# Elastic–Inelastic and Inelastic–Elastic Transitions in ZrO<sub>2</sub> Materials

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## Abstract

It has been found that in four-point bending the first inelastic–elastic transition in TS-grade ceramics starts during cooling to ~50°C, whereas in Y–Ce–PSZ single crystals the first elastic–inelastic transition occurs below 200°C, the second one at about 1000°C, and inelastic–elastic transition occurs at a temperature above  $800^{\circ}$ C. © 1996 Elsevier Science Limited.

The possibility of predicting the mechanical behaviour of ceramics from specific features of their deformation was shown in Refs 1 and 2. This is worthwhile when analysing the results of investigations and when assessing performance under mechanical or thermal loading.

In the present work, tests were performed in four-point bending with the distance between the load application points being 20/40 mm. Specimens were cooled by liquid nitrogen vapours, whereas for heating a high-temperature chamber was used.<sup>3</sup>

The materials studied were Mg–PSZ (Nilcra Ceramics, Australia, TS-grade) ceramics containing 9 mol% MgO, and Y–Ce–PSZ crystals grown by skull melting technique and containing 3 mol%  $Y_2O_3$ , as well as ceria and neodymia technological (about 0.3%) additives.<sup>4</sup> The orientation of crystals specimens was determined by Laue back-reflection which revealed that all their surfaces approximately coincided with the (001) planes.

The analysis of the results was based on brittleness measure  $\chi$ ,<sup>2</sup> which is equal to the ratio of specific elastic energy accumulated in the material by the moment of its fracture to the total specific energy spent in its deformation by the same moment:

$$\chi = \sigma_{\rm u}^2/2 \cdot E \cdot \int_0^{\epsilon_{\rm u}} \sigma d\epsilon ,$$

where  $\sigma_u$  is the ultimate strength, *E* is elasticity modulus,  $\epsilon_u$  is the ultimate strain, and  $\sigma$  are the stresses at the current strain values  $\epsilon$ .

Testing of the first batch of specimens revealed that, with a decrease of the test temperature down to -140 to -150°C and with the test machine crosshead speed, v, being 0.5 mm/min, specimens of this ceramic, which is inelastic at ambient temperature (AT), exhibited a linear dependence between the loading force and deflection (a similar effect was discussed in Refs 5 and 6 for temperatures below -80 to  $-100^{\circ}$ C). While testing the second batch of Mg-PSZ ceramic specimens at v=0.1 mm/min, it was found (Fig. 1) that it exhibited a pronounced decline in inelasticity at about -45 to -55°C and became elastic ( $\chi = 1$ ) at lower temperatures (Fig. 2). Using the SENB methods at temperatures of -40 to -80°C it was found (see Table 1) that the ceramic's fracture toughness increased as compared to that at AT (such fact was drawn attention also in Ref. 5). At AT the fragments of tested specimens were indented using a Vickers indentor and their surface layer was ground. In this way a decrease in the degree of the cooling-induced damage of ceramics was registered (Fig. 3). It should be noted that a similar effect and a decline in the steepness of R-curves, mentioned for the case considered in Refs 5 and 6, are always characteristic of any microfracturing ceramic<sup>2</sup> if an increase in its brittleness measure  $\chi$ is registered (particularly a transition from  $\chi < 1$ to  $\chi = 1$ ).

If the previously published data to the effect that an inelastic-elastic transition occurs at a temperature below  $300^{\circ}$ C and the second elastic-inelastic one at approximately  $1000^{\circ}$ C<sup>7</sup> are considered when analysing the above results, it becomes clear that it is not a simple matter to have a good understanding of real possibilities of such types of ceramics, which change their fracture toughness



Fig. 1. Stress-strain diagrams of Mg-PSZ ceramics.

three times, as regards thermal shock resistance and other features of mechanical behaviour with temperature.<sup>2</sup>

In the present investigation of Y-Ce-PSZ, as in the case of our previous testing under three-point bending, the first elastic-inelastic transition was observed at a temperature of below 200°C and an inelastic-elastic one below 800°C which does not contradict the data of Ref. 8 whose author revealed a nonlinear load versus axial compression relation for Y-PSZ crystals at 700°C for specimens with similar crystallographic orientation. The second elastic-inelastic transition in Y-Ce-PSZ was

Table 1. Fracture toughness of Mg-PSZ ceramics

Temperature (°C)	# Specimens	K <sub>1c</sub> MPa m <sup>1/2</sup>	
		Average	min-max
AT	7	12,1	9,9 – 12,9
-50	3	13,8	13,2 - 14,3
-80	3	13,2	11,2 - 14,7
-140	4	12,0	11,8 - 12,6

observed at about 1000°C (see typical stress-strain diagrams in Fig. 4). In other words, deformation behaviour of crystals (if the problem is considered from engineering standpoint) may turn out to be nearly the same as that of ceramics, although they differ in composition, structure and the discussed mechanisms which govern their mechanical behaviour.

If for crystals it is possible as a first approximation to explain the mechanisms which control elastic-inelastic transitions on the basis of work<sup>8</sup> and for Mg-PSZ ceramics from Refs 5 and 6, though the first is based on the effect of tetragonal-monoclinic transformation and the second on tetragonalorthorhombic transition, the reasons for inelastic-elastic transitions call for additional studies.

As a whole, the results of this work (whose details are to be published) demonstrate the necessity of more extensive investigation into deformation behaviour of advanced ceramics and crystals since without their consideration a reliable evaluation of real serviceability of the material types considered is not always possible.



Fig. 2. Brittleness measure-temperature diagram of Mg-PSZ ceramics (every point is average of 1-4 experimental results).



Fig. 3. Vickers impression of Mg-PSZ ceramics after grinding off its surface at 500 N load. Temperatures: (a) AT, (b) -73 and (c)  $-150^{\circ}$ C.



Fig. 4. Stress-strain diagrams of Y-Ce-PSZ crystals.

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