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Effect of surface treatments on strength of PZT polycrystalline ceramics

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In bending strength determinations of ceramics, the fracture usually initiates from the tensile surface or the edges. The edge effect could be minimized by carefully rounding off the edges of specimens subjected to four-point bending. The effect of tensile surface polishing, polishing and rounding off the edges, and the acid treatment of the polished surface on the strength of PZT polycrystalline ceramics was determined along with the failure mechanism.

1. Introduction

In the ceramic industry surface finishing of component parts is very important, particularly from the point of view of strength. A wide range of surface finishes can be generated by grinding, abrading and polishing. Grinding and abrading may produce extrinsic surface cracks, whereas polishing may remove some of these cracks. In addition, there may be some intrinsic flaws in the polycrystalline ceramics. The strength of ceramics is sensitive to these types of flaws, and their removal should improve the strength.

These extrinsic or intrinsic flaws in polycrystalline ceramic materials are related to the microstructure, particularly to the grain size, second phase and the grain boundary structure. The classical strength–grain size relationship is shown in Fig. 1. Region I, which follows the Griffith–Orowan strength relation, is represented by $\sigma_f = K_1 d^{1/2}$ where σ_f is the strength, K_1 is a constant, and d is the average grain size. In this region, previous experimental results suggest that for some polycrystalline ceramic materials the largest grain size is the most severe flaw leading to failure. The failure mechanism consists of propagation of these existing flaws. Region II, which follows the Hall–Petch strength relation, is represented by $\sigma_f = \sigma_0 + K_2 d^{1/2}$. Here, the generated flaw mechanism is based on the microplastic flow nucleated fracture due to dislocation processes. Application of this equation to fine-grained ceramic materials assumes that the presence of microplastic flow or dislocation activity leads to crack nucleation. A combination of both relations was observed by Carniglia [1] for polycrystalline Al_2O_3. Some ceramic materials exhibit only the Griffith–Orowan relation, and some only the Hall–Petch relation [2], but in most cases a combination of the two relations is observed.

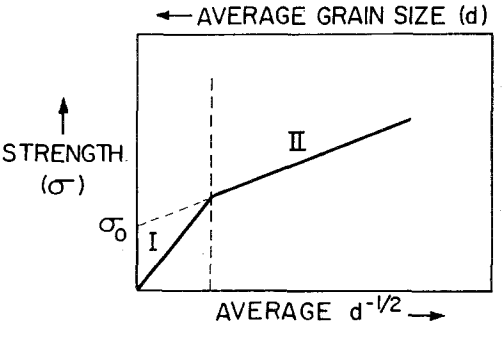

Figure 1 Strength–grain size relationship.

Recently Bradt and Tressler [3] observed that surface finish has a definite effect on strength of

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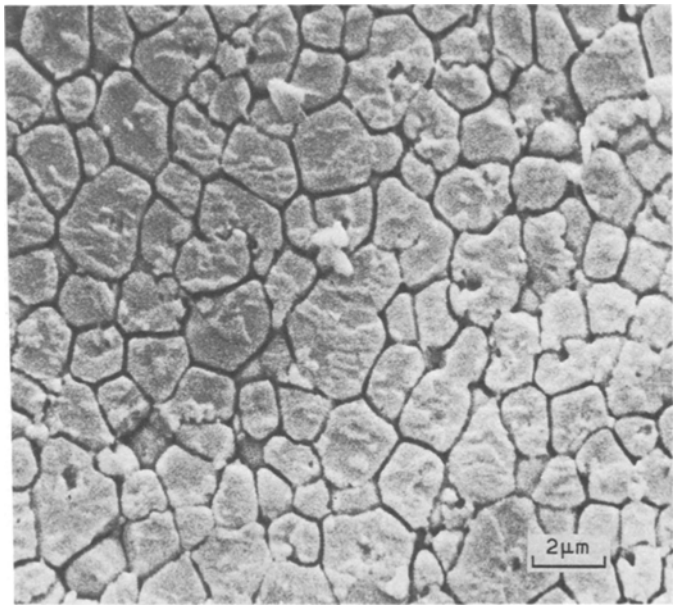

Figure 2 Typical microstructure of dense PZT ceramic specimen used.

fine-grained Al_2O_3 and SiC ceramics whereas surface finish has no apparent effect on strength of coarse-grained materials. In this case, the intrinsic microstructural flaw was operative in Region I, and machining operations controlled the strength in Region II instead of the microplastic flow processes. As the surface finish by the diamond grit size became finer, they found that the strength increased considerably.

The purpose of this study was to determine the effect of various surface treatments on strength of a highly dense polycrystalline lead zirconate–titanate (PZT) ceramic, and its failure mechanism.

2. Experimental procedure

The material used was stoichiometric PZT. The samples were fine grained in which the average grain size was controlled by doping with one mole per cent niobium oxide, Fig. 2. The samples were fabricated with 5.5 wt% excess PbO and sintered at 1200° C for 8 h in one atmosphere pressure of oxygen. The excess PbO enhaces the sintering process and densities over 99% of the theoretical value were obtained. In sintering the specimens were embedded in a packing powder of $PbZrO_3$ + 5 wt% ZrO_2 which is used to absorb excess PbO and control the PbO content.

A wide range of surface treatments was given to the bend specimens after slicing from the sintered pellets by using a precision diamond blade. A set of eight specimens were used for each series of (1) as-cut, (2) as-cut + rounded edge (by using a rotating 1 μm diamond wheel), (3) polished tensile surface, (4) polished tensile surface + rounded edge, and (5) polished tensile surface + rounded edge + etching (HF/HCl solution). All the specimens (2.54 cm x 0.3 cm x 0.2 cm) were tested in a four-point bending machine with an overall span of 1.9 cm. The fracture origin was detected by scanning electron microscope (SEM) examination.

3. Results and discussion

Table I shows the strength values of the different surface treated specimens. It is clear that the strength variation with respect to different surface treatments is insignificant. If the polishing removed surface cracks produced during machining, the strength should have increased. In this case, however, there is no change in strength even after

TABLE I Effect of surface treatments on strength of PZT ceramics

Surface treatments	Strength (MN m^{-2})
As-cut	82.70 ± 5.5
As-cut + rounded edges	85.65 ± 4.4
Polished tensile surface	79.30 ± 9.1
Polished tensile surface + rounded edges	79.68 ± 7.8
Polished tensile surface + rounded edges + etching	79.92 ± 4.1

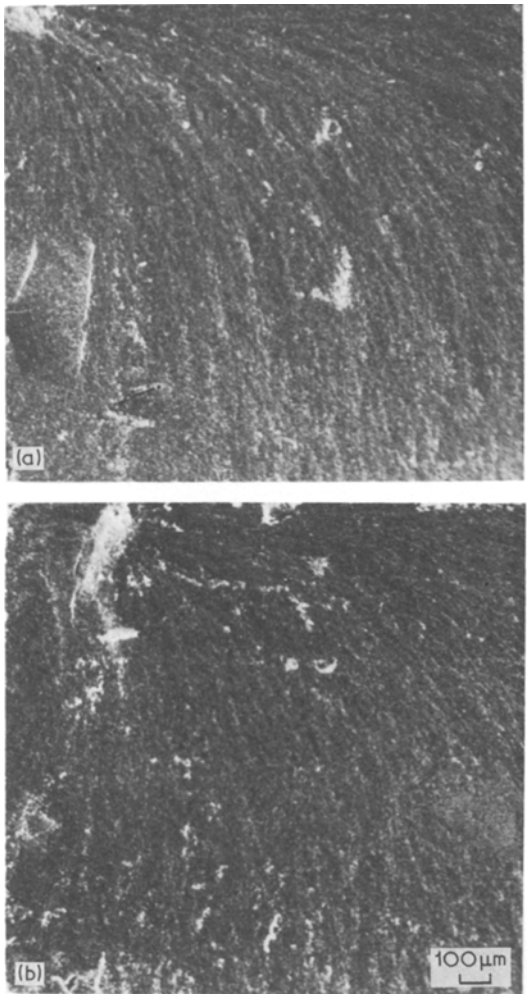

Figure 3 Fracture surfaces of as-cut PZT specimens.

removal of the extrinsic flaws indicating that the intrinsic flaws are more severe than the introduced surface flaws.

SEM examination indicated that intergranular fracture was the primary fracture mode with some transgranular fracture. By tracing back the river pattern, the fracture origin was detected. In as-cut specimens, the fracture initiation was at the edges of the specimen (Fig. 3a) and/or at the width of the bend specimen (Fig. 3b). Rounding off the edges of the specimen minimized the edge failure (Fig. 4a). In the cases of the polished tensile surface, polished tensile surface + rounded edge, and polished tensile surface + rounded edge + etched specimens, the failure occurred at or near the tensile surface (Figs. 4b, c and d, respectively). It is interesting to note that the crack initiation morphology in most cases is very similar and the crack size is very large (around 200 to 300 μm in length or in depth). This behaviour indicates that the failure mechanism is the same for all specimens irrespective of surface treatments.

It has been observed that materials (e.g. ZrO_2, $MgTi_2O_5$) with low elastic modulus and high internal friction contain many microcracks [4]. Previous experimental results on the PZT ceramic showed [5] that it has a low elastic modulus, $E = 6.89 \times 10^4\ MN\,m^{-2}$, and a high internal friction. It is then expected that it had many inherent microcracks. Two reasons for the presence of microcracks are proposed. The first cause could be the presence of some excess PbO along grain boundaries if it is not completely evaporated into the packing powder during sintering. The remaining PbO during cooling from the sintering temperature remains at the grain boundaries and it might be the source of microcracks. Because of the thermal expansion anisotropy and differences between the PZT and PbO phases, boundary stresses can develop which produce the microcracks. Secondly, in the presence of excess PbO, rapid and more complete densification could occur in a localized region, where material shrinks more than in other regions developing sufficient stress to form microcracks. In bending, as the stress level increases, these microcracks are linked up to form a large crack that causes failure.

To support the existence of microcracks in the presence of a small amount of PbO, a simple experiment was conducted. A small amount of PbO was placed on top of a sintered PZT piece and heated at 950° C (m.p. of PbO is 888° C) for one hour in air. After cooling, the specimens on examination with the SEM indicated large cracks as shown in Figs. 5a and b. The liquid PbO penetrates through grain boundaries or through the microcracks, and during cooling, because of the thermal expansion differences between the phases, produces large cracks. Similar experimental evidence of microcrack formation in SiC and B compacts to which B was added as a sintering aid was observed by Evans [6].

From the size of the fracture initiating flaw, the fracture toughness, K_{IC}, was calculated by using the Griffith–Irwin equation [7];

$$a = \frac{Z}{Y}\left(\frac{K_{IC}}{\sigma_f}\right)^2 \qquad (1)$$

where σ_f is the fracture stress, a is the flaw depth

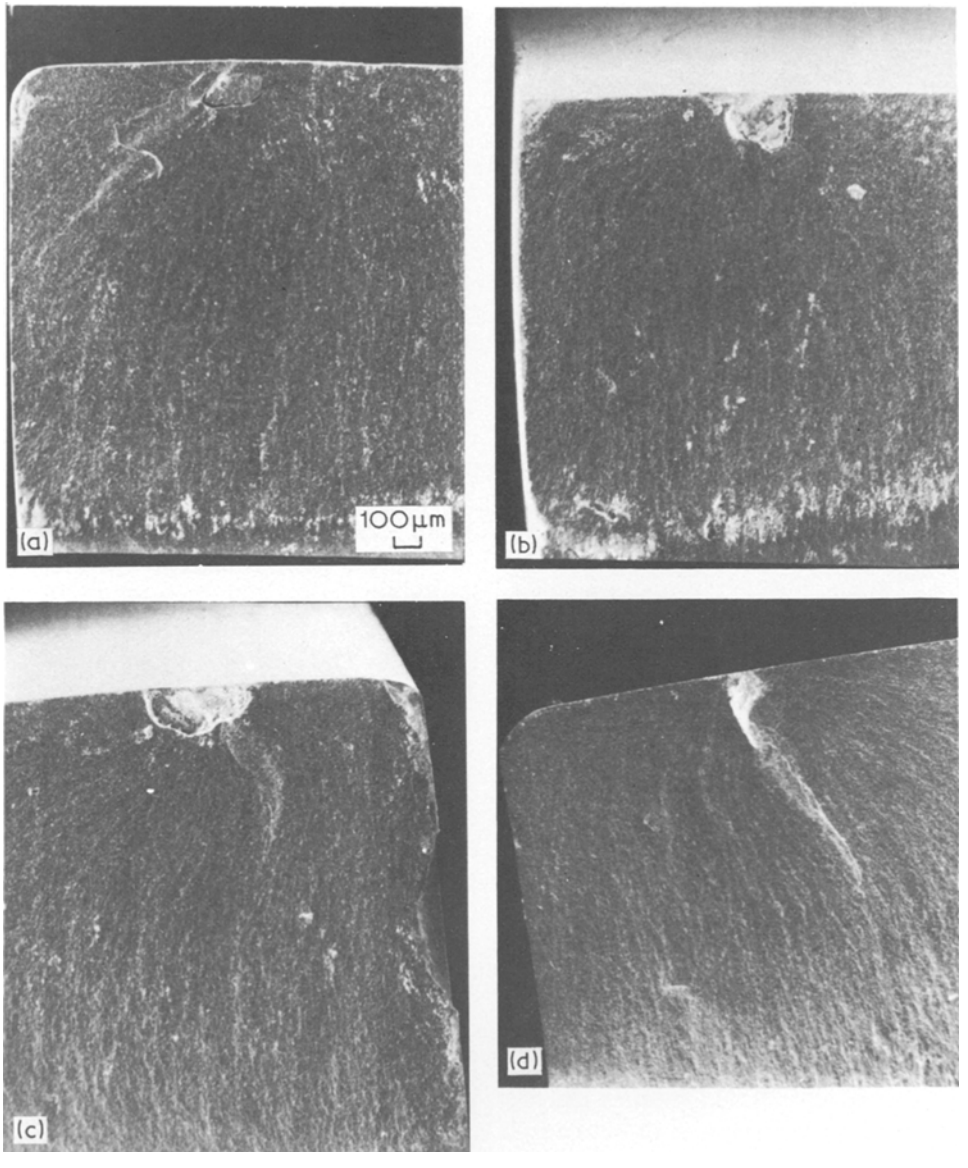

Figure 4 Fracture surfaces of specimens: (a) as-cut + rounded edges, (b) polished tensile surface, (c) polished tensile surface + rounded edges, and (d) polished tensile surface, rounded edges + etched.

in case of surface flaw, Z is the flaw shape parameter, and Y is a geometrical constant. The "a" value was measured from the flaw configuration of Figs. 4a, b and c, and Z and Y values were taken from [8]. Using the σ_f values from Table I, the K_{IC} value was calculated from Equation 1 and found to be approximately $1.40\,MN\,m^{-3/2}$. The experimentally measured [9] K_{IC} value was $1.60\,MN\,m^{-3/2}$ for this ceramic. Therefore, using the flaw size measurement the calculated fracture toughness matches the experimental value well. The uniformity of flaw configuration is in accordance with the proposed mechanism for failure of PZT ceramics.

4. Conclusion

Surface finish could improve the strength of some polycrystalline ceramics, whose strength is controlled by extrinsic surface flaws which can be removed by surface polishing or other treatments. In the case of polycrystalline fine-grained PZT ceramics with some excess PbO as a second phase, surface treatments of bend specimens do not affect the strength because the extrinsic flaws generated

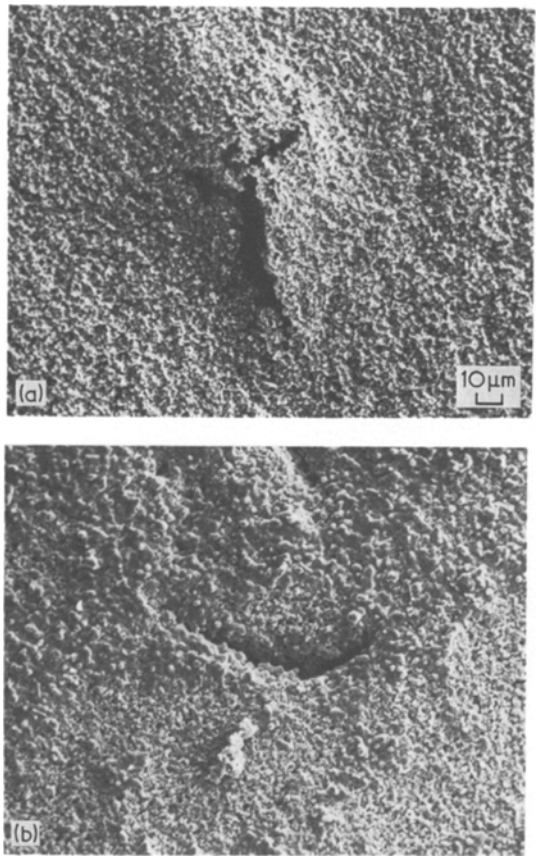

Figure 5 Large cracks in the presence of PbO in PZT specimens.

by machining are less sensitive than the intrinsic microcracks produced by the presence of a small amount of PbO phase.

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