(NASA-CR-178116) PROTECTIVE COATINGS FOR COMPOSITE TUBES IN SPACE APPLICATIONS (Boeing Aerospace Co., Seattle, Wash.) 12 p CSCL 11B
N87-18669
Unclas
G3/27 43648

PROTECTIVE COATINGS FOR COMPOSITE TUBES IN SPACE APPLICATIONS

Harry W. Dursch and Carl. L. Hendricks
Boeing Aerospace Company
Seattle, Washington 98124

Abstract

Protective coatings for graphite/epoxy (Gr/Ep) tubular structures for a manned Space Station truss structure were evaluated. The success of the composite tube truss structure depends on its stability to long-term exposure to the low Earth orbit (LEO) environment with particular emphasis placed on atomic oxygen. Concepts for protectively coating Gr/Ep tubes include use of inorganic coated metal foils and electroplating. These coatings were applied to Gr/Ep tubes and then subjected to simulated LEO environment to evaluate survivability of coatings and coated tubes. Evaluation included: atomic oxygen resistance, changes in optical properties and adhesion, abrasion resistance, surface preparation required, coating uniformity, and formation of microcracks in the Gr/Ep tubes caused by thermal cycling. Program results demonstrated that both phosphoric and chromic acid anodized Al foil provided excellent adhesion to Gr/Ep tubes and exhibited stable optical properties when subjected to simulated LEO environment. SiO_2/Al coatings sputtered onto Al foils also resulted in an excellent protective coating. Electroplated Ni exhibited unacceptable adhesion loss to Gr/Ep tubes during atomic oxygen exposure.

This work was funded by NASA Langley Research Center, Contract NAS1-16854, titled "Development of Composite Tube Protective Coatings".

1.0 INTRODUCTION

Gr/Ep tubular struts comprise the baseline design for the large "dual keel" rectangular truss structure of the manned Space Station. These tubular struts will be approximately 2-in-diameter and up to 23-ft-long. There are many requirements for these tubes including high stiffness, dimensional stability, close dimensional tolerance and long life in LEO. The success of the composite tube truss structure depends on its ability to endure long-term exposure to various LEO environmental factors such as atomic oxygen, thermal cycling, charged particle radiation, ultraviolet radiation, micrometeriods and space debris. Atomic oxygen environment at LEO is especially severe. The recombination of atomic oxygen absorbed on unprotected Gr/Ep surfaces causes substantial erosion of the composite. Combined effects must also be taken into account. For example, micrometeroid penetration of protective coatings would allow a mechanism for atomic oxygen degradation of the Gr/Ep tube.

This paper describes the development and evaluation of LEO protective coatings applied to Gr/Ep tubular struts. Candidate coatings included anodized Al foil, sputtered SiO_2/sputtered Al/Al foil, bare Al foil and electroplated Ni. Adhesive systems and fabrication techniques were evaluated for bonding foils to the tubes and surface treatments were evaluated for promotion of electroplating adherence. Evaluation of coatings and coated tubes included resistance to atomic oxygen (using Boeing's large scale plasma atomic oxygen screening facility), thermal cycling, abrasion resistance, bond surface preparation, formation of microcracks in tubes, changes in optical properties and adhesion after testing, and coating uniformity.

Four 2-in-diameter by 8-ft-long Gr/Ep tubes, fitted with representative space-erectable truss structure end fittings, were fabricated with the selected protective coating to verify full scale fabrication techniques.

2.0 GR/EP COMPOSITE SELECTION AND PROCESSING

The composite material selected for tube fabrication was P75S/934 Gr/Ep manufactured by Fiberite Corporation. It was supplied as a unidirectional prepreg tape with a per ply thickness of 0.005-in. This material meets the primary design requirements of high composite stiffness (longitudinal tensile modulus > 45 Msi), relatively large data base, and commercial availability. P75S is a high modulus graphite fiber manufactured by Amoco Performance Products (formally Union Carbide) and 934 is an epoxy resin manufactured by Fiberite.

Composite tube ply orientation selected was $(0_2,\pm 20,0_2)_s$. This selection was based on analysis of P75S/934 composite ply orientations using a Boeing-developed computer program called INCAP. Analysis methods contained in INCAP are based on classical lamination theory; the base material properties of P75S/934 were developed from results of industry test data. Three basic ply stacking techniques were analyzed: $(0_2,\pm\theta,0_2)_s$, $(\theta,0,-\theta,0,\theta,0,)_s$ and

$(\pm\theta,0_2,\mp\theta)_s$. Table 1 shows the results of this analysis. The orientation $(0_2,\pm 20,0_2)_s$ was selected based on possessing a minimum modulus of 40 Msi and adequate crushing strength.

Construction technique selected for fabrication of the required composite tubes was convolute wrapping. Prepreg piles were wrapped onto the mandrel using a rolling table. Before the initial ply is wrapped, the mandrel has had several coats of releasing agents applied to ensure release of the cured composite tube. The wrapped mandrel was vacuum bagged, thermocoupled and then cured at 350^{o}F for 2 hours.

3.0 PROTECTIVE COATING EVALUATION

Several concepts for protectively coating Gr/Ep tubes were evaluated including; anodized Al foil, Al foil sputter-coated with Al and SiO_2, electroplated Ni (with and without SiO_x coatings) and inorganic sol gel solutions. Except for the large area SiO_2 coatings and SiO_x depositions, all the above coatings (along with the Gr/Ep tubes) were deposited or fabricated in Boeing facilities. The coatings were required to meet targeted optical values of an AM-0 solar absorptance = 0.20 to 0.35 and a thermal emittance = 0.15 to 0.25. Low absorptance values reduce the maximum tube temperatures when exposed to direct or albedo radiation and low values of emittance reduce minimum temperatures when exposed to deep space. A low specular reflectance was also a design goal. This would provide astronauts with a non-mirror like surface when conducting EVA. Figure 1 shows the predicted temperature range that a Gr/Ep tube, wrapped with Al foil possessing the required optical values, would undergo in LEO orbit. The maximum temperature is on the front side and is predicted to be +65F and the minimum temperature would be -55F on the backside of the tube.

Screen testing consisted of establishing a coating's ability to achieve the desired optical ~~properties and possessing processing~~ parameters that are compatible with the Gr/Ep tubes. Results of screen testing narrowed the initial list of coatings down to the following five: chromic and phosphoric acid anodized Al foil, sputtered SiO_2/sputtered Al/Al foil and electroplated Ni with and without an SiO_x coating.

Various thicknesses and tempers of Al foil were evaluated. All foil evaluated was Al alloy 1145. The 0.002-in-thick, 1145-H19 (fully strain-hardened) Al foil was selected as the lightest weight Al foil that could be consistently wrapped onto the 2-in-diameter tubes without tearing or pinholes caused by handling. Thicker Al foil can be bonded to the tubes to improve the resistance to impact damage, if it is required.

3.1 Anodized Al Foil

Anodizing of Al foil was performed initially using Boeing specifications. After anodizing, optical properties of the foil were determined. This established what could be achieved using Boeing specifications. Follow-up samples were then fabricated using modified anodizing parameters in an attempt to achieve the targeted optical values. Because anodizing was performed in production tanks, it was impossible to modify various acid solution/water percentages. Parameters that were varied included immersion time in the acid solution and/or ramp time to the desired voltage. The foils were not sealed because of expected adverse effects on the foil to Gr/Ep bonding strengths (ref. 1).

3.2 Sputtered SiO_2/Sputtered Al/Al Foil

Several iterations of sputtered SiO_2 and sputtered Al were deposited onto Al foil to determine the thicknesses required to obtain required optical values. Emittance was tailored by controlling thickness of the deposited SiO_2, and absorptance was tailored by controlling thickness of sputtered Al. It was found that, using Al foil as a substrate, sputtered Al layers of less than 1000-angstroms exhibited little if any grain growth, therefore no change in reflectance. Sputtered Al layers greater than 1000-angstroms developed increasing grain structure and, as a result, reflectance decreases (increased absorptance) as the grain structure increases. The optimized thickness proved to be 1-micron of SiO_2 and 3000-angstroms of Al.

Flexibility of the 1-micron layer of SiO_2 on Al foil was a concern because the coated foils would be wrapped around 2-in-diameter tubes. However, testing showed that no crazing of the SiO_2 took place unless the foil was actually creased by folding it in half.

3.3 Electroplated Nickel with and without SiO_x

Electroplating was selected as potential coating because of its low cost application methods, good corrosion resistance, good uniformity and ability to coat irregular-shaped surface such as tube end fittings. The exterior surfaces of the Gr/Ep tubes were sanded prior to plating to improve adhesion. These tubes were immersed in an electroless copper solution to provide the conductive surface required for electroplating. Because of expected degradation during atomic oxygen testing, SiO_x coatings were deposited onto Ni. The SiO_x also increased the emittance of Ni to within the targeted range.

4.0 COATING EVALUATION TEST RESULTS

The five selected coatings were bonded to 1-ft-long tubular sections of P75S/934 Gr/Ep tubes. These coatings were then subjected to LEO environmental testing that included atomic oxygen testing, thermal cycling, adhesion testing and abrasion resistance. Criteria used for evaluating coatings was change in optical properties and change in coating adherance.

4.1 Thermal Cycling

Coated tubes were initially thermal cycled in an LEO environment by placing them in a vacuum chamber with AM-0 solar simulation capabilities. Solar simulation was generated with xenon lamps providing a 35-in-diameter uniform beam. The heat sink of space was simulated using LN_2 shrouds. The tubes were subjected to 50 thermal cycles with each cycle consisting of 57-mins of solar and 37-mins without solar radiation. This cycle closely matches that of the Space Station at LEO. Using this testing technique, each coated tube was allowed to seek its own temperature versus time profile. Several of the tubes had thermocouples bonded to their surfaces. These profiles were used to verify analytical predictions of the temperatures during thermal cycling of the tubes. After completion of the cycling the tubes were optically evaluated, checked for formation of microcracks and evaluated for coating and foil adhesion. There were no detectable changes in any properties including formation of microcracks. Because of the unexpected lack of microcracking, further thermal cycling was undertaken. The tubes that had been exposed to the previous 50 cycles were subjected to an additional 500 56-min, +120F to -150F thermal cycles. This testing was performed in a thermal cycling chamber and not under vacuum as the initial 50 cycles were. After completion of 500 cycles, the tubes were re-examined for microcracks using 200X photomicrographs and X-ray analysis. Again, no microcracks were found in any specimens.

4.2 Atomic Oxygen Testing

The coated tubes were exposed in the Boeing built large-scale plasma atomic oxygen materials screening (PAMOS) test facility. Tube specimens were exposed in the PAMOS facility for 11-hours and then removed for evaluation. The specimens were then placed back in the chamber for an additional 22-hours of exposure. This would be an equivalent of 10 months at a 305-km orbit for Kapton-H. The tubes were placed parallel to the flow to minimize turbulence. Edges and interior surfaces of the tubes were left exposed. Anodized and SiO_2/Al coated specimens had minimal changes in optical and adhesion properties. All samples exhibited loss of Gr/Ep on the unprotected surfaces. Edges that were once flush with Al foil at the specimen ends, had recessed 1/16-in during the 33-hour exposure. The downstream edges degraded at similar rates as the upstream edges. Electroplated Ni exhibited total adhesion loss to the Gr/Ep. The SiO_x coating did prevent degradation of optical properties that took place on uncoated tubes but this coating did not improve adhesion of Ni to the composite. During atomic oxygen testing, one of the tubes was pre-punctured to produce a 0.015-in-diameter pin hole through the coating and foil to simulate potential damage caused by micrometeroids. Figure 2 shows a photomicrograph of a cut made through the pinhole after 22-hours of exposure in the PAMOS facility. The photo shows that 2 of the 12 plies were eroded away. Because the Al foil is inert to effects of atomic oxygen, the diameter of the pinhole through the foils remains constant. This limits the flux of atomic oxygen to the composite. Therefore, it is expected that while continued exposure would erode the Gr/Ep at a constant mass loss, because of increasing surface area, the rate of penetration would be expected to decrease. No structural testing was performed to determine the effect of erosion on mechanical properties of the tube section.

4.3 <u>Adhesion Testing</u>

Anodized and unanodized foil bonded to Gr/Ep specimens were subjected to 80 72-min, +250F to -250F thermal cycles to determine bond strengths before and after cycling. The Al foil had primer sprayed to the backside prior to bonding with 0.005-in-thick epoxy sheet adhesive to the Gr/Ep substrate. Testing of control specimens showed that while unanodized foil (backside of SiO_2 coated foil) was able to be peeled off the composite (average peel strength of 4-in-lb/in), the peel strength of anodized foil exceeded the tensile strength of the 0.002-in foil. Peel testing of the cycled specimens showed no decrease in peel strengths.

4.4. <u>Abrasion Resistance</u>

Abraiding the tubes by rubbing tubes with like coatings together caused the SiO_2/Al/Al foil tube to become darkened along the line of contact. There was no change in any of the anodized foil tubes even after being aggressively rubbed together.

5.0 SUMMARY AND CONCLUSIONS

Both phosphoric and chromic acid anodized Al foil proved to possess very good durability to LEO environment (no UV testing was performed) and also possessed excellent adhesion to Gr/Ep tubes. Chromic acid anodizing can be easily tailored to meet a variety of optical values by varying anodizing parameters. Phosphoric acid anodizing was not as versatile. Anodized Al foil possesses an additional benefit of being produced in large volume without a major R&D effort.

Sputtered SiO_2 and sputtered Al onto Al foil also possessed environmental stability similar to anodized foils although the bond strength to the composite was not as high. During abrasion testing the coating showed signs of optical degradation, but this would be a small percent of total area. A major disadvantage is the need to have large area vacuum coaters to deposit these coatings onto Al foil.

While electroplated Ni has the potential of providing conformal coatings to tubes and any irregular shaped surfaces, such as end fittings, adhesion loss during exposure to LEO environment needs to be improved. SiO_x coatings demonstrated the capability of improving the durability of the electroplated Ni and also improved the ability to tailor optical properties of the Ni.

No microcracks were found in any of the P75S/934 tubes after undergoing 50 94-min, +175F to -180F thermal cycles and 500 56-min, +120F to -150F themal cycles. The use of low angle off-axis plies required to meet stiffness requirements of the Space Station truss structure minimizes microcracking.

As part of this effort, four 8-ft-long Gr/Ep tubes, wrapped with chromic acid anodized 0.002-in Al foil were fabricated and latched together using a typical space-erectable structural end fitting. The foil surface was textured during tube fabrication to increase diffuse reflectance as shown in Figure 3. The hub and stud assembly shown in Figure 4 represents a corner of an interlocking network of the Gr/Ep struts and aluminum hubs that can easily be erected by a single astronaut without tools. Threaded aluminum inserts are bonded to the inside of each Gr/Ep tube. The latching mechanism is then screwed into the tube and a locking ring is tightened to hold the devise in place. The four tubes with the latching mechanism in place are shown in Figure 5. Strut attachment to the hub is accomplished by latching the strut and hub assemblies together and then sliding the locking collar foward and rotating to secure the strut to the hub. Figure 6 shows the four 8-ft-long tubes latched together.

Chromic acid anodized 0.002-in Al foil was selected as the best coating for protecting the tubes from LEO environment due to:

- o Environmental durability in LEO including retention of foil to Gr/Ep bond strength and retention of optical properties during LEO exposure.
- o Excellent adhesion to Gr/Ep.
- o Optical tailorability.
- o Ease of manufacture and low cost.
- o Excellent handling properties.

6.0 REFERENCES

1. Wernick, S. and Pinner, R., "Surface Treatment of Aluminum", Vol. 2, 1972, pp. 725.

Layup	Ex (Msi)	Ey (Msi)	Gxy (Msi)	αx (μin/in)	αy (μin/in)	F_L^{tu} (ksi)	F_T^{tu} (ksi)
$(0_2, \pm 10, 0_2)_s$	45	0.8	1.1	-0.8	17.5	105	2.4
$(0_2, \pm 15, 0_2)_s$	42.5	0.9	1.6	-1.0	17	100	2.5
$(0_2, \pm 20, 0_2)_s$	40	1.0	2.2	-1.1	14	95	2.7
$(0_2, \pm 30, 0_2)_s$	35	1.7	3.5	-1.2	8	80	4.0
(10, 0, -10, 0, 10, 0)s	42.5	0.8	1.4	-0.9	17	95	2.5
(20, 0, -20, 0, 20, 0)s	35.5	1.0	2.9	-1.3	13.5	80	2.7
(30,0, -30, 0, 30, 0)s	28	1.8	4.5	-1.3	7	65	4.0
$(\pm 10, 0_2, \mp 10)_s$	42.5	0.8	1.6	-1.0	16.5	102	3.0
$(\pm 20, 0_2, \mp 20)_s$	32	1.0	3.8	-1.6	12.5	74	4.4
$(\pm 30, 0_2, \mp 30)_s$	22	1.8	6.2	-1.6	6	52	8.6

Table 1. Composite Matrix Properties

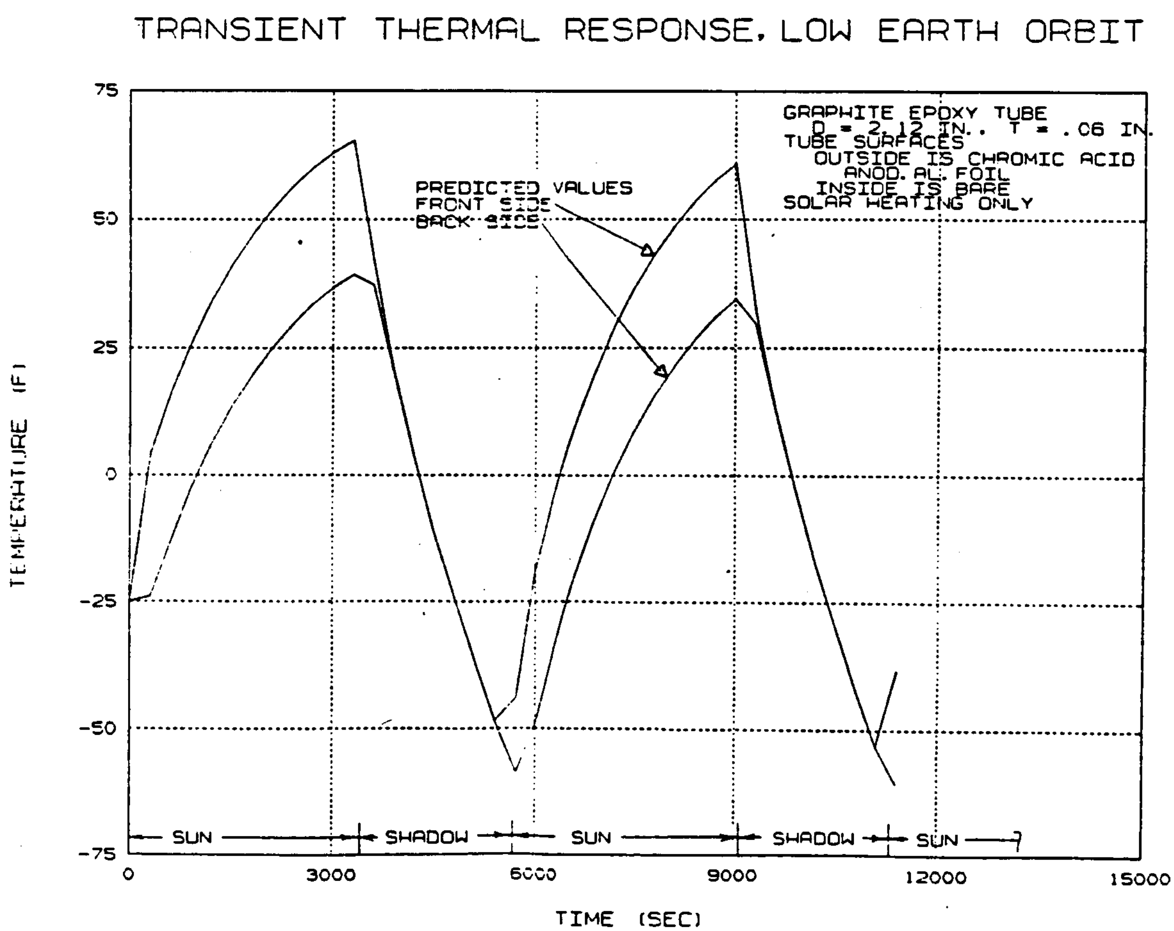
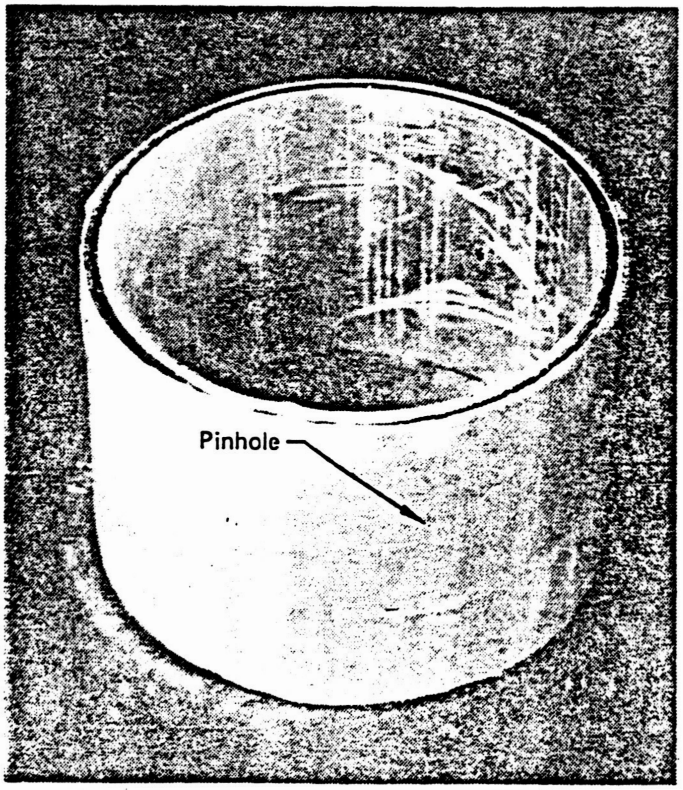
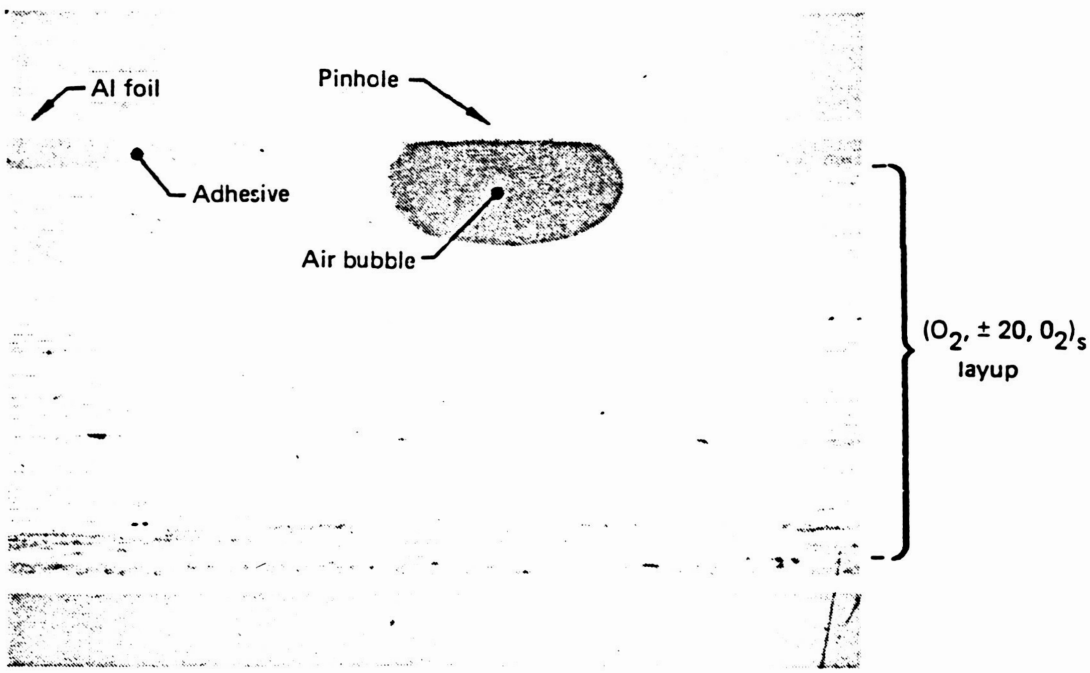


Figure 1. Predicted LEO Temperature Range

ORIGINAL PAGE IS
OF POOR QUALITY

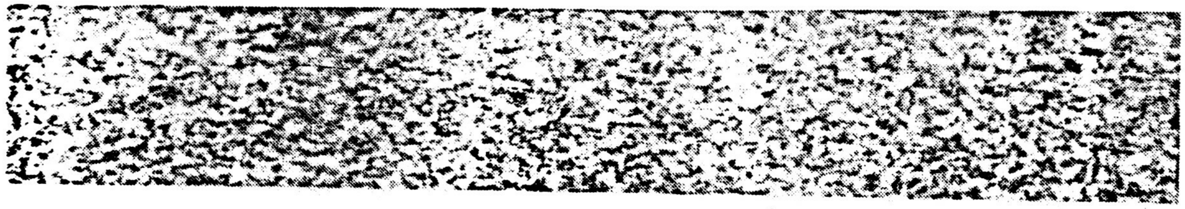


1.4X magnification



30X magnification

Figure 2. Effects of Pin Holes During Atomic Oxygen Exposure



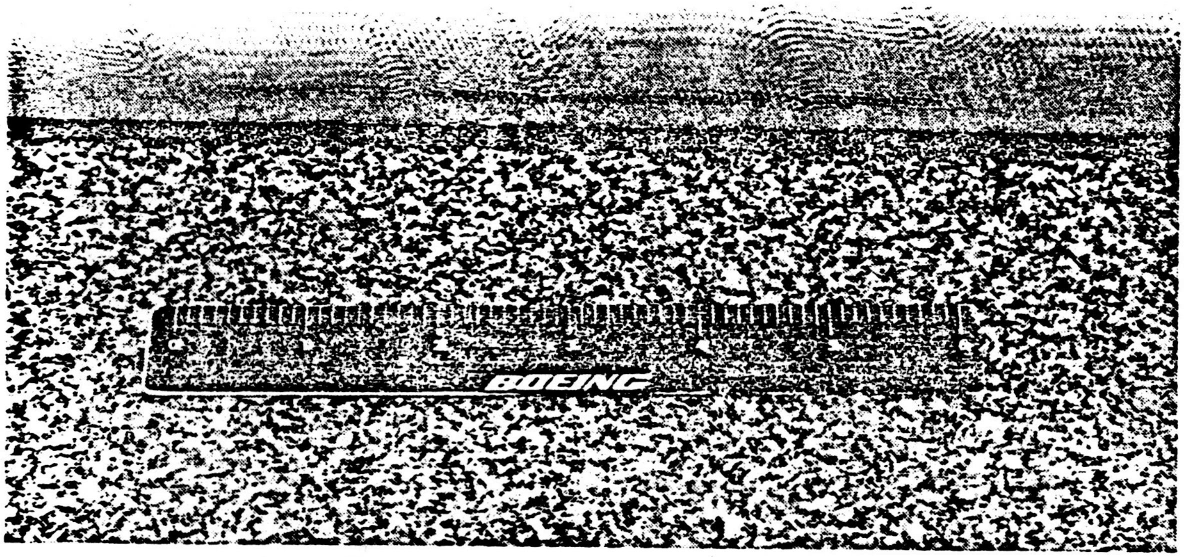


Figure 3. Chromic Acid Anodized Foil With Textured Surface

RIGINAL PAGE IS
OF POOR QU...

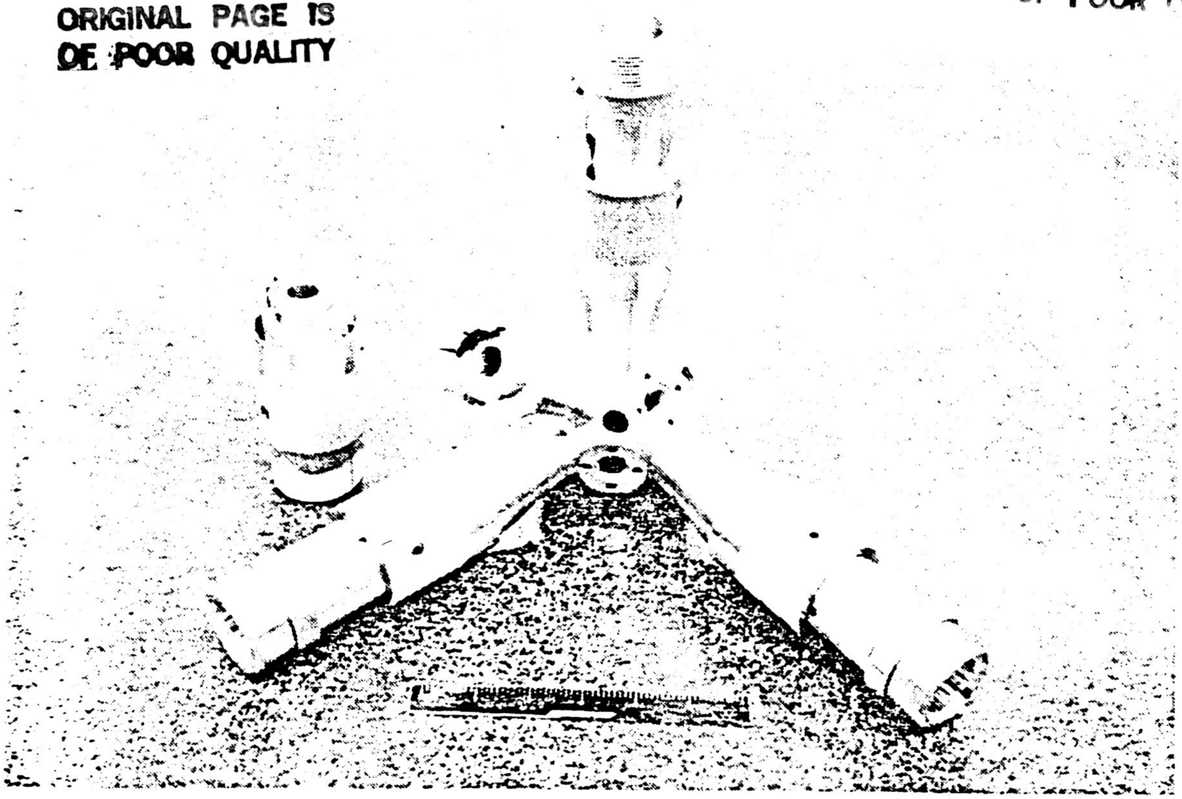


Figure 4. Space-Erectable End Fitting

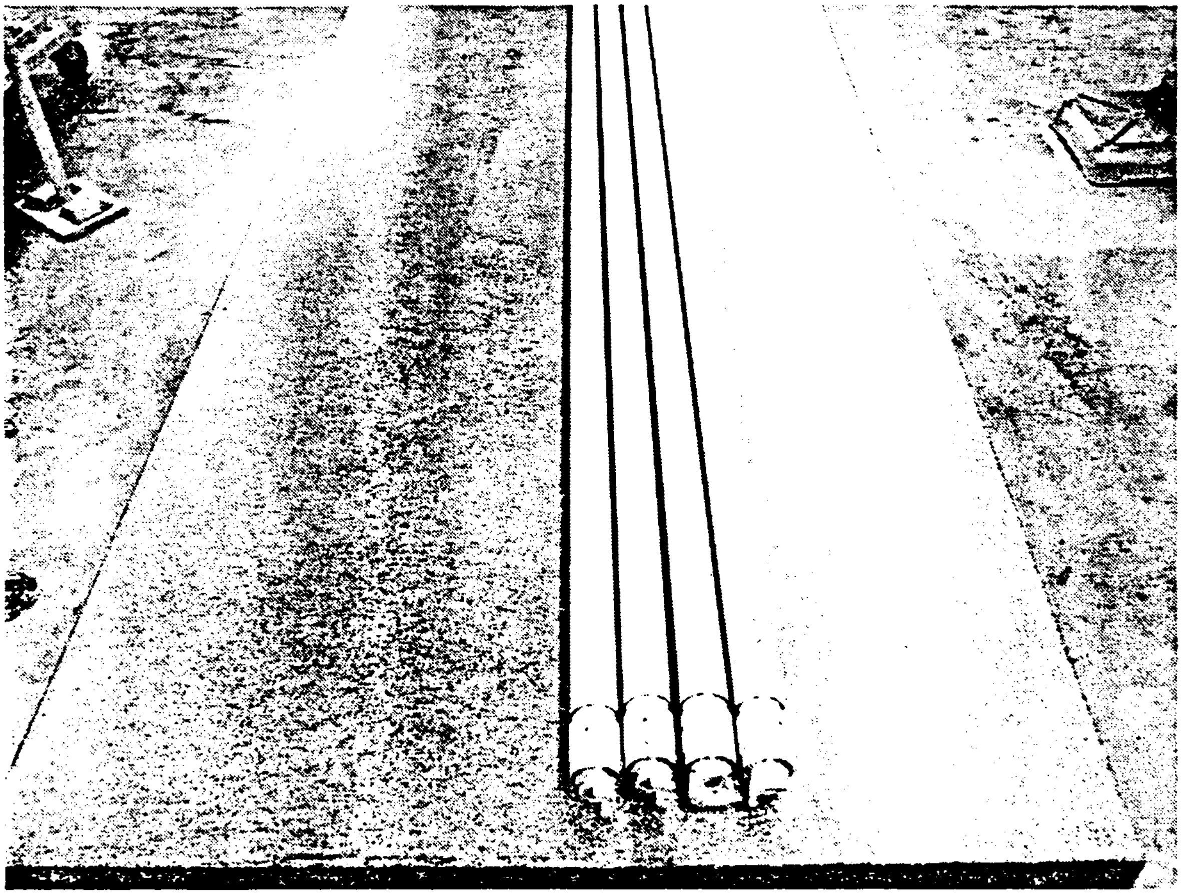


Figure 5. Four 8-ft-Long Gr/Ep Tubes Wrapped With Aluminum Foil

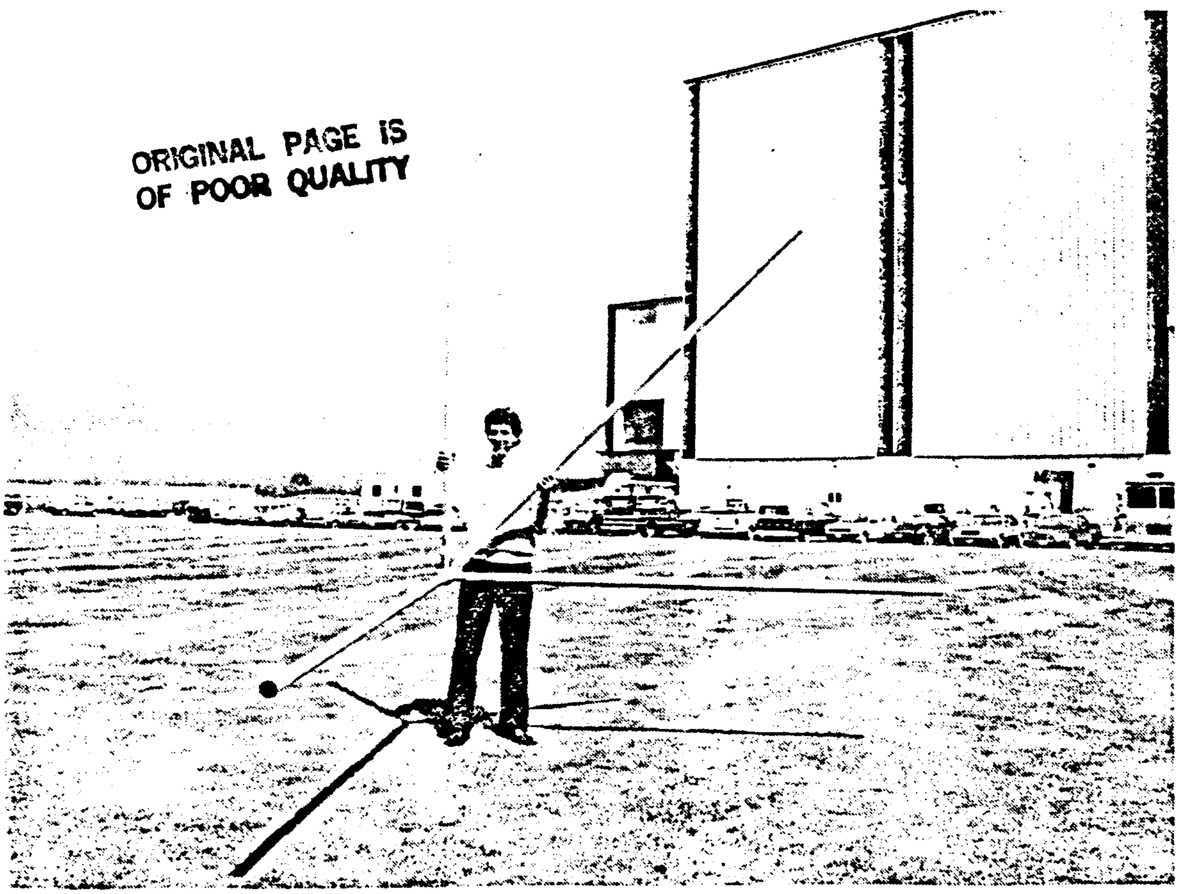


Figure 6. Four 8-ft-Long Gr/Ep Tubes Latched Together