Journal of the European Ceramic Society **11** (1993) 201–204

Static-Fatigue Life of a TZP/Alumina Composite

Jeffrey J. Swab & Steven C. Stowell

US Army Research Laboratory, Watertown Site, Massachusetts 02172-0001, USA

(Received 29 January 1992; revised version received 21 April 1992; accepted 12 May 1992)

Abstract

Stepped-temperature stress–rupture tests were used to assess the static-fatigue life of a TZP/alumina ceramic between 800°C and 1200°C. These tests were carried out with and without a 100 h treatment at 1000°C in air. Stress rupture performance of the as-received material was superior between 800°C and 1000°C, while both the as-received and heat-treated materials had essentially the same performance at 1100°C and beyond. Fractographic analysis was used to elucidate failure mechanisms. This analysis revealed that slow crack growth was the primary failure mechanism at 1100°C and higher.

Um die Lebensdauer einer TZP/Al_2O_3-Keramik im Bereich zwischen 800°C und 1200°C für den Fall statischer Ermüdung zu bestimmen, wurden Zeitstandversuche bei verschiedenen Temperaturen durchgeführt. Diese Versuche wurden vor und nach einer 100-stündigen Wärmebehandlung des Materials bei 1000°C an Luft durchgeführt. Die Lebensdauer des unbehandelten Ausgangsmaterials war im Bereich zwischen 800°C und 1000°C dem wärmebehandeltem Material überlegen. Bei Temperaturen um 1100°C und höher zeigen beide Materialien jedoch weitgehend gleiches Verhalten. Fraktographische Untersuchungen wurden herangezogen, um die Bruchmechanismen zu klären. Diese Analysen zeigten, daß langsames Rißwachstum den primären Versagensmechanismus bei 1100°C und höheren Temperaturen darstellt.

Des tests de rupture sous contrainte menés à différentes températures entre 800 et 1200°C ont permis d'évaluer la durée de vie sous fatigue statique d'une céramique TZP/alumine. Ces tests ont été effectués avec et sans un traitement préalable de 100 heures à 1000°C dans l'air. La performance (exprimée en terme de rupture sous contrainte) des matériaux testés sans traitement préalable est meilleure entre 800 et 1000°C, tandis qu'à 1100°C et au-delà, les deux matériaux (traité et non traité) présentent des performances très semblables. L'analyse fractographique a été utilisée pour identifier les mécanismes d'endommagement. Cette analyse révèle qu'une croissance lente des fissures est à l'origine du mécanisme principal de rupture à 1100°C et au-delà.

1 Introduction

Yttria–tetragonal zirconia polycrystal (Y-TZP) materials have been considered for high-temperature structural applications because high strength and toughness values can be achieved as a result of transformation toughening. Despite the aforementioned properties, the prospects for using Y-TZP materials in these applications have not been realized. This is due to a significant decrease in strength and toughness after long-term exposure to elevated temperatures,[1,2] after exposure to 200–300°C in the presence of water vapor,[3–6] and at elevated temperatures.[1,7,8] In all cases the decrease in properties can be attributed to reduced transformation toughening. Recently, studies[8,9] at TOSOH Corporation (formerly Toyo Soda Manufacturing Company) have shown that room-temperature three-point flexure strength values as high as 2400 MPa could be attained with a Y-TZP containing 20–30 vol.% α-Al_2O_3. This strength is approximately twice the strength values reported for Y-TZP materials without Al_2O_3.[1,2,8]

In order to determine if the improved room-temperature strength would translate into improved high-temperature performance elevated-temperature tests needed to be conducted. The present paper provides preliminary information on the static-fatigue life and failure mechanisms of a TZP/Al_2O_3 between 800°C and 1200°C.

2 Material

The material evaluated was produced by TOSOH Corporation of Tokyo, Japan using TOSOH TZ-3Y20A 'Super Z' zirconia/alumina powder. The powder was formed in billets 15·3 cm × 15·3 cm × 1·3 cm in size through a sinter/hot isostatic pressing (HIPing) process. Primary sintering was done at ≈1400°C followed by HIPing at ≈1500°C. The composition of the powder, according to company literature, is 75·7 wt% tetragonal zirconia (partially stabilized by 4·2 wt% yttria) with 20 wt% α-alumina. Typical impurity content is as follows: SiO_2 (0·01 wt%), Fe_2O_3 (0·005 wt%) and Na_2O (0·007 wt%).

3 Experimental Procedure

Billets of the 'Super Z' material were obtained and machined into type 'B' flexure bars (3 mm × 4 mm × 50 mm) according to MIL-STD 1942A. Half of the bars were heat treated for 100 h at 1000°C, in air, at laboratory ambient humidity.

Stepped-temperature stress–rupture (STSR) testing was done to analyze the static-fatigue life of the material between 800°C and 1200°C. STSR testing was carried out in four-point flexure following the procedure outlined by Quinn & Katz.[10] A bar is loaded onto a four-point-flexure fixture that is in a furnace and heated to 800°C in ≈2 h, in air, with no applied stress to the bar. Upon reaching the required temperature, a predetermined stress is applied and the bar is allowed to soak under load, for up to 24 h. If the bar survives this step, then the furnace is heated to 900°C (in ≈10 min), while under the same applied stress and again allowed to soak for up to 24 h. This cycle is repeated for 1000°C, 1100°C and 1200°C. If the bar fractures or excessive creep occurs the power to the furnace is automatically shut off by a microswitch. The time of fracture is denoted on the STSR plot using an arrow, with the applied stress that caused fracture above the arrow. Optical and scanning electron microscopy was done in an attempt to characterize the fracture origins in the material. Fractographic analysis was carried out according to Ref. 11.

4 Results and Discussion

The static-fatigue life with and without heat treatment is shown in Fig. 1. In both cases the static-fatigue life decreases as the temperature increases. This is not unexpected, since as the temperature approaches 1000–1100°C (the temperature range at which the tetragonal phase is stable), the driving force for the tetragonal-to-monoclinic transformation gradually decreases.

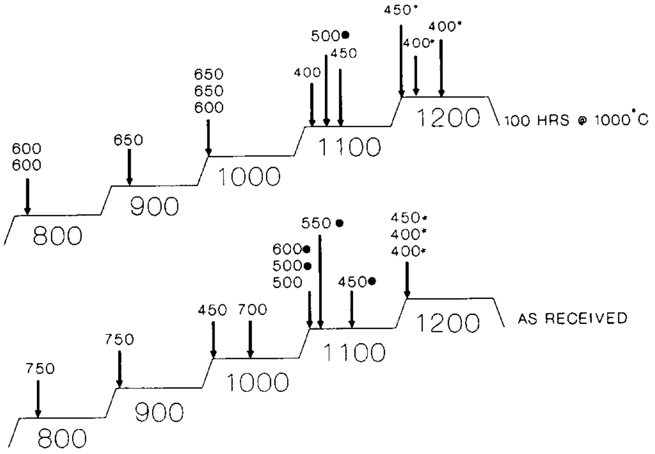

Fig. 1. Stepped-temperature stress–rupture results with and without heat treatment at 1000°C for 100 h. The arrow denotes the time of fracture and the number above it the applied stress that caused fracture. Each stress above the arrow is for a different bar. Stress in MPa; temperature in °C. ★, Test terminated due to creep; ●, creep fracture. 24 Hour hold at each temperature; 10 min between steps.

Between 800°C and 1000°C the material in the as-received condition performs slightly better than the heat-treated material. This may be due to compressive stresses created in the surface of the bar during machining. At elevated temperatures, thermal relaxation of these stresses is expected, but studies[12,13] have shown that it is a time-dependent process, and in this study exposure times are relatively short.

Because the driving force for the tetragonal-to-monoclinic transformation decreases as the temperature increases the strength-at-temperature becomes dependent on the microstructural parameters, such as grain size and grain size distribution.[7] Since there is no noticeable grain growth even after the heat treatment, the static-fatigue life

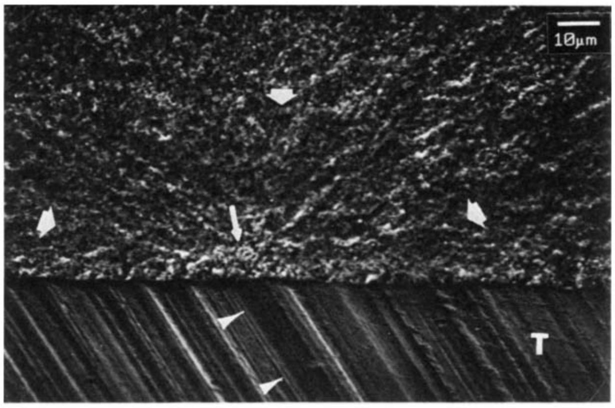

Fig. 2. SEM micrograph of the fracture surface of an as-received specimen which failed after 0·1 h at 1000°C under a 650 MPa applied stress. Surface machining damage is noted by the arrowheads and the semi-elliptical crack by the small white arrow. Large white arrows outline the mirror region and T denotes the tensile surface.

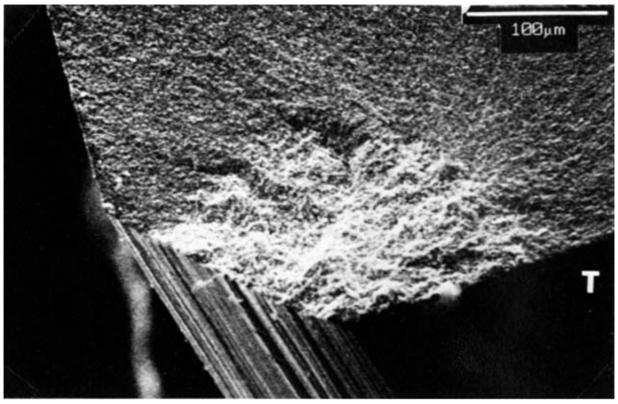

Fig. 3. SEM micrograph of the slow crack growth region of an as-received specimen which failed after 0·3 h at 1200°C under a 450 MPa applied stress. T denotes the tensile surface.

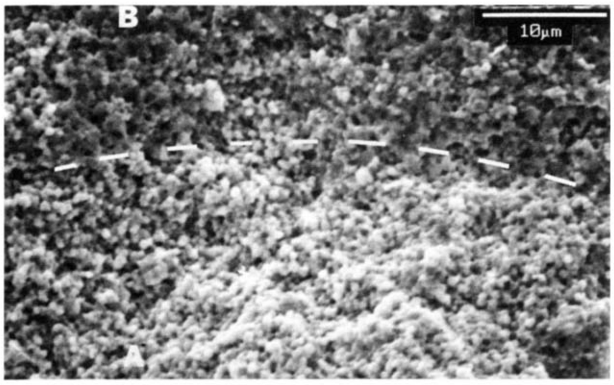

Fig. 5. SEM micrograph showing the transition from (A) intragranular fracture in the slow crack growth region to (B) transgranular fracture outside this region.

for both conditions is expected to be similar. Indeed, this is the case for temperatures of 1100°C and higher.

Permanent strain was measured on all specimens which survived into the 1100°C and 1200°C steps. This value ranged from a low of 0·27% at 1100°C to a high of 4·7% at 1200°C. The large amount of permanent strain at 1200°C can be attributed to the phenomenon of superplasticity which has been found in this ceramic.[14–16] Permanent strain was not observed for either the as-received or heat-treated material below 1100°C.

Fractographic analysis indicates that during the first three steps of this test, fracture initiates at damage created during machining, see Fig. 2. Starting at 1100°C, the failure mechanism changed to slow crack growth, which is presumed to have started from machining damage, as seen in Figs 3–5. This is the case for both conditions. To the authors' knowledge, this is the first instance where slow crack growth has been found in TZP materials.

There are differences in the static-fatigue life and fracture mechanisms of this composite when compared to Y-TZPs without Al_2O_3 but with similar grain sizes,[7] see Figs 1 and 6. The composite has a static-fatigue life approximately twice that of the Y-TZP without Al_2O_3. This appears to be the result of both very small flaws and the addition of the Al_2O_3. The best Y-TZP in Ref. 7 failed from large porosity-related flaws. A comparable amount of plastic deformation was found at 1100°C and higher in the Y-TZPs without Al_2O_3 but no slow crack growth was observed.

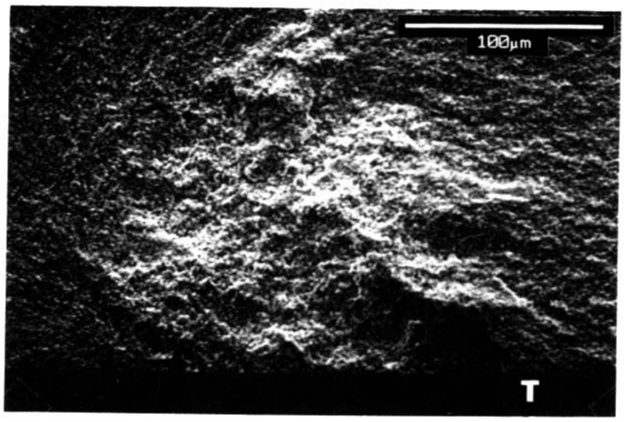

Fig. 4. SEM micrograph of the slow crack growth region of a specimen which failed after 6 h at 1100°C under a 500 MPa applied stress. Specimen was heat treated before testing. T denotes the tensile surface.

Although the static-fatigue life of this composite is markedly better than previously tested Y-TZPs,[7] it still may not be sufficient for certain high-temperature structural applications. This is the case for the next generation of diesel engines which have a strength-at-temperature requirement of >800 MPa at >982°C.[17]

5 Conclusions

The static-fatigue life of a TZP/Al_2O_3 ceramic between 800°C and 1200°C was found to be significantly better than that of a Y-TZP without Al_2O_3. However, it still fell short of the strength-at-temperature requirements for some high-temperature structural applications.

Fractographic analysis revealed that at temperatures below 1100°C, fracture occurred by fast fracture stemming from machining damage. At 1100°C and above, slow crack growth became the

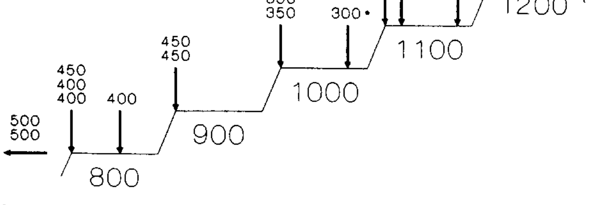

Fig. 6. Stepped-temperature stress–rupture results for a TZP without Al_2O_3, from Ref. 7. Strength at 25°C = 1169 MPa. Stress in MPa; temperature in °C. ★, Test terminated due to creep; ●, creep fracture. 24 Hour hold at each temperature; 10 min between steps.

dominant failure mechanism. This is the first time slow crack growth has been observed in a TZP material.

References

1. Larsen, D. C. & Adams, J. W., Long-term stability and properties of zirconia ceramics for heavy duty diesel engine components. NASA-Lewis Research Center CR-174943, prepared for the US Department of Energy under Contract DEN 3-305, September 1985.
2. Swab, J. J., Properties of yttria–tetragonal zirconia polycrystal (Y-TZP) materials after long-term exposure to elevated temperatures. US Army Materials Technology Laboratory TR 89-21, prepared for the US Department of Energy under Interagency Agreement DE-AI05-84OR21411, NTIS Access No. ADA-207064, March 1989.
3. Watanabe, M., Iio, S. & Fukuura, I., Aging behavior of Y-TZP. In *Advances in Ceramics, Science and Technology of Zirconia II*, Vol. 12, ed. N. Claussen, M. Ruhle & A. H. Heuer. The American Ceramic Society, Columbus, OH, 1984, pp. 391–398.
4. Lu, H. Y. & Chen, S. Y., Low temperature aging of *t*-ZrO_2 polycrystals with 3 mol.% Y_2O_3. *J. Am. Ceram. Soc.*, **70** (1987) 537–41.
5. Sato, T., Ohtaki, S., Endo, T. & Shimada, M., Humidity dependence of phase transformation from tetragonal to monoclinic structure in yttria-doped tetragonal zirconia. In *High Tech. Ceram.*, ed. P. Vincenzini. Elsevier Science Publishers, B.V., Amsterdam, The Netherlands, 1987, pp. 281–8.
6. Swab, J. J., Low temperature degradation of Y-TZP materials. *J. Mat. Sci.*, **26** (1991) 6706–14.
7. Swab, J. J., Performance of Y-TZP materials between 800°C and 1200°C, US Army Materials Technology Laboratory TR 90-3, prepared for the US Department of Energy under Interagency Agreement DE-AI05-84OR21411, NTIS Access No. ADA-217723, January 1990.
8. Tsukuma, K., Takahata, T. & Shiomi, M., Strength and fracture toughness of Y-TZP, Ce-TZP, Y-TZP/Al_2O_3, and Ce-TZP/Al_2O_3. In *Advances in Ceramics, Science and Technology of Zirconia III*, Vol. 24, ed. S. Somiya, N. Yamamota & H. Hanagida. The American Ceramic Society, Columbus, OH, 1988, pp. 721–8.
9. Tsukuma, K., Ueda, K. & Shimada, M., Strength and fracture toughness of isostatically hot-pressed composites of Al_2O_3 and Y_2O_3-partially-stabilized ZrO_2. *J. Am. Ceram. Soc.*, **68** (1985) C-4–C-5.
10. Quinn, G. D. & Katz, R. N., Stepped temperature stress rupture testing of silicon-based ceramics. *Am. Ceram. Soc. Bull.*, **57** (1978) 1057–8.
11. Quinn, G. D., Swab, J. J. & Slavin, M. J., A proposed standard practice for fractographic analysis of monolithic advanced ceramics. *Fractography of Glasses and Ceramics II, Ceramic Transactions*, Vol. 17, ed. V. Frechette & J. Varner. The American Ceramic Society, Westerville, OH, 1991, pp. 309–61.
12. Green, D. J., Critical microstructures for microcracking of Al_2O_3–ZrO_2 composites. *J. Am. Ceram. Soc.*, **65** (1982) 610–14.
13. Green, D. J., Lange, F. F. & James, M. R., Residual surface stresses in Al_2O_3–ZrO_2 composites. In *Advances in Ceramics, Science and Technology of Zirconia II*, Vol. 12, ed. N. Claussen, M. Ruhle & A. H. Heuer. The American Ceramic Society, Columbus, OH, pp. 240–50.
14. Wakai, F., Sakaguchi, S. & Matsuno, Y., Superplasticity of yttria-stabilized tetragonal ZrO_2 polycrystals. *Adv. Ceram. Mater.*, **1** (1986) 259–63.
15. Wakai, F. & Kato, H., Superplasticity of TZP/Al_2O_3 composite. *Adv. Ceram. Mater.*, **3** (1988) 71–6.
16. Nieh, T. G., McNally, C. M. & Wadsworth, J., Superplastic behavior of a 20% Al_2O_3/YTZ ceramic composite. *Scripta Met.*, **23** (1988) 457–60.
17. Bryzik, W. & Kamo, R., TACOM/Cummins adiabatic engine program. SAE Paper 830314 in SP-543—The adiabatic diesel engine. SAE International Congress and Exposition, Detroit, MI, 1983, pp. 21–45.