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FINAL TECHNICAL REPORT

TECHNOLOGICAL DEVELOPMENT OF HIGH ENERGY DENSITY CAPACITORS

FEBRUARY 1976

NASA LEWIS RESEARCH CENTER, CONTRACT NAS 3-18925

ELECTRO-OPTICAL DATA
SYSTEMS GROUP
AEROSPACE GROUPS

HUGHES

HUGHES AIRCRAFT COMPANY
CULVER CITY, CALIFORNIA

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Project Manager, Norman T. Grier, NASA Lewis Research Center, Cleveland, Ohio

16. Abstract

The objective of this program was to develop cylindrical wound metallized film capacitors rated 2 μF 500 VDC that had energy densities greater than 0.1J/g. Polysulfone (PS) and polyvinylidene fluoride (PVF2) were selected as dielectrics. Single film PS capacitors of 0.2J/g (uncased) were made of 3.75 μm material. Single film PVF2 capacitors of 0.19J/g (uncased) were made of 6.0 μm material. Corona measurements were made at room temperature, and capacitance and dissipation factor measurements were made over the ranges 25°C to 125°C and 120 Hz to 100 kHz. Nineteen of twenty PVF2 components survived a 2500 hour DC plus AC life test. Failure analyses revealed most failures occurred at wrinkles, but some edge failures were also seen. A 0.989g case was designed. When the case was combined with the PVF2 component, a finished energy density of 0.11J/g was achieved.

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FOREWORD

This report documents all work performed by the Hughes Aircraft Company during the period 23 September 1974 to 23 October 1975 for NASA Lewis Research Center under Contract NAS 3-18925. The NASA Project Manager at Lewis Research Center was Norman T. Grier. The Program Manager at Hughes Aircraft Company was Dr. Robert D. Parker. A majority of the capacitor fabrication was conducted at Component Research, Incorporated, Santa Monica, California.

ABSTRACT

The objective of this program was to develop cylindrical wound metallized film capacitors rated 2 μF 500 VDC that had energy densities greater than 0.1J/g. Polysulfone (PS) and polyvinylidene fluoride (PVF2) were selected as dielectrics. Single film PS capacitors of 0.2J/g (uncased) were made of 3.75 μm material. Single film PVF2 capacitors of 0.19J/g (uncased) were made of 6.0 μm material. Corona measurements were made at room temperature, and capacitance and dissipation factor measurements were made over the ranges 25°C to 125°C and 120 Hz to 100 kHz. Nineteen of twenty PVF2 components survived a 2500 hour DC plus AC life test. Failure analyses revealed most failures occurred at wrinkles, but some edge failures were also seen. A 0.989g case was designed. When the case was combined with the PVF2 component, a finished energy density of 0.11 J/g was achieved.

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I. INTRODUCTION

Much research into the problems of decreasing the size and weight of electronic components for spacecraft power converters has been done in the past decade. This effort has been directed primarily at the largest and least efficient components, transformers. Not as much research has focused on capacitors, with the result that today's state-of-the-art capacitor has become, on the basis of weight and volume, the least efficient component in spacecraft power conditioners. Present and foreseeable systems dictate filter and commutator capacitor ratings of a few microfarads at voltages between 500V and 2kV. In this range of ratings the most commonly used capacitors for this application have energy densities of about 0.01 J/g. (Not a true density, the Joule per gram rating is a measure of weight efficiency.) The primary goal of this program was to increase this energy density to 0.1 J/g [45 J/lb.] in a representative 2 μF 500V rating.

THEORY

In theory, the design of a high energy-density capacitor should be quite simple. The energy density D is the stored energy divided by the weight W:

$$D = CV^2/2W \qquad (1)$$

where C is the capacitance and V the applied voltage. For the most elementary case, that of a planar capacitor of two plates where edge corrections are neglected, it is possible to write this equation as:

$$D = 4.425 \times 10^{-14} \kappa A V^2/[tW] \qquad (J/g) \qquad (2)$$

where κ is the relative dielectric constant, A the area of one plate, and t the dielectric thickness (all dimensions in centimeters). If the assumption is made that most of the capacitor weight is in the dielectric, this equation becomes:

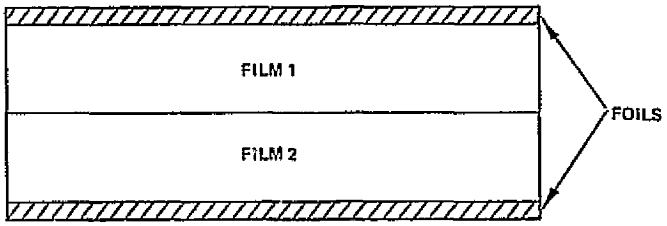
$$D = 4.425 \times 10^{-14} \kappa E^2/d \qquad (J/g) \qquad (3)$$

where E is the electric field between plates and d is the density of the dielectric. While the assumption made to derive this equation is not a particularly good one for the case of medium-voltage components, the resulting simplified equation is helpful in keeping the end goal firmly in mind. It is necessary to achieve an energy density in the dielectric greater than the density specified for the capacitor in order to build a complete working capacitor.

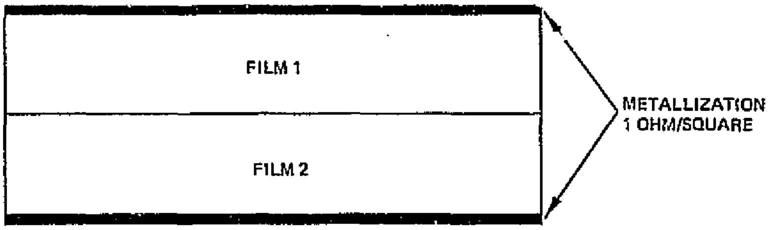
CAPACITOR CONSTRUCTION

Two constructions are commonly used in components designed for this service: ceramic, and wound plastic film. The ceramic capacitors are typically monolithic structures of barium titanate or mixtures of a variety of other high dielectric constant materials. Energy densities in these materials are controlled by the limited ability of the dielectric to support large electric fields, and further progress is likely only when advances in hot processing of these difficult materials allow the production of pore-free insulating bodies. Because of the difficult technological problems associated with improving the energy density of ceramic components, this program concentrated on film-dielectric capacitors.

The plastic film capacitors are made by a very old and well-known process. Either metallized films or unmetallized films plus foil electrodes may be used. Normally two film layers are used between electrodes, resulting in a layer construction shown in Figure 1. The finished cylindrical unit is terminated using flame-sprayed metal, giving a final unit configuration shown in Figure 2. The working core is encased and may either be impregnated or not. Design electric fields are usually well below 8×10^4 V/cm (200 V/mil).



(a) TWO-FILM/FOIL CONSTRUCTION



(b) TWO-FILM/METALLIZATION CONSTRUCTION

Figure 1. Typical two-film capacitor construction.

FAILURE MECHANISMS

It has been repeatedly shown in experiments that the intrinsic dielectric strength of a polymeric film is between 4.8×10^6 V/cm (12 kV/mil) and 7.2×10^6 V/cm (18 kV/mil). Since it has been shown that in theory the realization of a high energy density capacitor is feasible, it is fair to ask why this goal has not been achieved. After all, if one could operate a Mylar dielectric at 4.8×10^6 V/cm, one could achieve an energy density of 2.5 J/g. The answer is really three-fold. First, for 500 V applied, many materials are not made thin enough to develop large fields. Second, flaws in the dielectric itself limit the applied field that can be reliably used. Finally, most manufacturing processes yield components so imperfectly configured that they fail well below the voltage limitations of even the imperfect dielectric. In the end, both of the last two types of defects result in electrical failure. Thermal failure from dielectric heating would not be expected at these voltages and powers.

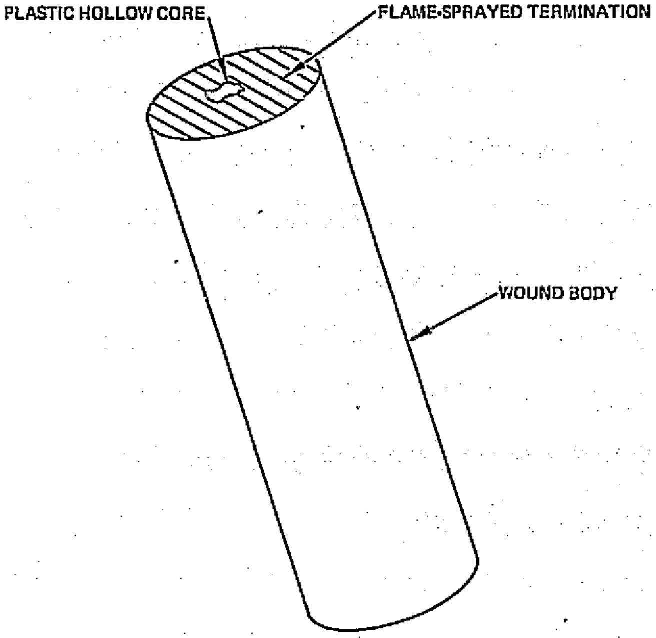


Figure 2. Typical finished capacitor active portion, cylindrical film construction.

Electrical Failure

Electrical failure in these components always takes the form of corona leading eventually to breakdown and a shorted component. The corona that causes breakdown is not exactly the same electrical discharge one hears and sees on power line insulators. True corona, such as that on power line insulators, requires at least one metallic electrode in the cavity where the discharges occur. The effect in capacitors is usually what is known as "partial internal discharges." An electric field across the dielectric produces an electric field in the gas that fills any voids, bubbles or other insulation imperfections. The gas breaks down at a much lower electric field than the other dielectric and, in addition, the field in the gas is enhanced somewhat by the difference between the dielectric constants of the gas and of the other insulation. At some point in the increase of the applied field a breakdown, or partial internal discharge, will occur in the void. The breakdown, called corona for short, produces a high temperature plasma that tends to carbonize the surrounding insulation and enlarge the void. The next time the field is applied, the void will fire at a lower field strength, and a larger current will flow, since the void is now larger. Of course, a larger amount of damage is done, and the situation never gets better. Failure occurs by shorting across the enlarged carbonized void.

A slightly different picture is needed for fluid dielectrics. If the dielectric is organic, corona carbonizes the fluid. The particles of conductive carbon line up along the field lines, causing repeated breakdowns at lower and lower fields, and finally failure. Very high electric fields or large field divergences sometimes cause the release of bubbles, either from solution or as the result of dielectric decomposition, depending upon the particular fluid and temperature.

Philosophy

Given the apparent simplicity of the theory and the contrasting difficulty raised by the observed failure mechanisms, how should this problem be approached? The conventional way high energy density capacitors are designed is to use configurations similar to previous units, build and test a large number of components, and statistically analyze the results. The usual conclusion is that a particular design has a certain life under the test conditions. Unfortunately, it is difficult to make significant advances using this method.

The method developed at Hughes for the solution of high-voltage component design problems is more phenomenological than the approach described above. This method stresses the importance of determining the causes of failure and of understanding each failure mechanism, so that components may be designed to prevent failures while still achieving other goals. An important part of this process is to build components to the best design, test to failure, perform detailed failure analyses to identify the failure mechanism, adjust the design, and begin the cycle again. Several iterations of this process may be necessary to achieve a reliable design which also meets the required specifications.

DENSITY CRITERION

As can be seen from the original statement of work, Appendix I, the energy density goal for this program was originally set down as 0.1 J/g (45 J/lb) for an assembled component. Early on it was agreed between the NASA Project Manager and the Hughes Program Manager that the principal effort should be in increasing the energy density in the dielectric, rather than in designing a lightweight case. Accordingly, it was agreed that an equivalent goal could be the design of an uncased capacitor of energy density 0.2 J/g. This would include winding, foils or metallization, and end termination, but would not include case or leadwires. For a program of this nature, it was felt that this was a more meaningful goal.

For the 2 μF 500 V component to be designed, this criterion would specify:

Winding weight:	1.25 g
Case weight:	1.25 g
Total:	2.50 g

The Hughes Program Manager does not feel a 1.25 g case is impractical, and a section has been included in this report on the fabrication of such a case. Capacitors for life tests were mounted in conventional military hermetic packages, which weigh 3 grams.

II. DIELECTRIC SYSTEMS SELECTION

The selection of dielectric materials for a high energy density 500V component is a particularly difficult task, since the relatively low operating voltage adds the problems of obtaining and working with very thin polymeric films to an already well-filled chamber of horrors. A series of simple calculations yields the curves of Figure 3, which show that a 500V component constructed of a $\kappa = 3$ film needs about a 3.75 µm (0.15 mil or 15 gauge) total dielectric thickness, while the same component made from a $\kappa = 11$ film needs a 5.9 µm (0.24 mil or 24 gauge) total dielectric thickness. The applied voltage will create a field in these thin dielectrics much larger than

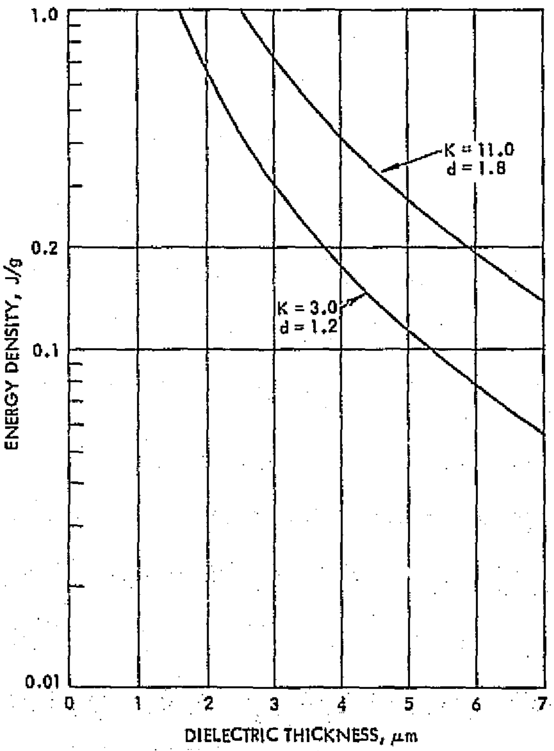


Figure 3. Energy density vs. dielectric thickness, for 500V applied

that at which conventional capacitors operate. This being so, the scope of this part of the program (Task 1 in the statement of work) was expanded from film selection to dielectric systems selection. Large electric fields imply the use of an impregnant or adhesive to fill voids and prevent corona. The intended eventual application and the extraordinary electrical conditions make selection of film, impregnant, case material, and terminations together as a system mandatory.

The statement of work required the consideration of seven different dielectric films. After some thought, the list was expanded, so that at one time eighteen materials were under consideration. These materials, their dielectric constants and their densities, are shown in Table I.

PRINCIPAL CRITERIA FOR FILM SELECTION

The two criteria used to winnow the best candidates from this array are straightforward:

1. Is the film obtainable in such a thickness that a 2 μF 500 V component with energy density 0.2 J/g or greater can be made?
2. Which two films support the lowest electrical fields when used in such a component?

The reason for the first criterion is apparent. Many films, designed principally for use in low-stress capacitors, tapes, and greenhouse coverings are not manufactured in thin enough a gauge for this application. Because this program did not encompass the manufacture of special films, such thick films were eliminated as candidates. Parylene, a vapor-phase-deposited polymer that polymerizes in situ, is out of place here. The unique problems and advantages of Parylene are discussed on page 46. The remaining films and their minimum thicknesses, are listed in Table II.

The second criterion, that of minimum necessary electric field, is based upon observation of failure mechanisms. In manufacture, most capacitors acquire some imperfections, and the starting materials have built-in defects. Since failures are always the result of electric breakdown, and since such a breakdown is a field-strength-dependent effect, it behooves the designer to work at as low a stress as possible, consistent with other

TABLE I. CANDIDATE DIELECTRIC FILMS

Material	Dielectric Constant	Density
*Polyvinylidene Fluoride (PVF2)	11.0	1.80
*Polysulfone	3.1	1.24
*Polycarbonate	3.0	1.20
*Teflon (PTFE)	2.0	2.20
*Polyethylene Terephthalate (Mylar)	3.2	1.40
*Polyimide (Kapton)	3.5	1.43
*Parylene N	2.65	1.1
*Parylene C	3.15	1.29
Nylon 6	3.7	1.15
Nylon 12	3.8	1.0
Polyethylene	2.2	0.9
Polypropylene	2.0	0.9
Polystyrene	2.4	1.0
Vinyl Chloride/Acetate	4.0	1.2
Vinylidene Chloride (Saran)	4.5	1.7
Polyvinyl Chloride	4.0	1.0
Polyvinyl Fluoride	8.5	1.8
Polyurethane	5.2	1.5

*Materials required to be considered by statement of work.

TABLE II. CANDIDATE THIN DIELECTRIC FILMS

Material	Minimum Thickness, μm	Supplier
Polyvinylidene Fluoride	6.0	Kureha Chemical
Polysulfone	2.0	Peter J. Schweitzer
Polycarbonate	1.5	Peter J. Schweitzer
Mylar	3.75	DuPont

requirements. Table III shows the thickness of dielectric required, and the electric field, for a 0.2 J/g density in each of the four films from Table II.

By this criterion it is clear that the best film is polyvinylidene fluoride (PVF2), and tied for second best are polysulfone and polycarbonate. Choice between these two materials must be on secondary considerations.

SECONDARY CRITERIA

To select from among a number of nearly identical films called out by the principal tests, a number of secondary criteria may be used. These criteria are, in approximate order of importance for this application: service temperature, dissipation factor, and fabrication ease. It should be remembered that these are secondary criteria only for this program, where the attainment of the desired energy density is the primary goal. In a subsequent program secondary criteria may become primary goals.

Service Temperature

The statement of work specifies a service temperature limit of 125°C. Upper service temperature is important for several reasons. As is well known, the insulation resistance of most materials decreases with increasing temperature, and this results in decreasing dielectric strength with increasing temperature. Experience with standard film capacitors

TABLE III. ELECTRIC FIELD FOR 0.2 J/g ENERGY DENSITY

Material	Required Thickness	Electric Field
Polyvinylidene Fluoride	5.81 µm	8.6×10^5 V/cm
Polysulfone	3.72 µm	1.34×10^6 V/cm
Polycarbonate	3.72 µm	1.34×10^6 V/cm
Mylar	3.56 µm	1.4×10^6 V/cm

The values in this Table were computed using equation 3, and 60 Hz 25°C values of κ and density.

shows an increase in leakage current and a decrease in average life for elevated temperature service. Second, each material has as an intrinsic property a temperature above which irreversible physical and chemical changes occur. Obviously, the operation of a material above this temperature will lead to failure.

Finally, plastic films have a unique thermally-triggered failure mechanism: the thermal activation of flaws in the film. This mechanism is related to the decrease in dielectric strength, the difference being that dielectric strength is a bulk property while flaws are local phenomena. The flaws are as large as 625 μm (25 mils) in diameter and show up well as inhomogeneous areas when observed by polarized light microscopy. The flaws are sites of excessive localized conductivity and eventual thermal breakdown of the dielectric.

On the basis of these criteria, the films would rank in order of desirability: PVF2, polysulfone, polycarbonate, Mylar. Mylar has a higher service temperature than polycarbonate, but is known to have flaws. Polysulfone has the highest service temperature of all but PVF2. PVF2 can be used to 160°C, but does exhibit some problems, to be discussed below.

Dissipation Factor

In this application dissipation factor is important only in that the components must not overheat at the maximum rated AC current. This rating is 5 amperes at 10 kHz.

The equivalent series resistance of the capacitor may be found from the dissipation factor as:

$$R_s = DF/C_s \omega \tag{4}$$

And the average power loss is simply:

$$\overline{P} = I^2 R_s \tag{5}$$

These components are the physical size of a five-watt resistor, and can safely dissipate powers of the order of a watt. This implies that the dissipation factor must be of the order of 1 percent or smaller at 10 kHz. This requirement is met by all films under consideration, as will be shown.

Fabrication Ease

This is a large category, and the judgements made are purely subjective. The basic criterion is, can capacitors be made out of this material repeatably and reliably. After some experience, the films of Table II can be ranked, with polysulfone, polycarbonate, and Mylar almost equal, and PVF2 a distinct second. PVF2 is related to Saran, and is therefore extremely limp and difficult to work with.

CHEMICAL COMPATIBILITY

Because of the complicated interactions between materials in capacitors during long service in a space environment, it was decided to investigate the chemical compatibility of various candidate materials. This study addressed directly the problems involved, but it did have certain limitations. The length of the study was short compared to typical spaceflight component system life. Additionally, most of the tests were run in air, rather than in the type of environment that might be expected in a hermetic component case; cost was a factor here.

A series of experiments was conducted to establish the compatibility of capacitor films and liquid dielectric impregnants. The objectives were to establish compatible combinations, to identify service temperature limits, and to identify the kinetic rates associated with deterioration processes. Four films and three liquids were studied. The films were:

- Cast polycarbonate - P. J. Schweitzer
- Cast polysulfone - P. J. Schweitzer
- Extruded polysulfone - P. J. Schweitzer
- Polyvinylidene fluoride - Kureha Chemical

The liquids were:

- Mineral oil (transformer grade) - GB-100M, Golden Bear Division, Witco Chemical Company
- Alkylated benzene - D. O. 100, Chevron Chemical Company
- Aryl-alkyl silicone compounds - SF-1050, Silicone Products Division, General Electric Company

To evaluate the interactions between the films and liquids, the following determinations were made. On films,

- I-V characteristic
- tensile strength
- weight change
- appearance

On liquids,

- volume resistivity
- electrical breakdown strength
- water concentration
- appearance

The following sections summarize the evaluation procedures used in the studies as well as the experimental results including the compatible film-fluid and temperature combinations.

Experimental Procedure

Following are summaries of the test procedures and exposure conditions used in the compatibility evaluations.

Film Electrical Resistivities

The resistances were determined using a voltmeter-ammeter method. In these determinations five layers of the film were retained between unguarded, 2-inch diameter disc electrodes. Voltages of 100 VDC and 1000 VDC were applied and the leakage currents recorded after an eight minute settling time. The volume resistivities were then calculated using the expression:

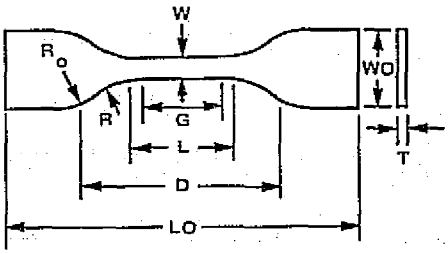
$$\rho_e = \frac{V}{i} \cdot k \text{ (ohm-cm)} \qquad (6)$$

where $k = \frac{A}{t}$, A the electrode area, and t the combined thickness of the five layers. For the post-exposure determinations, the samples were first rinsed with hexane and oven dried 30 minutes at 75°C. This was to remove any surface oil films which could have functioned as a shunt resistance. The initial determinations prior to exposure were made on as received, unconditioned film samples.

Film Tensile Strength

The test sample configurations used in these determinations conformed to those described in ASTM D638-71a, Tensile Properties of Plastics, Type IV, for samples less than 0.16 inch thick. This configuration, shown in Figure 4, provides a narrow section length of 1.3 inches and a corresponding width of 0.25 inch. All samples were cut with the major axis parallel to the machine direction of the film. The film strengths were measured on individual samples at a strain rate of 0.5 inch per minute. The strengths recorded were ultimate or break values. The tensile strengths were calculated using the expression:

$$\sigma_B = \frac{L}{0.25 \times t} \text{ (psi)} \qquad (7)$$



W–WIDTH OF NARROW SECTION	0.25
L–LENGTH OF NARROW SECTION	1.30
WO–WIDTH OVER-ALL	0.75
LO–LENGTH OVER-ALL	4.5
G–GAGE LENGTH	1.00
D–DISTANCE BETWEEN GRIPS	2.5
R–RADIUS OF FILLET	0.56
RO–OUTER RADIUS	1.00

Figure 4. Film tensile strength test sample configuration (per ASTM D-638)

where σ_B is the tensile strength at break, t is the film thickness in inches, and L is length in inches. Post exposure preparation was similar to that performed on the film resistivity samples. The initial determinations were made using the as-received film which had no prior special conditioning. Two samples were used in both the pre- and post-exposure determinations.

Weight Change Determinations.

These determinations were made using five 2-1/4 x 2-1/4 inch pieces in combination. These were weighted on an analytical balance to an accuracy of 0.5 milligram with weights recorded to the nearest milligram. The post-exposure samples were prepared similar to the film resistivity samples to eliminate surface absorbed liquid, thereby providing a more accurate determination of the weight changes of the film. As in the other determinations, the film samples were used in the as-received condition.

Appearance

The general appearance including color and physical character were noted before and after the exposures.

Fluid Volume Resistivity

The resistivities were determined using the voltmeter-ammeter method and a Balsbaugh guarded electrode dielectric test cell. The fluid was placed in the cell, 100 VDC applied, and the current recorded after 8 minutes. The resistivity was calculated using (6) with k now equal to the cell constant of the liquid test cell. The cell constant here was 1100. Single measurements were made on the pre- and post-exposure samples.

Fluid Electrical Breakdown Strength

These determinations were made using spherical electrodes and a 60 Hz potential. The electrodes were gapped at 25 mils and immersed in a 40 milliliter sample of the fluid. The voltage was increased continuously at 0.5 kV per second until breakdown. Five such breakdowns were conducted on each sample, allowing a two minute interval between events. The electrical breakdown strength was then reported based on the average of five values and is expressed as the average-field strength:

$$E_{avg} = \frac{V_{ac}}{a} \text{ (volts per mil)} \tag{8}$$

where V_{ac} is the average rms voltage and a the electrode spacing, here 25 mils. Only one sample was measured for each fluid both pre- and post-exposure.

Fluid Water Concentration

Water determinations were made using the Karl Fischer method. The procedure is described in ASTM E203-64, "Water Using Karl Fischer Reagent." Three samples were measured for each fluid before and after exposure. The reported values are the average for them.

Fluid Appearance

The general appearance including color, viscosity, sedimentation, and gellation were recorded for each fluid before and after exposure. It should be noted that the fluids were all used as received.

The film-fluid exposures were conducted at 25°, 75° and 125°C. Samples of the films were placed in glass containers and filled with the fluid. All of the films were tested together. The evaluated combinations are shown in Table IV. The tests were conducted in covered but unsealed containers and were continually exposed to air throughout the test period. The exposure period used in these evaluations was 640 hours. Each of the determinations previously described was performed at the beginning and end of the exposure period.

Experimental Results

The experimental results of the capacitor film-dielectric fluid compatibilities are summarized in Tables V through XX. Tables V through XVI provide summaries of the film characteristics while Tables XVII through XX describe the fluid properties. A brief review of the results follows.

TABLE IV. TEST COMBINATIONS USED IN FILM-FLUID COMPATIBILITY STUDIES

Exposure Temperature Test Fluid	25°C	75°C	125°C
Alkylated benzene	CPC, CPS, EPS, PVF	CPC, CPS, EPS, PVF	CPC, CPS, EPS, PVF
Mineral oil	CPC, CPS, EPS, PVF	CPC, CPS, EPS, PVF	CPC, CPS, EPS, PVF
Silicone fluid	CPC, CPS, EPS, PVF	CPC, CPS, EPS, PVF	CPC, CPS, EPS, PVF

CPC - cast polycarbonate
CPS - cast polysulfone
EPS - extruded polysulfone
PVF2 - polyvinylidene fluoride

The film resistivity determinations were made as a means of identifying decomposition products of reduced or non-dielectric character. Such materials could result from decomposition of the film, or the absorption of fluid into the film which was itself of reduced properties, or reaction with the film to yield degraded products. A review of the experimental results in Tables V, VI, and VII indicate some changes have occurred to all of the films in each of the fluids. The slight reductions noted are probably primarily due to liquid absorption. If uniformly distributed at low concentrations, the absorbed liquid having a lower resistivity would act as a series-parallel resistance in combination with that of the film as shown in Figure 5. The result would be the lowering of the film resistance as observed experimentally. The higher resistances noted at 100 VDC and 75°C are attributed to reduced absorbed moisture in these samples, resulting from the higher temperatures. At 125°C, the lower resistance values are attributed to the absorbed fluid which may have undergone some deterioration at this temperature. The lower resistivities noted at 1000 VDC are attributable to two possible effects: electrode edge effects, or additional charge injections — both because of the higher electric field. The 1000 VDC measurements are

TABLE V. RESISTIVITY PROPERTIES OF CAPACITOR FILMS EXPOSED TO AN ALKYLATED BENZENE

Capacitor Film	Exposure Temperature °C	Volume Resistivity, ohm-cm			
		Applied Voltage - 100 VDC		Applied Voltage - 1000 VDC	
		Before	After	Before	After
polysulfone, extruded	25°	4×10^{16}	7×10^{15}	—	2×10^{15}
	75°		8×10^{15}		2×10^{15}
	125°		6×10^{15}		*
polysulfone, cast	25°	3×10^{16}	6×10^{15}	—	2×10^{15}
	75°	—	6×10^{15}		2×10^{15}
	125°	—	4×10^{15}		2×10^{15}
polycarbonate, cast	25°	4×10^{16}	7×10^{15}		2×10^{15}
	75°		2×10^{16}		6×10^{15}
	125°		3×10^{15}		2×10^{15}
polyvinylidene fluoride	25°	1×10^{16}	4×10^{15}		5×10^{14}
	75°		2×10^{15}		3×10^{14}
	125°		3×10^{15}		2×10^{14}

* - Electrical breakdown occurred through sample

TABLE VI. RESISTIVITY PROPERTIES OF CAPACITOR FILMS EXPOSED TO MINERAL OIL

Capacitor Film	Exposure Temperature °C	Volume Resistivity, ohm-cm			
		Applied Voltage - 100 VDC		Applied Voltage - 1000 VDC	
		Before	After	Before	After
polysulfone, extruded	25°	4×10^{16}	7×10^{15}	—	2×10^{15}
	75°	—	8×10^{15}		2×10^{15}
	125°	—	7×10^{15}		2×10^{15}
polysulfone, cast	25°	3×10^{16}	4×10^{16}		2×10^{15}
	75°	—	7×10^{15}		2×10^{15}
	125°	—	5×10^{15}		8×10^{15}
polycarbonate, cast	25°	4×10^{16}	5×10^{15}		2×10^{15}
	75°	—	6×10^{15}		4×10^{15}
	125°	—	2×10^{16}		2×10^{15}
polyvinylidene fluoride	25°	1×10^{16}	7×10^{15}		4×10^{14}
	75°		2×10^{15}		2×10^{14}
	125°		3×10^{15}		3×10^{14}

TABLE VII. RESISTIVITY PROPERTIES OF CAPACITOR FILMS EXPOSED TO SILICONE FLUID

Capacitor Film	Exposure Temperature °C	Volume Resistivity, ohm-cm			
		Applied Voltage - 100 VDC		Applied Voltage - 1000 VDC	
		Before	After	Before	After
polysulfone, extruded	25°	4×10^{16}	9×10^{15}	—	2×10^{15}
	75°	—	1×10^{16}		3×10^{15}
	125°	—	4×10^{15}		2×10^{15}
polysulfone, cast	25°	3×10^{16}	5×10^{15}	—	2×10^{15}
	75°		2×10^{16}		4×10^{15}
	125°		6×10^{15}		2×10^{15}
polycarbonate, cast	25°	4×10^{16}	9×10^{15}		2×10^{15}
	75°		2×10^{16}		4×10^{15}
	125°		1×10^{16}		5×10^{15}
polyvinylidene fluoride	25°	1×10^{16}	2×10^{15}		4×10^{14}
	75°		2×10^{15}		3×10^{14}
	125°		4×10^{15}		3×10^{14}

TABLE VIII. CHANGES IN TENSILE STRENGTHS OF CAPACITOR FILMS RESULTING FROM EXPOSURE TO AN ALKYLATED BENZENE

Capacitor Film	Tensile Strength - PSI								
	Exposure - 25°C			Exposure - 75°C			Exposure - 125°C		
	Before	After	Δ, %	Before	After	Δ, %	Before	After	Δ, %
polysufone, extruded	10,600	10,000	-6	see 25°C	10,000	-6	see 25°C	11,200	+6
polysulfone, cast	11,700	9,000	-23	see 25°C	9,700	-17	see 25°C	10,000	-14
polycarbonate, cast	25,600	28,300	+10	see 25°C	30,000	+17	see 25°C	24,600	-4
polyvinylidene fluoride	21,400	20,000	-6	see 25°C	24,200	+13	see 25°C	23,800	+11

TABLE IX. CHANGES IN TENSILE STRENGTH OF CAPACITOR FILMS RESULTING FROM EXPOSURE TO MINERAL OIL

Capacitor Film	Tensile Strength - PSI								
	Exposure - 25°C			Exposure - 75°C			Exposure - 125°C		
	Before	After	Δ, %	Before	After	Δ, %	Before	After	Δ, %
polysulfone, extruded	10,600	11,700	+10	see 25°C	10,900	+3	see 25°C	13,100	+24
polysulfone, cast	11,700	10,600	-9	see 25°C	12,000	+2	see 25°C	13,000	+11
polycarbonate, cast	25,600	32,500	+27	see 25°C	27,200	+5	see 25°C	23,800	-7
polyvinylidene fluoride	21,400	16,900	-21	see 25°C	25,500	+19	see 25°C	16,600	-22

TABLE X. CHANGES IN TENSILE STRENGTHS OF CAPACITOR FILMS RESULTING FROM EXPOSURE TO A SILICONE COMPOUND

Capacitor Film	Tensile Strength - PSI								
	Exposure - 25°C			Exposure - 75°C			Exposure - 125°C		
	Before	After	Δ, %	Before	After	Δ, %	Before	After	Δ, %
polysulfone, extruded	10, 600	9, 200	-13	see 25°C	10, 000	-6	see 25°C	10, 400	-2
polysulfone, cast	11, 700	11, 000	-6	see 25°C	9, 400	-20	see 25°C	11, 000	-6
polycarbonate, cast	25, 600	23, 800	-7	see 25°C	27, 500	+7	see 25°C	20, 800	-19
polyvinylidene fluoride	21, 400	23, 800	+11	see 25°C	23, 300	+9	see 25°C	23, 000	+7

TABLE XI. WEIGHT CHANGES OF CAPACITOR FILMS EXPOSED TO ALKYLATED BENZENE

Capacitor Film	Film Weight* - Milligrams								
	Exposure Temp. - 25°C			Exposure Temp. - 75°C			Exposure Temp. - 125°C		
	Before	After	Δ, %	Before	After	Δ, %	Before	After	Δ, %
polysulfone, extruded	136	138	+1.5	138	139	+0.7	136	137	+0.7
polysulfone, cast	183	184	+0.5	181	184	+1.6	182	192	+5.5
polycarbonate, cast	126	127	+0.8	126	128	+1.6	126	133	+5.6
polyvinylidene fluoride	353	357	+1.1	339	340	+0.3	335	336	+0.3

* - Combined weight of (5) 2-1/4 x 2-1/4 inch pcs.

TABLE XII. WEIGHT CHANGES OF CAPACITOR FILMS EXPOSED TO MINERAL OIL

Capacitor Film	Film Weight* - Milligrams								
	Exposure Temp. - 25°C			Exposure Temp. - 75°C			Exposure Temp. - 125°C		
	Before	After	Δ, %	Before	After	Δ, %	Before	After	Δ, %
polysulfone, extruded	138	140	+1.4	162	166	+2.4	153	139**	**
polysulfone, cast	178	180	+1.1	180	182	+1.1	178	179	+0.6
polycarbonate, cast	125	125	0	128	130	+1.6	127	133	+4.7
polyvinylidene fluoride	328	329	+0.3	276	278	+0.7	335	338	+0.8

* - Combined weight of (5) 2-1/4 x 2-1/4 inch pcs.

** - Weighing error.

TABLE XIII. WEIGHT CHANGES OF CAPACITOR FILMS EXPOSED SILICONE COMPOUND

Capacitor Film	Film Weight* - Milligrams								
	Exposure Temp. - 25°C			Exposure Temp. - 75°C			Exposure Temp. - 125°C		
	Before	After	Δ, %	Before	After	Δ, %	Before	After	Δ, %
polysulfone, extruded	136	138	+1.5	137	139	+1.4	139	140	+0.7
polysulfone, cast	176	179	+1.7	178	182	+2.2	180	181	+0.6
polycarbonate, cast	127	128	+0.8	126	128	+1.6	125	126	+0.8
polyvinylidene fluoride	330	331	+0.3	336	338	+0.6	351	357	+1.7

* - Combined weight of (5) 2-1/4 x 2-1/4 inch pcs.

TABLE XIV. CHANGES IN CAPACITOR FILM APPEARANCE RESULTING FROM EXPOSURE TO AN ALKYLATED BENZENE

Capacitor Film	Appearance Before Exposure	Post Exposure Appearance		
		Exposure Temp. - 25°C	Exposure Temp. - 75°C	Exposure Temp. - 125°C
polysulfone, extruded	Clear, colorless, transparent film	No visual change	No visual change	No visual change
polysulfone, cast	Clear, transparent light blue cast	No visual change	Very slight loss of color, no other changes	Almost complete loss of color, no other changes
polycarbonate, cast	Clear light green, slightly less transparent than blue	No visual change	Very slight loss of color, no other changes	Complete loss of color, some shrinkage
polyvinylidene fluoride	Clear, colorless transparent	No visual change	No visual change	Very slight yellowish tint, no other changes

TABLE XV. CHANGES IN CAPACITOR FILM APPEARANCE RESULTING FROM EXPOSURE TO MINERAL OIL

Capacitor Film	Appearance Before Exposure	Post Exposure Appearance		
		Exposure Temp. - 25°C	Exposure Temp. - 75°C	Exposure Temp. - 125°C
polysulfone, extruded	See Table XIV	No visual change	No visual change	No visual change
polysulfone, cast	See Table XIV	No visual change	Very slight loss of color, no other changes	Almost complete loss of color, no other changes
polycarbonate, cast	See Table XIV	No visual change	Very slight loss of color, no other changes	Complete loss of color, some shrinkage
polyvinylidene fluoride	See Table XIV	No visual change	No visual change	Slight amber tint, no other changes

TABLE XVI. CHANGES IN CAPACITOR FILM APPEARANCE RESULTING FROM EXPOSURE TO A SILICONE COMPOUND

Capacitor Film	Appearance Before Exposure	Post Exposure Appearance		
		Exposure Temp. - 25°C	Exposure Temp. - 75°C	Exposure Temp. - 125°C
polysulfone, extruded	See Table XIV	No visual change	No visual change	No visual change
polysulfone, cast	See Table XIV	No visual change	Very slight loss of color, no other change	Moderate loss of color, no other change
polycarbonate, cast	See Table XIV	No visual change	Very slight loss of color, no other change	Moderate loss of color, some shrinkage
polyvinylidene fluoride	See Table XIV	No visual change	No visual change	No visual change

TABLE XVII. ELECTRICAL RESISTIVITY PROPERTIES OF THE THREE FLUIDS USED IN THE COMPATIBILITY DETERMINATIONS

Dielectric Liquid	Volume Resistivity - ohm-cm								
	25°C Exposure			75°C Exposure			125°C Exposure		
	100 Volts Before	100 Volts After	1000 Volts After	100 Volts Before	100 Volts After	1000 Volts After	100 Volts Before	100 Volts After	1000 Volts After
Alkylated Benzene	4×10^{15}	3×10^{15}	7×10^{15}	See 25°C	4×10^{15}	1×10^{16}	See 25°C	1×10^{14}	2×10^{14}
Mineral Oil	3×10^{15}	1×10^{15}	2×10^{15}	See 25°C	2×10^{15}	5×10^{15}	See 25°C	7×10^{13}	2×10^{14}
Silicone Compound	2×10^{13}	2×10^{13}	1×10^{14}	See 25°C	2×10^{13}	7×10^{13}	See 25°C	9×10^{12}	5×10^{13}

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TABLE XVIII. ELECTRICAL BREAKDOWN CHARACTERISTICS OF DIELECTRICS USED IN FILM EXPOSURE STUDIES

Dielectric Liquid	Electrical Breakdown Voltages and Stresses								
	25°C Exposure			75°C Exposure			125°C Exposure		
	Before 1 kVac . . . 5 Avg Stress*	After	Δ% - Stress	Before	After	Δ% - Stress	Before	After	Δ% - Stress
Mineral Oil	18.0	17.5		See 25°C ↓	18.0		See 25°C ↓	18.5	
	19.5	17.5			21.0			20.0	
	20.0	17.0	-6.		20.5	+4.		20.0	-3.
	19.0	18.5			16.0			16.5	
	17.0	17.0			21.5			15.5	
	748	700			776			724	
Alkylated Benzene	23.0	19.0			19.0			20.0	
	18.0	23.0			23.0			21.0	
	16.0	22.0	+12.		19.0	+15.		16.0	+3.
	20.0	22.0			23.0			20.5	
	15.0	17.5			22.0			17.5	
	736	828			848			760	
Silicone Compound	13.0	18.0			18.0			15.0	
	16.5	17.5			18.5			7.0	
	15.0	18.5	+4.		18.0	+15.		6.0	-32.
	17.0	14.0			18.0			7.5	
	17.0	14.0			17.5			18.0	
	628	656			720			428	

* = $kV_{(AVG)} \div 25$ mils = volts (RMS)/MIL

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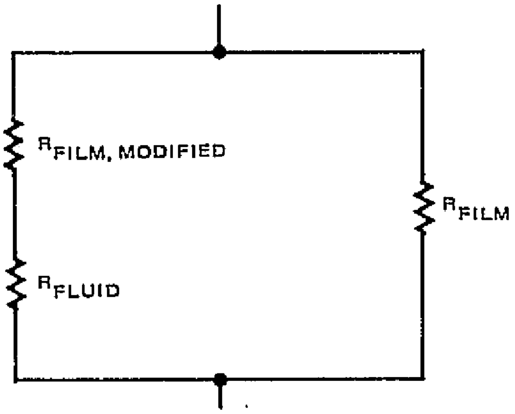
TABLE XIX. MOISTURE CONTENT OF DIELECTRIC LIQUIDS USED IN FILM EXPOSURE STUDIES

Dielectric Liquid	Water Concentration - Parts Per Million								
	25°C Exposure			75°C Exposure			125°C Exposure		
	Before	After	Δ%	Before	After	Δ%	Before	After	Δ%
Alkylated Benzene	70	78	+11	See 25°C	71	+1	See 25°C	74	+6
Mineral Oil	55	68	+24	See 25°C	53	-4	See 25°C	71	+29
Silicone Compound	79	105	+33	See 25°C	60	-24	See 25°C	51	-23

TABLE XX. GENERAL CHARACTERISTICS OF DIELECTRIC LIQUIDS

Dielectric Liquid	Initial Characteristics	Characteristics After Exposure		
		25°C	75°C	125°C
Alkylated Benzene	Clear Water - white, low viscosity	No change - color or viscosity	No change - color or apparent viscosity	Some darkening to a light yellow, also some tanish striations. No apparent viscosity change.
Mineral Oil	Very light golden-yellow	No change - color or viscosity	Some darkening to a dark golden yellow. No viscosity change	Darkening to a light tan with some dark tan striations. No apparent viscosity change.
Silicone Compound	Clear water - white, very low viscosity	No change - color or viscosity	No change - color or apparent viscosity	No change in color or apparent viscosity



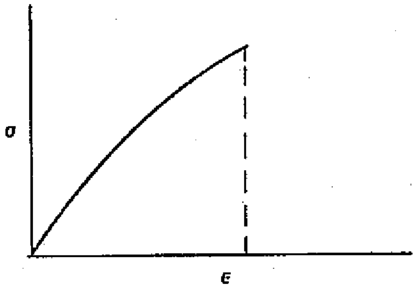
$(R_{FLUID} + R_{FILM, MOD}) < R_{FILM}$

Figure 5. Equivalent resistance circuit for film containing absorbed dielectric fluid

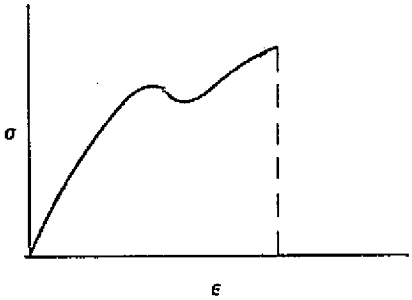
probably more representative of the film performance in a device since these stresses more closely correspond to the stress levels within the devices. The changes noted for any of the film-fluid combinations are not considered of a magnitude that would be catastrophic to the performance of a capacitor, but do reflect the occurrence of changes which, if of a continuing nature, could be eventually deleterious to such devices.

The film tensile strengths are summarized in Tables VIII, IX, and X. Like the volume resistivity determinations, these measurements were performed as a means of identifying physical changes of the films which would indicate a change in the basic structure of the films.

Some significant observations were made. First, the elongation of the cast polysulfone films was measurably reduced in all of the fluids at the elevated temperatures. This suggests a molecular reorientation which would probably affect the dielectric constant and hence the capacitance of a device exposed to such conditions. Some changes in the elongation characteristics and plastic region were also noted for the cast polycarbonate at 125°C in each of the fluids. Here the elongation was slightly reduced but the ultimate values became measurably higher than the yields. The shape of the stress-strain curve changed as shown in Figure 6. As with the cast polysulfone such changes are probably associated with molecular rearrangements which



(a) 25° AND 75°C EXPOSURE



(b) 125°C EXPOSURE

Figure 6. Stress-strain characteristics for the cast polycarbonate film

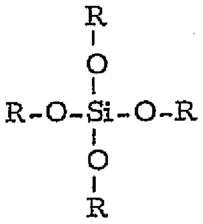
could alter the capacitance value of a device. The cause of significant reductions in the strength of the polyvinylidene fluids in mineral oil at 25° and 125°C, but not 75°C, is not known. In all cases the values reported in the tables are ultimate values and in most cases are within 15 percent of the yield strengths. Of the four films the extruded polysulfone yielded the least change in the three fluids at the three temperatures.

The weight variations are summarized in Tables XI, XII, and XIII. These test determinations were intended to merely indicate whether weight varying processes, either additive or subtractive, were occurring. Weight loss phenomena would be associated with physical processes such as moisture elimination or dissolution, and chemical processes such as reactive dissolution. Weight gain could also be physical, as in fluid absorption, or chemical, as in absorption followed by in situ reaction. The results in these experiments indicate primarily physical phenomena with two processes occurring. The first involves the moisture elimination resulting in weight reduction. The other is fluid absorption, acts concurrently and increases the weight. As seen in the tables, the fluid absorption dominates with the net effect of an increase in sample weight. These weight increases do confirm the absorption of fluid, and thus support the postulates for the causes of the results observed in the resistance and tensile strength determinations.

The changes in the physical appearance of the films, given in Tables XIV, XV, and XVI, also support the contention that variations did occur. The loss of color noted for the cast polycarbonate and the cast polysulfone could be indicative of a chemical reaction with the fluids as a thermal degradation of the dye. The shrinkage noted for the cast polycarbonate at 125°C is further evidence of structural rearrangements of this film.

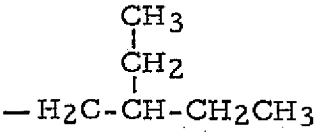
Variations in fluid properties are also important for liquid impregnated film capacitors. Like the film resistivity determinations, the fluid resistance measurements were conducted to identify changes which could be deleterious to an impregnated capacitor. A review of the results in Table XVII shows all of the fluids to be essentially unchanged at the 25° and 75°C conditions. However, at 125°C, both the alkylated benzene and the mineral oil show significant changes. These changes are probably caused by oxidative reactions which yield acids having low ionic character. Such are known to occur with hydrocarbons in an oxidizing environment similar to those existing in these experiments. It should be noted that in most capacitor applications such severe oxidizing conditions would not exist and that these results can be considered to represent the most severe set of conditions.

Thermally, the silicone fluid was the most stable, exhibiting essentially no changes which are manifest by reduced resistivities. It should be noted that the silicone compound used in these evaluations may have contained up to one percent of an orthosilicate ester of the type:



$$\begin{array}{c} R \\ | \\ O \\ | \\ R\text{-}O\text{-}Si\text{-}O\text{-}R \\ | \\ O \\ | \\ R \end{array}$$

where R is primarily



$$\begin{array}{c} CH_3 \\ | \\ CH_2 \\ | \\ -H_2C\text{-}CH\text{-}CH_2CH_3 \end{array}$$

This silicate ester may account for the lower resistivity of the silicone compound.

Finally, the higher resistivities produced by each of the fluid at 1000 VDC are attributed to space charge phenomena. As the electric field increases the space charge region at one or both electrodes increases in dimensions, and with this increase there is a corresponding reduction in the current. Such effects would be expected to be even greater in a capacitor where the electric fields could be as much as 5-10 times larger than those occurring in these determinations.

The electrical breakdown determinations for the fluids were performed to identify changes which could measurably alter this property. While the electrical breakdown properties of the fluid impregnant are not usually of paramount concern in the materials selection and design of a capacitor, major reductions in this property during the life of a device would be considered in the selection. The changes in the breakdown characteristics given in Table XVIII show only the silicone fluid at 125°C to have undergone a significant reduction. The behavior here is somewhat similar to that of both dimethyl and methyl phenyl silicone fluids of the extended chain type. These materials will commonly demonstrate a 40 to 60 percent reduction in the second arc potential. The cause for this behavior in these experiments may result from some polymerization occurring at 125°C which was absent at the lower temperatures. However, if only the first arc potentials are considered, then all of the fluids at the three temperatures can be considered to have experienced negligible changes.

Table XIX presents a summary of the water contents for each fluid and exposure condition. These were taken initially to provide a reference point which might be used in the control of materials for use in manufactured assemblies. The post exposure results were to act as indicators of the conditions occurring during the exposure. The results with some exceptions are as would be predicted. The 25°C room temperature exposure at 50 to 60 percent R. H. generally showed some increase in water content while the elevated temperature samples at the lower humidities generally had lower moisture contents. The principal exception is the mineral oil at 125°C which showed an approximate 30 percent increase. Such an observed increase may be due to procedural errors which limit the accuracy of this method to about ±10 to 15 parts per million. The results do, however, provide the general reference range originally intended.

Table XX presents a summary of the changes in the physical appearance of the fluids resulting from the exposures. At the lower temperatures, 25 and 75°C, no changes were noted in any of the three fluids. At 125°C the alkylated benzene and the mineral oil were measurable discolored, which is typical of hydrocarbon oxidation. The silicone, while not discolored at 125°C, seems to have increased slightly in viscosity; this observation was not verified quantitatively. In all cases these visual changes support the changes indicated in other fluid property determinations.

The experimental results summarized in terms of film-fluid compatibilities are presented in Tables XXI and XXII. The results show only marginal compatibilities for selected combinations at 125°C. For these marginal combinations the fluid seems to be the limiting material. At the lower exposure temperatures, 25 and 75°C, the observed changes in both films and fluids are minimal and all of the combinations are considered compatible under these conditions.

Finally, attempts to identify activation energies for the processes producing the observed changes have not been effective. The changes have generally been sufficiently small and varied (i. e., non-directional) so as to

TABLE XXI. FILM-FLUID COMPATIBILITIES AT 125°C

Film	Liquid		
	Mineral Oil	Alkyl Benzene	Silicone Compound
Extruded polysulfone	I (L)	M (L)	M (L)
Cast sulfone	I (F, L)	M (L)	M
Cast polycarbonate	I (F, L)	I (F)	I (F)
PVF2	I (L)	M (L)	I (F, L)

C - Compatible
M - Marginal
I - Incompatible

( ) - failing component
F - film
L - liquid

TABLE XXII. FILM-FLUID COMPATIBILITIES AT 25° and 75°C

Film	Liquid		
	Mineral Oil	Alkyl Benzene	Silicone Compound
Cast carbonate	C*	C	C
Cast sulfone	C	C	C
Extruded sulfone	C	C	C
PVF2	C	C	C

*See Table XVIII key for definitions

eliminate the identification of the energies associated with the changes. To identify these energy values would require considerably more experiments.

Conclusions

Based on these experiments, the following conclusions are drawn:

None of the combinations appears suitable for extended or continuous use at 125°C in open air. If such requirements were to exist the extruded polysulfone carbonate-silicone fluid would have the highest probability of performing under these conditions. At the lower temperatures, all of the combinations appear capable of extended use. However, because these experimental conditions do not precisely reflect the conditions in a capacitor, test capacitors employing the films and liquids should be exposed to thermal conditioning and the performance monitored as the final indicator of compatibility.

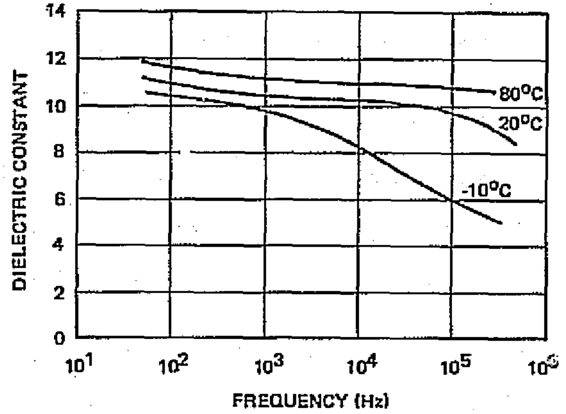
FINAL DIELECTRIC SELECTIONS

The statement of work (Appendix I) required that two different film dielectrics be chosen for further study. On the basis of the work reported above, the dielectrics chosen were polyvinylidene fluoride (PVF2) and polysulfone. This section discusses the rationale for these choices, displays the rest of the dielectric system, and sets down other pertinent information gleaned during this work.

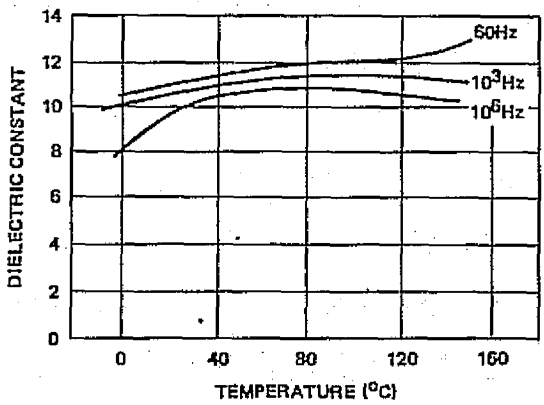
Polyvinylidene Fluoride

This material, primarily used for coverings of greenhouses, is attractive as a capacitor dielectric because of its high dielectric constant (shown in Figure 7). The stability of this property with temperature and frequency is remarkable, given the highly polar nature of the molecules needed to produce it to begin with.

It should be emphasized here that this data was taken on polyvinylidene fluoride produced by Kureha Chemical Industry of Japan under the name KF Polymer, and distributed in this country by Kreha Corporation. This material appears to have different properties than the polyvinylidene fluoride resin produced by Pennwalt in this country. The electrical properties of the



(a) DIELECTRIC CONSTANT VS FREQUENCY



(b) DIELECTRIC CONSTANT VS TEMPERATURE

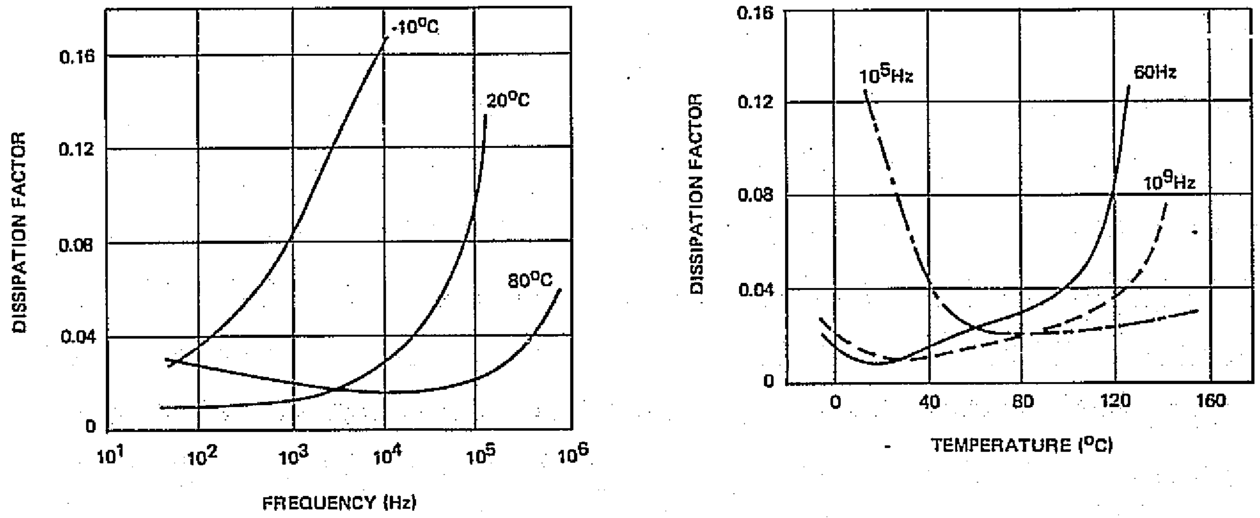
Figure 7. Dielectric constant of KF polyvinylidene fluoride

Pennwalt resin more closely resemble those of polyvinyl fluoride than of KF Polymer. The KF Polymer is an extruded biaxially oriented film; the biaxial orientation is achieved by stretching the film more than 50 percent in each direction after extrusion. This stretching and the molecular chain orientation it produces appear to be responsible for some of the unusual electrical properties of this material. The films available in this country, made from the Pennwalt resin, are solvent cast.

The large dielectric constant immediately implies a lower electric field to meet the required energy density compared to materials of lower dielectric constant. This film is obtainable in 6.0 µm (0.24 mil) thickness. In that thickness, the energy density at 500 VDC is 0.188 J/g (84.6 J/lb.), and the required electric field is 8.33×10^5 V/cm (2083 V/mil).

The dissipation factor of PVF2 is quite large and variable with temperature and frequency (Figure 8). For this application, the large dissipation factor is not critical, but for other applications it may become so.

It was previously mentioned that the KF film is a chemical relative of polyvinylidene chloride, from which Saran wrap is made. The KF polymer inherits the family tendency toward limpness, and in addition exhibits enhanced static electricity problems during fabrication, because of the combination of high dielectric constant and high volume resistivity. Thus, especially in the 6 µm gauge, PVF2 is difficult to handle during the common manufacturing processes. It did prove possible, however, to fabricate components with it with repeatability, after the initial problems were worked out.



(a) DISSIPATION FACTOR VS FREQUENCY

(b) DISSIPATION FACTOR VS TEMPERATURE

Figure 8. Dissipation factor of KF polyvinylidene fluoride

The service temperature of this material seems to be about 160°C, on the basis of insulation resistance (Figure 9) and other measurements. In the form in which it was used in this application, use at high temperatures is governed by the irreversable shrinkage, which, if unconstrained, reaches 2 percent at 125°C.

Polysulfone

Of a large class of dielectrics with similar dielectric constants and densities, polysulfone is the best for this application. Polysulfone in capacitor grade is made in this country only by Peter J. Schweitzer, in a high molecular weight grade branded Kimfone, and is available in various gauges down to 2.0 μm (0.08 mil or 8 gauge). The available film is of high quality with low numbers of defects.

The thickness chosen for this application was 3.75 μm (0.15 mil or 15 gauge), at which the energy density for 500 VDC was 0.2 J/g. The electric field was 1.33×10^6 V/cm (3333 V/mil), substantially higher than that required for PVF2.

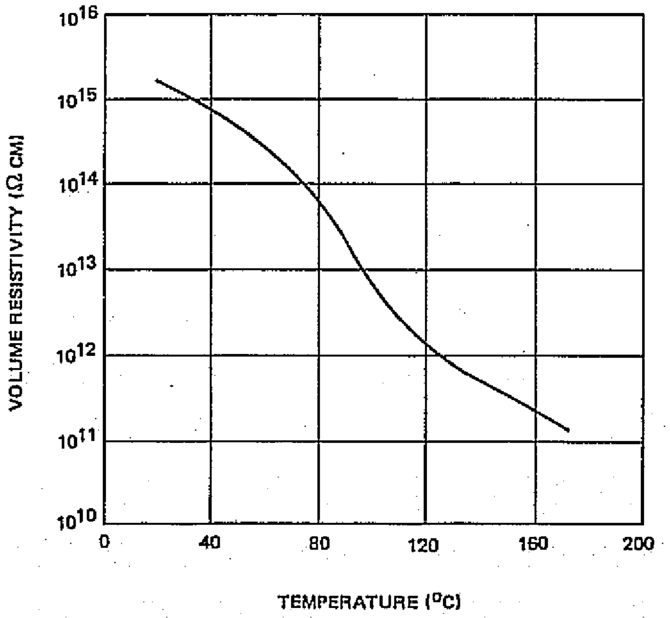


Figure 9. Volume resistivity of KF polyvinylidene fluoride

The dielectric constant of polysulfone declines slightly with temperature (Figure 10) and is stable with frequency. The dissipation factor shows a rather unusual double inflection with frequency, but is exceedingly low at the frequencies of interest. Dissipation factor characteristics are shown in Figure 11.

Polysulfone has essentially the same energy density as polycarbonate, but has a high service temperature, a quality necessary for this program. The service temperature is 125°C. Additionally, polysulfone is easy to fabricate into capacitors.

Fluids

Any one of a number of fluids could have been used in these components with only slight differences. From the point of view of electrical properties, it would be desirable to have a fluid with a large dielectric constant, to force most of the electric field into the strongest member of the dielectric system, the film. Unfortunately, no practical fluid with these properties was uncovered.

Therefore, choice of fluid was made on the bases of good long term life and ease of use. A mixture of several silicon fluids, Dow Corning DC-200, was used. This fluid has a service temperature substantially in excess of any program requirement. Its dielectric constant is only 2.0,

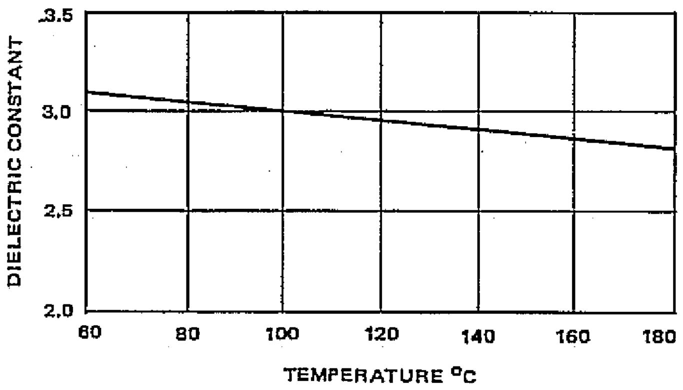
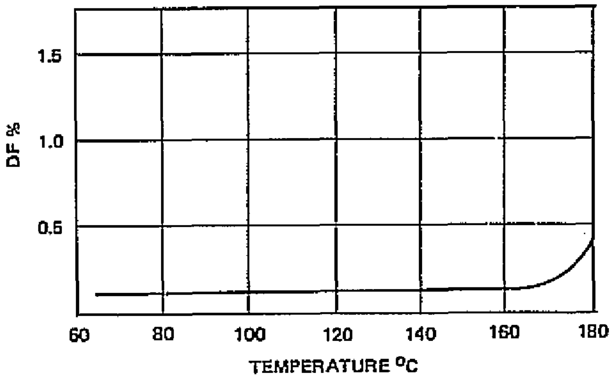
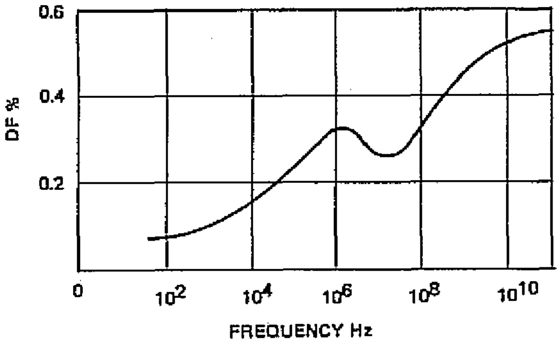


Figure 10. Dielectric constant of polysulfone



(a) DISSIPATION FACTOR AT 60 Hz VS TEMPERATURE



(b) DISSIPATION FACTOR VS FREQUENCY AT 25°C

Figure 11. Dissipation factor of polysulfone (P. J. Schweitzer)

which is not as high as one would desire. Experiments with the polychlorinated biphenyls, fluids with dielectric constants of about 6, were not conducted because of environmental problems.

Other Materials

It is useful to put down here briefly the reason other film materials were not chosen.

Polycarbonate was eliminated because of a 105°C service temperature.

Teflon (PTFE) is not available in extremely thin films, and has a very low dielectric constant/density ratio. It does possess the lowest dissipation factor, especially at high frequencies.

Kapton would be an excellent candidate for higher voltages, but it is not available in sufficiently small thicknesses for this application.

Mylar has a lower energy density than polysulfone.

Parylene is a special case. Parylene is vapor deposited polymer that is used in the Kemet brand of capacitors. There are two ways to use Parylene. The Kemet capacitors are made by depositing the dielectric film on aluminum foil and winding the resultant layered structure into a cylinder. Alternatively, it would be possible to put the film down continuously onto a glass plate, strip it from the plate, metallize it, and make capacitors the conventional way.

There were several reasons this was not done. Hughes is not equipped to produce long continuous films of Parylene, or to continuously coat Al foil. The film as presently produced has electrical properties that vary too much, batch to batch, for results of the desired repeatability. And finally, although a superior film in terms of physical construction may be produced, the achievable energy density is not better than polysulfone.

III. DESIGN AND PRODUCTION

To achieve the lightest weight capacitor it was necessary to optimize not only the dielectric system but also the physical configuration, winding procedures, end terminations, and case. This section describes the designs and methods used to build the components that were life tested later in the program. While no lightweight cases were ever fabricated, designs for a practical case weighing about 1.0 gram are presented in the final part of this section.

CONFIGURATIONAL CONSIDERATIONS

If the capacitors to be built were all in the form of perfect planar units with no margins (inactive portions of the dielectric at the edges), the design of the proper aspect ratio for the completed structure would be simple indeed. Unfortunately, a number of interacting problems not present in the simple planar model complicate the design process.

To begin with, it is clear that, whatever their aspect ratios, all capacitors of the same capacitance value had ought to require the same plate area for a given thickness of dielectric. Since the thickness has already been selected, it is a simple matter to find this area:

$$A = Ct/(8.85 \times 10^{-14}\kappa) \tag{9}$$

where C is the capacitance, t the thickness, and κ the dielectric constant. From the area and thickness the weight of the active portion of the dielectric is easily determined:

$$\omega_A = Atd \tag{10}$$

where d is the density of the dielectric. The metallization on the dielectric film creates a negligable weight addition. Therefore, this weight ω_A is the absolute minimum weight for this capacitor.

The total weight of the finished capacitor is larger than ω_A, and is composed of the weights of the following elements in addition to the "active weight":

- Margin weight
- Termination weight
- Core weight
- Outer wrap weight
- Case weight
- Beginning or leader weight
- Lead wire weight

Each of these except the lead wire weight is a function of the aspect ratio, or the length and width chosen to achieve the active area. In this minimization the weight of the lead wires will not be considered, since a user preference determines them.

Margins

Metallized capacitor film is commonly supplied with either 0.32 cm (0.125 inch) margins or 0.16 cm (0.0625 inch) margins. In these designs the 0.16 cm margins were used, to reduce margin weight. While it would be possible to adjust margin widths for each design to correspond to the voltage used, the weight saving would not be large and the problems of obtaining quantities of such non-standard film would be overwhelming.

In this type of capacitor, metallized films are wound in pairs, right margin and left margin, as shown in the crossection of Figure 12. The result of this is that the effective margin is really twice that of an individual film. This must be taken into account in the calculations.

It is then possible to write down an expression for the margin weight in terms of winding width and required active area:

$$\omega_m = \frac{2A \times M \times t \times d}{W - 2M} \qquad (11)$$

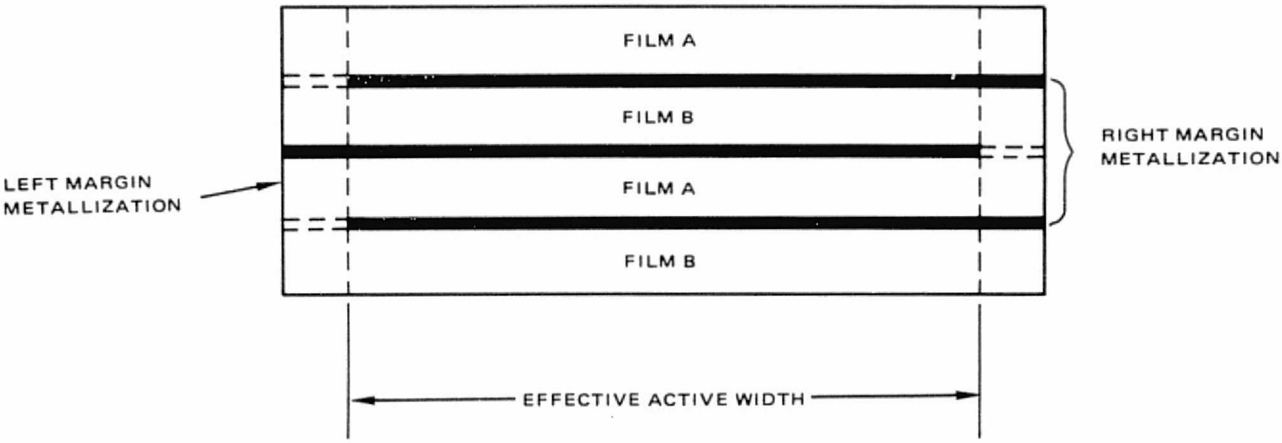


Figure 12. Cross-section showing left hand and right hand metallizations.

where

$$\frac{A}{W - 2M} = L \qquad (12)$$

and where M is the margin width, t the film thickness, d the film density, W the total film width, and L the required winding length. The expression (11) can be used with other terms to be derived to determine minimum weight configurations.

Terminations

The medium used to contact the exposed ends of the extended metallization is flame-sprayed metal — in the present case Sn, for its weldability. This metal layer is applied to approximately the same thickness regardless of the finished capacitor diameter. Therefore, its weight may be represented as:

$$\omega_T = 2d_T \times Lt \qquad (13)$$

with L as in equation (12), d_T the density of the termination layer (different units: d_T is grams/cm^2, a surface or sheet density), and t the film thickness. This calculation assumes that the area of the core is negligable compared to the total area covered, that the winding is sufficiently tight, and that the end area of the winding corresponds to the surface area of the unwound edge.

Core Weight

Each capacitor is wound about a core in this design. The core weight is simply:

$$\omega_c = W \times d_c \tag{14}$$

where W is the winding width and d_c is the core weight per unit length.

Outer Wrap Weight

The outer wrap secures the films and provides an abrasion-resistant surface to facilitate handling during the case assembly process. The wrap is usually a single layer of Kapton tape, so its weight is:

$$\omega_w = 2\pi r d_w t_w W \tag{15}$$

where r is the radius of the finished winding, found from:

$$r = \sqrt{\frac{Lxt}{\pi} + \text{core radius}^2} = \sqrt{\frac{At}{(W - 2m)\pi} + \text{core radius}^2} \tag{16}$$

where d_w is the wrapper density, t_w the wrapper thickness, and W the width.

Case Weight

This discussion applied to standard MIL - Specification cases only, and not to the special cases discussed in detail below. Standard cases consist of a tube of the correct length and diameter, and two end seals. The equation for standard case weight is therefore:

$$\omega_c = \omega_{tube} + 2\omega_{seal} \tag{17}$$

The seal weight is

$$\omega_{seal} = 2\pi r^2 d_s \tag{18}$$

where d_s is a surface density characteristic of the seals. The tube weight is:

$$\omega_{tube} = 2\pi rLd_{tube}t_{tube} \tag{19}$$

where d_{tube} is the density of the wall material and t_{tube} the thickness.

Leader Weight

A standard length leader is used, so that weight is only a function of width:

$$\omega_L = 4L_sWtd \tag{20}$$

for a two-film winding, where L_s is the standard leader length.

Minimization

To begin with, it is simple and illustrative to find the minimum weight of the winding itself. This process involves the following weights:

$$\begin{aligned} \omega_{tot} &= \omega_M + \omega_T + \omega_C + \omega_L \\ &= \frac{2AMtd}{W - 2M} + 2d_TLt + Wd_c + 2L_sWtd \end{aligned} \tag{21}$$

This can be written in a form to eliminate L:

$$\omega_{tot} = \frac{2AMtd}{W - 2M} + \frac{2d_TAt}{W - 2M} + Wd_c + 2L_sWtd \tag{22}$$

The reason the parameter W is displayed here is that one designs and manufactures capacitors by selecting the width. The winding machine automatically selects the proper length. This expression is easily minimized with respect to W:

$$\frac{d\omega_{tot}}{dW} = 0 \tag{23}$$

The result is:

$$W = 2M + \sqrt{(2AMtd + 2d_T tA)/(d_c + 2L_s td)} \qquad (24)$$

It is clearly possible to write an equivalent expression for any configuration or design. For example, the equation analogous to (22) involving all terms is:

$$\omega_{tot} = \frac{2AMtd + 2d_T At}{W - 2M} + W(d_c + 2L_s td) + \pi d_s \left(\frac{At}{\pi(W - 2M)} + r_{core}^2\right)$$

$$+ \left[2\pi(d_w t_w W + \frac{d_{tube} t_{tube} A}{W - 2M})\right] \left(\frac{At}{(w - 2M)\pi} + r_{core}^2\right)^{1/2} \qquad (25)$$

This expression is messy to minimize for the general case. However, for a particular design application trade-off studies may be conducted quickly and easily on a computer.

Practical Constraints

There are a number of considerations which are purely practical that may restrict the designer. These restrictions principally concern the availability of materials. Capacitor film is commonly available with certain margins and in certain widths and thicknesses. It is thus necessary to make material choices based upon a series of energy density calculations combined with a knowledge of material availability.

Additionally, there is one factor which does not appear in the above calculations but which argues for a relatively larger width-to-length ratio. This is edge corona. If edge corona occurs, it occurs at random sites along the edge of the metallization, at the margin. Therefore, the shorter the edge the lower the probability of edge corona, everything else being equal The designer must take this into account.

Numerical Calculations

The film widths used for capacitor construction in this program were derived from this type of calculation. As an example, the calculation for the PVF2 component is displayed:

$A = 1232\ \text{cm}^2$ $\qquad L_s = 10\ \text{cm}$

$t = 6 \times 10^{-4}\ \text{cm}$ $\qquad d_c = 1.96 \times 10^{-2}\ \text{g/cm}$

$M = 0.159\ \text{cm}$ $\qquad d_T = 5.0 \times 10^{-2}\ \text{g/cm}^2$

$d = 1.8\ \text{g/cc}$

Ideal Width = 2.82 cm (1.112 inch)

The actual width chosen was 2.699 cm (1.0625 inch), because of the corona effect discussed above and because the film was available in 2.06 cm (0.8125 inch) or 2.699 cm only. Similar calculations were made for other materials.

WINDING TECHNIQUE

The basic principal in winding high density capacitors is to try to make the winding as nearly perfect as possible. Because of the large electric fields necessary, any sort of imperfection in the winding contributes to early failure. Among the practical problems to be worked on are particulate contamination, wrinkles in the film, stretched film, edge alignment (run-out), start and finish, termination, and core. Once this principal is accepted, the elimination of problems is basically an experimental manufacturing task. While it was not possible on this program to eliminate all the defects, considerable progress was made, as evidenced by the test results.

Core

The first item to be investigated is the core upon which the winding is made. This core must be lightweight, producible, and as cylindrical as possible. The last requirement listed is very important, since any core imperfections have been found to propagate wrinkles into the overlying film.

The core used on this program consisted of a pair of Teflon "D"s, as illustrated in Figure 13. This core has the advantage of being relatively

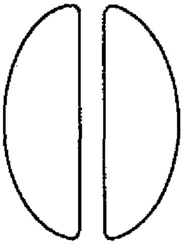


Figure 13. Cross-sectional view of capacitor care.

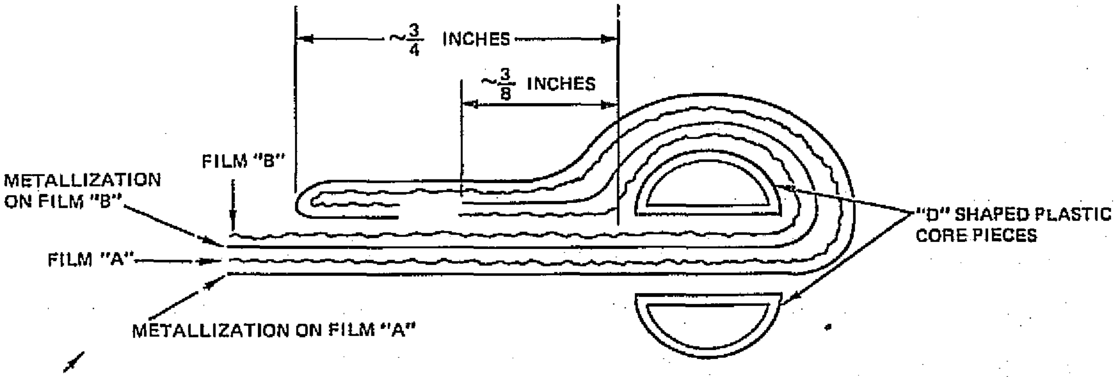
lightweight, and it fits over the two prongs of many conventional small capacitor winders. Additionally, it is approximately cylindrical.

Winding

The secret to winding is, for small windings, to maintain constant tension during the winding process. The reason for this is to maintain film characteristics, including thickness, as constant as possible. Additionally, this method seems to help to eliminate wrinkles and other similar defects. Tension is selected as that stress that just does not stretch the film.

The start of the winding is accomplished as shown in Figure 14. Ideally, the film that is wrapped between the core halves should be unmetallized so that no electrical breakdown may occur over the inevitable wrinkle at the initial fold. This was not done in the present case because of the problem of removing the metallization.

The finish of the winding is accomplished simply by cutting the films off the supply roll in such a way that no shorting occurs, and then wrapping



THIS END IS THEN WOUND IN A COUNTER-CLOCKWISE DIRECTION AROUND THE TWO "D" SHAPED PIECES.

Figure 14. Starting wrap.

the whole winding with Kapton tape. In a full production situation this is usually thick tape, to provide protection during subsequent assembly operations. If a limited production situation were used, a lighter and thinner final wrap might be feasible.

Two considerations should be mentioned here. The first is edge alignment or runout. If the films are not perfectly lined up in the winding, the result is that the effective margin is reduced. To compensate for this, the designer must increase the real film margin to give some minimum effective wound margin, and this increases the weight. Pronounced run-out also makes termination more difficult. Finally, the winding operation should be performed in a clean area, particularly when the thinner films are used.

End Termination

The conventional end termination and electrical connection that is used for metallized film capacitors is flame-sprayed metal. In a common construction, the entire end of the capacitor is sprayed, and the wire is soldered onto this coating as shown in Figure 15. The original termination design used on this program differed slightly, and is shown in Figure 16.

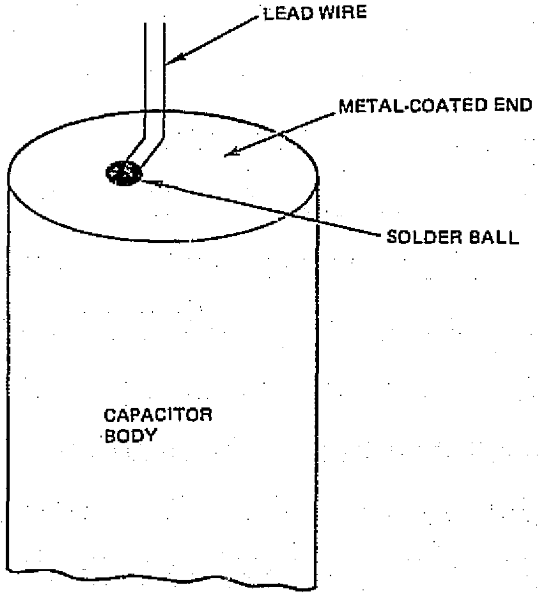


Figure 15. Common end termination.

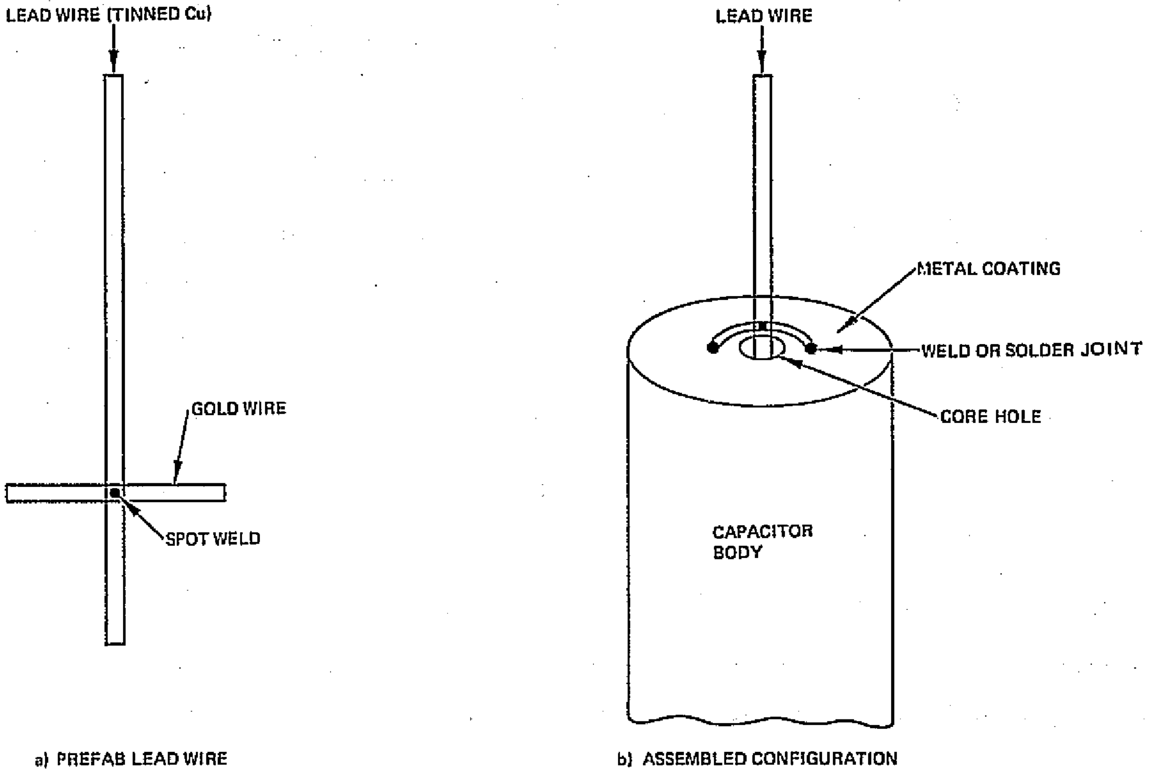


Figure 16. End termination used on this program.

There was suspicion that this coating might not be as porous as it needed to be to insure complete impregnation, so an investigation was conducted using the Scanning Electron Microscope (SEM) facility. Figure 17 shows an overview of the end of a standard test specimen. It is important to remember that the gray scale that appears in this and subsequent SEM photographs does not correspond with normal visual experience. Here the bright white area (in this case, the core material) is not optically white, but instead is an insulator. Figure 18 shows a low magnification view of a part of the metal coating. Notice the areas that show splattering of larger portions of metal. Figure 19 shows a very high magnification view of a portion of the area shown in the previous view. This area does not look particularly porous. An x-ray analysis of the flame-sprayed material was conducted, and the material was found to be pure Sn, as thought.

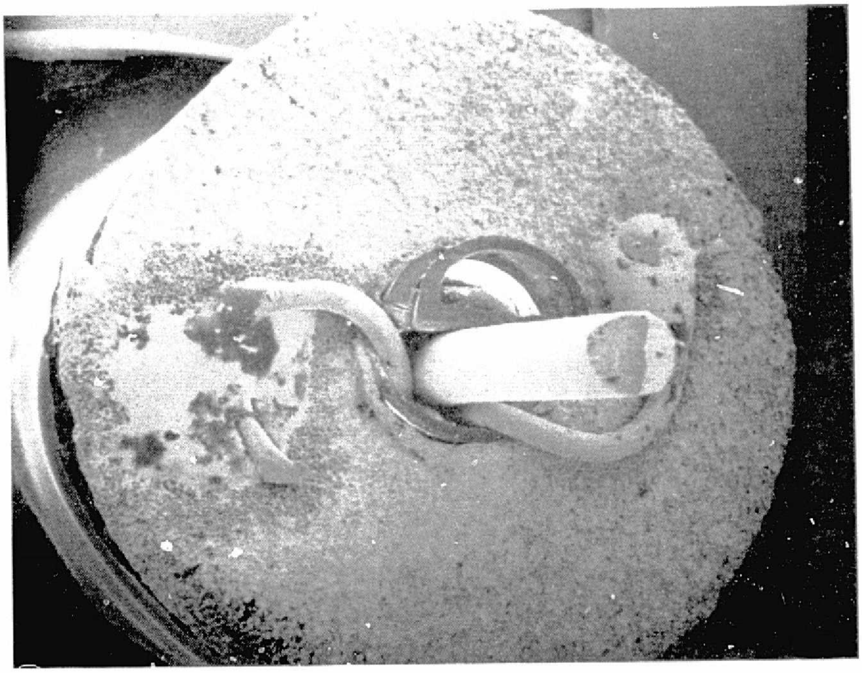


Figure 17. Scanning electron micrograph (SEM) of termination and end structure, 16X.

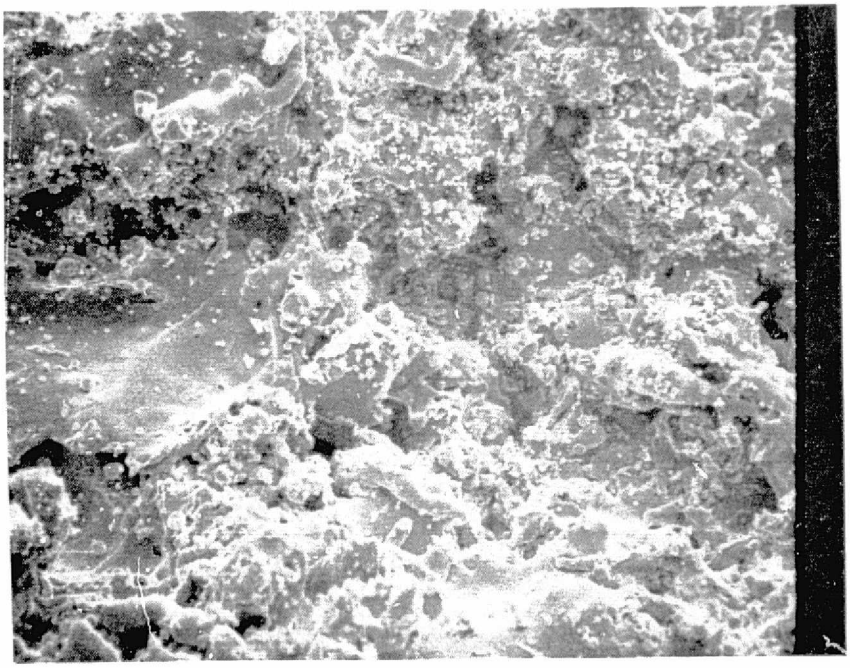


Figure 18. SEM of termination metal, 530X.

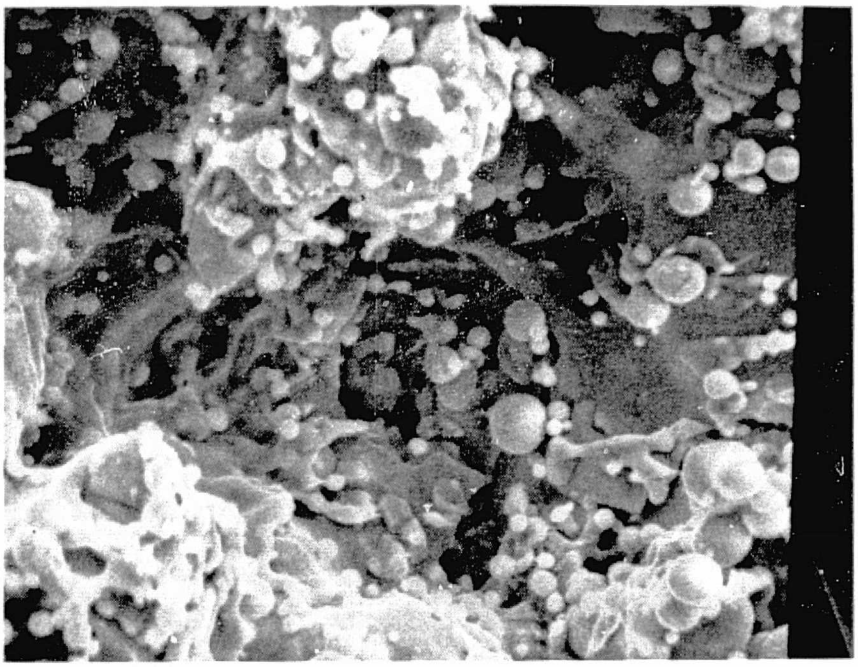
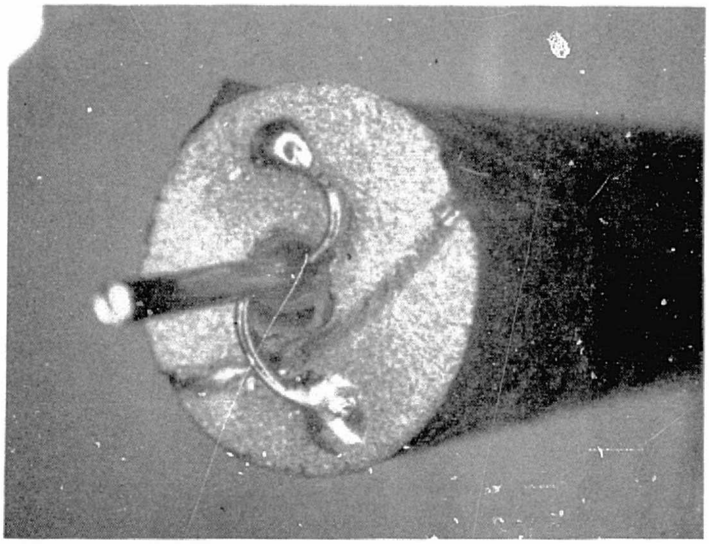


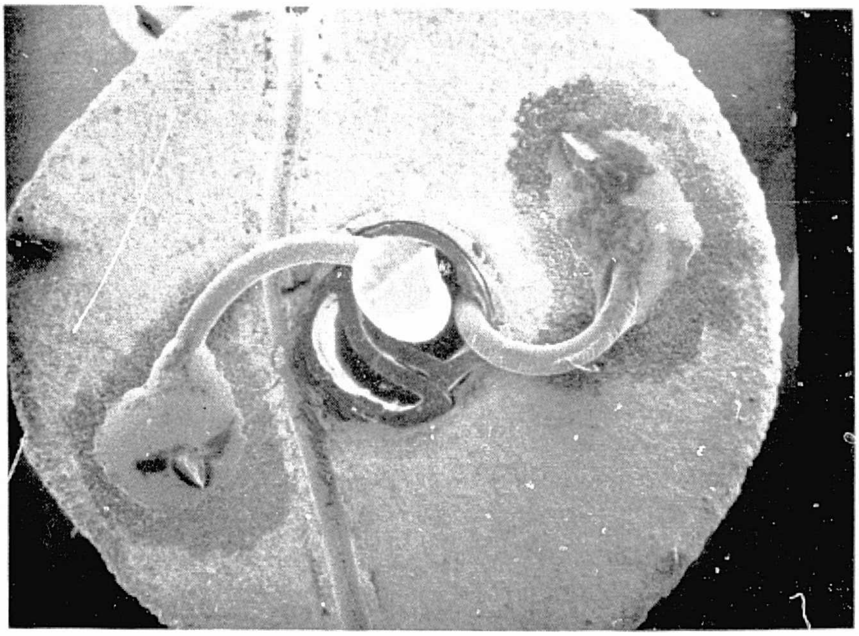
Figure 19. SEM of termination metal, 2100X.

Experiments were conducted using flame-sprayed metal coatings deposited on membrane type filter sheets, to try and get a more quantitative estimate of the porosity. These experiments were judged inconclusive, because in the process of deposition of the coating some melting of the filter media occurred, and it was impossible to determine how much of the change in fluid flow rate was due to the metal coating.

It was known that some commercial capacitor manufacturers mask a wide stripe across the center of each end, so that uncoated access to each layer interstice is available to the impregnant. However, it was also known that such a configuration causes an increase in the dissipation factor, particularly at the higher frequencies. As a compromise, a very narrow stripe was masked across each end of the final set of capacitors, as shown in Figure 20. Figure 20a is an optical photograph of this modification. Figure 20b is a SEM photograph, showing the increased porosity. Figure 20c is a SEM photograph of a portion of the stripe shown in 20b, and Figure 20d is an enlarged picture of the bottom of the stripe channel, showing the access to the layer interstices. This increased the impregnation weight change slightly

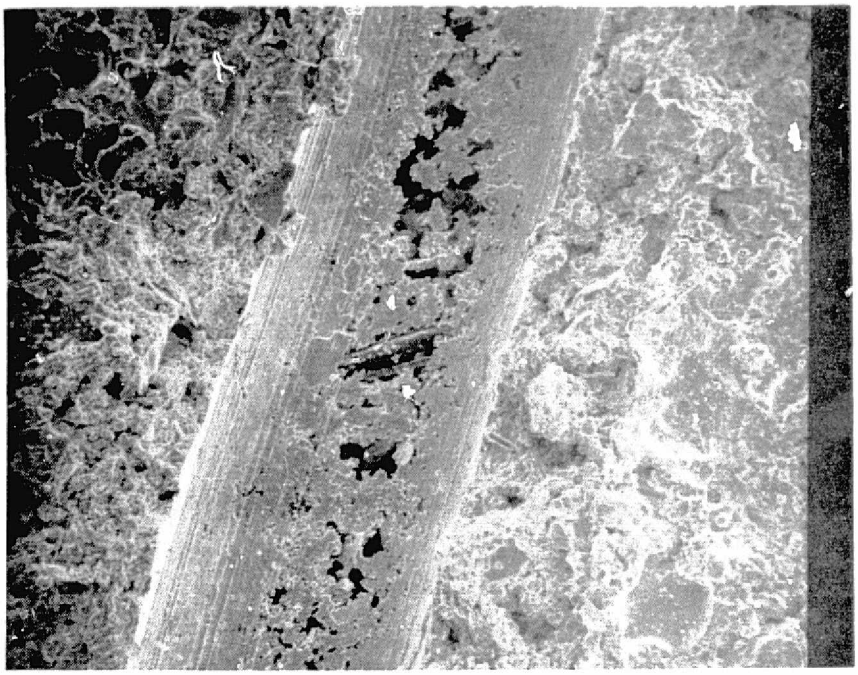


a. Optical photograph of capacitor end metallization, showing stripe, 8X.

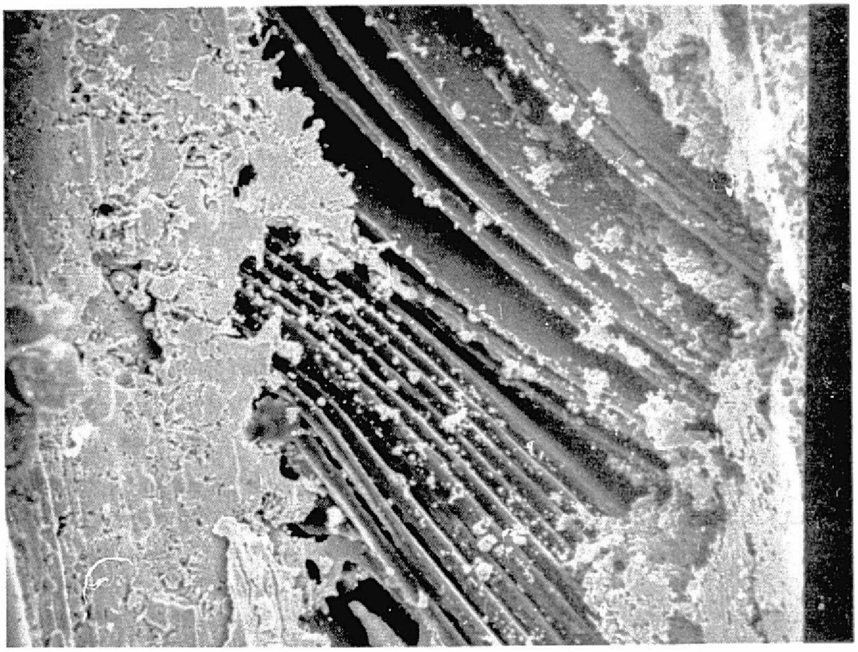


b. Scanning electron micrograph of end metallization, showing stripe, 18X.

Figure 20



c. Detail SEM view of stripe, with film ends showing through. Magnification 65X.



d. Detail SEM view of bottom of stripe channel, showing ends of film layers. Magnification 320X.

Figure 20

but significantly, indicating better impregnation over the unstriped situation. No significant change in electrical parameters was observed.

Reports of other government-sponsored capacitor development work frequently discuss the breakdown of dielectric material in the margin as a principal failure mechanism. This is usually attributed to the large electric field at the edge of the metallization. However, in some cases it appears that the method of termination used causes overheating of the dielectric in this critical area, and that electrical failure follows in the most highly damaged areas. Test units made using the techniques discussed above were carefully dissected, and some damage was found. However, it was confined to the region next to the flame-sprayed termination, and in no case did the damage extend further into the margin than 0.016 cm (6.25 mil), or one tenth of the margin width.

DESIGN OF LIGHT-WEIGHT CAPACITOR CASES

The objective of this section is to design a moderately light-weight case suitable for multi-purpose capacitors of high energy density. The major design goals are:

- Weight of case 1.25 gm or less
- Two terminal case with both terminals insulated
- Reasonable manufacturability

The design should be capable of meeting MIL environmental requirements. The capacitor may be impregnated with oil or other dielectric fluid. This necessitates that the case be completely leak-free, bakeable at high temperatures, and constructed from materials which are compatible with the impregnant. Finally, the construction and assembly operations should not require the invention of new technology.

Some of the designs which were considered may be briefly mentioned. An all plastic case would be extremely light-weight and low cost, and for many applications it would be satisfactory. The major limitations are the high permeation of gases through plastics and the difficulty of meeting the environmental requirements. It is felt that a light-weight, all glass case could be designed, except it would be very fragile. Other designs envisaged indium and gold diffusion seals for the end caps. Such seals would be delicate and not necessary to make a moderately light-weight case.

The design which is proposed is conceptually like ordinary hermetically sealed commercial and MIL capacitors. These usually consist of a metal cylinder with glass-to-metal end caps soft soldered to the cylinder. The major change to achieve light weight is redesign of the end cap.

Proposed Design

It is convenient first to discuss briefly the construction of a typical hermetically sealed capacitor. A cross-section view is shown in Figure 21. This design is used for high reliability military applications and is made by Components Research, Inc. The total weight of the case is 3 gm. The case cylinder is nickel alloy tubing. The end caps are kovar-glass. The capacitor section has extended foils which are spray metallized on each end. Wires are soft soldered to the metallization and the wire lead for the electrical connection.

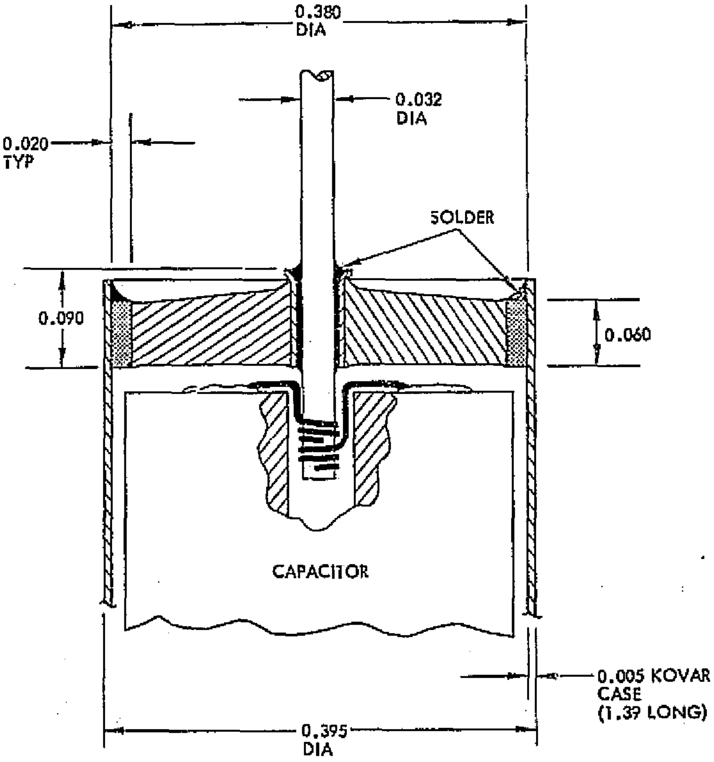


Figure 21. Standard MIL-type capacitor case — view of one end.

To make the hermetic seal the end caps are soft soldered to the case. After impregnation the lead and end cap are sealed with soft solder.

It is desirable to design a case for the capacitor which is substantially lighter in weight. The commercial case described above weighs 3 gm. To meet the energy density required the case must weigh 1.25 gm or less. Several approaches may be taken to achieve this. The one selected is to design a case similar to the typical case shown in Figure 21. It is apparent that the case cylinder can be made from thinner wall material. The two end caps account for two-thirds of the total weight. It is obvious the end caps have to be redesigned to be lighter, as they alone weigh more than is allowed for the whole case.

The proposed light-weight capacitor case design is shown in Figure 22. For convenience the case cylinder is Kovar with a wall thickness of 127 µm (5 mils). Kovar is used for the cylinder to permit joining the Kovar end cap and cylinder by welding. Either heliarc or resistance welding are

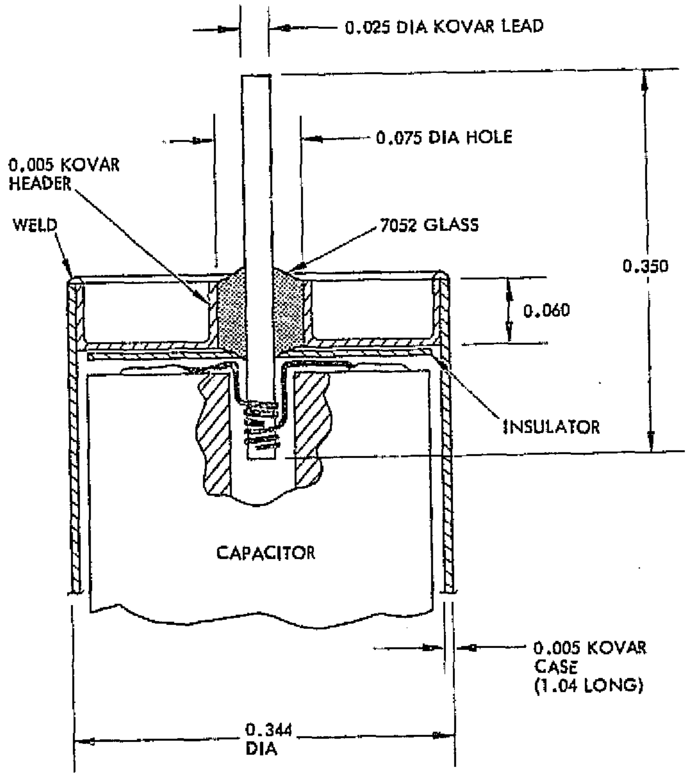


Figure 22. Improved very lightweight case.

feasible; for small quantities the operation may be performed manually and for production automatically. Alternatively, the end cap and cylinder may be soft soldered. This might be desirable for prototype and development work, but it would be slightly heavier. On the other hand, the case might be disassembled easily and reused. No special tools or equipment are needed. It should be pointed out that the clearance required for welding and soft soldering are different. For welding a tight or interference fit is necessary. On the other hand, a clearance of approximately 0.004 in. is needed for soft soldering.

The end cap is specially designed for this application. The Kovar is designed to be made in lathe or screw machine. With some changes it could also be stamped. The sealing of the glass to the Kovar is normally done in a high temperature furnace. It is possible to make prototypes or small quantities by hand or in a vertical glass lathe. A very large variety of standard feedthroughs are commercially available, so by compromising the design and with some increase in weight a standard feedthrough could be accommodated.

The processing to assemble the capacitor is an important factor in achieving high energy density and reliable performance. The design of the above case is consistent with these stringent requirements. The construction is entirely cf metal and glass. The case may be chemically cleaned and vacuum baked out at elevated temperatures. The Kovar and glass are compatible with normal impregnants. For convenience the leads are soft soldered but they could be welded to eliminate the lead-tin solder.

The calculated weight of the proposed light-weight capacitor case, shown in Figure 22, is summarized in the following table:

Item	Number Required	Material	Weight (gm)
Case	1	0.005 Kovar	0.749
Kovar Lead	2	0.025 Dia Kovar	0.023
Glass Bead	2	Type 7052 Glass	0.010
Kovar Cup	2	0.005 Kovar	0.079
Insulator	2	0.005 Kapton	0.008
		Total Weight	0.989 gm

FINAL CONFIGURATIONS

The capacitors that were fabricated (by Component Research) for the electrical testing were of two different film materials, and of different thicknesses. The specifications were as follows:

Material	Thickness	Width	Margin
Polysulfone	3.556 μm	2.699 cm	0.159 cm
PVF2	5 μm	2.699 cm	0.159 cm

Both types of capacitors were impregnated with a silicone fluid of the DC-200 series. A complete description of the manufacturing details is contained in Appendix II, Detailed Procedures.

UNIQUE DEVELOPMENTS

Two different approaches to the problem of high energy density medium voltage components were developed on the periphery of the main program effort, and deserve mention here. These are the super-thin windings, and the flat flexible capacitors.

As was previously discussed, extremely thin polymeric films of very high quality are now available. In particular:

Material	Minimum Thickness
Polycarbonate	1.52 μm (0.06 mil or 60 microinches)
Polysulfone	2.0 μm (0.08 mil or 80 microinches)

It was decided to fabricate two types of experimental thin-film capacitors: a single 8 gauge (2.0 μm) polysulfone, and a double layer component of the same material. The double layer components were not as promising as had been hoped, showing large amounts of corona (sometimes accompanied by visible corona!) at relatively low voltages, and will not be reported further. The single layer components, however, gave extremely good results. Twenty of them were given a step-stress DC life test, and the results are reported on page 91.

In searching for a better way to construct a component for this purpose, it was thought that perhaps layers of metallized film could somehow be laminated together to form an essentially monolithic structure that might not require a case and certainly would not require an impregnant. As a first experiment, sheets of metallized 18 µm (0.75 mil) PVF2 were cut to a size of about 10 cm x 12 cm. A nylon epoxy was prepared, coated onto the sheets as thinly as possible, and allowed to dry. A number of sheets of the coated material were stacked up, and placed in a heated press. Pressure and heat were applied, until the epoxy had fully cured.

This capacitor had a corona inception voltage (CIV) greater than 700 volts, DC. Therefore, it was decided to try to make a capacitor from 6 µm (0.24 mil) PVF2, which is a considerably more difficult task, since wrinkles must be prevented. The design parameters of this capacitor were:

- Material — Polyvinylidene Fluoride
- Thickness — 6 µm
- Plate size — 10 x 12 cm
- Number of sheets — 10
- Capacitance — 2.0 µF

Unfortunately, with the small amount of effort devoted to this configuration, it was not possible to build an acceptable capacitor. Everything seems to be fine about the process, except the thin sheets are very difficult to handle, and very wrinkled capacitors are the result. However, it is thought that this problem could be solved with additional effort.

Were it possible to build an electrically acceptable capacitor to these specifications, several unique properties and advantages would result. The capacitor has an energy density of about 0.18 J/g in its finished configuration at 500 V, has an unusual form factor (10 x 12 cm x 60 µm) that admits incorporation into circuit boards, can be made with screened electrodes to produce multiple capacitors connected in a variety of configurations, and is flexible. It should be noted that there is a multiplicity of patents in the area of stack-type capacitors, some of which may cover this concept in whole or in part.

IV. PARAMETER MEASUREMENT

The requirement of this set of tasks was to electrically characterize 100 capacitors fabricated for the life tests. These characterizations included capacitance, dissipation factor, leakage current, and corona spectrum for each capacitor, over a range of temperatures. At the beginning, it was not expected that any problems would arise from these relatively routine measurements, but unfortunately that was a misapprehension.

The detailed measurement requirements of Task 6 are as follows:

- Estimate of Energy Density
- Maximum Operating Voltage
- Capacitance vs Frequency to 100 kHz at 25°C, 75°C, and 125°C
- Dissipation Factor vs Frequency to 100 kHz at 25°C, 75°C and 125°C
- DC Resistance at 25°C, 75°C, and 125°C
- Corona Inception Voltage and Quantitative Pulse Count

The first two of these are derived parameters, found from the CIV. They will be treated at the end of this section. The techniques and results for the direct measurements are discussed below.

CAPACITANCE AND DISSIPATION FACTOR

At first glance, the measurement of capacitance and dissipation factor is straightforward. There is a bridge, the General Radio 1615A, that measures C and DF from 0 to 100 μF up to 100 kHz. However, at frequencies larger than 1 kHz, the bridge error is:

$$\left[\pm 3 \times 10^{-5}\% + 2(C_F) \times 10^{-3}\% \pm 3 \times 10^{-7}\ \text{pF}\right] \times (f_{kHz})^2 \qquad (26)$$

This error, caused principally by residual inductance in the bridge transformer and lead wires, is quite large at 100 kHz. Figure 23 shows a frequency response curve made by the Hughes Primary Standards Laboratory, using a 2.0 µF silver mica capacitor calibrated by the National Bureau of Standards. This capacitor is very stable with frequency, and so, were the measuring apparatus accurate, the curve would be a straight line. The deviation of the curve from a straight line represents the inaccuracy. The values found are within the stated accuracy, but this was nevertheless judged unsuitable for use on this program.

An alternative method using the same bridge but with auxiliary equipment was developed in consultation with General Radio. This method has an accuracy of about ±2.5 percent and a precision better than 0.1 percent, but both the measurement and the calculations are involved. The circuitry is shown in Figure 24. A phase-locked amplifier is used to obtain the null in place of the usual detector. The calculations then are:

$$C_x = C_{EM} + C_B - \frac{C_{EM}\, C_B\, D_E^{\,2}}{C_B + C_{EM}} \tag{27}$$

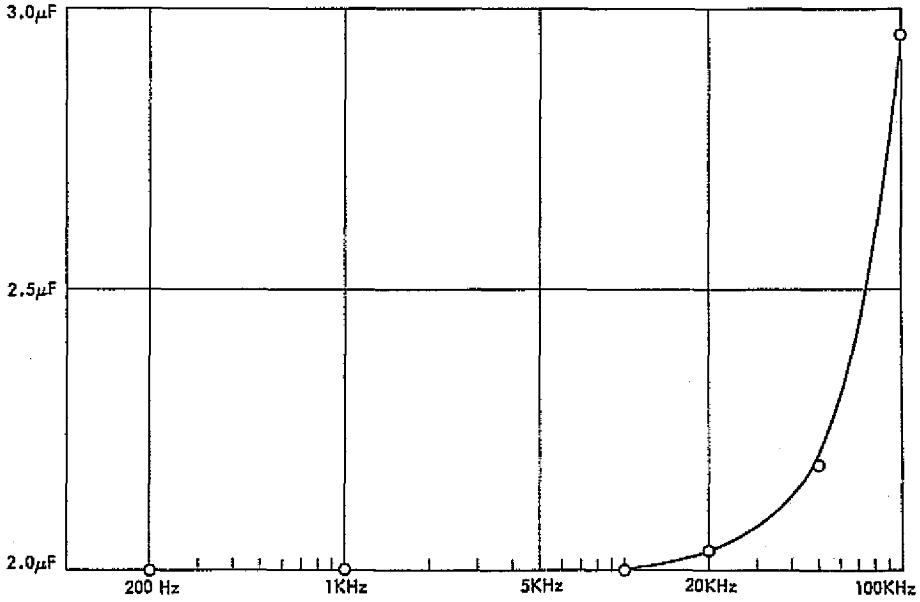


Figure 23. GR 1615A frequency response.

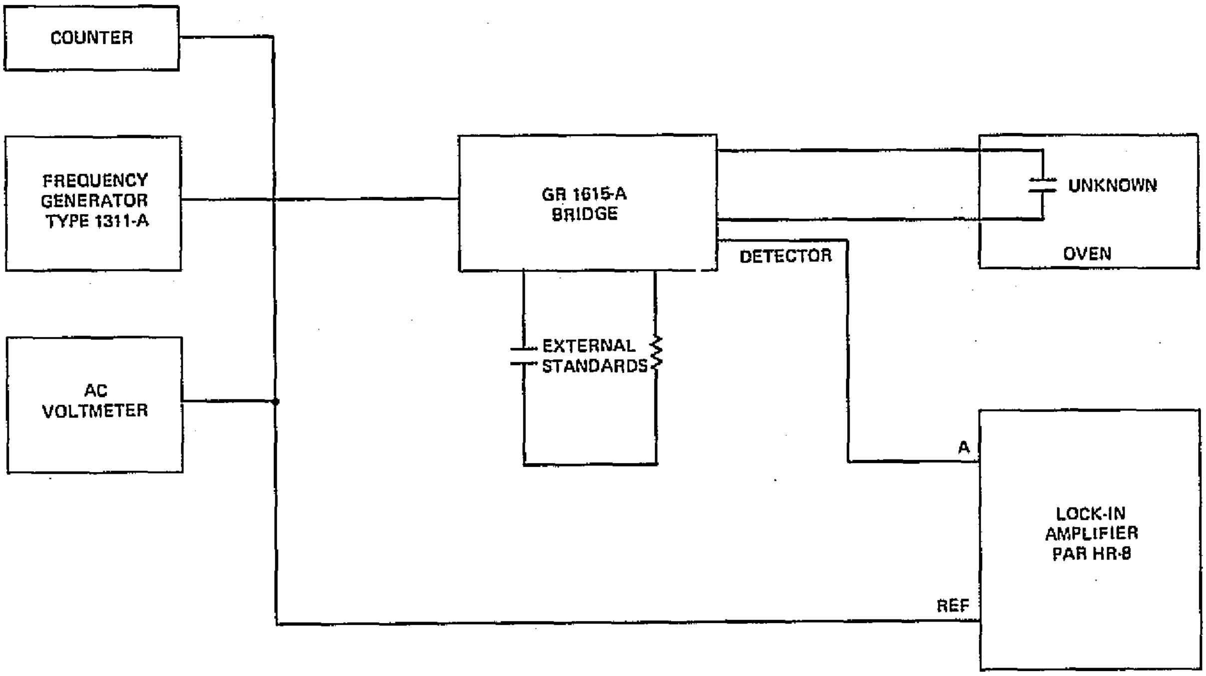


Figure 24. Precision parameter measurement circuit.

and

$$\text{DF percent} = \frac{C_{EM}\, D_E}{C_B + C_{EM}} \times 100 \text{ percent} \qquad (28)$$

where:

$D_E = R_E \times 2\,\pi f \times C_E$
$C_{EM} = C_E \times$ multiplier (farads)
$C_E =$ External Capacitor (farads)
$C_B =$ Bridge Capacitance Reading (farads)
$C_X =$ Unknown Capacitance (farads)
$R_E =$ External Resistance Standard (ohms)
percent DF = DF of C_x (percent)

At frequencies above 10 kHz, additional corrections, shown in Appendix III, must be used.

Because of this difficulty, it was decided, with the approval of the Project Monitor, to make the following measurements on the capacitors for life test:

- Complete curves on 20 components
- Measurement at 1 kHz 25°C on 80 components

In future experiments, it might be possible to make measurements faster and easier using a system containing an impedance comparator such as the GR-1654, modified for four terminal use (to eliminate much of the residual inductance). This would limit frequencies to 100 Hz, 1 kHz, 10 kHz, and 100 kHz, however.

Results

The results of the parameter measurements are presented in a series of tables in this section. These measurements were made on components that had previously been corona tested. The correspondence between component type, serial number, and internal sample number is shown in Table XXIII. Table XXIV contains the 1 kHz results for the 80 components; these measurements were made on a GR-1673 automatic bridge. Notice the much lower DF for the polysulfone components. Tables XXV, XXVI, and XXVII present the detailed results for 20 components at 25°C, 75°C, and 125°C, respectively. None of this data shows anything that was not expected from analysis of the constituent material.

CORONA MEASUREMENTS

Measurement of corona has been routinely applied as a quality control technique on high voltage components such as transformers and cables, and to a lesser extent on capacitors. With components that have relatively low inter-electrode capacitances such tests have been successful, giving reliable nondestructive indications of maximum operating voltage and life at voltage. However, problems arise with the testing of capacitors of large capacitance, such as those in this program. These problems are sensitivity and AC power.

TABLE XXIII. RELATION BETWEEN SPECIMEN NUMBER, SERIAL NUMBER, AND FILM TYPE, FIRST 100 LIFE TEST SPECIMENS

Specimen Number	S/N	Type	Specimen Number	S/N	Type	Specimen Number	S/N	Type
1	20	PVF2	35	66	PVF2	68	38	PS
2	21	PVF2	36	68	PVF2	69	41	PS
3	22	PVF2	37	69	PVF2	70	42	PS
4	23	PVF2	38	72	PVF2	71	43	PS
5	24	PVF2	39	78	PVF2	72	46	PS
6	25	PVF2	40	79	PVF2	73	47	PS
7	26	PVF2	41	81	PVF2	74	51	PS
8	27	PVF2	42	84	PVF2	75	52	PS
9	29	PVF2	43	85	PVF2	76	53	PS
10	31	PVF2	44	86	PVF2	77	54	PS
11	32	PVF2	45	87	PVF2	78	55	PS
12	33	PVF2	46	88	PVF2	79	56	PS
13	34	PVF2	47	92	PVF2	80	57	PS
14	36	PVF2	48	93	PVF2	81	59	PS
15	37	PVF2	49	95	PVF2	82	60	PS
16	39	PVF2	50	96	PVF2	83	61	PS
17	40	PVF2	51	06	PS	84	62	PS
18	41	PVF2	52	07	PS	85	63	PS
19	42	PVF2	53	08	PS	86	64	PS
20	43	PVF2	54	10	PS	87	65	PS
21	44	PVF2	55	12	PS	88	66	PS
22	45	PVF2	56	13	PS	89	72	PS
23	46	PVF2	57	15	PS	90	76	PS
24	47	PVF2	58	16	PS	91	78	PS
25	54	PVF2	59	18	PS	92	80	PS
26	55	PVF2	60	19	PS	93	81	PS
27	56	PVF2	61	20	PS	94	82	PS
28	57	PVF2	62	21	PS	95	83	PS
29	58	PVF2	63	22	PS	96	85	PS
30	59	PVF2	64	31	PS	97	87	PS
31	60	PVF2	65	33	PS	98	92	PS
32	62	PVF2	66	34	PS	99	94	PS
33	64	PVF2	67	36	PS	100	95	PS
34	65	PVF2						

Note: PVF2 type No. XT1032-1
PS type No. XT1032-3

Extra specimens 101-114 PVF2
115-129 PS

TABLE XXIV. PARAMETER RESULTS AT 1kHz 25°C

Specimen Number	C μF	DF %	Specimen Number	C μF	DF %	Specimen Number	C μF	DF %
13	1.996	1.42	*53	2.057	0.36	*100	2.069	0.30
14	1.962	1.18	*54	2.083	0.33	101	2.194	1.20
*15	2.179	1.20	*55	2.068	0.41	102	2.171	1.26
16	2.021	1.21	*56	2.070	0.35	103	2.174	1.26
17	1.993	1.39	*57	2.078	0.32	104	1.995	1.17
*18	1.975	1.40	*58	2.086	0.32	105	2.209	1.19
*19	2.090	1.30	*59	2.035	0.33	106	1.991	1.17
20	2.169	1.29	*60	2.083	0.32	107	2.210	1.20
21	1.963	1.63	*71	2.078	0.34	108	2.210	1.20
22	2.195	1.27	*72	2.088	0.32	109	2.166	1.28
23	2.190	1.24	*73	2.036	0.35	110	2.168	1.28
24	1.989	1.19	*74	2.063	0.39	111	2.204	1.18
*25	1.969	1.17	*75	1.871	0.29	112	2.211	1.19
26	1.966	1.22	*76	2.076	0.36	113	2.178	1.22
27	2.160	1.52	*77	2.061	0.37	114	2.128	1.18
28	2.179	1.23	*78	2.091	0.36	115	2.080	0.40
*29	2.153	1.13	*79	2.064	0.36	116	2.075	0.35
*30	2.194	1.21	*80	2.063	0.41	117	2.102	0.38
*31	2.002	1.26	*81	2.066	0.35	118	2.087	0.33
32	2.175	1.40	*82	2.093	0.30	119	2.042	0.40
33	2.175	1.25	*83	2.066	0.34	120	2.086	0.34
34	1.967	1.19	*84	2.071	0.33	121	2.120	0.40
*35	2.187	2.47	*85	2.077	0.32	122	2.085	0.35
36	1.986	1.36	*86	2.062	0.36	123	2.096	0.34
*37	2.164	1.24	*87	2.065	0.41	124	2.070	0.40
38	2.210	1.28	*88	2.063	0.36	125	2.070	0.40
39	1.980	1.28	*89	2.087	0.33	126	2.075	0.37
40	1.981	1.21	*90	2.085	0.34	127	2.070	0.40
*41	1.994	1.12	*91	2.079	0.32	128	2.064	0.36
*42	2.060	1.21	*92	2.087	0.33	129	2.087	0.33
43	2.184	1.22	*93	2.080	0.30			
44	2.160	1.28	*94	2.055	0.31			
*45	1.976	1.16	*95	2.070	0.29			
46	2.106	1.18	*96	2.074	0.36			
47	2.009	1.17	*97	2.104	0.40			
48	1.986	1.18	*98	2.117	0.38			
*49	1.983	1.20	*99	2.063	0.32			
50	1.985	1.19						
*51	2.067	0.40						
*52	2.085	0.38						

Note: *Denotes capacitor subsequently damaged in corona test.

All measurements made on GR-1673 Automatic Capacitance Bridge.

TABLE XXV. PARAMETER MEASUREMENTS AT 25°C

Specimen	310 Hz C(µF)	310 Hz DF(%)	500 Hz C(µF)	500 Hz DF(%)	1 kHz C(µF)	1 kHz DF(%)	2 kHz C(µF)	2 kHz DF(%)
1	2.0016	0.9237	1.9966	0.9783	1.9881	1.124	1.9844	1.373
2	2.0134	0.9212	2.0079	0.9743	1.9998	1.118	1.9971	1.350
3	2.1449	0.9023	2.1395	0.9588	2.1318	1.106	2.1272	1.342
4	2.1842	0.8321	2.1694	0.9369	2.1559	1.145	2.1509	1.331
5	1.9775	0.9230	1.9724	0.9742	1.9639	1.115	1.9609	1.347
6	2.0078	0.9287	2.0027	0.9832	1.9941	1.127	1.9914	1.361
7	2.0087	0.9214	2.0037	0.9732	1.9953	1.116	1.9926	1.350
8	1.9819	0.9458	1.9735	0.9945	1.9634	1.113	1.9596	1.332
9	2.0299	0.9147	2.0243	0.9972	2.0155	1.099	2.0124	1.487
10	2.178	0.9134	2.1735	0.9672	2.1643	1.118	2.1616	1.354
11	2.1694	0.9138	2.1640	0.9685	2.1549	1.116	2.1524	1.360
12	2.1761	0.9146	2.1709	0.9683	2.1618	1.119	2.1583	1.367
61	2.0707	0.1547	2.0694	0.1972	2.0688	0.354	2.0675	0.423
62	2.0827	0.1625	2.0929	0.2001	2.0809	0.301	2.0790	0.451
63	2.0559	0.1471	2.0592	0.2040	2.0542	0.324	2.0523	0.420
64	2.0594	0.1591	2.0583	0.1899	2.0575	0.368	2.0558	0.439
65	2.0670	0.1623	2.0661	0.1921	2.0653	0.375	2.0641	0.411
66	2.0647	0.1479	2.0640	0.1932	2.0631	0.298	2.0620	0.424
67	2.0767	0.1761	2.0756	0.1944	2.0745	0.311	2.0733	0.476
68	2.0709	0.1605	2.0699	0.1926	2.0689	0.329	2.0677	0.434
69	2.0587	0.1472	2.0579	0.1890	2.0577	0.320	2.0559	0.426
70	2.0634	0.1500	2.0625	0.2011	2.0618	0.369	2.0608	0.471

Specimen	10 kHz C(µF)	10 kHz DF(%)	20 kHz C(µF)	20 kHz DF(%)	50 kHz C(µF)	50 kHz DF(%)	100 kHz C(µF)	100 kHz DF(%)
1	1.9739	3.083	1.9761	4.832	1.9862	10.85	2.0855	35.76
2	1.9884	3.067	1.9877	4.764	1.9961	10.73	2.1042	34.82
3	2.1162	3.088	2.1216	4.869	2.1302	11.17	2.2142	38.65
4	2.1400	2.959	2.1454	5.169	2.1521	11.82	2.2172	41.08
5	1.9490	3.038	1.9506	4.718	1.9583	10.55	2.0719	33.19
6	1.9799	3.093	1.9815	4.834	1.9893	10.95	2.0958	35.12
7	1.9817	3.090	1.9836	4.842	1.9873	11.30	2.0955	35.78
8	1.9483	3.124	1.9497	4.516	1.9525	10.80	2.0728	32.87
9	2.0012	3.051	2.0033	4.884	2.0063	11.42	2.1185	36.02
10	2.1498	3.151	2.1550	5.011	2.1563	11.59	2.2436	39.42
11	2.1413	3.135	2.1467	5.009	2.1479	11.08	2.2492	38.96
12	2.1481	3.154	2.1531	5.019	2.1542	11.64	2.2544	38.75
61	2.0769	0.514	2.0780	0.561	2.1615	0.622	2.0185	0.714
62	2.0882	0.582	2.0894	0.610	2.1715	0.697	2.0311	0.731
63	2.0615	0.540	2.0613	0.585	2.1412	0.646	1.9786	0.698
64	2.0655	0.547	2.0661	0.614	2.1454	0.650	1.9882	0.689
65	2.0737	0.497	2.0741	0.549	2.1551	0.601	2.0115	0.627
66	2.0666	0.568	2.0711	0.602	2.1531	0.667	2.0003	0.720
67	2.0825	0.514	2.0831	0.559	2.1639	0.616	2.0214	0.680
68	2.0723	0.527	2.0775	0.563	2.1606	0.621	2.0281	0.676
69	2.0594	0.564	2.0651	0.591	2.1470	0.643	1.9920	0.692
70	2.0697	0.601	2.0701	0.664	2.1500	0.675	2.0007	0.721

Measurements above 10 kHz use parallel configuration of external standards. DF calculated from conductance.

Samples maintained at 25°C ± 0.5 by oven.

Higher frequencies corrected for cable and residual bridge inductance, and skin resistance.

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TABLE XXVI. PARAMETER MEASUREMENTS AT 75°C

	310 Hz		500 Hz		1 kHz		2 kHz	
Specimen	C(µF)	DF(%)	C(µF)	DF(%)	C(µF)	DF(%)	C(µF)	DF(%)
1	2.0846	1.725	2.0709	1.663	2.0540	1.577	2.0407	1.590
2	2.0952	1.711	2.0819	1.655	2.0649	1.573	2.0518	1.580
3	2.2282	1.691	2.2120	1.640	2.1944	1.564	2.1809	1.579
4	2.2743	1.726	2.2474	1.651	2.2229	1.714	2.2073	1.682
5	2.0601	1.717	2.0472	1.661	2.0307	1.581	2.0178	1.595
6	2.0935	1.735	2.0799	1.676	2.0626	1.599	2.0494	1.611
7	2.0904	1.713	2.0772	1.656	2.0603	1.579	2.0473	1.594
8	2.0576	1.716	2.0417	1.632	2.0230	1.602	2.0097	1.612
9	2.1076	1.710	2.0951	1.603	2.0779	1.535	2.0649	1.720
10	2.2655	1.651	2.2538	1.598	2.2364	1.528	2.2234	1.574
11	2.2504	1.702	2.2375	1.649	2.2195	1.575	2.2061	1.598
12	2.2589	1.696	2.2462	1.643	2.2283	1.574	2.2152	1.585
61	2.0415	0.1551	2.0381	0.1983	2.0384	0.327	2.0381	0.419
62	2.0519	0.1634	2.0643	0.1975	2.0511	0.295	2.0475	0.401
63	2.0250	0.1500	2.0315	0.1994	2.0267	0.309	2.0221	0.400
64	2.0291	0.1611	2.0297	0.1864	0.0271	0.347	2.0346	0.415
65	2.0386	0.1634	2.034	0.1931	2.0344	0.340	2.0364	0.399
66	2.0329	0.1504	2.0382	0.1925	2.0340	0.291	2.0321	0.417
67	2.0481	0.1782	2.0463	0.1949	2.0450	0.299	2.0440	0.446
68	2.0410	0.1634	2.0401	0.1930	2.0399	0.304	2.0397	0.425
69	2.0236	0.1491	2.0265	0.1901	2.0269	0.315	2.0263	0.413
70	2.0351	0.1523	2.0333	0.1896	2.0322	0.344	2.0311	0.451

	10 kHz		20 kHz		50 kHz		100 kHz	
Specimen	C(µF)	DF(%)	C(µF)	DF(%)	C(µF)	DF(%)	C(µF)	DF(%)
1	2.0190	2.383	2.0081	3.648	2.0635	8.141	2.2438	19.71
2	2.0297	2.398	2.0190	3.585	2.0754	7.928	2.2403	20.75
3	2.1579	2.451	2.1464	3.731	2.2112	8.422	2.3532	22.64
4	2.1832	2.457	2.1712	3.727	2.235	9.151	2.3549	20.21
5	1.9926	2.420	1.9848	3.731	2.0385	7.897	2.2194	19.12
6	2.0239	2.460	2.0156	3.826	2.0713	8.172	2.2414	20.36
7	2.0225	2.434	2.0150	3.822	2.0717	8.353	2.2395	20.91
8	1.9843	2.436	1.9771	3.645	2.0301	7.782	2.2084	18.79
9	2.0401	2.466	2.0316	3.814	2.0882	8.398	2.2534	20.99
10	2.1984	2.490	2.1874	3.911	2.2528	8.662	2.3705	23.85
11	2.1805	2.453	2.1704	3.714	2.2344	8.750	2.3654	23.27
12	2.1894	2.454	2.1790	3.868	2.2436	8.506	2.3725	23.04
61	2.0475	0.501	2.0479	0.599	2.1216	0.641	1.9998	0.695
62	2.0591	0.562	2.4601	0.578	2.1320	0.654	2.0114	0.687
63	2.0310	0.530	2.0304	0.612	2.1147	0.651	1.9473	0.649
64	2.0363	0.514	2.0373	0.601	2.1139	0.629	1.9542	0.637
65	2.0441	0.507	2.0454	0.544	2.1250	0.617	1.9920	0.626
66	2.0365	0.561	2.0429	0.597	2.1200	0.643	1.9701	0.653
67	2.0541	0.500	2.0567	0.543	2.1349	0.625	1.9923	0.634
68	2.0417	0.517	2.0491	0.555	2.1303	0.617	1.9987	0.659
69	2.0299	0.546	2.0354	0.586	2.1154	0.636	1.9414	0.685
70	2.0384	0.583	2.0441	0.629	2.1201	0.662	1.9700	0.701

Samples maintained at 75°C ± 1°C by oven.

Higher frequencies corrected for cable inductance, skin resistance, bridge residual inductance.

TABLE XXVII. PARAMETER MEASUREMENTS AT 125°C

	310 Hz		500 Hz		1 kHz		2 kHz	
Specimen	C(μF)	DF(%)	C(μF)	DF(%)	C(μF)	DF(%)	C(μF)	DF(%)
1	2.0593	1.888	2.0481	1.921	2.0289	2.015	2.0231	2.177
2	2.0678	1.816	2.0573	1.861	2.0398	1.979	2.0318	2.146
3	2.1957	1.831	2.1843	1.867	2.1663	1.982	2.1577	2.151
4	2.2421	1.926	2.2227	1.994	2.2002	1.994	2.1870	2.178
5	2.0408	1.884	2.0297	1.918	2.0124	2.019	2.0018	2.159
6	2.0679	1.862	2.0610	1.898	2.0397	2.008	2.0301	2.156
7	2.0681	1.850	2.0575	1.890	2.0407	2.000	2.0310	2.150
8	2.0368	1.882	2.0211	2.037	2.0019	2.055	1.9916	2.136
9	2.0809	1.911	2.0691	2.018	2.0513	2.199	2.0400	2.229
10	2.2377	1.856	2.2250	1.895	2.2056	1.986	2.1921	2.104
11	2.2194	1.814	2.2077	1.853	2.1907	1.977	2.1807	2.141
12	2.2324	1.836	2.2210	1.870	2.2034	1.9886	2.1909	2.145
61	2.0219	0.1789	2.0189	0.2045	2.0198	0.381	2.0170	0.441
62	2.0331	0.1801	2.0416	0.2056	2.0317	0.324	2.0299	0.474
63	2.0069	0.1746	2.0116	0.2081	2.0024	0.346	2.0016	0.448
64	2.0101	0.1794	2.0089	0.1989	2.0096	0.399	2.0055	0.461
65	2.0157	0.1821	2.0174	0.2004	2.0124	0.385	2.0192	0.427
66	2.0154	0.1763	2.0157	0.2016	2.0137	0.324	2.0098	0.435
67	2.0273	0.1830	2.0268	0.2047	2.0224	0.340	2.0246	0.486
68	2.0206	0.1724	2.0190	0.2038	2.0199	0.373	2.0187	0.441
69	2.0111	0.1701	2.0009	0.1976	2.0070	0.349	2.0060	0.439
70	2.0127	0.1747	2.0176	0.2078	2.0101	0.387	2.0114	0.485

	10 kHz		20 kHz		50 kHz		100 kHz	
Specimen	C(μF)	DF(%)	C(μF)	DF(%)	C(μF)	DF(%)	C(μF)	DF(%)
1	1.9954	3.104	1.9737	4.32	2.0209	9.030	2.1924	21.31
2	2.0049	3.145	1.9832	4.468	2.0337	9.445	2.1914	21.80
3	2.1293	3.134	2.1068	4.532	2.1624	9.926	2.2995	24.16
4	2.1568	3.153	2.1329	4.563	2.1865	10.349	2.2845	26.79
5	1.9735	3.113	1.9522	4.181	2.0014	8.58	2.1636	21.02
6	2.0022	3.169	1.9799	4.405	2.0317	8.995	2.1855	22.54
7	2.0035	3.145	1.9823	4.347	2.0341	9.278	2.1850	23.02
8	1.9634	3.132	1.9426	4.092	1.9896	8.377	2.1614	19.92
9	2.0127	3.086	1.9912	4.287	2.0429	9.098	2.1883	23.05
10	2.1645	3.048	2.1420	4.232	2.2010	9.120	2.3334	23.90
11	2.1521	3.206	2.129	4.525	2.1873	9.472	2.3197	24.06
12	2.1618	3.245	2.1383	4.431	2.1953	9.502	2.3166	24.48
61	2.0271	0.527	2.0292	0.586	2.1012	0.641	1.9699	0.734
62	2.0394	0.601	2.0400	0.614	2.1120	0.704	1.9804	0.740
63	2.0150	0.562	2.0114	0.604	2.1247	0.661	1.9275	0.715
64	2.0147	0.571	2.0179	0.628	2.0900	0.682	1.9394	0.709
65	2.0248	0.523	2.0256	0.584	2.0946	0.636	1.9626	0.654
66	2.0133	0.584	2.0209	0.630	2.1057	0.697	1.9517	0.746
67	2.0327	0.533	2.0347	0.596	2.1024	0.640	1.9720	0.702
68	2.0241	0.549	2.0290	0.584	2.1140	0.654	1.9786	0.696
69	2.0100	0.589	2.0146	0.604	2.1126	0.667	1.9409	0.721
70	2.0189	0.622	2.0186	0.681	2.0914	0.690	1.9504	0.756

Samples maintained at 125°C ± 1% by oven.

Higher frequencies corrected for cable inductance, skin resistance, and residual bridge inductance.

ORIGINAL PAGE IS OF POOR QUALITY

The sensitivity problem affects both AC and DC measurements. The instrument used on this program was a 60 kV Biddle equipped with pulse counting facilities. This is a single-ended detector, so the sensitivity is proportional to $1/C_S$, where C_S is the sample capacitance. Figure 25 shows a calculated sensitivity versus C_S curve for this equipment, derived from values supplied by Biddle. In practice, the sensitivity is somewhat better than this, being about 200 pC at 1 μF.

The second problem is the AC power required to drive the capacitor to voltage. The 2 μF capacitors have an equivalent series resistance of 1325 ohms at 60 Hz. The Biddle test set has a 1 kVA capability, which the 2 μF capacitor exhausts at 35 VAC. In practice, it was never possible to obtain repeatable readings much above 15 VAC, and the operation of the AC plus DC test was rendered very difficult.

The corona test that should be run, given the life test criterion, is an AC plus DC test, of course. The voltage levels required are normally determined by experience and life test. The statement of work required an

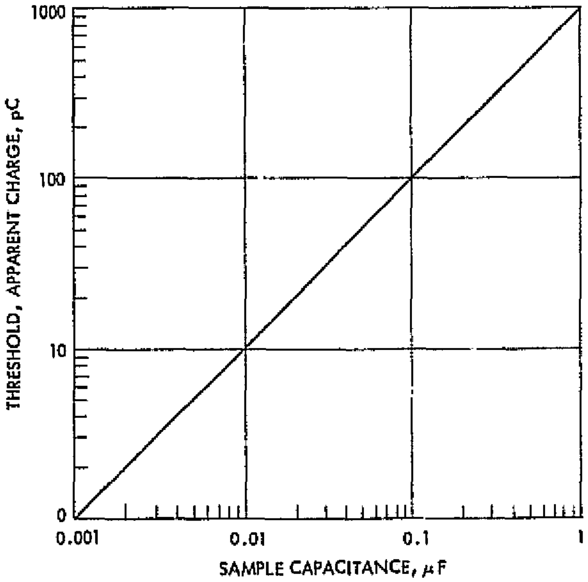

Figure 25. Corona test set sensitivity.

AC test to 110 percent of the rated peak AC voltage, which in this situation would be 44 VAC. Additionally, the following tests were required:

- AC and DC CIV
- Quantitative Pulse Count
- Integrated Energy

The AC and DC CIV is a very straight forward measurement once CIV is defined. The IEEE defines the CIV as:

> "The lowest voltage at which partial discharges exceeding a specified level are observed under specified conditions when the voltage applied to the test object is gradually increased from a lower value. This voltage is expressed as the peak divided by the square root of two."

and it is obvious they were thinking of AC tests only. At Hughes, the CIV is usually defined as that voltage where continuous discharges are observed to occur (on AC), that voltage where bursts of discharges occur (on DC), or that voltage where more than 10 pC per second of apparent charge transfer occurs. Because of the sensitivity problem, the first two of these definitions were adopted for tests tests.

The quantitative pulse count is usually used as a production screen rather than as development tool. The operator does not find the CIV. Instead, he sets the machine to a specified voltage and takes a pulse count. This tends to reduce operator-dependent test results. The pulse counts reported here were all taken at the apparent DC CIV, and are therefore usually quite small.

The integrated energy of corona is a new concept, over which there is some disagreement. It has been hypothesized that the real indicative parameter for corona damage is the apparent charge transfer observed multiplied by the total voltage applied. This product has units of energy and, so the argument goes, represents the amount of energy dissipated in the dielectric. Knowing this number and the bond energies of the dielectric, one may then calculate the dielectric lifetime. Unfortunately, no conclusive set of experiments has been reported to substantiate the theory, and there exist some conceptual difficulties in its application to complex component structures. Therefore, only the CIV and corona spectra are reported.

Because of all of these factors, some experiments on lower-valued capacitors of the same general type were conducted. This work showed nearly identical AD and DC CIVs. The corona versus voltage curves measured had sharp knees at the CIV, so that an increase of only a few volts was required to cause a large increase in apparent charge transfer. This being the case, the CIV was rather insensitive to minimum corona detection threshold. From these results, it was determined that DC CIVs would be sufficient.

An alternative test should be discussed here. Corona in a capacitor on DC voltage causes an increase in the leakage current. Experiments were conducted on several 2.0 µm polysulfone capacitors to obtain leakage current versus voltage curves. A typical curve, from P/N XT 1032-2, S/N 001, is shown in Figure 26. The initial part of the curve where current is insensitive to voltage evidences ionic conduction. The flat sloped part of the curve is a normal ohmic conduction, and the final portion displays the non-ohmic behavior associated with corona and breakdown. The difficulty with this test is that it generally causes irreversible damage to the component if allowed to go into the non-ohmic region.

Preliminary Results

An early experimental design was tested unimpregnated on DC, and then the units were impregnated with Freon and retested. Freon would never be used for production units because of its low boiling point, high vapor pressure, and chemical composition, but it is a convenient high dielectric strength fluid that is easily removable after the test. The data for the unimpregnated units is shown in Table XXVIII; some of the units are bad and others are good, as might be expected. Table XXIX shows the test results of four of these units impregnated and retested. Note that all of the capacitors except one improved their CIVs to greater than 800 VDC. This data proves the value of some sort of liquid impregnant.

A number of unumpregnated 2.0 µm polysulfone 2 µF capacitors were also tested on DC. The results are displayed in Table XXX. A quick calculation yields the extraordinary result that at 700 volts a field of 3.5 MV/cm

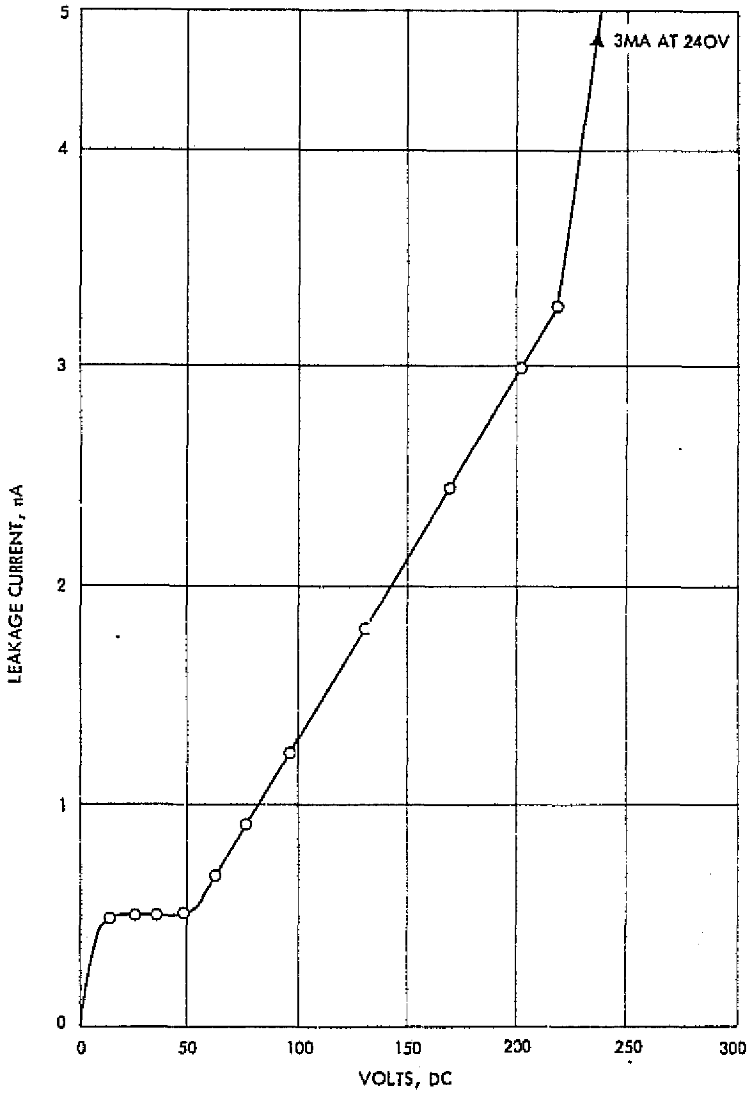

Figure 26. I-V curve for 2 μm 2 μF capacitor.

TABLE XXVIII. UNIMPREGNATED PC CAPACITOR CORONA DATA

Unit	DC CIV	Pulse Counts in 30 Seconds				
		2pC	5pC	10pC	25pC	50pC
1	350	4595	1799	520	64	4
2	>800	0	0	0	0	0
3	>800	0	0	0	0	0
4	500	732	555	468	130	35
5	500	3916	2015	1085	298	29
6	700	318	59	18	1	0
7	700	8543	3610	1081	127	8
8	>800	0	0	0	0	0
9	>800	0	0	0	0	0
10	>800	0	0	0	0	0

TABLE XXIX. CORONA – PC CAPACITORS IMPREGNATED WITH FREON

Unit	DC CIV	Pulse Counts in 30 Seconds				
		2pC	5pC	10pC	25pC	50pC
1	330	1298	213	63	44	0
2	>800	1	0	0	0	0
4	>800	2	0	0	0	0
6	>800	2	0	0	0	0

TABLE XXX. CORONA DATA, 2.0 μm POLYSULFONE CAPACITORS

Unit	DC CIV	Number of Counts in 30 Seconds				
		2.0 pC	5.0 pC	10.0 pC	25.0 pC	30.0 pC
1	530	182	44	9	2	0
2	450	194	37	2	0	0
3	360	94	0	0	0	0
4	340	169	4	0	0	0
5	430	110	37	7	0	0
6	700	1	0	0	0	0
7	700	0	0	0	0	0
8	700	0	0	0	0	0
9	390	234	11	2	0	0
10	700	0	0	0	0	0

was being supported by the dielectric, with no evidence of deterioration. Subsequent tests revealed that each component had been broken down in the corona tests, without the operator even noticing.

The way that this came about was fairly easy to diagnose. The particular technician was used to making AC acceptance-type tests, where the voltage is set and the counter then run for 30 seconds to record the corona. In DC tests, the burst of corona only occurs at inception for a brief period, and was therefore never seen because the technician did not have the counter on and was not looking at the scope. This was unfortunate, but it was a learning experience.

The present DC test set-up utilizes a step-type DC supply, because that was the quietest supply available. It was very hard to recognize corona at the time the voltage was turned up, because of the noise associated with the step. The modified test apparatus now being constructed for development work uses a very heavily filtered continuously variable DC supply for DC and AC plus DC work.

Final Results

One hundred capacitors were tested, in conformance with the contract specification. The results for the PVF2 components are shown in Table XXXI, and the results for the polysulfone components are shown in Table XXXII.

At the conclusion of these tests, insulation resistance (IR) tests at 45 VDC were conducted. These revealed that several PVF2 components and almost all of the polysulfone components had been damaged in the corona testing, despite the fact that the operator had been warned of the problem. Therefore, additional components of both types were obtained for the life tests. These components were characterized by IR only.

INSULATION RESISTANCE

All components were measured for leakage current (insulation resistance) at 45 VDC. Table XXXIII displays these readings for all components. Those capacitors with specimen numbers 100 and greater are replacement components as described above.

ESTIMATES OF ENERGY DENSITIES AND MAXIMUM OPERATING VOLTAGE

Maximum operating voltages are usually derived from the CIV by reducing the voltage allowed from the CIV to some lower value. Because of the uncertainty about the corona results, these values are presented for the PVF2 unit only, in Table XXXIV.

The energy densities at the required voltage were as follows:

PVF2 – 0.188 at 500 V
Polysulfone – 0.2 at 500 V

for uncased components with terminations but without leads.

TABLE XXXI. 2.0 μF POLYVINYLIDENE FLUORIDE CORONA DATA, 30 SECOND COUNT

Hughes Serial	VDC	2 pC	5 pC	10 pC	25 pC	50 pC
001	700	0	0	0	0	0
002	700	0	0	0	0	0
003	700	3	0	0	0	0
004	700	0	0	0	0	0
005	700	0	0	0	0	0
006	700	0	0	0	0	0
007	700	0	0	0	0	0
008	700	44	2	0	0	0
009	700	0	0	0	0	0
010	700	0	0	0	0	0
011	700	0	0	0	0	0
012	700	0	0	0	0	0
013	700	0	0	0	0	0
014	700	0	0	0	0	0
015	700	0	0	0	0	0
016	700	0	0	0	0	0
017	700	3	0	0	0	0
018	700	0	0	0	0	0
019	700	0	0	0	0	0
020	700	0	0	0	0	0
021	700	8	0	0	0	0
022	700	0	0	0	0	0
023	700	0	0	0	0	0
024	700	0	0	0	0	0
025	700	0	0	0	0	0
026	700	314	16	7	0	0
027	700	0	0	0	0	0
028	700	0	0	0	0	0
029	700	0	0	0	0	0
030	700	0	0	0	0	0
031	700	0	0	0	0	0
033	700	0	0	0	0	0
034	700	1	0	0	0	0
035	700	0	0	0	0	0
036	700	0	0	0	0	0
037	700	0	0	0	0	0
038	700	0	0	0	0	0
039	700	0	0	0	0	0
040	700	119	79	38	3	0
041	700	0	0	0	0	0
042	700	0	0	0	0	0
043	700	0	0	0	0	0
044	700	341	4	1	0	0
045	700	0	0	0	0	0
056	700	0	0	0	0	0
047	700	0	0	0	0	0
048	700	0	0	0	0	0
049	700	0	0	0	0	0
050	700	0	0	0	0	0

TABLE XXXII. 2.0 μF POLYSULFONE CORONA DATA, 30 SECOND COUNT

Hughes Serial	VDC	2 pC	5 pC	10 pC	25 pC	50 pC
051	700	2	0	0	0	0
052	700	0	0	0	0	0
053	700	1	0	0	0	0
054	700	0	0	0	0	0
055	700	0	0	0	0	0
056	700	0	0	0	0	0
057	700	1	0	0	0	0
058	700	0	0	0	0	0
059	700	2	0	0	0	0
060	700	0	0	0	0	0
061	700	0	0	0	0	0
062	700	0	0	0	0	0
063	700	0	0	0	0	0
064	700	0	0	0	0	0
065	700	0	0	0	0	0
066	700	0	0	0	0	0
067	700	0	0	0	0	0
068	700	0	0	0	0	0
069	700	0	0	0	0	0
070	700	0	0	0	0	0
071	700	0	0	0	0	0
072	700	247	99	31	4	0
073	700	0	0	0	0	0
074	700	1	0	0	0	0
075	700	77	3	0	0	0
076	700	0	0	0	0	0
077	700	0	0	0	0	0
078	700	0	0	0	0	0
079	700	0	0	0	0	0
080	700	0	0	0	0	0
081	700	0	0	0	0	0
082	700	0	0	0	0	0
083	700	0	0	0	0	0
084	700	2	0	0	0	0
085	700	1	0	0	0	0
086	700	0	0	0	0	0
087	700	1	0	0	0	0
088	700	0	0	0	0	0
089	700	0	0	0	0	0
090	700	0	0	0	0	0
091	700	0	0	0	0	0
092	700	0	0	0	0	0
093	700	0	0	0	0	0
094	700	0	0	0	0	0
095	700	0	0	0	0	0
096	700	2	0	0	0	0
097	700	0	0	0	0	0
098	700	0	0	0	0	0
099	700	0	0	0	0	0
100	700	0	0	0	0	0

TABLE XXXIII. SPECIMEN INSULATION RESISTANCE

Specimen Number	IR (MΩ)			Specimen Number	IR (MΩ)		
	25°C	75°C	125°C		25°C	75°C	125°C
1	563	136	9	26	346	137	8
2	360	150	9	27	375	115	7
3	360	125	10	28	346	137	9
4	385	140	8	29	346	148	9
5	450	118	9	30	409	166	9
6	391	176	10	31	346	148	9
7	391	173	10	32	375	209	10
8	404	147	9	33	346	156	10
9	416	160	8	34	346	142	8
10	290	230	13	35	375	136	8
11	346	155	9	36	387	137	9
12	360	114	17	37	298	117	7
13	350	137	9	38	312	130	8
14	367	146	10	39	360	166	10
15	412	117	11	40	360	125	9
16	375	167	12	41	385	140	10
17	346	143	9	42	321	136	8
18	346	157	8	43	346	155	8
19	351	141	10	44	382	167	10
20	351	132	8	45	310	118	7
21	409	166	12	46	318	121	7
22	382	167	9	47	321	141	8
23	507	145	10	48	351	132	10
24	316	136	8	49	449	176	9
25	316	147	8	50	385	226	12

(Continued next page)

(Table XXXIII, concluded)

Specimen Number	IR (kMΩ)			Specimen Number	IR (kMΩ)			Specimen Number	IR (kMΩ)		
	25°C	75°C	125°C		25°C	75°C	125°C		25°C	75°C	125°C
51	18.8	15.0	3.1	77	30.0	23.3	4.1	103	3.46	1.47	0.13
52	37.5	18.5	2.6	78	37.5	28.1	5.7	104	3.46	1.59	0.12
53	30.0	21.0	7.6	79	25.0	17.6	2.9	105	4.09	2.30	0.19
54	30.0	22.0	5.4	80	45.0	32.1	6.1	106	3.46	1.36	0.11
55	45.0	31.2	9.6	81	25.0	20.0	2.9	107	3.75	1.67	0.14
56	25.0	17.9	3.1	82	23.7	16.7	2.7	108	3.46	1.14	0.10
57	23.7	18.6	2.7	83	34.6	17.8	2.6	109	3.46	1.46	0.13
58	34.6	21.0	8.0	84	25.0	19.6	2.7	110	3.46	1.35	0.10
59	30.0	20.6	7.6	85	32.1	26.9	4.1	111	3.46	1.33	0.11
60	25.0	18.0	3.1	86	32.1	25.3	4.0	112	3.46	1.43	0.10
61	32.1	23.2	6.2	87	37.5	28.4	6.3	113	3.75	1.56	0.14
62	32.1	23.2	5.4	88	45.0	34.4	7.1	114	3.75	1.67	0.12
63	18.8	16.1	2.6	89	45.0	31.8	6.8	115	40.9	29.6	8.3
64	37.5	25.0	7.2	90	28.1	17.9	2.3	116	30.0	15.2	4.2
65	45.0	31.0	10.0	91	34.6	26.9	5.7	117	23.7	18.6	2.6
66	34.6	22.5	4.2	92	30.0	22.1	4.3	118	28.1	19.8	2.8
67	28.1	18.8	2.6	93	23.7	16.8	2.4	119	34.6	21.0	5.9
68	30.0	22.0	2.6	94	32.1	24.4	3.6	120	25.0	17.8	4.8
69	40.9	31.0	5.4	95	32.1	26.9	3.8	121	45.0	83.4	10.1
70	34.6	20.0	2.7	96	25.0	21.2	4.2	122	37.5	26.0	8.4
71	32.1	17.6	2.6	97	18.8	14.6	2.3	123	32.1	19.4	3.7
72	18.8	14.7	2.1	98	18.8	14.7	2.3	124	37.5	21.6	5.8
73	45.0	32.1	4.7	99	37.5	29.9	7.1	125	30.0	17.1	2.6
74	45.0	31.0	4.5	100	21.0	14.1	4.1	126	30.0	19.2	2.6
75	40.7	28.6	6.1	101	3.26	1.27	0.11	127	18.8	15.5	2.1
76	18.8	14.7	3.2	102	3.75	2.01	0.15	128	32.1	23.0	4.9
								129	32.1	22.6	4.7

TABLE XXXIV. PVF2 OPERATING VOLTAGE

Specimen Number	VDC	Specimen Number	VDC
01	500	26	250
02	500	27	500
03	500	28	500
04	500	29	500
05	500	30	500
06	500	31	500
07	500	32	500
08	300	33	500
09	500	34	500
10	500	35	500
11	500	36	500
12	500	37	500
13	500	38	500
14	500	39	500
15	500	40	300
16	500	41	500
17	500	42	500
18	500	43	500
19	500	44	200
20	500	45	500
21	500	46	500
22	500	47	500
23	500	48	500
24	500	49	500
25	500	50	500

Operating voltage for 2 years life, derived from corona data, Table XXXI.

V. LIFE TESTS

The life testing prescribed in the statement of work was simple and straightforward. The requirements were:

- 20 capacitors, 10 each dielectric
- 500 VDC plus 20 V 10 kHz AC
- 2500 hours, 25°C exposure

In addition to this life test, a step-stress DC test was run on 20 components made from very thin polysulfone. The results of this extra work are reported after the main life test results.

CIRCUITRY

The design of the life test circuit requires one main inverter and five secondary inverters. In Figure 27, the main inverter and one of the secondary inverters are shown; the four remaining inverters attach to the four sets of windings on T_1. This inverter design, as is, produces 10 kHz sine waves across the test components. If the filters between T_2 and the test components are removed, the voltage across the test components is a clean 10 kHz square wave.

INFANT MORTALITY

In a previous section, it was mentioned that it was found possible to destroy components in corona testing by application of stepped DC voltages. As the life test was begun, it was found that some components expired within a few minutes of application of the DC voltage. This indicates that the correct predictive test for operating voltage and life has yet to be devised and implemented.

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C.2

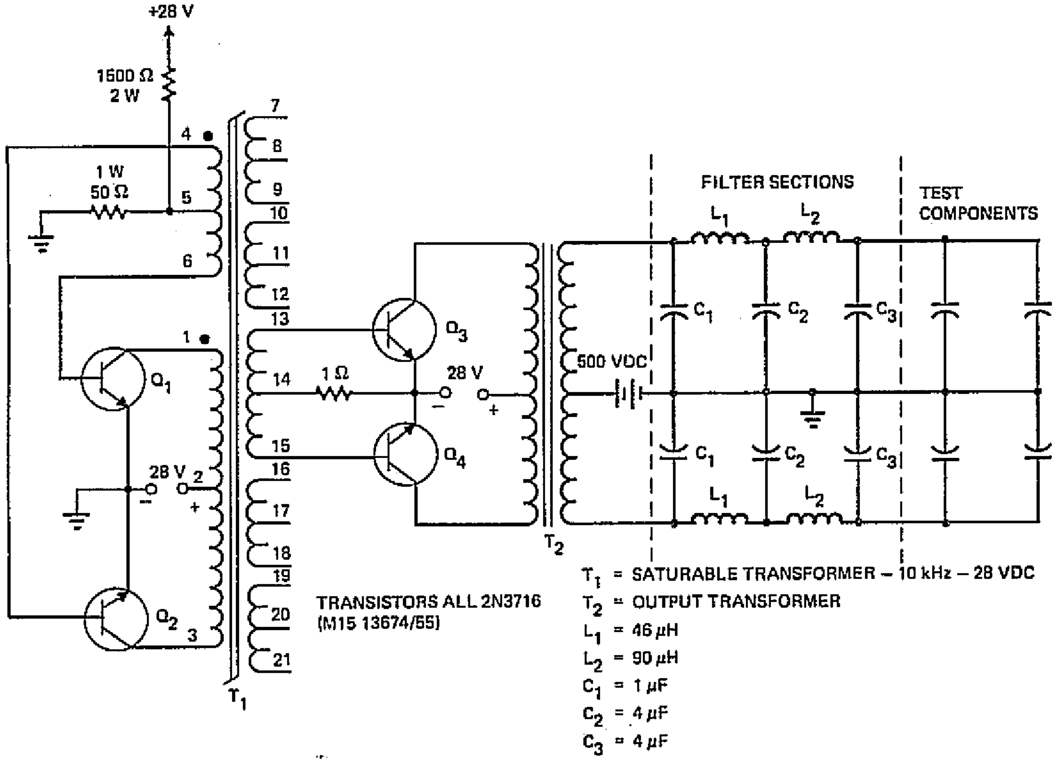

Figure 27. Life test circuit.

At the beginning of the test, 10 polysulfone and 10 PVF2 capacitors were placed in the inverters, and the DC voltage was slowly turned up, no AC being applied. After a few hours all remaining polysulfone components had been destroyed. The inverter was then completely filled with PVF2 components; the PVF2 infant mortality is shown in Table XXV. The infant mortality of the PVF2 components appears excessive but this is easily explained. The specimens numbered 101-124 were additional components, obtained after damage has been done to some of the original components during corona test. These additional capacitors were not given the usual tests to screen out the weak units to which the original components (01-50) had already been subjected. Thus, the failures. After these failures, 20 PVF2 components were in the inverters with 500 volts DC across them. The AC power was then applied. During this test, none of the capacitors became warm to the touch, ever though 2.5A flowed continuously through each of them.

TABLE XXXV. PVF2 INFANT MORTALITY

Speciman	Failure Type	Time to Failure (Hours)
117	A	<1
109	A	<1
120	A	<1
114	A	<1
116	A	<1
124	A	<1
119	A	<1
107	B	3
104	B	24

Legend: A = Failure on DC voltage
B = Failure at 500 VDC

LIFE TEST RESULTS

The 2500 life test had one failure at 696 hours. The remaining 19 components passed the life test and retained their original electrical values. The components tested are shown in Table XXXVI.

STEP STRESS DC TEST

Twenty 2μF capacitors made from very thin polysulfone (2μm) were put on a step-stress DC life test. In this test, voltage is applied at a low value and then increased in uniform steps at regular intervals. Table XXXVII shows the results of this test.

TABLE XXXVI. PVF2 FINAL LIFE TEST SPECIMENS

Specimen	Failure Type	Hours
023	C	696
013	N	2500
014	N	2500
016	N	2500
020	N	2500
022	N	2500
024	N	2500
026	N	2500
028	N	2500
029	N	2500
030	N	2500
101	N	2500
102	N	2500
103	N	2500
105	N	2500
108	N	2500
110	N	2500
111	N	2500
113	N	2500

Legend: C = Failure under life test conditions

N = No failure

TABLE XXXVII. DC STEP-STRESS LIFE TEST RESULTS, 2.0 μm COMPONENTS

Voltage	Hours	Number of Failures
100	0-168	0
125	169-336	0
150	337-504	0
175	505-672	0
200	673-840	0
225	841-1008	1
250	1009-1176	5
275	1177-1344	5
300	1345-1512	9

VI. FAILURE ANALYSIS

Numerous failure analyses were performed on failed capacitors throughout the development program. Every failure was found to be a corona-induced puncture on the dielectric film. The majority of these failures were found to occur between 2 and 60 cm from the start of the winding, centering on a wrinkle; isolated instances of failure at the metallization edge were also found. One capacitor suffered a cracked glass seal.

TECHNIQUE AND RESULTS

Failed capacitors were normally recognized by large measured DF, low DC resistance, or by blown fuses in the life test apparatus. What follows is a detailed description of the failure analysis of three PVF2 2.0 μF capacitors.

The first step in the failure analysis is physical examination and electrical test. The results of this were:

Serial Number	DC Resistance	Appearance
106	100 kΩ	Cracked end seal
109	700Ω	Normal
114	1.5 kΩ	Normal

Next, the capacitors were x-rayed. The x-ray examination revealed the presence of several dark spots on the radiograph near the end of the unit that had leaked (the radiograph is not presented here because it would not reproduce satisfactorily).

Each capacitor was then opened by grinding off one end of the case. Units 114 and 109 appeared normal, but unit 106 was badly burned, as Figure 28 shows. Because of the extensive damage to 106 it was impossible to

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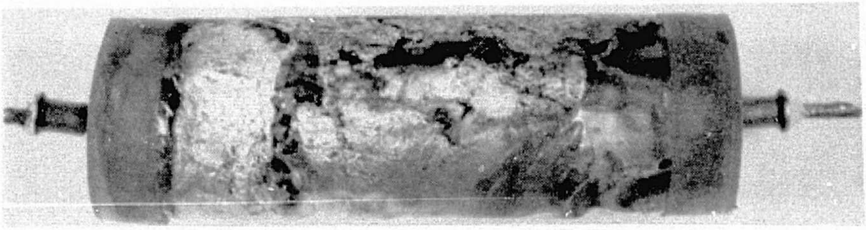

Figure 28. Sample No. 106, heavily damaged, magnification 2.9X.

unroll it, so it was cut open. This operation revealed extensive internal charring, more than would normally be expected from a failure at 500 VDC. This evidence led to the installation of fault current limiters in the life test apparatus, so that more artefacts of the original failure would be preserved.

Unit 109 was unrolled, and the failure was found very close to the start of the winding, as is shown in Figure 29. The dotted line in the figure is the point to which the films inserted through the core are folded back. Since one of the insulation punctures is located at this discontinuity, and the others lie immediately above on successive layers, this is the most probable cause of failure.

Unit 114 was unrolled, and two areas of insulation damage were found. One area, shown in Figure 30, was about 73 cm from the start of the film (total film length was about 450 cm). This damage appears to be centered on a wrinkle, although that does not show clearly in the photograph. A second area of damage was found 61 cm from the start of the film, and is shown in Figure 31. This is a small burn that occurs exactly on a wrinkle.

As a consequence of this work, it was found that the large wrinkles in these components are caused primarily by the two grooves on the surface of the core.

OTHER RESULTS

Not all capacitors failed because of large burned areas centered on wrinkles. One capacitor assembled from the 2 μm film showed an defect neither on a wrinkle nor near a metallization edge. In general the 2 μm capacitors had far fewer wrinkles than the components made from thicker film.

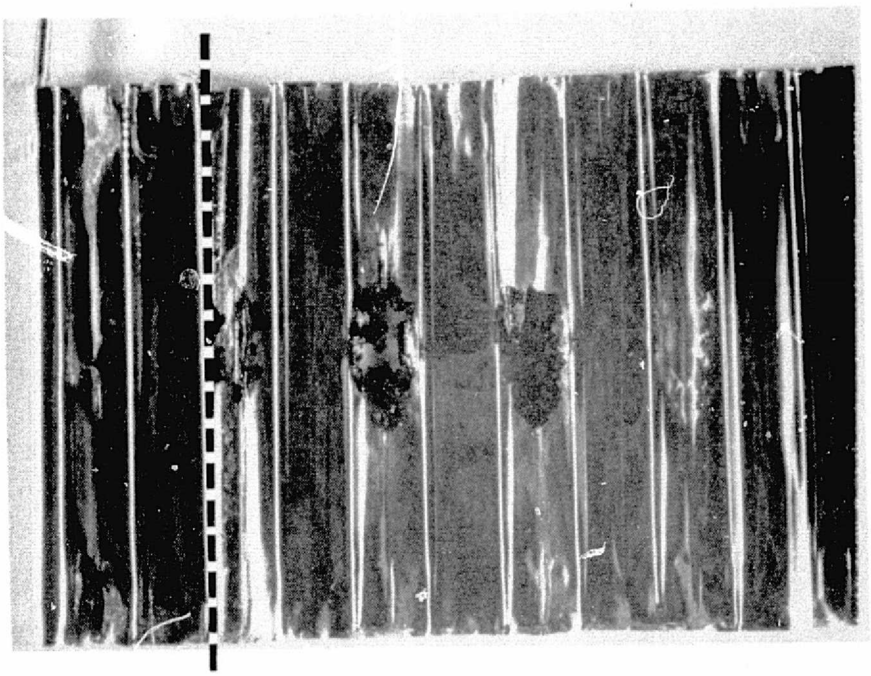

Figure 29. Sample 109, failure near core. Core is to the left. Dotted line shows end of folded start leader, 2.7X.

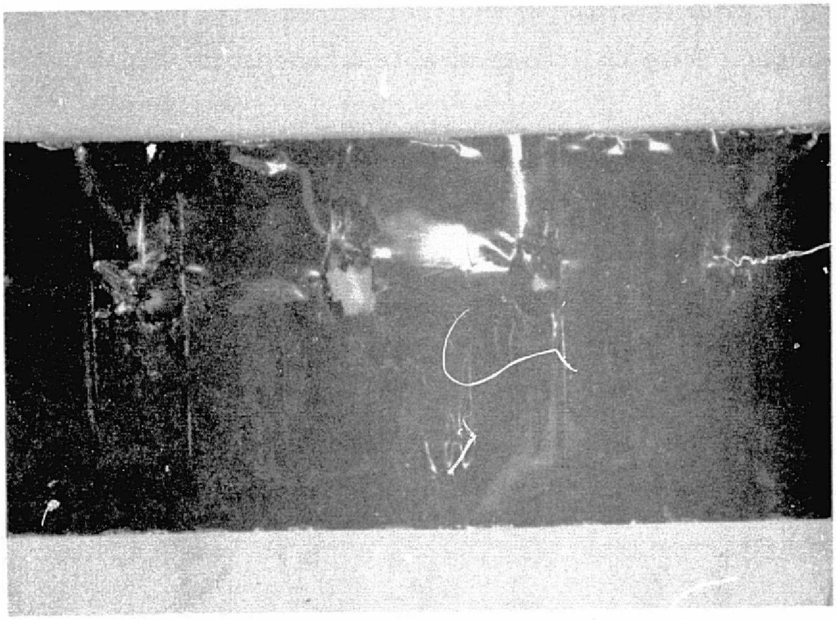

Figure 30. Sample 114 failure 73 cm from start, 2.0X.

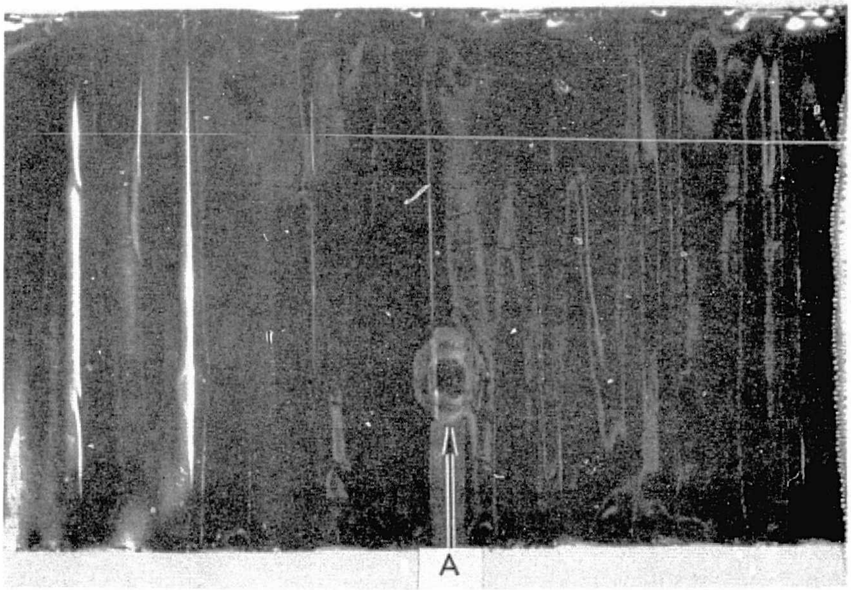

Figure 31. Sample 114, failure 61 cm from start, marked by arrow, 2.8X.

One edge failure was found, in a 3.56 μm polysulfone component. This is illustrated in Figure 32, and is relatively self-explanatory.

Some contamination of the metallized film surface was discovered in the PVF2 units. This is demonstrated in the scanning electron micrograph of Figure 33, which is at an enlargement of approximately 500X. The feature running across the micrograph is a wrinkle, and the small humps are contamination. It is not thought that any of the failures were caused by such contamination.

Finally, it was found that the PVF2 in the finished components is much more wrinkle-free than the polysulfone. Part of this is assuredly caused by the thinner polysulfone being more wrinkle-prone. Part of it may be attributable to too high and long a bake temperature cycle (bake cycles are used to increase the IR). This effect is illustrated in Figure 34, where Figure 34a is PVF2, and Figure 34b polysulfone, at the same magnification.

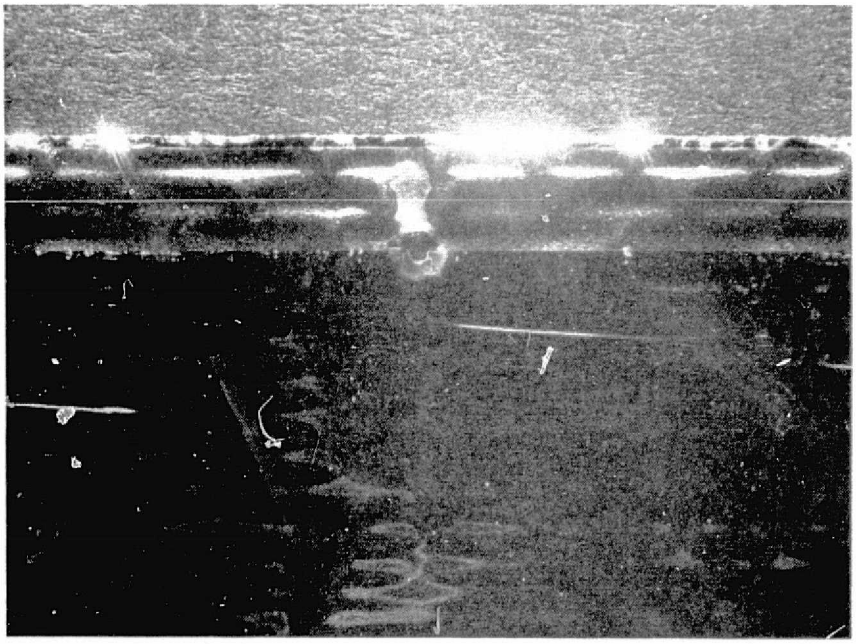

Figure 32. Edge failure in polysulfone, 55X.

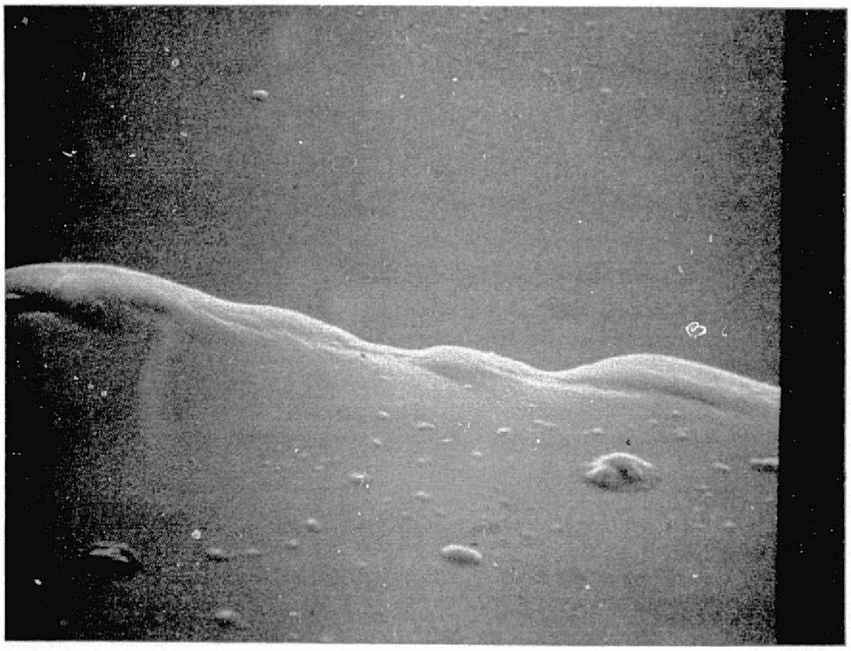

Figure 33. SEM photo of PVF2 film, showing wrinkle and contamination, 530X.

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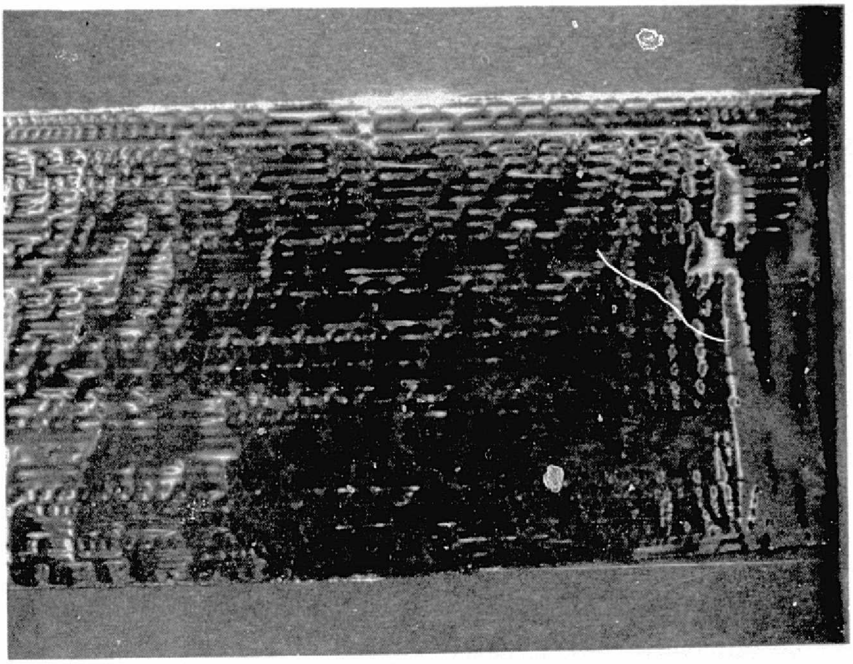
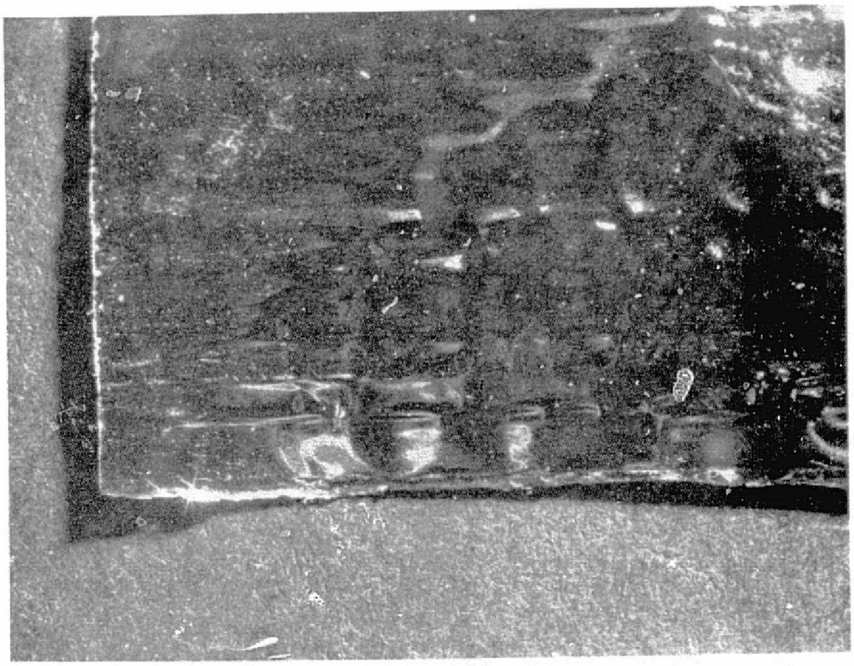


a. Polysulfone



b. PVF2

Figure 34. Difference in film surfaces.

VII. CONCLUSIONS AND RECOMMENDATIONS

This work has corroborated the original thesis that the most effective way to build high energy density capacitors for medium voltages is to make as perfect a component as possible. It was demonstrated that it is possible to build PVF2 capacitors with active densities of about 0.19 J/g that have life-times greater than 2500 hours. When these uncased components are combined with the proposed 0.989g case an effective cased density of about 0.11J/g is obtained.

Much was learned during the course of this program about capacitor failure mechanisms and ways to circumvent them. To control wrinkles, very careful assembly, constant dynamic tension winding, and carefully tailored baking cycles are needed. Some additional work needs to be done on the impregnation technology of small components.

One area of increasing concern is the problem of corona or other types of testing for reliability prediction. In this area Hughes has begun work on a highly modified corona test instrument that will have the following specifications:

Sensitivity — 10 pC across 4 μF
Power handling — 125 kVA

This will enable the testing of 1 μF capacitors to 15 kVAC. Progress thus far has been excellent, with present sensitivity 10 pC across 1 μF.

Finally, there are two items that have become clearer and clearer as this program has progressed. The first has to do with testing and failures. It is common practice to subject components intended for high reliability applications to severe electrical overstress to weed out weak units. This concept was fine when capacitors were designed to 25×10^3 V/cm (10 V/mil). But it will not work when the operating stress is 1.3×10^6 V/cm, since any sort of severe overstress will simply cause wholesale avalanches and breakdown of the insulation. Some other means of finding potential failures must be found. When it becomes possible to make a perfectly physically configured capacitor, these techniques will still be needed, since at these stresses some failures caused by film imperfections would still occur.

The second thought is really a forewarning to component engineers and system designers. To achieve the very maximum energy density, it will be found necessary to specifically tailor components, cases, and in some cases circuits, to the particular application. No longer will one component be optimum for all applications.

VIII. ACKNOWLEDGEMENTS

Dr. Norman Bilow, Mr. Richard J. Holbrook, and Mr. John Mosher performed much of the work on Chemical Compatibility, assisted in the laboratory by Joseph F. Biro and Walter Butterfield. Water determinations were performed by Marcia Verzwyvelt. Mr. Holbrook wrote the section on Chemical Compatibility. Mr. Robert D. Gourlay and Mr. Allan Lange worked on the life tests and parameter measurements, with the laboratory work being performed by Mr. Don L. McWilliams. Mr. Edward E. G. Wong performed corona tests and fabricated prototype capacitors. Mr. Robert S. Buritz designed the cases and wrote the section on case design. Mr. Harry Ashe designed and tested the inverters for the life tests. Mr. Alfred M. Schwider designed the adhesive system and application method for the flat flexible capacitor. Analyses on the Scanning Electron Microscope were performed by Mr. Roy L. Hackett. Capacitor failure analyses were performed by Mr. James Erickson. Mr. Robert W. Gerbode and Mr. Arnold Tuckerman were the cognizant Department Managers.

Mr. David Kellerman and Mr. John Conklin of Component Research, Incorporated gave technical assistance and produced many of the capacitors used on this program.

APPENDIX I — STATEMENT OF WORK

1.0 Scope of Work

The objective of this investigation is to develop compact, high storage energy, low weight capacitors for a wide variety of applications. The capacitors will have the following characteristics as a design goal:

A. energy density - greater than 0.1 joule/gram

B. lifetime - 2 years or greater at continuous use

C. operating condition - 500 Volts dc, 5A. ac, at frequencies up to 100 KHz

D. operating temperature range - -30° to 125°C

2.0 Specific Tasks

The contractor shall furnish all personnel, supplies, equipment, and facilities needed to complete all of the work described in each of the tasks listed below:

TASK 1. The Contractor shall examine different dielectric materials, including but need not be limited to these listed below, and recommend two materials to be used in the development of capacitors that will operate at 500 Volts at frequencies up to 100 kHz, and that will result in an energy density greater than 0.1 joule/gram for the assembled capacitors.

A. polysulfone
B. polyvinylidene Fluoride
C. polycarbonate
D. Teflon
E. Kapton
F. Mylar
G. Parylene

TASK 2. The contractor shall develop detailed procedure for building capacitors that have an energy density greater than 0.1 joule/gram for assembled capacitors using the two dielectrics recommended in TASK 1 to conform to the following specification goals:

A. Rating: 2 microfarads, 500 V dc, 5 A ac, 10 kHz

B. Case Temperature: The rated operating case temperature range of all capacitors shall be -30°C to 125°C.

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C. Design life: The design life shall be at least two (2) years of continuous operation at rated conditions.

D. Design of capacitor packaging: The capacitor packaging shall be a simple two terminal design with both terminals insulated from the case. The packaging shall be of a type generally suited for use in space or aircraft environments.

The detailed procedure shall be approved by the NASA Project Manager before proceeding to TASK 3.

TASK 3. The contractor shall construct 50 samples using each of the two dielectrics recommended in TASK 1, for a total of 100 capacitors. The specifications listed in TASK 2 shall apply.

TASK 4. The Contractor shall subject 10 samples of each of the two dielectrics used to a combined dc-ac 2500 hour continuous test consisting of 500 V dc with a 20 V, 10 kHz ac voltage superimposed. Description of all circuits, instrumentation, and facilities used for this test shall be submitted to the NASA contract manager for approval. Life tests shall not be started until approval is given.

TASK 5. The contractor shall analyze all capacitors that failed, to determine the nature and cause of failure. The analysis shall, as a minimum, include photographs that illustrate the capacitor's internal damage and examinations and tests to determine the point of initial failure.

TASK 6. Capacitor Characteristics

For each of the 100 capacitors constructed in TASK 3, the contractor shall provide the energy density in units of joules/gram, a recommended maximum operating voltage for two (2) year life, and curves showing the following information:

A. Capacitance versus frequency up to 100 kHz at temperatures of 25°C, 75°C and 125°C.

B. Dissipation factor (DF) versus frequency up to 100 kHz at temperatures of 25°C, 75°C and 125°C.

C. The dc resistance at temperatures of 25°C, 75°C and 125°C.

Corona Information

Using a suitable corona test set, each of the 100 capacitors built under TASK 3, shall be tested for existence of corona at voltages up to 110 percent of rated ac peak voltage. The ac and dc corona inception voltage shall be recorded. A quantitative measurement of the corona intensity shall be made from two picocoulombs through 50 picocoulombs. The integreated energy of the corona shall also be measured over the corona test period. With the corona data an estimate of the lifetime of the capacitors shall be made.

APPENDIX II

DETAILED PROCEDURE FOR CAPACITOR FABRICATION

This detailed procedure is presented in response to the requirements of NASA Lewis Research Center Contract NAS 3-18925, Exhibit A, Paragraph 2.0, TASK 2. While not exhibiting formal manufacturing documents, it contains sufficient information to enable another contractor or agency to fabricate capacitors similar to those fabricated for this program.

The principal problem in designing a capacitor with an energy density greater than 0.1 J/g is in designing the working part, called the core, for some density in excess of 0.1 J/g. Because of this, and because case making and encasing are a standard well-known technology, discussion of the packaging is omitted except where it affects the energy density or electrical properties.

This process may be divided into five distinct parts: film cleaning, winding, termination, impregnation, and sealing. Each of these is more or less self-explanatory. Each part of the process will be recognized as a part of commercial capacitor manufacture. However, upon examination of the detailed procedure important differences will be found. These differences all lead to the manufacture of a more reliable component of higher energy density.

FILM CLEANING

Whether metallized or unmetallized polymeric film is used, it must be cleaned prior to winding. As received from the manufacturer, the film is usually "dirty", and winding experiments have shown a much higher failure rate for components made of the "as-is" film as contrasted to that for components made of cleaned film. Mass-spectrographic analyses done on small areas of film after solvent extraction have revealed no particular impurity, so the film probably is not uniformly contaminated. Additionally, much of the contamination is thought to be particulate matter.

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Film cleaning must be done either on a winding machine or on a specially constructed cleaning machine. This latter tool consists of a reeling and un-reeling mechanism, tensioners, and a solvent bath with wipers. The important specification is: Edge wander < 12 µm. Tension is adjusted so that the film does not stretch. The solvent used may be any of a number compatible with the films. In the present case, isopropyl alcohol or Freon is used. The solvent bath is continuously recirculated and filtered to remove impurities. The wipers remove the solvent from both sides of the film.

WINDING

Winding is done on a special machine that maintains constant static tension on the film, and has edge wander smaller than 12 µm. For this program, the following film combinations were used to fabricate capacitors as required in TASK 3:

Film	Thickness	Width	Margin
Polycarbonate	3.566 µm	2.699 cm	0.15875 cm
Polyvinylidene Fluoride	6 µm	2.699 cm	0.15875 cm

These films are Al metallized on one side in a vacuum metallizer, and have a clear margin on one edge, as indicated. The metallization is 1 to 2 ohms per square. The sections that result from these specifications are about 0.2 J/g at 500 V.

A standard single dielectric layer construction was employed. As wound, therefore, two layers with opposite margins were used. Capacitance was continually monitored in this process, so that the winding could be terminated at the proper point. The configuration of the finished winding was cylindrical, with a 0.2159 cm core. This core was of a type compatible with the winding machine. In the present case, it was a Teflon double-D core, which fits on the two D-shape fingers of the winding machine. The starting films secured to it by interleaving between the Ds.

The winding was done in a positive-pressure controlled-access clean room. The operator controlled the winding machine manually, and wound slowly to keep edge runout small (below 12 µm). Slow winding also allows the static film tension to be maintained constant. This tension was adjusted so that during the starting operation the film is not quite stretched. After the section was wound, it was secured with Kapton tape and passed on to the next operation.

TERMINATION

The electrical connection to the metallization was accomplished by a variant of the extended-foil construction common to larger components. Flame-sprayed tin was used to connect to alternate layers, as in Figure 1. A wire with two gold leads welded on was inserted into one of the "D" openings at each end, and the two gold leads were ultrasonically soldered to the tin, as in Figure 2. The larger wires protruded through the case for the final termination.

This termination was not particularly porous to capacitor impregnants, so stripes were masked off on each end, and remained unsprayed to enhance impregnation.

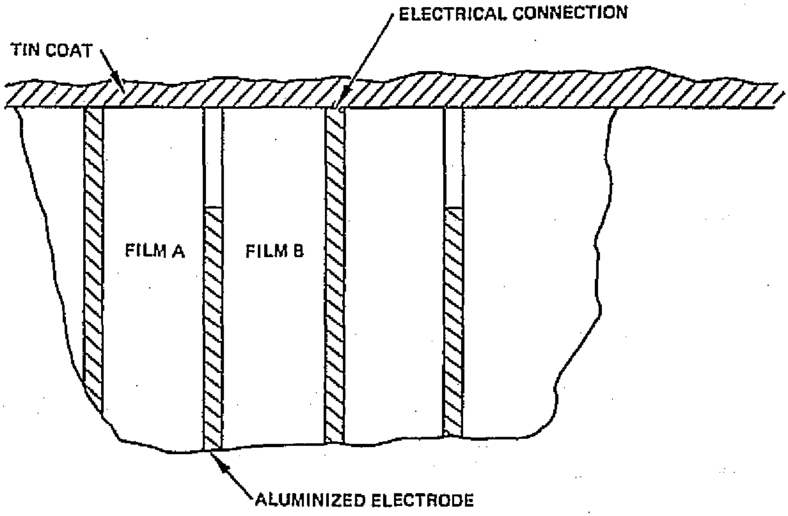

Figure 1. Detail of metallized film connections.

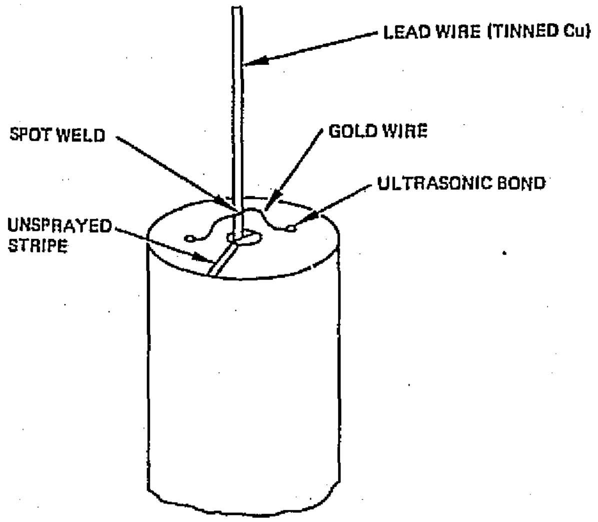

Figure 2. End connection detail.

IMPREGNATION

Numerous fluids are used for capacitor impregnation, so that the impregnation process is necessarily tailored to the peculiarities of the particular fluid. The process described here is for mineral oil. Very slight modifications are used for silicone fluids.

The capacitor sections were placed in a vacuum oven and baked at a temperature above 100°C for at least 24 hours. The temperature depended on the material, and was about 110°C for both materials to be used here. The length of time for the bake was determined by monitoring the insulation resistance (IR) of the components. When the IR stopped changing, the components were sufficiently dry.

While the sections were being baked, the fluid was treated to remove particles, moisture, and ionic contaminants. Particles were removed by filtering, moisture by vacuum baking, and ionic contaminants by resin ion exchange columns. These treatments were necessarily severe, the end point specifications being:

particles — none larger than 0.4 μm
water ≤ 35 ppm
resistivity $\geq 10^{14} \Omega$-cm

During this process the impregnation fluid was also degassed by the vacuum-bake cycle. The cycle lasts 24 hours at 100°C and $\leq$10 microns pressure. Analyses for water and particles were made, and the resistivity measured, at the end of this time.

The baked components were removed from the vacuum oven and placed in an oil impregnation unit. This apparatus consisted of a heated vacuum chamber, an evacuated fluid storage reservior, connecting tubes, and miscellaneous fixtures. The apparatus in present use at Hughes is an un-modified Red Point ESL-2020. The components remained in the heated evacuated chamber for 3 hours, and were then filled with heated fluid from the side loader (fluid storage reservoir). Following this, the components were allowed to cool under vacuum to room temperature. Then the vacuum was broken and the component cases sealed.

SEALING

It is most desirable to seal the containers under vacuum at the end of the impregnation cycle. Where the cases are large, this may be possible. However, if it is not, the process must be designed to minimize the amount of gas/contaminants re-introduced into the oil.

APPENDIX III

ADDITIONAL HIGH-FREQUENCY BRIDGE CORRECTIONS

The additional corrections that are to be applied at frequencies above 10 kHz are:

1. Series bridge transformer inductance
2. Transformer series resistance
3. Cable and connector skin effect.

SERIES INDUCTANCE

The capacitance corrections can be made from data supplied on the primary standards test report. Series bridge inductance is:

$$L = \frac{C_M - C_A}{\omega^2 C_M C_A} \qquad (1)$$

where C_M and C_A are the measured and actual capacitances values at ω. This calculation gives:

$$L = 4.108 \times 10^{-7} \text{ H} \qquad (2)$$

This test was conducted without the connecting coaxial cable, which has an inductance of 1.55×10^{-7} H. Therefore, the total inductance is:

$$L = 5.2630 \times 10^{-7} \text{ H} \qquad (3)$$

The correction formula is:

$$C_A = \frac{C_M}{1 + \omega^2 L C_M} \qquad (4)$$

where L is as above and C is in units of farads.

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RESISTANCE

The correction for series resistance is:

$$D_R = D_M - \omega R C_A \tag{5}$$

and all that remains is to estimate R. The data supplied by GR indicate R = 0.010 ohm within the bridge. To this must be added the resistance caused by skin effect at high frequencies in the coaxial cable.

$$r_s = 8.3\sqrt{f}\left(\frac{1}{d} + \frac{1}{D}\right)L \tag{6}$$

This gives:

$$D_R = D_M - \omega C_A\left[0.01 + 8.3\sqrt{f}\left(\frac{1}{d}+\frac{1}{D}\right)L\right] \tag{7}$$

At the frequencies of interest here, r_s is important only above 10 kHz, where the values are:

$r_s = 2.1 \times 10^{-2}$ at 100 kHz

$r_s = 1.48 \times 10^{-2}$ at 50 kHz

$r_s = 9.39 \times 10^{-3}$ at 20 kHz

The correction as a whole is very small below 20 kHz.