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INSITU-IMPREGNATED CAPACITOR FOR PULSE-DISCHARGE APPLICATIONS

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INTRODUCTION

Capacitor designs for DOE and/or DoD applications are now driven by two major factors; first, the need to reduce component volumes (attain higher energy density) to permit inclusion of additional components and/or sensors in systems and second, the continuing budget constraints. The reduced volume and cost must be achieved with no sacrifices in functionality, reliability and safety. (Sandia capacitors utilized as the capacitor discharge unit, CDU, for firing sets must often supply the weak-link function for accident or terrorist scenarios).

Dry Wrap and Fill Capacitors

Many CDUs have utilized a "dry wrap and fill" design, i.e. the dielectric system is composed of a Mylar[1] film and air, the amount of which is determined primarily by the winding tension.[2, 3] The capacitor ends are filled with an epoxy for protection and handling. If the output current rise time requirement was not too fast, buried-foil capacitors with symmetrically spaced taps for external connections could be designed with reasonable volumes. However, the newer slapper or silicon bridge detonators require very fast current rise times, less than 100 nseconds, and hence very low internal inductance for the capacitor (less than 10 nH). To achieve these low inductance values, an extended foil capacitor design is required. For extended foil capacitors, the margin requirements are prohibitive (0.75 to 1.5 inch) for dry wrap & fill in the 3-6 kV designs.

Fluorinert-Impregnated Capacitors

Sandia solved this problem in the 1980s by developing liquid Fluorinert-impregnated capacitors that were hermetically sealed in stainless steel cans. [4, 5, 6, 7] By substituting the Fluorinert ($k = 1.95$) for air, the margin requirements were reduced substantially (to about 0.25 inch) and the standard deviations for breakdown distributions were also reduced to about 0.3 kV, as compared to the typical 1kV for dry wrap & fill capacitors. DOE's required temperature extremes (-55 °C to +74 °C) and the high expansion coefficients for Fluorinert required the use of a compensating bellows in these capacitors, usually located in a hollow mandrel. This and the hermeticity requirements resulted in very expensive processes and costs; about $2500/capacitor for small development quantities (less than 50) and around

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$1000/capacitor for production quantities.

Solid-Impregnant Capacitors

To reduce these prohibitive development and production costs and to achieve the same approximate energy densities as the Fluorinert-impregnated capacitors, we investigated several rigid, solid-impregnate designs, but with inconsistent results. We then began investigating the feasibility of gels, primarily due to their higher dielectric constants (in the 6-9 range) and their higher elasticity. (Our capacitors must still function reliably after exposure to a number of temperature cycles, typically at least 10). Reliable performance with respect to ultimate breakdown strength and pulse-discharge life was never achieved with the rigid, solid-impregnant capacitors after exposure to this cycling.

Insitu-Impregnated Gel Capacitors

We are now achieving increasing success with these capacitors utilizing a gel/Mylar film as the dielectric system. Process details will be given later, but the extended-foil capacitor is wound on a mandrel, and ends are electroded via a commercially available, arc spray process. The electroded rolls are then placed in an evacuated chamber and pressure filled using a liquid gel. The temperature is then raised to achieve cross-linking of the gel. The capacitors are then "cut out" of the resulting gel block and cleaned for subsequent electrical test and evaluation. This is, of course, a suitable process for fabrication of small development quantities (now 10 capacitors) per run, but it must be substantially improved to become a viable production process.

We have utilized 0.3 μF and 2.0 μF extended-foil designs. These capacitors are intended to operate in the 3-6 kV range with output currents of a few kA, having rise times of less than 100 nsec. Three layers of 40 gage (g) Mylar (0.001 inch = 100 g) are wound on a 0.375-inch diameter mandrel at 80-100 grams of tension. For these studies, a 0.3 to 0.375-inch margin has been utilized. Several arc-spray electrode patterns (discussed later) have also been used.[8] We plan to later investigate other dielectric films with various gel compositions, but for now because of the weak-link requirements, we are only utilizing Mylar.

PROJECTED COST/VOLUME REDUCTIONS

The very large development and production costs, about $2500 and $1000 respectively, have already been cited for the Fluorinert-impregnated capacitors. These costs are used for comparison because only the Fluorinert capacitors have similar energy densities and performance characteristics (low standard deviations of the ultimate breakdown strength) to the gel capacitors. (This standard deviation or sigma is important because our acceptance requirements dictate that the average breakdown strength of a lot minus "k" times sigma must be greater or equal to capacitors' rated voltage value. For example, for a test lot of 20 units, k = 4). [9]

Experience to date indicates that the cost of our gel capacitors is about $300/capacitor when fabricated in lots of 20-40 units. For production quantities of greater than 1000 units, we believe costs in the range of $50 to $100 can be achieved, but this has yet to be demonstrated. To achieve these lower production costs, production lines or specific processes must be "qualified" to minimize in-process testing. Obviously, this approximate order of magnitude cost reduction would be significant.

To compare volumes (between the dry wrap & fill capacitors and the gel capacitors), we selected a 2.0 μF, 3.5 kV that utilized three layers of 48 g (3 X 48) Mylar. A 50% reduction in volume results. This occurs primarily because of the higher dielectric constant of the gel compared to air and because of a factor of three decrease in the margin. Most data for capacitors with the "better performing gel compositions" results in an approximate factor of three decrease in the standard deviation for the ultimate dielectric strength, i.e. short-time breakdown (STB). This destructive test (D-test) is performed by applying an approximate 250 volt/sec. ramp to the capacitor until breakdown occurs. Some data indicates that for a capacitor with a rated voltage of less than 4 kV, we could utilize a dielectric pad of 2 X 48 g. If this is the case, a volume reduction of 67% would result. (Additional units must be fabricated/tested to demonstrate this larger volume saving).

INITIAL FESIBILITY STUDIES

We began our studies utilizing various gel mixtures of three different plasticizers and three different cross-linking materials. The plasticizers were Di-n-Butyl Phthalate (DBP), Dioctyl Phthalate (DOP) and Phenyl Xylyl Ethane (PXE). The cross-linking materials were Diallyl Phthalate (DAP), Polybutadiene (PBD) and 1, 3, Butylene Glycol Diacrylate (212).

The capacitor rolls were wound and arc sprayed on the ends. Each roll utilized 2 x 48 g Mylar with 0.25-inch margins. This "dry" capacitor (before gel impregnation) design was set to be 0.3 μF. The gel mixture with a thermal initiator was heated to 85 °C and degassed and stirred in a container under vacuum. The capacitor rolls (20ea) were loaded into a separate chamber and vacuum baked also at 85 °C for one-half hour. This chamber was then back filled with the gel mixture, and the chamber was pressurized using a piston to 100 psig for one hour at the same temperature. The chamber was then vented to atmospheric pressure and the temperature raised to 125 °C for four hours to achieve insitu cross-linking of the gel. The capacitance increase after impregnation varied with gel compositions from 15% to 26%.

Ultimate Dielectric Strength at -55°C and +74 °C

These capacitors were destructively tested (D-tested) by determining their ultimate dielectric strength or short-time breakdown (STB) by applying a voltage ramp of about 250 volts/sec. to each capacitor until failure. The data is shown below in Table 1 with each capacitor group (gel composition) subdivided into two test groups, one at –55 °C and one at +74 °C. Note that the results are always better at "high temperature". Several gel compositions did, however, give encouraging "cold temperature" results. Relatively speaking, the "good" compositions were DBP/PBD and DOP/DAP. These initial results would influence our compositional investigations for the next several test groups.

Table 1

STB v. Temperature					
Composition	PXE/PBD	DBP/PBD	DBP/DAP	DOP/DAP	DBP/DAP/212
Avg. STB @ –55°C kV	3.20	6.26	2.99	6.74	4.45
Sample size	10	10	10	10	10
Avg. STB @ 74°C kV	8.24	8.03	7.79	8.85	9.47
Sample size	15	10	10	10	10

Although these results were somewhat encouraging, we had hoped to achieve higher STB values. We were concerned about any voids that might be created during the shrinking of the gel during cross-linking (this could explain lower the lower than expected results. We decided that a more optimum process would be to cross-link under pressure. A heating jacket was then added to the hydraulic cylinder that was utilized for the impregnation chamber. This permitted us to raise the temperature for the cross-linking operation while the capacitor rolls and gel were under pressure.

Ultimate Dielectric Strength vs Edge Margin at 23 °C

A second group of capacitors was wound, maintaining the 2 X 48 g Mylar pad. This time, however, we decided to see if a larger margin would produce higher STB values. Half of the units were fabricated with the identical 0.25-inch margin as per the prior test sequence and for the other half, the margin was increased to 0.375 inches. (The overall capacitor length was held constant, as the Mylar width was decreased. This lowered the "dry" capacitance of these larger margin units from 0.30 µF to about 0.18 µF). One of the prior gel compositions was repeated, several used slightly modified ratios of plasticizer/cross link and two new ones were tried. For this evaluation, we decided to only determine STB at room temperature.

The results were somewhat surprising in that the larger margin (0.375 inch vs. 0.,250 inch) had no effect on the STB values. In fact one 0.375-inch margin subgroup resulted in the lowest average STB (7.25 kV), and one 0.250-inch margin subgroup gave the highest STB average of 11.6 kV for six test capacitors. This particular test group was one of the ones with a "new" composition, i.e. DOP/259. The "259" cross-linking material is Polyethylene Glycol Diacrylate. These results indicated to us that if there were no gel voids in the margin region and if the integrity of the gel/electrode (aluminum foil) interface was good via adequate adhesion, excellent STB values could be achieved with a 0.250-inch margin.

Effects of Charge/Discharge Cycling on Ultimate Dielectric Strength at 23 °C

A second group of these DOP/259 units with the 0.250-inch margin were then tested to determine the effect, if any, of long-term pulse-discharge cycling (the 0.30µF "dry" design was maintained). This test, labeled as "operational life" or OPL is performed by repeatedly charging and then discharging the capacitor through a selected load to give the current output (with respect to rise time, peak amplitude and peak reversal (negative) current amplitude). For these tests, the capacitors were charged to 3.5 kV and the load was designed to yield at 20% peak reversal current. Of the group of ten test units, six were tested for STB with no pulse-discharge cycling. Four units were cycled for 100,000 times with no failures and then subjected to our standard STB test, again at 23 °C. The results are shown below in Table 2.

Table 2

Effects of Operational Life Testing		
	STB (no OPL Testing) (Sample size = 6)	STB (100k pulses) (Sample size = 4)
Average	9.94 kV	10.83 kV
Standard Deviation	1.35 k V	0.60 kV

We were very encouraged with these results as they indicated that even with the repeated cycling and the resulting electrical and mechanical stresses generated in the margins, adhesion was adequate to preclude formation of voids.

These were excellent results, at least on a comparative basis at this point in the development program and confirmed that the 0.25-inch margin was adequate.

Effects of Thermal Cycling on Ultimate Dielectric Strength at 23 °C

Another group of units were fabricated with the same dielectric pad (2 x 48 g Mylar) and margin (0.25 inch) to determine the effects of thermal cycling on the STB values. Fifty units were fabricated with half-tested for STB (virgin units) and half tested for STB after 10 temperature cycles. Each cycle was between –55 °C and +74 °C with a soak time at each temperature extreme of one hour and an approximate transition time of 2.8 hours. On all of our prior tests, the plasticizers DBP and DOP seemed to generally give the better performance. Since the best OPL results were obtained with the "259" cross-linking material, we decided to use DBP as the plasticizer for all 50 units and various percentages of "259" in three of the five test groups. For the other two groups we utilized "210" and "344" cross-linking materials. These are molecular-weight modifications of the "259" material. The "EO-TMPTA" is a triacrylate.

The results are summarized below in Table 3 with data as a function of gel composition. The "non-cycled" STB values were encouraging in that we were pleased to see an average for a larger group of test units (25) above 10 kV. The sigma or standard deviation of about 1 kV is similar to that achieved for our dry wrap and fill capacitors. We would prefer the sigma be less than 0.5 kV or closer to the values obtained for our Fluorinert-impregnated capacitors. (Of course to achieve the same sigma in the gel units considering that their margin is only one-third of that for the dry wrap and fill units may not be insignificant).

Table 3

Effects of Thermal Cycling on STB

Composition	DBP/259	DBP/259*	DBP/344	DBP/210/344	DBP/259/EO-TMPTA	Total
Thermal Cycled						
Average STB (kV)	7.10	6.08	5.90	6.46	6.53	6.39
Standard Deviation	1.92	0.50	0.85	1.79	1.78	1.39
Sample size	5	5	6	4	5	25
No Thermal Cycle						
Average STB (kV)	11.32	10.00	10.28	10.04	10.45	10.42
Standard Deviation	1.32	0.92	0.36	1.68	0.20	1.08
Sample size	5	5	5	5	5	25

*Different ratio

The STB results were, after thermal cycling, discouraging. Neglecting any probably small compositional effects, the average STB value for the thermal cycled units was only about 60% of the non-thermal cycled units. Based on our average STB minus four times sigma criteria (four is the appropriate multiplier for a sample size of about 20 test units)[9], the rated voltage of this capacitor after thermal cycling would be just under 1.0 kV. However, the corresponding value for the units not subjected to this environment is just over 6.0 kV for the rated voltage. This is quite a difference!

There is not a great deal of difference in the results as a function of these various gel compositions. Based on the limited sub-sample sizes, the first group of that particular DBP/259 ratio does give somewhat higher STB values by almost 1 kV for both the virgin and thermal cycled capacitors. Based upon our studies to date, it is now evident that thermal cycling is the most degrading and critical test of our gel capacitor design and fabrication processes. We decided to rely exclusively on the thermal cycle test as the evaluation method for future gel capacitor designs until we could achieve comparable performance between subsequent virgin and thermal cycled gel units.

PRESENT EQUIPMENT/PROCESSES DESCRIPTION

We next decided to concentrate on our design and fabrication of our impregnation equipment. A diagram of the resulting impregnation equipment is shown below in Figure 1 with a detailed description after the figure.

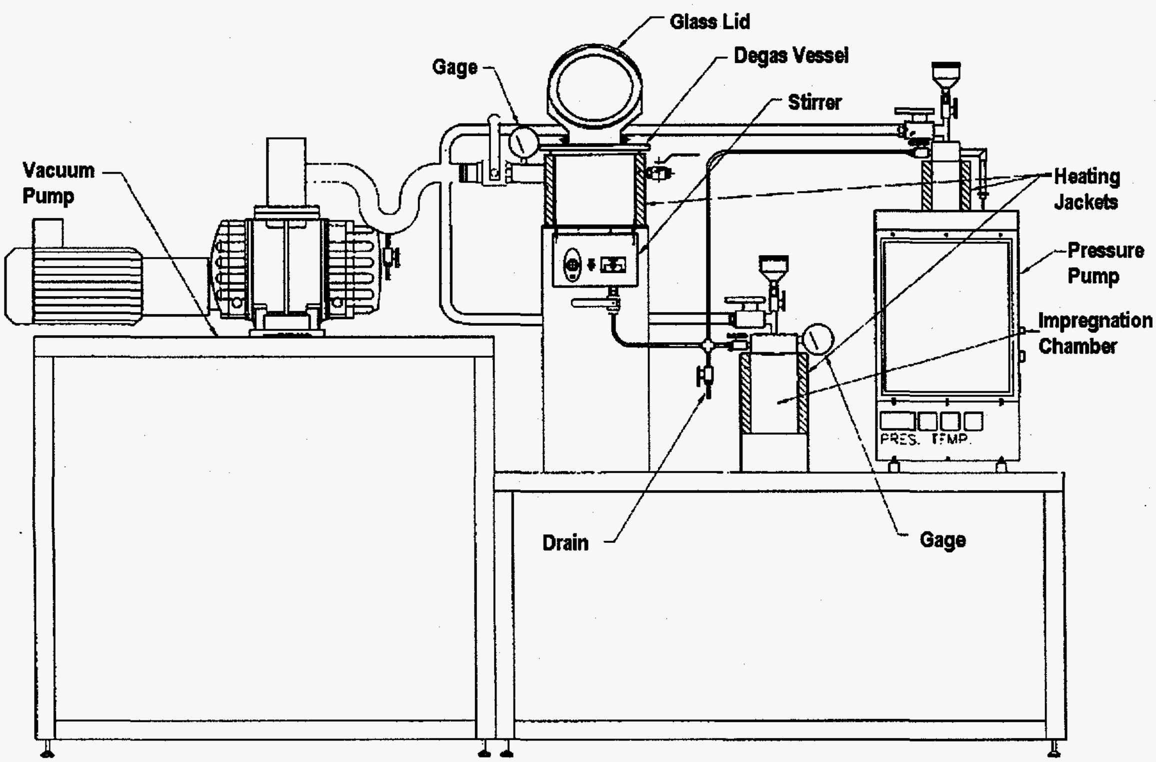

Figure 1

We should first emphasize that the impregnation equipment shown above is for processing small quantities of development and feasibility prototype capacitors. Extensive modifications will be required to achieve a production capability for larger quantities. Based on our several experimental prototype sizes, we can impregnate 8-20 capacitors during a single run. The capacitor rolls with their arc sprayed ends are loaded into the impregnation chamber. Holding fixtures for the rolls are utilized to protect the capacitor ends (exposed, extended foil edges and electrodes) from mechanical damage and to assure unobstructed gel flow. The basic process consists of vacuum baking the capacitor rolls, degassing the gel mixture, transferring the impregnant to the capacitor chamber, pressurization and curing.

A heating jacket around the impregnation chamber is used to increase the chamber temperature to about 80 °C. Simultaneously the chamber is evacuated using the vacuum pump to a range of 10 to 20 mtorr. The elevated temperature is maintained for four hours, and then the capacitors are allowed to cool to ambient temperature. The vacuum is, however, maintained overnight. The impregnation vessel must be isolated from the vacuum pump before degassing the gel mixtures. The appropriate gel mixture is put into the degassing vessel, and the materials are stirred under vacuum to achieve mixing and degassing at ambient temperature.

The vacuum is isolated from the degassing vessel, and then the pressure pump is evacuated. With the pressure pump under vacuum, the impregnation chamber is opened to the vacuum line to eliminate any out gassing that occurred during the gel degassing operation. The vacuum pump is next isolated from the system. The drain valve from the degassing vessel is opened to permit flow of the liquid gel mixture into the impregnation chamber and into the pressure pump.

The top of the degassing vessel is vented to atmospheric, pressure, which forces the liquid gel into the impregnation chamber and pressure pump. The impregnation chamber is then isolated from the degas vessel. The pressure is then increased to 100 psig minimum and held for about two hours to assure complete impregnation of the capacitors.

The pump is next isolated from the impregnation chamber, and the temperature of the chamber is increased (less than 80 °C) to a level to assure material cross-linking. The time at elevated temperature is a function of the gel composition. The chamber is permitted to cool to ambient temperature, and impregnated capacitors are removed, while still in their holding fixtures, by hand. After removing the capacitors from the fixtures, the excess gel is carefully removed from the exterior of the capacitor.

TEST RESULTS WITH CAPACITORS FABRICATED ON NEW EQUIPMENT

With better processing equipment, a larger group of gel units were processed in a continuing sequence. Based on customer interest in a slightly higher rated voltage capacitor (around 4 kV), the Mylar pad thickness was increased from two to three layers of 48 g.

1.9 μF Capacitor Ultimate Dielectric Strength Results (Effects of Thermal Cycling)

These test units utilized a pad thickness of 3 x 40 g, but the nominal 0.25-inch margin was retained. Based on this larger capacitor size, a maximum of eight units were process per "gel impregnation run". We again utilized DPB and DOP plasticizers, and for 6 of the 7 groups utilized "259" as the cross-linking material. The remaining run used PBD for cross-linking.

The STB comparisons of thermal cycled and virgin capacitors are shown below in Table 4. All of the 7 subgroups indicated improved performance (after thermal cycling) as compared to the prior capacitor test group. Failure analyses indicated that by far the predominant failure mode for the thermal cycled units was margin arc over. Since the margins on these units were the same as the prior smaller capacitors, we attribute this improved performance to better equipment and process improvements, rather than to the thicker dielectric pad (120 g vs. 96 g). Taken in the aggregate, the average subgroup decrease due to thermal cycling was only about 0.5 kV, as compared to the "virgin" units.

Table 4

Effect of Thermal Cycling on STB					
Composition	DBP/259	DOP/259	DBP/PBD	DBP/259*	DOP/259**
Thermal Cycled					
Average	7.31	8.66	10.63	9.46	7.33
Standard Deviation	1.03	0.67	1.33	0.92	0.94
Sample size	8	8	4	4	4
No Thermal Cycle					
Average	9.1	8.74	10.20	9.46	8.53
Standard Deviation	0.50	0.38	0.22	0.48	0.22
Sample size	8	8	4	4	3

*Different Composition

**Different thermal cure

We are "stretching the point" to be concerned with the sigma values for these small sample sizes (particularly the subgroups of 4). However, based on our average STB minus 4(σ) criteria which is representative of our sampling quantities for production lots, several of the above subgroups would yield rated voltages in the 5-6 kV range (if these numbers held for a sample size of 20 units). Results from this test group (Table 4) indicated significant performance improvements from the prior group (Table 3.)

<u>0.75 μF Capacitor Ultimate Dielectric Strength Results (Effects of Thermal Cycling)</u>

A factorial experiment was designed for the next test series. Based on results from the prior experiment (see Table 4), we decided to stay with the DBP/PBD gel composition because this mixture gave the highest STB results, even though we would like to have a sigma around 0.5 kV or less). We decided, however, to investigate a second type of PBD cross-linking material, labeled as Type II for this discussion. We also fabricated an equal number of units with the PBD, Type I (all PBD materials noted in prior tables were Type I). We wanted to see if gel/foil adhesion would be improved by the Type II material.

All of these test capacitors were wound and electroded at Custom Electronics, Inc. and then shipped to Coating Innovations for gel impregnation. Some earlier results indicated that good capacitor roll impregnation could still be obtained through the arc-sprayed pattern (even with complete end roll coverage). We felt that if the roll ends were completely covered with arc-spray, the mechanical integrity of the roll, and particularly of the somewhat delicate extended foils, would be enhanced. For that reason, we fabricated half of the units with 100% arc-spray coverage and the other half with a "stripe" on each end (about 30%) area coverage. As with the prior experiments, half of each group of units was subjected to our "standard" thermal cycle (TC) environment. The input factor variables were then (1) gel impregnation type, (2) end spray pattern and (3) TC or non-TC. The response functions were (1) STB, (2) capacitance increase, (3) change in dissipation factor and (4) insulation resistance. The results for the STB values are shown below in Table 5.

Table 5

Composition	Avg. STB (kV)	Standard Deviation	High STB	Low STB	Sample Size
DBP/PBD-type I					
Thermal Cycled	11.71	0.68	12.55	10.94	4
No Thermal Cycle	13.80	0.47	14.50	13.29	5
DBP/PBD-type II					
Thermal Cycled	11.72	0.83	12.49	10.55	4
No Thermal Cycle	13.95	0.82	14.80	12.80	5

First, we were pleased that the STB values increased with sigmas all less than 1 kV. We again see the effect of thermal cycling as evidenced by the lower STB values after TC. However, if we could maintain even these average STBs with the relatively low sigmas on larger lot samples, this would result in a capacitor with a rated voltage of greater than 7 kV (based on our "average - 4σ greater than or equal to rated voltage").

Note that there is one sample less in each of the TC groups. We decided to incorporate a 4.5 kV insulation resistance screening test. One unit from each composition group failed this screen. Defect analysis has not yet be performed on these two units. We've already discussed the effect of the TC response function. The only other response function that resulted in a noticeable effect on the STB values was the electrode pattern. On average, the "stripe pattern" resulted in about 1 kV higher STB values. This will have to be investigated further.

SUMMARY

Since this study was initiated, we have seen a general, continuous increase in resulting STB values, with particular improvements noted on thermal cycled capacitors. Process and equipment improvements have been, we believe, the main contributors. The results support our prediction that a 50%-65% volume reduction can be achieved with no reduction in performance and reliability. Larger lot sizes must be fabricated and tested to "prove this out". Obviously from the results in the tables, repeats on several gel compositions are in order, along with further study on the effect of arc spray pattern.

Since we believe we have demonstrated performance feasibility of an insitu-impregnated gel capacitor, we must also concentrate further efforts on making the process "production friendly" for larger lots (about 200 capacitors) and on reducing fabrication costs.

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REFERENCES

1. Registered trademark of the DuPont Company.
2. J. D. McBrayer and R. A. Brooks, *Dry Wrap and Fill Shaped Capacitors for Weak Link Applications*, **CARTS '92**, Tucson, AZ, March 16-19, 1992.
3. R. A. Brooks and L. R. Edwards, *A Close-Tap Design of Buried Foil Capacitors*, **CARTS '96**, New Orleans, LA, March 11-15, 1996.
4. G. H. Mauldin, *The Application of Perfluorocarbons as Impregnants for Plastic Film Capacitors,* **Proc. NASA/IEEE Symposium**, Huntsville, AL, Februrary 24-25, 1981.
5. J. O. Harris, Jr., J. C. Foster and R. W. DeLancey, *Process/Performance Comparisons of Two Electroding Systems for a Perfluorocarbon Liquid/Plastic Film Capacitor,* **CARTS Europe '88**, Dusseldorf, W. Germany, October 17-21, 1988.
6. J. O. Harris, Jr., J. C. Foster and R. W. DeLancey, *Effect of Winding Tension/Space Factor and Edge Margin on Performance of a Perfluorocarbon Liquid/Plastic Film Capacitor,* **CARTS '89**, Orlando, FL, March 13-16, 1989.
7. M. J. McDonald and M. L. Reichenbach, *Sandia's New Generation Winding Machine*, **CARTS '90**, San Francisco, CA, March 28-29, 1990.
8. J. D. McBrayer and J. S. Bowers, *Fractional-Factorial Process Development Techniques*, **CARTS '94**, Jupiter, FL, March 21-24, 1994.
9. L. R. Edwards, *Reliability Performance of Pulse Discharge Capacitors*, **CARTS '97**, Jupiter, FL, March 24-27, 1997.