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# NASA Technical Memorandum 79224

(NASA-TM-79224) INVESTIGATION INTO THE N80-13473 EFFECT OF PLASMA PRETREATMENT ON THE ADHESION OF PARYLENE TO VARIOUS SUBSTRATES (NASA) 20 p HC A02/NF A01 CSCL 11A Unclas G3/37 46306

# INVESTIGATION INTO THE EFFECT OF PLASMA PRETREATMENT ON THE ADHESION OF PARYLENE TO VARIOUS SUBSTRATES

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Prepared for the Seminar on Cleaning, Finishing and Coating Processes sponsored by the Society for the Advancement of Material and Process Engineering Los Angeles, California, February 5-6, 1979 INVESTIGATION INTO THE EFFECT OF PLASMA PRETREATMENT

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ON THE ADHESION OF PARYLENE TO VARIOUS SUBSTRATES\*

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#### ABSTRACT

An experimental effort has been undertaken to examine the effect of plasma pretreatment of various substrate materials coated with a polymer in the parylene family. This report describes the procedure and discusses initial data which indicate using plasmas of argon and oxygen to promote adhesion of parylene coatings upon many difficult-to-bond substrates. Substrates investigated were gold, nickel, kovar, teflon (FEP), kapton, silicon, tantalum, titanium, and tungsten. Without plasma treatment, 180° peel tests yield a few g/cm (oz/in) strengths. With dc plasma treatment in the deposition chamber, followed by coating, peel strengths are increased by one to two orders of magnitude.

## INTRODUCTION

When various types of materials are used for the protection of electronic circuits, it is important that a good bond be affected between the coating and the substrate to take full advantage of the coating's protective qualities. If the coating contains a substance that may interact with the substrate, or sufficient roughness exists, reasonable adhesion may result. In addition certain types of interface treatments such as chemical or mechanical may be employed to promote bonding. These approaches however are not always technically possible due to sensitivity of the substrate circuitry.

The purpose of this investigation was to examine the bond strengths possible using plasma surface treatment prior to

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coating with a unique vacuum polymerized material, parylene (polymonochloro-para-xylylene). This material has received attention as a coating in hybrid microelectronics (1 to 7) and is specified in Mil-I-46058, "Military Specification Insulating Compound, Electrical (for coating printed circuit assemblies)." Because of these wide applications, it was felt important to investigate the bondability of parylene to some of the difficult-to-bond substrates encountered in electronics. Although it is known that silane will enhance the bond strength of parylenes to many substrates (8) only nominal improvements are possible with the substrates of this study.

Today the use of plasma to prepare substrates for bonding is a widely accepted technology, particularly in the microelectronics industry. Plasma treatment of substrates is a process requiring no post treatment such as drying; the byproducts of the plasma process are typically nonreactive gases. Further, the process yields only small ash residue, whereas vast quantities of spent solvents and chemicals are left over after liquid cleaning. The polymerization of parylene is done at vacuum levels within an order of magnitude of typical plasma pressures,  $10^{-1}$  to  $10^{-2}$  torr. It seems only natural that these two process sequences should go together. While limited work dealing with plasma pretreatment of substrates prior to parylene deposition has been reported, bond strengths were not presented (9 and 10).

The usual mode of industrial plasma cleaning is by the use of rf induction. All the work reported here was done under dc conditions. The reason for this was twofold. The rf equipment can be very complicated and unless properly designed it radiates unwanted interference to neighboring equipment. In addition, there is the possibility that stray rf signals may be induced in the substrate. If there are sensitive electronics present such as CMOS devices, damage could result.

The primary objective of this investigation was to demonstrate a trend of improvement in the bond strength between an electronic substrate and its protective parylene overcoating. To obtain these data several substrates, plasma gases and electrode configurations were investigated. The bond strengths of the parylene coatings to various substrates were documented by peel tests.

#### APPARATUS AND PROCEDURE

#### The Parylene Process

In order to coat an object with parylene, a vacuum system is required which is capable of achieving  $10^{-2}$  to  $10^{-3}$ 

torr pressure. Figure 1 illustrates the NASA Lewis Research Center experimental facility used for this investigation. White parylene dimers whose molecular weights range from 208 to 344, resembling soap powder, are heated to a temperature in excess of 100° C (212° F) in vacuum. Under these conditions the dimer sublimes, travels through the pyrolizer zone at 700° C (1300° F) where it is cleaved to monomer after which it polymerizes upon cooling, forming a molecule of average molecular weight of 500 000. Depending on facility design and quantity of substrates in the deposition chamber, a portion of the original mass ends up coated all over the parts in the deposition chamber. The polymer products that pass through the deposition zone are trapped in the condenser to protect the pump. Because it is a gas, the parylene monomer has the ability to penetrate minute openings and cost uniformly high and low points while being pinhole free down to 100 angstrom thickness (11). In addition the coating process is a linking of molecules end to end with no cross linking involved and therefore the substrate is not stressed. There are three parylenes commercially available as shown in figure 2. The more common parylene C was used for this investigation.

#### Plasma Electrode Configuration

<u>Initial configuration</u>. - The entire deposition chamber (fig. 1) is made of pyrex glass and the top and bottom flanges are aluminum. The first attempt at dc plasma cleaning used high voltage applied across the flanges while bleeding plasma gas through a valve located on the chamber bottom center. At potentials of 500 to 1000 V dc it was possible to generate a plasma, at pressures above 0.1 torr. The discharge was very nonuniform with glow discharges moving around on the cathode flange. In addition, a large glow emanated from the gas inlet tube, resulting in a cone shaped discharge. These observations indicated that uniform plasma conditions would not be possible with this configuration.

<u>Second configuration</u>. - To eliminate the problems encountered with the inital configuration, the arrangement illustrated in figure 3 was assembled. A very uniform plasma was observed in the neighborhood of the cathode-parts area. Operating under plasma conditions described before, two samples were fabricated and peel tested. Using this set up it was possible to clean and coat approximately 413 cm<sup>2</sup> (64 in<sup>2</sup>) of area. Attempts to increase the treated area by utilizing more cathode/parts combinations in parallel resulted in only one group being at the proper plasma conditions.

Third configuration. - In order to increase cleanable area, a set up shown in figure 4 was fabricated and tested.

A majority of the data for this report was obtained from specimens prepared using this basic configuration. The parts to be cleaned and parylene coated were mounted to a blade assembly (fig. 4) which was rotated past the cathode electrode at a rate of 5 rpm. As many as eight electrically floating blades were available for mounting of parts. Two locations for the cathode were investigated. Figure 5 show these locations. This arrangement, using a 5 cm (2 in.) wide by 30 cm (12 in.) high cathode, enabled as much as  $3600 \text{ cm}^2$ (576 in<sup>2</sup>) to be treated in a single run on both sides.

With the cathode located at position "A", byproducts from cleaning must travel at least the distance across the chamber before being removed by the vacuum pump and cryocondenser. The possibility exists that blades may become cross contaminated by each other, particularly after plasma extinction. Cathode location "B" enables the contaminants to be swept out quickly. Cleaned surfaces are also surrounded with inert gas before ionization.

<u>Sample preparation</u>. - Sample substrates were obtained from various sources. Gold coated kovar hybrid lids were used as substrates. Nickel and kovar samples were obtained from rolls of foil. Teflon (FEP) and kapton (polyimide) films were from virgin rolls, directly from the manufacturer. The silicon samples were obtained by stripping the front contacts and antireflective coatings from solar cells. The history of the refractory metals is unknown as was that of the titanium foil. All samples were in the range of 0.125 to 0.5 mm (5 to 20 mils) thick. No solvent cleaning or other special substrate preparation was performed before plasma exposure.

The sample substrates were mounted either directly to the blade frame or smaller samples were mounted to a piece of polypropylene, which in turn was mounted to the blade frame. The samples of parts to be coated were electrically floated by isolating the rotating blade assembly. To investigate positional influence, one group of samples was mounted diagonally. Thus it was possible to rate the value of being nearer to the cathode. Another group of parts were mounted parallel to the cathode, at various distances away. The initial peel test samples were obtained by prying at the parylene-substrate interface. Considerable difficulty was experienced in grasping the parylene, especially with the well bonded samples. A modified method, using premounted tape, was used and is illustrated in figure 6.

<u>Plasma cleaning and coating</u>. - After the test substrates were mounted, the entire blade assembly was loaded into the deposition chamber of the coater. The top flange, complete with anode and cathode, was lowered into place. The vaporizer-plasma gas inlet tube assembly was charged with dimer and mounted to the facility. During the cleaning procedure, the discharge voltage, current and deposition chamber pressure were nominally 500 to 900 V, 90 to 50 mA and  $2\times10^{-1}$ torr. The cleaning procedure, for the most part, varied from 12.5 to 60 minutes in length. If more than one set of blades and parts were to be cleaned under the same set of conditions, the drive motor was turned on. After the pressure had been stabilized to 0.15 torr in the deposition zone, an open circuit voltage of up to 900 V dc was applied to initiate a discharge. Adjustments were made so that a constant 45 W was maintained in the discharge. After the plasma was completed, high voltage and plasma gas were turned off. The next step was to start the coating run by installing the vaporizer heater on the facility and setting it for the required temperature. The coating process then proceeded as described previously. After the coating was completed, the facility was returned to atmospheric pressure and the coated parts removed for testing.

<u>Testing</u>. - In order to compare the relative bonding strengths of parylene to different surfaces, the force required to peel the parylene layer from the surface was measured. This was done using a Mettler digital readout balance equipped with a hook on the underside of the pan. The specimen hung from the balance and force was applied as necessary to peel the parylene from the substrate. Specifically the sequence was as follows (refer to fig. 6):

a. Mask samples of the substrates to be studied, 0.64 cm (1/4 in.) along one edge with tape and mount in the coater.

b. Clean and coat with at least 25 microns (1 mil) thickness of parylene to minimize failure within the coating.

c. After coating, cut the substrate (or at least the parylene layer) into 2.5 cm (1 in.) wide strips for testing, with the masked edge at one end.

d. Peel the masking tape from the substrate, thereby beginning the peeling of the parylene from the substrate surface.

e. Tape the previously masked edge of the substrate to a plate hung from the underside of the balance.

f. Zero the balance.

g. Tape the free edge of the parylene layer to a second plate. This plate has a hook on its lower end for the addition of weight.

h. Pull gently downward on the lower plate by hand, watching the specimen and the balance output to establish that the weight of an empty beaker could be supported by the bond or else record the approximate force applied which resulted in failure.

i. If no failure occurred, a beaker was hung from the lower hook and slowly filled the water until the parylene peeled from the substrate.

In cases where the peeling force was less than 10 g (1/4 oz) the peel strength was considered zero. In cases where the bond strength was greater than the parylene, the maximum reading was recorded. These data are signified in figure 7 by vertical arrows. Based on the ultimate tensile strength of parylene films, a force of about 4500 g (10 lb) is necessary to fail a 2.5 cm (1 in.) wide parylene strip whose thickness is 25 microns (1 mil).

<u>Selected electrical substrates.</u> - To insure that this plasma cleaning technique would not harm microelectronics, two types of devices were investigated. A group of thin film nichrome resistors, mounted on hybrid packages, were processed along with the peel samples. Their resistances were measured before and after treatment. Some biomedical components and circuits were also exposed to plasma cleaning/ parylene coating cycles like the conditions used for the bond samples.

### RESULTS AND DISCUSSION

All tensile peel data are summarized in table I and, where possible, trends are presented in figures 7 and 8. The effect of plasma cleaning and parylene coating on the nichrome resistors is presented in table II. The effect of hot water exposure on some of the processed resistors is shown in table III.

The effect of several variables were investigated to demonstrate the advantages of promoting adhesion of parylene to selected surfaces. Two gas plasmas were evaluated to assess effects of a passive gas (argon) and a reactive gas (oxygen). The effects of position, total time of exposure, and the relation to the cathode were also of importance. The effect of handling, time between cleaning and coating, and sequence of cleaning although important, were not studied.

### Peel Samples

Table I summarizes the effect of changing various parameters on parylene peel strength. Basically it shows that the closer to the cathode that the substrates are located, the greater is the improvement in bond strength. Much higher bond strengths are possible when parts face the cathode, rather than the anode. Both of these results should be expected in a dc plasma since the release of volatile products generally occur in the neighborhood of the cathode. It was necessary however, for this work to examine locations other than right on the cathode.

If the substrates to be plasma treated were metallic, then the more efficient approach would be cathode mounting.

However, this work was to explore corditions typical of electronic circuits, that is, metal and nonmetals. Some of the effects of two different plasma gas types on treatment of teflon and kapton are shown in figure 7. The oxygen plasma produces parylene peel strengths in the order of 500 g/cm (1 lb/in) for both kapton and teflon substrates. The argon plasma has little effect on kapton.

Some of the peel data are not maximum values, that is, the parylene films failed before peeling occurred. Thus, some of the data of figure 7 represent lower limits on the parylene peel strength. Based on bulk properties, one would expect parylene film failure at approximately 4500 g/cm (10 lb/in). Further work with a large number of samples is required to generate final values.

The purpose of this investigation was to indicate potential trends. In order to expedite substrate processing four different treatments, that is, two gases at two different exposure times, were performed in the same coating run, to obtain data shown in figure 7. This means, for instance, the chance of contaminating all other sample groups while cleaning one group exists. This condition would not normally happen in a production run, since all parts would be cleaned with the same set of conditions. Reference to figure 8 illustrates this point. Mounted parts were rotated in and out of the plasma field such that the total plasma exposure was 12-1/2 minutes. Comparing oxygen plasma curves of figures 7 and 8 shows that surfaces up to 10 cm (4 in.) away from the cathode still receive sufficient plasma to be well bonded. The lower peel values of figure 8 against figure 7 could result from the fact that total treatment time differed by a factor of two.

Improvements in parylene bonding were found in almost every metal substrate studied using plasma cleaning with the exception of the refractories tungsten and tantalum. For example, the peel strengths of parylene bonded to kovar and gold increased by a factor greater than 100 when pretreated by an argon plasma (table I). Plasma cleaning of silicon increased the parylene bond strength by a factor of 23. Thus, a dc plasma may be utilized to successfully prepare several metals, semiconductors and polymers prior to parylene deposition. Such materials exhibit bond strengths at least an order of magnitude lower using conventional cleaning techniques.

#### Surface Topography Study

Whenever plasma etching of a surface takes place, the material removal may be accomplished by either physical sputtering of atoms or compounds, as well as chemical reactions which produce volatile products. Since this technique is intended for electronics which could possibly contain integrated circuit chips, sputter erosion could produce deleterious effects. A series of treated and untreated surfaces were examined by scanning electron microscopy at magnifications to 3000 X. No visible change was observed.

### Resistor Samples

Although peel tests are a good method of investigating process variables, a second method was utilized in this work. Since one of the principal uses of coatings in electronics is to protect the circuits from environment induced shorting, a series of thin film resistors were cleaned, coated, and immersed in hot water. Two events must occur during an electronic assembly coating schedule. The first and by far the most important step, is the complete removal of conductive surface contaminants. Water, which by itself is an excellant dielectric, will eventually diffuse through most coatings. If the interface under the coating contains conductive contaminants, the H2O will form an electrolyte of very high concentration. If, on the other hand, a majority of the surface contaminants are removed, thereby initiating a bondable surface, H2O cannot form a highly conductive comtinuous media in the coating-circuit interface.

To demonstrate bonding, as well as show the effect of plasma cleaning, a group of thin film resistors were exposed to plasma and coating treatment. Table II contains the post treatment resistance of various size devices as a result of argon or oxygen exposure before coating. Argon plasma treatment produces greater resistance increases than oxygen plasma processing of thin film resistors. Argon apparently removes conductive surface contaminants faster than oxygen.

Table III shows what happens to plasma treated, parylene coated resistors after immersion in tap water at temperatures from 43° to 82° C (110° to 180° F) and times up to one week. The magnitude of the changes is considered minor, especially since the presence of any conductive contaminants would cause the resistors to short. These times and temperatures are sufficient for transmission of 0.1 mg at 43° C (110° F) in one day to 35 mg at 82° C (180° F) in one week (12). Table III further indicates that the parylene matrix only transmitted nonconductive H<sub>2</sub>O, not the attendant conductive ions typically found in tap water.

### CONCLUSIONS

The following conclusions can be drawn from the data presented in this report:

1. A dc plasma may be utilized to prepare several different types of surfaces of electronic substrates for parylene coating, which are regarded as nonbondable. Substrates investigated were gold, nickel, kovar, teflon, kapton, and silicon. Without plasma treatment, 180° peel tests yield a few g/cm strengths. With dc plasma treatment in the deposition chamber followed by coating, peel strengths are increased by one to two orders of magnitude.

2. The substrates, with the exception of refractory metals, that were plasma cleaned exhibited coating adhesion superior to uncleaned.

3. If more parts are to be treated than can be arrayed within a few inches of the cathode, a carrousel arrangement is suggested. In addition, the cathode should be located near the low pressure (vacuum pump port) side of the chamber so that freshly desorbed contaminants may be removed without recontaminating another surface.

4. Plasma treated, parylene coated electrical substrates, such as nichrome resistors are protected from moist environments for periods of at least one week at a temperature up to  $82^{\circ}$  C (180° F).

5. A schedule of dc plasma treatment of 30 minutes at 45 W cleaned all nonrefractory substrates tested without damage to usually sensitive thin film nichrome resistors.

6. The combining of plasma cleaning with a coating process such as vacuum polymerization is efficient because individual equipment requirements (vacuum of  $10^{-2}$  to  $10^{-1}$ torr) are compatible.

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Pary iene run number	Substrate Deterial	F1	Relation of sample to cathode	Length of esponsore, osa	Average discharge power,	180° Pull etrength, ges	Coment s
	letion	Ar / None	2.5 cm away	20	15	230/145	
	Silicon	Ar /None		ю	-?	232710	
	A1203	A.				250	
	Au	A1				250	
	800					1900	
•	<b>H</b> 4	None / Ar	7.5 cm away	30		335/10)	(.),(.)
	Kover	hone/Ar	1.5 cm away	10		3/480	(.),(.)
	A.,	None / Ar	7.5	ы	•3	37355	(
c	Berosilicate Glass	None /Ar	7.5	30 	Ĩ	557/205	(a),(s) + Another plasms ciested sample could not be peeled
	T.					93/93	
	т.			i		27/750+	
	-					1680/10)	
	•					40/205	<ul> <li>Probably not high density enough plasma or contamination</li> </ul>
	4		1 +			100/()	

#### TABLE 1. - Parylene Peet Strength Summery

Parylene failed.

"Befer to figure 5, cathode position A.

Substrates were stationary during plasma treatment

221	Bubetrete Gateriei	F1.0000	Bristian of Baspie to Cathode	Length of capacete.	Average discharge poet.	185° 5.11	Company a	
•	tel tun .nd Rapt en	Ar and 07	Peretiet to et vertes.	ъ	••	See 11g. 7	(	
	101 1	*; ***	Wert scal to at variant distant	л	•"	14. I		
•	Lap1 en	**	West	46/1 x	**	1244/853	(er.(e),(e)	
	Repton	•7		69/29		1026/8 10		
	lefion	**				• 32/234		
	let ion	•,		-0.70				
	Sapten	-				> > 1 + + + +		
	tes ion	-		•••••		10		
•	Repton	*	Wert	60.15	**	1300/1022	ter, (a), (s)	
	Esptan	w7						
	letion	**		Sec. 15		100/20-		
	Teliun	0,		60/JU			· Loss aperianas (	•
	Repton	Bune				<b>*</b>	Ļ	

#### 14812 1. - Continued. Parylene Peel Stremth B

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Peoplem failed. Treated with All'S scale scattom to provide adversion, Ibefor to figure 2, cathomic position 8.

Pary lower run number	Substrate Motorial	P1.0000 800	Relation of sample to cathode	Length of esposure, Bin	Average diacharge peetr, U	180° Pull strength, gns	Comont s
7 (con't)	4	A.	Wertical, 5 cm away	60/15	43	348/148	(w),(s) 
	4	07	Nortical. 5 cm away	40/20	45	408/828	
	~	( <b>a</b> )	0000011- 0100000			130	
÷	Ta	A.	Vertical. 5 cm away	00/15	45	217/506(e)	
	T.	υ,		-0/20		680(e)/270	
	Ti	A.		+0/15		(e)/628(e)	
	Ti	U2		60/20		(•)/(•)	
	T1	(g)				76	•
6	Kapton	•2	Vertical. 5 cm away	10/60	• 3	736/1290	(*),(*),(*)
	Kapion	Hone	Oppnaile plasme area	•••		177	(e),(i),(e)
	Tellon	•2	Vertical, 5 co away	10/60	43	557/1210	(.),(.)
	Tellon	Hong	Opposite plasse area			348(+)	(=),(1)

# TABLE 1. - Continued. Parylene Peel Strength Summery

•Peryless failed. •Befer to figure 5, cothede position A.

SAlcohol weah. Substrates were stationary during plasma treatment.

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Pary lene run number	Substrate moterial	Plasma gas	Relation of sample to cathode	Length of esposure, ein	Average discharge puwer, U	180° Pull strength, ges	Coment s
c	Rover, Hi. Hickrow	₽2	Vertical, 5 cm away	10/60	+3	All three (e)	(.),(.)
	Kover, Hi, Nichrome	None	Opposite plosmo area			Kovar (e) Ní, 900(e) Níchrome 1400(e)	(.),(1)
•	Kapton (16 samples)	Ar	Vertical, 5 cm away	8U(H)	43	585/477(K)	(n), (j)
	Tefion (16 samples)	AT	Vertical, 5 cm away	80(H)	43	108/194(K)	(#),(j)
	Ni (4 semples)	AT	Vertical, 5 cm away	BU(N)	45	1630(e)/1478(k)(e)	(n),(j)
	Ni (6 samples)	Nune	Opposite plasme area			557	(R),(j)
	To (16 samples)	A7	Vertical, 5 cm away	BU(N)	•	112/171	(n),(j)
	Ta	None	Opposite plasma area			105	(m),(j)

#### Table 1. - concluded. Parylene feel Strength Summery

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\*Parylene failed.

Refer to figure 5, cathode position A.

Refer to figure 5, cathode position B.

"Substrates were stationary during plasma treatment.

Mparts actually in the plasma a total of 12.5 minutes.

Kupper value from leading edge samples, lower trom trailing edge.

RSubstrates were stationary during plasma "reatment.

# TABLE II. - Change of Value of Thin Film Nichrome Hybrid Resistors Treated with Plasma and Parylene Coated

Plasma treatment	Percent resistance increase (for nominal values listed)				
	100	kΩ	71 K	1 <b>00</b> Ω	
	1	2			
Ar, 15 min	0.13	0.15	0.14	0.11	
Ar, 60 min	.24	. 21	. 16	.11	
02, 20 min	.07	.07	.07	.11	
02, 40 min	.18	. 19	.15	.11	

(*)					
Test condition <sup>®</sup>	Percent total resistance change after				
in H <sub>2</sub> O	exposure (for nominal values listed)				
	63 kΩ	20 kΩ	100 <u>Ω</u>		
24 hr at 43° C (110° F)	+0.04	0	0		
24 hr at 66° C (150° F)	0	0	+.02		
24 hr at 77° C (170° F)	01	+.04	+.01		

# Table III. - Effect of Hot Water Exposure on Adhesion Promoted and Coated Nichrome Resistors

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	<b>N</b> 1
•	••

Test condition <sup>®</sup>	Percent total resistance change after				
in H <sub>2</sub>	exposure (for nominal values listed)				
	102 kΩ	69 kc. <sup>c</sup>	20 kΩ	900 £	
24 hr at 43° C (110° F)	+0.03	+0.02	+0.05	-0.11	
24 hr at 66° C (150° F)	01	01	+.05	11	
24 hr at 77° C (170° F)	03	07	+.01	11	

#### (c)

Test condition <sup>d</sup> in H <sub>2</sub> O	Percent total resistance change after exposure (for nominal values listed)					
	120 ks	66 12	21 kΩ	a 006		
7 days at 55° C $(130^{\circ} F)$ 7 days at 70° C $(160^{\circ} F)$ 7 days at 82° C $(180^{\circ} F)$	+0.02 +.01 06	-0.09 +.06 06	0 +.01 0	-0.22 11 +.11		

(d)

Test condition <sup>e</sup> in H <sub>2</sub> O	Percent total resistance change after exposure (for nominal values listed)			
	20 kΩC	900 P		
7 days at 55° C $(130^{\circ}$ F) 7 days at 70° C $(160^{\circ}$ F) 7 days at 82° C $(180^{\circ}$ F)	+0.07 +.10 01	0 0 Sample damaged during handling		

<sup>a</sup>After argon plasma for 15 minutes followed 24 hr later with 38 mm of parylene coating. <sup>b</sup>After argon plasma for 60 minutes followed 24 hr later with 38 mm of parylene coating. <sup>c</sup>Average of two resistors. dAfter oxygen plasma for 20 minutes, followed 2 hours later

with 1.5 mils of parylene coating. <sup>e</sup>After oxygen plasma for 40 minutes followed 1 hr later with 1.5 mils of parylene coating. ;







(a) PARYLENE N.

n > 5000

(D) PARYLENE C.

n > 5000 CI

(C) PARYLENE D.

Figure 2. - Three commercially available parylenes.

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Figure 4. - Deposition chamber cross section.

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Figure 5. - Deposition chamber, top view.







(b) AFTER PARYLENE COATING.

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(c) TRIMMED, READY FOR TENSILE PEEL TEST.

Figure 6. - Steps in preparation of adhesive peel sample.





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ing O<sub>2</sub> plasma treatment. Samples ro-tated during plasma exposure. Total plasma treatment time, 12 J/2 minutes.