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Uniaxial stress behaviour of Y–TZP

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Uniaxial stress behaviour of yttria–tetragonal zirconia polycrystal (Y–TZP) was investigated using a novel split Hopkinson pressure bar technique. The Y–TZP specimens were loaded by a single compressive pulse of predetermined shape and duration. The stress–strain response of the material revealed a linear elastic regime followed by an inelastic regime during which tetragonal to monoclinic transformation occurs. After the transformation was complete, the stress–strain behaviour was found to be elastic with a slope similar to that of the untransformed material. This behaviour suggests that the modulus of the transformed monoclinic zirconia is essentially the same as that of the untransformed tetragonal zirconia, and microcracking during transformation does not significantly affect the subsequent uniaxial stress–strain relation in Y–TZP. Ultrasonic measurements before and after the experiments, and microscopic observations on transformed zirconia specimens also support this conclusion, the latter revealing a very small number of nanometre-size cracks where martensitic laths in partially transformed grains interface with grain boundaries.

1. Introduction

Zirconia ceramics have received considerable attention in the literature due to their ability to undergo stress-induced martensitic transformation from tetragonal to monoclinic crystal structure. The transformation behaviour is studied in depth in bending [1, 2], tension [3], compression [4–6], and indentation experiments [7] on various zirconia ceramics, such as partially stabilized zirconia (PSZ), tetragonal zirconia polycrystals (TZP), and zirconia toughened ceramics (ZTC). This inelastic behaviour is also found to be responsible for the high fracture toughness observed in these ceramics [8–10].

The transformation from tetragonal to monoclinic structure is dilatational in nature. The stress required to induce transformation in these ceramics under compressive loading is generally many-fold greater than that in tension. This introduces considerable difficulty in experimental evaluation of dynamic properties of these ceramics because of their high compressive strength. In the case of yttria–tetragonal polycrystalline materials, the dynamic compressive stress required to induce transformation is found to be around 4 GPa [4]. In addition, some zirconia ceramics such as Mg–PSZ undergo extensive microcracking during their inelastic behaviour and therefore, recovering a specimen after extensive damage becomes very difficult. Suitable modifications need to be made in the testing procedure and in the equipment to test these materials successfully and recover them intact after the experiment for microscopic examination. A novel stress-reversal Hopkinson bar technique developed at the University of California, San Diego (UCSD) has been successfully used to achieve these high stress levels and completely recover a ceramic specimen even after inducing extensive damage during the stress-induced transformation. Details of this technique are available elsewhere in the literature and will not be discussed here [11, 12].

The objective of this paper is to report the results of our investigation on the dynamic uniaxial stress behaviour of yttria–tetragonal zirconia polycrystals (Y–TZP) under compressive loading. Limited information is available in the literature on the dynamic response of these ceramics. To the best of our knowledge, experimental evidence is also unavailable on the post-transformation behaviour of these ceramics, even though certain mechanisms of microcracking in this material are well established [13]. It is also the objective of this paper to address the complete stress–strain behaviour of these ceramics and provide experimental and microstructural evidence on the effect of microcracking on the post-transformation constitutive behaviour of Y–TZP ceramics.

2. Experimental procedure

3 mol % Y–TZP was obtained from Nilcra Ceramics (Elmhurst, IL) in bar stock. It has an average grain size of 0.5 μm and its fracture toughness is reported to be 6 MPa $m^{1/2}$. The bars were cut into specimens of 4.1 mm × 4.1 mm × 8.1 mm size. They were loaded on their square faces in the Hopkinson bar. Strain gauges were mounted on the lateral rectangular faces of the specimen in order to measure axial and transverse strains during the deformation. The loading surfaces were kept parallel to within ± 0.0025 mm to avoid

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unacceptable non-uniform strains which can cause premature failure in the specimen during the loading.

A modified Hopkinson bar technique was used. Details of the modifications of the Hopkinson bar to test hard ceramics are given elsewhere [11]. It is worthwhile to mention that the modifications ensure that the ceramic specimen is subjected to a single triangular compressive pulse (60 μs duration, in the present case). Indentation of the hard ceramic into the bars is also avoided by placing impedance-matched tungsten carbide pieces between the specimen and the steel bars. The technique has been successfully used on Mg–PSZ ceramics, as reported elsewhere in the literature [4, 12].

Because it is well known that zirconia ceramics undergo extensive microcracking during stress-induced transformation, the amount of input energy is controlled by subjecting the specimen to a prescribed triangular pulse. The triangular pulse contains half the input energy of the traditionally used square pulse. The loading is adjusted such that the onset of transformation occurs close to the peak input stress. Hence the remaining duration of the pulse is short, so that the microcracks which accompany the transformation have very little time to propagate and cause failure of the specimen. Thus, it is possible to recover a specimen intact for microstructural observations after transformation is complete and extensive damage has been introduced. The above modifications in the testing procedure are important for recovery experiments, as well as for ceramics which undergo extensive cracking under applied loads.

After the experiments, the specimens were ultrasonically cut into long cylinders of 3 mm diameter and then sliced into thin discs of 200 μm thick. They were then ground and polished to 100 μm thick, dimpled to 50 μm, ion-beam thinned to perforation, and then carbon coated to prevent charging under an electron beam. Transmission electron microscopy (TEM) was performed on these specimens in a Phillips CM30 operating at 300 keV.

3. Results and discussion

A typical stress–strain curve for a Y–TZP specimen subjected to loads at room temperature in a Hopkinson bar at a strain rate of 1100 s^{-1} is shown in Fig. 1. It has an initial elastic regime (denoted by OA) with Young's modulus of 210 GPa, followed by an inelastic regime (AB) starting at 3.3 GPa. This inelastic portion represents stress-induced transformation. The transformation continues under increasing stress and saturates after exhaustion of transformable (tetragonal) zirconia. The transformation plasticity which is responsible for the inelastic portion (AB) of the stress–strain curve is generally accompanied by extensive microcracking in magnesia-partially stabilized zirconia and strongly affects the post-transformation behaviour of these ceramics [4, 12, 13]. Once the transformation is complete, the stress–strain curve begins to ascend (BC). The slope of this portion of the curve now depends on the modulus of the transformed material and the density of microcracks and their distribution in the specimen. In the case of Y–TZP, the slope of the stress–strain curve after the saturation of transformation is found to be the same as the initial elastic portion of the curve represented by OA. Hence, it can be concluded that the transformed monoclinic zirconia has the same modulus as that of the initial transformable tetragonal zirconia, and also that in Y–TZP ceramics the amount of microcracking is negligible. Ultrasonic measurements were also performed on these specimens before and after the experiments. The longitudinal wave velocity measurements did not reveal any significant decrease after the test in the Hopkinson bar, indicating that the amount of microcracking is indeed negligible in Y–TZP under uniaxial compressive loading.

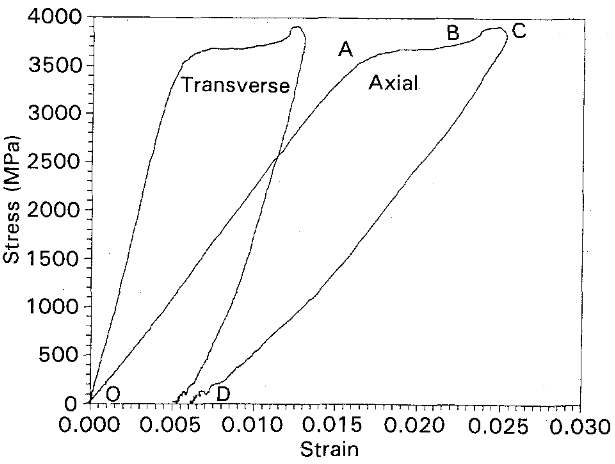

Figure 1 A typical stress–strain curve for Y–TZP in uniaxial compression at a strain rate of 1100 s^{-1}, representing various regimes of constitutive behaviour: OA, elastic; AB, transformation and microcracking; BC, elastic; CD, unloading. Axial strains are compressive, Transverse strains are tensile.

Section CD of the stress–strain curve in Fig. 1 corresponds to unloading. As the load is released, the curve tends to lean towards the origin, representing the strain recovered due to possible reverse transformation. Reverse transformation in zirconia ceramics during unloading is well documented in the literature [14–16]. The stress–strain curve shown here is complete in the sense that it reveals all the dominant mechanisms in the constitutive response of Y–TZP. During loading the response is tri-linear, i.e. initially elastic, then inelastic (transformation plasticity and microcracking), and then finally, elastic again, which represents the response of the transformed zirconia which may be affected by the amount of microcracking. In Mg–PSZ ceramics, the microcracking is so dominant that the slope corresponding to the portion BC of the corresponding curve is significantly affected by these microcracks [4]. Any attempt to load the specimens beyond the stress level shown in the figure resulted in the complete failure of the specimen due to the growth of axial compressive cracks [17–21].

Microscopic analysis was conducted to quantify the amount of microcracking during the transformation plasticity. Scanning electron microscopic observations did not reveal any microcracks on the tested specimens. TEM studies were then conducted to observe

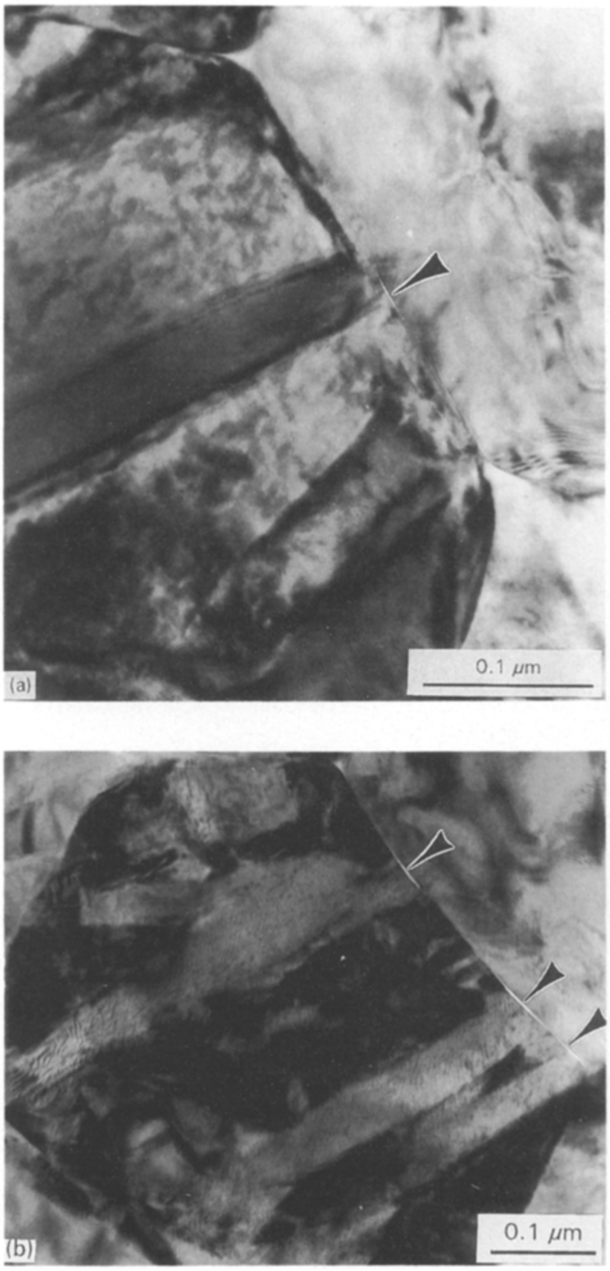

Figure 2 Transmission electron micrographs of microcracks in partially transformed grains at intersections of martensitic laths and grain boundaries.

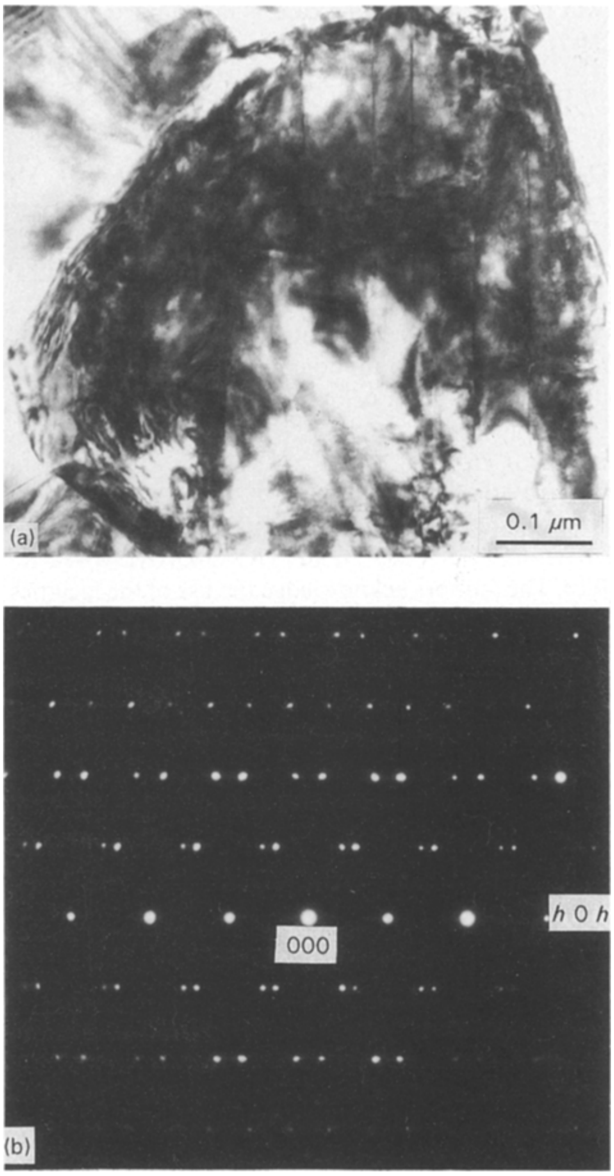

Figure 3 (a) Transmission electron micrograph of a transformed grain and (b) its selected-area diffraction pattern in $\langle 1\,1\,1 \rangle$ zone axis. Unsplit row corresponds to $(2\,0\,2)$ type twin planes.

the microstructural features in transformed grains. Fig. 2 shows transmission electron micrographs of partially transformed grains. The transformed monoclinic regions are revealed as thin strips in each grain. At the intersection of these martensitic laths and the corresponding grain boundary, small cracks are observed. These cracks are several nanometres in size and are few in number. Hence they do not significantly affect the overall modulus of the material after the transformation is complete. This results in a steep rise in the stress–strain curve (BC) with the same modulus as the initial elastic portion (OA). Microcracks at the intersection of the grain boundary and a martensitic lath have also been observed under electron beam irradiation [22, 23]. The transformation in Y–TZP is found to initiate at the grain boundaries [22]. Even though the shear strain associated with the transformation is accommodated by thinning, it is conceivable that the shear strain could be large during the nucleation stage of transformation. This can induce local tension at the grain boundary and initiate microcracks. In fact, it can be noticed in the above micrographs that for every martensitic lath in a grain, there is a corresponding microcrack at the grain boundary. These cracks are very limited in number, and due to their small size the overall macroscopic behaviour is not affected. This microcracking in the partially transformed grains may also prevent the reverse transformation upon unloading [23].

Fig. 3 is a transmission electron micrograph of a completely transformed grain and its selected-area diffraction pattern. The martensitic transformation is shear dominant, and in zirconia the shear strains are accommodated by twinning of the transformed regions. In the above micrograph the transformed grain contains many twinned regions. The boundary between these regions is a twin plane which is revealed as a thin vertical line because the grain is oriented in such a way that the twin planes are parallel to the electron beam direction. The diffraction pattern in this configuration shows one unsplit row corresponding to the twin plane which in this case is determined to be of (2 0 2) type.

4. Conclusions

The dynamic uniaxial stress–strain response of Y–TZP shows a tri-linear behaviour. The post-transformation behaviour is strongly dependent on the amount of microcracking which in the case of Y–TZP is found to be negligible. Microcracks are seen at a very limited number of places where the martensitic laths intersect the grain boundaries. The twin plane in a transformed grain is found to be of (2 0 2) type.

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