# Indentation fatigue of ceramic nuclear fuels

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The possibility of using an indentation technique for the determination of fatigue behaviour of sintered ceramic nuclear fuel pellets has been explored. A relation between load and number of cycles to failure has been derived. The underlying principle of this technique has also been explained.

## 1. Introduction

Ceramic materials are increasingly used for critical high-strength applications in the field of aerospace, heat engines, nuclear reactors, aircraft components, etc. The strength of ceramics is time dependent due to the propagation of surface microcracks. Failure predominantly occurs from the stress-dependent growth of a pre-existing flaw to a dimension critical for spontaneous crack propagation [1]. Because the ceramics would be subjected to cyclic loading for prolonged periods, it is essential to know their fatigue behaviour [2]. While considerable work has been done in the field of creep and fracture of ceramics, comparatively little is known about their behaviour under cyclic loading conditions. Hence, a study was undertaken to estimate the fatigue behaviour of ceramics in which fatigue properties of ceramic nuclear fuel pellets are estimated at room temperature using a new technique called indentation fatigue [3]. In this technique a sharp indentor (Vickers) is indented on the sample under constant load for a particular dwelling time and then it is withdrawn. Indentations are repeated at the same point without disturbing the set up until it results in the formation of chips from the corners of the indentation which are called lateral chips. The advantages of this technique lie in its simplicity and minimal material usage compared to the conventional fatigue testing which is time consuming and requires large size samples.

This paper deals with the fatigue studies of sintered  $UO_2$  fuel pellet by the above-mentioned technique. A relation between load and number of cycles required for the formation of lateral chips has been derived. The underlying principle of this technique has also been studied and presented in this paper.

#### 2. The principle

The indentation technique has been established as a simple, but important, technique for the estimation of many mechanical properties of ceramic materials [4–7]. Many authors [8, 9] have studied the stress pattern under a sharp indentor. The nature of the stress field around a Vickers indentor is essentially elastic plastic. Just below the indentor, there is a highly localized plastic deformation zone which is surrounded by the elastic matrix.

The crack pattern produced by a standard Vickers indentor under the application of load, can be classified as follows.

1. Radial/median crack on symmetry planes normal to the surface and containing the load axis.

2. Palmqvist crack or surface crack.

3. Lateral cracks on shallow sub-surface planes normal to the load axis.

Fig. 1 shows a schematic view of the indentation fracture pattern for the Vickers indentor. On application of a load, P, to the specimen, a well-developed radial crack of dimension 2C is produced. In addition to this radial crack, a second type of crack, called a "lateral crack", is also produced. These lateral cracks originate from the deformation zone just beneath the indentation surface and spread outwards [10]. On cyclic loading these cracks grow further outwards and intersect the surface thereby causing chipping of the surface (Fig. 2).

#### 3. Experimental procedure

Uranium dioxide pellets were prepared from a single batch of green powder (ammonium diuranate route) by cold compaction and sintering at 1650°C for 4 h.

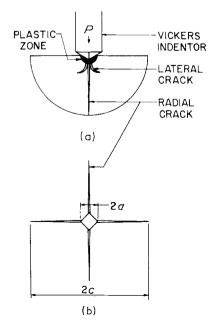


Figure 1 A schematic view of indentation showing different crack systems: (a) sectional view, (b) top view.

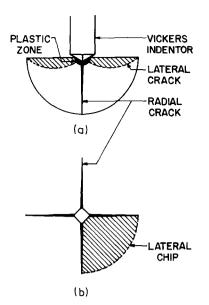


Figure 2 Illustration of indentation crack geometry after repeated indentations: (a) sectional view, (b) top view.

The pellets were about 12 mm diameter and 14 to 16 mm high with a density in the range 92 to 94% theoretical density (TD). The sintered pellet samples were metallographically polished to obtain a flat surface.

The experiment was conducted using the following loads: 600, 300, 200, 100, 50, 10, 7.5 and 5 N. In each case, the number of cycles required to cause lateral chips was noted. For loads above 30 N, an Instron testing system and for lower loads Shimadzu microhardness tester were used. In both cases, the loading rate and dwelling time were kept identical. A typical

load application pattern is shown in Fig. 3. The experiment was repeated five times for each load and the number of cycles required for the formation of chips was noted. The average of these with its standard deviation are used to obtain a plot of load against number of cycles (Fig. 4). A load of 5 N did not cause lateral chipping even after 100 cycles.

#### 4. Results and discussions

The load (S) against number of cycles (N) curve for sintered UO<sub>2</sub> pellet can be best represented by the following equation

$$S = 704.39 \,\mathrm{N}^{-1.12} \tag{1}$$

with the coefficient of regression r = 0.95, where S is the load (N), and N is the number of cycles.

The formation of the different types of cracks and their propagation leading to failure as loading cycles continue are shown in Figs 5a to f. During the first cycle, very well developed radial cracks were formed (Fig. 5a). Subsequently, with a greater number of load cycles, the radial crack grew further and secondary cracks were also generated as shown in Fig. 5b. Further, as the load cycles continued, fissures were formed near one of the corners of the indentation. The secondary crack grew further but the primary crack stopped growing. The fissure became widened on subsequent cycles as shown in Fig. 5d. The secondary crack grew further and underwent branching. A small area was chipped first on further loading and finally resulted in a complete lateral chip as shown in Fig. 5f. A cross-section of the indentation shows the lateral cracks (Fig. 6). This lateral crack grows further out-

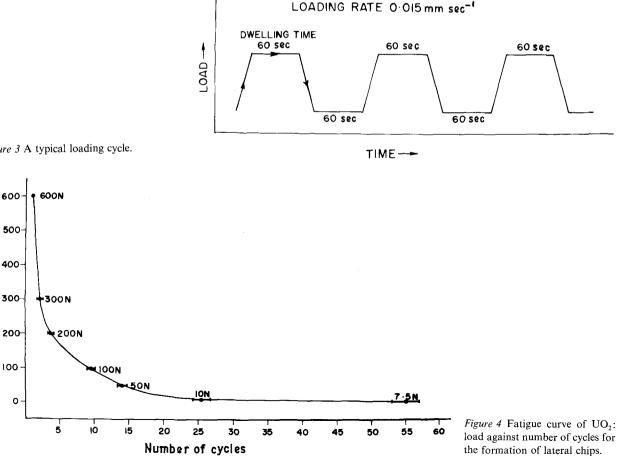


Figure 3 A typical loading cycle.

(N) poor

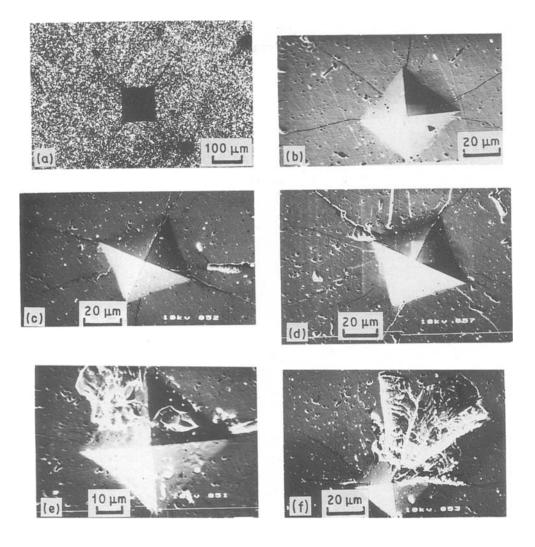
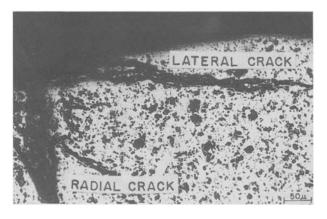


Figure 5 Microstructural changes resulting from repeated indentations with 10 N load: (a) 1 cycle, (b) 3 cycles, (c) 7 cycles, (d) 12 cycles, (e) 20 cycles, (f) 25 cycles.

wards on subsequent cycling and intersects the surface, resulting in the formation of a lateral chip (Fig. 5f).

Sample preparation is very important to obtain good results. Indentation fracture behaviour is extremely



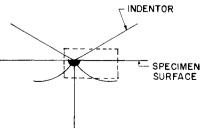


Figure 6 A sectional view of indentation showing lateral crack.

sensitive to surface preparation [11]. The surface may contain compressive residual stresses which might have developed from the grinding and polishing procedure. To minimize these effects, samples were ground using successively a series of silicon carbide abrasive papers with grit size 240, 320, 400 and 600 mesh and then polished with 8 and  $2 \mu m$  diamond paste, respectively. Care must also be taken to maintain the indentation axis parallel to the surface normal.

# 5. Conclusion

A novel technique for the evaluation of fatigue properties of ceramic nuclear fuel has been explained. This technique can also be used for the estimation of fatigue behaviour of other brittle ceramic materials. It may be concluded that the indentation technique using a Vickers indentor can be usefully employed to evaluate the fatigue behaviour of ceramic material such as compacted and sintered  $UO_2$ .

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## References

- K. JAKUS, D. C. COYNE and J. E. RITTER JR, J. Mater. Sci. 13 (1978) 2071.
- 2. J. R. MATHEWS and T. PREUSSER, Nucl. Engng Design 101 (1987) 281.
- 3. D. A. J. VAUGHAN, F. GUIU and H. R. DALMAN, J. Mater. Sci. Lett. 6 (1987) 689.
- B. R. LAWN and R. WILSHAW, J. Mater. Sci. 19 (1975) 1049.
- Hj. MATZKE, in Proceedings of the International Workshop on "Indentation Fracture and Mechanical Properties of Ceramic Fuels and of Waste Ceramics and Glasses", Karlsruhe, 11 to 12 November 1985, edited by Hj. Matzke, (Harwood Academic Publishers, Chur, Switzerland, 1986) pp. 1007-27.
- 6. M. T. LAUGIER, *ibid.* pp. 1171-82.
- 7. T. R. G. KUTTY, K. N. CHANDRASEKHARAN,

J. P. PANAKKAL and J. K. GHOSH, J. Mater. Sci. Lett. 6 (1987) 260.

- 8. D. B. MARSHALL and B. R. LAWN, J. Mater. Sci. 14 (1970) 2001.
- 9. G. R. ANSTIS, P. CHANTIKUL, B. R. LAWN and D. B. MARSHALL, J. Amer. Ceram. Soc. 64 (1981) 533.
- C. A. BROOKS and J. D. J. ROSE, in Proceedings of the International Workshop on "Indentation Fracture and Mechanical Properties of Ceramic Fuels and of Waste Ceramics and Glasses", Karlsruhe, 11 to 12 November 1985, edited by Hj. Matzke, (Harwood Academic Publishers, Chur, Switzerland, 1986) pp. 1147-57.
- 11. D. K. SHETTY, I. G. WRIGHT, P. N. MINCER and A. H. CLAVER, J. Mater. Sci. 20 (1985) 1873.

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