The Korean Journal of Ceramics, 5[4] 386-389 (1999)

Reliability Improvement of Titania Ceramics with Surface Flaw Through High Voltage Screening

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(Received September 23, 1998)

Effect of high voltage screening was examined on mechanical strength of titania ceramics with two different surface roughness. Roughly finished sample showed degraded mechanical strength meaning that the introduced flaw played the role of starting point of mechanical fracture. On such sample, electrically weak parts were eliminated by applying a screening field. Mechanical strength measurement on survived parts revealed that after screening the Weibull plots bended to become a convex curve while plots at high strength region were almost the same. This result means that relatively low mechanical strength parts were eliminated by the electrical method As a result the Weibull modulus calculated from all the data increased, demonstrating the effect of high voltage screening on titania ceramics containing fracture controlling surface flaws Roles of the surface flaw such as a common weak spot for both failures are disscussed in relation to the electric field concentration similar to that of mechanical stress.

Key Words: Screening weibull wodulus, Dielectric breakdown, Surface flaw

I. Introduction

Ceramic materials, behave as brittle materials, have considerable scatter in their mechanical strengths due to the existence of defects. Therefore, in the case of using ceramic parts as structural materials, it is necessary to narrow the strength distribution by eliminating relatively low strength parts. To improve the reliability of ceramic materials, mechanical screening or proof test is mainly adopted. However, this method is said to have demerits to require a lot of time and to induce crack growth under the loading.[1-2] Hence, an alternative method to stress screening had been required.

Dielectric breakdown strength of ceramic insulation materials is determined by microstructures such as pores and surface flaws similar to machanical strength. One of the authors has already reported that mechanical strength distribution and dielectric strength distributions are substantially the same.[3-5] In accordance with the results, analogy of mechanical strength and dielectric strength could be derived, which can be adopted to improve the reliability of mechanical strength by an electrical method.

Based on this scheme, we examined the possibility of electric screening (named high voltage screening) using TiO_2 ceramics.[6-7] As a result low mechanical strength ceramic parts were selectively eliminated by the high voltage screening instead of applying screening stress. However, decisive factors to lead such effect remained unproved. The present study concerned with surface flaws as possibly the common weak spot for both mechanical and dielectric failures, and the influence of surface flaws on mechanical distribution after high voltage screening was examined.

II. Experimental Procedure

1. Sample Preparation

Titanium dioxide ceramics were employed. Titanium dioxide powder (Kojundo Chemical Co. Ltd, Japan, rutile phase, purity 99.99%) was used as starting material. Powder compacts were first formed by uniaxial pressing (30 MPa for 60 s) and subsequently fabricated by hydrostatic pressing (200 MPa for 90 s). The compact bodies were sintered at 1450 °C for 4 h in air. The resultant sintering bodies had relative densities around 98.5%. The bodies were cut into rectanglar bars of $13 \times 3894 \times 0.3$ mm^3 with a precision cutting machine (Maruto Co.Ltd, Japan, MC-603).

Cut ends of the specimens were comparable or smoother than the surface finished with the 800-grit abrasive paper. Coarse surface specimens were processed by grinding their surfaces by 400-grit abrasive paper.

Two kinds of test pieces, coarse and fine, were obtained. Half of both coarse and fine test pieces were subjected to mechanical and dielectric strengths measurement. The other half were used for high voltage screening test. This experimental procedure is schematically illustrated in Fig. 1.

2. Strength measurement and High voltage screening

For dielectric strength measurement, silver electrodes with a diameter of 2.5 mm were attached on both sides of the test pieces. Breakdown test were carried out by applying d.c. voltage at the increasing rate of 50V/s. The elec-

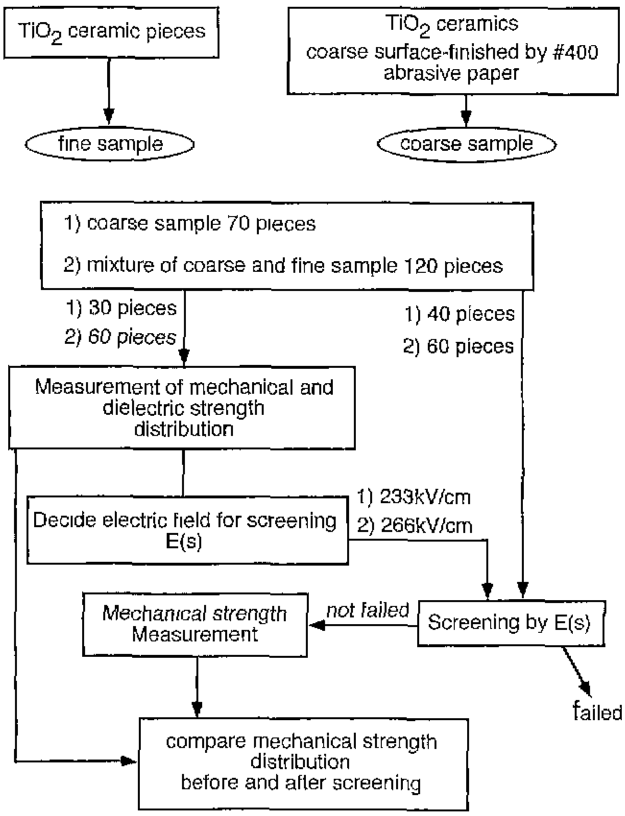

Fig. 1. Schematic diagram for experimental procedure.

trodes were made to have diffused edges to prevent concentration in electric field at the edge of electrode. Test pieces were placed in silicon oil to prevent the surface flash-over. The electric field at which a current abruptly increased was regarded as dielectric strength (Eb).

Mechanical strength was measured by three point bending test, in which span length is 10 mm and crosshead speed is 0.5 mm/min. Obtained mechanical and dielectric strength distributions were compared and screening field (Es) was determined from the dielectric strength distribution.

Silver electrodes with a diameter of 2.5 mm were attached to the approximately center point of both sides on the test pieces for screening. The electroded test pieces were subjected to breakdown test, the electric field increased up to the screening field. The broken pieces during screening test were eliminated. Mechanical strength were measured on the survived samples, on which the maximam stress was applied at the center line of the electrode. Mechanical strengths before and after screening were compared using the Weibull statistics.

3. Evaluation of strength distribution

Distribution of mechanical strength and dielectric strength were estimated to use the function of two-parameter Weibull distribution, as follows,

$$F = 1-\exp[-(\sigma/\sigma_0)^m V] \quad (1)$$

where σ is the mechanical strength or dielectric strength,

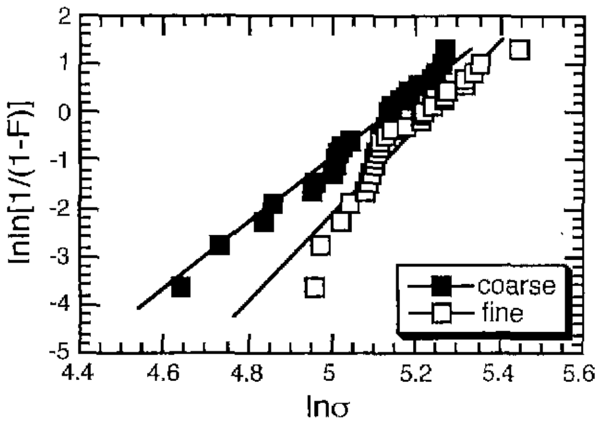

Fig. 2. Weibull plots of mechanical strength for differently surface finished TiO_2 ceramics.

σ_0, m, V are the Scale parameter, shape parameter (=Weibull modulus), and effective volume for each strength, respectively. Cumulative probability, F, was calculated using the mean rank method. On the mechanical stress screening, the failure probability after screening, F(a) is expressed as,

$$F(a) = (F_{total}-F_s)/(1-F_s) \quad (2)$$

where F_{total} is the failure probability without screening and F_s is the failure probability with a screening stress. According to this equiation, strength distribution after screening shows convex curve approaching to the screening stress, σ_s.

III. Results and Discussion

Fig. 2 shows the Weibull plots of mechanical strength for the surface-finished (coarse) and unfinished (fine) specimens. Both plots show good linearity (the correlation coefficient r>0.95) , indicating that the scattering of each data set can be expressed by a single-mode Weibull distribution function. The coarse finished test pieces ground by No.400 abrasive paper shows relatively small Weibull modulus and average strength (Table 1), indicating that surface flaws, which play a role of mechanical fracture origin, have been introduced by grinding.

Fig. 3 shows the Weibull plots of dielectric strength for the different surface-finished specimens. Coarsely surface finishing leads to a similar tendency in dielectric strength, that is, decreasing both Weibull modulus and mean strength. As a result, distribution shapes of both mechanical and dielectric strengths shows good resemblance for

Table 1. Average Strength and Weibull Modulus for Different Surface-finished TiO_2 Ceramics

	Mechanical strength	Dielectric strength
Average strength (coarse/fine)	160/178(MPa)	261/293(kV/cm)
Weibull modulus (coarse/fine)	6.9/8.8	6.1/8.3

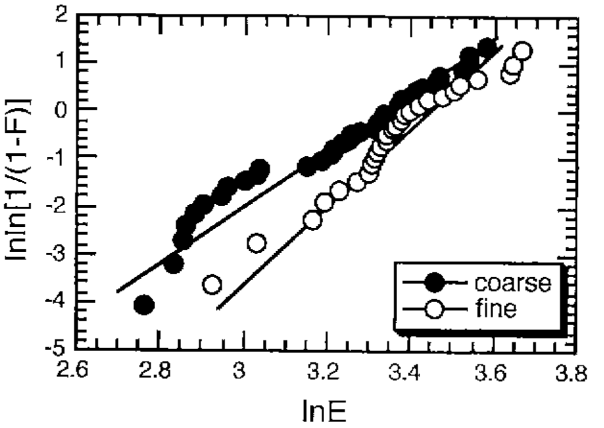

Fig. 3. Weibull plots of dielectric strength for differently surface finished TiO_2 ceramics.

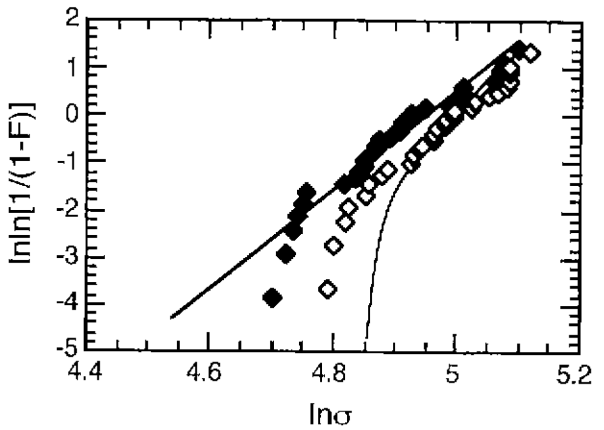

Fig. 4. High voltage screening for coarsely surface finished TiO_2 ceramics.

both coarse and fine samples (Table 1). Such similarity in both strength distributions has already been reported by one of the authors, indicating the analogy of weak spot distribution for both failures.[3-5]

The high voltage screening was conducted on the strength degraded samples of which mechanical and dielectric failures are both governed by the surface flaw. Seventy coarsely surface finished test pieces were prepared; 30 of which were used for dielectric strength measurement and the other 40 for screening. From the dielectric strength distribution, screening field was determined as 233 kV/cm at which cumulative dielectric failure probability was 30%.

Fig. 4 illustrates the Weibull plots of mechanical strengths before and after high voltage screening. Mechanically low strength parts are eliminated selectively by the high voltage screening, and the Weibull plot after high voltage screening is bended to become a convex curve while plots at high strength region are almost the same. This result means mechanical strength degraded samples have been removed by the electric method, in other word, severe mechanical flaw introduced sample tends to be weak in dielectric strength.

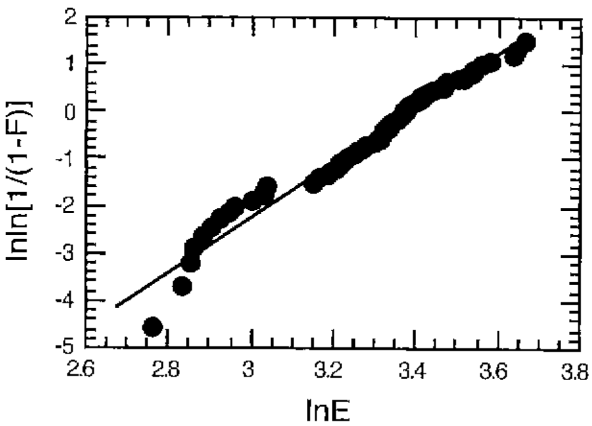

Fig. 5. Weibull plots of dielectric strength for mixture of differently surface finished TiO_2 ceramics.

Then the coarsely and fine finished samples were mixed and subjected to the high voltage screening. Fig. 5 shows the Weibull plots of dielectric strength for the mixture of different surface-finished specimens. The plots should be composed of two different distribution functions, however, they can roughly be estimated by a single distribution. This is probably because the two distributions overlap considerably as shown in Fig. 2. From this distribution figure, screening field was determined as 266 kV/cm, at that field 35% of samples should have been broken electrically. If the field were applied independently to the coarsely and fine surface finished samples, cumulative dielectric failure probabilities should have been 50% and 20%, respectively.

Fig. 6 shows the distribution of mechanical strength before and after high voltage screening for mixed samples composed of the same number of coarsely and fine surface test pieces. Similar to the result for uniform surface samples, the lower mechanical strength parts are eliminated by high voltage screening and the Weibull plot after the high voltage screening bends to become a convex curve. After the high voltage screening, 15 out of 30 coarse surface samples and 5 out of 30 fine surface samples have been removed, which accords with the above mentioned expectation. As a result, two kinds of different surface roughness samples were left in the survived samples.

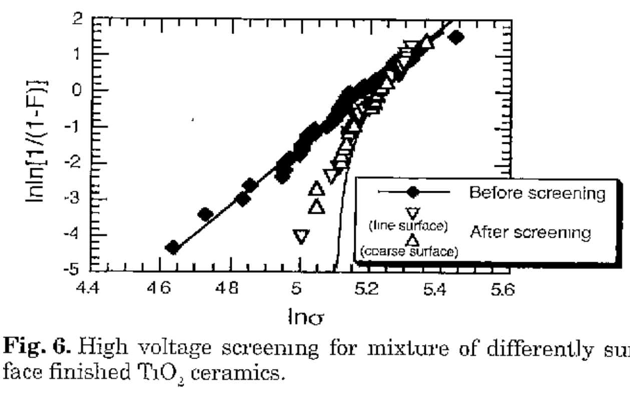

Fig. 6. High voltage screening for mixture of differently surface finished TiO_2 ceramics.

Surface flaws, especially those introduced during grinding or polishing processes, have widely been accepted as affecting the mechanical strength of ceramic material and its distribution. Matsuo et al. has reported that median cracks in scratch groove introduced during grinding are effective on its mechanical strength.[8] The coarse abrasive particle should increase a compressive weight by an abrasive particle and depth of grinding groove, so that depth and number of those flaws are increasing. By this reason, it is considered that strength distribution spread to lower region and average strength decreased.

However, there are few papers relating between breakdown strength and surface morphorogy. Surface bumps and flaws are known to weak spots on discharge along the surface.[9] There are some papers in literature that gas discharge induced dielectric breakdown.[10,11] If dielectric breakdown occurs by initiating a discharge in surface pit, groove and other flaws, only coarsely surface-finished sample could be selectively eliminated by the high voltage screening regardless of the mechanical strength. Some authors has reported that dielectric breakdown occurs when electric field concentrated to surface concave tips reaches critical electric field to induce electric avalanche.[5,11]

In the present result, mechanically weak samples were selectively eliminated by the high voltage screening irrespective of surface roughness, indicating the surface crack beneath the groove is the breakdown decisive flaw similar to mechanical fracture. In analogous with the stress concentration on mechanical fracture, electric field concentration should occur at the tip of the crack due to the permittivity difference, leading the analogy of mechanical and dielectric strengths.

By the way, the convex curves drawn in solid lines in Fig. 4 and 6, are theoretical lines of mechanical strength after stress screening at which 30 and 35% of the sample should have failed, denoted as σ_{s30} and σ_{s35}, respectively. On high voltage screening, there are some specimens with strengths lower than σ_{s30} or σ_{s35}. These results can be explained that correlation between mechanical and dielectric strength is not perfect. It is been already reported that correlation coefficient between mechanical and dielectric strength were 0.77 in TiO_2 ceramics (7). The reason why the correlation between mechanical and dielectric strength is not perfect is thought to be as follows. The effect of a flaw for both failure is not equivalent, for example mechanical strength depends on the direction of a crack in a surface plane, which should not be suited to dielectric strength taking into account of the field concentration. The areas at which stress and electric field have been applied, are not equivalent.

IV. Conclusions

Effect of high voltage screening have been confirmed on titania ceramics of which mechanical strength was degraded by coarsely surface grinding When the high voltage screening was conducted on mixture samples with coarse and fine surface, mechanically weak samples were selectively eliminated irrespective of surface roughness. The electric breakdown governing flaw should not the surface bump or groove but the surface crack similar to mechanical fracture, which leads the analogy between mechanical and dielectric strength.

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