A STUDY ON THE MECHANICAL BEHAVIOUR OF PSZ (5%WT. YTTRIA) DURING BENDING TESTS COMPARING WEIBULL STATISTICAL MODEL AND ACOUSTIC EMISSION RESULTS

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ABSTRACT

One of most promising possibilities to increase toughness of advanced ceramics consists in stabilizing a phase within the material by addition of suitable amounts of a second constituent. This is what happens in zirconia in which we obtain a more relevant toughness due to cubic phase stabilization by adding yttria. In our tests we deal with a PSZ (partially stabilized zirconia) that contains 5% yttria. A wide problem is therefore to evaluate in a reliable way that the toughness of the specimens doesn't spread upon a large field, according to the use we aim to do with our material. In our work a large set of specimens was tested, carrying out four points bending tests up to ultimate load in a very short time (15 sec.), so to ponder particularly with main crack propagation and phenomena linked with failure. During the whole test acoustic emission was monitored, in order to evaluate the energy released during critical crack propagation and duration of failure phenomena for each specimen. M.O.R. data were then put in correlation not only with deformation data, leading to Young's modulus determination, but also in a two parameters Weibull distribution.

Acoustic emission variables (energy, counts, duration etc.) analysis allows a quantitative evaluation of toughness in our material, which complete efficaciously mechanical and statistical features. Further studies can be carried out on other typical brittle materials with acoustic emission monitoring, furnishing an alternative way to evaluate whether an appreciable decreasing of brittleness has been obtained with phase stabilization or other processes.

INTRODUCTION

One of major limitations to the diffusion of advanced ceramic materials for structural elements comes from their origin from high melting point oxides, nitrides and carbides, that are inherently brittle in addition with the presence in them of a variety of surface and volume defects. Thus the material presents a low tenacity, that induces during mechanical testing a linear behaviour up to ultimate load.

By adding a stabilizing oxide (1), such as magnesia or yttria to pure zirconia, it is possible to retain it in its cubic grain structure even at room temperature. A much stronger structure is supplied however partially by stabilized zirconia that contains a lower concentration of the stabilizing oxide and presents, during cooling down from sintering temperature, the triggering of tetragonal grains inside the cubic grains. The promising effects of this phase transformation are indeed very variable with the single situation of the specimen. Fracture behaviour is often unpredictable and M. 0 .R. values are typically rather dispersed, even for materials coming from the same source and subjected to the same manufacturing process (2); this leaves of course a great deal of doubts for as concern mechanical validity of a ceramic materials based-structure.

Such characteristics enhance the capability of designing ceramic components starting from a probabilistic point of view rather than from criteria connected with the maximum value of allowed stresses. From these considerations comes the requirement of elaborating probabilistic models, which can supply a reasonable evaluation of factors that lead us from fracture mechanics up to the real behaviour of such materials. Most models nowadays disposable are based on the "weakest ring" theory proposed by W.Weibull in 1939 (3,4), which supposes the structure fails with its weakest part failure.

This theory presents however some limitations (5), considering the independence of all failure events when loading a volume V with uniaxial tension S 1 .We suppose that density of the defects is equal to g(S)dS per volume with a critical tension between S and S+dS. In a ceramic material we can put G(S) =(S/So)m and observe that in the case of multi axial tension problems are mainly two:

-to calculate m and So values

-to evaluate probabilities of multiaxial failure.

In fact, limitations of Weibull approach, although improved by Freudenthal e Barnett (6), are connected to the difficulty to relate efficaciously different ways of crack opening and propagation. In a structural ceramic we can discern surface and volume defects: the first ones are easily detectable also before bending test, especially using optical microscopy, the other are inclusions and porosities that can be observed only after failure.

Acoustic emission monitoring allows instead the detection of defects that, apart from their morphology, result in a criticality condition for structural integrity of the material, giving the opportunity to trace the dynamic development of the cracks and to adequately control the surface properties of technical ceramics (7).

The effectiveness of such an approach, relating probabilistic behaviour with energetical considerations supported by acoustic emission can lead finally to a sure discrimination of brittleness level of each subject in a population of ceramic material specimens.

MATERIALS AND METHODS

Bending tests were performed on partially stabilized zirconia with 5% yttria. A number of thirty specimens by TEMA V coming from different batches and realized from Tosoh TZ-3YB powders, was tested, using an universal testing machine with a 2 kN-load cell and a four-points bending support. Load application speed was 2.5 mm/min so to carry out tests in a very short time (approx. 15 sec. from first load application and material collapse).

Acoustic emission system was LOCAN -AT by PAC, which is an ICC (Independent Channel Controller) based system. This means that the acoustical signals from the loaded structure are converted into electrical signals by the sensors, amplified to usable voltage levels by the preamplifiers and measured in two -channel computerized modules (ICC). We used two 1220A preamplifiers with an internal band-pass filter (100-~:)O kHz). Acquisition was carried out by two PZT R15 type- piezoelectric sensors with a peak frequency of 150 kHz, held in place by silicon grease. Two square little plates placed at both extremities of stranded copper wires were used as waveguide, because the specimen was otherwise not accessible by AE sensors. Two further guard sensors were placed on bending support, so to avoid to detect signals due to background noise coming from universal testing machine.

Specimen dimensions (mm50x3x5) respond to standards not exactly suitable for use of Acoustic Emission (AE), thus the use of waveguides was required so to reach an acceptable connection from sensors to specimen. This of course reduces part of released mechanical energy detected as acoustic emission, but we can imagine that percentage of lost AE is equal for all specimen. AE setup was oriented to the recognition of mere damage phenomena connected with main crack propagation, so to assess the capacity of this monitoring technique to detect the defects that bring this material to breakdown.

With this goal, signal definition times: PDT (Peak Definition Time), HDT (Hit Definition Time) and HL T (Hit Lockout Time) were chosen much longer than suggested values respectively 500, 2000 and 2000 μs, so to lock in few complex AE events the whole acoustical activity related with failure and simplify statistical analysis. A 40 dB-floating, i.e. variable with background noise, was finally chosen so to allow a relevant filtering of continuous noise signals carried on by waveguides.

RESULTS AND DISCUSSION

The free potential energy of the elastic deformation in the grains by the high level of thermal anisotropy in the three axes is equal to or larger than the energy necessary for the formation of new fractive surfaces, as showed by the following balance of the energies:

$V_{cum}=V_o-V_{ee}+V_{ne}$

where V cum is the cumulative energy of the microstructure, Vo is the energy of a body without microcracks, Vee the free elastic energy and Vne the fracture surface energy.

The higher values of the cumulative AE energy can be related to the availability of a large number of micro and macrocracks in the ceramic material, formed during its cooling, because of the high levels of thermal orthotropy. AE waves can originate at the large number of microcracks which have a cumulative energy proportional to their number (8). This means the existence of a correlation between ultimate load and released energy i.e., AE cumulative energy that has not enough significance, however, to explain alone the material behaviour in our tests.

Beyond to this order of considerations, the more or less relevant presence of defects, i.e. in this material mostly porosities can influence the propagation of crack leading to failure. A lower concentration of greater porosities causes in effect a slower diffusion of fatal crack through the material and vice versa a higher concentration of little porosities allows the crack to travel over the defects with a greater speed. This of course has consequences on the failure time (usually a very short time for a ceramic material), i.e. the time from the first onset of damage to the final large energy burst that signifies material breakdown.

Let's see now more in detail mechanical, probabilistic and acoustic emission (AE) data.

Weibull modulus value obtained for our specimens was:

$m=(\ln(\ln(q+l))-\ln(\ln((q+l)/q))/\ln(\sigma max/\sigma min) = 4.789$

where σmax= maximum ultimate load

σmin = minimum ultimate load

q= number of tested specimens

In fig. 1 the interpolation curve is described, leading to Weibull plot of PSZ strength data.

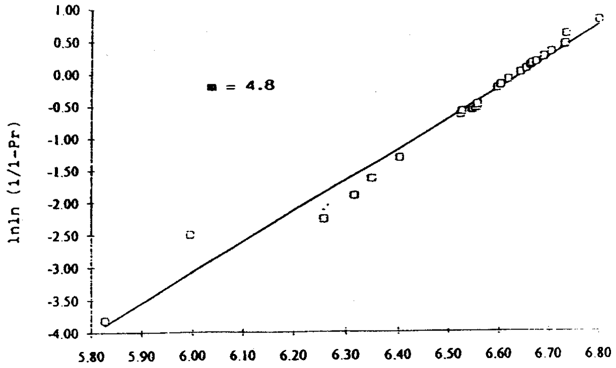

fig. 1 Weibull distribution

Table 1 depicts ultimate load data in close comparison with acoustic emission results, for as regards cumulative energy, time of duration for failure, obtained by considering the period of continuous acoustic emission immediately before collapse. The last three columns show respectively the ratio between the two previous variables, the probability of failure and the maximum voltage reached during failure, i.e. the stronger burst of acoustic emission due to mechanical collapse.

Table1: Specimen	MOR	AE cum.energy	Failure time	Ce/Ft	% Pf	RMS (Volts)
160	339.5	46116	0.127	363118.1	8.19	2.45
4	401.3	90756	0.489	185595.1	9.63	2.75
179	521.6	102684	0.2	513420	17.07	3.6
62	552.8	59121	0.272	217366.6	28.65	2.85
219	571.0	82181	0.069	1191029	29.25	4.1
285	602.5	74612	0.39	191312	39.66	2.8
253	679.6	101883	0.356	286188.2	40.39	2.95
69	681.7	66360	0.139	477410.1	41.99	2.8
36	695.0	77828	0.228	341350.9	42.45	4.35
139	696.0	51059	0.213	239713.6	42.88	2.35
151	699.1	107714	0.446	241511.2	43.55	3.1
132	701.6	84015	0.29	289706.9	43.81	3
229	701.8	96387	0.424	227327.8	45.24	3.5
79	702.5	39581	0.292	135551.4	45.18	2.6
205	730.5	104957	0.2	524785	54.15	4.7
130	735.0	60863	0.122	498877	55.7	3.1
266	736.6	97797	0.253	386549.4	56.22	4.2
191	747.8	122648	0.183	670207.7	59.28	3
47	766.4	113767	0.394	288748.7	62.99	3.65
278	775.4	110117	0.468	235292.7	65.48	3.25
233	782.2	83592	0.153	546352.9	67.28	3.4
234	784.0	123912	0.465	266477.4	68.27	5.5
6	790.9	73896	0.291	253938.1	69.05	3.1
330	803.3	90339	0.082	1101695	71.73	2.45
201	803.4	81762	0.32	255506.3	72	3
64	815.6	136137	0.408	333669.1	74.65	3
83	837.7	143911	0.387	371863	77.66	-[1]
204	838.2	34295	0.09	381055.6	78.41	2.55
269	839.5	114183	0.45	253740	80.57	3.05
300	896.7	107613	0.444	242371.6	87.13	4.6

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[1] In this case it was not possible to recognise acoustic emission events associated to main crack propagation

The above described situation, though it presents some non ideal aspects, can be regarded as a normal feature when compared with an anomalous behaviour (strong decrease of M.O.R. value) like we can observe in specimen n.253 (Fig. 4-5): in this case, failure initiation is generated by a superficial crack induced by machining. So the M.O.R. is relatively low and we suppose even a scarce tenacity. A deeper observation shows instead that the crack proceeds during the initial part of its propagation following an extremely irregular path with both porosities and scaling off: after a 0.05

distance, however, there is a sudden collapse. This seems to be the effect of an unexpected deal of tenacization: like AE confirms, the maximum of energy is stored within the structure, nevertheless the already mentioned presence of a dramatic fabrication defect, and released quickly, inducing the breakdown. Here, only the synergy between AE and fractography can discover the presence of the effect of yttria, that a mere bending test is not able to detect.

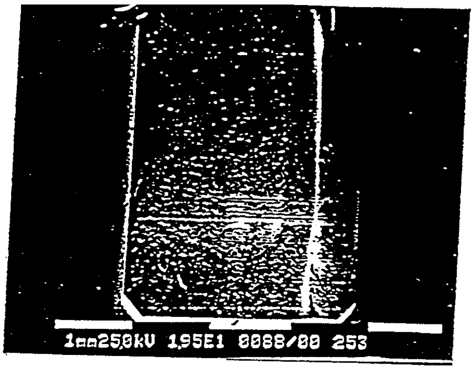

Fig. 4 Specimen no.253

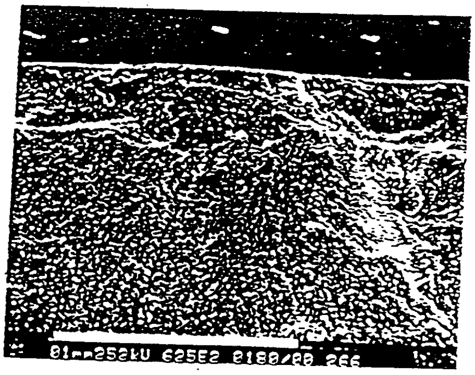

Fig. 5 Specimen no. 266

Another superficial crack is the cause of failure in specimen no.4 (fig.6-7); the main difference lays on the greater extension of fracture surface, that influences negatively M.O.R. value, and is due to diffuse porosity that leads to a real chipping of the material. To this situation is ascribed the association of a very low ultimate load, a quite strong amount of released energy and a not relevant RMS voltage.

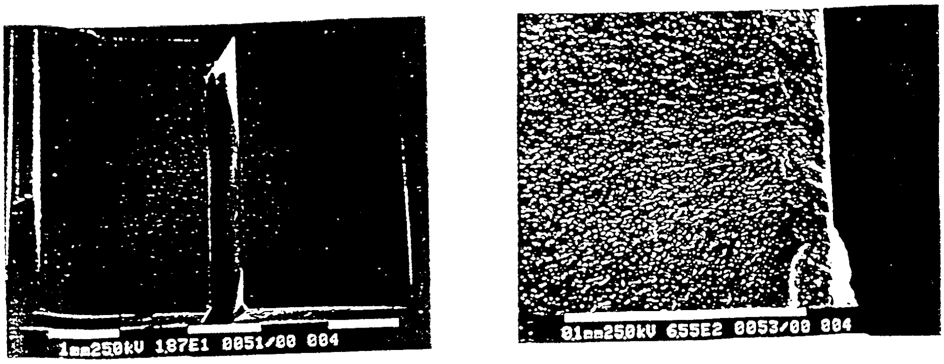

Fig. 6 and 7 Specimen no. 4

These considerations have of course a significance closely connected to the preliminary character of this study, that aims especially to focus our attention on the capacity of arriving to ceramic properties prediction by starting with a simple measurement carried out on released energy, duration of release and presence (or not) of a sudden stress wave emission, comparable with a burst.

CONCLUSIONS

The attempt to relate the results obtained from a typical NDT technique, such as acoustic emission, here used in order to predict the level of brittleness of a specimen by the evaluation of mechanical energy released out of main crack propagation, has sorted interesting results, showing the possibility to proceed to other tests so to quantify with mathematical correlations the relation between M.O.R., brittleness level and released energy.
Surely, a larger database is needed, so that the acoustic emission system can be much more oriented in the sense of a sharper detection of phenomena involved in ceramic materials sudden fracture.

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