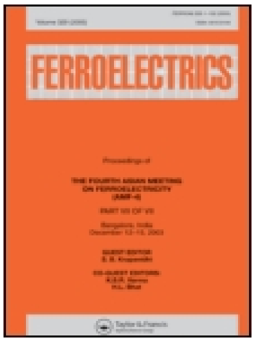

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FRACTURE OF FERROELECTRIC CERAMICS

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Abstract This paper surveys the temperature, microstructural and environmental variations of the fracture properties of ferroelectric ceramics. Earlier work shows that fracture toughness decreases on heating through the Curie temperature. There is also anomalous behavior in the strength at small crack sizes, indicative of a grain size effect. Further, the strength properties are known to be adversely affected by the presence of water in the atmosphere. Data from recent indentation studies on barium titanate are used to investigate these phenomena.

INTRODUCTION

Ferroelectric ceramics such as barium titanate ($BaTiO_3$) and lead zirconate titanate (PZT) are susceptible to brittle failure from small processing and handling flaws. To characterize the strength properties of brittle materials we need to understand the nature of all forces acting on the flaws. We shall address these issues in terms of data from indentation fracture studies.

INDENTATION TESTING

Reproducibility in strength data may be optimized by introducing controlled indentation flaws into prospective test specimens.[1] The driving force on such flaws comes from two main sources, the externally applied stress field and a local residual field about the elastic/plastic impression. Under equilibrium fracture conditions the strength is given by

$$\sigma = AK_c^{4/3}/P^{1/3} \qquad (1)$$

where A is a dimensionless constant, K_c is the material toughness and P is the original indentation load. Departures from the predicted variation of σ on P in Eq. (1) could be effected either by a third, unaccounted driving force on the indentation flaw, or by a systematic dependence of K_c on crack size.

Accordingly, controlled flaw tests were run on $BaTiO_3$. Indentations were made on annealed bar specimens with a Vickers diamond pyramid, in air, at prescribed loads. Failure was produced in four-point flexure in a controlled environment. At low loads, failure tended to initiate from natural rather than introduced flaws, thereby imposing a lower limit on the data range.

TEMPERATURE EFFECTS

Strength variations were followed as a function of temperature for a $BaTiO_3$ material of nominal grain size 7 μm. The indentation load for these tests was fixed at 30 N, so that the cracks produced were well in excess of the grain size. The flaws were introduced at room temperature, and the strength tests were run in a heated oil bath. Figure 1 shows the results. There is a monotonic decrease in strength to the vicinity of the Curie temperature, T_c, then an apparent levelling out. The falloff between 25°C and 150°C corresponds to a reduction of $\approx$40% in K_c (see Eq. 1), consistent with trends noted previously in monocrystalline $BaTiO_3$.[2,3] Specimens tested after cycling through T_c showed the same strengths, within experimental scatter, as those heated directly from room temperature.

In view of this reversibility, it can be concluded that the results in Fig. 1 reflect intrinsic thermal effects in the toughness parameter. The reported appearance of domain wall

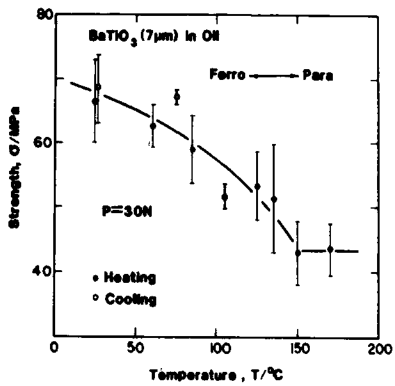

FIGURE 1 Strength as function of temperature.

markings on fracture surfaces produced below T_c suggests that crack/twin interactions could account for the trends observed here[2]; the falloff of K_c with temperature would then be attributable to the thermally activated annihilation of domain walls as the transition is approached.[4]

Analogous thermal effects have been reported for PZT.[5,6]

MICROSTRUCTURAL EFFECTS

The influence of microstructure on strength properties was investigated by systematically reducing the controlled-flaw size, via the indentation load. Data are plotted in Fig. 2 for the 7 μm (Fig. 1) and a 1 μm $BaTiO_3$. Several points may be noted:

(i) At 150°C the data fit the dependence predicted by Eq. (1) over the entire load range, for both materials. Moreover, the representative straight lines are indistinguishable, indicating that K_c is the same in both cases. This grain-size invariance of toughness above the Curie point is consistent with data on other cubic ceramics.[7]

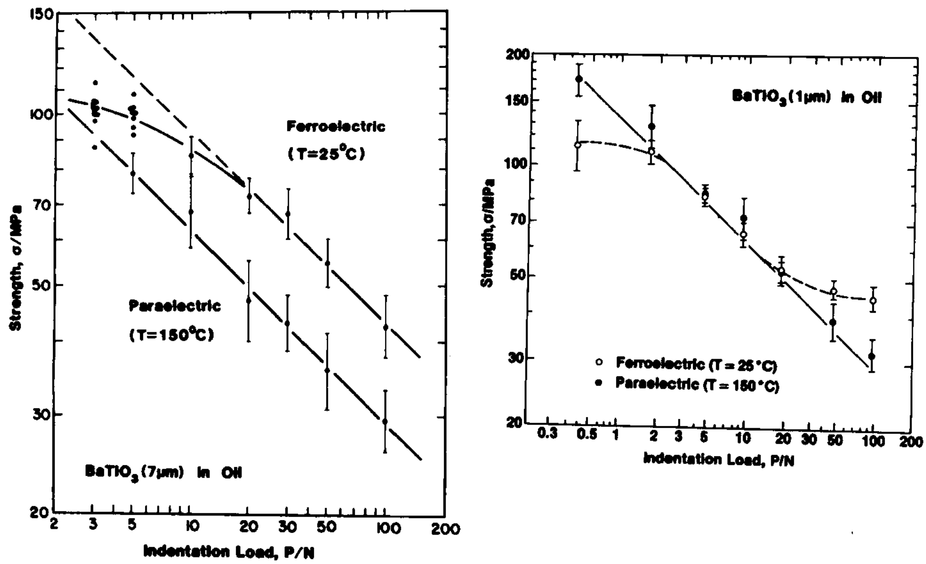

FIGURE 2 Strength as function of load.

(ii) At 25°C the data are well behaved over only part of the load range. In this well-behaved region there is overlap with the 150°C data for the 1 μm material, whereas for the 7 μm material the temperature dependence noted in the strength data of Fig. 1 is apparent. These results imply a grain-size dependence in K_c below the Curie point. A systematic study of this dependence in $BaTiO_3$ materials over a grain-size range 1-150 μm shows that K_c can vary by as much as a factor of two.[2]

(iii) At low loads the 25°C data for both materials drop off below the theoretical line. As alluded earlier, such a departure could be due either to the presence of an additional driving force on the cracks or to a tendency for K_c to increase in a systematic manner as the cracks intersect a greater number of grains. Of these two possibilities, only the first appears to be capable of accounting for the reversion to ideal behavior at the elevated temperature, since the grain geometry remains invariant on traversing the Curie point. Thus it has been suggested that the room temperature data are consistent with the existence of local internal stresses associated with a non-cubic (tetragonal) polycrystal.[2,4] These stresses are felt more strongly as the cracks become smaller, approaching the scale of the microstructure; in this context, it is noted in Fig. 2 that the deviation occurs at a lower level of P in the 1 μm material. (iv) At high loads the 25°C data for the 1 μm material again deviates from ideal behavior. The cause of this anomaly is not understood at present, although analogous behavior has recently been reported in a La-doped lead titanate.[8]

ENVIRONMENTAL EFFECTS

The access of water to crack tips in brittle ceramics can cause subcritical growth, i.e. growth at stress levels less than that needed to maintain the equilibrium configuration implied in Eq. (1). The rate of subcritical crack growth increases dramatically with stress intensity, approximately to some power N, where N >> 1 typically. Accordingly, specimens loaded at low stressing rates are subject to more extensive subcritical growth, and hence to a greater degradation in strength. For specimens with indentation flaws, the "fatigue susceptibility" parameter N can be measured directly from the (inverse) slope of a strength vs stressing rate plot.[9]

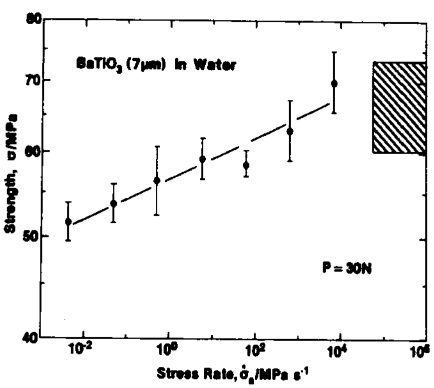

FIGURE 3 Strength as function of stressing rate.

Accordingly, such a plot is shown in Fig. 3 for the 7 μm $BaTiO_3$ tested in water at 25°C. The strength indeed falls off significantly as the stressing rate decreases (or, alternatively, as the time to failure increases). The value N = 67 obtained from these data is considerably higher than that for soda-lime glass (N = 18, representing one of the most susceptible of all brittle materials), but is within the range found for a broad spectrum of ferroelectric ceramics. Small changes in composition, particularly in the form of grain boundary impurities, could well give rise to deleterious decreases in the N parameter.

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