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METEOROID SENSOR MATERIAL DEVELOPMENT

By

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THE FRANKLIN INSTITUTE RESEARCH LABORATORIES
Philadelphia, Pennsylvania 19103

Final Report

**CASE FILE
COPY**

September 20, 1968 to September 20, 1969

Prepared under

Contract No. NAS-9-8830

for

NASA Manned Spacecraft Center
Space Physics Division
Meteoroid Sciences Branch
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ABSTRACT

Several dielectrics have been evaluated for use in micrometeoroid sensor capacitors. Dielectrics evaluated included Parylene N and C, Polysulfone, Lexan, Estane, Tyril, Acryloid, Penton and aluminum oxide in various composite arrangements. The capacitor sensors fabricated were subjected to electrical testing, which included the measurement of dielectric properties, electrode resistance, D.C. leakage current and the effect of polarity reversal, shelf-life tests, environmental tests, mechanical tests, and particle impact tests.

The most satisfactory micrometeoroid sensors were made from films of Parylene. Parylene performed well in all the tests. Other materials did not give reliable sensors as frequently, although Lexan and Polysulfone composites with Estane performed satisfactorily in all tests but the environmental tests and the impact tests. Only one polymer dielectric, Acryloid, was found to be completely unusable.

It was concluded that the higher reliability of Parylene was due to the greater precision of control in the Parylene film process over that in the film casting process used for the other materials. Thus the test results in the report do not reflect on the material properties of the dielectrics tested, but only the present feasibility of making sensitive units using fabrication processes at hand.

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I. INTRODUCTION

This project was undertaken to evaluate materials for fabricating capacitors with large areas and thin dielectrics for use in sensors to measure meteoroid velocity, speed and direction. Since the particle must penetrate at least one dielectric without altering its trajectory, the capacitor with its electrodes must be thin and light (200 micrograms per cm^2), and its total thickness cannot exceed 1 micron. The units must be stable to the mechanical perturbations experienced in liftoffs of space vehicles and to degradation from solar irradiation, high vacuum and electric fields of 10,000 volts per cm.

II. FABRICATION OF MICROMETEOROID SENSORS

1. Preparation of Dielectric Films

The majority of the dielectrics were prepared by casting film-forming polymers, dissolved in solvents non-miscible with water, upon a quiet water surface. After the films spread and solidified, they could be lifted off the water. Even polymers which do not make flexible thick films can be treated in this way. Parylene N and C films were obtained from Union Carbide Corporation, Chemicals and Plastics Division, Bound Brook, New Jersey. The materials selected for study, with their symbols used in this report, are shown in Table I. The concentrations of the casting solutions for the polymer dielectrics are also shown in Table I.

Table I
DIELECTRIC MATERIALS AND CASTING SOLUTIONS

Polysulfone (Union Carbide Resin P-47)	P
Parylene N	(PN)
Parylene C	(PC)
Aluminum Oxide	(Al ₂ O ₃)
Lexan, General Electric Co. polycarbonate resin	(L)
Tyrl-767, Dow-Chemical Co. styrene- acrylonitrile copolymer	(T)
Estane, B.F. Goodrich polyurethane elastomer 5740X071	(E)
Acryloid, Rohm and Hass acrylic ester resin B72	(A)
Penton, Hercules Powder Co. chlorinated polyether elastomer	(Pn)

Acryloid was rejected very early on the basis of its poor film strength.

The best casting solutions were prepared as follows:

<u>Polymer</u>	<u>Weight</u>	<u>Solvent</u>
Lexan	5 g	50 ml cyclohexanone
Estane	10 g	100 ml tetrahydrofuran
Tyrl-767	5 g	50 ml cyclohexanone
Polysulfone	5 g	50 ml cyclohexanone
Penton	6 g	50 ml cyclohexanone

Most of the film structures evaluated were composites of Estane and other polymers fabricated on a supporting frame which was designed to utilize the elastomeric properties of Estane. Filtered solutions were cast on water surfaces in a laminar flow work station and lifted off onto the Lucite frame shown in Figure 1. Polymer solutions were filtered through an ultrafine sintered glass funnel (pore size 0.9 to 1.4 microns). The laminar flow work station was equipped with a 0.3 micron air filter. Films from resinous polymers were lifted onto the flat side of the frame which was then again submerged under the water surface to lift off an Estane film onto the beveled side. Only an elastomer will stretch into the bevel to contact the film on the other side. In general it was found that single films have pinholes and did not make capacitors with high leakage resistance.

Parylene films were metallized with gold electrodes at the Union Carbide facility and mounted onto the frame shown in Figure 1. They were then shipped to The Franklin Institute for test evaluation. Parylene films were bonded onto the flat side of the frame, with one metallized surface at the interface between the surface of the Lucite frame and the Parylene film.

Al_2O_3 films were obtained by anodizing commercial Reynolds Wrap sheet aluminum (1.5 mil thickness) on both sides using a 0.4% by weight aqueous solution of a 1:1 by weight citric acid: ammonium citrate dry mixture. The anodized sheet of aluminum, approximately 1-1/2" x 1-1/2" in area, was bonded on to the flat surface of the Lucite frame with GE RTV-103 silicone cement, covering the open area, 1-1/4" x 1-1/4" in the frame. The top layer of Al_2O_3 was removed with a 42% NaOH solution and the aluminum substrate was removed with a 1:3 HCl water solution. The remaining Al_2O_3 film-Lucite frame composite was washed in distilled water and could be composited with cast polymer films in the usual way or used alone. When a higher purity aluminum sheet was used an uneven oxide coating was obtained which could not be used in the fabrication of capacitor dielectric films.

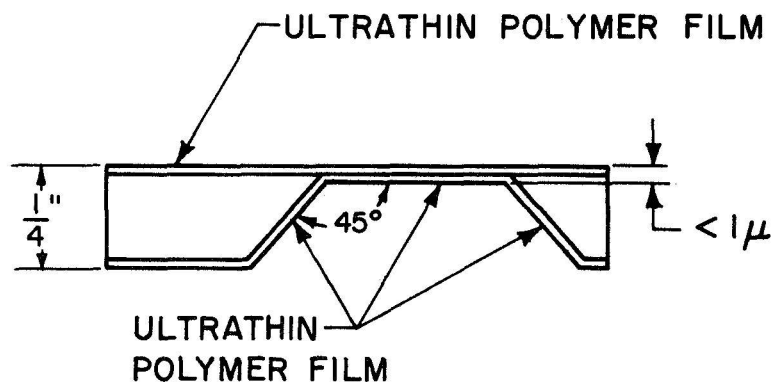
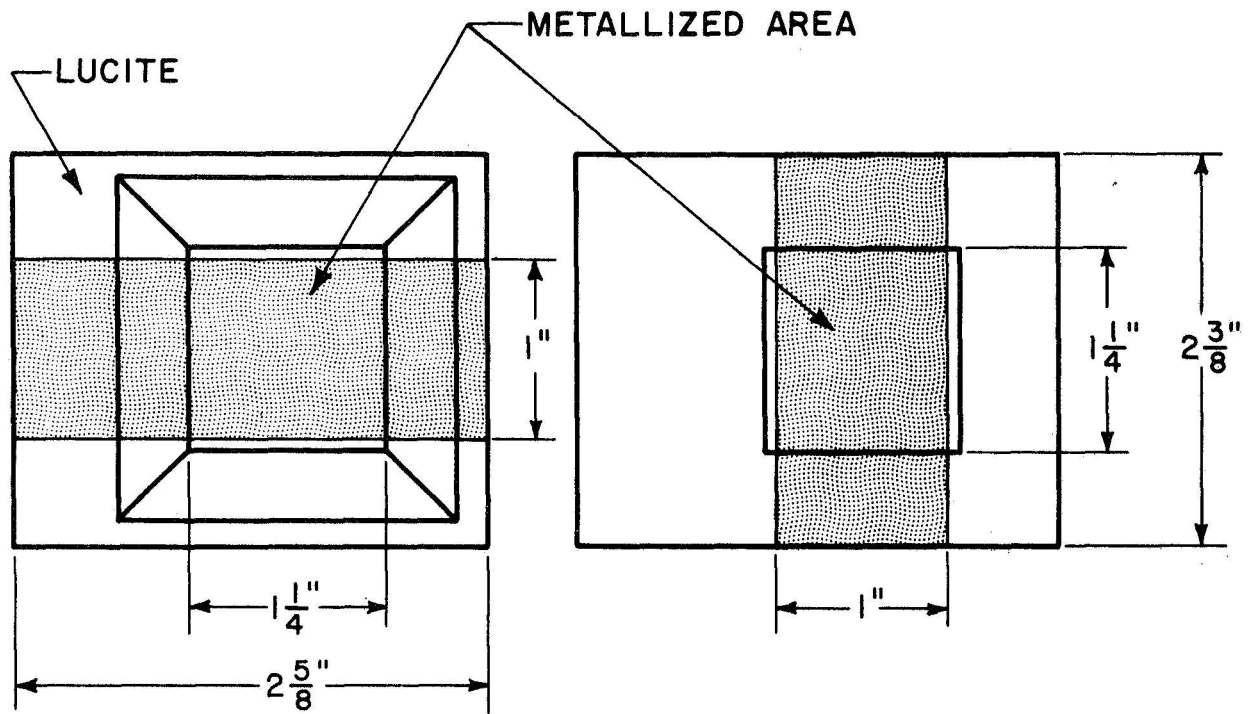


Fig. 1 Sample Holder - Ultrathin Polymer Film Meteoroid Sensor

2. Electrodes

The bevel makes it easy to apply an electrode to the Estane side. A continuous surface is needed to provide a continuous thin film electrode leading from the unsupported part of the film to the part of the film supported by the frame. When the frame bears only one film, there is a crack between the inside of the cutout in the frame and the film. This crack results in an open circuit when one attempts to make contact from the metal deposited onto the frame surface, even though it seems to lead onto the film surface. Using Estane to bear the electrode on the back side obviates this difficulty, since there is then no crack. Electrode 1 refers to electrodes on the bevelled surface. Electrode 2 refers to electrodes on the flat surface. Contact to the metallized areas or the Lucite frame was made by bonding fine copper wire with conductive silver paint.

In the latter stages of the program another method was used to eliminate Estane from the structure and also the possibility of a few pinholes forming during the metallization step. A film was picked up on a large Lucite frame and metallized on one side. It was then floated off this frame onto a water surface, metallized side down, and picked up again on the flat side of a smaller frame which had a metallized surface. The result was a metallized film in intimate contact with a metal layer on a Lucite frame. Succeeding films were then picked up over this one. The last film can be metallized in situ or before being set in place on the frame. Fabrication of capacitors from previously metallized films was generally considered to be the preferred method.

Metal electrodes were applied to the films by vapor deposition. Two metals were tried: a very stable gold electrode which requires a high temperature for vaporization and silver, which vaporizes at a lower temperature and forms better films. The durability of these electrodes are compared in Table II, which reports the surface resistivity at the beginning and at the end of a three month shelf-life test. Gold electrodes do appear to be more

stable than the silver. The resistance of the latter, however, was constant enough to make it questionable whether it would be worth the greater difficulty of applying the more refractory gold electrodes. Certainly the performance in the environmental tests, in which samples were irradiated with ultraviolet light and cycled between room temperature and -40°C was not dependent upon electrode material (Table IV). On the basis of this experience all subsequent samples bore silver electrodes. Many of the samples had, however, been fabricated and tested at this point. Thus testing data in the report is equally distributed between samples with gold and silver electrodes.

Table II
ELECTRODE RESISTANCE AFTER AGING
(Resistance in ohms)

<u>Electrode</u>		<u>Gold</u>	
		<u>1</u>	<u>2</u>
Sample			
1	0 mo. 3 mo.	6 ohms 5	4 ohms 5
2	0 3	5 5	3 3
4	0 3	6 5	10 10
5	0 3	4 3	5 4
26	0 3	40 10	30 10
28	0 3	20 20	45 50
		<u>Silver</u>	
33	0 mo. 3 mo.	50 60	12 40
34	0 3	10 30	45 100

III. MICROMETEOROID SENSOR TESTS

In order to evaluate the micrometeoroid sensors a series of tests were applied. The tests included electrical characteristic measurement, environmental testing, mechanical testing and measuring the effect of impacting simulated micrometeoroid particles. The testing procedure is detailed in the Appendix.

1. Bench, Leakage, Polarity and Shelf-Life Tests

After passing bench tests, samples were tested for leakage when biased at 6, 12 and 18 volts. dc for 24 hours. Two series of samples were submitted to these tests, one at normal room temperature, pressure and humidity and another in a vacuum (10^{-3} - 10^{-4} torr). Polarity tests were then applied. Shelf-life tests were carried out for selected samples. Typical results are reported in Table III. All samples tested in vacuum performed satisfactorily except aluminum oxide, while only the Parylene samples gave acceptable performance in room air. An occasional Lexan or polysulfone film was also satisfactory in air (Samples 26, 28, 25 and 33). It is seen also from Table III that neither the capacitance nor the dielectric loss changed during the aging. The dielectric properties at the beginning and the end of the shelf-life testing period, 3 months, are given in the table. No significant changes from these values occurred at intermediate times in the testing period. Samples, 4, 5, and 28, which were aged under 12 volts bias, also remained unchanged.

Dielectrics from Parylene, Lexan and polysulfone and their various combinations with Estane performed successfully in the materials screening tests and were so submitted to environmental testing.

Table III
MATERIALS SCREENING TESTS

	Maximum current observed during leakage and polarity tests (amp)		Capacitance (Farad x 10 ⁹)		Dissipation Factor	
	Air	Vacuum	0 Month	3 Month	0 Month	3 Month
	1 (PN)		1.0 x 10 ⁻¹³	15	15	0.004
2 (PN)	6 x 10 ⁻⁷		15	15	0.004	0.004
3 (PN)	1.3 x 10 ⁻¹¹					
4 (PN)		1.6 x 10 ⁻¹³	17	17*	0.008	0.009*
5 (PC)	1.9 x 10 ⁻¹³		23	23*	0.03	0.03*
6 (2L+E)	1 x 10 ⁻⁶					
10 (2L+E)	1 x 10 ⁻⁶					
13 (2L+E)		2 x 10 ⁻⁹				
14 (2L+E)	3.4 x 10 ⁻⁶					
17 (2L+2E)		5 x 10 ⁻¹²				
18 (2L+2E)		5 x 10 ⁻¹²				
22 (2L+E)	1 x 10 ⁻⁵					
23 (2L+E)	8 x 10 ⁻⁶					
26 (2L+E)	1 x 10 ⁻⁷		33	34	0.08	0.07
27 (2L+E)	1 x 10 ⁻⁶					
28 (2L+E)	1.4 x 10 ⁻¹²		26	27*	0.10	0.09*
29 (2L+E)	1.3 x 10 ⁻⁶					
30 (2L+E)	5 x 10 ⁻⁶					
25 (L+E+T)	3.8 x 10 ⁻¹¹					
32 (2P+E)	1 x 10 ⁻⁶					
33 (2P+E)	8.5 x 10 ⁻⁶		26	24	0.19	0.18
34 (3P+E)		1.8 x 10 ⁻¹³	45	47	0.08	0.10
36 (3P+E)		1.8 x 10 ⁻³				
39 (Al ₂ O ₃)		1.1 x 10 ⁻⁵				

*Aged under 12 v bias.

2. Environmental Tests

The environmental testing consisted of evaluating the effect of simulated solar irradiance (light from a Xenon source having intensity 0.14 watts/cm^2) and temperature change on the leakage current at 12 volts.

Results of such tests are shown in Table IV for 5 samples. In general, the leakage current remained below 10^{-7} amp through the cooling sequence. On irradiating, however, large increases in leakage current occurred, ranging from a factor of 10 to a factor of 10^5 . In the case of the Lexan-Estane samples, the current level increased even further during the irradiation and then remained at this level after the irradiation was removed. In the case of the two Parylene samples, the current level did not increase significantly above 10^{-7} amps under irradiation. In one case the current level did not increase significantly above 10^{-7} amps under irradiation. In one case the current level decreased to the dark current level on removing the irradiation, suggesting that the effect of radiation was to produce a reversible photocurrent. In order to test whether the increased irradiation-induced current was due to photoemission of electrons from the metal electrode, an irradiation sequence was carried out in a nitrogen atmosphere on (P+E) sample No. 32. A permanent conductivity increase was produced in this case also, indicating that the effect is probably due to photodegradation of the polymer film. The capacitance and dissipation factors were found to have changed markedly for this sample. The capacitance was reduced to 2 picofarads and the dissipation factor became unmeasurable; it may have opened up on handling.

Minor changes in the capacitance and dissipation factors were found for Parylene samples 1 and 2, as a result of the thermal-irradiation treatment. Samples 18, 17, and 30 all showed increase in dissipation factor. Sample 17 showed a marked decrease in capacitance.

Table IV
ENVIRONMENTAL TESTS*

	1 (PN) Au		2 (PN) Au	
	Current (amp)	Dissipation Factor	Current (amp)	Dissipation Factor
	Capacitance (Farad x 10 ⁹)		Capacitance (Farad x 10 ⁹)	
Dark, 25°C	1 x 10 ⁻¹²	0.004	1 x 10 ⁻¹⁰	0.004
Dark, -40°C	1 x 10 ⁻¹⁵		8 x 10 ⁻⁹	
Xe Arc, Start, 25°C	3 x 10 ⁻¹¹		3 x 10 ⁻⁷	
Xe Arc, 2 hr, 30°C	2 x 10 ⁻⁷		2 x 10 ⁻⁷	
Dark, 25°C	3 x 10 ⁻¹²	0.004	2 x 10 ⁻⁷	0.004
				<u>17 (2L+2E) Ag</u>
				38.0
Dark, 25°C			3 x 10 ⁻¹¹	0.06
Dark, -40°C			1 x 10 ⁻¹³	
Dark, 25°C	2 x 10 ⁻⁷	0.13	2 x 10 ⁻¹²	
Xe Arc, Start, 25°C	9.5 x 10 ⁻⁶		3 x 10 ⁻⁹	
Xe Arc, 2 hr, 30°C	9.5 x 10 ⁻⁶		2 x 10 ⁻⁴	
Dark, 25°C	9.5 x 10 ⁻⁶	0.002	2 x 10 ⁻⁴	0.08
		Not measurable		28.0

Table IV (Cont.)

ENVIRONMENTAL TESTS*

	18 (2L+2E) Ag		30 (2L+E) Ag			
	Current (amp)	Capacitance (Farad x 10 ⁹)	Dissipation Factor	Current (amp)	Capacitance (Farad x 10 ⁹)	Dissipation Factor
Dark, 25°C	4×10^{-11}	36.0	0.5	4×10^{-11}	53.0	0.08
Dark, -40°C	$<10^{-12}$			$<10^{-12}$		
Dark, 25°C	1×10^{-12}			1×10^{-11}		
Xe Arc, Start, 25°C	2×10^{-8}			1×10^{-6}		
Xe Arc, 2 hr, 30°C	3×10^{-4}			9×10^{-3}		
Dark, 25°C	3×10^{-4}	37.0	0.13	4×10^{-3}	49.0	0.19

*Currents at 12 v DC.

**Temperature cycling not carried out; sample in nitrogen (1 atmosphere).

In conclusion, polymer films of Lexan or polysulfone in combination with Estane do not appear to withstand the effects of simulated solar irradiation as well as Parylene films, permanent increases in leakage current magnitude being generally obtained. This effect may be due in large part to opacity of the heavier gold electrodes that were on the Parylene samples, but even sample 1 (PN) showed some photoconductivity effect, which was reversible.

A few experiments have been done using thicker metal electrodes on the cast films. In general thicker electrodes are poorly matched to the mechanical properties of the film, which tears and displays greater fragility in handling than with thinner electrodes.

3. Mechanical Tests

The mechanical tests involved acceleration and vibration.

i. Acceleration Tests

In this mechanical testing procedure, meteoroid sensors were subjected to at least 7 g's normal to the capacitor surface and at least 4 g's lateral to the capacitor surface. In the centrifuge arrangement used, 7 g's corresponded to 261 rpm and 4 g's to 150 rpm. In initial tests samples were used which had been only subjected to bench tests. This procedure was adopted in order to test whether gross mechanical damage occurred before proceeding to test samples which have passed leakage, polarity, shelf-life and impact tests.

The effect of acceleration testing at 7 g's normal to the capacitor surface is summarized in Table V for several samples. The effect of 4 g's lateral to the capacitor surface is summarized in Table VI. Table VII summarizes test data on samples subjected to 7 g's normal to capacitor surface with 12 volts dc bias applied during the acceleration. Table VIII summarizes data on samples which had passed leakage, polarity, shelf-life and impact tests.

Table V

ACCELERATION TEST (7 g Normal to Capacitor Surface)

Sample No.	RPM	Capacitance (Farad x 10 ⁹)	Dissipation Factor	Electrode Surface Resistance - 1 (Ω)	Electrode Surface Resistance - 2 (Ω)	Remarks
79 (L+2E)	300	42.0 58.0	2.6 0.33	80 100	10 10	Before After
29 (2L+E)	300	18.2 18.1	0.07 0.073	22 22	22 22	Before After
30 (2L+E)	300	56.0 73.0	0.7 0.12	10 10	100 100	Before After
32 (2P+E)	300	36.4 36.4	0.15 0.15	50 50	50 50	Before After
59 (2T+E)*	300	42.2	0.4	3	20	Before
Sample lost contact (failed)						
52 (Pn+Al ₂ O ₃ +P)	300	--	--	1	2	Before
		--	--	1	2	After
84 (E+P+L)	300	55.7 56.2	0.032 0.032	20 20	3 3	Before After
85 (2E+2P)	300	55.0 55.7	0.04 0.04	5 5	4 4	Before After
86 (3E+T)	300	54.0 55.2	0.092 0.094	10 10	17 17	Before After
75 (2E+2L)	300	65.6 65.5	0.09 0.087	5 5	7 7	Before After

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*Tytil 780

Table VI
ACCELERATION TEST (4 g Lateral to Capacitor Surface)

Sample No.	RPM	Capacitance (Farad x 10 ⁹)	Dissipation Factor	Electrode Surface Resistance		Remarks
				- 1 (Ω)	- 2 (Ω)	
79 (2E+L)	150	58.1	0.328	100	2	Before
				100	3	After
29 (2L+E)	150	18.0	0.071	26	24	Before
				26	24	After
30 (2L+E)	150	54.0	0.723	10	100	Before
				11	100	After
32 (2P+E)	150	36.1	0.15	75	100	Before
				75	100	After
84 (E+P+L)	150	55.8	0.032	20	3	Before
				20	3	After
85 (2E+2P)	150	55.0	0.04	5	4	Before
				5	4	After
86 (3E+T)	150	54.2	0.092	10	17	Before
				10	17	After
80 (P+E)	150	36.7	0.031	2	25	Before
				(Film Torn (failed))		
75 (2E+2L)	150	65.0	0.091	5	7	Before
				5	7	After

Table VII
ACCELERATION TEST (7 g Normal to Capacitor Surface with 12 Volt dc Bias Applied)

Sample No.	RPM	Capacitance (Farad x 10 ⁹)	Dissipation Factor	Electrode Surface Resistance - 1 (Ω)	Electrode Surface Resistance - 2 (Ω)	Remarks
84 (E+P+L)	285	56.5	0.031	20	3	Before
		--	--	20	3	After
Capacitance and dissipation factor could not be measured after test. Bulk resistance was found to be 3 Ω (failed).						
85 (2E+2P)	285	56.1	0.041	3	4	Before
		--	--	--	--	After
Two large holes were found in sample after test (failed).						
90 (P+E)	300	40.0	0.23	10	50	Before
		42.0	0.22	15	50	After
26 (2L+E)	285	33.8	0.072	10	50	Before
		33.8	0.072	10	50	After

Table VIII

ACCELERATION TESTS WITH 12 V dc BIAS

Sample	Capacitance (Farad x 10 ⁹)		Dissipation Factor		Electrode Resistance Before (flat side/ angled side) (ohm)		Electrode Resistance After (flat side/ angled side) (ohm)	
	Before	After	Before	After	Before (flat side/ angled side)	After (flat side/ angled side)	Before (flat side/ angled side)	After (flat side/ angled side)
1 (PN)	15.1	15.1	0.0040	0.0040	5/5	5/4	5/4	5/4
2 (PN)	15.2	15.2	0.0041	0.0040	5/3	5/4	5/4	5/4
4 (PN)	17.4	17.4	0.0090	0.0090	10/10	10/7	10/7	10/7
5 (PC)	22.8	22.3	0.0340	0.0340	3/3	5/3	5/3	5/3
26 (2L+E)	33.9	33.8	0.0720	0.0730	10/10	10/10	10/10	10/10
28 (2L+E)	26.6	26.3	0.0900	0.0890	50/20	50/20	50/20	50/20
34 (3P+E)	47.2	47.2	0.0590	0.0620	30/100	30/100	30/100	30/100
33 (3P+E)	23.5	23.5	0.1800	0.1820	40/60	35/70	35/70	35/70
4 g Lateral to Film Plane								
1 (PN)	15.1	15.1	0.0040	0.0070	5/4	5/5	5/5	5/5
2 (PN)	15.2	15.2	0.0040	0.0080	5/4	5/3	5/3	5/3
4 (PN)	17.4	17.4	0.0090	0.0100	10/7	10/5	10/5	10/5
5 (PC)	22.8	23.0	0.0340	0.0400	5/3	3/4	3/4	3/4
26 (2L+E)	33.8	33.8	0.0720	0.0900	10/10	10/10	10/10	10/10
28 (2L+E)	26.3	26.4	0.0890	0.0900	50/20	50/20	50/20	50/20
34 (2P+E)	47.2	47.1	0.0620	0.0600	30/100	30/100	30/100	30/100
33 (2P+E)	23.5	23.5	0.1820	0.1800	35/70	35/70	35/70	35/70

In general the samples without bias survived the acceleration testing without any major effects. Bench test data were substantially unchanged for the samples listed in Table V. In one case contact to the sample was broken as a result of the acceleration treatment. Similarly, samples subjected to 4 g's lateral to the film surface appeared to be little affected by the treatment. In one case a mechanical tear appeared in the film. A Parylene sample which had developed a short circuit was also subjected to 7 g's without bias (sample 62). No bench test data were taken for the sample in view of the low bulk resistance. No discernible mechanical damage was found, however, after the acceleration testing.

In the case of samples with 12 volt bias two samples out of four developed flaws as a result of normal accelerations. The samples tested had leakage current greater than 10^{-7} amp at 12 volts in all cases, however. None of the films were affected by lateral accelerations.

ii. Vibration Tests

Samples on their mounting frames were attached to a vibration table and shaken in directions parallel to and then normal to the film plane. The results are listed in Table IX. It is seen that with the exception of one case the dielectric properties of the samples were virtually unchanged. Sample No. 34 was damaged after lateral vibration, leaving an open circuit on the angle side of the film (Estane side). The break was not visible to the eye and probably occurred at the angle itself.

Table IX

VIBRATION TESTS OF CAPACITOR FILMS
LATERAL AND NORMAL MOVEMENT

Sample	Bias (volt)	Capacitance (Farad x 10 ⁹)		Dissipation Factor		Electrode Resistance Before (Flat side/ angled side) (ohm)	Electrode Resistance After (Flat side/ angled side) (ohm)
		Before	After	Before	After		
1 (PN)	12	15.1	13.8	0.0070	0.0037	5/5	5/5
2 (PN)	12	15.2	15.4	0.0040	0.1100	5/3	5/4
28 (2L+E)	12	26.4	26.6	0.0900	0.0890	50/20	50/20
34 (3P+E)	12	47.0	open	0.0600	open	30/100	35/00
4 (PN)	0	17.4	17.4	0.0090	0.0090	10/5	10/5
5 (PC)	0	23.0	22.8	0.0400	0.0340	3/4	5/3
26 (2L+E)	0	33.8	36.0	0.0900	0.0730	10/10	10/10
33 (3P+E)	0	23.5	24.0	0.1800	0.2000	35/70	35/70

4. Simulated Micrometeoroid Impact Tests

In operation the capacitors are penetrated by a particle traveling at high velocity, causing a temporary short circuit.

Actually the lowering of the resistance after impact does not amount to a complete short; resistances of the order of 10^8 ohms are typical. To test the performance of the units under these conditions two series of tests were run in a converted Van de Graff accelerator at NASA/MSU. In this unit small iron particles of mass about 10^{-11} grams and velocities of 5-10 km/sec are selected to bombard the particles. It is possible to select the particles singly in this facility, measure their velocity, mass and charge for each impact, using in-line detectors mounted in the acceleration tube. The second series of tests was slightly different from the first in that masses of the particles in the first test were 10^{-11} grams whereas those in the second series of tests were typically 5×10^{-12} grams. As seen in Table X the energies were somewhat lower in the second series of tests also.

The capacitors were connected into the circuit in Figure 2. In general no correlation could be seen between pulse heights and properties of the particles: mass, velocity, momentum, energy or charge. The heights generally correlated only with the bias. The very large pulse in the aluminum oxide sample may indicate dependence on dielectric constant also. In other words the pulse height depends on the energy stored in the charged capacitor. Leaky capacitors gave no pulses, as would be expected, but good capacitors did not always give pulses. Thus the Lexan-Estane samples No. 26, 27, 18, 28 and 110 all gave no pulses, although No. 27, 26 and 18 had currents lower than 10^{-8} amp at the bias voltage. The same is true of the polysulfone-Estane sample No. 34, Tyril-Estane-Lexan No. 25 and polysulfone-Estane No. 37.

Table X
PARTICLE-IMPACT TESTS

Sample	Particle*** Energy (10^{-2} erg)	Pulse Height (mv)	Bias (v)	Current at 18 v (amp)
1 (PN) Au*	98	0.5	6	3×10^{-12}
	117	0.7	6	
	111	1.5	12	
	98	>2.0	12	
	117	2.2	18	
2 (PN) Au*	191	2.3	12	2×10^{-7}
	105	0.3	12	
	108	0.4	12	
	105	0.6	6	
	105	0.9	6	
	111	0.8	18	
	114	1.2	18	
2 (PN) Au**	64	<0.4	18	6×10^{-7}
	107	<0.4	30	
4 (PN) Au*	290	0.14	12	4×10^{-12}
	148	0.08	12	
	136	0.12	18	
	~100	<0.01	18	
	130	0.03	3	
	123	0.36	6	
	4 (PN) Au**	174	0.4	
115		0.3	12	
94		0.4	18	
126		0.5	18	
131		0.6	18	
5 (PC) Au*	105	1.1	6	3×10^{-10}
	130	2.0	6	
	102	2.3	18	
	123	2.2	18	
5 (PC) Au**	120	<0.6	12	3×10^{-9}
	72	1.8	12	
	51	2.0	12	
	86	2.0	18	
	70	2.2	18	

Table X (Cont.)

PARTICLE-IMPACT TESTS

Sample	Particle*** Energy (10^{-2} erg)	Pulse Height (mv)	Bias (v)	Current at 18 v (amp)
13 (2L+E) Ag*	~100	<0.05	6	4×10^{-8}
	~100	<0.05	12	
	~100	<0.05	18	
	98	<0.05	18	
	~100	<0.05	24	
	~100	<0.05	30	
	~100	<0.05	30	
17 (2L+2E) Ag*	~100	<0.05	6	3×10^{-10}
	~100	<0.05	12	
	~100	<0.02	18	
	~100	<0.02	18	
	117	<0.04	29	
	~100	<0.02	29	
18 (2L+2E) Ag*	~100	<0.01	6-12	2×10^{-10}
25 (L+E+T767)*	~100	<0.05	6	2×10^{-10}
	~100	<0.05	12	
	~100	<0.05	18	
26 (2L+E) Au*	~100	0	6	1×10^{-9}
	~100	0	6	
	~100	0	12	
	~100	0	12	
	~100	0	18	
	~100	0	18	
27 (2L+E) Au*	~100	0	6	4×10^{-12}
		0	6	
		0	12	
		0	18	
28 (2L+E) Au*	~100	<0.01	6-18	1×10^{-8}
33 (P+E)**	62	<0.1	12	8×10^{-6}
	107	<0.1	18	

Table X (Concl.)

PARTICLE-IMPACT TESTS

Sample	Particle*** Energy (10^{-2} erg)	Pulse Height (mv)	Bias (v)	Current at 18 v (amp)
34 (3P+E) Ag*	~100	0	6	3×10^{-10}
		0	12	
		0	18	
		0	18	
		0	18	
		0	28	
37 (3P+E) Ag*	~100	<0.02	6-40	5×10^{-11}
51 (P+Al ₂ O ₃ +T) Ag**	83	2.0	18	1×10^{-7}
100 (2L+E)**	158	<0.1	6	2×10^{-5}
	142	0.3	6	
	80	0.6	6	
	115	0.8	18	
	75	1.0	18	
102 (2L+E) Ag**	75	1.2	12	1×10^{-11}
	78	1.4	12	
	64	1.4	18	
	118	0.6	18	
	128	0.6	18	
	94	0.6	18	
107 (2L+EL)**	83	<0.1	18	1×10^{-4}
110 (2L+E) Ag**	169	<0.1	18	8×10^{-5}
		<0.1	12	

*First test series, March 27, 1969.

**Second test series, August 8, 1969.

***Particle size was limited to a pre-set range of approximately the energy indicated.

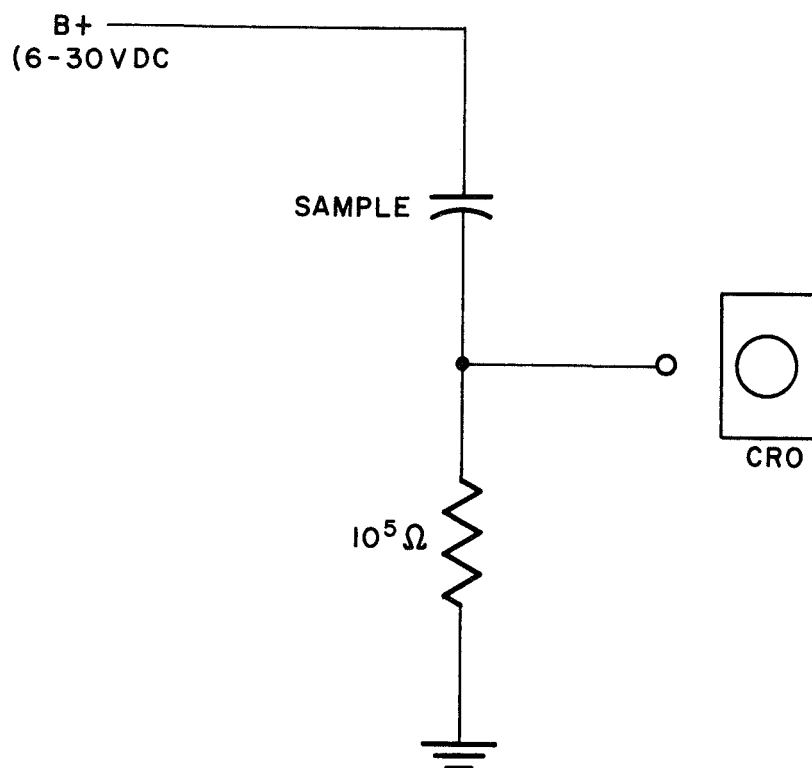


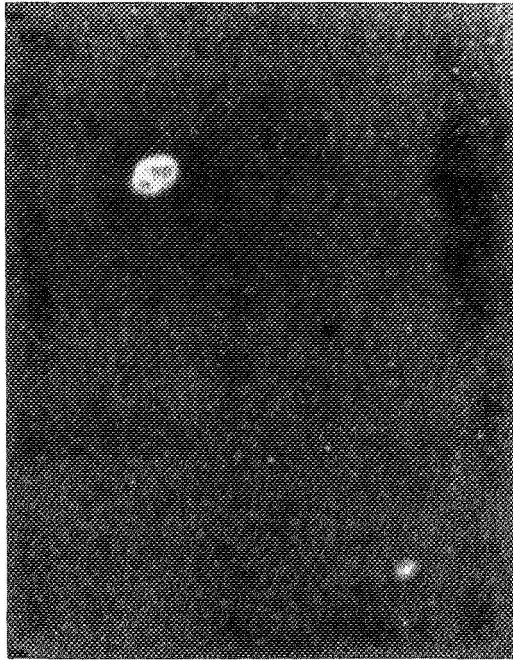
Fig. 2. Circuit for Impact Testing

An occasional "temperamental" sample was found. Sample 13, a Lexan-Estane sample, usually gave no pulses. In seven tries it gave one small one. Sample No. 4 usually gave pulses, but occasionally did not. Sample No. 17 gave a small pulse when biased at 30 volts, but only once. Sample No. 5 occasionally gave no pulse but usually gave strong ones. These observations are all recorded in Table X.

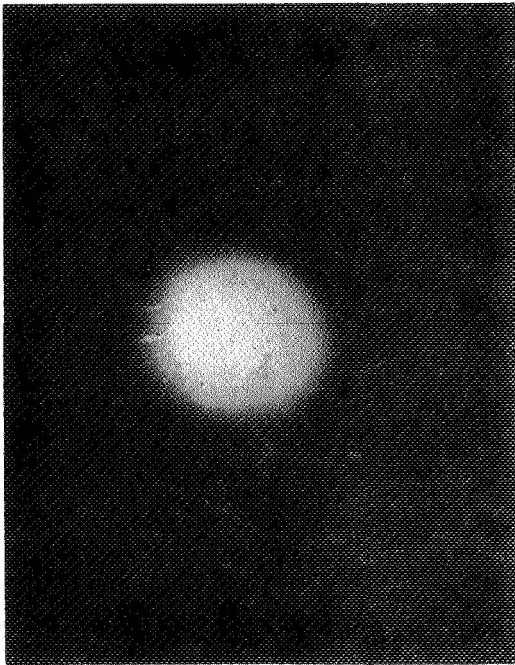
5. Film Defects

During latter phase of the project many films were made of various compositions, all based on the Estane composite principle. These included Estane-aluminum oxide, Estane-polysulfone, Estane-aluminum oxide-Estane, Estane-Lexan-Estane, etc. Although the procedures for making capacitors were the same as before, very few of the units were usable, most having leakage resistances less than 10^{-7} amps at 18 volt bias. In fact only 3 out of about 50 had leakage current between 10^{-7} and 10^{-9} amps. In order to elucidate any effects due to film sub-structure, certain of these films were examined by optical and electron transmission microscopy without shadowing. The results were unexpected. The surface of the film was much different from those of the cast polyphenyleneoxide film, for example, which we had examined in this way previously, or a Parylene film, both of which show a relatively smooth surface, sometimes with low parallel ridges (Kronick, P. L., Serafin, F. L., and Jesch, K., "Organic Films for Discrete Elements for R-C Networks". AD-824-179). A micrograph of an Estane film is shown in Figure 3A. The surface has many small craters with occasionally a large one giving it a rough texture. In addition thin spots are noted, with round contours resembling broken or collapsed bubbles (Figure 3B). Occasionally tiny angular sub-micron particles are seen in the middle of each, resembling inorganic crystals sometimes found in dust (Figure 3C). These could be elsewhere in the film but would not be visible because of the rough texture.

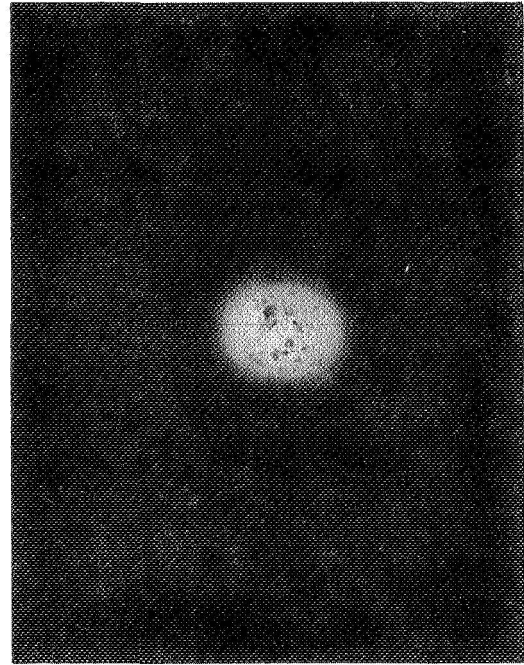
These defects are much more frequent than previously found in thin plastic capacitors prepared using similar techniques. The craters, if they were metallized, could form electric field-concentrating spikes, which might be responsible for the high leakage current. Precautions aimed at reducing sub-micron particulate matter, such as carefully filtering the polymer solutions, were explored, but the micrography was not carried forward. The texture of the film may also be modified by using a different solvent.



(A) 2000X



(B) 10,000X



(C) 10,000X

Fig. 3. Estane Film Micrographs

IV. DISCUSSION

Of the materials evaluated for use in particle sensor capacitors, i.e., Parylene, aluminum oxide, polysulfone, Lexan, Tyril, Estane, Acryloid, and Penton, only one, Acryloid, was found to be completely unusable. A large number of capacitors were made from Lexan and Estane in combination, but polysulfone was also found to be usable. Toward the end of the program it was found easier to make pinhole-free films from polysulfone than from Lexan.

All of the materials stood up well to the mechanical and electrical stresses encountered in the tests. In the environmental tests, however, large permanent changes in resistance arose after two hours illumination using xenon lamps when Lexan-Estane and polysulfone-Estane composites were exposed. Parylene, which showed also large changes during the exposure, returned to its highly resistive state after the light was extinguished (Table IV). One of the Parylene samples became leaky after low-temperature cycling. The resistivity of even this one was higher than that of the cast film samples when exposed to the light. The superior performance of Parylene may be, in part, due to the opacity of the heavy gold electrodes on the Parylene samples. The observation of photoconductivity indicates, however, that significant amounts of radiation are falling on the polymer substrate.

Some samples of Lexan, of polysulfone, and of Parylene passed all the other tests, including the impact demonstration. However, all the samples of Parylene were satisfactory, while very few of the others were. Only the Parylene was satisfactory in the environmental tests. The reasons for the higher reliability of Parylene probably lies in the greater precision of control in the Parylene process over that in the film casting process for the other materials. Defects in the Estane, as mentioned in an earlier section on film defects, may also play a role. Thus it is not felt that the test results in this report reflect on the material properties of the

dielectrics tested, but only the present feasibility of making sensitive units using fabrication processes at hand. Even the photosensitivity of the Lexan-Estane composites could be due to impurities in the polymers themselves, which might act as photosensitizers for degradation. The polymers used in this project were purified by reprecipitation, but that would not attain the high degree of purity needed to avoid photosensitization. The dielectrics performance in the test sequence is summarized in Table XI.

Table XI
SUMMARY OF PERFORMANCE OF DIELECTRICS

<u>Test*</u> →	<u>Bench</u>	<u>Leakage</u>	<u>Polarity</u>	<u>Environmental</u>	<u>Mechanical</u>	<u>Impact</u>	<u>Shelf-Life</u>
Material**							
PN	✓	✓	✓	✓	✓	✓	✓
PC	✓	✓	✓	0	✓	✓	✓
L+E	✓	✓	✓	X	✓	✓	✓
P+E	✓	✓	✓	X	✓	X	✓
T+L+E	✓	✓	✓	0	✓	X	0
T+E	✓	X	0	0	0	0	0
Al ₂ O ₃	✓	X	0	0	0	0	0
Al ₂ O ₃ + Pn + T	✓	X	0	0	0	✓	0
Al ₂ O ₃ + E	X	0	0	0	0	0	0
Pn + L + E	X	0	0	0	0	0	0
A + E	X	0	0	0	0	0	0

✓ = at least one sample passed test.

X = all samples failed test.

0 = not tested.

* = see Appendix.

** = see Table I.

V. CONCLUSIONS

Satisfactory meteoroid sensors can be made from films of Parylene bearing opaque gold electrodes. Other materials did not give reliable sensors as frequently, although the occasional successes achieved with Lexan and polysulfone indicate that they might be developed in the future as suitable thin film dielectrics.

VI. DELIVERABLE ITEMS

In accordance with the terms of the contract ten meteoroid sensors fabricated from the material which performed most satisfactorily in the test sequence are to be delivered. Ten Parylene samples, five of Parylene C and five of Parylene N, fabricated according to the specifications used for the samples described in the tests, have been obtained from Union Carbide for this requirement.



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APPENDIX
TEST PROCEDURES

1. BENCH TESTS

Bench tests were carried out immediately following the electrode metallizing procedure and consisted of measuring the electrode surface resistance, for both electrodes, the capacitance at 10 KHz and the capacitor dissipation factor at 10 KHz. The measurements were made with the sample in air. The sample was considered to have passed the test if the electrode surface resistance was less than 100 ohm and stable capacitance data could be obtained in the range $10\text{--}50 \times 10^{-9}$ Farad.

2. LEAKAGE TESTS

The leakage test consisted of applying successively a 6, 12 and 18 volt D.C. bias and monitoring the D.C. leakage current continuously for the first five minutes that bias was applied and again after 1, 4 and 24 hours of continuous biasing. Leakage tests were carried out at normal room temperature, pressure and humidity and in vacuum (10^{-3} – 10^{-4} torr).

3. POLARITY TESTS

The polarity test consisted of monitoring the leakage current at +12 volts bias for 5 minutes, reversing the bias voltage to -12 volts and monitoring the leakage current for a further five minutes. The reverse polarity was monitored for 24 hours and the leakage current measured after this time. The original bias polarity was then reapplied and the leakage current measured. Samples were considered to have passed the leakage and polarity tests if the current did not exceed 1×10^{-7} amp during the test sequence. Samples which had leakage currents up to 1×10^{-6} amp were considered marginal.

4. SHELF-LIFE TESTS

The shelf-life test consisted of performing bench tests each week on eight samples, three biased at 12 volts continuously and five

unbiased, for a period of three months. The samples were maintained at normal room temperature, pressure and humidity during the three month period. Samples were considered to have passed the shelf-life test if no significant change in bench test data occurred during this time.

5. SUBMISSION OF SAMPLES FOR IMPACT, MECHANICAL AND ENVIRONMENTAL TESTING

The majority of samples subjected to Impact, Mechanical and Environmental Testing had passed test procedures 1, 2 and 3. In some cases marginal samples, i.e., with leakage current greater than 1×10^{-7} amp were tested. This was done in the case of mechanical testing, for example, where we wished to ascertain whether gross physical damage would occur, before testing valuable samples which had passed test procedures 1, 2 and 3. Some marginal samples were also subjected to impact testing.

6. IMPACT TESTS

Impact tests were carried out at the Van der Graaf microparticle facility at NASA, MSC Houston. The test chamber was cubical with an eighteen inch evacuable interior diameter with provision for electrical feedthroughs and target translation in two directions. Four samples were mounted and rotated into the particle path and impacted one at a time after the chamber had been evacuated. The particles had energies ranging from 0.5 to 1.7 erg. The particle penetration signals were measured across a 100 K ohm resistor. Samples were considered to have passed the impact test if one or more signals were obtained in a series of several impacts.

7. ENVIRONMENTAL TESTS

The environmental test consisted of evacuating the sample to 10^{-4} - 10^{-5} torr, cooling to -40°C , and maintaining the sample at this temperature for at least two hours. After warming up to room temperature the sample

was irradiated with a Xenon light source through quartz optics for a period of two hours at an intensity of 0.14 watts/cm^2 . A circular region of the film approximately 1.5 in diameter was irradiated. The leakage current at 12 volts bias was monitored during the temperature cycling, and irradiation sequence and bench tests were applied before and after the environmental testing. The temperature was monitored by means of a thermocouple bonded with conductive silver paint on the metallized Lucite surface on the periphery of the unsupported capacitor film. Samples were considered to have passed the test if the leakage current did not exceed 1×10^{-7} amp at any point during the test sequence.

8. MECHANICAL TESTS

Acceleration Tests

Acceleration tests consisted of subjecting the samples to at least 7 g's normal to the capacitor surface and at least 4 g's lateral to the capacitor surface in normal ambient conditions in a centrifuge. Some samples were biased at 12 volts and some unbiased. Bench tests were carried out before and after the treatment. Samples were mounted in a centrifuge arrangement in the required configuration and the required revolutions per minute established. The rpm was verified by means of an electronic stroboscope and a tachometer. The sample was subjected to the acceleration for 1 minute and then removed. Samples were considered to have passed the test if no significant changes occurred in the bench test data.

Vibration Tests

Samples were attached to a vibration table and subjected to sinusoidal vibration in directions parallel to and then normal to the film plane in normal ambient conditions. The frequency of the vibration table was swept from 5 to 2000 Hz with dynamic force amplitudes not exceeding 1 g. Samples were biased at 12 volts or unbiased during the vibration. Bench tests were carried out before and after the treatment. Samples were considered to have passed the test if no significant changes occurred in the bench test data.