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LEAM SENSOR FRONT FILM DEVELOPMENT REPORT

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Introduction

The principle of operation of the Lunar Ejecta and Meteorites (LEAM) experiment sensor is based on the fact that when a hypervelocity particle impinges upon a media, some or all of the particle's energy is lost by producing ionization of the particle or media.

The media used on LEAM is a very thin metallic film. This film is supported, for practical reasons, by a thin film of Parylene C⁽¹⁾. Parylene C has been found in previous experiments⁽²⁾ to be the best available material for this application.

The LEAM experiment is a development, for lunar applications, of a highly successful satellite experiment which has flown on Pioneer satellites 8 and 9. The LEAM sensor is identical to the one on the Pioneer experiment except in application. LEAM will be subjected to the lunar environment of approximately fourteen days of sun and fourteen days of lunar night. The Pioneer satellite was spin stabilized, giving uniform solar illumination, thus the thermal problems of the two experiments are entirely different.

A development program was defined to determine the characteristics required by the front film to survive the new environment and also to benefit from any recent developments in the state of the art of Parylene deposition to produce a thinner film which could detect smaller or lower energy particles.

(1) A patented product of Union Carbide Corporation.

(2) Meteoroid Sensor Material Development Report - Franklin Institute Research Labs. (1969)



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2.0 Program Description

The film development program was conducted in conjunction with Union Carbide Corporation, Chemicals and Plastics Division, Bound Brook, New Jersey. The program plan was defined in the Statement of Work to Union Carbide Corporation, SW-A-120.

2.1 Objectives

The objectives of the program, as defined by the Statement of Work were:

- a) To develop a thin Parylene C film laminate and structural attachment which will withstand the prelaunch, test, storage and translunar flight environments, and perform normally for two years under lunar environmental conditions.
- b) To develop a thin Parylene C film laminate and structural attachment which will yield a maximum supply of ions and electrons when impacted by hypervelocity microparticles.
- c) To develop a thin Parylene C film laminate and structural and attachment which will satisfy (a) and (b) and result in the smallest mass cross section; thus minimizing the microparticle kinetic energy threshold for penetration of the film.

2.2 Development Approach

Twenty-one different film laminates, mounted on various sizes of frames, were procured. These film samples were used to evaluate the optical, mechanical, thermal and scientific (electrical) properties of the film laminates. Table I defines the structure of the film samples used.

In addition Union Carbide evaluated the possibility of using an alkali metal salt as the ionizing medium on the film.



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Gold was the first metal used as the ionization material on all samples except those with the alkali metal salt deposit, because it was considered to have the best ionization properties of all the metals that could be easily vacuum deposited. As the program progressed various other deposits and film samples were used. A considerable amount of work was done using silver as a deposit material due to its good thermal/optical properties. In addition, due to its good optical and mechanical properties, samples of Silicon Oxide over Aluminum were tested. Flight configuration samples, on 1 x 4 inch frames were mounted on beryllium copper grids, 0.006 inches thick and with approximately 97% optical transparency.

2.3 Test Facilities

2.3.1 Optical Properties Measurements

The optical properties were all measured at Goddard Space Flight Center using equipment under the management of Dr. J. Heaney. Confirmatory measurements on a few samples were made at Marshall Space Flight Center and Bendix.

2.3.2 Scientific Electrical Measurements

Ionization properties were determined by the Principal Investigator using his particle accelerator at Goddard Space Flight Center.

2.3.3 Mechanical Measurements

The vibration tests were performed on a small vibrator at the Bendix facility. The test set up is described in ATM 1011, LEAM Film Development Vibration Test Report.

2.3.4 Thermal Calorimeter Measurements

The calorimeter tests were performed in the 4 x 8 foot vacuum chamber at the Bendix facility, using a calorimeter designed and fabricated at Bendix.

The calorimeter, and test configuration are described in ATM 995, LEAM Film Development Test Report.

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2.3.5 Thermal Shock Tests

Some samples of both the silver and silicon oxide coated aluminum films were subjected to thermal shock and thermal soak. These tests were conducted by Dr. Mark Spivack of Union Carbide using a methanol and dry ice bath, a liquid nitrogen bath and a vacuum oven.

3.0 Evaluation Results

3.1 Evaluation of Alkali Metal Salts

Union Carbide Corporation screened the salts of the alkali metals (lithium, sodium, potassium, rubidium and cesium) by reviewing available technical literature. Sample depositions of the recommended salt were made, with and without gold in combination.

The objectives of the salt evaluation were:

- a) They shall be capable of controllable deposition onto Parylene C. That is, repeatable deposits of thickness ranging between 100 Angstrom units to 1000 Angstrom units.
- b) The alkali metal, with or without gold in combination shall have high electrical conductivity in the plane of the film. The resistance of a 1" x 4" film strip shall not exceed 10 ohms along the 4" length.
- c) The compound deposit characteristics (physical, chemical and optical) should be completely stable when subjected to the lunar environments of pressure, 10^{-11} torr, solar radiation and temperature range of -300°F to $+250^{\circ}\text{F}$, for periods of two years or longer. In addition, the deposit should exhibit no degradation of characteristics when subjected to storage, test, prelaunch or launch environments.

Sodium fluoride was recommended as being worthy of evaluation as an ionization material in the LEAM detector. The Deposition Development Report (Union Carbide, October 1970) describes the salt evaluation.



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3.2 Optical Sample Measurements

The samples were measured for values of reflectance, solar absorptance, transmittance and infrared emittance.

The gold samples exhibited large ratios of solar absorptance to infrared emittance (α/ϵ) and were thus rejected for use on the LEAM.

The samples of silver gave promising values for α/ϵ but were later rejected for other reasons. The variables which affect the value of α/ϵ were found to be many and were difficult to control to give reliable flight films. The purity of the silver, the deposition rate, the parylene deposition method and silver tarnishing were all found to contribute to deficiencies in the optical properties. In addition, thermal cycling of the silver films produced stress cracks in the film. (See ATM 1019, LEAM DVT Thermal Test Report).

The final samples tested used a silicon oxide coat over a deposition of aluminum. This combination gives adequate values for α/ϵ and withstands the thermal cycling. The deposition rates are not so critical and there are no tarnishing problems associated with aluminum and silicon oxide.

The results of the optical measurements are tabulated in Table II.

3.3 Mechanical Properties Evaluation

The purpose of the tests was to determine the mechanical properties of the film and verify the necessity for a support grid.

The vibration tests indicated that the beryllium copper grid is required to support the film during the launch, and lunar descent phase of the lunar landing mission. A full description of the tests is given in ATM 1011.

3.4 Thermal Properties Evaluation

Calorimeter tests were performed in a vacuum chamber to provide data that would allow computations to be made of the film temperature and verify the measured optical properties of the film. The effects of using a beryllium copper support grid were also to be evaluated.

The tests confirmed, within experimental limits of error, the optical properties of the film determined by optical methods. The tests also showed that the support grid is a desirable element as it reduces the film temperatures considerably when compared to an unsupported film, which cannot be used with incident radiation above 0.2 solar constants. The full test and results are described in ATM 995.

3.5 Ionization Characteristics

Samples of the film laminates used in the other test phases were used by the Principal Investigator to evaluate their capability to produce ionized plasma when impacted by hyper-velocity particles. The samples were tested using a 2 MeV electrostatic accelerator.

All the samples tested produced essentially the same amounts of ionized plasma as the original Pioneer films. Thus, all films are considered suitable for use on LEAM from the aspect of ionization.

3.6 Thermal Shock Tests

Samples of both silver and silicon oxide coated aluminum were subjected to hot and cold temperature soak and thermal shock.

3.6.1 High Temperature Test

3.6.1.1 The silver film was left overnight in a vacuum oven, at a temperature of 75°C and exhibited no changes in physical properties.



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3.6.1.2 The silicon oxide coated aluminum film was left in the oven at 160°C. The lexan frame warped slightly but there were no signs of change in the film.

3.6.2 Low Temperature Tests

3.6.2.1 The silver film was first placed, in a test tube, into a methanol and dry ice bath, which is at approximately -78°C, for thirty minutes, after which 3 of the 1/8 x 1/8 inch squares had crazed or torn, on one of the 1 x 1 inch squares. The damage occurred, one at each remote corner and one in the center of the square.

The film was then placed into a liquid nitrogen bath for fifteen minutes, when 50 squares became broken, mainly around the edges of the frame. The silver film became visibly cloudy and taut.

3.6.2.2 The silicon oxide coated aluminum film showed no signs of change in either the dry ice or liquid nitrogen baths.

4.0 Conclusions

The conclusions made from the evaluations described above are that:

- a) The deposit should be 3000 to 4000 Angstrom units thickness of Silicon oxide over 600 to 700 Angstrom units of Aluminum on a 1500 to 2000 Angstrom units thick Parylene substrate. The laminate is to be mounted on a 97% transparent, beryllium copper grid. The aluminum and Silicon oxide are to be vacuum deposited while the Parylene C and grid are free standing on frames. This laminate has the optical and thermal properties required to endure the lunar environment and to allow thermal control of the experiment electronics.
- b) A support grid is required to provide mechanical strength and to provide thermal conductivity from the film to the film support frame.



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- c) The deposition provides adequate ionized plasma upon impact by a hypervelocity particle.
- d) Free standing deposition resulting in slightly wrinkled film is desirable for low temperature survival.

5.0

Bibliography

- a) Meteorite Sensor Material Development Report; Franklin Institute Research Laboratories. (1969)
- b) Statement of Work for Front Film Sensor (LEAM) experiment; Bendix, SW-A-120.
- c) Bendix, ATM 1011, LEAM Film Development Vibration Test Report.
- d) Bendix, ATM 995, LEAM Film Develop Test Report.
- e) Bendix, ATM 1019, LEAM DVT Thermal Test Report.
- f) Union Carbide, Deposition Development Report, (October 1970)



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TABLE I

Film No.	Quantity	Substrate Thickness (Angstroms)	Deposit Material	Thickness (Angstroms)	Outer Layer		Utilization
					Material	Thickness	
1	4 ea.	1000	Gold	400	Parylene	1000	Eng. Evaluation
2	4 ea.	1000	Gold	1000	Parylene	1000	Eng. Evaluation
3	4 ea.	1000	Gold	400 (1)	Parylene	None	Vibration
4	4 ea.	1000	Gold	1000 (1)	Parylene	None	Calorimeter
5	4 ea.	1000	Gold	400	Parylene	None	Vibration
6	4 ea.	1000	Gold	1000	Parylene	None	Eng. Evaluation
7	4 ea.	2000	Gold	400	Parylene	1000	Vibration
8	4 ea.	2000	Gold	1000	Parylene	1000	Eng. Evaluation
9	4 ea.	1000	Gold	400	Parylene	None	Eng. Evaluation
9A	2 ea.	1000 (2)	Gold	400	Parylene	None	Optical
10	4 ea.	1000	Gold	1000	Parylene	None	Calorimeter
10A	2 ea.	1000 (2)	Gold	500 (3)	Parylene	None	Optical
10(b)	1 ea.	2000	Silver	1000	Parylene	less than 500	Ionization Tests
11(b)	1 ea.	2000 (2)	Silver	1000	Parylene	less than 500	Optical
11(c)	1 ea.	2000 (2)	Silver	1000	None		Optical
11(d)	1 ea.	2000 (2)	Silver	800	None		Optical
11A	2 ea.	1000 (2)	Gold	200 (3)	None		Optical
12(a)	1 ea.	2000 (2)	Silver	1200	None		Optical
12(b)	1 ea.	2000 (2)	Silver	1000 (4)	None		Optical
12 (c)	1 ea.	2000 (2)	Aluminum	700-1000(3)	Silicon Oxide	2000-4000	Optical
12(d)	1 ea.	2000(2)	Silver	1000	None		Optical
12A	2 ea.	2000(2)	Gold	500(3)	None		Optical
13	4 ea.	1000	Gold	400	Parylene	1000	Eng. Evaluation
13A	2 ea.	1000 (2)	Sodium Flouride	1400	None		Optical
14	4 ea.	1000	Gold	1000	Parylene	1000	Calorimeter
14A	2 ea.	1000(2)	None		None		Optical
15	4 ea.	2000	Silver	1000(1)	Parylene	200	DVT I
15A	2 ea.	2000 (2)	Gold	400	Aluminum	200-500	Optical
16(a)	2 ea.	2000(2)	Aluminum	1000	None		Optical
16(b)	2 ea.	2000	Aluminum	1000(1)	None		Ionization Test
16A	2 ea.	2000(2)	Gold	500(5)	None		Optical
17	2 ea.	1000	Gold	400	None		Ionization Test
18	2 ea.	1000	Gold	1000	None		Ionization Test
19	2 ea.	2000(2)	Silver	1000	Parylene	500	Optical
20	2 ea.	2000(2)	Silver	1000(1)	Parylene	500	Ionization Test



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TABLE I

<u>Film No.</u>	<u>Quantity</u>	<u>Substrate Thickness (Angstroms)</u>	<u>Deposit Material</u>	<u>Thickness (Angstroms)</u>	<u>Outer Layer</u>		<u>Utilization</u>
					<u>Material</u>	<u>Thickness</u>	
21-1	1 ea.	Thermocouple	Deposition				Calorimeter
21-2	1 ea.	2000 (2)	Silver	1000	Parylene	less than 200	Optical
21-3, 4, 5, 6	1 ea.	2000	Silver	1000 (1)	Parylene	less than 200	DVT I
21-7	1 ea.	2000 (2)	Silver	1000	Parylene	less than 200	Optical Sample for 15 and 21-3, 4, 5, 6
21-8	1 ea.	1500-2000	Aluminum	600-800(1)	Silicon Oxide	3000-4000	DVT II and 1 for Ionization Test
21-17, 18	1 ea.	1500-2000 (2)	Aluminum	600-800(1)	Silicon Oxide	3000-4000	Optical for 21-8 thru 16

Notes: (1) Copper support grids 0.006 inches thick and with approximately 99% optical transparency were bonded to the 4" x 1" film laminate, and electrically connected to the metallic deposit.

(2) Two sizes of frames were used for these films. One was 1" O.D. and the other 1.3" I.D. These frames did not have electrical contact between the film and the frames. The deposition side of any asymmetric film was mounted in contact with the frame. All other films were on 1" x 4" frames.

(3) Deposit applied to one side only.

(4) Deposited at high deposition rate (40 $\overset{\circ}{\text{A}}$ /second)

(5) Substrate surface was given a matted finish.



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TABLE II
LEAM FILM
OPTICAL TEST RESULTS

FILM	α	E	α/E	SECTION OF FILM	COMMENTS	α	E	α/E
Measured from this side				Thickness Dimensions and Materials		Measured from this side		
9A	Est. .3	.026	11.5	2 9.38 2 Au, P, Au	Au at 4 Å/Sec.			
10A	.281	.02	14.0	5 9.38 Au, P	Au at 4 Å/Sec.			
11A	.339	.04	8.5	2 9.38 Au, P	Au at 4 Å/Sec.			
11b	.12			6 17 20 P, Ag, P	Ag at 17 Å/Sec.	.12		
11c	.09			17 20 Ag, P	Ag at 17 Å/Sec.	.125		
11d				15.15 20 Ag, P	Ag at 17 Å/Sec.	.125		
12A	.297	.02	14.8	2 9.38 Au, P	Au at 4 Å/Sec.			
12A		.08			MSFC Measure- ment			
12a				24.5 20 Ag, P	Ag at 17 Å/Sec.	.125		
12b	.06	.02	3	17 20 Ag, P	Ag at ≈ 40 Å/Sec.	.09		
12c	.25	.45	.55	180 7 21.35 SiO, Al, P				
12c	.19	.28			MSFC Measure- ment			
13A	.017	.008	2.1	7 9.38 7 NaF, P, NaF	NaF at 5.2 Å/Sec.			
14A	.013	.005	2.6	9.38 P				
15A	.363	.033	11.0	2 2 22.3 2 Al, Au, P, Au				
16A	.233	.023	10.1	5 22.3 Au, P				



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TABLE II
LEAM FILM
OPTICAL TEST RESULTS

FILM	L	E	L/E	SECTION OF FILM	COMMENTS	L	E	L/E
Measured from this side			Thickness Dimensions and Materials			Measured from this side		
16(a)	.13	.03	4.3	5 20 5 Al, P, Al	Wrinkled			
16(a)	.12				MSFC Measurement			
19	.59	.15	3.9	5 5 20 5 P, Ag, P, Ag	Impure Ag			
19	.113	.02	5.7	5 5 20 5 P, Ag, P, Ag	Pure Ag	.12	.02	6.0
21-2A				1.3 10 21.35 P, Ag, P				
21-2B	.085	.02	4.25	100 10 1.55 P, Ag, P		.114		
21-2B	.09				MSFC Measurement			
21-7	.128	.025	5.1 6.4	1.75 10 20 P, Ag, P	For DVT-I AG at > 25 Å/Sec.			
21-17- 18	.4	.085 .10	4 4.7	31 16-7 18 SiO, Al, P	Some DVT-II SiO=10 Å/Sec 6.5 Å/Sec			
21-18	.35	.042			Bendix Measurement			

Notes: (1) Thickness Dimensions Are In 100 Å Units
(2) Measurement apparatus at MSFC failed before completion of emittance readings.

(3) Symbols Ag - Silver NaF - Sodium Fluoride
 Al - Aluminum P - Parylene C
 Au - Gold SiO - Silicon Oxide