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Pulsed Dielectric Breakdown of Aluminum Oxide (ALOX) Filled Epoxy Encapsulants: Effects of Formulation and Electric Stress Concentration

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**Pulsed Dielectric Breakdown of
Aluminum Oxide (ALOX) Filled Epoxy Encapsulants:
Effects of Formulation and Electric Stress Concentration**

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Abstract

Aluminum oxide (ALOX) filled epoxy is the dielectric encapsulant in shock driven high-voltage power supplies. ALOX encapsulants display a high dielectric strength under purely electrical stress, but minimal information is available on the combined effects of high voltage and mechanical shock. We report breakdown results from applying electrical stress in the form of a unipolar high-voltage pulse of the order of 10- μ s duration, and our findings may establish a basis for understanding the results from proposed combined-stress experiments. A test specimen geometry giving approximately uniform fields is used to compare three ALOX encapsulant formulations, which include the new-baseline "459 epoxy resin" encapsulant and a variant in which the Alcoa T-64 alumina filler is replaced with Sumitomo AA-10 alumina. None of these encapsulants show a sensitivity to ionizing radiation. We also report results from specimens with sharp-edged electrodes that cause strong, localized field enhancement as might be present near electrically-discharged mechanical fractures in an encapsulant. Under these conditions the 459-epoxy ALOX encapsulant displays approximately 40% lower dielectric strength than the older Z-cured Epon 828 formulation. An investigation of several processing variables did not reveal an explanation for this reduced performance. The 459-epoxy encapsulant appears to suffer electrical breakdown if the peak field anywhere reaches a critical level. The stress-strain characteristics of Z-cured ALOX encapsulant are measured under high triaxial pressure and we find that this stress causes permanent deformation and a network of microscopic fractures. Recommendations are made for future experimental work.

Acknowledgments

Several Sandia Laboratories personnel contributed toward this research effort: Breakdown tests were performed at the Materials Radiation Science Department's flash-x-ray test facility, with Grant Lockwood and Mike Selph. A pulsed high-voltage power supply constructed and maintained by Rick Howe and Mike Vickers (Neutron Generator Development Dept.), was crucial to these experiments, as was the help we received from Dave Beutler and Ted Parson (Simulation Technology Research Dept.) in data acquisition software and hardware. Sprytron switch tubes contributed by Gordon Boettcher (Firing Set, Fuze & Switch Tube Dept.) were utilized in the high-voltage pulse source and Otis Solomon Jr. (Sandia's Primary Standards Laboratory) calibrated the voltage and current data channels. We also thank Mark Grazier (Tribology, Mechanics & Melting Dept.) and Paul Hatch (Geomechanics Dept.) for performing the high-pressure, low-temperature deformation experiments on 828-Z alumina-filled encapsulant, Mark Grazier and Lawrence Carlson (Solid Mechanics Engineering Dept.) for sectioning and dyeing the resulting specimens, and Howard Anderson (Geochemistry Dept.) for help with scanning electron microscopy. Discussions with Doyle Morgan (Neutron Generator Development Dept.) have been informative.

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I. Introduction

A. Background

The requirements for high-voltage components are subject to change, as are the materials used to fabricate them. In the past such changes have been accommodated by costly cycles of build and test. A serious effort is now underway at Sandia to develop numerical models of component operation, to be used as cost-saving engineering tools. Predicting the likelihood of electrical failure is an important element in this ambitious task.

Aluminum oxide (ALOX) filled epoxy is the dielectric encapsulant in shock driven high-voltage power supplies. ALOX encapsulants display high dielectric strength under purely electrical stress, and fields near 200 kV mm^{-1} are usually withstood by small test specimens when the voltage is applied as a brief, single pulse. At present, however, the available breakdown information is too rudimentary to be incorporated into a predictive model of power-supply electrical reliability. Very little is known about the inherent breakdown statistics of ALOX encapsulant and how the breakdown probability is influenced by the distribution of electric stress. Furthermore, high voltage and transient high mechanical stress are simultaneously present when a power supply functions, and this situation may be difficult to realize in practical test-coupon arrangements.

As a first step in characterizing dielectric breakdown in ALOX encapsulants we have fabricated and tested simple specimens in which the electric field distributions were readily calculated. All measurements involved applying individual, unipolar pulses with duration of the order of 10 microseconds. These were “passive” tests, in that no mechanical stress was applied during the high-voltage pulse. We used electrode arrangements which produced a region of approximately uniform peak field, as well as electrode shapes that resulted in strong, localized field concentrations. The latter arrangements were intended to mimic the electric stress concentration at sharp corners and edges in actual power supplies. Data from these specimens also provided information on how a small mechanical fracture might affect the breakdown strength, because an electrically polarized fracture could give rise to strong field enhancement.

We have found evidence of large quantities of microscopic fractures remaining in ALOX specimens that have experienced large, static mechanical stress. These specimens displayed a residual shape deformation. Breakdown tests of such damaged encapsulant material were proposed, but these were not completed during the funding period. Also, a new test specimen geometry has been conceived and partially developed, in which a pair of mechanically compliant electrodes are cast into a block of ALOX encapsulant. This arrangement is intended for breakdown tests in a pressure cell, and the compliant electrodes avoid the severe mechanical-stress distortion that would be expected if rigid electrodes were embedded in the encapsulant material. Completion of these experiments is recommended as a simple extension of the work reported here, to be done before much more difficult “active” tests with simultaneous high voltage and shock stress are attempted.

B. Pertinent Knowledge and Published Data

There is a longstanding interest in using epoxy resins loaded with dielectric fillers as insulation or energy-storage media in high-voltage pulsed systems. A 1965 publication on the effects of various fillers on the electrical properties of casting resins [1] found that alumina filler had no effect on the breakdown voltage of non-irradiated test specimens, but improved the dielectric strength of irradiated ones. In 1970, Tse et al. [2] reported breakdown measurements on cast epoxy with tabular alumina and silica fillers subjected to 40- μ s-duration voltage pulses. Their specimens had large electrically stressed volumes, in the range 10 to 100 cubic centimeters, and relatively low breakdown fields (typically below 100 kV mm⁻¹) were found.

During this same era, Doyle Morgan (Neutron Generator Development Dept.) tested aluminum-oxide filled 828 epoxy in a wire-and-plane electrode configuration (wire electrode oriented parallel to a planar electrode) [3]. He determined, by using a stepped “up-and-down” test-voltage method [4] that stressed each specimen with only one high-voltage pulse, that a 1% failure probability (95% confidence limit) occurred at 219 kV. The pulse duration was of the order of 10 μ s, or possibly somewhat longer. The spacing between the wire and plane was approximately 12 mm which gave a rather low line-averaged electric field of 18 kV mm⁻¹. This value is comparable to a 19 kV mm⁻¹ (485 volts per mil) dielectric strength, measured per ASTM D149 (using ac stress, often applied for time periods of the order of seconds), which is reported in Sandia’s *Encapsulating Resins Properties Chart* [5]. We note, however, that ASTM D149 tests can yield low breakdown strengths compared with tests employing a brief, unipolar voltage pulse. The wire diameter is thought to have been about 2 mm, and the peak electric field at the wire surface can be estimated by invoking coaxial-cylinder geometry,

$$E_{\max} = V / [\rho_1 \ln(\rho_2/\rho_1)] . \quad (1)$$

If inner and outer cylinder radii are 1 and 12 mm, the peak field at 1% breakdown probability would have been about 90 kV mm⁻¹. The estimated peak field would increase to near 140 kV mm⁻¹ if the wire diameter were 1 mm, rather than 2 mm.

A small amount of component-test data is pertinent to the effective breakdown strength of ALOX encapsulant material under simultaneous mechanical and electrical stress. These data gave support to an established “zero failure” rule-of-thumb, that the electric stress in the ALOX encapsulant should not exceed 11.8 kV mm⁻¹ (300 volts per mil).

The very low dielectric strengths implied by these results are in strong contrast with higher values, generally well above 100 kV mm⁻¹, that are recorded during tests using pulsed, uniform-field, purely electrical stress. It seems that the combined effects of strong field intensification and high mechanical stress can lower the effective dielectric strength of ALOX encapsulant by at least an order of magnitude.

Recent publications from Asia and Europe have indicated a current interest in loaded epoxies for high-voltage dielectric applications. A small (10 to 20%) *increase* in

breakdown voltage has been reported, in epoxy resins containing a few vol% of alumina filler, compared with results from unfilled-resin specimens [6-10]. Above 5 to 10 vol% of filler, however, the breakdown voltage gradually declined to levels comparable to the results from the unfilled-resin specimens. The test arrangements typically used a sharp needle as one electrode, which was oriented perpendicular to a planar opposing electrode (“needle-plane” electrode geometry), and specimens were subjected to multiple voltage pulses that were gradually increased in amplitude until breakdown occurred. Such testing procedures may place a greater emphasis on high-voltage aging effects than in our tests, which stressed each specimen with only a few voltage pulses, and it should be noted that data obtained under our test conditions may not agree with the results reported above. For example, we found substantially higher dielectric strength in unfilled epoxy than when it contained the usual, approximately 45 vol% of Alcoa tabular alumina (discussed in Section III.C). Other published findings seem more consistent with our data; Henk et al. [11] have reported a reduced resistance to partial discharges when epoxy resins are loaded with conventional dolomite, alumina or silica fillers, but an opposite effect was found with very fine particles of titanium dioxide or amorphous silicon dioxide.

The Japanese research team also reported a reduction in the dielectric strength [6] and an increase in the thermally stimulated current [7] above 150 °C, which they state to be associated with the glass transition temperature of the resin. The breakdown mechanism near and above this temperature is attributed to a thermal runaway process, while electron avalanches are presumed to be causing breakdown at lower temperatures [12]. The effects of large gamma-ray doses were also investigated, and no reduction in breakdown strength was observed at accumulated doses below 2 MGy (200 Mrad) [9].

II. Experimental Details

A. Encapsulant Formulation

Many of the ALOX encapsulant specimens we investigated contain 46.7 vol% of alumina filler (Alcoa T-64 Tabular Alumina, -325 mesh) in a “459 epoxy resin” (Sandia Laboratories terminology). This resin consists of Shell Epon 826 (di-glycidyl ether of bisphenol A type liquid epoxy resin) and two amine curing agents (Jeffamine D-230 from Huntsman Petrochemical Corp. and Ancamine 2049 from Air Products and Chemicals, Inc.) A very small amount, about 25 ppm, of an additive (KF-865 from Shin-Etsu Chemical, Ltd.) is included to facilitate removing bubbles from the liquid resin via vacuum treatment. This formulation has been proposed as Sandia’s new baseline alumina-filled encapsulant, and it will be referred to in this report as “459” ALOX.

An older encapsulant, used for many years to encapsulate high-voltage power supplies and other components, is composed of 43.2 vol% of the same alumina filler in a different bisphenol A type liquid epoxy. The resin is Shell Epon 828, which is cured with a mixture of two aromatic amine curing agents (Epi-Cure Z Curing Agent from Shell Oil Company). Several specimens containing this material, referred to here as “828-Z” ALOX, were included in the study for comparison purposes. Encapsulant formulations are summarized in Table I. Note that these two versions of ALOX encapsulant differ in resin, curing agent, precise alumina amount, and oven-cure schedule, and an anti-foaming agent has been added to the newer formulation.

Table I: ALOX Encapsulant Formulations

Test Specimen Designation	Filler Manufacturer and Product Name	Filler vol. %	Epon Epoxy Resin	Curative	KF Anti-Foaming Agent
“459”	Alcoa T-64 tabular alumina	46.7	826	459	yes
“828-Z”	Alcoa T-64 tabular alumina	43.2	828	Z	no
“459-AA10”	Sumitomo (Japan) advanced alumina AA10	46.7	826	459	yes

Specimens were also tested in which the Alcoa alumina filler was replaced with 46.7 vol% AA10 “Advanced Alumina” from Sumitomo of Japan, but the composition otherwise remained identical to the 459 ALOX encapsulant. The AA10 filler is a vapor-deposited alumina consisting of faceted, but otherwise nearly spherical particles having diameters ranging from 6 to 12 microns. By contrast, the Alcoa alumina is produced by

crushing fired ceramic and contains particles of highly irregular shape. The “459-AA10” ALOX encapsulant is of interest for several reasons, including a reduced viscosity of the uncured mixture. A few test specimens were also fabricated which contained resins without the alumina filler, in order to determine the dielectric strengths of the resins.

B. Specimen Fabrication

Our test coupons consisted of aluminum electrodes which were embedded in cubes of cured encapsulant. These were fabricated by pouring the uncured mixture into pre-heated molds that held the electrodes rigidly in position. Trapped air was removed by vacuum degassing and the specimens were cured in an air-circulating oven. The temperature schedule used for the 459 epoxy specimens lasted 8.5 hours and consisted of 2.5 hours at ambient temperature, a 3 hour ramp to 93 °C, a 2 hour isothermal period at 93 °C, and a 1 hour cool to ambient temperature. A few 459 specimens were cured by using the longer, 29-hour “anhydride curing schedule” (Sandia Laboratories terminology) currently used with 459-ALOX encapsulated high-voltage power supplies, to determine if this affected the breakdown strength. The temperature schedule used to cure the 828-Z specimens lasted 32 hours and consisted of 2 hours at ambient temperature, a 10 hour ramp to 93 °C, an 18 hour isothermal period at 93 °C, and a 2 hour cool to ambient temperature. This schedule simulated the curing and post-curing of 828-Z-ALOX encapsulated power supplies. Our efforts to avoid bubbles in critical high-field regions were apparently successful, as no large voids have been found when broken-down samples are dissected.

Two basic sample geometries were tested, which are depicted in Fig. 1(a) and 1(b): “Uniform-field” coupons contained a pair of 1/4” diameter aluminum-rod electrodes in which the opposing electrode ends were smoothly rounded with 1/4” radii. The narrowest gap between these electrodes, which occurred along the axis of cylindrical symmetry, was set at 0.5 mm. This arrangement resulted in a nearly uniform electric field being applied along the symmetry axis, and a field strength up to 370 kV mm⁻¹ (9.4 kV per mil) could be realized within the voltage limit of the high-voltage pulse source. “Enhanced field” coupons were fabricated by replacing one of the two rounded-end aluminum rods with an 1/8” diameter cylinder having a square-cut end. If a geometrically perfect edge had been achieved on the square-cut cylinder, a mild electric-field singularity would have occurred there. (One can readily show, by applying conformal mapping techniques, that near the sharp edge the field becomes inversely proportional to the cube root of distance from the edge.) In reality, the effective edge radius was found to be in a 10- to 30-μm range (very roughly 1 mil), which limited the maximum field. Two different symmetry-axis gaps were used with this type of sample, 0.9 and 2 mm, in order to test the effects of field distribution in the inter-electrode gap. Because these samples provide high fields at only one of the two electrodes, the possibility of a polarity dependence exists; in all of these tests the sharp edged electrode was chosen to be positive.

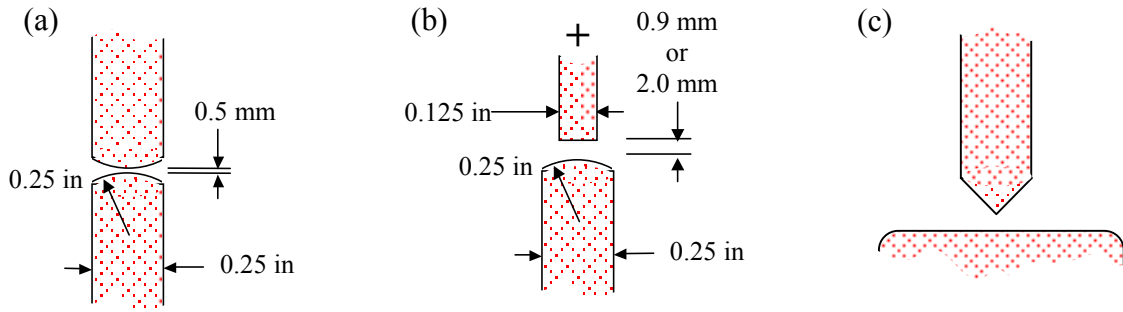


Figure 1. Test-coupon electrode geometries: (a) “Uniform-field” electrode geometry with cylindrical rods having 0.25-inch radius spherically rounded ends. (b) Square-cut cylindrical anode, which produced strong field intensification near the sharp edge of the cylinder. Two centerline gaps were used. (c) Conical point (a 90-degree included angle is shown). This geometry would replicate, with fair accuracy, the electric stress distribution near a 3-D rectangular corner of a conductive or high-permittivity feature.

A third electrode geometry is being developed for future coupon tests. As shown in Fig. 1(c), one of the electrodes is sharpened to a conical point. This results in a highly localized electric stress concentration near the cone tip, which should severely test any breakdown criterion. Note that a cone with an included angle of 90° approximately replicates the electric stress distribution near a 3-dimensional rectangular corner of a conductive or high-permittivity feature.

C. Breakdown Testing

Breakdown data were acquired at the test facility operated by Grant Lockwood and Mike Selph (Materials Radiation Science Dept.), using high-voltage pulse equipment constructed by Neutron Generator Development Dept. personnel. A 20-ns flash-x-ray source having photon energies in the 100 to 200 keV range was available, and it could be synchronized with the midpoint of an applied high-voltage pulse.

In the breakdown tests we report here, the encapsulant specimens were immersed in Galden® (Ausimont) fluorinated dielectric fluid to prevent external breakdown, and were subjected to nominally flat-topped, unipolar high-voltage pulses. These waveforms were generated by an inductive-capacitive pulse forming network, and typically had rise and fall times of approximately 2 microseconds and duration of the order of 10 microseconds. Pulse amplitudes were adjustable to maximal amplitudes approaching 200 kV, at which the immersion fluid was stressed near its practical limit.

Test coupons were subjected to individual high-voltage pulses, roughly once per minute. After an initial shot at a voltage about 20% below the expected breakdown level, the voltage was increased in roughly 10% steps until breakdown occurred. Usually two to five pulses were applied before breakdown was achieved. It is not known if these

multiple pulses may have influenced the final breakdown strength (as a result of either conditioning or aging effects), but the very modest scatter in breakdown voltages argues against any significant effect.

Small voids are typically found inside the Alcoa alumina particles when cured encapsulant is sectioned and examined under a microscope, and some relatively large, 300 μm (10 mil) voids have been discovered in cured 459 ALOX encapsulant (but not in the 828-Z ALOX encapsulant). It was of interest to determine if gas-discharges in these voids could affect the dielectric strength of the encapsulant. Some of our coupons were therefore tested with concurrent x-ray bursts, each of which gave an integrated dose of roughly 2 rad silicon at the sample location. These x-ray bursts, which were as large as practically achievable at the test facility, should have triggered discharges in many of the voids. However, no obvious radiation effects were observed during any of these tests.

We note that a very large current anomaly, which might be misinterpreted as dielectric breakdown, is sometimes recorded when a specimen is pulsed near the equipment voltage limit. This is caused by a luminous arc external to the specimen, through the Galden® dielectric fluid in the high-voltage tank. More frequent, but less luminous, corona-like discharges into the dielectric fluid give rise to small current anomalies.

III. Results

A. Uniform-Field Coupons

Four uniform-field coupons from each of the three ALOX encapsulant formulations were tested. Several of these twelve similarly constructed specimens were dissected after testing and all breakdown tracks were close to the electrode centerline, as intended. These microscopic examinations also allowed the electrode spacing to be measured, and the mean spacing was approximately 0.55 mm. We used this averaged value in converting the breakdown voltage from each specimen to breakdown field (ratio of voltage and electrode spacing).

Five of these twelve specimens were subjected to concurrent irradiation, but none of them broke down in synchrony with the timing of an x-ray burst. The highest breakdown field, in fact, was recorded with an irradiated 459-AA10 ALOX coupon. One of the two irradiated 828-Z coupons gave the highest breakdown field of the four of this type, and the breakdown fields from the two irradiated 459 coupons fell near the average from this specimen type.

Because the irradiation had no discernable effect, the breakdown voltages from all four specimens of each encapsulant formulation were pooled. The mean breakdown fields and standard deviations were $248 \pm 7 \text{ kV mm}^{-1}$ for 459 specimens, $218 \pm 39 \text{ kV mm}^{-1}$ for 828-Z specimens, and $312 \pm 25 \text{ kV mm}^{-1}$ for 459-AA10 specimens. These uniform-field results are listed in Table II. From these very limited data one might hypothesize that the more regularly shaped Sumitomo AA10 filler particles have improved the dielectric strength by 20 to 30%.

B. Enhanced-Field Coupons

All further ALOX-encapsulant tests used coupons having the Fig. 1(b) electrode arrangement, which gave locally enhanced electric stress at the sharp edge of the cylindrical anode. Post-testing dissections of some of these were done and the breakdown track was found, as expected, to be attached to the sharp edge of the square-cut electrode. Also, the electrode spacings were close to the intended spacings.

In the first of these tests the electrode spacing was nominally 0.9 mm, and the breakdown voltages of 459 and 459-AA10 specimens, four of either type, were compared. As found with the uniform-field specimens, there was no clear indication that irradiation influenced breakdown. One of the irradiated 459 specimens broke just after the x-ray burst and gave the lowest breakdown voltage of the four in this group; however, the other irradiated one gave the second highest breakdown voltage in this group. Any radiation effects were therefore ignored, and the mean breakdown voltages and standard deviations of the pooled data were $79 \pm 8 \text{ kV}$ for 459 specimens and $70 \pm 4 \text{ kV}$ for 459-AA10 specimens. According to these results, the dielectric strengths of these two formulations do not differ significantly. The modest advantage of the AA10 filler that was found with the uniform-field coupons apparently does not apply when localized high fields are present.

Table II: Breakdown Strengths of ALOX-Filled Epoxies

Electrode Arrangement and Other Information	Nominal Electrode Spacing (mm)	"459"	"828-Z"	"459-AA10"
		1st line: Number of Specimens in Test		
		2nd line: Mean BD Field (or Voltage) \pm Standard Dev.		
		3rd line: Mean Peak Field at Breakdown		
"uniform field" (initial tests)	0.5	4 248 \pm 7 kV mm ⁻¹ -----	4 218 \pm 39 kV mm ⁻¹ 1 -----	4 312 \pm 25 kV mm ⁻¹ 1 -----
square-cut anode (initial tests)	0.9	4 79 \pm 8 kV \approx 230 kV mm ⁻¹	----- ----- -----	4 70 \pm 4 kV \approx 200 kV mm ⁻¹
square-cut anode (initial tests)	2.0	4 111 \pm 15 kV \approx 210 kV mm ⁻¹	3 179 \pm 11 kV \approx 340 kV mm ⁻¹	----- ----- -----
square-cut anode (matched anodes) R. Lagasse's lab	2.0	8 129 \pm 13 kV \approx 240 kV mm ⁻¹	8 173 \pm 15 kV \approx 330 kV mm ⁻¹	----- ----- -----
square-cut anode (matched anodes) H. Arris's lab	2.0	8 108 \pm 14 kV \approx 200 kV mm ⁻¹	----- ----- -----	----- ----- -----
square-cut anode (combined data)	2.0	27 112 \pm 17 kV \approx 210 kV mm ⁻¹	14 176 \pm 13 kV \approx 330 kV mm ⁻¹	----- ----- -----
square-cut anode (“anhydride cure”) H. Arris's Lab	2.0	4 121 \pm 4 kV \approx 230 kV mm ⁻¹	----- ----- -----	----- ----- -----

Numerical computations of the electric field distribution were performed in order to analyze these results. The high spatial resolution we used allowed the inclusion of an appropriate edge radius, $\approx 20\text{ }\mu\text{m}$, on the square-cut anode electrode. These computations indicated that a one-volt potential difference between the electrodes would result in a peak electric field, at the edge of the anode electrode, of approximately 2.9 V mm^{-1} . This quantity was used to convert the measured breakdown voltages to approximate peak fields at breakdown, which are shown in Table II. We observe that these converted peak fields are comparable to the results from the uniform-field specimens, and that this is particularly true with the 459 specimens, where the mean value of peak field is approximately 230 kV mm^{-1} and the uniform-field averaged result is 248 kV mm^{-1} .

The remaining tests of ALOX-filled epoxy used the Fig. 1(b) arrangement with a nominally 2-mm electrode spacing. For this wider spacing, we computed a peak electric field of approximately 1.9 V mm^{-1} from a one-volt potential difference. Note that a more than doubling of the electrode spacing has lowered the peak field by only one third.

The initial tests with these wider gaps compared the breakdown voltages of 459 and 828-Z specimens, and gave unexpected results. The mean breakdown voltage of the 459 specimens was 111 kV, but was 179 kV for the 828-Z specimens. From these voltages the peak fields at breakdown are approximately 210 and 340 kV mm^{-1} , respectively. These very limited data imply that the older encapsulant formulation, with Z-cured Epon 828 resin, is superior under localized stress intensification. Furthermore, the mean value of the peak field from the 828-Z specimens is 50% above the mean result from the uniform-field tests of this formulation. This suggested that the electrical breakdown of the 459 formulation may respond to the peak field, regardless of the amount of material that is stressed at that field level, but that the 828-Z formulation behaves differently. Its effective dielectric strength depends on the electric field distribution and a greater peak field is withstood when only a small volume of encapsulant is stressed at that level.

C. Unfilled Resins

One might expect that the difference in dielectric strength of the 459 and 828-Z formulations, if real, might also be found in test specimens which contained unfilled epoxy. Therefore, a few specimens of either formulation were made to represent the three electrode variations previously used. When these were oven cured, the reaction exotherm was found to be more troublesome. With the full volume of the mixture producing heat during the curing process, rather than approximately half the volume when the alumina filler is present, the internal temperature of the more active 828-Z formulation may have peaked substantially above the oven temperature. The consequences were obvious distortions to the overall shape of these test coupons and, in some of these, small but visible separations between the electrode faces and the encapsulant.

Breakdown voltages, however, seemed not to be greatly perturbed by the curing-induced imperfections. All of the uniform-field specimens of either encapsulant formulation survived the maximum voltage available from our test equipment, 193 kV, which was

sometimes applied more than once, and a few of these specimens were also subjected to concurrent x rays. The electrode spacings were nominally 0.5 mm, which translates to very high breakdown fields greater than 380 kV mm^{-1} . There were no breakdowns in specimens with a square-cut cylinder and a 2-mm electrode spacing, and this also implies that peak fields near 400 kV mm^{-1} were withstood. These results show that the alumina filler markedly reduces the high dielectric strengths of the unfilled epoxies.

Two non-uniform-field coupons with 0.9-mm electrode spacings, Fig. 1(b), were tested from either encapsulant formulation. Both of the 828-Z specimens survived 193 kV, at which the peak field is estimated to be near 550 kV mm^{-1} . However, both of the 459 specimens broke, at a slightly lower voltage, each after first surviving one pulse at this voltage level. These unfilled resins are again found to have very high intrinsic dielectric strengths, but the 828-Z resin may be superior. These results also demonstrate that breakdown tests of the unfilled resins are made difficult by their very high dielectric strengths.

D. Matched Anode Electrodes

We were concerned that consistent differences in the edge radii of the square-cut anode electrodes, or some processing variable, might have contributed to the substantial difference in the measured dielectric strength of the 459 and 828-Z ALOX encapsulant specimens. To resolve this, a batch of new anode electrodes were fabricated with the intention of holding the edge radius close to a nominal 10- to 30- μm range, and each of these was carefully examined under the microscope. A set of 24 anode electrodes judged to have relatively consistent edge radii around the cylinder circumference were selected, and this collection was then randomly split into three groups of eight electrodes. In a further attempt to eliminate uncontrolled variables all of the encapsulant formulations were blended using a Hobart mixer, but two batches of the 459 were prepared, one in Bob Lagasse's laboratory and the other in Howard Arris's laboratory (Organic Materials Dept.).

The test specimens containing these matched anodes were again configured with a nominally 2-mm electrode separation. Mean breakdown voltages of the three groups were 129 kV for 459 encapsulant mixed in Lagasse's laboratory, 108 kV for the same encapsulant type mixed in Arris's laboratory, and 173 kV for the 828-Z group of coupons. This confirmed that 828-Z displays substantially higher pulsed dielectric strength with sharp-edged anode electrodes. These results are listed in Table II.

Table II also shows the combined breakdown results from all tests of standard 459 or 828-Z ALOX encapsulants that used the 2-mm electrode separation (which includes seven 459 tests and three 828-Z tests not mentioned above). The nearly 60% greater mean dielectric strength of the 828-Z encapsulant under these test conditions cannot be accounted for by scatter in the data. According to the number of measurements contributing to the means and the standard deviations, the uncertainties in mean value are only about $\pm 5\%$, at a 95% confidence level, for either set of combined data.

E. Investigation of Other Variables

In a final round of tests, four batches of 459 encapsulant were hand-mixed by Howard Arris and were cast into test specimens with square-cut cylindrical anodes and 2-mm electrode separations. The first of these contained the standard formulation, and the four specimens using it showed a mean breakdown voltage of 95 kV, which is below the combined-data mean of 112 kV but not greatly so. In the second batch the alumina content was lowered by 7.5% to match the volume fraction of alumina in the 828-Z formulation. This resulted in a mean breakdown voltage of 106 kV from four specimens. The third batch was prepared without the KF degassing aid, and four specimens gave a mean breakdown voltage of 103 kV, which is not substantially different. Finally, four 459 encapsulant specimens were cured according to the 29-hour “anhydride curing schedule” (Sandia Laboratories terminology), which is currently used with 459-ALOX encapsulated high-voltage power supplies. This resulted in a mean breakdown voltage of 121 kV, which is not significantly above the combined mean of the other 459 encapsulant specimens with this electrode arrangement (see Table II). While this small amount of data does not eliminate the possibility of measurable dependence on formulation or processing variables, it does show that several of them are not critically sensitive.

F. Discussion

Our data suggests that the new 459 ALOX encapsulant suffers breakdown when the electric field anywhere reaches a critical level in the 210 to 240 kV mm⁻¹ range, regardless of the volume of encapsulant stressed at that level. In fact, the breakdown voltage with a particular electrode geometry might be predictable from a computation of peak electric stress. A reasonable extension of this argument would suggest that 459 ALOX is also highly sensitive to microscopic field-enhancing defects, an example of which would be a stress-induced mechanical fracture that becomes electrically conductive by way of a partial discharge. A newly created fracture would be filled, for a brief time period, with “fracto-emission” electrons and ions [13], which would trigger a surface discharge if the electric field component in the plane of the fracture were sufficiently large. It must be pointed out that *we have no evidence, from either modeling or diagnostic tests, that such fractures are opened by the passage of power-supply shock waves*. This is simply a possibility that is not readily eliminated.

One might suppose that the reduced sensitivity of the 828-Z ALOX encapsulant to field enhancement near a sharp electrode corner is primarily a statistical “weakest-link” effect, such as apparently governs the pulsed breakdown of GMB-filled epoxy [14]. The underlying principle is that the volume of the encapsulant that is exposed to the highest fields is very small, and this reduces the risk of breakdown initiation. However, another possibility suggests itself: Perhaps the 828-Z epoxy has greater high-field conductivity [15] than the 459 epoxy, and the high fields in the near vicinity of sharp electrode features are able to relax on a time scale comparable to the voltage risetime. It is clear that elucidating the responsible physical process would require further research.

The AA10 filler resulted in the highest dielectric strength when uniform-field specimens were tested, but this advantage was apparently lost in non-uniform-field specimens. Explanations for this effect can also be hypothesized: We presume that breakdowns start in the polymer constituent. There is a relatively large volume of highly stressed encapsulant in the uniform-field coupons, and it may contain a sampling of particularly large and irregularly shaped particles when the standard alumina filler is used. The encapsulant dielectric strength might be influenced by the locally enhanced fields around these particles. Alternatively, there may be a sensitivity to discharge events occurring within certain alumina particles that contain fractures or voids. The highly regular AA10 filler would be beneficial in either case. A relatively small volume of encapsulant is involved when the highest fields are localized near an electrode edge, and it may be unlikely that a large, irregular, or flawed alumina particle is present.

G. GMB-filled 459 Epoxy Encapsulant

It is of interest that 459 epoxy resin with glass microballoon (GMB) filler also appears to show a sensitivity to sharp-edged electrodes. In this case the reference material was the GMB-filled encapsulant that has been used for many years in high-voltage components. (The formulation contains Shell Epon 828 epoxy, B. F. Goodrich CTBN 1300X8 elastomeric modifier, and a diethanol amine curative.) In non-radiation tests of coupons that gave strong field intensification at the anode electrode (the anode consisted of a thin metallic “fin” that was oriented perpendicularly to the surface of a planar cathode) the 459 GMB encapsulant gave an approximately 30% lower mean breakdown voltage than the reference encapsulant. Eight or nine specimens of each type were tested.

This finding, when combined with our data from the alumina-filled specimens, suggests that 459 based encapsulants have a lower breakdown strength than traditionally used encapsulants when significant electric field enhancement is present, whether the filler is alumina or GMB.

IV. Triaxial Compressive Deformation of ALOX Encapsulant

A. Stress-Strain Characteristics

Three specimens of Z-cured, alumina filled encapsulant were deformed in triaxial compression at high confining pressures ($\sigma_3 = 100$ or 200 MPa), at temperatures of -30 or -60 °C, and with a compressive strain rate of 0.01 s^{-1} applied parallel to the axis of maximum compressive stress, σ_1 . (Following the usual rock mechanics convention, compressive stresses and strains are reckoned *positive* in this report.) Test specimens and strain instrumentation, test apparatus, and experimental procedures were virtually identical to those employed earlier for ALOX encapsulant characterization [16]; however, a 500 MPa pressure vessel capable of operating at temperatures as low as -65 °C [17] was substituted for the 1000 MPa vessel used in the earlier investigation. The right, square prisms of encapsulant ($10.8 \text{ mm} \times 10.8 \text{ mm} \times 25.4 \text{ mm}$) were compressed along the long axis to strains approaching 20%. Data for these three tests are shown in Fig.2, in which we plot the stress difference, $\sigma_1 - \sigma_3$, *versus* the compressive axial strain. The stress difference is twice the maximum resolved shear stress applied to the specimens. These specimens retained a visible barrel-shaped distortion after they were removed from the pressure vessel.

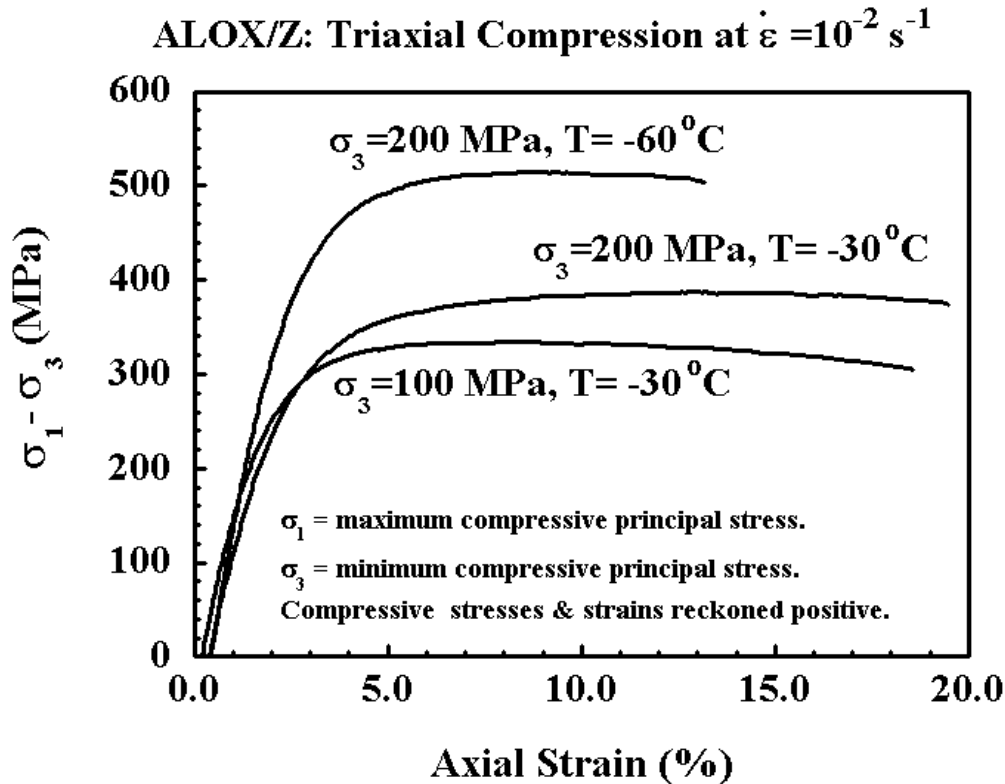


Figure 2. Stress-strain plots for three Z-cured ALOX encapsulant specimens. These were deformed in compression at the confining pressures (σ_3) specified on the figure [17]. The hydrostatic pressure was applied prior to the deviatoric loading.

In the earlier investigation into the mechanical properties of Z-cured ALOX encapsulant (and other ALOX formulations) at room temperature [16], only those specimens deformed without confining pressures showed definitive evidence of compressive failure. Under all other stress conditions the specimens continued to harden. However, the stress-strain curves shown in Fig. 2 clearly show signs of flattening (specimen softening) and, possibly, incipient failure. Fracturing was not detectable visually, even in the specimen deformed at the lowest confining pressure ($\sigma_3=100$ MPa). We therefore investigated the microstructure of these deformed specimens, looking for indications of damage at the microscopic scale. The obvious effects of temperature and confining pressure on sample strength are discussed elsewhere [17].

B. Evidence of Micro-Fracturing

Specimens were sectioned parallel to the axis of compression and the newly exposed surfaces were lapped smooth. The prepared faces were then immersed in a fluorescent dye penetrant (Zyglo, Magnaflux® Div. Illinois Tool Works, Inc.) for about one minute and then wiped clean. The barreled regions showed an area of staining (see Fig. 3) indicative of dye uptake in a network of microscopic fractures or voids. This was not found in similarly prepared, but undeformed material; nor is this damage visible in the less-deformed ends of the specimens shown in Fig. 3, which were comparatively protected from deformation by frictional effects at the specimen-endcap interface [17]. Note that the specimen on the far right in Fig. 3, deformed at $\sigma_3=200$ MPa and $T=-60$ °C, absorbed the least dye and was deformed only to about 13% axial strain. The two specimens on the left were deformed to axial strains of about 18.5 and 19.5% and absorbed substantially more dye. Despite the obvious penetration of the specimens by the dye, however, no fractures or voids were detectable using ordinary incident light microscopy.

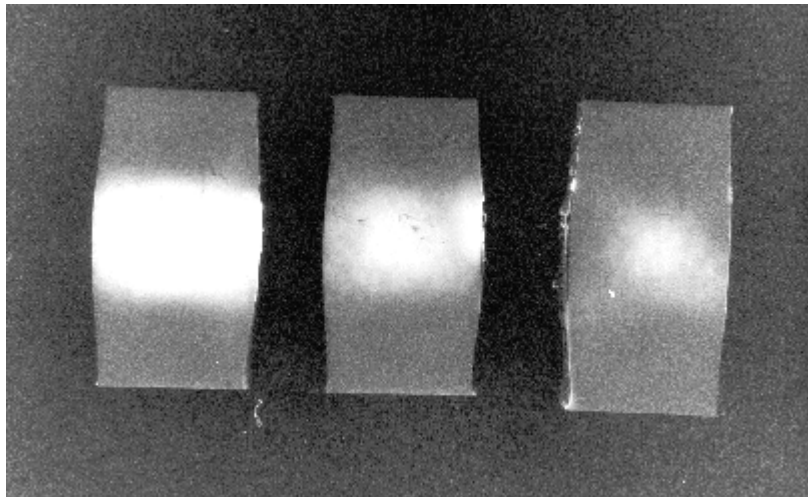


Figure 3. Ultraviolet light photograph of dye-impregnated specimens. The bright regions indicate dye penetration. From left to right, the specimens were deformed at confining pressures (σ_3) of 100, 200 and 200 MPa and temperatures of -30, -30 and -60 °C, respectively. Stress-strain plots for these specimens are given in Fig. 2.

The microstructure of the residual stress damage in these ALOX encapsulant specimens may hold an important clue to understanding the effects of stress on dielectric strength, at least at low temperatures. Sectioned specimens were therefore examined using the Geochemistry Dept. scanning electron microscope in an attempt to image the microscopic fractures that were responsible for the dye uptake. Remarkably, however, the deformed material showed no obvious indication of damage at any magnification, and the photomicrographs we obtained from deformed specimens were virtually indistinguishable (except for a very vague indication of greater amounts of fractures within grains of alumina filler) from the photomicrographs of undeformed specimens.

Why the microscopic fractures were so difficult to image is unknown. It may be necessary to combine dye penetration and microscopy in order to characterize the damage. Two approaches are: 1) using a high-power optical microscope with bright ultraviolet illumination to reveal the location of the fluorescent dye, or 2) using a penetrant fluid (if such exists) which provides contrast in a scanning electron microscope.

V. Recommendations for Further Work

A. Breakdown of Deformation Damaged ALOX Encapsulant

Highly damaged ALOX encapsulant may show a large reduction in dielectric strength. We found, while performing the tests reported in Section IV, that substantial microscopic damage can occur without being readily visible. The absence of obvious macroscopic damage, however, does not guarantee that the dielectric strength is unaffected, and we recommend an additional experiment to evaluate the effects of the deformation damage. The proposed test specimen would contain a wafer of the order of 10-mm square and 1-mm thick cut from the permanently deformed volume of a stressed encapsulant specimen. This material would be held between two smoothly rounded electrodes and cast inside a block of clear epoxy resin. Dielectric strengths of wafers cut normal to the compression axis would be compared with the strengths of wafers sectioned parallel with the compression axis and wafers from undeformed encapsulant. Some of these ALOX specimens have already been prepared, but none have been fabricated into practical test specimens. We recommend that these relatively simple breakdown tests be tried before embarking on complicated (but realistic) experiments that involve synchronized electrical and mechanical stress.

The preparation of thin test specimens from this microscopically damaged and quite possibly mechanically fragile material raises two important experimental issues: 1) An open fracture, which develops during the wafer-cutting process and penetrates the thickness of the wafer, would very likely cause a large and unrepresentative reduction in dielectric strength by encouraging a flashover breakdown along its surfaces. It is not clear how such damage might be prevented or detected. 2) Epoxy resin wicking into the fractures might lead to a large effect in the opposite direction, in which the open fractures would be filled and the resulting dielectric strength would be measured too high. To prevent this, the wafer surface might be sealed with a thin coating of a highly viscous or a partially gelled resin before the clear-resin casting step.

B. Pressure Cell Breakdown Tests

Static-stress breakdown tests of ALOX encapsulant have also been contemplated that would use the Geomechanics Dept. high-pressure triaxial test cell [17]. That apparatus is capable of applying realistically high stresses which include a large deviatoric component, but its high-pressure electrical feedthroughs would limit the electrical stress to about 10 kV and applying a short high-voltage pulse might not be practical. However, if applying a quasi-static deviatoric load can cause a large reduction (approximately tenfold) in dielectric strength and test specimens with sub-millimeter electrode spacings can be fabricated, breakdowns might actually be achieved.

Avoiding experimental artifacts would be a critical issue. For example, the dissimilar elastic properties of the encapsulant and the electrodes inside it may produce a mechanical stress distortion that unintentionally fractures the encapsulant. Electrical failure along a fracture could easily be misinterpreted as a true electrical breakdown.

Our scheme for minimizing mechanical stress enhancement is to use electrodes in the form of thin metallic films which are effectively suspended in the cured encapsulant. A method to realize this would be to cast electrodes of the desired shape (smooth-surfaced hemispheres, for example) which are, themselves, made of cured ALOX encapsulant. Alternatively, the electrode shapes could be machined from blocks of the encapsulant material. Each pre-electrode would be cleaned and inspected, and a thin metallic layer would then be applied to its surfaces by evaporation or sputtering in vacuum. The metallized hemispheres would be electrically attached to external conductors; a loop of wire, for example, could be secured to a hemisphere's equator with conductive silver-loaded epoxy. These electrodes would then be installed in a mold and additional encapsulant mix would be cured around them. We recommend that a few of these breakdown tests also be completed before proceeding with difficult combined-stress experiments.

C. Breakdown from Purely Electrical Stress

Another economical extension of the present work would be to proceed with developing an ALOX-encapsulant predictive breakdown model that considers pulsed electrical stress alone. Although the full benefits of such a computational tool are unclear, it would apply to practical situations in a shock driven power supply. For example, there are sharp edges and corners on the active ceramic and associated electrodes that give strong field intensification, and we already know that field intensification in the encapsulant might raise the risk of breakdown. Some of these high fields would be switched on in regions not yet affected by the shock wave.

Experiments that are needed to develop the model would involve specimens having large volumes of stressed encapsulant (but with electrode gaps less than 1 mm to stay within high-voltage capabilities), and specimens using our well-characterized conical points. Either of these would probe the dependence of breakdown strength on stressed volume; using a conical-point electrode, for example, would greatly reduce the stressed volume compared with using a square-cut cylinder.

D. Simultaneous Mechanical-Shock and Electrical Stress

Breakdown tests in which a realistic stress wave in the ALOX encapsulant is synchronized with a high-voltage pulse eventually must be performed, if the effects of shock stress on breakdown are to be understood. Gas-gun experiments may be the most convenient way to achieve the combined stress environment, but these would be difficult as well as expensive. Nevertheless, it must be emphasized that simpler but less realistic experiments are unlikely to yield the crucial information. As mentioned in Section III.F, real time fractures would be accompanied by coincident bursts of fracto-emission electrons and ions released into the fracture, which would trigger an electric discharge if an appropriate electric field were present at the same time. Tests on previously damaged material would lose that potentially critical feature.

Combined stress experiments would include test coupons with uniform-field electrode arrangements as well as coupons with rigid, rectangularly shaped electrodes. The intent of the latter specimens would be to create representative electric field intensification and realistic interactions with the mechanical shock wave. This would allow the possibility of real-time, millimeter scale encapsulant fractures (if such might occur in shock driven power supplies). An informative control experiment would be to replace the rigid rectangular electrodes with ones of the same shape that are mechanically matched to the surrounding encapsulant (Section V.B, above).

A thorough investigation as proposed here would greatly improve our understanding of electrical breakdown in ALOX power-supply encapsulant. This new knowledge would immediately provide engineering guidance, and might ultimately result in a predictive breakdown model.

VI. Summary

Test specimens containing various electrode arrangements have been used to investigate the microsecond-pulsed dielectric strength of three ALOX-encapsulant formulations. These studies were intended to provide a basis for understanding the results of more difficult breakdown tests, in which high voltage and mechanical shock stress would simultaneously be present. Although our data are limited, several informative conclusions can be drawn. The most general of these is that the dielectric strength of ALOX encapsulants is affected by the epoxy resin and curative mixture, and to some degree by the properties of the alumina filler. We note that unfilled resin has substantially higher dielectric strength under our pulsed-voltage test conditions.

When electrical stress having approximate spatial uniformity is applied to the new-baseline “459 epoxy resin” Alcoa ALOX encapsulant, fields of 240 to 250 kV mm⁻¹ (6.1 to 6.3 kV per mil) are required to cause breakdown. Comparable results are obtained from test specimens of the older 828-Z ALOX encapsulant formulation under these conditions. Substituting an alternative type of alumina filler (Sumitomo AA-10), which has roughly spherical particles with a relatively narrow size dispersity, increases the dielectric strength of the 459-resin formulation by perhaps 25% compared with results from using the standard alumina filler. Although voids are sometime observed when cast ALOX is sectioned, we find no sensitivity to concurrent x-ray irradiation.

The peak field in the 459-epoxy encapsulant appears to be the critical quantity when highly non-uniform fields are present, and the dielectric strength when referred to this peak stress (210 to 240 kV mm⁻¹) is comparable to that obtained with uniform fields. This encapsulant is therefore highly sensitive to small regions in which the field is high, which differs markedly from the behavior of GMB-filled epoxy under similar conditions [14]. Our very limited data from specimens fabricated with 459 epoxy and AA-10 filler indicate that the same conclusion applies, and that the apparent advantage of this filler in uniform-field specimens is lost when highly non-uniform fields are present.

Compared with the older 828-Z ALOX encapsulant formulation, the new-baseline 459 epoxy ALOX encapsulant gives roughly 40% lower dielectric strength when stressed with highly non-uniform electric fields (by using a square-cut anode electrode). These results are consistent throughout several different experiments, including one in which anodes were selected at random from a carefully matched set. We have determined that none of several formulation variables are responsible for the dielectric strength difference, including the mixing method used to prepare the encapsulant, where and by whom the mixing was done, and whether or not the KF anti-foaming agent was added. Substituting the anhydride curing schedule also had no discernible effect. The difference in dielectric strength is apparently caused by the different resin and/or curative. We speculate that a greater high-field conductivity [15] may be present in the older 828-Z formulation, which allows the high electric fields near electrode edges to relax on the time scale of the high-voltage pulse. Further experiments would be needed to confirm this hypothesis.

We point out that an analogous sensitivity to non-uniform electric stress is found with GMB-filled 459 epoxy, when that encapsulant is compared with an earlier formulation using Epon 828 plus CTBN resin cured with diethanol amine.

Recommendations for future work include a relatively simple breakdown test using wafers of ALOX material that have been deformed using high triaxial pressures. This would determine if the network of fractures, found with Zyglo dye penetrant, affect the dielectric strength. Pressure vessel breakdown tests of specimens containing mechanically compliant, metallic film electrodes are also recommended before the “realistic”, but much more difficult, studies of the effects of synchronized electrical and mechanical-shock stress are attempted.

Finally, we conclude that tests in which mechanical stress is also present must consider the possibility of stress-induced micron scale encapsulant fractures. Partial breakdowns occurring there may intensify local electric fields (as with the sharp-edged electrodes in some of our test coupons) and launch a catastrophic breakdown.

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