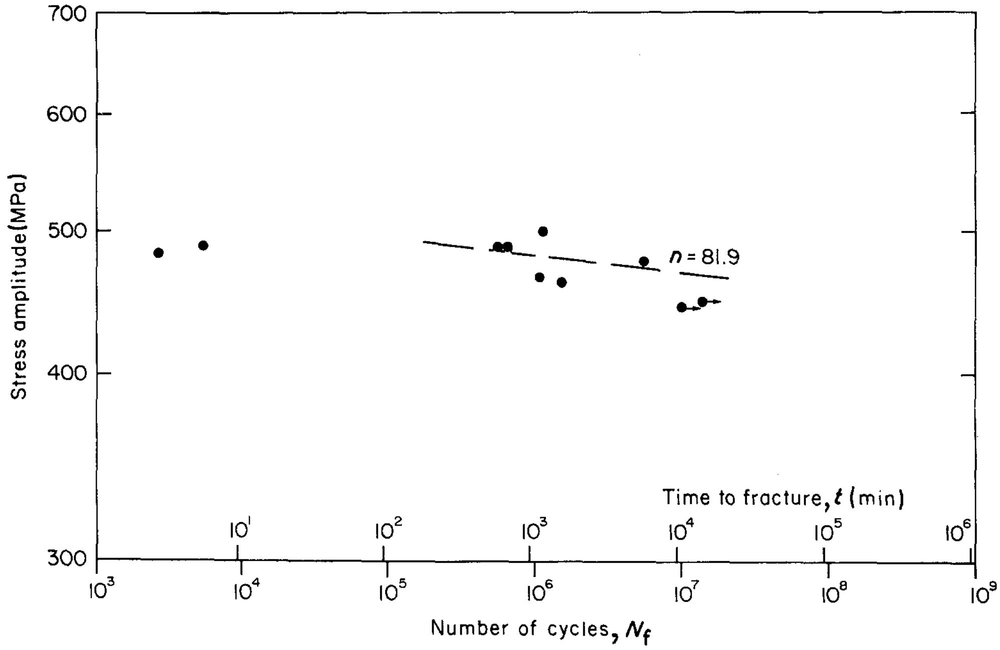


Figure 4 Observed strength–lifetime or cycles to failure of specimens axially ground, tested at 15.5 Hz.

PSZ more metallic in behaviour, and presumably results in cyclic fatigue being more deleterious than static fatigue. Direct measurements of crack velocity in conventional double cantilever beam (DCB) geometry specimens gave n values of 110 to 120 for the Mg-PSZ MS material tested, much higher by a factor of two than the present estimates of n [14]. Close scrutiny of the axial-ground results at 200 and 15.5 Hz shows that the number of cycles to failure is less at the lower frequency. However, when both sets of results are plotted against time to failure rather than cycles to failure reasonable superposition of the data occurs (Fig. 5).

A previous study of rotating bending fatigue of Mg-PSZ by Rauchle and Thieman [15] found significantly different results from those presented here. They observed a much greater reduction in strength with number of cycles when tested at 100 Hz in air. The retained strength after 10^8 cycles was only ± 200 MPa and the slope of the data gave an n value of 20. Engel *et al.* [16] in a study of the microstructure of a number of ceramics reported that the Mg-PSZ material they investigated contained substantial silicious glassy phases at grain boundaries and triple points. In contrast, the material evaluated in this study has minimal glass at the grain boundaries because of specific additives [17]. The presence of the glass at grain boundaries, which is known to have lower values of n (typically 15 to 30 [18]), may account for the lower stress corrosion index of the Mg-PSZ material evaluated by Rauchle and Thieman [15]. More recent studies of cyclic flexural fatigue of Mg-PSZ [19] have

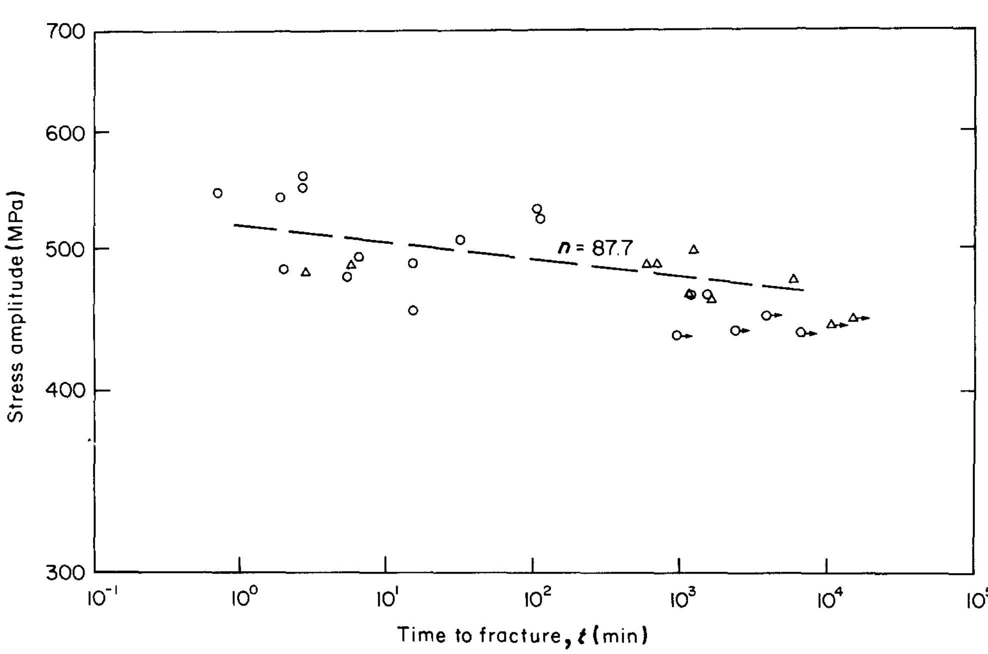


Figure 5 Comparison of the strengths of axially ground specimens tested by cyclic flexure at (△) 15.5 and (○) 200 Hz when plotted using time to failure as the horizontal axis.

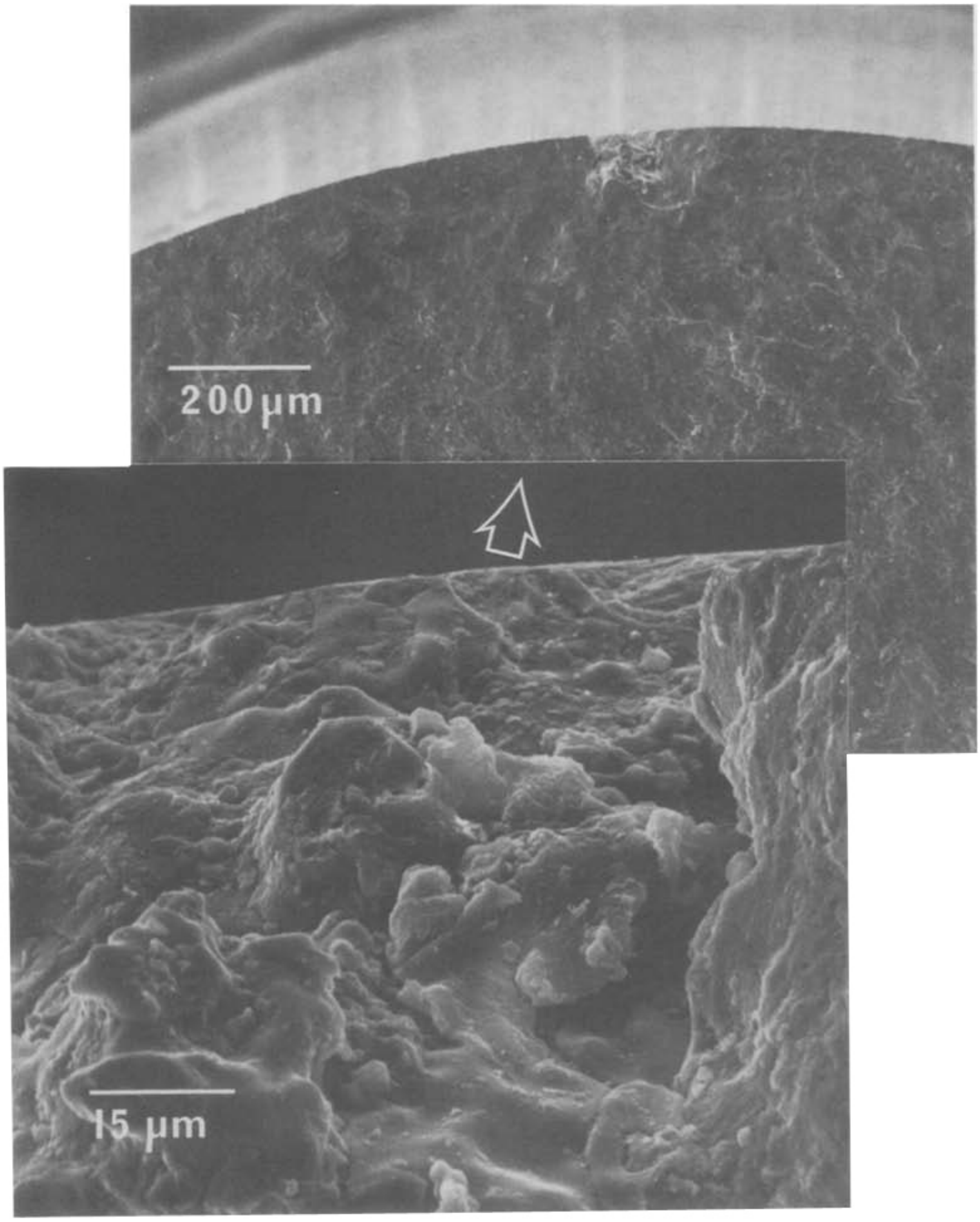


Figure 6 Scanning electron microscope observation of the fracture surface and origin of a specimen that failed at a stress of 453 MPa after 1.9×10^5 cycles when tested at 200 Hz.

found lower n values than those reported in this work. Such observations may suggest that cyclic fatigue crack propagation similar to that in metals does take place in transformation-toughened Mg-PSZ.

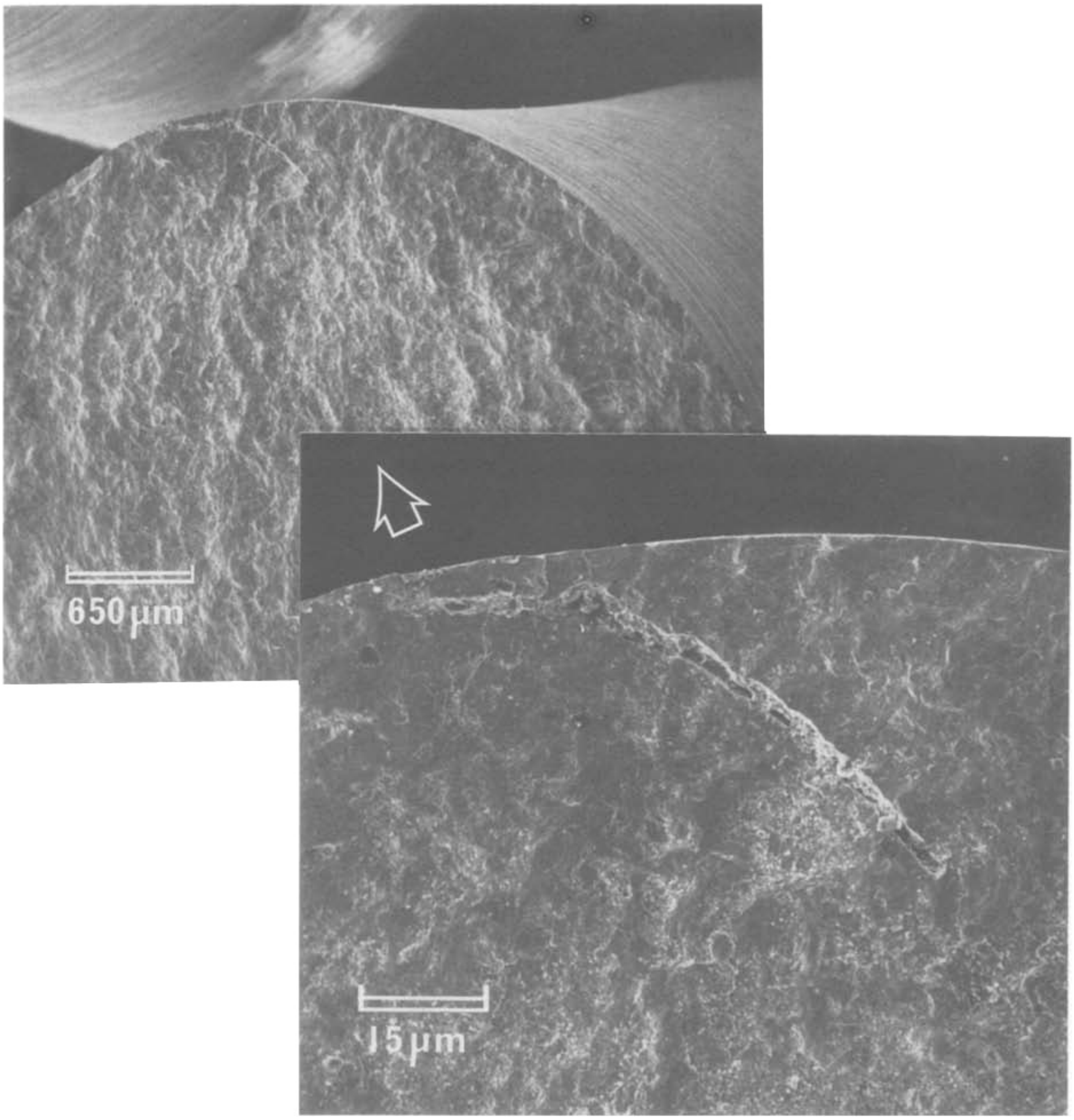
Fracture surfaces of specimens that failed during the course of these tests are shown in Figs 6 and 7. In no instances were fatigue striations present as observed in metals. In some circumstances a lip developed on the fracture surface near the edge on the side opposite the fracture origin. As seen in Figs 6 and 7, the fracture surface was very rough with poorly defined Wallner lines radiating from the fracture origin. The origins of the fracture were sometimes well defined as in Fig. 6, which consisted of a porous glassy region. Other fracture origins included fibre-like defects, larger pores and occasionally surface cracks as in Fig. 7.

An estimate of the critical stress intensity factor for the flaws and fracture origins may be had from the fracture mechanics expression

$$K_c = 1.3\,\sigma a^{1/2}$$

where 1.3 is the geometrical parameter for a semicircular surface flaw of dimensions $2a$ and subjected to a stress of σ. Using a failure stress of 400 MPa and the flaw sizes measured from Figs 6 and 7, namely $2a$ ranging from 130 to 170 μm, this leads to typical values of K_c from 4.2 to 4.8 MPa m$^{1/2}$. These values are somewhat towards the lower range of measured K_c in R-curve estimates of the MS material evaluated [20], and lower than anticipated for steady-state subcritical crack growth of large cracks [14].

In further publications the influence of temperature and environment on the rotating bending fatigue of Mg-PSZ heat-treated to various fracture toughnesses will be presented [19].



Acknowledgements

The authors wish to thank Nilcra Ceramics for supply of the materials tested.

References

1. N. CLAUSSEN, in "Advances in Ceramics", Vol. 12, edited by N. Claussen, M. Ruhle and A. H. Heuer (American Ceramic Society, 1984) p. 325.
2. R. C. GARVIE, *ibid.* p. 465.
3. P. F. BECHER, M. V. SWAIN and M. FERBER, *J. Mater. Sci.* **22** (1987) 63.
4. D. B. MARSHALL, *J. Amer. Ceram. Soc.* **69** (1986) 173.
5. R. W. HERTZBERG, "Deformation and Fracture Mechanics of Engineering Materials", 2nd Edn (Wiley, 1983).
6. H. N. KO, *J. Mater. Sci. Lett.* **5** (1986) 464.
7. *Idem, ibid.* **6** (1987) 175.
8. M. V. SWAIN, *Mater. Forum* **9** (1986) 34.
9. D. A. KROHN and D. P. H. HASSELMAN, *J. Amer. Ceram. Soc.* **55** (1972) 208.
10. R. H. J. HANNINK and M. V. SWAIN, *J. Aust. Ceram. Soc.* **18** (1982) 53.
11. C. A. ANDERSON and R. J. BRATTON, in "The Science of Ceramic Machining and Surface Finishing II", edited by B. J. Hockey and R. W. Rice, NBS Special Publication 562 (1979) p. 463.
12. A. G. EVANS and E. R. FULLER, *Metall. Trans.* **5** (1974) 27.
13. D. B. MARSHALL and M. R. JAMES, *J. Amer. Ceram. Soc.* **69** (1986) 215.
14. P. F. BECHER, *J. Mater. Sci.* **21** (1986) 297.
15. W. RAUCHLE and K. H. THIEMAN, unpublished poster presented at Zirconia 83, Stuttgart, June 1983.
16. L. ENGEL, V. LEYDENROTH and K. H. THIEMAN, *Ber. Dt. Keram. Gesell.* **9** (1984) 7.
17. J. DRENNAN and R. H. J. HANNINK, *J. Amer. Ceram. Soc.* **69** (1986) 541.
18. S. M. WEIDERHORN, in "Fracture Mechanics of Ceramics", Vol. 4, edited by R. C. Bradt, D. P. H. Hasselman and F. F. Lange (Plenum, New York) p. 549.
19. V. ZELIZKO and M. V. SWAIN, to be published.
20. M. V. SWAIN, *High Technol. Ceram.* in press.

Received 4 June
and accepted 27 July 1987