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RAI RESEARCH Corporation

DEVELOPMENT OF A PROTOTYPE PLASTIC SPACE ERECTABLE SATELLITE

Contract No. NAS5-3923

with the

National Aeronautics and Space Administration Goddard Space Flight Center Greenbelt, Maryland

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#### 1.0 INTRODUCTION

During this reporting period tests were performed on Marlex 6002, a high density polyethylene film, manufactured by the Phillips Joanna Chemical Company. The evaluation of test data indicated that the film was not an acceptable material and it was therefore eliminated from further consideration. A new standard polyethylene film made by Sea Space (1 mil, density 0.935) was selected for consideration. Samples of the Sea Space film have now been received and evaluated. Tests indicate that the film has acceptable properties. Samples of Sea Space standard polyethylene film were irradiated and heat-treated under different ambient conditions (1.e., nitrogen, air and oxygen) to determine the effects of atmosphere on the processed film. The results indicate that the effect of atmosphere has little influence on the mechanical properties of the processed film.

Companies have been chosen to do some of the processing on the deliverable items. Sea Space Systems, Inc., has been chosen to extrude the thin biaxially oriented polyethylene film to be used to construct a cap section then heat-treat it and perforate it. National Metallizing Division, Standard Packaging Co., has been chosen to coat the film with aluminum by vacuum deposition. The processed gore sections will be sealed together by ultrasonic bonding. The ultrasonic bonding unit will be purchased or rented from Ultra-Sonic Seal Inc., Ardmore, Pa.

Work has been started on the fabrication of the deliverable items. Sea Space Systems, Inc., has been selected to deliver extruded high density biaxially oriented polyethylene

-1-

film. This will be used to fabricate the final cap sections. The non-oriented standard polyethylene film which will be used to make one cap section has been irradiated at Electronized Chemical Corporation, Burlington, Mass. During the irradiation process the film fused together, rendering the lot of film processed unacceptable. A new batch of film will be irradiated after the cause of the first run problem has been determined by Electronized Chemical. Finally, calculations to determine the thickness of film required to withstand a buckling pressure of 5 times solar pressure have been completed. It was found from the most reliable of four trial calculations that the thickness required to sustain the calculated buckling pressure is in the range of 0.50 to 1.48 mils.

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# 2.0 FILM EVALUATION

# 2.1 Phillips Joanna - Marlex 6002

Tensile tests were performed on the Phillips Joanna high density polyethylene film as received. The tensile tests in the direction of extrusion indicated satisfactory uniformity and strength. Tensile tests in the transverse direction, however, showed poor uniformity and poor strength (see Table 1).

#### Table 1

Transverse Yield Force and Maximum Elongation for Phillips Marlex 6002 (900) Polyethylene Film

Sample No.	F (1b <sup>y</sup> )	€ max (%)	Comments
1	3.2	80	spike, some strength, little elongation
2	2.65	76	spike, some strength, little elongation
3	2.85	100	spike, some strength, little elongation
4	3.0	372	acceptable strength and elongation
5	3.0	612	acceptable, but some irregularities in plastic region
6	3.2	444	acceptable strength and elongation
7	2.90	484	acceptable strength and elongation
8	3.0	360	acceptable strength and elongation
9	3.2	116	acceptable, but weakness in elongation
10	3.2	188	acceptable, but weakness in elongation

Upon closer examination and discussions with the manufacturer it was found that the material was extruded at

-3-

a low temperature (most likely below the crystalline melting point), leaving small particles of unextrded resin within the film. This is further substantiated by the facts that the film is cloudy, irradiates poorly, and shows small tears at 200X magnification when it is strained to its plastic range.

# 2.2 Phillips Joanna - Marlex 600B2

An additional sample of 1 mil high density polyethylene film was obtained from Phillips Joanna. Upon evaluation, tensile tests in the direction of extrusion proved satisfactory but tensiles in the transverse direction were very poor and irregular (see Table 2). Visual examination of the film indicated that stress lines were present in the direction of extrusion, Figure 1.



# Figure 1 PHILLIPS FILM SHOWING STRESS LINES

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# Table 2

Sample No.	Fy (1b.)	€ max (%)	Comments
1	3.75	388	acceptable strength and elongation
2	3.15	372	acceptable strength and elongation
3	3.20	264	acceptable, but weakness in elongation
4	3.50	172	acceptable, but weakness in elongation
5	2.50	80	spike, some strength, little elongation
6	3.20	92	spike, some strength, little elongation
7	1.80	48	spike, some strength, little elongation
8	2.50	52	spike, some strength, little elongation
9	3.15	64	spike, some strength, little elongation
10	0.50	16	spike, little strength or elongation
11	0.05	16	spike, little strength or elongation
12	1.80	40	spike, little strength or elongation

Transverse Yield Force and Maximum Elongation for Phillips 600B2 Polyethylene Film (90°)

The extrusion lines had high stress concentrations, so any tensile sample which included one or more of these lines gave very poor strength and elongation. The results obtained on these samples precluded the acceptance of this material.

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2.3 <u>Sea Space - Standard Density Polyethylene Film</u>

Quantities of Sea Space 1 mil standard density polyethylene film were obtained and tested. The film was found to be satisfactory. Table 3 lists the initial properties of the film.

#### Table 3

Initial Properties of 1 Mil Standard Density Sea Space Polyethylene Film

¥

<b>G</b> y <sub>0</sub> (psi x 10 <sup>3</sup> )	$\frac{E}{(\text{psi x } 10^3)}$	Oy90 (psi x 10 <sup>3</sup> )	<sup>E</sup> 90 (psi x 10 <sup>3</sup> )	Tm (°C.)	
1.15 <sup>±</sup> 0.12*	26 <b>.</b> 3±2 <b>.</b> 9	1.11±0.09	20.4±4.9	110**	

All tensile tests performed on a Table Model Instron with a strain rate of 2"/min. 5 pounds full scale load.

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\*\* The average result of four different tests. See Section 3.0

3.0

# CRYSTALLINE MELTING POINT (T<sub>m</sub>) OF SEA SPACE STANDARD DENSITY POLYETHYLENE FILM

The crystalline melting point of the Sea Space film was determined in the following four ways:

1. Immersion heating

2. Heating while restrained

- 3. Stress relaxation upon increased temperature
- 4. Modulus of elasticity changes with temperature.

#### 3.1 Immersion Heating

A small sample of film was immersed in silicone oil at room temperature and heated slowly. At 95°C. the sample lost some opacity and at 109°C. became completely transparent. In addition, at 109°C. the sample exhibited some fluid properties, i.e., flowing and fusion. This property change with temperature indicated that 109°C. was the crystalline melting point of the polyethylene film as determined by this method.

#### 3.2 Heating while Restrained

This experiment consisted of restraining 1 mil film, with seven 1.8 cm. diameter circles punched in a face-centered hexagonal array,<sup>1</sup> in a steel ring.<sup>2</sup> The restrained sample was then heated slowly from room temperature. As the temperature increased, the circles became elliptical in shape. At  $107^{\circ}$ C. the elliptical holes became more elongated and began to tear. This temperature, where large deformations begin to occur, is indicative of the T<sub>m</sub> (crystalline melting point).

## 3.3 Thermal Stress Relaxation

One inch wide samples of Sea Space film were elongated at 75°C. past their yield points on an Instron tensile tester operating at a strain rate of 20 in./min.

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The straining was then stopped and heating was begun. The force to keep the film at its constant elongation was then measured as temperature increased. The resulting data points are shown plotted in Figure 2. It can be seen that the force begins to drop to zero at 110°C. This temperature (110°C.) is indicative of the crystalline melting point temperature.

# 3.4 <u>Modulus of Elasticity Changes with Temperature</u>

The modulus of elasticity, E, of the film was determined from standard tensile tests at temperatures from 25°C. to 115°C. The results are plotted in Figure 3. The temperature where E approaches zero is considered the crystalline melting point, since this is the temperature where the material looses its solid properties and begins to behave as a fluid.

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#### RATIO OF FLEXURAL RIGIDITIES

Flexural rigidities were determined for various thicknesses of polyethylene plated with copper (5 x  $10^{-6}$  in.) in preparation for the thickness calculations to follow. In addition, the flexural rigidity of 2 mil copper was determined. The method used was the standard ASTM D1388 55T Beam Cantilever Test. The ratio G  ${}^{cu}_{GF} = K(t)$  was determined as follows:

$$K(t) = \frac{G^{cu}}{G^{F}} = \frac{W_{cu}}{W_{F}} \frac{(\frac{1}{2}cu)^{3}}{(\frac{1F}{2})^{3}}$$

where  $W_{cu}$  = area weight of copper = density of copper x thickness =  $P_{cu}t_{cu}$ 

> $W_F$  = area weight of film = density of film x thickness =  $\rho_F t_F$  $l_{cu}$  = length of overhang of copper

 $l_F$  = length of overhang of film

K(t) is then equal to

4.0

$$K(t) = \frac{\beta_{cu}}{\beta_{F}} \left(\frac{1_{cu}}{1_{F}}\right)^{3} \frac{t}{t_{F}}$$
(1)

Experimental values of the ratio of lengths of overhang and the corresponding values of K(t) are given in Table 4 and are plotted in Figure 4 for various film thicknesses.

	l <sub>cu</sub>	<b>T</b> (+)
(inch)	l <sub>F</sub>	K(t) dimensionless
0.30	4.17	4567
0.30	7.35	25077
0.55	3.33	1262
0.55	2.94	873.5
1.00	2,50	296.0
1.00	1.92	133.9
2.00	1.35	23.3
5.50	1.42	9.3

Flexural Rigidity Ratio K(t) Polyethylene Coated with 5 x 10<sup>-6</sup> inches of Copper

Table 4

¥

Polyethylene coated with  $5 \ge 10^{-6}$  inches of copper on on side



#### 5.0 THICKNESS CALCULATION

In the Quarterly Report for December 1965-February 1966 thickness calculations were performed using a theoretical approach<sup>3,4</sup> and an empirical approach based on derivations in Engineering Dynamics, a paper by Reiss, Greenberg and Keller,<sup>6</sup> Keller, 6 and an empirical form factor in the final report of "Development of an Inflatable Rigidizing Satellite," by G.T. Schjeldahl Company. As noted in the Quarterly Report for December 1965-February 1966, the theoretical approach is inaccurate for very thin films (large  $\frac{R}{t}$  ratios) where R is the radius and t the film thickness. Furthermore, the combined approach of the last report was only an approximation, based upon an empirical factor for another material. The methods to be used in this report will separately compare required thickness based on two models. One model is a uniformly loaded cap section, the other model is a point loaded cap section. For comparison the calculations have been performed with and without the material correction factor.

#### 5.1 Calculation Without Material Factor

## 5.1.1 Uniformly Loaded Sphere

The articles by Reiss,<sup>6,8</sup> deal with the buckling of a uniformly loaded spherical cap section of small curvature. The articles determine critical buckling pressure for spherical caps of various geometries. The results given in the articles and which are plotted in Figure 6, curve B were determined both numerically and experimentally. The critical buckling pressure is given in non-dimensional form:

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$$P_{crr}(\hat{p}) = \frac{P_{cr}}{2Ek^{\frac{1}{2}}(\frac{t}{R})^2}$$
(2)

or  $P_{cr/t^2} = \frac{2Ek^{\frac{1}{2}}}{R^2} P_{crr}(\rho) = U(\rho)$ where  $P_{crr}(\rho) =$  non-dimensional buckling pressure  $P_{cr} =$  actual buckling pressure (=6.5 x 10<sup>-9</sup>)  $k = \frac{2}{3}(1 - y^2) = 0.50$  y = Poisson's ratio (= 0.50) t = film thickness R = radius of spherical cap = 2550 inch E = Young's modulus = 1 x 10<sup>4</sup> psi

The non-dimensional pressure  $P_{crr}$  is plotted against a non-dimensional geometrical parameter, p, given as:

$$P = 2k^{-\frac{1}{2}} \frac{r^2}{R} \frac{1}{t}$$
(3)

where r = radius of deformation, see Figure 5. It was found convenient to plot  $\frac{Pcr}{+2}$  against  $\beta$ 

for all modes of deformation. The results are given in Figure 6, curve B. It can be seen from curve B that  $\frac{Pcr}{t^2}$ varies between 2 and 4.3. For a critical buckling pressure of 6.5 x 10<sup>-9</sup> psi, this range corresponds to thicknesses between 1.22 x 10<sup>-4</sup> inch to 1.72 x 10<sup>-4</sup> inch.

# 5.1.2 Point Loaded Sphere

In Engineering Dynamics,<sup>9</sup> critical buckling pressures have been theoretically determined for spherical cap sections of various geometries. The results are given

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in terms of a loading parameter ( $\mathbf{N}$ ) versus a geometrical parameter ( $\lambda$ ). They are defined respectively as follows:

$$\mu = \frac{\frac{\mathrm{RP}}{\mathrm{Cr}}}{\frac{\mathrm{Cr}}{\mathrm{Et}^3}}$$
(4)

$$\lambda = \frac{r^4}{t^2 R^2} \tag{5}$$

where  $P_{cr}^{c}$  = critical buckling force

If  $P_{cr}$  is concentrated as a force,  $P_{cr}^{c}$ , at a point on the center spherical cap, then

$$P_{cr}^{c} = \pi r^{2} P_{cr}$$
 (6)

This transformation is shown in Figure 5.



Figure 5 TRANSFORMATION FROM DISTRIBUTED LOAD TO POINT LOAD From the graph of  $\mu$  and  $\lambda$  in Engineering Dynamics,  $\mu$  may be represented by the equation:

$$\mu = \sqrt{0.093(\lambda + 11.5)} - 0.94 \approx 1 \times 10^{-2} \lambda + 1 \quad (7)$$

It should be noted that at  $\lambda > 500$  the approximation of equation (7) gives a high value of  $\mu$  which will provide an additional factor of safety for the designer. If equations (3)-(7) are used to solve for P<sub>cr</sub> in terms of  $\rho$  and t the following equation is obtained:

$$P_{cr} = \frac{2E}{\Pi R^2 k^2} \left( \frac{10^{-2}k}{4} \beta^2 + \frac{1}{\beta} \right) t^2$$
$$= F(\beta) t^2$$
(8)

 $\frac{P_{cr}}{t^2}$  was then plotted versus  $\rho$ . At  $\rho = 30$   $\frac{P_{cr}}{t^2}$  is a minimum. At the minimum  $\frac{P_{cr}}{t^2}$ , t is found by solving equation (8); the result is 0.0076 inch.

5.2 <u>Calculation with Material Factor</u>

5.2.1 Point Loaded Sphere

If equation (4) is written in terms of the uniform critical buckling pressure, using equation (6), the result is as follows:

$$P_{cr} = \frac{E \mu t^3}{\pi r^2 R}$$
(9)

It can be seen from this equation that the critical buckling pressure is proportional to the flexural rigidity, G, of the material since

$$G = EI \propto Et^3$$

A material correction factor ( $\frac{G^{F}}{c^{std}}$ ) then suggests itself

$$P_{cr}^{F} = \frac{G}{G^{std}} P_{cr}^{std} = \frac{G}{G^{std}} \frac{Eut^{3}}{\pi r^{2}R}$$
(10)

where  $P_{cr}^{F} = P_{cr} = 6.5 \times 10^{-9} \text{ psi.}$ 

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The standard material chosen is 2 mil copper, since an accurate value of G<sup>cu</sup> can be experimentally determined and equation (9) is known to apply for metal films in that range. In this case equation (10) is as follows:

$$P_{cr} = \frac{G^{F}}{g^{cu}} P_{cr}^{cu} = \left(\frac{G^{F}}{g^{cu}}\right) \left(\frac{E_{cu} \mu^{t} c_{u}}{\pi r^{2} R}\right)^{t^{2} cu}$$
(11)  
where  $t_{cu} = 2 \times 10^{-3}$  inch  
Using equation (8)  $P_{cr}^{cu}$  is  

$$P_{cr}^{cu} = \frac{2E_{cu}}{R^{2} k \frac{1}{2}} \left(\frac{10^{-2} k_{cu}}{4}\right)^{t} + \frac{1}{P} t_{cu}^{2}$$
(12)  

$$= F_{cu}^{cu} P_{cu}^{2} t_{cu}^{2}$$
(12)  
where  $E_{cu} = 2 \times 10^{7}$  psi  
 $k = 0.95$   
 $R = 2550$ 

It therefore follows that  $P_{cr}$  can be given as

$$P_{cr} = \frac{g^{F}}{g^{cu}} F^{cu}(\rho) t_{cu}^{2}$$
(13)

with  $t_{cu} = 2 \times 10^{-3}$  in.  $P_{cr} = 6.5 \times 10^{-9}$  psi.

With  $\frac{G^{cu}}{G^F} = K(t)$ , given in Section 4.0 for various

thicknesses of polyethylene and  $F^{cu}(\mathcal{P})$  given by equation (12) and plotted in Figure 7, curve A, the film thickness may then be calculated using equation (13) in the following form:

$$K(t) = 0.62 \times 10^{+3} F^{cu}(\mathbf{P})$$
 (14)

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Values of film thickness for various values of  $\rho$  are given in Table 5 below.

T	â	b	1	ê	-5
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٩	F <sup>cu</sup> ())	$K(t) \times 10^{-3}$	$t \times 10^{+3}$
10	.25	0.16	1.45
20	.24	0.15	1.48
30	.25	0.16	1.45
60	.38	0.24	1.15
100	.58	0.36	0.95
200	1.15	0.71	0.70
350	1.90	1.18	0.50

Final Thickness of Composite Film

# 5.2.2 Uniformly Loaded Sphere

If the critical buckling pressure in equation (2) is also corrected for rigidity using the factor

$$\frac{1}{K(t)} = \frac{g^{F}}{g^{cu}}$$

the modified equation is as follows:

$$P_{cr} = \frac{1}{K(t)} \left[ 2E_{cu} k_{cu}^{\frac{1}{2}} \left( \frac{t_{cu}}{R} \right)^2 P_{crr}(\rho) \right]$$

or  $1.63 \times 10^{-3} K(t) = U^{cu}(\rho)$  (15)

with K(t) given in Figure 4 and  $U^{cu}(9)$  plotted in Figure 7, it can be seen that with  $U^{cu}(9)$  varying between 11 and 5.5, for the values of  $\beta$  under consideration, t will vary between 0.25 x 10<sup>-3</sup> in. and 0.35 x 10<sup>-3</sup> in.



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-20-



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#### Comparison of Derived Thicknesses

5.3

Table 6 summarizes the values of the thickness for the various methods of calculation.

It is believed that 0.50 to 1.48 mil range is the most reliable of the four calculated thickness ranges for the following reasons. Using the ratio of flexural rigidities, K(t), the buckling pressure equation was corrected for the actual material to be used. In addition using a point loaded instead of a uniformly distributed load creates an additional safety factor, since a point load is a more severe condition to cause buckling than a distributed load. It is even possible that a point load buckling is in fact the correct way to describe critical buckling of a very thin walled sphere, since thesphere actually buckles in a "dimple-like" point fashion.<sup>10</sup>

Derived Thickness of Polyethylene Film

Table 6

.

1.Polyethyleneuncorrecteduniform( $6.5 \times 10^{-9}$ ) $1.22-1.72 \times 10^{-4}$ in.2.Polyethyleneuncorrectedpoint( $6.5 \times 10^{-9}$ ) $3.00-7.60 \times 10^{-3}$ in.3.Polyethylene( $+5 \times 10^{-6} \ln . Cu$ )corrected foruniform( $6.5 \times 10^{-9}$ ) $2.50-3.50 \times 10^{-4} \ln .$ 4.Polyethylene( $+5 \times 10^{-6} \ln . Cu$ )corrected forpoint( $6.5 \times 10^{-9}$ ) $0.50-1.48 \times 10^{-3} \ln .$ 4.Polyethylene( $+5 \times 10^{-6} \ln . Cu$ )corrected forpoint( $6.5 \times 10^{-9} \mathrm{mr}^2$ ) $0.50-1.48 \times 10^{-3} \ln .$		Material	Method	Load	Thickness Range
2. Polyethylene uncorrected point(6.5x10 <sup>-9</sup> $frr^2$ ) 3.00-7.60x10 <sup>-3</sup> in. 3. Polyethylene (+5x10 <sup>-6</sup> in.Cu) corrected for uniform(6.5x10 <sup>-9</sup> ) 2.50-3.50x10 <sup>-4</sup> in. copper 2.50-3.50x10 <sup>-4</sup> in. 4. Polyethylene (+5x10 <sup>-6</sup> in.Cu) corrected for point(6.5x10 <sup>-9</sup> $frr^2$ ) 0.50-1.48x10 <sup>-3</sup> in.	1.	Polyethylene	uncorrected	uniform(6.5x10 <sup>-9</sup> )	1.22-1.72x10 <sup>-4</sup> in.
<ul> <li>3. Polyethylene (+5x10<sup>-6</sup>in.Cu) corrected for uniform(6.5x10<sup>-9</sup>) 2.50-3.50x10<sup>-4</sup>in.</li> <li>4. Polyethylene (+5x10<sup>-6</sup>in.Cu) corrected for point(6.5x10<sup>-9</sup> Hr<sup>2</sup>) 0.50-1.48x10<sup>-3</sup>in.</li> </ul>	5	Polyethylene	uncorrected	point(6.5xl0 <sup>-9</sup> 4r <sup>2</sup> )	3.00-7.60×10 <sup>-3</sup> in.
4. Polyethylene (+5x10 <sup>-6</sup> 1n.Cu) corrected for point(6.5x10 <sup>-9</sup> 47r <sup>2</sup> ) 0.50-1.48x10 <sup>-3</sup> 1n. copper	<b>з.</b>	Polyethylene (	+5x10 <sup>-6</sup> in.Cu) corrected for copper	uniform(6.5x10 <sup>-9</sup> )	2.50-3.50x10 <sup>-4</sup> 1n.
	4.	Polyethylene (	+5x10 <sup>-6</sup> 1n.Cu) corrected for copper	point(6.5x10 <sup>-9</sup> Hr <sup>2</sup> )	0.50-1.48x10 <sup>-3</sup> 1n.

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# 6.0 IRRADIATION ATMOSPHERE TESTS

# 6.1 Initial Tests: Air, Nitrogen

A series of tests were performed to determine the effects of atmosphere and heat treatment on irradiated polyethylene film in preparation for the irradiation and heat treatment of the materials for the deliverable items. Various samples of film were irradiated to 15 Mrads. Some of the samples were irradiated in a nitrogen atmosphere, others in an air atmosphere. Half of the above irradiated samples were immediately heat treated to ca. 100°C. The irradiation, heat treatment atmosphere scheme is shown in Table 7.

# Table 7

Irradiation Heat Treatment Atmosphere Experiment Scheme (Air, Nitrogen) Irradiation of 1 Mil Sea Space Polyethylene Film to 15 Mrads

Run No.	Irradiation Atmosphere	Heat Treatment Atmosphere
1	Nitrogen	Nitrogen
2	Air	Nitrogen
3	Nitrogen	No Heat Treatment
4	Air	No Heat Treatment

Tensile tests were then performed about once every three days for almost a month. The tests which were repeated twice, A & B runs, (see Figures 8 - 11) in general show an increase in strength (reported as modulus of elasticity, E) with time which appears to persist. It is believed that the scatter and variations in the results are caused by the competing effects of oxygen degradation and post-irradiation crosslinking. Both effects are caused by residual postirradiation trapped free radicals. It therefore appears that the initial heat treatment given to some of the samples

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was not sufficient enough to decay the free radicals.

With the possibility in mind that the initial heat treatment was not sufficient to eliminate the free radicals, all four of the samples were carefully heat treated above their crystalline melting point on the 12th day (this means that two of the samples were heat treated twice). Tensile tests were then performed on the samples to check for variations with time. The results, shown in Figures 12 - 15 still in general appear to rise somewhat, although the variation is less than before the second heat treatment.

#### 6.2 Tests: Oxygen, Nitrogen, Air

To further evaluate the effects of atmosphere (especially the effect of oxygen) on polyethylene film during irradiation, samples of film were irradiated and heat treated in an oxygen atmosphere in combination with the other gases used in the first part of the experiment (nitrogen and air). The tests were conducted according to the scheme outlined in Table 8. Tensile tests were conducted approximately every three days for three weeks.

#### Table 8

Irradiation, Heat Treatment, Atmosphere Experimental Scheme (Oxygen, Nitrogen, Air) Irradiation of 1 Mil Sea Space Polyethylene Film to 15 Mrads

Run No.	Irradiation Atmosphere	Heat Treatment	
1	Oxygen	Oxygen	
2	Oxygen	Nitrogen	
3	Oxygen	None	
4	Nitrogen	Oxygen	
5	Air	Air	

The results of this set of tests are summarized in Figures 16 through 20. In general there is a fairly sharp increase in strength for all of the runs a few days after irradiation which then levels off approximately in 18 days. The samples show larger increases in strength in the 90° direction than in the 0° direction. In all cases there appears to be no degradation. In fact there is a net increase in strength for all tests. Also, the fact that there even was a net increase (although small) in strength with samples irradiated in oxygen and heat treated in oxygen, indicates with some certainty that the effect of oxygen is not critical in the irradiation or heat treatment of this polyethylene film under the conditions used in our evaluation. These results lead to the conclusion that irradiation and heat treatment in air will present no problem with regard to oxygen degradation. In fact, run no. 5, Figure 20, irradiation and heat treatment in air, shows approximately 10 psi increase at steady state over its initial unirradiated conditions.

#### 6.3 Effect of Dose

For informational purposes the initial tests listed in Table 7 were run at 30 Mrads to check the effect of increasing the dose on the strength time curves of the film. The results are given in Figures 21 - 24. The results in general increase sharply upon initial irradiation and gradually decrease. The decrease, although noticeable, does not indicate any serious degradation.

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#### 7.0 EXTRACTION STUDIES

The dose variation-extraction tests have been started on the 1 mil standard density polyethylene film. Samples of film have been irradiated from 5 to 75 Mrads, allowed to stabilize for approximately 1 month and the uncrosslinked fractions were extracted in xylene to constant weight. Additionally, tensile tests were run on the irradiated samples before and after extraction. With the completion of density measurements using density gradient columns  $E/\rho$ values will be reported. At present, work is being done on the setup of the density gradient columns for the density evaluation of the extracted film.

#### 7.1 Experimental Results

#### 7.1.1 Variation of Strength with Dose

Tensile tests have been run on samples of 1 mil standard density polyethylene film given doses of irradiation from 5 to 75 Mrads. The results, see Table 9 and Figure 25 indicate only slight changes in E. In fact a slight decrease with dose in the  $0^{\circ}$  direction is observed.

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# Table 9

Dose	Modulus of Elasticity E	Modulus of Elasticity <u>E/</u>
(Mrads)	(psi x 10+3)	(psi x 10+3)
0 5 10 15 20 30 40 50 60 70 75	$23.8 \pm 1.7$ $23.8 \pm 1.6$ $31 = 25.5 \pm 1.7$ $18.5 \pm 1.7$ $30.7 \pm 0.9$ $24.3 \pm 1.2$ $29.8 \pm 1.6$ $23.5 \pm 1.0$ $22.4 \pm 1.0$	$38.6 \pm 1.3$ $31.7 \pm 1.9$ $40 \pm 2.1$ $26.5 \pm 2.2$ $35.5 \pm 0.4$ $36.6 \pm 1.0$ $39.4 \pm 2.5$ $27.8 \pm 1.1$ $29.0 \pm 0.3$

Effect of Radiation\* Dose\*\* on the Strength of 1 Mil Standard Density Sea Space Polyethylene Film

\* Irradiated in Nitrogen, Heat Treated in Nitrogen \*\* Accurate to  $\frac{+}{-}$  1 Mr.

# 7.1.2 Solvent Tests

Samples of unirradiated polyethylene were extracted for one day in the various solvents at  $100^{\circ}$ C.  $\pm 2^{\circ}$ C. The results in order of extractibility are listed in Table 10 below.

#### Table 10

Extraction Efficiency of Various Solvents on Polyethylene

Solvent	Туре	% Extracted*
Decalin	Unsaturated	99.23
Tetralin	Saturated	99.19
Xylene	Saturated	99.08
O-Dichlorobenzene	Unsaturated-Polar	83.74
Butyric acid	Saturated	4.92
Dimethyl formamide	Polar	0.98
Dimethyl sulfoxide	Polar	-0.19

% Extracted = % dissolved

Figure 25 EFFECT OF RADIATION\* DOSE ON STRENGTH OF

1 MIL STANDARD DENSITY POLYETHYLENE FILM

 $\cdot$  0° direction to extrusion x 90° direction to extrusion

\* Irradiated in Nitrogen, Heat Treated in Nitrogen, 100°C.



The results indicate that solvents which have mainly saturated or unsaturated character with little or no polarity are the best solvents for extracting polyethylene which is itself mainly a saturated substance. The solution of the polyethylene at  $100^{\circ}$ C.  $\pm$  2°C. by the first three solvents indicate that they are very acceptable solvents for polyethylene extractions.

#### 7.1.3 Extraction

The extractible low molecular weight fractions in the irradiated polyethylene film samples were extracted in xylene at 100°C. for 4 days. The 4-day time was chosen to insure complete extraction. In addition, the antioxidant n-beta-phenylnaphthylamine was added to each solvent to retard oxidation during extraction.

# 7.1.4 Gel Fractions

The most effective way of determining the effect of irradiation on the crosslinking of a polymer is to determine its gel fractions, i.e.

# $\frac{\text{weight of non-extractible material}}{\text{weight of unextracted material}} = \frac{\text{wt. gel}}{\text{wt. sol}}$

The various gel fractions for the different doses of irradiation are plotted in Figure 26. It can be seen from the curve that pronounced gelation (crosslinking) occurs after 10 Mrads of dose. This result gives further verification that a dose of 15 Mrads is satisfactory for sufficient crosslinking. The curve furthermore shows that at ca. 75 Mrads the materials approach a constant maximum gel of 85%.

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## Figure 26 GEL FRACTIONS OF IRRADIATED POLYETHYLENE FOR VARYING RADIATION DOSES

# Irradiation: Nitrogen Atmosphere Heat Treatment: Nitrogen, 100°C. Samples Aged Approximately 1 Month After Irradiation



7.1.5 Strength Dose\_Variations of Extracted Material

Tensile tests have been run on samples of 1 mil standard density polyethylene film given various doses of irradiation and then extracted. The results for the 0<sup>0</sup> direction only, is given in Table 11 below and in Figure 27.

#### Table 11

# Effect of Complete Extraction\* on Strength of 1 Mil Standard Density Sea Space Polyethylene Film Given Various Doses of Radiation\*\*

Dose	Modulus of Elasticity, E
(Mrads)	(psi x 10 <sup>3</sup> )
0 5 10 15 20 30 40 50 60 70 75	0 0 3.76 5.85 24.3 26.3 19.5 24.0 18.9 20.2

Irradiated in Nitrogen, Heat Treated in Nitrogen, 100°C.

\*\* Extracted in xylene at 100°C. for 4 days with 0.1% n-beta-phenylnaphthylamine

Figure 27

## 27 EFFECT OF COMPLETE EXTRACTION ON STRENGTH OF 1 MIL STANDARD DENSITY SEA SPACE POLYETHYLENE FILM GIVEN VARIOUS DOSES OF RADIATION

Irradiated in Nitrogen Heat Treated in Nitrogen, 100°C. Extracted in Xylene at 100°C. for 4 days with 0.1% n-beta-phenylnaphthylamine



# 7.1.6 Projected Results of Extraction Program

With the completion of the density measurements E/9 can be evaluated. It may be expected that the E/9values resulting from increased irradiation and/or extraction will not be significantly improved, since the largest decrease in density expected will be no more than 20% and E, already evaluated has not significantly increased (in fact, in some instances it has actually decreased). In light of the -48above considerations it may be assumed at this time that irradiation-extraction methods are not efficient ways of increasing E/c to any substantial degree.

In conjunction with strength to density studies, RAI Research Corporation has recently undertaken an in-house research effort in methods and designs for obtaining a high strength low weight material for the satellite. The method consists of metallizing ultra thin ca. 0.10 mil polyethylene film with copper (or aluminum) to ca. 10 x  $10^{-6}$  in. on both sides. Preliminary tests indicate that the strength of the composite material is about 100-times stronger than the polyethylene alone. If the flexural rigidity of the ultra thin composite material increases accordingly, and is in the same flexural rigidity range as the film coated with 5 x  $10^{-6}$  in. copper, i.e.,  $10^2 \leq K(t) \leq 10^3$  (see Section 4.0) then it would be possible to substitute this thinner material and obtain a considerable savings in weight.

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# 8.0 DELIVERABLE ITEMS

Various companies have been contacted in conjunction with the fabrication of the deliverable items. The following section indicates the current status on these services and their prices.

## 8.1 Production Steps

8.1.1 Film Procurement

Source: Sea Space Systems, Inc.

- Material Types: a) biaxially oriented, standard density polyethylene film, 1 mil (\$660/10 rolls).
  - b) biaxially oriented, high density polyethylene film, 1 mil (\$710/10 rolls).

The biaxially oriented high density film will be produced on a best efforts basis. The standard density polyethylene film has already been received. Sea Space has already started the extrusion of the high density film.

# 8.1.2 Irradiation

The original source of irradiation chosen was Radiation Dynamics Incorporated (RDI). It was then further decided to use the services of Electronized Chemical Corp. since they have irradiation facilities which can handle thin films. Samples of standard density Sea Space 1 mil film, from 1 to 24 thicknesses were irradiated to 15 Mrads at 5 and 2.5 Mrads/pass. The results of the trial run indicated that there was no degradation, melting or sticking. The 1200 ft. of standard density film was then irradiated in a flat position 8 layers thick. The material fused in many places and was considerably wrinkled rendering it unsatisfactory for further processing. Electronized Chemical Corp. is at present

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trying to determine the cause of the problem. When the cause is discovered, a new lot of film will be irradiated. Tentatively, the film will be irradiated in a single thickness. The single thickness irradiation has two advantages. In the first place, the problem of film sticking will be eliminated. Secondly, this will prevent any wrinkling during irradiation since a single thickness film can easily be pulled taut and smooth while passing the accelerator beam. The cost for processing the film necessary for construction of the deliverable items in a single thickness is approximately \$2000.

8.1.3 <u>Heat Treatment</u>

Source: Sea Space Systems, Inc.

The costs for heat treatment are \$600 for first roll and \$350 for each additional roll.

## 8.1.4 <u>Metallic Coating</u>

Source: National Metallizing Div., Standard Packaging Co.

The coating will consist of about 2500 Å of aluminum vacuum deposited on the polyethylene continuously. The cost is \$250 per roll on a best efforts basis.

#### 8.1.5 <u>Ultrasonic Bonding</u>

A number of companies have been investigated as to their capabilities for ultrasonically bonding crosslinked polyethylene film. The companies investigated as well as their approximate prices are as follows:

Branson-Sonic Power, Danbury, Conn. Edison Instruments, Inc., Rawhay, N.J. Gultin Industries, Inc., Schiller Park, Ill. Ultra Sonic Seal, Inc., Ardmore, Pa. \$1500-2000 5000 400 2500 or \$300/month rental

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Samples have been sent out to the above companies for a feasibility study. Upon receipt of their samples it has been found that only Ultra Sonic Seal produces an ultrasonic bonding machine capable of satisfactorily bonding crosslinked polyethylene film.

# 9.0 FUTURE WORK

During the next reporting period work will be continued to fabricate the deliverable items. New quantities of standard density polyethylene film will be irradiated and then heat treated. Arrangements will be made with Ultra Sonic Seal to obtain an ultrasonic welding apparatus for the near future.

The density measurements required by the extraction program will be completed. Additionally, the ultra thin film-metallization approach to increasing  $E/\rho$  will be further pursued.

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#### 10.0 REFERENCES

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