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DRAFT

DEVELOPMENT OF NEW DIELECTRIC SYSTEMS TO PROVIDE A MINIMUM
VOLUMETRIC EFFICIENCY OF TEN JOULES PER CUBIC INCH

QUARTERLY PROGRESS LETTER #3

Period: August 8, 1968 to November 8, 1968

To Sandia Corporation
Albuquerque, New Mexico

Contract No. 57-5750

Buyer: J. L. Miller, 4312

Requester: A. F. Hurford, 1432

SPRAGUE ELECTRIC COMPANY
North Adams, Massachusetts

December 3, 1968

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SUMMARY OF PROGRESS

In the third quarter of this investigation of new dielectric systems for high energy storage capacitors the major effort has been concentrated on polymers and glasses loaded with barium titanate, with a lesser effort on nonferroelectric ceramics. The volumetric energy storage factor ($K_0 E_{BD}^2$, where K_0 is the dielectric constant at low field, and E_{BD} is the dielectric breakdown field) is now about 500-1000 for loaded polymers, 100-350 for loaded glasses, and 30-40 for barium tetratitanate. Preliminary measurements of actual energy discharge (in less than 100 nanoseconds) indicate that for both the loaded polymers and loaded glasses the amount of energy discharged is about half of that calculated from $K_0 E^2$.

In the polymer films loaded with barium titanate, chlorinated polyvinylchloride (Geon) continues to show certain advantages over polycarbonates: Geon films are flexible even at 67 volume % of barium titanate, though polycarbonates become brittle over 30 volume %; also Geon films usually have higher breakdown strengths, leading to $E_{BD}^2 K_0$ values of nearly 1000. Appreciable differences in dielectric effectiveness of different preparations of barium titanate are becoming clear; the most effective powders have the least porosity, which suggest that air pockets may develop with porous powders.

Studies of $B_2O_3-Al_2O_3-BaO$ glass filled with barium titanate show much promise over the earlier studied SiO_2 --containing glasses; at 85 volume % loading K_0 is 550 and E_{BD} is 500-800. One reason for this improvement in properties is the absence of new phases which develop in the SiO_2 glasses after heating with barium titanate.

Barium tetratitanate, a nonferroelectric ceramic, has been made with reproducible E_{BD} values of greater than 1000 Volts/mil in 10 mil discs. Breakdown is believed to result from porosity which may be eliminated by hot-pressing in vacuum.

CERAMIC-LOADED POLYMER DIELECTRICS

D. Kelley

Chlorinated PVC Binder Systems

Films were cast from both tetrahydrofuran (THF), and THF-cyclohexanone (CH) solutions of Hi-Temp Geon Vinyl 101-EP (B. F. Goodrich) mixed with milled dispersions of BaTiO_3 . All films are flexible up to the 67 volume percent BaTiO_3 , but tear resistance decreases slightly with higher loading.

Table I

Geon Binder Systems

<u>Volumetric</u> <u>Energy</u> <u>Storage*</u> <u>Factor</u> $K \cdot E_{BD}^2$	<u>BaTiO₃ Source</u>	<u>Volume %</u> <u>BaTiO₃**</u>	<u>Breakdown Voltage, Kv/mil</u>		<u>Dielectric</u> <u>Constant (K_o)</u>
			<u>Method #1</u>	<u>Method #2</u>	
565	Auty, Lot 4-4 ^a	36	4.0 $\pm$ 0.3	7.1 $\pm$ 2.4	11.3
536	TAM ^b Expt. Grade	39	4.9 $\pm$ 0.5	6.3 $\pm$ 1.2	13.6
965	"	57	3.8 $\pm$ 0.6	5.5 $\pm$ 1.3	32.2
950	"	67	3.5 $\pm$ 0.4	5.6 $\pm$ 0.8	30.6
675	---	0	15.0 $\pm$ 0.9	7.5 $\pm$ 2.5	ca.3

^a Rotary calcined

^b Titanium Alloy Mfg. Div., National Lead Co.

* Using the highest value of E_{BD} obtained

** Assuming no void content.

Electrical properties are summarized in Table I. Two different methods were used to measure breakdown voltages. In Method #1, the film specimen was clamped between spring-loaded point contacts and immersed in a silicone oil bath. D.C. voltage was continuously increased as in the ASTM "short-time" method. Method #2 was performed by A. Dequasie of the Engineering Laboratories. It involved placing the specimen on a flat copper plate and contacting the top surface with a spring-loaded 1/4 inch copper rod with a flat smooth end. Method #2 was carried out in air; the 30-80% higher values of breakdown voltage for loaded films might be attributed to a more rapid increase of applied electric field strength than in Method #1, since measurement in air usually results in slightly lower values (10-20%). (Note: Unloaded Geon films are much more sensitive to environment--the breakdown voltage in oil being more than 100% greater than in air.)

In an attempt to toughen the highly loaded film, Hycar CTBN liquid rubber (B. F. Goodrich) was incorporated in some formulations. Tear resistance was not noticeably improved and dielectric strength was affected only according to Method #2.

Table II

Effect of Hycar CTBN on 67 vol.% filled Geon

<u>Hycar Level, phr</u>	<u>Breakdown Voltage, Kv/mil</u>		<u>Dielectric Constant</u>
	<u>Method #1</u>	<u>Method #2</u>	
5	3.0 $\pm$ 1.0	4.8 $\pm$ 0.9	30.5
10	3.0 $\pm$ 1.1	3.8 $\pm$ 1.0	32.0
20	3.2 $\pm$ 1.3	3.0 $\pm$ 1.4	32.3

A series of four organic orthotitanate esters (Tyzors, duPont) were included during the milling of certain BaTiO₃ dispersions in an attempt to promote wetting of the BaTiO₃ surface by the binder. No improvement in dielectric strength was found.

Storage for several weeks produces an increase in E_{BD} of 15-25%, even though films were rigorously dried prior to the first measurements.

Polycarbonate Binder Systems

Data summarized in Table III and Figures 1, 2, and 3. Methods #1 and #2 for measuring breakdown voltage (E_{BD}) agree much better than in the case of Geon binder systems. Films loaded with greater than about 25 volume percent $BaTiO_3$ are very brittle when dried. This correlates with the decrease in dielectric strength beyond this point as seen in Figure 1. Even though dielectric constant continues to increase beyond the 25% level, lower values of E_{BD} prevent the volumetric energy storage factor from exceeding a value of 400 (Figure 3).

Films prepared using rotary-calcined $BaTiO_3$, Lot 4-4, (from Dr. Auty) and Sprague Production Type 644AB are exceptions due to generally higher dielectric strength and the greater slope of the K vs. $BaTiO_3$ level plot (broken curves in Figures 1, 2, and 3). Nevertheless, film brittleness beyond the 25% level is a serious limitation for handling and processing. (Note: The 60% level is the apparent limit for casting coherent films).

Dielectric constant appears to decrease after the 50% level. This was also found with Geon binder and might be explained by an increase in void content with loading. This is suggested by the observation that film thickness increases with loading for films cast under identical conditions.

In order to make useful comparisons of energy storage capabilities with that of unloaded polycarbonate film, one must know the relative dielectric strengths under the conditions of application. While measurements of E_{BD} under silicone oil and in air agree well for loaded polycarbonate films, they do not for unloaded films. Values of E_{BD} in oil are approximately twice as great as those measured in air (14.0 ± 1.4 and 7.3 ± 0.2 Kv/mil respectively). These values lead to volumetric energy storage factors of 610 and 164, respectively, and can be compared with data in Table III and Figure 3.

Ganex V-type polyvinyl pyrrolidone (GAF Corp.), Vitel PE-200 polyester (Goodyear), tricresyl phosphate, and tri (2-ethylhexyl) phosphate were incorporated in formulations in attempts to toughen loaded films. None were effective.

Polyurethane Binder Systems

Loaded films cast from THF solutions of Estane Type 5707F1 (B. F. Goodrich) had rougher surfaces than either polycarbonate or Geon composites. Therefore, the thickness values used to calculate E_{BD} are less reliable. One set of films contained

Table III

Polycarbonate Binder Systems

Binder Type	BaTiO ₃ Source	Volume % BaTiO ₃	Breakdown Voltage, Kv/mil		Dielectric Constant, K _o	Volumetric Energy Storage Factor, $\frac{K_o E_{BD}^2}{O}$
			Method #1	Method #2		
Merlon M-60	Rotary-Calcined, Lot PPl, 2-2, 4 (Auty)	8	4.4 $\pm$ 0.7		4.8	92
		22	5.4 $\pm$ 0.7			
		23	3.7 $\pm$ 0.9			
		27	5.7 $\pm$ 0.7		14.2	382
		34	2.4 $\pm$ 0.8			
		35	2.0 $\pm$ 0.8		21.6	86
Makrofol K-G	Same as above	29	3.7 $\pm$ 0.3		13.3	180
		42	2.0 $\pm$ 0.3		26.5	106
		50	3.2 $\pm$ 0.5	3.6 $\pm$ 0.8	32	326
		52	2.7 $\pm$ 0.4		--	
		59	3.9 $\pm$ 1.0	1.6 $\pm$ 0.3	24	356
Merlon M-60	Spray-calcined, Cyclone-Bowen, Run 3 (Auty)	22	3.3 $\pm$ 0.6		12.3	133
Lexan 101	TAM Exptl.	24	3.9 $\pm$ 0.5		10.4	158
		26	6.0 $\pm$ 0.3		10.9	392
		39	4.2 $\pm$ 0.5		20.7	362
Lexan 101	Sprague 644AB, Lot 10-11	23	6.0 $\pm$ 0.6			
		25	5.6 $\pm$ 0.4			
		38	5.4 $\pm$ 0.2			
M-60	Rotary-calcined, Lot 4-4 (Auty)	9	6.2 $\pm$ 1.2	5.6 $\pm$ 0.4	3.2	122
		20	6.3 $\pm$ 1.0	5.2 $\pm$ 1.1	7.3	292
		31	5.0 $\pm$ 1.0	4.2 $\pm$ 0.5	26.8	670
		34	4.7 $\pm$ 0.6	4.2 $\pm$ 0.8	35.2	780

* In report of August 26, 1968 to F. M. Fowkes

5 phr diphenylmethane-4, 4'-diisocyanate (Multrathane M) (MDI) and was cured at 135°; another contained 3 phr benzoyl peroxide (Bz_2O_2):

Table IV
Cured Estane Binder Systems

<u>Curing Agent</u>	<u>Volume % BaTiO₃</u>	<u>E_{BD}, Kv/mil (Method #1)</u>	<u>Dielectric Constant, K_O</u>	<u>$\frac{K_O E_{BD}^2}{10^6}$</u>
MDI	14	4.2 $\pm$ 0.8	14.5	250
MDI	20	3.2 $\pm$ 0.6	18.8	190
Bz ₂ O ₂	24	1.7 $\pm$ 0.3	--	--
MDI	33	2.2 $\pm$ 0.5	45.3	220
Bz ₂ O ₂	42	2.4 $\pm$ 0.4	38.1	220
MDI	48	0.9 $\pm$ 1.1	(shorted)	--
MDI (10 phr)	0	9.3 $\pm$ 0.4	--	--

Curing with 10 phr MDI increased the breakdown voltage of unloaded Estane film by only 25% (7.4 $\pm$ 0.6 to 9.3 $\pm$ 0.4 Kv/mil); and curing of loaded films did not appear to be beneficial.

Table V
Uncured Estane Binder Systems

<u>Vol. % BaTiO₃</u>	<u>E_{BD}, Kv/mil</u>	<u>Dielectric Constant</u>
0	7.4 $\pm$ 0.6	----
36	2.5 $\pm$ 0.2	42.2
44	1.7 $\pm$ 0.3	----

Significantly higher dielectric constants are obtained, however, compared with equivalent polycarbonate and Geon binder systems at lower BaTiO₃ levels. Estane films also exhibit a maximum dielectric constant in the range 30-35% BaTiO₃.

Capacitor Discharge for BaTiO₃-Filled Polymer Film Dielectrics

Discharge characteristics were measured for some of the film samples described above. The discharge current vs. time plot is sinusoidal in all cases. The time interval for complete discharge varies from 15 to 30 nanoseconds with 20 nanoseconds being the interval for the great majority of cases. (Some ringing of the circuit was noted, however).

Stored energy was calculated from the equation $J_o = 1/2 C_o V^2$. Energy discharged was calculated from the equation for a sinusoidal wave of duration time t :

$$J = R I_m^2 \frac{t}{2}$$

Average values of discharge efficiency are of the order of unity (Table VI).

Scanning Electron Micrographs

Four samples of polymer films loaded with barium titanate powder were examined by scanning electron microscopy. Surfaces and fractured edges were examined, and stereo pairs were furnished for a more detailed study of the degree of aggregation, the extent of air entrapment, and other structural information.

1. In polymer films loaded with powders having an adsorbed layer of another polymer the powders were badly agglomerated, and many agglomerates contained air pockets.
2. Heavily loaded polycarbonate films have a sponge-like structure of barium titanate with only part of the hole filled with polymer, but with the chlorinated polyvinylchloride the individual barium titanate particles appear to be polymer-coated and much less air is entrapped.
3. Barium titanate powders prepared by spray-calcination show flake-shaped aggregates of low porosity.

Table VI

Capacitor Discharge Characteristics of
BaTiO₃-Filled Polymer Dielectrics

<u>Binder</u>	<u>BaTiO₃-Type</u> <u>(volume %)</u>	<u>Applied</u> <u>Field</u> <u>(Kv/mil)</u>	<u>J/J₀</u>	
Polycarbonate	Lexan 101	TAM (24)	2.0	1.07
		(26)	3.0	1.16
		(39)	2.0	1.31
	M-60	PP (8)	2.3	0.42
		(35)	1.0	0.30
		(27)	2.4	1.30
	K-G	PP (50)	1.3	<u>1.72</u>
			Ave.	1.04
	Geon 101 EP	TAM (67)	1.3	1.43
			(57)	1.5
(39)			1.7	1.64
(67)			0.8	2.41
4-4 (36)		1.7	<u>1.39</u>	
		Ave.	1.79	
Estane 5707F1	644TX (14)	1.8	0.85	
		(20)	1.4	<u>1.12</u>
		Ave.	0.99	

Overall average 1.30
(J/J₀)

CERAMIC-LOADED GLASS DIELECTRICS

I. Burn

Since the last report was issued the influence of glass composition on the dielectric properties of barium titanate-in-glass composites has been studied further. The aim is to combine the high dielectric constant of barium titanate with the higher breakdown strength of glass, and the problem is what composition and what fraction of glass to use.

Presently composites are being prepared by conventional ceramic methods, i.e. cold pressing of powders or screening of slurries, followed by a firing cycle to produce sintering. With these methods, the materials slump or stick to the substrate before porosity is eliminated if the glass fraction is too high. When the barium titanate fraction is sufficiently high to support the glass by surface tension forces, densification occurs when a temperature is reached at which there is significant solution of the barium titanate in the glass. In cases where excessive solution occurs, new crystalline phases of low dielectric constant may precipitate on cooling with a resulting decrease in dielectric strength.

Dielectric Constant

As mentioned in an earlier report, the presence of large amounts of network-forming oxides in the glass (e.g. B_2O_3 or SiO_2) generally leads to excessive formation of borate or silicate phases during firing with accompanying decrease in the dielectric constant of the composite. Most work to date therefore has been carried out with systems in which glasses can form with a minimum (10 wt% or less) of B_2O_3 and/or SiO_2 . The range of dielectric constant usually obtained for the glass systems investigated so far, with 55 vol% $BaTiO_3$ ceramic, is illustrated in Fig. 4, which also shows the effect of a small D.C. bias. New phases were detected by X-ray diffraction to some extent in all cases.

Because glasses containing large amounts of lead or bismuth are very absorbent to high energy radiation, composites containing glasses which are less affected by irradiation are being investigated. Unfortunately for the present purposes, these glasses have high melting temperatures ($\geq 1300^\circ C$) and tend to be very reactive towards barium titanate at the temperatures necessary for firing. Earlier work with silica and borosilicate glasses indicated that the barium titanate was almost entirely consumed by reaction during sintering. More recent work with $BaO-Al_2O_3-B_2O_3$ glass, however, has shown that although the K of composites containing this glass is relatively low compared with those

containing the lead-bismuth glass (viz. approx. 150 for 55 vol% BaTiO₃) no new phases were detected after firing. Thus with higher BaTiO₃ loadings increased dielectric constant can be achieved without appreciable decrease in dielectric strength; for example, with 85 vol% BaTiO₃ (before firing), a K of 550 is obtained with a breakdown strength in the range 500-800 Volts/mil. So far measurements on this material have been carried out only on pressed discs but screened layers are presently being prepared for more detailed evaluation of the material. In addition, composites with similar glasses containing CaO, or CaO + MgO instead of BaO will be examined. Not only are these glasses resistant to radiation damage but they have good durability and have an electrical resistivity similar to that of fused silica.

Dielectric Strength

Problems are still being encountered with obtaining reproducible dielectric strengths. With screened layers the presence of fabrication flaws (pin-holes, etc.) is the main cause of failure of the dielectric. Accordingly, measured strengths for a given material generally decrease with increasing area and decreasing thickness of the dielectric. Most breakdown tests have so far been carried out with pressed discs (usually 20-30 mils thick) because of their ease of preparation. As can be expected, the strengths are very sensitive to porosity. This is illustrated by the results obtained with a selection of materials which was tested after firing under vacuum. The vacuum firing reduced the porosity from a typical 10-12% for air-firing to about 5%, and there was in almost every case an appreciable increase in breakdown strength, as shown in Table VII.

Table VII

Maximum Breakdown Strengths (Average
of 5 in parenthesis) in Volts/mil

<u>Material</u> <u>(Glass +55 vol% BaTiO₃)</u>	<u>Vacuum firing</u>	<u>Air firing</u>
PBB-3	1086 (787)	558 (499)
PBCB-2	811 (564)	721 (578)
PBZB-1	813 (687)	597 (519)

Material (Glass +55 vol% BaTiO ₃)	Vacuum firing	Air firing
C-glass	621	418
	(524)	(366)

Tests on screened layers generally give similar results to those in Table VIII if the dielectric thickness is about 2 mils or more (cf Fig. 5).

Energy Storage

Attempts are being made to evaluate the energy storage capabilities of the materials both by calculation and from discharge measurements. Using the usual notation, the energy stored by a capacitor is given by

$$J = \int Q \, dV = \iint C \, dV \, dV \quad (1)$$

and to evaluate J it is necessary to know how C changes with V . Although C is also a function of the charging time, the decrease in C with time is generally small (1-2%) compared with the change with V for the times involved in measurement (~ 1 min) unless there is obvious degradation of the material. Measurements of capacitance under a D.C. bias (e.g. Fig. 5) indicate that to a fairly close approximation, at least up to about 500 Volts/mil, and above 100 Volts/mil, (see Fig. 6).

$$CV = aV^n \quad (n < 1) \quad (\text{above } 100 \text{ Volts/mil}) \quad (2)$$

a and n are constants for a particular material; in the case of material B, for example, $n = 0.61$.

For field strengths less than 100 Volts/mil it is found that, for the present purposes, it is sufficiently accurate to assume a linear relationship between C and V .

$$C = C_0 - bV \quad (\text{below } 100 \text{ volts/mil}) \quad (3)$$

Thus the energy stored at a particular voltage can be evaluated by adding the contributions of the energy below and above 100 Volts/mil obtained by substituting (2) and (3) into (1). The

calculated energy as a function of V for a capacitor of material B is shown in Fig. 7 compared with the corresponding values of $1/2 C_0 V^2$ (C independent of V), and of $1/2 CV^2$ where C is the measured value at a particular V . As expected, the calculated values lie between the two extreme cases.

Several preliminary discharge tests have now been carried out on some of the materials and have revealed that, at least for the type of units examined so far, the discharge is very rapid (≈ 40 n sec). However, this has made precise evaluation of the energy discharged difficult as the rise time of our oscilloscope is of similar magnitude (~ 13 n sec). The results obtained, as yet only for units of relatively small capacitance (~ 100 pf) at low fields (≈ 350 V/mil), indicate a somewhat lower energy storage than the calculated values, e.g. Table VIII.

Table VIII Energy storage of capacitors comprising
55 vol% BaTiO₃-45 vol% C-glass

<u>Capacitance</u> <u>pf</u>	<u>Field Strength</u> <u>V/mil</u>	<u>$1/2 C_0 V^2$</u> <u>Joules ($\times 10^5$)</u>	<u>$J_{\text{meas.}}$</u> <u>Joules ($\times 10^5$)</u>	<u>$J_{\text{calc.}}$</u> <u>Joules ($\times 10^5$)</u>
610	300	7.6	5.0	6.5
700	345	8.75	6.2	7.5
643	315	8.0	4.5	6.9

Efforts are presently being made to improve the accuracy of the discharge measurements and it is hoped that, with tests on more materials, it will be possible to make a closer check on the validity of the energy storage calculations.

CERAMIC DIELECTRICS

R. P. Auty

The prediction is that there would be a thickness dependence of the breakdown voltage/mil thickness if voids constitute an appreciable part of the thickness of the dielectric. Since our previous work was on 25 mil discs, thinner discs were pressed, fired, lapped to 10 mils or less, annealed and tested with point contacts and 20 sq. mm. gallium electrodes. Results are tabulated in Table IX and demonstrate that the variations become much greater with point contacts and that higher values of breakdown voltage/mil are obtainable. The conclusion is that although the ultimate breakdown voltage of the material may be higher a practical limit may be 1000 Volts/mil.

Since a practical limit on high voltage capabilities has been reached, it seems reasonable to explore the relationship, if any, between breakdown strength and dielectric constant for high purity, single phase materials. As a first step in this direction, CaTiO_3 powder ($K = 150$) has been prepared and sintering runs have been initiated to determine optimum conditions for densification.

Table IX.

Breakdown Field for BT_4 ($K = 40$)

	<u>Sample</u>	<u>Thickness (mils)</u>	<u>Breakdown Field (Volts/mil)</u>	<u>Notes</u>
1.	20 sq. mm. Ga. electrodes	7.6	1050	Breakdown
2.	"	10.4	1250	"
3.	Point Contacts	8.6	1750	30 sec.
4.	"	8.6	1160	Breakdown
5.	"	9.7	1340	"

FIGURE 1. BREAKDOWN VOLTAGE vs. LOADING FOR POLYCARBONATE BINDER SYSTEMS

Figure 2 Dielectric Constant vs Length for Polyacetylene Under Stress

Dielectric Constant

See Legend to
Fig. 1

Figure 3

Winding energy, (W_e) in

See Legend A:
Fig. 1

FIG. 5

K v E for screened layers (55 vol% BaTiO₃)

A - with C-glass (premelts + PbO)

B - with C-glass

C - with lead-bismuth-cadmium borate glass

FIG. 6

10⁴ x KE

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