REPORT NO. P74-289

Final Report
Contract NAS8-29940

DEVELOPMENT FOR APPLICATION OF PARYLENE COATINGS

(NASA-CR-120536) DEVELOPMENT FOR
APPLICATION OF PARYLENE COATINGS Final
Report, 1 Jul. 1973 - 15 Jun. 1974
(Hughes Aircraft Co.)

N75-12208

Unclas
04251

REPRODUCED BY
U.S. DEPARTMENT OF COMMERCE
NATIONAL TECHNICAL
INFORMATION SERVICE
SPRINGFIELD, VA 22161

JUNE 1974

PRICES SUBJECT TO CHANGE

AEROSPACE GROUPS

HUGHES

HUGHES AIRCRAFT COMPANY
CULVER CITY, CALIFORNIA

HUGHES AIRCRAFT COMPANY

DEVELOPMENT FOR APPLICATION OF PARYLENE COATINGS

FINAL REPORT
NAS 8-29940

June 1974

F. W. Oberin

Materials Applications Department
Materials and Processes Laboratory

Aerospace Groups
Hughes Aircraft Company
Culver City, California

Prepared for
National Aeronautics & Space Administration
George C. Marshall Space Flight Center
Marshall Space Flight Center, Alabama 35812
Attention: M. J. Berkebile, Project Monitor S&E-QUAL-QT

Approved: G L Robinson
G. L. Robinson, Manager
Materials Applications Department

FOREWORD

This report was prepared by Hughes Aircraft Company, Culver City, California 90230, under NASA Contract NAS8-29940, "Development for Application of Parylene Coatings". The work was sponsored and administered by the George C. Marshall Space Flight Center, Marshall Space Flight Center, Alabama 35812, with M. J. Berkebile serving as the Contract Monitor.

The program manager and principal investigator on the program was F. W. Oberin, Head of the Dielectric Packaging Group. Professional assistance was received from Drs. T. C. Hall and D. A. Dimeo, R. Barch, R. Holbrook, F. Keister, J. Takayesu and L. Holness. Valuable assistance was also provided by R. Brown.

This report covers work conducted between July 1, 1973 and 15 June 1974.

ABSTRACT

The objective of this program was to develop a practical method of applying a parylene* (paraxylene) coating to hybrid microcircuit devices for the purpose of preventing circuit malfunctions due to foreign debris existing prior to package sealing or formed upon exposure to the service environment. A secondary objective, but equally important, was to determine if there were any deleterious effects on typical active and passive devices found in hybrid microcircuits due to the coating or any coating pertreatments (if used).

The development effort and study produced a practical coating and inspection technique, and determined that parylene and an associated primer (if used) had generally little or no adverse effect on the active and passive elements of typical hybrid microcircuits. Exceptions to this statement relate to high frequency circuitry, low resistivity thick film resistors, and highly sensitive PIN diodes. Some advantages are increased dielectric strength and increased interconnection bond strength.

Coating service life in nitrogen or nitrogen with low levels of oxygen (up to 3%) as a contaminant are in excess of 1000 hours at 150°C and can be projected to be in excess of 5000 hours at 125°C.

---

*Product of Union Carbide Corporation

CONTENTS (Continued)

LIST OF ILLUSTRATIONS

LIST OF ILLUSTRATIONS (Continued

LIST OF TABLES

LIST OF TABLES (Continued)

INTRODUCTION

One of the continuing problems in the hybrid microelectronic field is the incorporation of unwanted debris within the sealed package or the generation of debris during shock and vibration. Good inspection practices tend to reduce initial debris problems but subsequent creation of debris (i.e., detachment of interconnect wire "pigtails") is difficult to avoid and generally occurs during service use.

The purpose of this program was to supplement an earlier study which evaluated the use of parylene* (paraxylylene) coatings as a method of anchoring loose or potentially loose debris. The effort undertaken involved 1) a determination of a minimum serviceable coating thickness (intended to minimize coating cost), 2) development of a method of applying the coating prior to closing the package which would preserve the sealability of the package and the solderability of the leads, 3) determination of the expected life of the coating at elevated temperature and the coatings effects (if any) on the passive and active elements in the package.

The approach used to determine the above consisted of the selection and installation of active and passive devices into various types of packages for subsequent treatment and testing.

---

*Product of Union Carbide Corporation

SUMMARY AND CONCLUSIONS

The following final report covers the work done on NASA Contract NAS 8-29940 regarding the "Development for Application of Parylene Coatings." The objective of this study was to develop procedures for applying a minimum thickness parylene coating to hybrid microcircuits for the purpose of minimizing foreign debris problems; to study the effects of the coating on typical passive and active devices within the package under various environmental conditions as a function of time; and to determine the expected service life of the coating.

Results of the study indicate that in general a parylene film 0.0001 inch or more in thickness is sufficient to hold debris (or prevent formation of debris) in place under severe shock, vibration, and temperature cycling. Further, such a film can be applied in a practical manner prior to sealing the package and that inspection for improperly coated parts can be accomplished readily through the use of fluoroescent coating additives and ultraviolet illumination.

No adverse effects were noted on stress sensitive interconnection wires during temperature cycling. The coating improved joint strength initially and this improvement was retained during subsequent temperature cycling exposure.

The coating does not appear to adversely effect any of the semiconductor devices tested. These included a PIN diode, NPN and PNP transistors, a C-MOS, an operational amplifier, and an N-channel MOSFET. All of these devices were exposed to elevated temperature conditions (some under reverse bias) for 1000 hours. In addition, the use of a silane primer has no apparent effect, except in the case of the PIN diode where an increase in initial leakage current appears to have occurred.

The effect on passive devices were somewhat mixed. The coating appears to improve dielectric breakdown initially and this improvement is retained to varying degrees after exposure to elevated temperature. No effect was noted on insulation resistance or on low frequency (up to 100 megahertz) circuits. Coated high frequency curcuits (2 to 12 gigahertz) have greater line loss than their uncoated counterparts with the greatest difference occurring at the highest frequency. There was no apparent initial effect of the coating on film resistors, however, after thermal exposure the effects varied from virtually no effect on thin film resistors to a significant effect on unglazed, low resistivity (ohms/square) thick film resistors. The coatings effect on unglazed higher resistivity film resistors was noticeably less. The effect on glazed thick film resistors was similar to the effect on their unglazed counterparts except that the effect was of a more minor degree. Oxygen appears to have a stabilizing influence on coated thick film resistors although oxygen has a degrading effect on the mechanical properties of the film.

No significant thermal effects were noted which could have an adverse effect on interconnection wires or beam lead diodes and transistors.

Coating service life in nitrogen at 150°C, and in oxygen contaminated (up to several percent) nitrogen at 150°C is in excess of 1000 hours for holding debris in place. At 125°C the service life should be in excess of 5000 hours. In air the service life can be expected to not exceed 300 hours at 150°C or 1500 hours at 125°C.

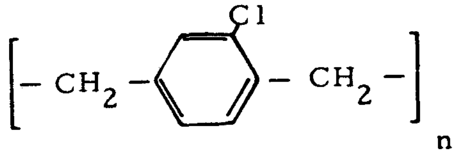
In conclusion, parylene appears to provide a suitable coating material for preventing damage or the formation of debris under typical hybrid operating conditions. The coating can be applied in a practical manner and the quality of the coating can be easily inspected. The effects on passive and active devices are negligible or tolerable except for low resistivity thick film resistors. This exception may be peculiar to the specific inks used and may not be true for other ink types of this resistivity level. Finally any of the effects noted in this study were generally determined during aging at 150°C up to 1000 hours. The effects at 125°C can be expected to be noticeably less.

TECHNICAL DISCUSSION AND EXPERIMENTAL RESULTS

PARYLENE AND ITS DEPOSITION

Parylene* is the generic name of a polymer series developed by Union Carbide Corporation. The polymer is a linear polymer of the p-xylylene series, formed by vapor deposition of xylylene radicals followed by chain growth in situ. As a vapor deposited material, it is capable of being formed in very thin pinhole-free films, is applied very evenly, and does not suffer from such flow problems as filleting or exposure of sharp points, which can be a problem with brushed, dipped, or sprayed coatings.

Two types of parylene are commonly used: parylene N is the basic polymer, poly(p-xylylene), while parylene C, poly(chloro-p-xylylene), has a chlorine atom substituted on each ring and thus has the formula



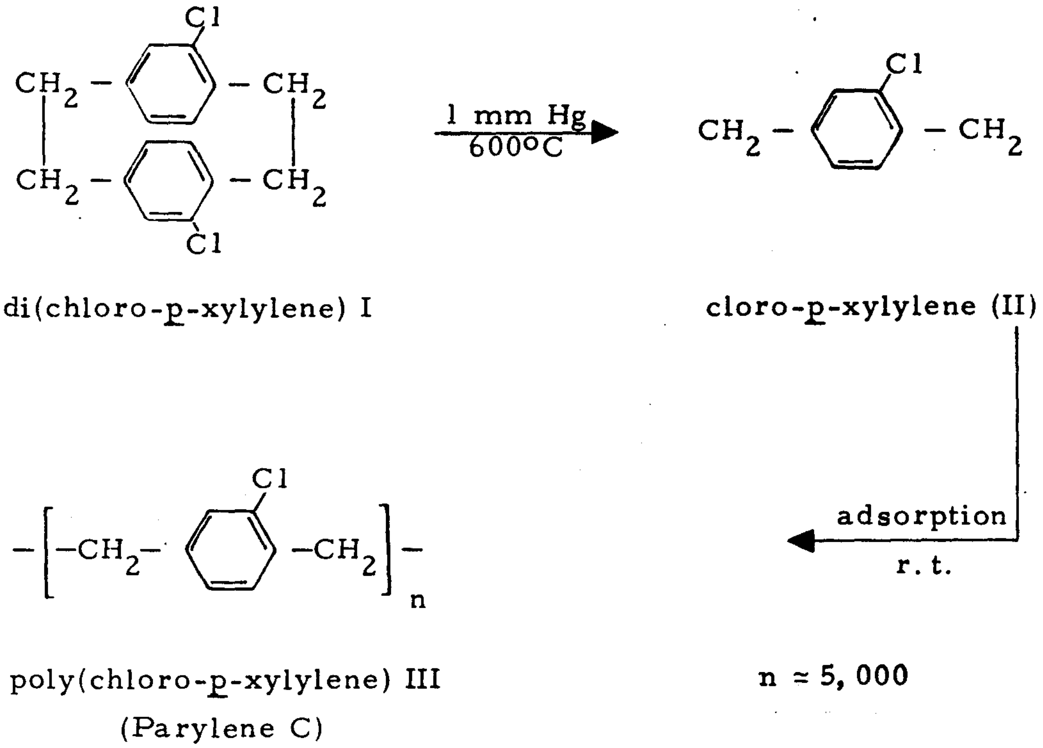
, where n is about 5, 000

Di(chloro-p-xylylene) is the starting material for the formulation of a parylene C film, This material is sublimed in vacuum, passing through a pyrolysis chamber. Thermal cleavage occurs at the methylene-methylene bridge, giving a nearly quantitative yield of cloro-p-xylylene, a diradical. These vapors pass into a deposition chamber at ambient temperature; deposition

---

*Product of Union Carbide Corporation

occurs on all surfaces encountered, with simultaneous free-radical polymerization. The mechanism for formation of parylene C is shown below:



di(chloro-p-xylylene) I

cloro-p-xylylene (II)

poly(chloro-p-xylylene) III
(Parylene C)

$n \approx 5,000$

Since the coating deposits from a vapor state and immediately forms a polymerized solid, no fluid flow occurs and therefore sharp points and edges remain coated and filleting does not occur. Further, since the vapor permeates all accessible areas, even cracks and crevices become coated, which although generally a desirable characteristic, makes it mandatory that good masking techniques be developed for areas intended to remain uncoated.

The coating of a substrate requires the use of an apparatus, consisting of a vacuum pump, trap, sublimation chamber, pyrolysis chamber, and polymerization chamber, as well as associated heaters, gauges, etc. A typical apparatus is shown in Figure 1.

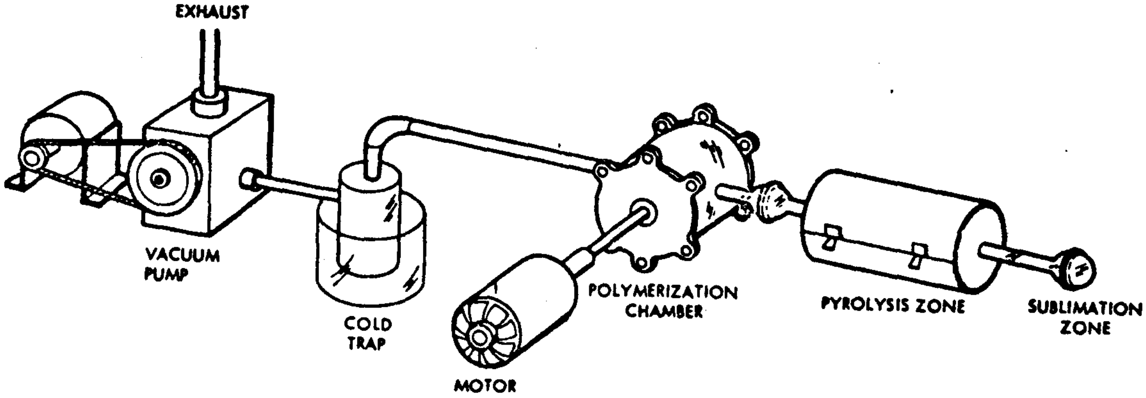


Figure 1. Apparatus for the vapor phase thermal polymerization of parylene films.

The film produced by the above process is non-glossy and transparent or nearly so. A number of physical properties have been measured by Union Carbide Corporation and are shown in Table I.

MINIMUM SERVICEABLE COATING THICKNESS

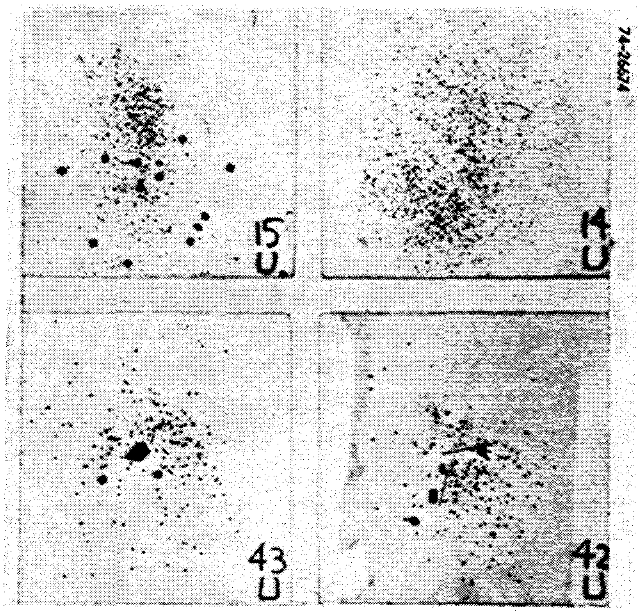
Debris Holding Ability

To determine the minimum serviceable coating thickness, debris specimens were fabricated using substrates which represent typical microelectronic hybrids. Several thicknesses of coating were studied along with different types and sizes of debris. In addition, the effect of primer and the effect of different nitrogen/oxygen ratios were also studied.

Figure 2 depicts the general appearance of the specimens with debris in place after the coating was applied and the specimen thermally aged. These specimens (or portions of them) were tested before and after exposure at 150°C. Testing consisted of exposure to ten shocks per MIL-STD-883

TABLE I. TYPICAL PHYSICAL, MECHANICAL AND ELECTRICAL PROPERTIES OF PARYLENE C, DATA PUBLISHED BY UNION CARBIDE CORPORATION

Properties	Parylene C
Secant Modulus, 1 percent strain, psi	400, 000
Tensile Strength, psi	13, 000
Elongation to break, percent	200
Density, gm/cm^3	1.289
Index of Refraction, n_D	1.629
T_m (melting) °C	280 - 300
T_g (glass transition) °C	80 - 100
T_5 (where modulus = 100, 000) °C	125
T_4 (where modulus = 10, 000) °C	250
Dielectric Strength, ST volts/ mi	3700
Corrected to 1/8 inch	520
Dielectric Strength, step by step, volts/mil	1200
Corrected to 1/8 inch	410
Volume Resistivity, at 23°C, ohms	8.8×10^{16}
Surface Resistivity, 23°C,	
50 percent RH, ohms	10^{14}
90 percent RH, ohms	7×10^{11}
Dielectric Constant	
60 cps	3.1
1 K cps	3.1
100 K cps	3.0
1000 K cps	2.9
Dissipation Factor	
60 Hz	0.02
1 KHz	0.0195
100 KHz	0.0156
1000 KHz	0.0128



0.001 and 0.004 inch parylene coated debris specimens thermally aged in 99.9% nitrogen at 150°C

0.001 and 0.004 inch parylene coated debris specimens thermally aged in 98.5% nitrogen 1.5% oxygen at 150°C

Figure 2. Typical debris specimens.

Method 2002 Condition B (1500 g's) and vibration per MIL-STD-883 Method 2007 Condition A at 20 g's between 20 and 2000 hertz for 12 sweeps with a duration of four minutes each sweep in each of two axis (within the plane of the specimen and perpendicular to the plane of the specimen). Table II indicates that sizes and weights of the debris. After thermal

TABLE II - DEBRIS DESCRIPTION

	Size	Weight
Tungsten	0.007 to 0.010 inch particles	0.0003 to 0.0006 grams
Gold wire	1/4 x 0.001 inch diameter	0.001 grams/inch
Lead shot	0.075 inch diameter	0.05 grams
Silicon chips	0.007 x 0.02 x 0.02 inches minimum dimensions.	0.00025 grams (minimum)

aging but prior to final shock testing, the parts were exposed to temperature cycling per MIL-STD-883 Method 1010 Condition C (Five cycles between -65 and 150°C) with no apparent effect.

Results of the testing are shown in Table III. Generally performance was satisfactory for coatings 0.0001 inch thick or greater. An exception to this relates to the lead shot particles which survived only part of the testing. The fact, however, that these particles were much larger than typical debris expected in hybrid packages and were of a difficult to hold shape, indicates a positive rather than negative performance of the coating. No significant difference in the performance of primed vs unprimed surfaces was noted. The partial failure or loss of silicon chips in the 0.0001 inch coated specimen consisted of losing approximately 20 percent of the chips during the shock testing. Loss occurred during the initial shock or during one of the 10 successive shocks prior to thermal aging. Other specimens of the same thickness gave similar results when shock tested after thermal aging. Parts exposed to thermal aging in nitrogen with 1.5 percent oxygen as a contaminant gave identical performance in all respects to parts aged in relatively pure nitrogen (99.99 percent N_2). The selection of 1.5 percent oxygen was based upon the intent to reproduce conditions that might be present in typical assembly and sealing facilities. The exposure method used during the test incorporated a much larger quantity of gas and thus the 1.5 percent level of oxygen is probably equivalent to an approximate 3 percent level of oxygen in a typical hybrid package. Some yellowing of the samples containing the oxygen contaminant was observed. Other samples tested early in the program which were exposed to much higher levels of oxygen during aging, exhibited a noticeably greater degree of yellowing. Since the oxygen content ($\approx$7 to 18 percent) was higher than intended, the results

TABLE III – RESULTS OF SHOCK AND VIBRATION TESTING OF DEBRIS SPECIMENS

Coating Thickness (inches)	Debris Type	Shock	Vibration	Both	Shock (After Thermal Aging 1000 Hours at 125°C)		Vibration (After Thermal Aging 1000 Hours at 125°C)		Both (After Thermal Aging 1000 Hours at 125°C)	
		(Prior to Thermal Aging)			N_2	N_2O_2	N_2	N_2O_2	N_2	N_2O_2
0.0001	Lead shot	F	—	—	—	—	—	—	—	—
	Tangsten	P	P	P	P	P	P	P	P	P
	Silicon chips	PF	P	PF	PF	PF	P	P	PF	PF
	Gold wire	P	P	P	P	P	P	P	P	P
0.00015	Lead shot	F	—	—	—	—	—	—	—	—
	Tungsten	P	P	P	P	P	P	P	P	P
	Silicon chips	P	P	P	P	P	P	P	P	P
	Gold wire	P	P	P	P	P	P	P	P	P
0.0002	Lead shot	F	—	—	—	—	—	—	—	—
	Tungsten	P	P	P	P	P	P	P	P	P
	Silicon chips	P	P	P	P	P	P	P	P	P
	Gold wire	P	P	P	P	P	P	P	P	P
0.0004	Lead shot	PF*	—	—	—	—	—	—	—	—
	Tungsten	P	P	P	P	P	P	P	P	P
	Silicon chips	P	P	P	P	P	P	P	P	P
	Gold wire	P	P	P	P	P	P	P	P	P

*1 out of 3 failed after six shocks.

P – Passed, PF – Partial Failure, F – Failed

N_2 – 99.9% Nitrogen

N_2O_2 – 98.5% N_2/1.5% O_2

were discounted and the test repeated. However these earlier tests do indicate that performance under shock and vibration were encouraging although somewhat inferior to that demonstrated with lower levels of oxygen.

Thermal/Mechanical Influence of the Coating.

Compatibility of the parylene coating with stress sensitive wire bonds was determined by exposing representative specimens to temperature cycling with pull strength tests performed prior to and after cycling.

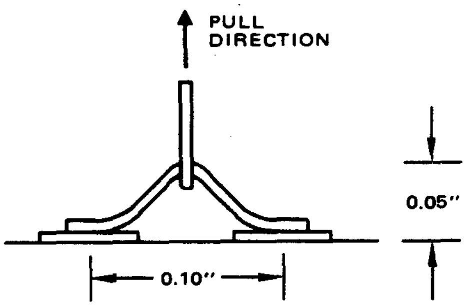
Coated and uncoated samples containing 0.001 inch diameter aluminum and 0.002 inch diameter gold wire bonds were subjected to 160 temperature cycles between -50°C and +125°C plus an additional 15 cycles between -65°C and +150°C. Dwell time at each extreme was 10 minutes with a 5 minute dwell at room temperature while transiting between these temperature extremes. Exposure rate to each temperature extreme after the 5 minute dwell at room temperature consisted of injecting the parts into the two extreme temperature environments in less than 5 seconds. Primer was used on some coated specimens. Coating thickness was 0.00024 inch.

Results indicate some degradation due to temperature cycling with both coated and uncoated specimens. The degradation approximates 5 to 15 percent of the initial strength. Coated aluminum wires retained a decided strength advantage over uncoated aluminum wires of approximately 40 percent. Coated gold wires retained a strength advantage over uncoated gold wires of approxmately 15 to 25 percent. The effect traceable to the use of primer appears to offer little advantage (if any) over coating without primer.

Table IV presents the pull test data and provides a pictorial view of the specimen and test technique. Since the specimen involves two wire bonds and are pulled simultaneously the values are controlled by the weaker bond.

TABLE IV. AVERAGE PULL STRENGTH

	Prior to Temperature Cycling (gms)	After Temperature Cycling (gms)	Percent Change	Retained Advantage Over Uncoated Specimen (gms)
Uncoated Aluminum	8.5	7.3	-14.1	—
Coated Aluminum	11.1	10.4	- 6.4	3.1
Primed and Coated Aluminum	12.3	10.2	-17.1	2.9
Uncoated Gold	28.0	26.8	-4.3	—
Coated Gold	34.4	31.2	-9.3	4.4
Primed and Coated Gold	34.7	34.0	-2.0	7.2



Test Specimen Configuration

Further the pull test values should be divided by two to arrive at the actual pull strength for a single bond.

PRETREATMENTS

Pretreatment prior to coating involves the potential need for cleaning and priming. Generally it is assumed that cleaning will be a desirable requirement — particularly if the hybrid device contains active or passive elements which are expected to be sensitive to surface contamination.

Further, it is generally good procedure to insure freedom from contamination which can interface with adhesion of the coating.

Cleaning procedures used on this program consisted of Freon vapor degreasing followed by thorough rinsing with electronic grade methanol. Results indicate that this is satisfactory for removal of adhesion preventing contaminants. For extremely sensitive semiconductor devices it may be advisable to add a hot dionoized water rinse if residual channeling types of contaminants are suspected.

Although mechanically, as noted in the previous discussion, primers did not appear to enhance the debris retention ability of the coating or improve wire bond strength, a primer does have advantages under certain circumstances where increased adhesion performs a desirable function. The one circumstance recognized in this study was during the evaluation of tape for masking purposes. It was found that during removal after coating there was less lifting of the local adjacent coating when primers were used. In all other respects, primers did not appear to provide any advantage. Finally, since the tape masking technique is not the recommended masking procedure, primers can probably be omitted as a pretreatment for most applications.

MASKING TECHNIQUES

The application of parylene to a partially completed hybrid assembly (i.e., substrates with components, interconnections, and lead frame) requires masking to prevent unnecessary coating of the lead frame or the associated leads. Since parylene penetrates small openings and crevices more readily than other coating materials, the masking technique must be capable of completely sealing the area intended to be left free of coating.

Several masking techniques were investigated. The first of these was the use of conventional pressure sensitive tapes. These tapes were typical of those used to mask printed wiring board assemblies parylene coated at Hughes. Strippable vinyl coatings were also investigated. Another technique investigated was the use of semipermanent masking fixtures in which hybrid packages can be installed for the coating process. These fixtures also served as holding fixtures and were refurbishable as necessary to preserve their effectiveness.

Pressure Sensitive Tape

Pressure sensitive tapes are capable of providing the masking necessary to prevent coating of areas intended to remain bare. As expected, however, difficulties were encountered when using this masking approach. The first difficulty lies in the difficulty of effectively applying tape to all the surfaces desired. A good deal of time and care is required to do a complete job and to avoid inadvertently damaging parts. In addition removal of the tape requires further care so as to avoid severely distorting the leads. Practically speaking some distortion appeares unavoidable. Basically only flat or near flat surfaces lend themselves well to this method of masking.

Strippable Liquid Maskants

A latex liquid maskant was used on some specimens. Although this material will prevent parylene from depositing on the surface to which it is applied, the material is difficult to apply in controlled amounts. After drying and parylene coating, the removal of the maskant is somewhat tedious in that the parylene overcoating on the maskant makes it difficult to grip the maskant and remove it along with the unwanted parylene. A thicker maskant deposit tends to improve the strippability but controlling the deposit area remains a problem. Finally, the amount of handling required for this method limits its use on a practical basis as well as increasing the potential for damage.

Semipermanent Masking Fixtures

Because of the demonstrated shortcomings of masking tape and liquid maskants, a somewhat novel approach was evaluated which held the promise of being more controllable, more effective, and more convenient to use. This technique involves the use of a two part metal fixture with foam rubber inserts. The technique is adaptable to horizontal and vertical lead hybrid packages, as well as "TO" type packages. A cross sectional view is depicted in Figure 3, and a picture of the actual tooling is shown in Figure 4.

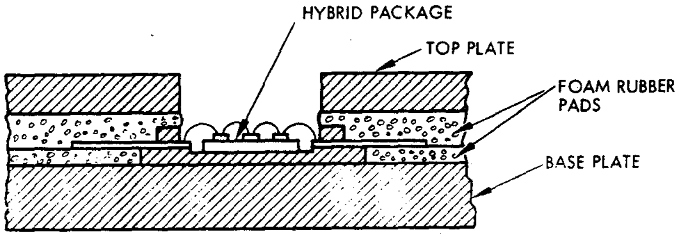


Figure 3a. Cross-sectional view of hybrid masking tooling for horizontal lead hybrid package.

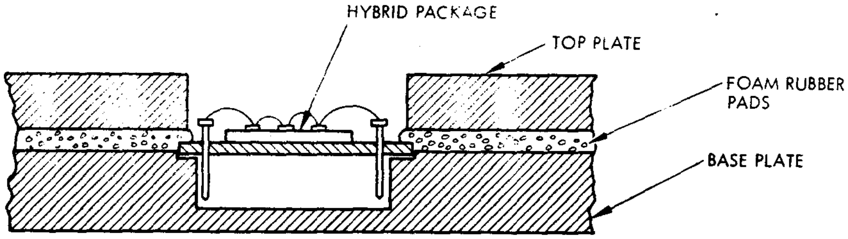


Figure 3b. Cross sectional view of hybrid masking tooling for vertical lead hybrid package.

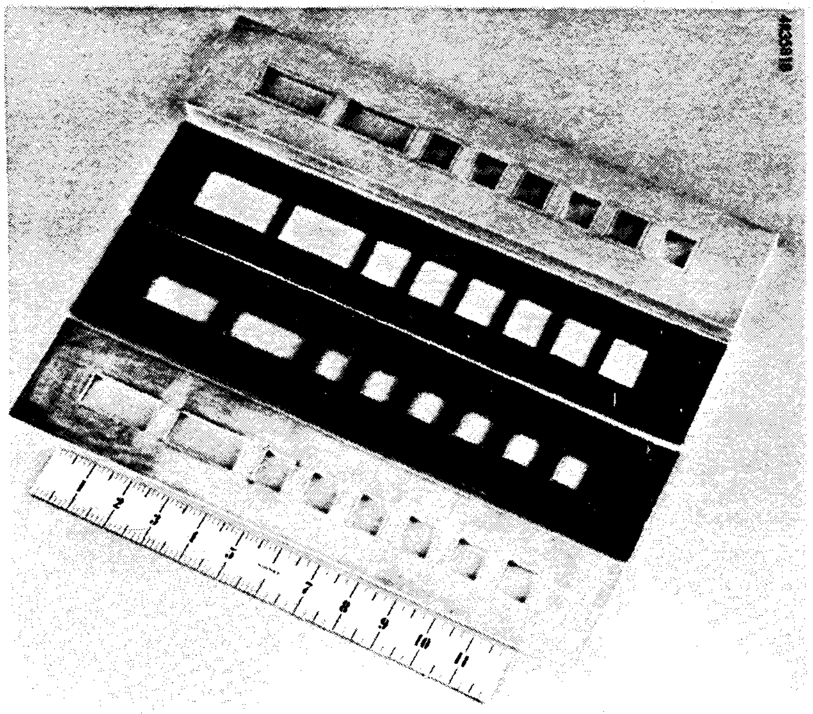


Figure 4a. Hybrid masking tooling disassembled.

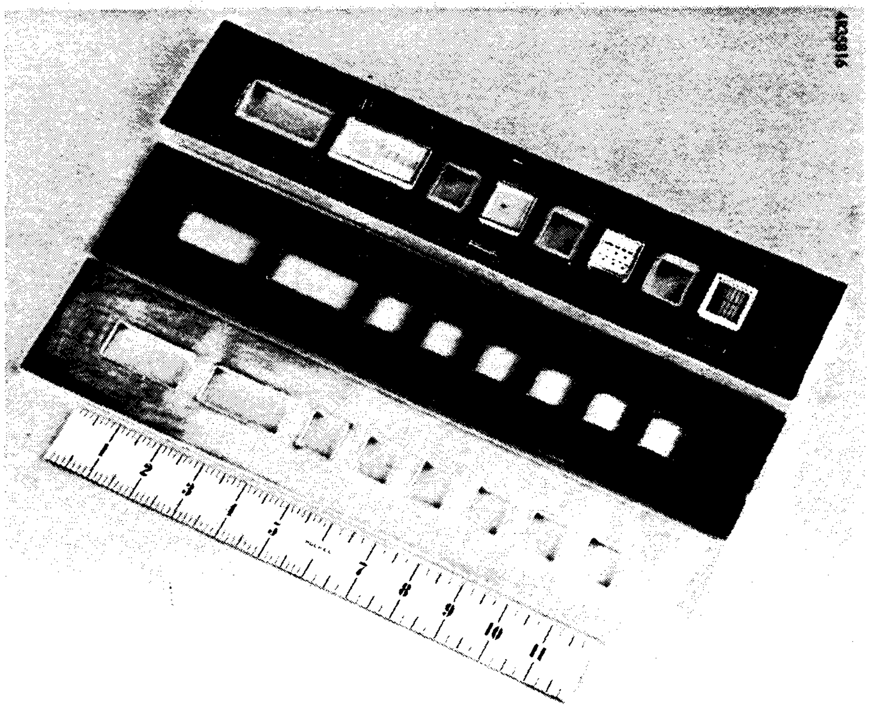


Figure 4b. Hybrid masking tooling, partially assembled with foam rubber matt installed under typical parts.

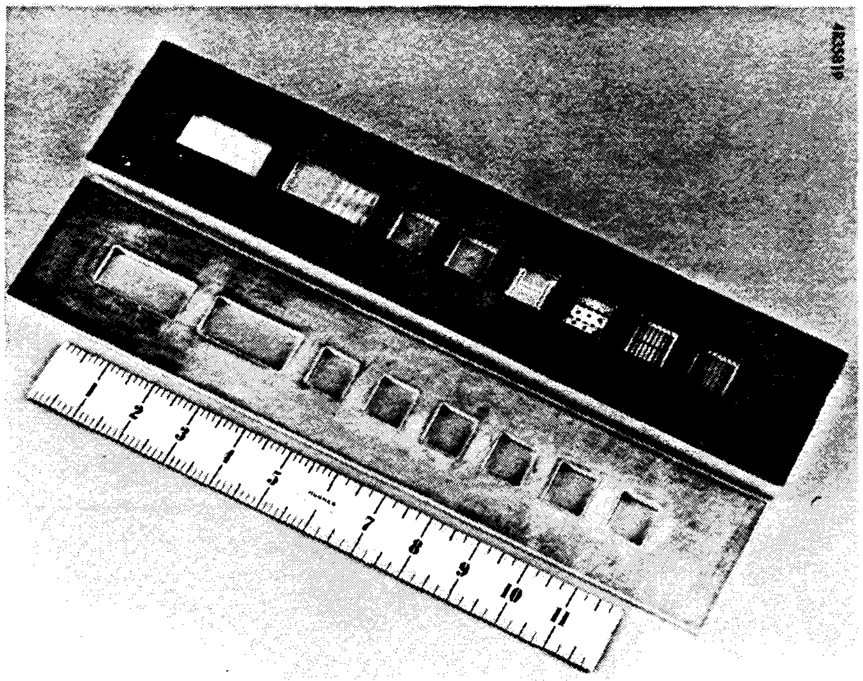


Figure 4c. Hybrid masking tooling, partially assembled with foam rubber matts installed both under and over typical parts.

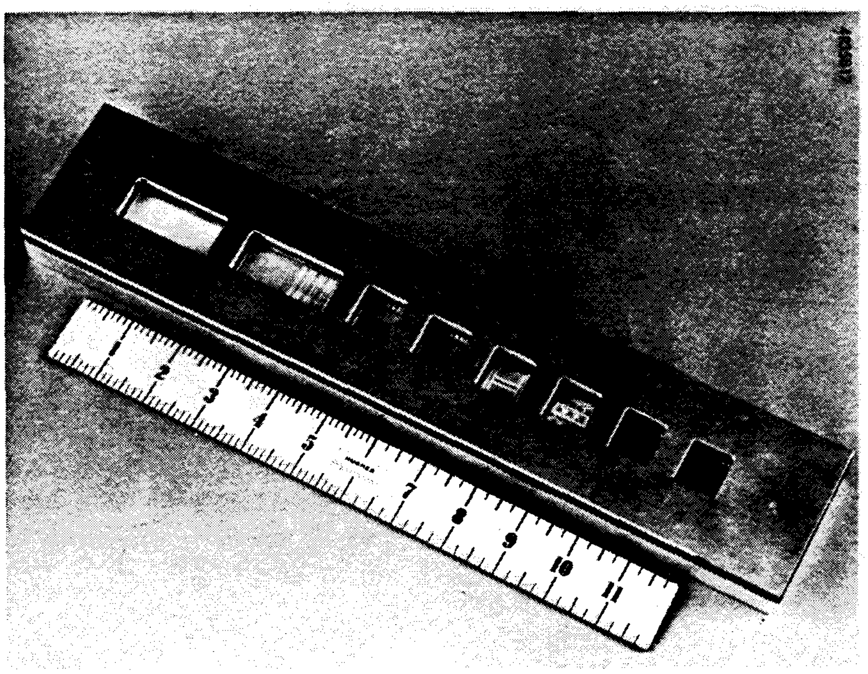


Figure 4d. Hybrid masking tooling, completely assembled with typical parts in place. (Fastening devices to hold tooling together not shown.)

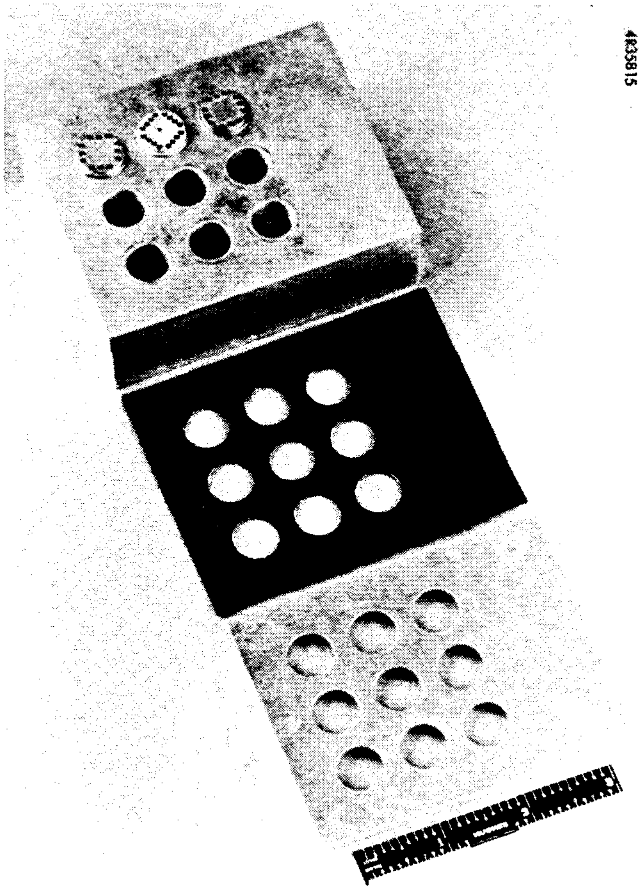


Figure 4e. TO type masking tooling, disassembled with typical parts in place.

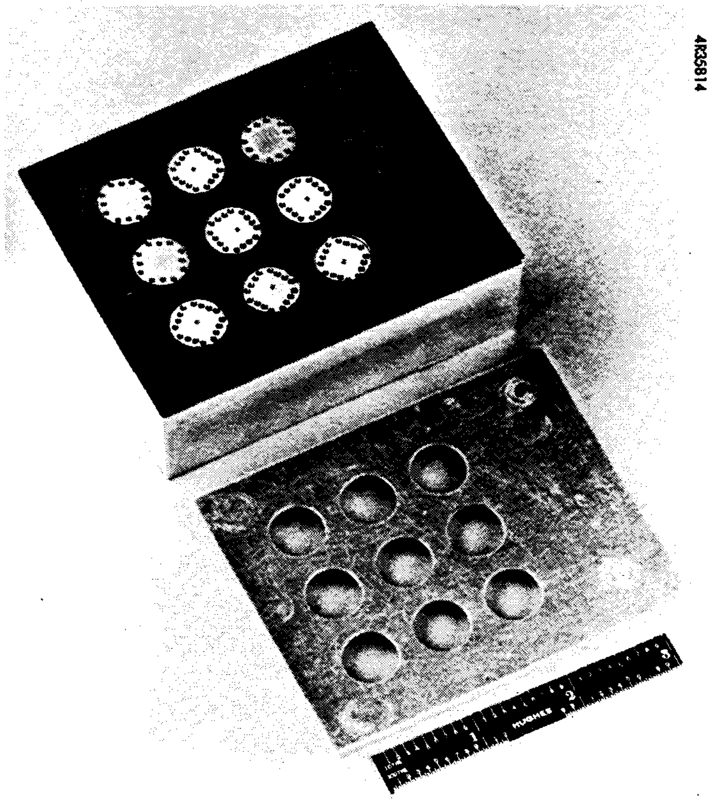


Figure 4f. TO type masking tooling, partially assembled with typical parts in place.

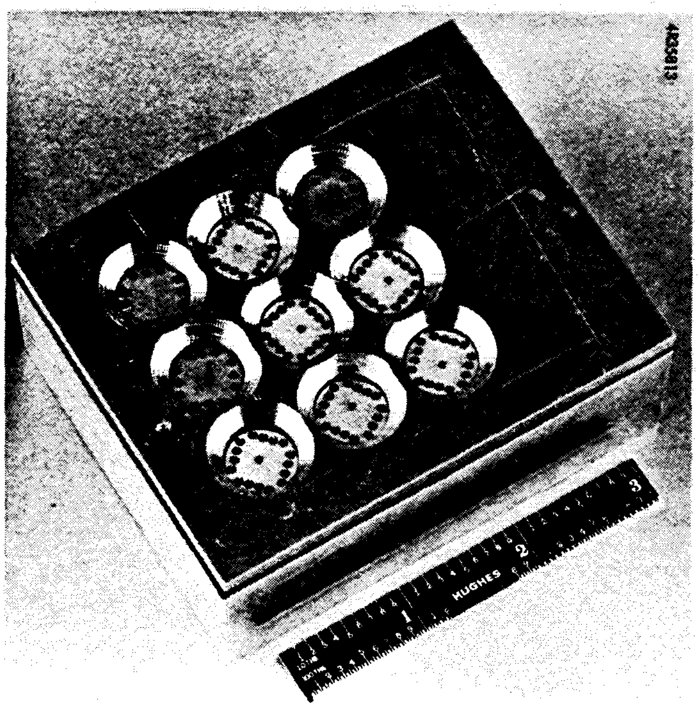


Figure 4g. TO type masking tooling, completely assembled with typical parts installed

The tooling shown in Figure 4a, b, c and d and 4e, f and g was used on actual hybrid specimens and TO type headers and was found to work with relative ease. The masking was effective and the parts were readily removable from the tooling after coating with minimal handling of the part. The tooling shown in Figure 3a, b, c, and d was designed to handle both vertical and horizontal leaded devices as well as two different sizes of each. In actual production use it would probably be desirable to fabricate tooling which would handle only one size and type of package. This would avoid the additional cost of fabricating the more universal type of tooling which would inherently limit the numbers of parts of a given size which could be accommodated at one time.

COATING DETECTION

Hughes has for several years used a fluorescent additive in its parylene coating work. Under UV light the fluorescent response made inspection for improperly coated or masked parts relatively easy. This approach was re-evaluated because of the fact that thinner coatings than used on printed wiring boards were desired and as a consequence the fluorescent response might be insufficient for inspection purposes.

Three additives were selected: Anthracene, cloroanthracene, and bromoanthracene. All three materials provided similar results producing a blue fluorescence under a UV source after coating. Cloroanthracene and bromoanthracene were investigated in addition to anthracene because of their initial green response to UV. It was felt that since UV sources tend to produce some visible blue light that a green response would be less likely to be interpreted incorrectly during examination. The material is however apparently altered chemically in the pyrolysis zone during the coating operation resulting in a change of the green response to blue.

With all three materials giving the same UV response after application, the remaining evaluation work was limited to determining the adequacy of only anthracene. The coating of actual hybrid specimens with 0.0001 to 0.0002 inch thick parylene containing 0.75 percent by weight anthracene in the dimer change was undertaken. The results of the coating application provided convincing evidence that anthracene provides a suitable tracer material for the visual detection of parylene. With the aid of conventional UV sources and typical magnification (≈ 10 x) inproperly masked areas are readily apparent. Further the extent of coating and its uniformity can be judged with this inspection method. Suitable UV source equipment is readily available and can be used conveniently in a production operation.

IN-PROCESS COATING THICKNESS CONTROL

Since coating with parylene involves a closed system, desired coating thickness is normally attained by selecting an appropriate sized charge. The charge size is selected on the basis of empirical data established during the initial few coating runs with a given piece of coating equipment. The technique is satisfactory for applications requiring coating thicknesses between 0.0005 and 0.001 inch thick where accuracy is not important.

On this program, however, thinner coatings were desired and accuracy was important. Further, knowing the thickness build-up as it was occurring was also valuable since having such information allowed discontinuing a coating run when the proper thickness is attained.

Several methods were considered for obtaining this information. Briefly these consisted of light interference patterns, frequency change of an exposed oscillating crystal and film capacitance change. All three methods are feasible, however the crystal oscillation and the capacitance method are the most practical. For this study the capacitance method was selected because the sensor and instrumentation were more readily available.

Capacitor patterns were etched from vapor deposited aluminum on quartz substrates. Figure 5 shows a typical pattern while Figure 6 shows the change in capacitance from a bare condition to one with a parylene coating thickness of approximately 0.0015 inch.

The capacitance pattern consisted of 500 intermeshed 2-1/2 inch long conductors of 0.002 inch width with 0.002 inch spacing between conductor

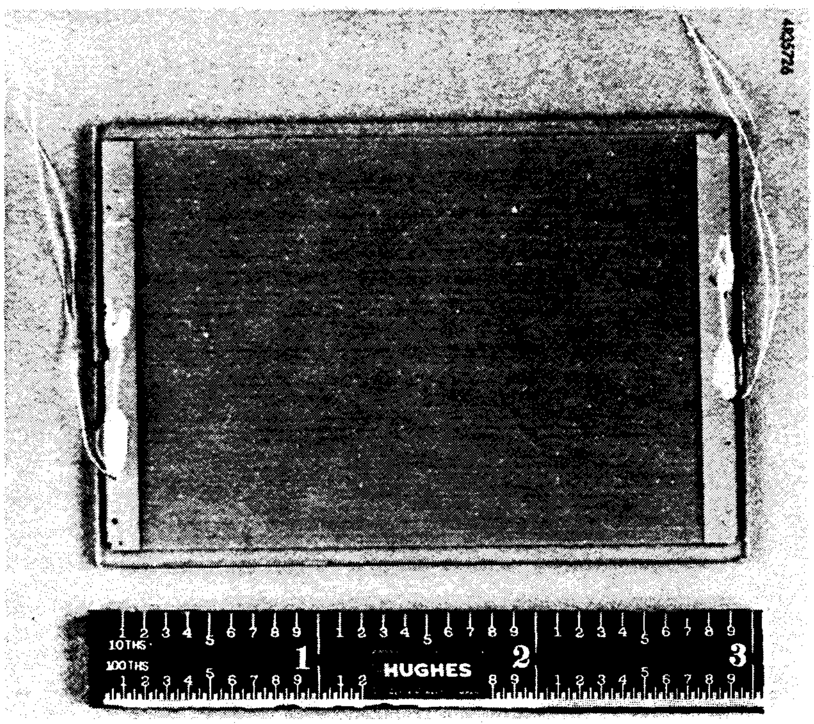


Figure 5. Typical capacitance pattern.

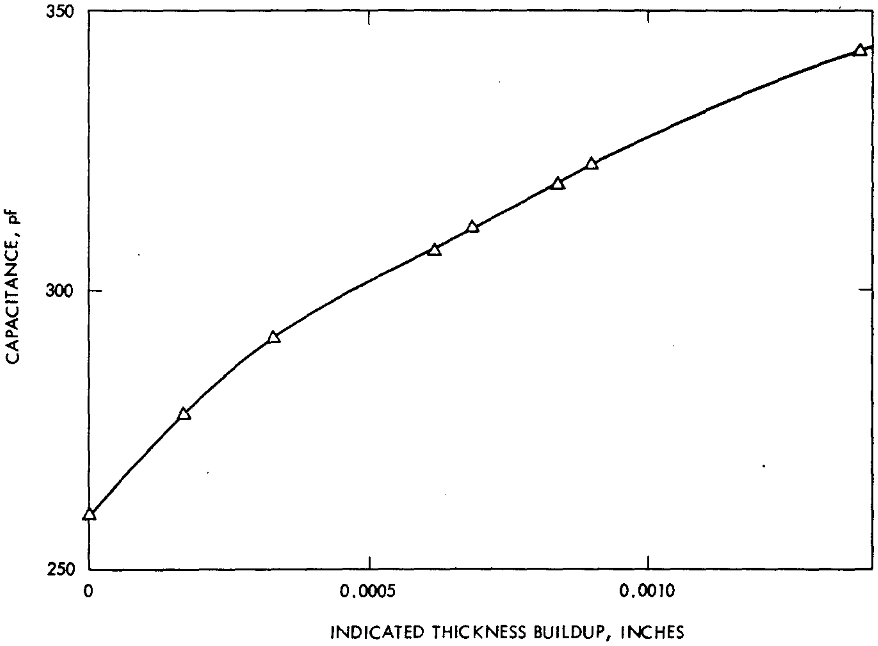


Figure 6. Coating thickness as a function of capacitance change (accumulative indicated total of 0.00138 inches compares with mechanical measured value of 0.00135 inches).

edges. The object of the large number of lines with close spacing was to produce as sensitive a capacitor as possible. As the results show (Figure 5 graph) this pattern gave good sensitivity over a 0.0015 inch thickness range. Thus a capacitor of this design has a useful life of approximately ten coating runs if the target coating thickness is 0.00015.

Considering the results, it appears that a capacitor design using fewer lines and wider spacing on glass rather than quartz would still be functional but probably for fewer coating runs. Such a pattern would be easier and less expensive to make and might be preferrable to the more sensitive version.

On a number of coating runs using the above technique, coating accuracies within approximately 10 percent of the desired thickness was achieved. A digital capacitance bridge provided the most convenient instrument for monitoring capacitance change. With a capacitance pattern, calibrated by measuring the results of previous runs on a different but identical pattern, the capacitance change corresponding to the associated coating thickness buildup could be predicted and the coating run terminated when the capacitance value was achieved.

One of the associated merits of this technique is the fact that unnecessary consumption of parylene can be minimized — in other words there is no need to overshoot to be sure of achieving a predetermined minimum.

EFFECTS UPON THERMAL DISSIPATION

Interconnect Wires

To study the thermal effects of parylene coating on interconnect wires, specimens containing 0.05 inch and 0.10 inch spans of 0.002 inch diameter gold wires were chosen. Some of the specimens were left bare while others were either coated or primed and coated. Figure 7 shows a typical specimen.

Using an infrared microscope, the radiant energy emitted from coated and uncoated gold interconnect wires was compared for various current settings. The tests were performed in an air environment with the substrate maintained at approximately 15°C. Initially approximately five times as much radiant energy was detected from the coated wire as compared to the uncoated wire. This difference varied, however, depending upon the current

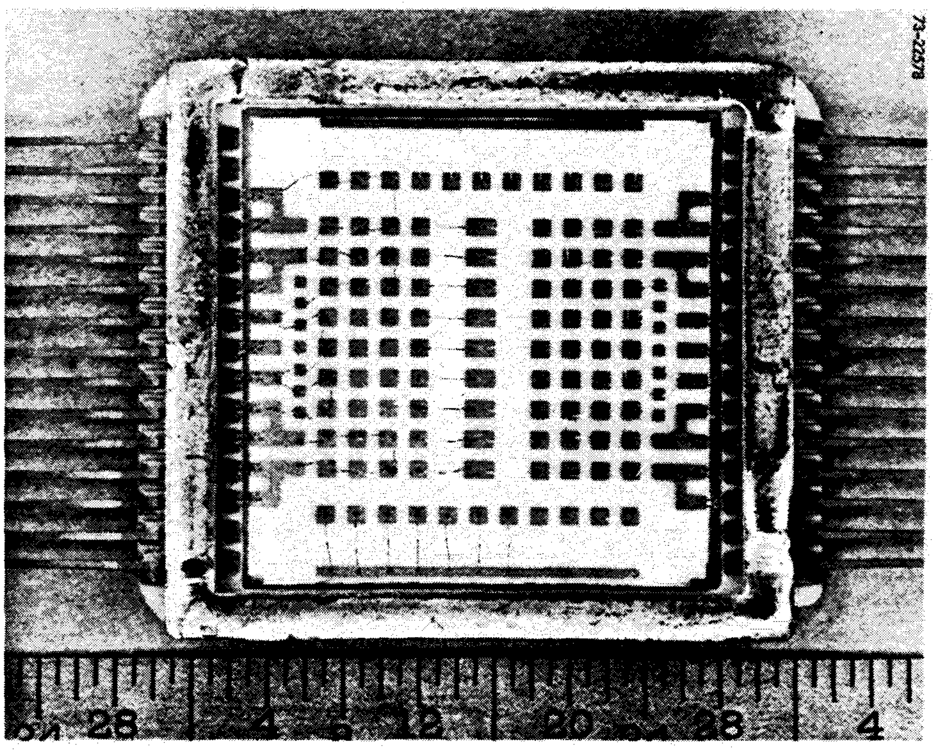


Figure 7. Thermal dissipation specimen.

level as well as the length of time it was subjected to temperatures near the melting point of the coating. Near the melting point the coating was observed to darken and then gradually disappear, indicating oxidation and an eventual conversion to CO or CO_2, and/or evaporation. Above the melting point the evaporation was rapid enough to cause the coating to disappear before much darkening occurred. In either case the wire returned to approximately the emittance level of an uncoated wire. When subjected to higher current, both coated and uncoated wires burned out in similar fashion at approximately the same current level.

The results can be interpreted two ways. One, the coating reduces convection cooling and as a consequence results in increased wire temperature as indicated by higher detected radiant energy. The alternate explanation (and the more accurate) is that the coating reduces convection cooling but increases the surface emissivity resulting in increased radiation cooling. Earlier emissivity measurements indicated the 0.00024 inch thick parylene on gold increased the emissivity from 0.04 to 0.16. The net effect, then, on wire temperature is difficult to determine but since reduced convection cooling is being more or less offset by increased radiant cooling, wire temperature probably remains nearly the same as with the uncoated counterpart.

Because of difficulty in interpreting the results from radiant energy measurements, other than to say that the effect was minor, additional tests were performed using a different technique. These tests were performed on aluminum and gold wires to determine effects of the coating on wire burnout current. Coating thickness used was again 0.00024 inch. In these tests, coated, primed and coated, and uncoated wires were subjected to rapid increases in current to achieve burn-out in approximately five seconds. No significant effect traceable to the coating was noted with either aluminum or gold wires. The data summarized in Table V indicates, however, a possible slight increase in burn-out current for primed and coated aluminum wires while a slight decrease in burn-out current for coated or primed and coated gold wires is shown. Since only three specimens of each type were tested these slight differences may not be real, although among each sample of three, the values were quite consistent.

TABLE V. WIRE BURNOUT CURRENT

	0.001 Inch Diameter, Aluminum Wire	0.002 Inch Diameter, Gold Wire
	Average Burnout Current (amps)	Average Butnout Current (amps)
	<u>0.10 Span Length</u>	<u>0.10 Span Length</u>
Uncoated	0.77	3.45
Coated	0.74	3.27
Primed and Coated	0.79	3.25
	<u>0.05 Span Length</u>	<u>0.05 Span Length</u>
Uncoated	1.23	All specimens tested to 5 amps without failure. Exceeding this value caused excessive heating of external leads which prohibited the use of higher current to burn-out.
Coated	1.23	
Primed and Coated	1.35	

Beam Lead Devices

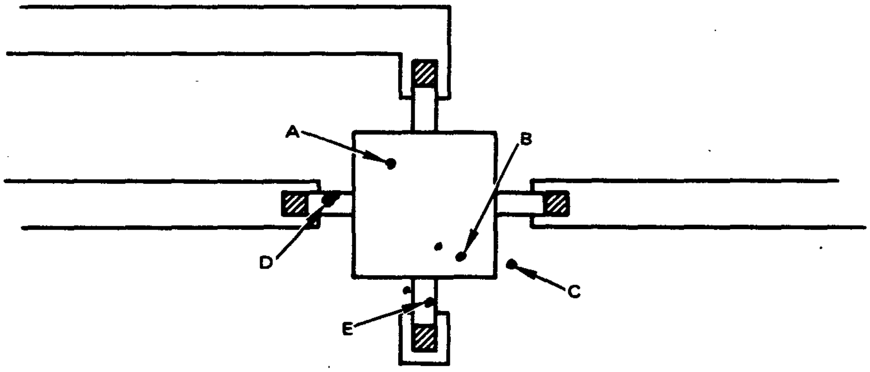
Determination of thermal effect on beam lead devices was accomplished using several mounted Texas Instrument 2N2907 transistors. Devices were tested in the uncoated, coated, and primed and coated condition. Two methods were used to heat the devices.

The first used an external heat source to provide uniform heating of the hybrid package while the second method involved electrically energizing the device while holding the substrate at 15°C. The reason for heating the device two ways was to enable the comparison of emitted energy of surfaces known to be at uniform temperatures versus the same device internally heated where temperatures would not be uniform. To provide a data reference point, the electrically energized device was powered up until the hottest point on the bare chip reached the value observed with the corresponding uniformly heated device. Coated or primed and coated parts were then subjected to the same uniform heating conditions or powered up to the same level and the emitted energy monitored using a Barnes scanning infrared microscope. Table VI presents the data with the chip shown pictorially with labeled points of reference. Interpreting the data indicates the following.

Only a slight difference was observed in emitted radiant energy between coated (or prime and coated) and uncoated devices with either heating method. Coated devices appeared to emit slightly more radiant energy than uncoated devices. Since convection cooling probably contributes little in the cooling of beam lead devices as compared to conduction cooling via the leads, the increased radiant energy is probably due to the coating's effect of increasing surface emissivity. If this analysis of the data is correct and convection cooling is unchanged or minimal, then the results can be interpreted as a slight

TABLE VI. BEAM LEAD THERMAL DISSIPATION

		Radiant Energy Observed (See Keister for Unit of Measurement)	
	Point Observed	Uniformly Heated	Electrically Energized 70 Volts 18.0 ma (Substrate at 15°C)
Bare	A	120 (80°C)	120 (80°C)
	B	118 (80°C)	060 —
	C	113 (80°C)	042 (15°C)
	D	54 (80°C)	034 —
	E	53 (80°C)	038 —
Coated	A	132 (80°C)	120 (<80°C)
	B	125 (80°C)	060 —
	C	116 (80°C)	040 (15°C)
	D	070 (80°C)	038 —
	E	070 (80°C)	040 —
Primed and Coated	A	130 (80°C)	130 (80°C)
	B	125 (80°C)	065 —
	C	118 (80°C)	044 (15°C)
	D	062 (80°C)	040 —
	E	062 (80°C)	042 —



lowering of temperature of coated devices as compared to uncoated devices when operated under equal electrical loads. If convection cooling is more significant than suggested, the above conclusion may only be accurate for a zero gravity space environment where convection cooling is not a factor. In any event, coating thicknesses up to 0.00024 appear to have only a minimal effect on beam lead devices.

Finally attempts were made to determine burnout current. Parts were subjected to current levels considerably above the rating for the device. Since it was found that the coating evaporated before burnout occurred this method of testing was discontinued on the basis that results were expected to duplicate uncoated devices.

THE EFFECTS OF PARYLENE COATING ON INSULATION RESISTANCE, VOLTAGE BREAKDOWN, AND PLANAR CAPACITANCE

FABRICATION OF TEST PATTERNS

The test pattern used for this portion of the program was a multi-purpose pattern. A photograph of a typical pattern is shown in Figure 8. The two inter digitated comb patterns on the right in Figure 8 serve a dual purpose: 1) for insulation resistance (IR) measurements; and 2) for capacitance (C) measurements. The larger comb pattern has 10-mil lines and spaces (36 planar capacitors in parallel). The smaller comb pattern has 5-mil lines and spaces (18 planar capacitors in parallel).

On the left in Figure 8 are a series of voltage breakdown (VB) patterns having line spacings of 1-mil, 2-mil, 5-mil, 10-mil, and 15-mil. Since a voltage breakdown pattern can be used only once, a series of these patterns were photoetched on the same substrate, so that a different series could be used for VB measurements at 0,168,500, and 1000 hours exposures to +150°C.

This pattern was photoetched from nichrome-nickel-gold thin film metalization on a 99.5 percent alumina substrate, 3/4 inch x 1-3/4 inch x 0.025 inch thick. Eight of these patterns were fabricated and evaluated. The substrates were bonded inside 1 inch x 2 inches ceramic hybrid packages. Metal covers were hermetically solder sealed to certain of the packages in a 99.9 percent pure nitrogen atmosphere. Other packages were simply left unsealed and thus exposed to an air environment.

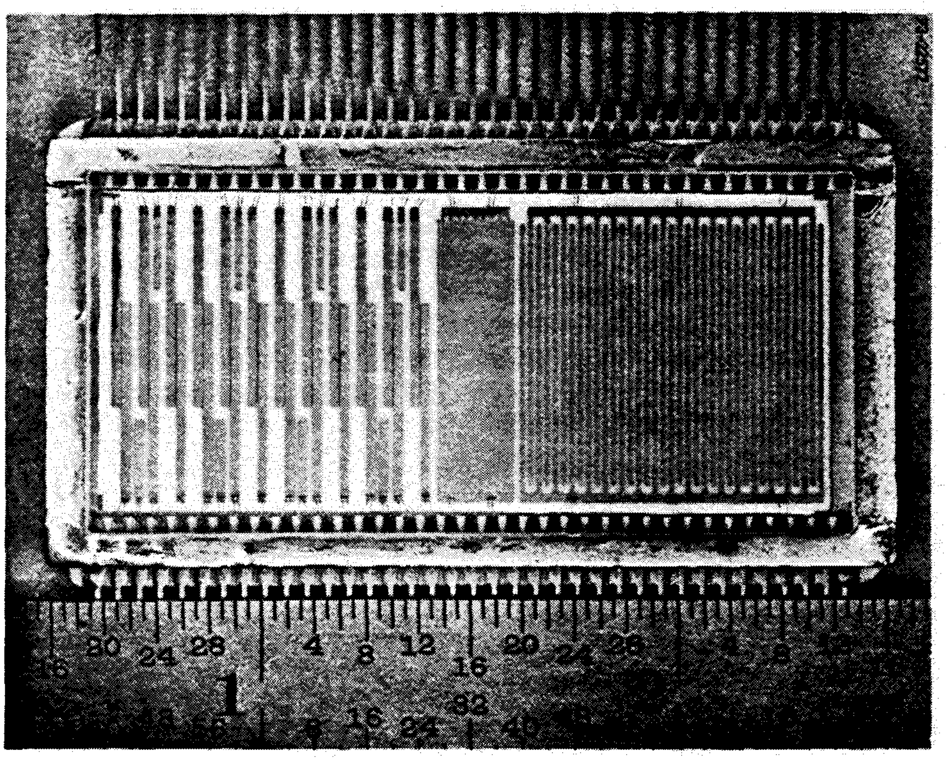


Figure 8. Insulation resistance, dielectric breakdown and low frequency capacitance patterns.

TESTING OF IR/VB/C PATTERNS

Voltage breakdown tests were done in accordance with MIL-STD-202C, Method 301 using ac applied voltage. An Assoc. Research HYPOT tester was used. Values were recorded when actual breakdown was observed and when the first signs of leakage occurred. Breakdown was observed visually (by seeing the arc-cover), by hearing the crackling noise characteristic of arc-over, and by observing the leakage and breakdown lights on the Hypot tester.

Insulation resistance measurements were made per MIL-STD-202C, Method 302, Condition B. A General Radio Type 1862-B Megohm Bndye was used.

Capacitance measurements were made per MIL-STD-202C, Method 305. Capacitance and dissipation factor measurements at 1000 Hz were done using a General Radio Model 1673 Automatic Capacitance Bridge. Capacitance measurements at 1 MHz and 100 MHz were done using a Boonton Model 33A RF Admittance Bridge, Dissipation factor could not be measured at 1 MHz or 100 MHz.

Electrical measurements of these 8 test patterns were made before and after parylene coating and after exposure to +150°C for 168, 500, and 1000 hours.

TEST PROCEDURE

The block diagram in Figure 9 shows the test procedure used for the 8 IR/VB/C test patterns. Two of the patterns were used as controls and were left uncoated. The remaining six were parylene coated. Of these six, two were primed prior to parylene coating. The parylene coating was 0.2-mil thick.

TEST RESULTS AND DISCUSSION

Does Parylene Coating Affect Insulation Resistance?

Table VII shows the IR test results for the pattern having 10-mil line widths and spacings. The 5-mil IR test pattern showed similar results.

From Table VII it is obvious that parylene coating had no degrading effect on insulation resistance whether the test specimens were exposed to an air or nitrogen environment. All readings were greater than 10^{14} ohms. The alumina ceramic is rated at 10^{14} ohm-cm. Volume resistivity at 25°C The surface resistivity of Parylene C is also rates at 10^{14} ohms at 230°C. Thus the IR results were as expected.

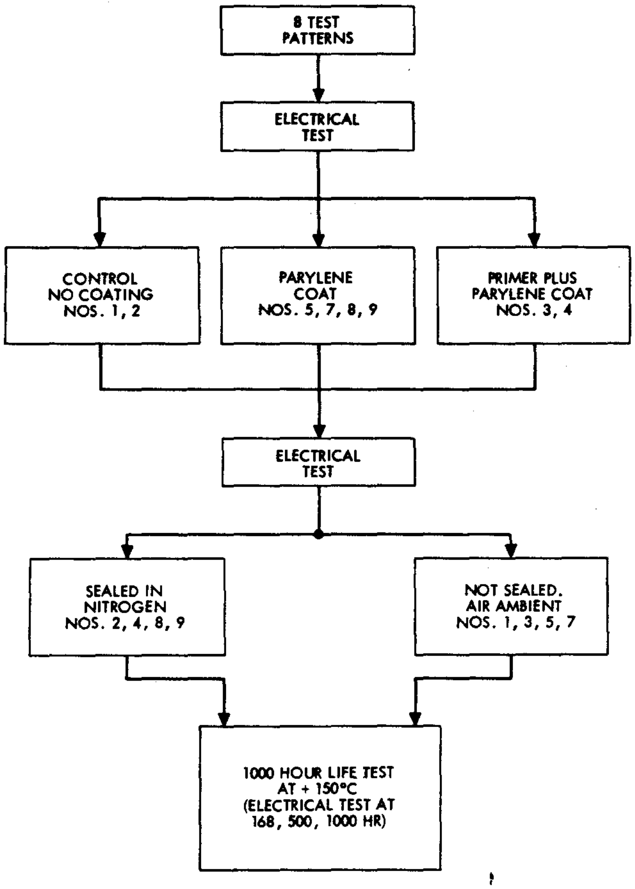


Figure 9. Block diagram of test procedure.

TABLE VII. INSULATION RESISTANCE TEST RESULTS FOR 10-MIL PATTERN

Type Specimen	Insulation Resistance (ohms)			
	Initial	After Cleaning and/or Coating	After 1000 hrs Exposure at 150°C	Percent Change
Uncoated air control	$>10^{14}$	$>10^{14}$	$>10^{14}$	–
Uncoated N_2 control	$>10^{14}$	$>10^{14}$	$>10^{14}$	–
P + P/air	$>10^{14}$	$>10^{14}$	$>10^{14}$	–
P + P/N_2	$>10^{14}$	$>10^{14}$	$>10^{14}$	–
P/air	$>10^{14}$	$>10^{14}$	$>10^{14}$	–
P/N_2	$>10^{14}$	$>10^{14}$	$>10^{14}$	–
Note: P + P = Primer plus parylene coating P = Parylene coating only. No primer.				

How Does Parylene Coating Affect Planar Capacitance?

The measurement of planar capacitance is another indication of the quality of the parylene film. In most hybrid microcircuit applications, conductors run parallel to each other and even cross over each other in multilayer designs. The capacitance between adjacent conductors is an important consideration is many circuit designs involving sensitive parameters such as noise, coupling, shielding, crosstalk, etc. Ideally this capacitance should be minimized.

Table VIII presents the capacitance values for the 8 test patterns. The values are those of the planar capacitor having 5-mil line widths and spacings. The 10-mil capacitor patterns could not be measured at 100 MHz because the range of the bridge would only permit capacitance values less than 30 pf at that frequency.

Dissipation factor values are not included in Table VIII, since these values were all extremely low and appeared not to increase measureably as a result of high temperature life testing. Typical dissipation factors ranged from 0.0001 to 0.006 (i.e., 0.01 percent to 0.6 percent).

The capacitance increased slightly with an increase in frequency. The capacitance of the uncoated 5-mil pattern was nominally about 15-16 pf. The capacitance of the uncoated 10-mil pattern was approximately twice as high, being about 31-34 pf.

The capacitance increased slightly with the addition of the parylene coating. This is expected, since the dielectric constant of parylene is 3.1. This is three times higher than that of air (k = 1). The primer has no effect on capacitance.

TABLE VIII. CAPACITANCE DATA FOR THE 5-MIL TEST PATTERN AT 1000 HZ, 1 MHZ AND 100 MHZ

Type Specimen	Frequency	Capacitance (pf) After Cleaning and/or Coating	Capacitance (pf) After 1000 hours at 150°C	Capacitance (pf) Percent Change In Capacitance
Uncoated air control	1000 Hz	15.40	15.51	+0.71
Uncoated N_2 control	1000 Hz	15.59	15.46	-0.83
P + P/air	1000 Hz	16.04	15.88	-1.00
P + P/N_2	1000 Hz	15.77	16.25	+3.04
P/air	1000 Hz	17.11	16.43	-3.97
P/N_2	1000 Hz	15.57	15.63	+0.39
Uncoated air control	1 MHz	16.37	16.25	-0.73
Uncoated N_2 control	1 MHz	16.55	16.64	+0.54
P + P/air	1 MHz	17.25	16.95	-1.74
P + P/N_2	1 MHz	17.17	17.64	+2.74
P/air	1 MHz	17.46	16.94	-2.98
P/N_2	1 MHz	17.02	16.82	-1.18
Uncoated air control	100 MHz	20.36	20.47	+0.54
Uncoated N_2 control	100 MHz	20.34	20.47	+0.64
P + P/air	100 MHz	21.50	21.90	+1.86
P + P/N_2	100 MHz	21.06	21.32	+1.23
P/air	100 MHz	22.08	21.87	-0.95
P/N_2	100 MHz	21.55	20.91	-2.97

Note: P + P = Primer plus parylene coating.
P = Parylene coating only. No primer.

After exposure to +150°C for 1000 hours, the uncoated specimens generally showed less percentage capacitance change than any of the coated specimens regardless of whether they were in an air or N_2 environment. Considering only the parylene coated specimens, neither the primer nor the environment (i.e., air or N_2) showed a consistently higher or lower percentage capacitance change. Any differences observed were so minor as to be within the measuring accuracy of the particular bridge used. In some cases the changes were positive, while in others they were negative.

Thus it could be concluded that the capacitance of both the coated and uncoated specimens was relatively unaffected by exposure to +150°C for 1000 hours regardless of whether the parts were sealed in air or nitrogen. If anything, it could be said that the parylene coated parts (whether primed) or not) showed slight decreases in capacitance after exposure to +150°C in an air environment. Those coated parts exposed in a nitrogen environment predominantly showed slight increases in capacitance. However these increases and decreases, taken as a whole, averaged only ±2 percent.

What is the Effect of Parylene Coating on Voltage Breakdown?

Ideally dielectric breakdown strength should be as high as possible. Although most hybrid microcircuits are low voltage, the line spacings are very close. Spacings of 1 to 2-mils are not uncommon. Spacings on integrated circuit chips are even closer, being in the order of 0.5 to 1-mil. The dielectric strength of 99.5 percent pure alumina is about 800 to 900 volts per mil (31 to 35 kilovolts per mm). The short time dielectric strength of Parylene C is 5600 volts per mil at 1-mil. Thus it would be expected that parylene would assist in voltage breakdown strength across the surface of the substrate. Cleanliness of the spacing between adjacent conductors must be maintained. Any small metal particles or unetched material between the lines could also serve as a stepping stone for a shortened breakdown path.

Table IX presents the voltage breakdown data in reduced form. Only the data for the 1-mil, 5-mil, and 10-mil spacings are included. The 2-mil and 15-mil spacings followed a similar trend. In some instances, breakdown first occurred between the leads on the outside of the hybrid package, especially on those tests involving the 10-mil and 15-mil spacings. This is because the metallization spacing on the packages is a minimum of 10-mil and could easily break down first, especially if the outside of the package was not as clean as the substrate inside.

Although leakage readings and actual voltage breakdown readings were recorded during the actual measurements, only voltage breakdown is included in Table IX. In many cases, leakage and breakdown occurred simultaneously. In general, both leakage and breakdown followed similar trends.

Regardless of whether the specimens were exposed to air or nitrogen and regardless of whether the readings were taken at 0, 168, 500, or 1000 hours, the order of highest voltage breakdown was:

Best — Parylene coating only.
2nd Best — Primer plus parylene coating.
Poorest — Uncoated patterns.

This ordering was also fairly consistent for 1, 5, and 10-mil gap spacings. Thus it seems that parylene only is superior to the others. The addition of a primer decreases the dielectric strength of the film. The patterns coated

TABLE IX. VOLTAGE BREAKDOWN TEST RESULTS

Test Pattern Identification	Spacing Gap (mils)	Voltage Breakdown (volts)			
		After Cleaning and/or Coating	After 168 hours at +150°C	After 500 hours at +150°C	After 1000 hours at +150°C
Uncoated control. Air exposure.	1	2600	1500	1800	1300
	5	700	800	900	700
	10	1000	1100	1200	900
Uncoated control. Sealed in Nitrogen.	1	3000	1400	1800	1200
	5	1000	900	900	700
	10	1000	1100	1100	900
Primer + Parylene air exposure.	1	>2000	>2000	2500	2100
	5	>2000	>2000	1000	700
	10	>1800	>1800	1400	1200
Primer + Parylene. Sealed in Nitrogen	1	>2100	>2100	3100	1900
	5	>2100	>2300	3000	800
	10	>1800	>1400	1500	1200
Parylene only. Air exposure.	1	>3000	>2400	2600	2200
	5	>3100	>2000	1400	1000
	10	>1400	>1400	1450	1350
Parylene only. Sealed in Nitrogen.	1	>2100	3200	3000	2250
	5	>2200	>2900	2500	3050
	10	>2200	2350	2000	1800

with only parylene showed average breakdown strengths of 2500 volts/mil prior to life testing and 2200 volts/mil after life testing.

For both air and nitrogen ambients, all patterns showed consistently lower breakdown strengths after being exposed to +150°C for 1000 hours.

The breakdown strengths in nitrogen were slightly higher than those in air, although the difference was slight and inconsistent.

CONCLUSIONS

Effects on Insulation Resistance

Parylene coating caused no change in insulation resistance (neither improvement nor degradation), even after 1000 hours exposure to +150°C. This applies regardless of whether a primer is used or whether the parylene coated patterns are exposed in an air or nitrogen environment.

Effects on Planar Capacitance

The addition of a parylene coating to adjacent conductors causes a slight increase in coupling capacitance. This capacitance increases slightly with an increase in frequency from 1000 Hz to 100 MHz. The capacitance of parylene coated patterns (whether primed or not) is, for all practical purposes, unaffected by exposure to +150°C for 1000 hours in either air or nitrogen. The parylene coated patterns did, however, exhibit a greater percentage change in capacitance after life testing than the uncoated patterns. These percentage changes were so small as to be considered almost insignificant.

Effects on Voltage Breakdown Strength

The parylene coated patterns had consistently higher voltage breakdown strengths than the uncoated patterns in both air and nitrogen environments. Parylene unprimed was superior to the primed patterns. The voltage breakdown strength decreased after exposure to +150°C for 1000 hours in both air and nitrogen environments. A nitrogen environment showed no significant advantage over an air environment except for the unprimed paraylene coated specimen, except for the unprimed parylene coated specimen.

THE EFFECTS OF PARYLENE COATING ON MICROWAVE INTEGRATED CIRCUITS

FABRICATION OF TEST PATTERNS

The Microwave Integrated Circuit (MIC) test pattern is essentially a microstrip transmission line with a 0.025 inch conductor width and a conductor length of 5.285 inches. Photographs of the 8 test patterns are shown in Figure 10.

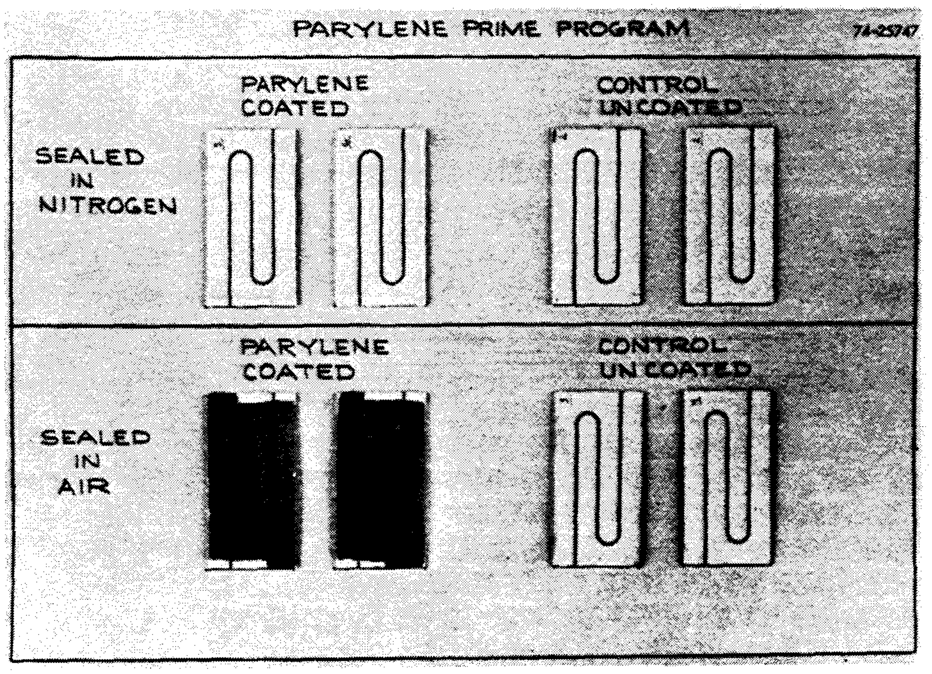


Figure 10. Microwave integrated circuit patterns.

The test patterns were photoetched from 1 x 2 x 0.025 inch thick substrates procured from TEK-WAVE (Part No. 15-2514-1212). This material is 99.5 percent pure alumina with a 10 microinch surface finish. The thin film metallization is chromium-copper-gold. The copper layer is 500 microinch thick.

TESTING OF MIC PATTERNS

All 8 MIC patterns were tested for VSWR and insertion loss over the frequency range of 2 to 12 GHz in increments of 500 MHz using an HP 8541A Automatic Network Analyzer. Measurements were made before and after parylene coating and after exposure to +150°C for 168 hours, 500 hours, and 1000 hours. Typical raw data sheets from the computer print-out are included in Appendix A. VSWR and loss measurements were made in both the forward and reverse directions. A total of 3,360 VSWR and loss measurements were made on the 8 specimens during the course of this program over a period of 5 months. The calculated error for these measurements in terms of line loss per inch is ±1 percent.

TEST PROCEDURE

The block diagram in Figure 11 shows the test procedure used for the 8 MIC patterns. Four patterns were used as controls and were uncoated. The other four were parylene coated. No primer was used. Four of the patterns were exposed to +150°C in air. The other four were exposed to +150°C in a 99.9 percent pure nitrogen atmosphere.

VISUAL APPEARANCE AFTER LIFE TEST

At the start of the +150°C exposure, all 8 test patterns visually appeared identical. After 168 hours exposure, the parylene coated specimens

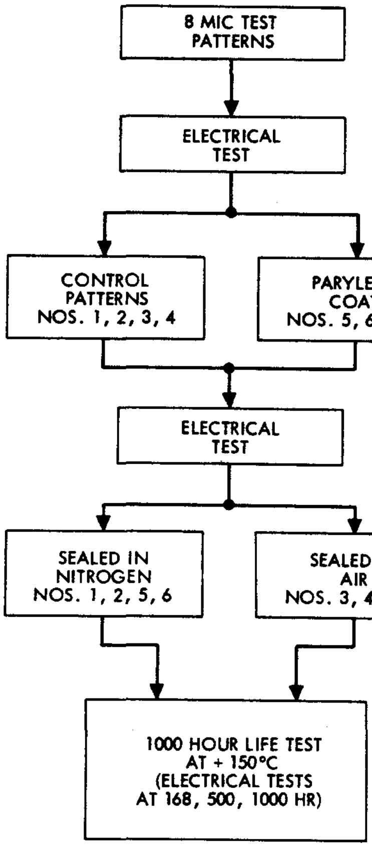


Figure 11. Test procedure for microwave integrated circuit patterns.

sealed in air began to discolor. The coating turned a light brown. In addition, some of the parylene started to flake off. The adhesion was especially poor to the gold conductors. The other patterns were essentially unchanged. Figure 12 shows all 8 test patterns after the 168 hour exposure.

After 500 hours exposure, the parylene coated specimens sealed in air had turned a darker brown. The uncoated conductor patterns sealed in air also began to noticeably discolor. The gold conductors showed evidence of diffusion of the copper film beneath the gold.

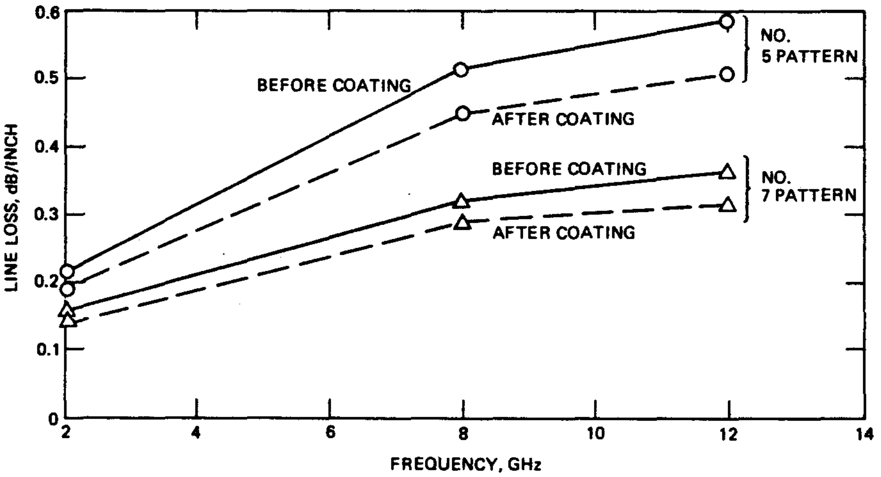


Figure 12. Plot of line loss vs. frequency for two parylene coated test patterns.

After 1000 hours exposure the two parylene coated specimens sealed in air had discolored to a tobacco shade. Discontinuities in the parylene over the gold conductors were evident. The parylene became brittle and could be easily scraped off the substrate. The parylene specimens sealed in nitrogen did not discolor, but did suffer a slight loss in adhesion. However, the parylene film was still flexible and showed no signs of embrittlement. The uncoated control patterns sealed in air showed definite signs of conductor discoloration, probably due to copper diffusing through pores in the gold film.

ELECTRICAL TEST RESULTS

In the analysis of the test results, gain (or insertion loss) was selected as the most meaningful criteria rather than VSWR. All gain measurements were converted into values of insertion loss in dB/inch using the REVERSE direction measurements. Although measurements were made at 21 different frequencies between 2 GHz and 12 GHz, only the values at 2, 8, and 12 GHz were selected for analysis. This selection was arbitrary, but nevertheless valid, since the values at the other frequencies showed similar trends. Table 1 in Appendix B lists all of the line loss data for each test pattern.

The percentage changes in line loss were calculated as a result of the 1000 hour exposure to +150°C. The end points of 0 hours and 1000 hours were selected for analysis rather than any intermediate time intervals. These calculations are given in Table 2 in Appendix B.

The percentage changes in line loss were also calculated using the line loss values before and after parylene coating. These calculations will allow a comparison of the coated versus the uncoated specimens at the different frequencies prior to any environment exposure. These values are given in Table 3 in Appendix B.

DISCUSSION OF TEST RESULTS

How Does Parylene Coating Affect Loss at Microwave Frequencies?

The 4 parylene coated samples were compared with the 4 uncoated samples. The comparison is shown in Table X. The minus sign (-) indicates a percentage decrease in loss, thus an improvement. The change in the uncoated specimens is due only to a time lapse of 65 days between measurements.

TABLE X. COATED VERSUS UNCOATED SPECIMENS

	Avg. Percentage Change in Line Loss		
Identity	2 GHz	8 GHz	12 GHz
Uncoated	-4.02	-5.08	- 8.37
Coated	-5.56	-8.63	-13.68

Figure 13 is a plot showing loss v. frequency before and after parylene coating for 2 typical test patterns. The other 2 coated patterns showed a similar trend.

From Table X and Figure 14, it appears that parylene coating does not adversely affect line loss. In fact, if anything, the loss improves after coating. The improvement in loss is more pronounced as the frequency increases. For example, at 12 GHz the percentage improvement in loss is greater than at 2 GHz or 8 GHz. Normalizing the coated and uncoated samples shows the overall improvement in loss to be 1.5 percent at 2 GHz, 3.6 percent at 8 GHz, and 5.3 percent at 12 GHz.

Coated v. Uncoated Test Patterns After Air Exposure

A comparison was made between the coated and uncoated test patterns after being exposed to an air environment for 1000 hours at +150°C. The results are presented in Table XI and Figure 15.

The parylene parts are definitely worse than the uncoated parts. This conclusion is justified, since the coated parts have a much higher percentage

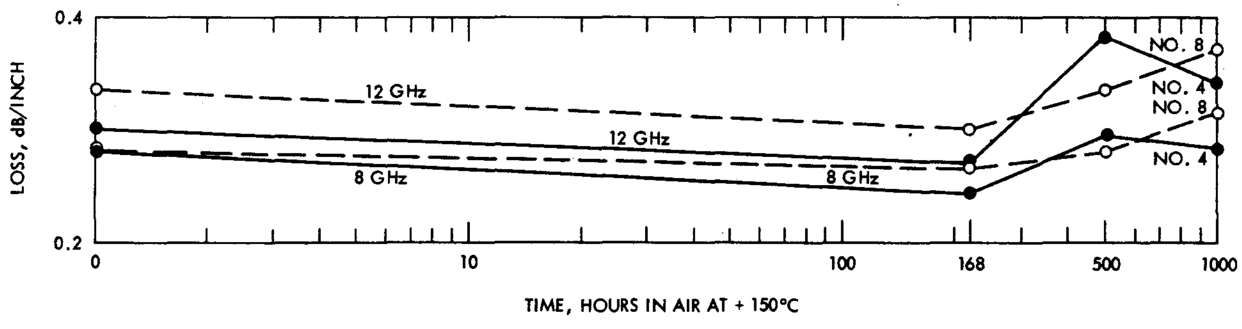


Figure 13. Plot of coated pattern (No. 8) v. uncoated pattern (No. 4) during 150°C air exposure at 8 GHz and 12 GHz.

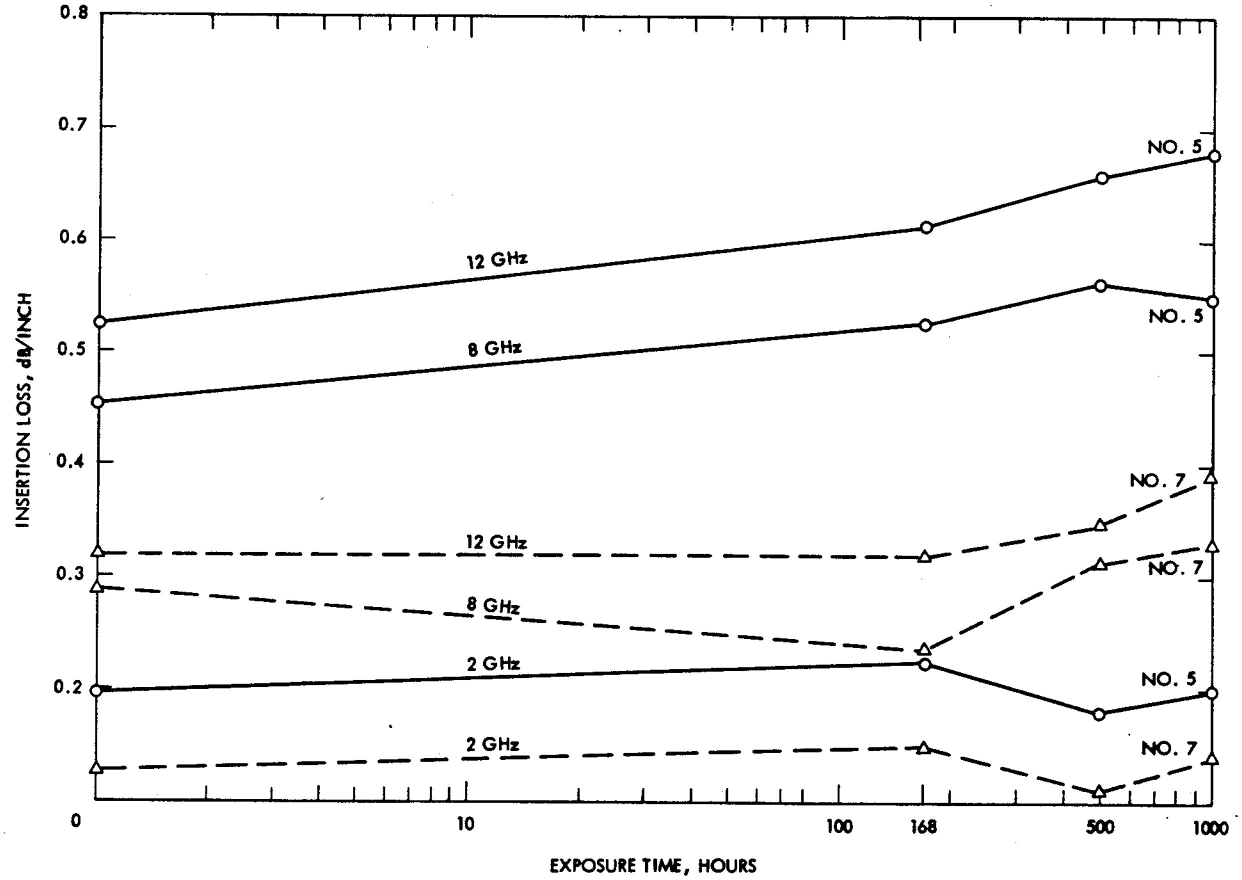


Figure 14. Plot of insertion loss v. exposure time in nitrogen (No. 5) and air (No. 7) for parylene coated test patterns.

TABLE XI. COATED V. UNCOATED PATTERNS AFTER 1000 HOURS AT +150°C IN AIR

Identity	Avg. Percentage Change in Loss		
	2 GHz	8 GHz	12 GHz
Coated	+8.03	+10.54	+11.38
Uncoated	-1.50	+ 5.45	+ 8.08

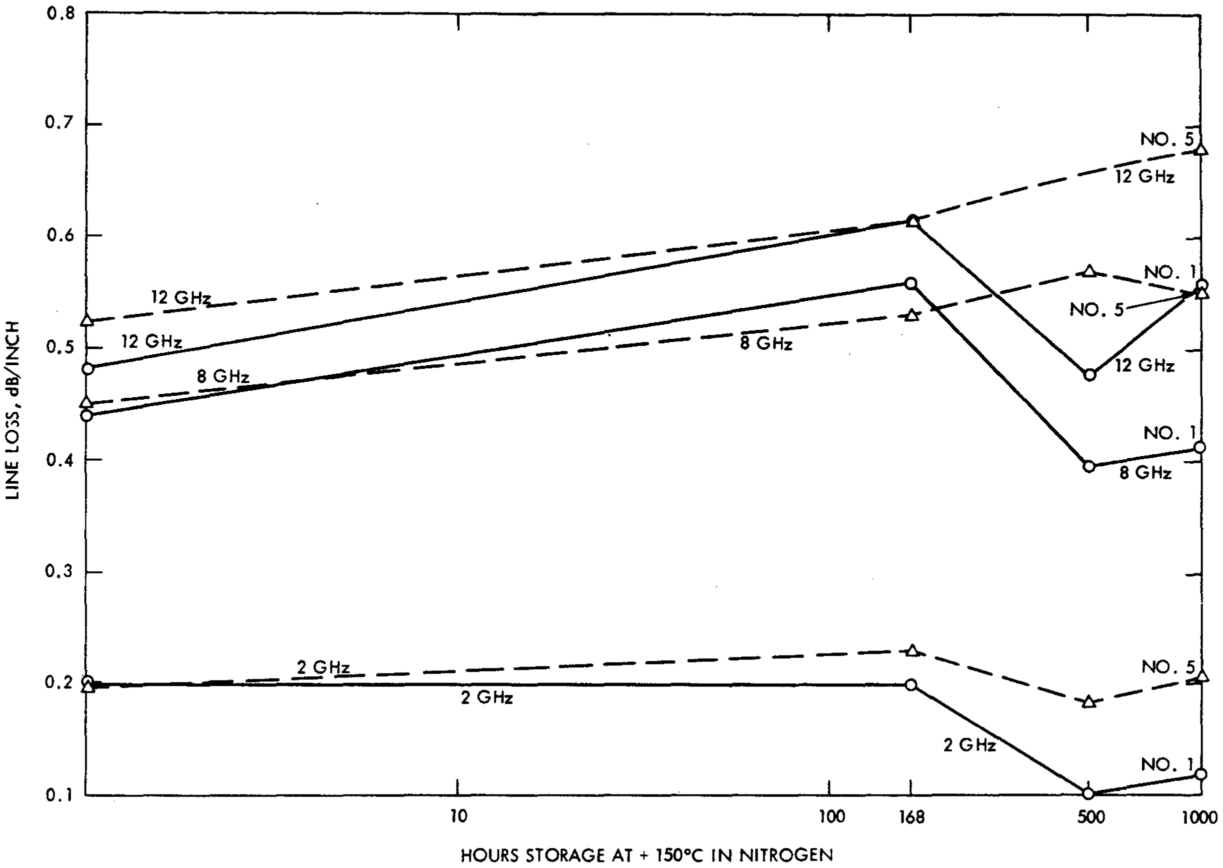


Figure 15. Plot of parylene coated pattern (No. 5) and uncoated pattern (No. 1) during 150°C nitrogen exposure at 2, 8, and 12 GHz.

change in loss at all frequencies. The change is positive, thus in the unfavorable direction.

Both coated and uncoated test patterns get worse as the frequency increases. This conclusion was consistent with all patterns, whether coated or uncoated.

Considering loss only, from Table 2 in Appendix B, the parylene coated patterns also exhibit greater absolute line loss than the uncoated parts after the 1000 hour air exposure.

Coated v. Uncoated Test Patterns After Nitrogen Exposure

A comparison was made between the parylene coated test patterns and the uncoated test patterns after exposure to +150°C for 1000 hours in a 99.9 percent pure nitrogen atmosphere. Table XII shows the results of this comparison. The values in Table XII are combined averages taken from Table 2 in Appendix B.

TABLE XII. COATED V. UNCOATED PATTERNS AFTER 1000 HOURS AT +150°C IN NITROGEN

	Avg. Percentage Change in Loss		
Identity	2 GHz	8 GHz	12 GHz
Coated	- 3.6	+22.76	+27.60
Uncoated	-35.26	- 6.14	+ 5.40

Figure 15 is a plot of actual line loss from Table 1 in Appendix B for a typical coated and uncoated test pattern. Plots of the other coated and uncoated patterns showed consistently similar trends.

From Table XII and Figure 15, it appears that the coated parts are worse than the uncoated parts at all frequencies. That is, the losses for the coated parts are higher and the average percentage change in loss is greater in a positive (or adverse) direction. As the frequency increases, both the coated and uncoated patterns show increased degradation. Considering only absolute line loss, after 1000 hours exposure in N_2 to +150°C, both the coated and uncoated patterns have approximately equal losses on the average at all frequencies. This is a realistic conclusion, since both coated and uncoated patterns had approximately equal loss values at zero hours prior to any environmental exposure.

Which is More Harmful to Parylene Coated Parts — Air Exposure or Nitrogen Exposure?

A comparison was made between those parylene coated test patterns exposed to air and exposed nitrogen for 1000 hours at +150°C. Table XIII presents a summary of this data analysis.

TABLE XIII. AIR V. NITROGEN EXPOSURE COMPARISON FOR PARYLENE COATED TEST PATTERNS

Identity	Avg. Percentage Change in Loss After 1000 Hr. at +150°C		
	2 GHz	8 GHz	12 GHz
Nitrogen Ambient	+9.3	+22.8	+27.6
Air Ambient	+8.0	+12.8	+15.9

Figure 14 is a plot of two typical parylene coated test patterns — one of which was contained in a nitrogen environment during the 1000 hours at +150°C while the other was exposed to an air atmosphere (i.e., 78 percent N_2, 21 percent O_2).

From an examination of Table XIII and Figure 14, it is clear that the parylene coated parts suffered less percentage change in loss in an air environment than in a nitrogen environment. This conclusion was consistent at all frequencies. This was somewhat unexpected, since the parylene parts exposed to air at +150°C discolored, flaked, and generally degraded in physical appearance.

All parts (whether exposed to N_2 or air) suffered greater degradation in line loss as the frequency increased.

Considering only absolute loss values (i.e., dB/inch), the nitrogen exposed parts were lossier than the air exposed parts after 1000 hours at +150°C.

CONCLUSIONS

1. Parylene is degraded physically and chemically by exposure to +150°C in an air environment. This degradation takes the form of discoloration, loss of adhesion, and embrittlement. Degradation was not evident for similar parylene coated parts protected by nitrogen.
2. Coating a microwave integrated circuit transmission line with parylene will not cause any increase in insertion loss. In fact, loss is slightly improved at frequencies from 2 to 12 GHz.
3. After exposure to +150°C for 1000 hours, parylene coated parts are lossier than uncoated parts at all frequencies. This applies regardless of whether the parts are maintained in an air or nitrogen environment.
4. Percentage change in line loss for both coated and uncoated gets worse as the frequency increases. This applies regardless of whether the parts are maintained in an air or nitrogen environment.
5. An air environment causes less percentage change in loss than a nitrogen environment for parylene coated parts. Nitrogen exposed parts were lossier than air exposed parts at all frequencies from 2 to 12 GHz.

EFFECTS ON FILM RESISTORS

FABRICATION AND PROCESSING OF FILM RESISTOR SPECIMENS

Thin film patterns were made from 99.5 percent alumina substrates previously metalized by vapor deposition techniques followed by electro plating with gold, the gold being retained for conductors and nicrome retained for resistor elements. Thick film patterns (used only for thick film resistors and associated conductors) utilized the following materials on 96 percent alumina substrates:

Conductor Ink – ESL #8835 gold
Overglaze Ink – ESL #4771B
Resistor Ink – ESL #3811 ($10\,\Omega/\Box$)
Resistor Ink – ESL #38-44 ($1000\,\Omega/\Box$)
Resistor Ink – ESL #38-47 ($100,000\,\Omega/\Box$)

All resistor patterns were stablized at 325°C for one hour prior to trimming. Trimming of thin film resistors was accomplished by use of YAG laser while thick film resistors were trimmed with an air/partical abrasion method. All resistors were trimmed to values five percent higher than their initial value to introduce the typical condition of trimmed resistors. After trimming all substrates patterns were bonded into hybrid packages using an epoxy adhesive. Functional electrical interconnections were made with 0.002 inch diameter gold wire. Figures 16a, b show the various resistor patterns used. Prior to sealing, the packages were vapor degreased in electronic grade freon, primed and/or coated with parylene, baked one hour in nitrogen at 125°C and then sealed as required to provide an atmosphere of dry nitrogen, approximately 96 percent N_2 –/4 percent O_2 or air. Parts were then leak checked to verify sealing.

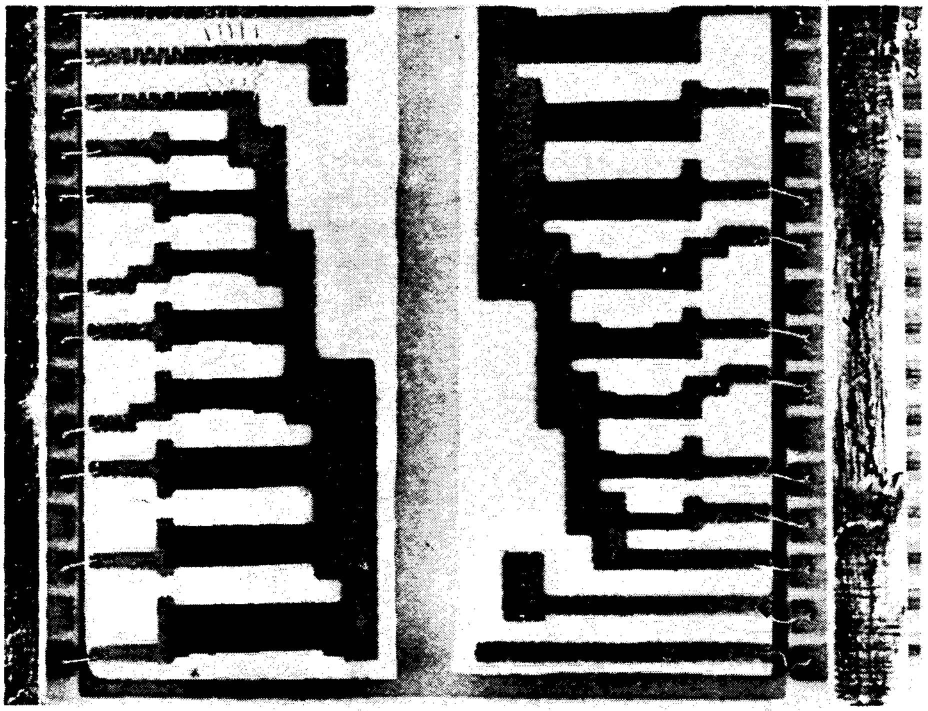


Figure 16a. Thick film resistor patterns.

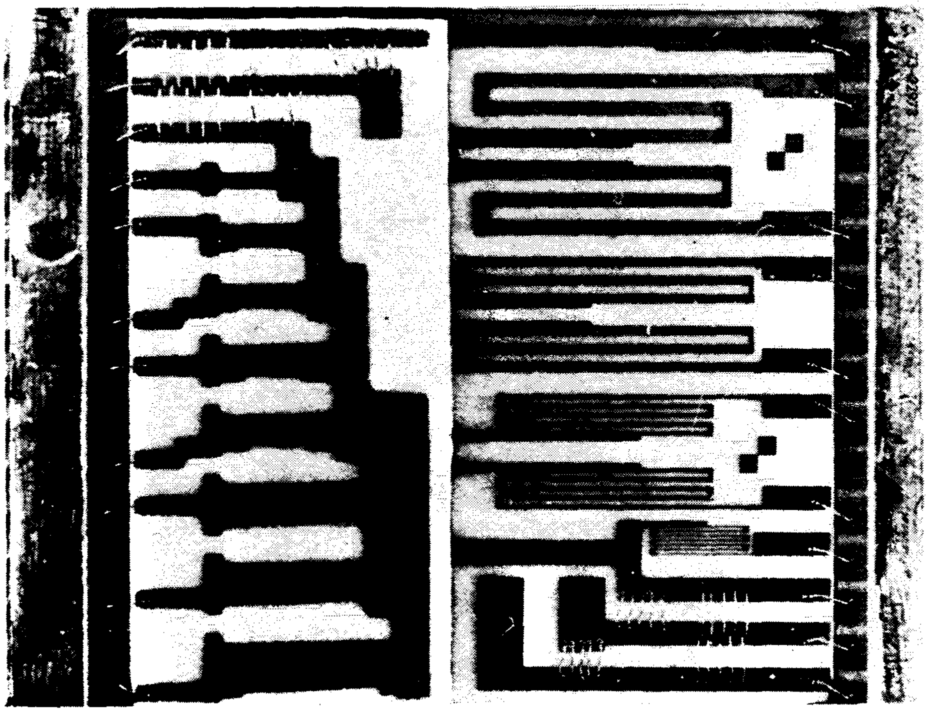


Figure 16b. Combination thick and thin film resistor patterns.

RESISTOR TEST RESULTS

This program involved the testing of 144 thin film resistors and 336 thick film resistors. A total of 2400 resistance measurements were made throughout the evaluation.

Appendix C contains the data sheets which represent a statistical data reduction of all the resistor information. Included are separate tables for:

1. Thin film resistors
2. Glazed thick film resistors
    a. 10 Ω/□ ink
    b. 1 KΩ/□ ink
    c. 100 KΩ/□ ink
3. Unglazed thick film resistors
    a. 10 Ω/□ ink
    b. 1 KΩ/□ ink
    c. 100 KΩ/□ ink

The data in these tabulations represents the average percentage change in resistance value:

1. after cleaning and/or parylene coating
2. after exposure to +150°C for 1000 hours in either air, 99.9 percent nitrogen, or 95.3 percent N_2 - 4.7 percent O_2.

Each individual value in the data sheets in Appendix C represents an average percentage change in resistance of 8 resistors (thick film data) or 16 resistors (thin film data).

A separate column in each table in Appendix C shows the approximate percentage of total resistance change which occurred between 500 and

1000 hours exposure to +150°C. It is interesting to note that in practically all cases, the majority of resistance change (usually from 75 percent to 90 percent) occurred during the last 500 hours of exposure. This was true regardless of whether the resistors were thick or thin film types, glazed or unglazed, or were parylene coated or uncoated. The only exceptions to this phenomenon were with the 10 Ω/□ thick film resistors.

ANALYSIS AND DISCUSSION OF RESISTOR TEST RESULTS

Thin Film Resistors

From Table 1 in Appendix C, it is obvious that coating the thin film resistors with parylene (primed or not) has no effect on the resistance value. The largest changes, 0.03 percent to 0.05 percent, occurred on the uncoated resistors.

Analyzing the percentage changes in resistance after life testing reveals that neither parylene coating, nor the use of a primer, nor the sealing atmosphere has any appreciable effect on the thin film resistors. All resistors increased slightly in value after life testing. However these increases, which ranged from a low of 0.30 percent to a high of 0.47 percent, were not significantly different in any category. For example, the uncoated resistors changed as much as the coated, and resistors sealed in a pure nitrogen atmosphere changed as much as those sealed in air.

The only significant thing which can be deduced from this data is that, taken as a whole, the thin film resistors exhibited less percentage change than thick film resistors after life testing. Only one type of thick film resistor (i.e., glazed 100 KΩ/□ ink) showed percentage changes comparable to those of thin film resistors.

Thus parylene has not helped stabilize the thin film resistors, nor has it degraded them. Nor does priming help or harm the thin film resistors. Nor are the thin film resistors sensitive to the sealing atmosphere. In other words, nothing appears to affect their stability.

Glazed Thick Film Resistors

From Tables 2, 3, and 4 in Appendix C, it appears that coating a glazed thick film resistor with parylene causes no appreciable change in resistance value (usually from 0 percent to 0.02 percent). These small changes are within the error of the last digit on the Automatic Digital Ohmmeter used for resistance measurements. A few larger changes occurred with the 10 Ω / □ ink on those substrates sealed in air (from 1 percent to 3 percent). This anomaly is unexplained, but cannot be attributed to the parylene coating. Rather it would appear to be related to the air ambient within the package.

Analyzing the life test data, the following trends are observed:

a. The coated resistors exhibit less percentage change than the uncoated resistors.

b. The parylene coated resistors without primer exhibit less percentage change than the primed resistors.

c. For the 1 KΩ/□ and 100 KΩ/□ ink types, the 4.7 percent O_2 ambient is superior to a pure nitrogen ambient.

d. For the 10 Ω / □ ink type, those resistors sealed in an air ambient exhibited less percentage change than those sealed in pure nitrogen or 4.7 percent O_2.

Thus it can be concluded that parylene coating of the glazed thick film resistor definitely helps stability. It would be better if the primer were omitted. The more oxygen in the sealing atmosphere the better.

Furthermore, it is observed that, regardless of the other parameters, the 100 KΩ/☐ ink is the most stable, exhibiting resistance changes less than 0.35 percent after 1000 hours exposure to +150°C. The 10 Ω/☐ ink was the least stable.

Unglazed Thick Film Resistors

From Tables 5, 6, and 7 in Appendix C, it appears that coating of the thick film resistors with parylene causes no appreciable change in resistance value (usually from 0.02 to 0.05 percent) for the 1K and 100 KΩ/☐ inks. The 10 Ω/☐ ink, however, exhibited percentage changes up to 2.5 percent simply due to cleaning or coating. This same anomaly was observed previously with the glazed 10 Ω/☐ resistors. The change was the greatest with those resistor sealed in the air ambient.

Analyzing the life test data, the resistors tended to increase in value as a result of life testing. This was also true of the glazed thick film resistors. Another general observation is that the unglazed resistors were less stable than the glazed resistors, regardless of whether they were parylene coated or not, primed or not, and regardless of the sealing atmosphere.

For all three ink types, the uncoated resistors exhibited less percentage resistance change after life testing than the parylene coated resistors. Plain parylene was slightly better than the primer-plus-parylene. The results for the different sealing atmospheres were inconsistent and no definite conclusions could be drawn.

As with the glazed resistors, the 100 KΩ/□ ink was the most stable, with changes averaging less than 2 percent after life testing. The 10 Ω/□ ink was extremely poor, showing resistance increases as high as 23 percent after life testing in a pure nitrogen atmosphere.

CONCLUSIONS

General

Coating (before life testing) of the thick or thin film resistors with parylene (whether or not a primer was used) caused no appreciable change in resistance value — usually about 0.02 to 0.05 percent. After life testing the thin film resistors were more stable than the thick film resistors, with average percentage resistance increases less than 0.5 percent after 1000 hours at +150°C. Of the three thick film resistor ink types, the 100 KΩ/□ ink was the most stable. The 10 Ω / □ ink was the least stable. The glazed thick film resistors were more stable than the unglazed resistors.

Thin Film Resistors

Thin film resistors were not significantly affected by parylene, by a primer, or by either air, nitrogen, or 4.7 percent oxygen sealing ambient. Parylene had no effect — it neither helped nor harmed them.

Glazed Thick Film Resistors

A parylene coating aided in the stability of these resistors. The stability was better without the primer. The more oxygen in the sealing atmosphere the better. Although parylene proved helpful, the stability was only improved by less than 0.2 percent. The expense of parylene coating may not justify its use simply to improve stability.

Unglazed Thick Film Resistors

A parylene coating decreased resistor stability slightly. However the average reduction in stability due to parylene coating was only 0.5 percent after life testing for the 1 KΩ/□ and 100 KΩ/□ inks. As with the glazed resistors, the use of a primer is not recommended. Thus, it could be concluded that if parylene is used with thick film resistors, the primer should be omitted and the resistors should be overglazed. Sealing in pure nitrogen is of no help in improving resistor stability.

SEMICONDUCTOR DEVICE SURFACE EFFECTS

GENERAL DISCUSSION

The principal objective of this portion of the study was to evaluate the effects of parylene coating on the electrical performance and reliability of semiconductor devices.

All anticipated device effects are associated with changes occurring at or near the semiconductor surface. These changes relate to shifts in the Fermi level at the surface due to changes in surface state density and energy distribution. Such changes in surface Fermi level are reflected in corresponding modifications of the magnitude and sign of the surface conductivity as well as the value of minority carrier lifetime.

A shift of the Fermi level due to surface state changes may move it away from the midgap position to produce an enhancement layer. A movement toward the midgap position will produce a depletion layer. A movement across the midgap position will produce an inversion layer at the surface. The creation of an inversion layer which may be a topological extension of bulk junctions at the semiconductor surface has the effect of adding to junction area thus increasing junction capacitance and reverse leakage. A reverse biased inversion layer is termed a "channel." The reverse bias required to produce such a channel from a simple unbiased inversion layer is available from the reverse voltage applied to an associated bulk p-n junction. A surface channel is thus topologically an extension of and electrically in parallel with the reverse biased bulk junction. The depletion region associated with the channel is generally narrower than that of the associated bulk junction and hence the reverse breakdown of the surface channel portion of the total junction area will be lower. Channels, then, act to reduce reverse breakdown and increase reverse leakage and capacitance of junction containing devices.

In addition decreased minority carrier lifetime at the surface also affect device characteristics by increasing junction leakage according to the well known p-n junction diode law:

$$I = I_o(e^{qV/kt}-1)$$

in which:

$$I_o = q\left[\sqrt{\frac{D_p}{\tau_p}}\, Pno\ t \sqrt{\frac{D_n}{\tau_n}\, Npo}\right]$$

where:

V = Applied voltage

I = Diode current

I_o = Diode reverse saturation current

D_p and D_n = Diffusion constants for holes and electrons respectively

P_{no} and N_{po} = Equilibrium densities of holes and electrons respectively as minority carriers

p and τ_n = Minority carriers lifetime of holes and electrons respectively

q = Electronic charge

k = Boltzman's constant

T = Absolute temperature (^{o}k)

The above discussion treats the effects of surface channels on diode characteristics. The existence of surface channels in transistors will produce various effects, upon electrical characteristics depending upon whether or not the conductive channel bridges the base of the transistor. Collector channels produce the usual diode degradation of the collector junction as described above. Base channels produce emitter – collector leakage paths and degraded transistor gain (reduced β values).

FABRICATION AND PROCESSING OF ACTIVE DEVICES

All semiconductor chips were mounted on TO-100 or TO-8 headers. Chips requiring more than four leads were mounted in individual headers while those with four leads or less, were mounted with several chips per header. Chips were bonded to the headers using a silver filled epoxy intended for semiconductor use. Sufficient chips for each type of device and variable studied were prepared to produce five working chips each. Interconnections were made with 0.001 inch diameter aluminum wire. All mounted and wired devices were then vapor degreased in electronic grade freon followed by thorough rinsing in electronic grade methanol. Parts were then blown free of methanol with dry nitrogen followed by a two hour bake in a clean air oven at 125°C.

Cleaned parts were then stored in sealed bags during the short interval of time required to transport and hold the parts for coating with parylene. Those parts requiring priming were primed with Union Carbide's A-174 silicon primer from a 0.1 percent by volume electronic grade methanol/distilled water solution. A typical mounted chip is shown in Figure 17 as it appears in the masking tooling just prior to placing in the coating chamber for coating.

After coating the parts were baked for one hour at 125°C in dry nitrogen. Using an integral glove box/resistance welding station the parts were sealed by having lids welded in place while the glove box atmosphere was controlled at 99.9 percent N_2 or approximately 96 percent N_2/4 percent O_2. Subsequently all parts were leak check to determine that adequate sealing had occurred. To interpret the symbology used in the following data analysis and graphical presentation refer to the list of abreviations on page 79.

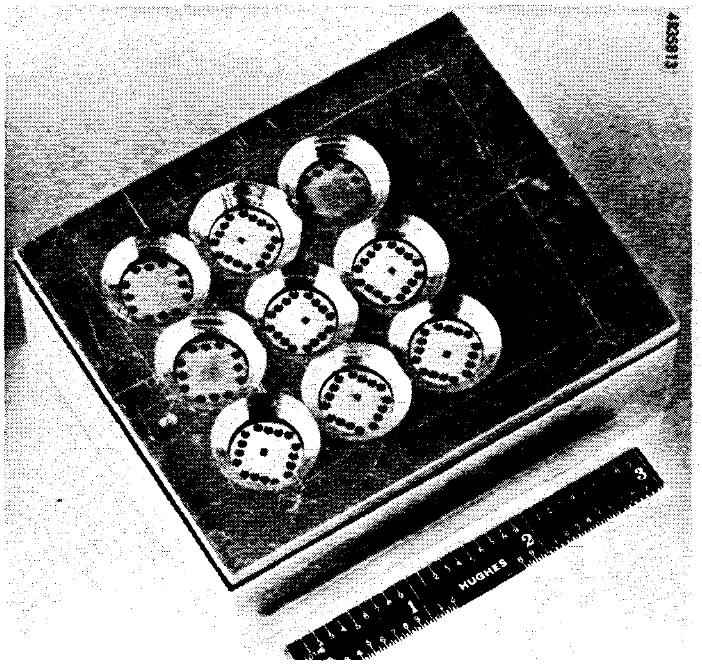


Figure 17. Active device mounted on TO-8 header ready for coating.

Discussion of Experimental Results

In order to determine the possibility of parylene surface treatments leading to such surface modifications and consequent degradative device parameter changes, a set of selected test devices were chosen for study. These devices were selected for their high sensitivity to surface condition.

A tabulation of the device types employed and the specific reasons for their selection is presented in Table XIV.

TABLE XIV. ACTIVE DEVICE SELECTION CRITERIA

Device	Reason for Selection
PIN Diode	High resistivity I layer is relatively easily modified in surface conductivity magnitude and sign.
PNP and NPN High gain Transistor	The high B values of these devices are sensitive to surface state condition and the existence of conductive channels over the base. Complementary devices chosen to determine effective n or p type doping effect of applied parylene film.
MOSFET (N channel)	The action of a MOSFET device is wholly dependent upon surface condition in the gate region.
C-MOS Device (N and P channel)	This device was selected primarily because of the fact that it contains both p and n channel IGFET gates. Thus a given surface impurity which could not shift threshold voltage of one gate type might cause shifts in the other gate type.
Operational Amplifier	This integrated circuit consists of a large number of active device elements arrayed together in a single circuit (op-amp). A shift in one or more of these elements due to surface contamination will sensitively affect the overall operation of the whole device.

In evaluating the effects of paralyene coating on each device type, three types of uncoated control(untreated)units where included. One type was intended for room temperature exposure as a calibration standard or test equipment check. The other two were intended for exposure at elevated temperature along with coated devices in either N_2 or 96 percent N_2/4 percent O_2.

Elevated temperature controls and separate groups of coated and primed and coated units for each device type were then subjected to high temperature reverse bias (HTRB) in high temperature in inert gas (N_2) and in N_2 containing O_2 (four percent) in order to simulate air contamination during sealing the device package. The HTRB condition facilitates the growth of inversion layers and channels if the contamination condition of the surface is such as to allow it.

ANALYSIS OF DATA

A. PIN Diode (Hewlett Packard — HP5082-3046)

As indicated in the data graphically presented in Figure 18 this device type shows an increase in reverse leakage for all treated and untreated (control) units with a slow and partial recovery approaching the original low leakage values. An important observation here is that all devices behave similarly and thus the observed increased leakage during HTRB cannot be due primarily to the primer or parylene treatments but to the normal behavior of the device-package complex itself. The observed behavior is normal for such highly surface sensitive devices. The observed drifts do not appear to be due to the treatments but rather to the normal behavior of the very sensitive PIN device itself. The primer however appears to have an initial effect on these devices and may be cause to question its use.

B. NPN-PNP Transistors (Motorola 2N3501 and Motorola 2N3637 respectively)

Both of these devices exhibited some variation in collector reverse leakage current (I_{CBO}) during 1000 hours HTRB as shown in Figure 19a and 19b. No consistent trend is apparent that is traceable to the coating or the use of the

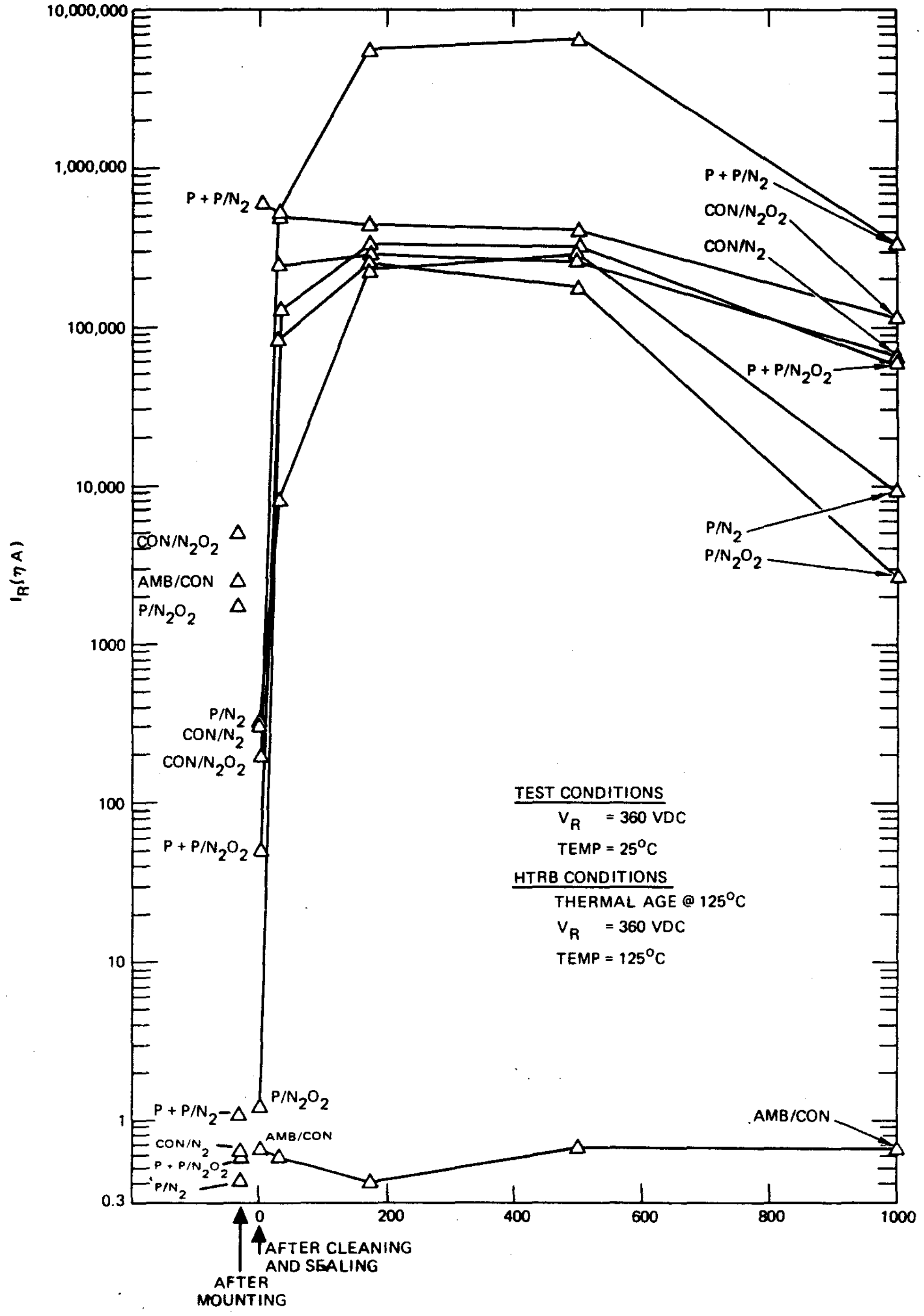


Figure 18. Pin Diode - Hewlett Packard (HP 5082-3046) (Requirement: $I_R \leq 10,000$ ηA)

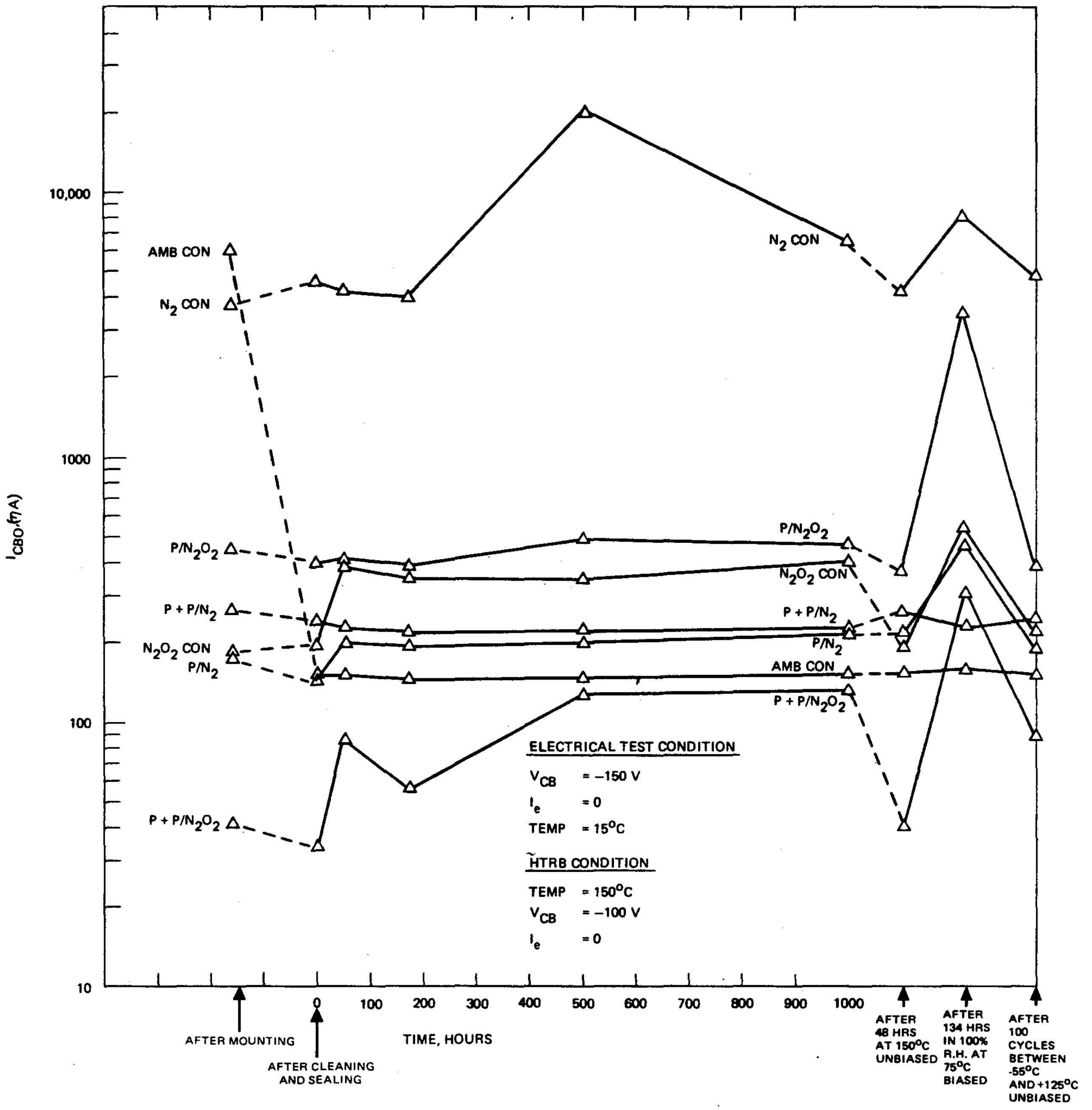


Figure 19a. PNP transistor (2N3637) collector to Base Cutoff Current. (Requirement: $I_{CBO} \leq 1000\ \eta A$)

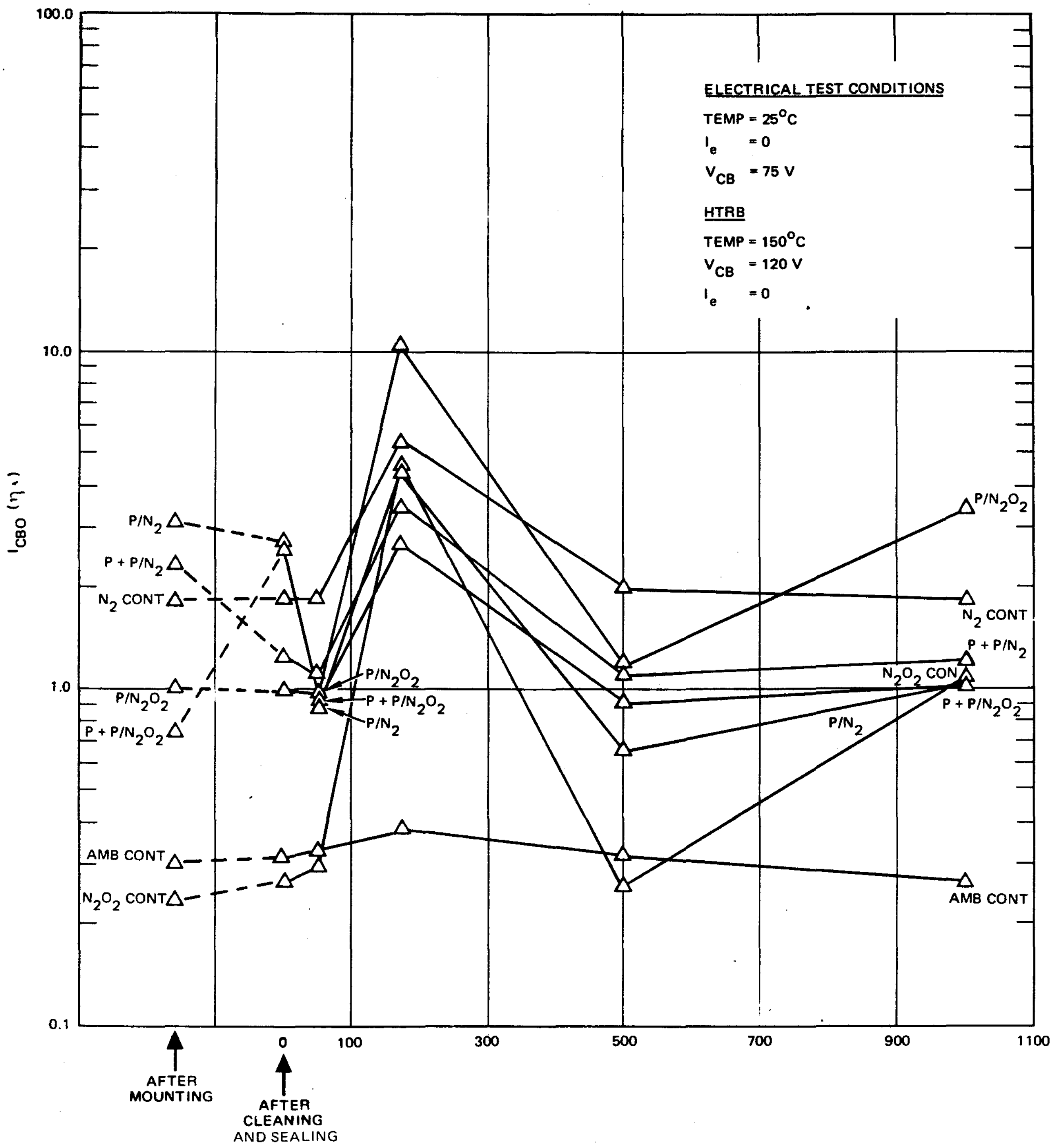


Figure 19b. NPN transistor (2N3501) (933012-4C or 4D) collector to base cutoff (I_{CBO})
(Requirement: ≤50 ηA)

primer prior to coating. Further, the presence of oxygen in nitrogen did not appear to produce different results from that obtained with parts exposed to relatively pure nitrogen. All parts appear to follow some pattern over time, but when compared to the various controls this pattern appears to be due to minor variations in test conditions rather than surface conditions.

The transistor gain (h_{fe}) shown in Figure 20a and 20b exhibits a similar performance as above with a pattern over time, probably again the result of minor variations in test conditions. In no case do there appear to be any obvious deleterious effects traceable to the coating or primer.

The PNP device was selected for additional testing which consisted of the following exposures after completion of the initial 1000 hours of HTRB:

48 hours at 150°C unbiased

134 hours at 75°C, biased under 100% R. H.

100 temperature cycles between -55°C and +125°C
(12 minutes at each extreme and 6 minutes at R. T.)

The 48 hour exposure at 150°C without bias was intended to return the device characteristics to the initial state. As can be seen in Figure 19a, I_{CBO} did return to levels very close to the original 0-hour values. h_{fe} either returned to near 0-hour values or fell slightly below these values.

Subsequent to this unbiased exposure to 150°C, the seals on all devices were intentionally broken to allow exposure while biased to high humidity at elevated temperature. I_{CBO} was monitored during the test at temperature in addition to measurements at room temperature before and after exposure. As expected, I_{CBO} was greater at 75°C as compared to values recorded at room temperature. Generally this increase ranged between 200 and 700 percent, with coated or primed and coated parts responding similarly to

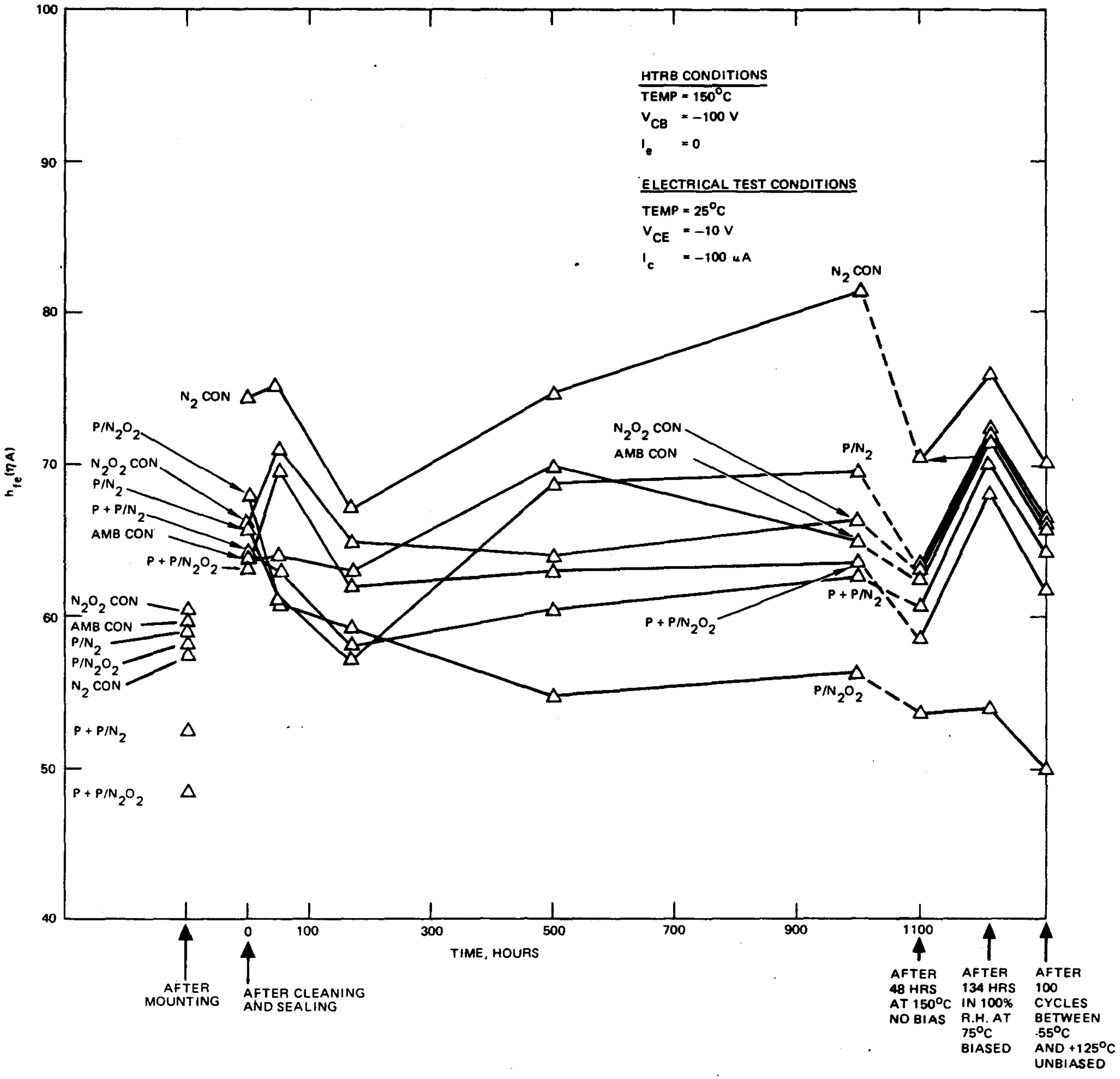


Figure 20a. PNP transistor - Motorola (2N3637) (933013-1C, 1D)
DC forward current gain (h_{fe})
(Requirement: $h_{fe} \geq 40$)

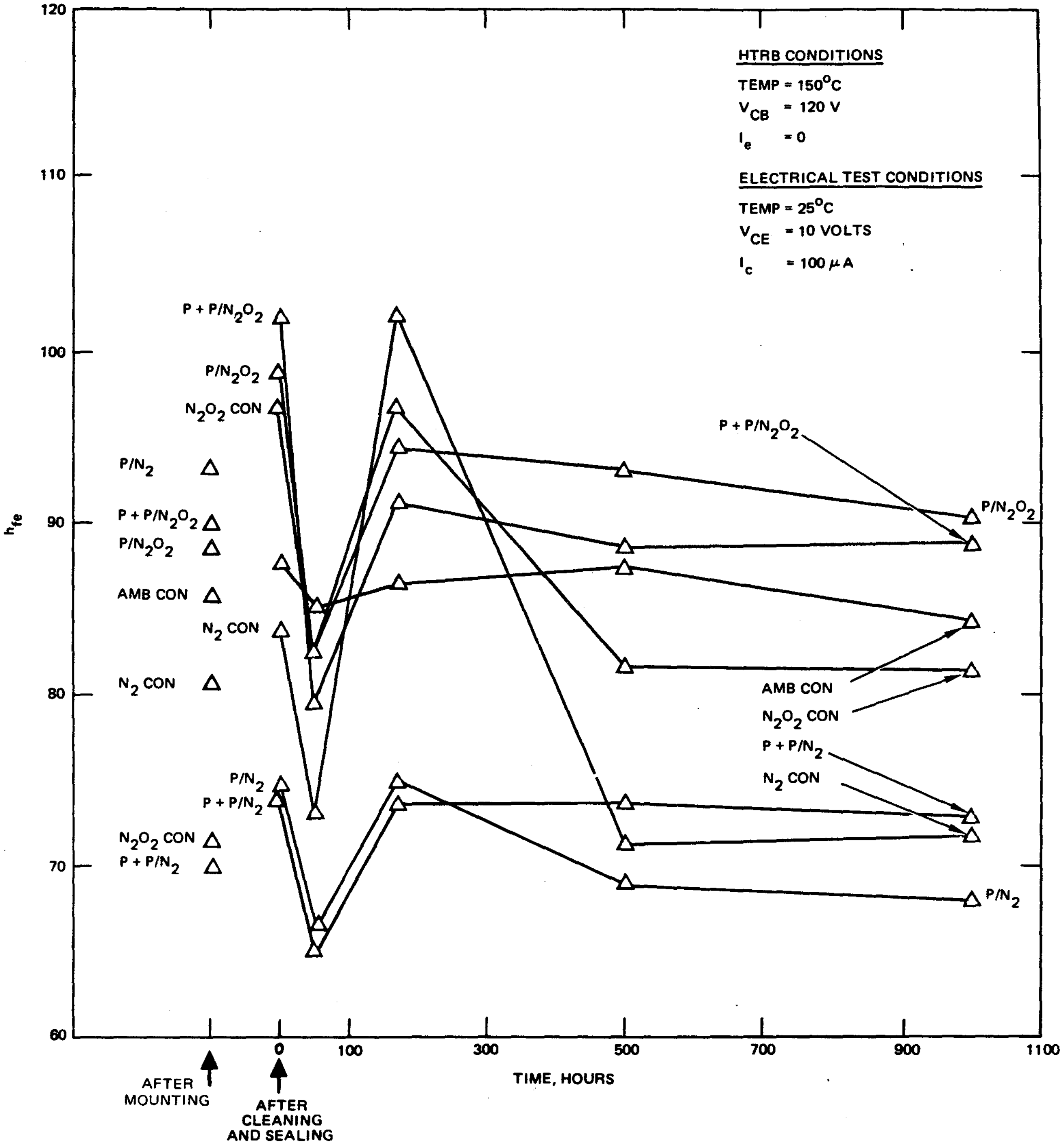


Figure 20b. NPN transistor (2N3501) DC forward current gain (h_{fe}) (Requirement: $h_{fe} \geq 35$)

uncoated parts. The subsequent exposure at 75°C in 100% relative humidity had varying effects, with those samples which were previously exposed to a nitrogen environment during initial HTRB testing showing the least additional change (averaging ±20%). The samples which were previously exposed to oxygen contaminated nitrogen appeared to be somewhat more affected by humidity showing an additional change of approximately +100% or a doubling of the I_{CBO} at 75°C without humidity. This effect on elevated temperature I_{CBO} is not shown in Figure 19a. No difference, however, was noted between the coated, primed and coated, or uncoated specimens. Figure 19a shows the R. T. change in I_{CBO} after this humidity exposure. All specimens showed an increase except the specimens which were primed, coated and subjected to the initial 1000 hours of HTRB in nitrogen. No explanation is apparent for why this combination appeared more immune to humidity. As shown in Figure 20a, h_{fe} also increased, but since a similar increase occurred with the ambient control, the change can be considered to be due to slightly different measuring conditions.

As a final test the PNP devices were exposed unbiased to 100 cycles of temperature cycling between -55°C and +125°C. In general this exposure tended to return all parts to their original values which is not unexpected. It was expected, however, that condensation would occur during the cycling and that the aluminum to gold wire interconnections might be damaged due to corrosion. This was felt to be particularly true for the uncoated specimens; however both coated and uncoated parts survived without apparent damage. A more severe exposure to liquid moisture would probably have shown this expected difference.

It can be concluded that although no beneficial effects of the coating were disclosed from this testing, coated parts or primed and coated parts performed at least as well as uncoated parts during HTRB, high humidity and temperature cycling conditions.

C. N Channel MOSFET (Motorola, MFE 3004/3005)

The source drain resistance (r_{ds}) and the gate leakage current (I_{gss}) and the zero-gate-voltage drain current (I_{Dss}) for a set of N channel devices as a function of time to 1000 hours as shown in Figures 21 and 22a and b.

Again, as in previous devices, a systematic change in r_{ds} and I_{gss} is observed in both treated and untreated control units. These changes are due to "normal" shifts in device behavior and/or minor changes in test conditions and are not principally due to the surface coating treatments since similar changes are observed in untreated control devices. Again there appears to be no obvious difference between coating with or without a primer nor any obvious difference in exposing the parts to nitrogen or nitrogen contaminated with approximately 4 percent oxygen. r_{ds}, I_{gss}, and I_{Dss} improved somewhat over time for all parts when compared to the untreated room temperature control.

D. Operational Amplifier (National Semiconductor CD-108)

The data graphically shown in Figure 23a and b showing the effects of surface priming and paralyne treatments on device electrical characteristics with controls illustrate again the systematic behavior of the device itself independently of the surface treatments. Both treated devices and elevated temperature controls exhibit similar behavior. The observed shifts can therefore be attributed to the peculiarities of the device itself and/or minor

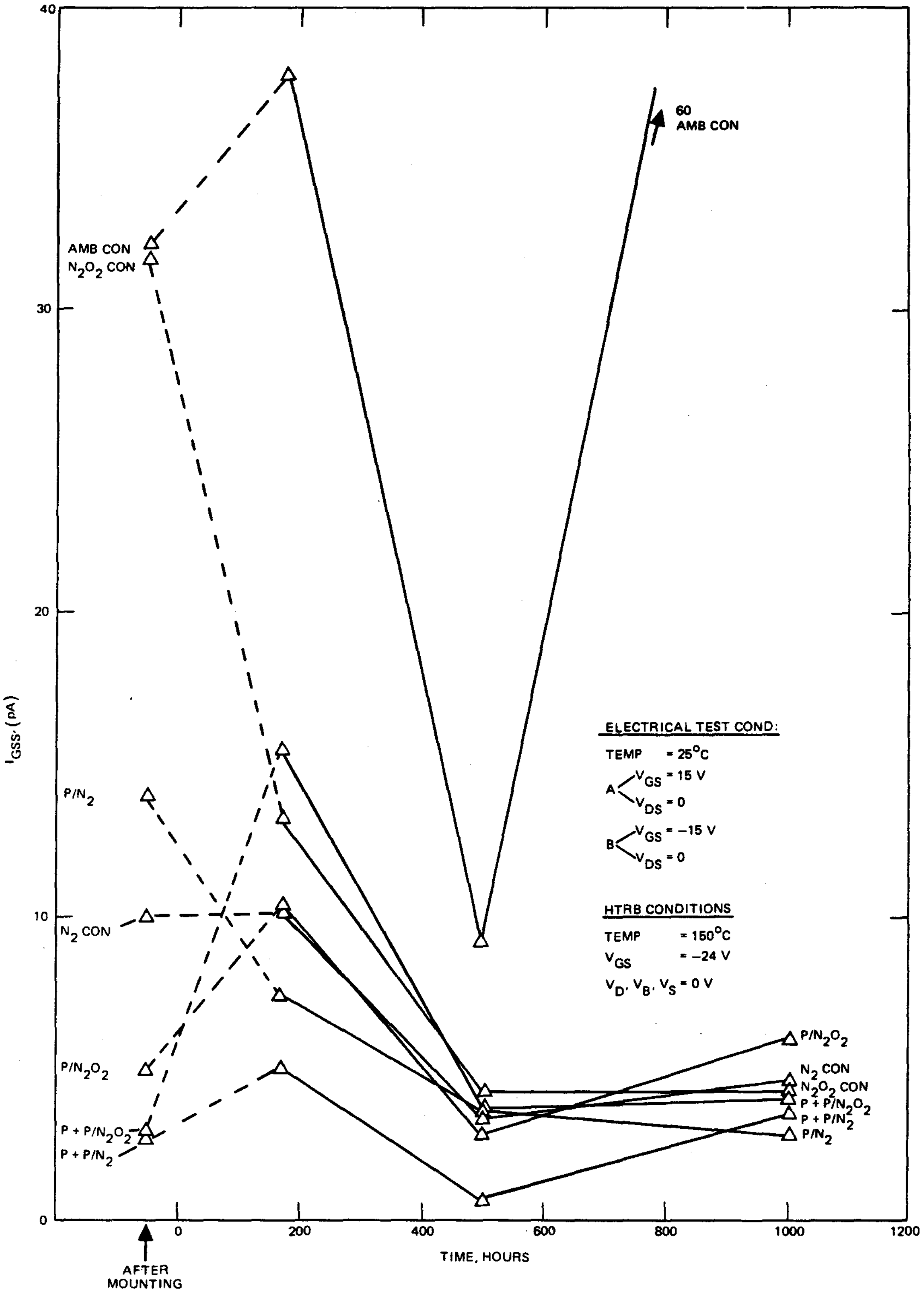


Figure 21. N-channel MOSFET gate reverse current (I_{GSS}) (Requirement: $I_{GSS} \leq 50$ pA)

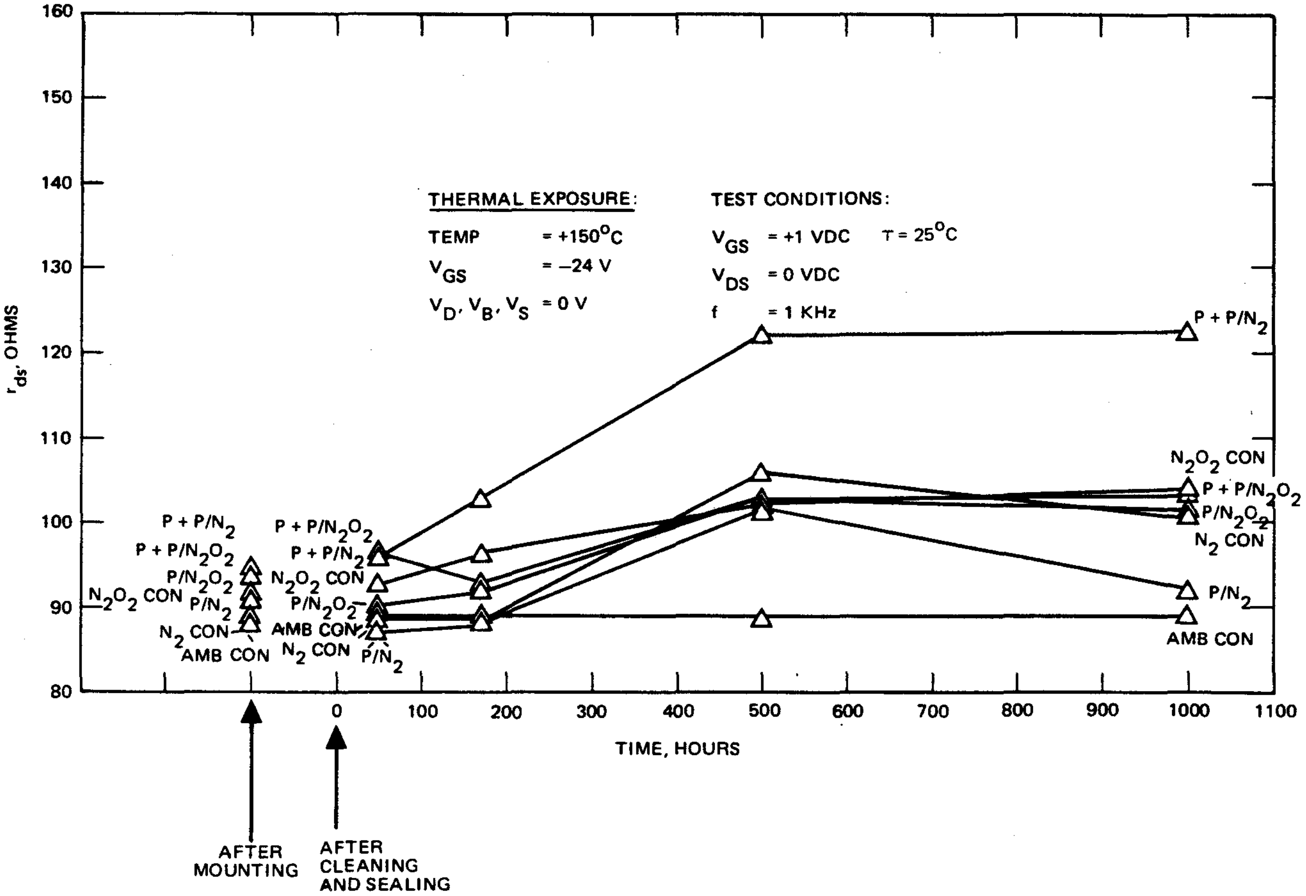


Figure 22a. N-channel MOSFET (Motorola MFE 3004/3005) drain to source on-state resistance (r_{ds}) (Requirement: $r_{ds} \leq 100$ ohms)

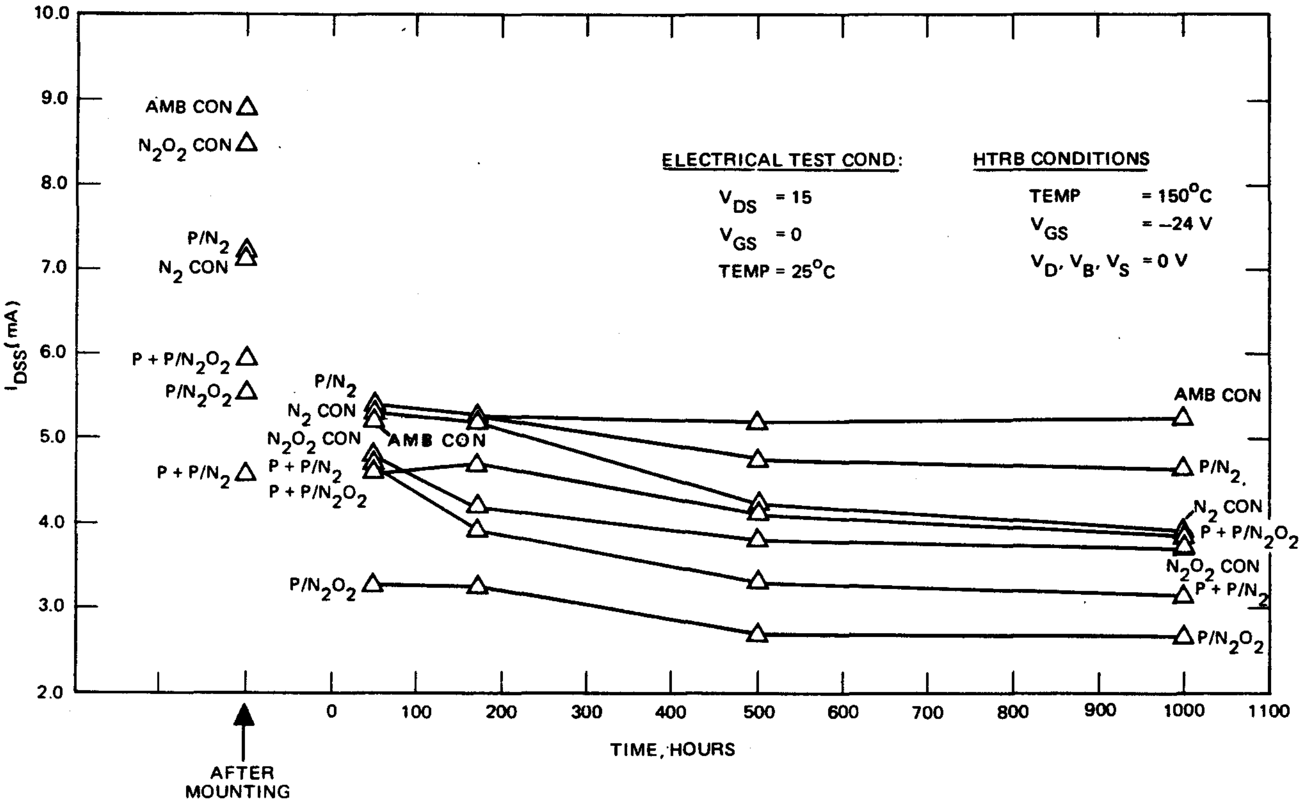


Figure 22b. N-channel MOSFET zero-gate-voltage drain current (I_{DSS}) (Requirement: $I_{DSS} \leq 10$ mA)

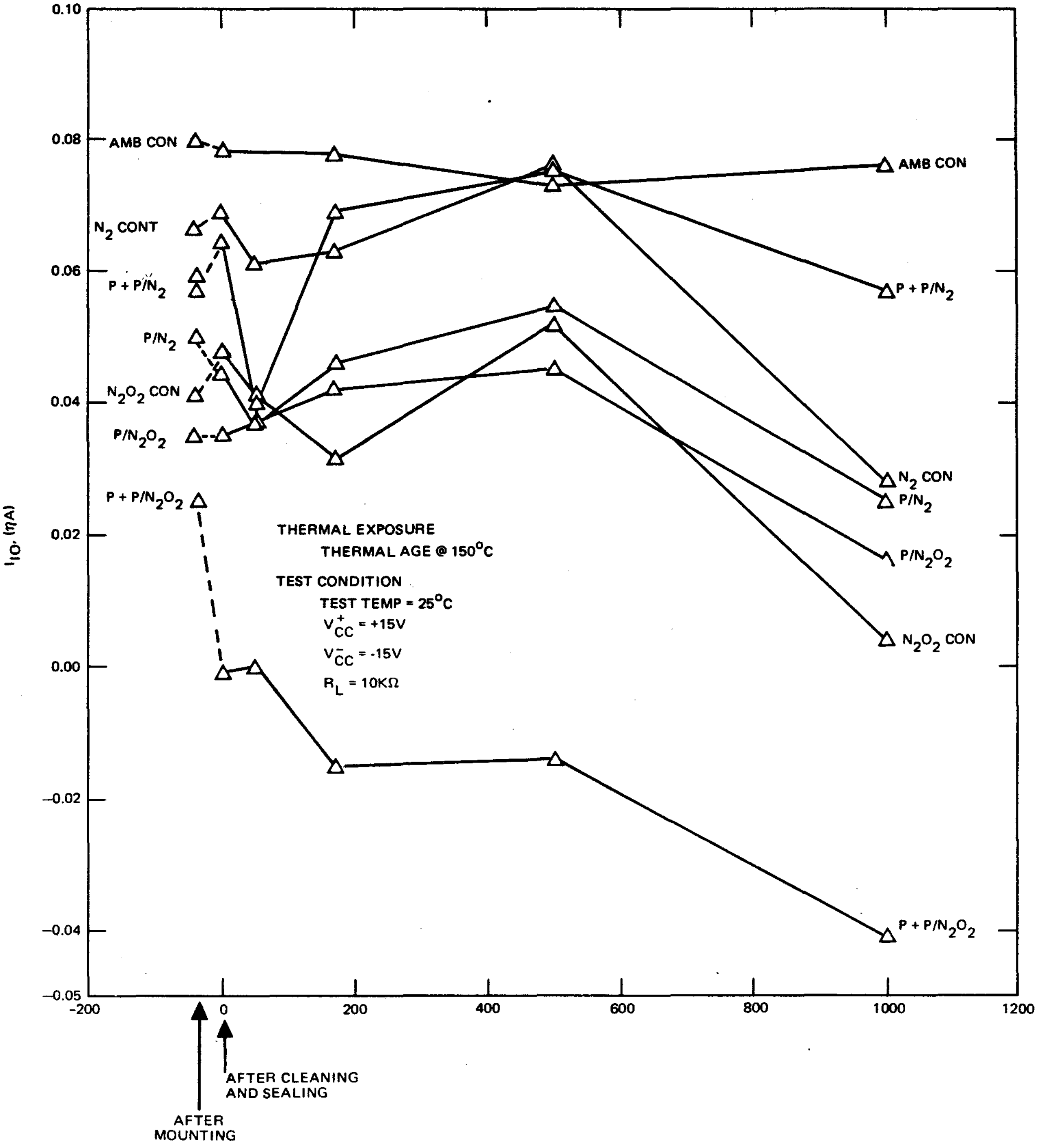


Figure 23a. OP AMP - NAT semiconductor (CD-108) (933429) input offset current (I_{10})
(Requirement: $I_{IO} \leq 0.2$ ηA)

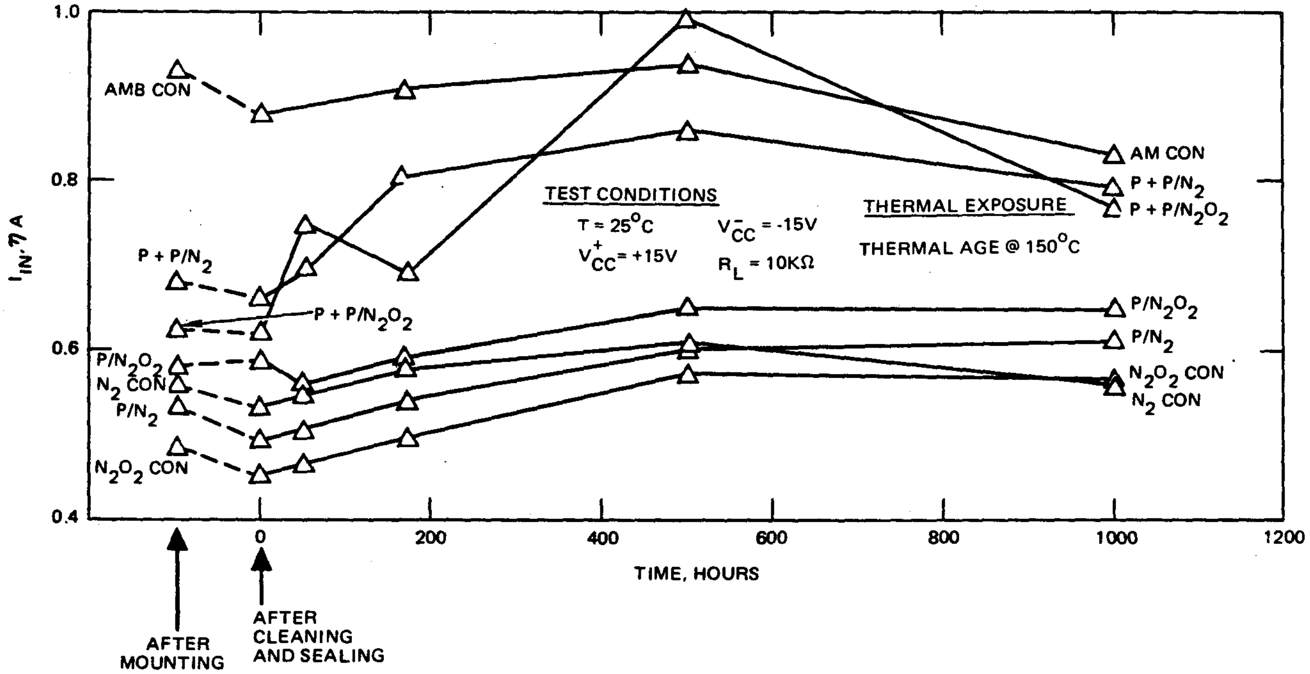


Figure 23b. OP AMP (CD-108) (933429) input bias (I_{IN})
(Requirement: $I_{IN} \leq 2\ \eta A$)

changes in test conditions rather than the effect of priming and coating. Again there appeared to be no obvious difference between exposure to nitrogen or nitrogen containing approximately 4 percent oxygen. Changes in input offset current (OI_{IO}) and input bias (I_{IN}) were generally minor and well within requirements.

E. C-MOS Device (N and P channel) (RCA CD4007AH)

These devices were not biased during 1000 hour high temperature treatment. Neither were the control units subjected to extensive cleaning as were the surface treated units. Even a casual inspection of the data tabulated in Table XV shows results essentially in conformity with those obtained on other devices studied in the program. The drain leakage current and the gate threshold voltages for both N and P channel IGFET's were monitored.

TABLE XIV - C-MOS (N and P channel) (RCA CD4007AH)

Specimen Description	Average Initial Value (mA)	Effect of Cleaning and/or Coating (% Change in Absolute Value)	Subsequent Change after Exposure to 150°C (% Change in Absolute Value)
I_D^P – Req: -1.1 mA Minimum			
Amb Control	-2.410	+2.49	-3.24
N_2O_2 Control	-2.385	-4.32	+4.43
$P+P/N_2O_2$	-2.386	-5.87	+5.39
P/N_2O_2	-2.492	-6.38	+5.87
N_2 Control	-2.407	-4.24	+2.43
$P+P/N_2$	-2.453	-1.83	-0.08
P/N_2	-2.415	-4.76	+2.35
I_D^N – Req: +1.3 mA Minimum			
Amb Control	3.020	+1.32	-2.61
N_2O_2 Control	2.964	+1.18	-5.00
$P+P/N_2O_2$	3.019	+3.246	-1.03
P/N_2O_2	2.908	+3.78	-2.15
N_2 Control	3.035	+0.49	-3.18
$P+P/N_2$	2.82	+4.61	-2.44
P/N_2	2.948	+5.46	-2.67
V_{TH}^P – Req: -1.5V Typical			
Amb Control	-1.55	-0.32	-0.32
N_2O_2 Control	-1.51	-0.40	-0.40
$P+P/N_2O_2$	-1.52	-0.26	+0.33
P/N_2O_2	-1.51	-0.26	-0.06
N_2 Control	-1.53	-0.13	0.00
$P+P/N_2$	-1.51	-0.60	0.40
P/N_2	-1.52	-0.40	0.13
V_{TH}^N – Req: +1.5V Typical			
Amb Control	+2.32	+0.43	-0.86
N_2O_2 Control	2.26	-0.18	-0.09
$P+P/N_2O_2$	2.25	-1.20	+0.90
P/N_2O_2	2.32	+0.08	-0.30
N_2 Control	2.25	+0.13	-0.27
$P+P/N_2$	2.30	-0.13	-0.26
P/N_2	2.28	+0.04	-0.44
I_L – Req: 0.1 μA Maximum			
Amb Control	≤ 0	Not Detectable	Not Detectable
N_2O_2 Control	≤ 0	Not Detectable	Not Detectable
$P+P/N_2O_2$	≤ 0	Not Detectable	Not Detectable
P/N_2O_2	≤ 0	Not Detectable	Not Detectable
N_2 Control	≤ 0	Not Detectable	Not Detectable
$P+P/N_2$	≤ 0	Not Detectable	Not Detectable
P/N_2	≤ 0	Not Detectable	Not Detectable

The behavior of the I_D^P and I_D^N is "complementary" as would be expected. A slight initial increase and recovery is observed for the P channel device and a slight initial decrease and recovery is observed for the N channel device. This is observed for both experimental treated as well as control devices.

A similar "complementary" behavior of the respective gate threshold voltages is also observed as would be expected. The observed shifts of V_{Th} for both N and P channel devices are not severe.

In both drain current (I_D) and gate threshold voltage (V_{TH}) the observed shifts affect both treated and control devices, are slight and are recovered. Again as with previous devices such changes affecting both controls and treated devices indicate systematic behavior of the devices themselves rather than paraylene process induced effects.

CONCLUSIONS

In all of the parylene or primed and parylened treated active devices as well as with their control counterparts, shifts in electrical behavior within acceptability limits were observed. In general any changes observed were essentially common to both experimental and control devices. These shifts are characteristic of the type of surface sensitive devices selected for this study and are not principally due to the parylene treatment or to the priming pretreatment. The P+N diode may be a partial exception with an apparent initial effect from the primer.

In conclusion, the effects of parylene and an optional primer upon device electrical behavior are essentially negligible. If these observations and conclusions are valid for the unusually surface sensitive devices selected for this study, they should be at least as valid for less sensitive "run of the mill" devices.

List of Abbreviations

P+P/N_2O_2 - Primer plus Parylene in 96% N_2, 4% O_2

P/N_2O_2 - Parylene only in 96% N_2, 4% O_2

P+P/N_2 - Primer plus Parylene in 99.9% N_2

P/N_2 - Parylene only in 99.9% N_2

N_2O_2 CON - Bare control specimen in 96% N_2, 4% O_2

N_2 CON - Bare control specimen in 99.9% N_2

AMB CON - Bare ambient temperature control

APPENDIX A

TYPICAL RAW DATA SHEETS FROM AUTOMATIC NETWORK ANALYZER PRINT-OUT FOR MICROWAVE INTEGRATED CIRCUIT TEST PATTERNS

NOTE: Only Substrate No. 8 is included. The data sheets for the other seven variables were similar.

TABLE 1. DATA TAKEN BEFORE PARYLENE COATING

*** HUGHES AIRCRAFT COMPANY *** 12/ 7/74

SUBSTRATE S/N 8

	FORWARD		REVERSE	
FREQ	VSWR	GAIN	VSWR	GAIN
2000.00	1.056	-.64	1.060	-.65
2500.00	1.295	-.84	1.313	-.82
3000.00	1.106	-.71	1.102	-.77
3500.00	1.061	-.87	1.043	-.86
4000.00	1.385	-1.04	1.374	-1.07
4500.00	1.219	-.97	1.210	-1.01
5000.00	1.247	-1.10	1.261	-1.13
5500.00	1.261	-1.28	1.238	-1.22
6000.00	1.117	-1.09	1.154	-1.07
6500.00	1.289	-1.52	1.244	-1.49
7000.00	1.230	-1.30	1.263	-1.30
7500.00	1.025	-1.47	1.062	-1.40
8000.00	1.265	-1.72	1.207	-1.68
8500.00	1.235	-1.75	1.324	-1.78
9000.00	1.073	-1.82	1.127	-1.81
9500.00	1.402	-2.12	1.560	-2.09
10000.00	1.090	-2.17	1.039	-2.19
10500.00	1.196	-2.39	1.424	-2.37
11000.00	1.069	-2.42	1.118	-2.40
11500.00	1.226	-2.67	1.060	-2.65
12000.00	1.274	-2.55	1.246	-2.57

REPRODUCIBILITY OF THE
ORIGINAL PAGE IS POOR

TABLE 2. DATA TAKEN AFTER PARYLENE COATING

*** HUGHES AIRCRAFT COMPANY *** 2/12/74

SUBSTRATE S/N 8

	FORWARD		REVERSE	
FREQ	VSWR	GAIN	VSWR	GAIN
2000.00	1.083	-.68	1.064	-.67
2500.00	1.344	-1.00	1.330	-.98
3000.00	1.101	-.79	1.099	-.79
3500.00	1.088	-.90	1.109	-.87
4000.00	1.392	-1.17	1.367	-1.16
4500.00	1.214	-1.00	1.233	-1.00
5000.00	1.269	-1.12	1.291	-1.09
5500.00	1.218	-1.15	1.208	-1.18
6000.00	1.130	-1.08	1.127	-1.11
6500.00	1.347	-1.46	1.357	-1.50
7000.00	1.229	-1.25	1.244	-1.27
7500.00	1.135	-1.30	1.121	-1.31
8000.00	1.186	-1.53	1.172	-1.52
8500.00	1.289	-1.40	1.281	-1.43
9000.00	1.111	-1.60	1.123	-1.62
9500.00	1.443	-1.61	1.422	-1.62
10000.00	1.026	-1.57	1.038	-1.64
10500.00	1.253	-1.61	1.240	-1.65
11000.00	1.160	-1.64	1.101	-1.68
11500.00	1.054	-1.67	1.078	-1.71
12000.00	1.346	-1.76	1.266	-1.76

TABLE 3. DATA TAKEN AFTER EXPOSURE FOR 168 HOURS AT +150°C IN AIR AMBIENT

*** HUGHES AIRCRAFT COMPANY *** 3/14/74

SUBSTRATE S/N 8

	FORWARD				REVERSE			
FREQ	VSWR	ANG	GAIN	ANG	VSWR	ANG	GAIN	ANG
2000.00	1.620	117.8	-.67	20.9	1.070	104.2	-.68	21.0
2500.00	2.945	-162.3	-.29	113.8	1.497	19.3	-.29	113.8
3000.00	1.997	124.0	-.85	-150.7	1.166	123.3	-.88	-150.8
3500.00	3.415	-156.6	-.84	-59.4	1.094	-124.7	-.87	-59.3
4000.00	2.617	128.7	-1.33	36.3	1.597	125.5	-1.35	36.3
4500.00	3.585	-156.4	-1.32	123.8	1.335	-150.1	-1.31	124.1
5000.00	2.586	127.8	-1.23	-141.6	1.413	114.5	-1.20	-141.3
5500.00	3.636	-158.0	-1.57	-57.1	1.446	-145.2	-1.54	-56.9
6000.00	1.855	114.6	-.56	30.5	1.169	-61.8	-.53	30.3
6500.00	3.418	-163.7	-2.40	120.7	1.484	-159.6	-2.41	120.7
7000.00	1.554	116.5	-1.25	-152.9	1.391	-72.2	-1.24	-152.9
7500.00	3.424	-169.1	-1.58	-63.0	1.169	-178.8	-1.58	-63.0
8000.00	1.417	115.8	-1.43	20.7	1.316	-70.2	-1.41	20.8
8500.00	3.128	176.7	-1.02	107.8	1.281	7.6	-1.03	108.0
9000.00	1.303	129.1	-1.61	-168.9	1.133	-71.9	-1.59	-168.7
9500.00	3.082	159.0	-1.18	-86.8	1.547	-26.4	-1.18	-86.9
10000.00	1.296	164.8	-1.80	-1.1	1.112	153.1	-1.80	-1.1
10500.00	3.180	146.5	-1.32	79.1	1.224	-23.5	-1.36	78.5
11000.00	1.596	-162.4	-1.70	160.0	1.154	128.7	-1.77	160.3
11500.00	3.299	137.9	-1.76	-119.5	1.187	115.1	-1.84	-118.6
12000.00	2.037	-173.1	-1.77	-38.4	1.330	50.0	-1.62	-38.0

TABLE 4. DATA TAKEN AFTER EXPOSURE FOR 500 HOURS AT +150°C IN AIR AMBIENT

*** HUGHES AIRCRAFT COMPANY *** 4/ 8/74

SUBSTRATE S/N 8

	FORWARD		REVERSE	
FREQ	VSWR	GAIN	VSWR	GAIN
2000.00	1.053	-.44	1.668	-.55
2500.00	1.620	-.21	2.117	-.28
3000.00	1.086	-.57	1.347	-.66
3500.00	1.060	-.58	2.018	-.66
4000.00	1.272	-1.14	1.349	-1.08
4500.00	1.213	-1.04	1.580	-1.06
5000.00	1.295	-1.19	1.594	-1.25
5500.00	1.172	-1.03	1.208	-1.11
6000.00	1.140	-.98	1.202	-1.06
6500.00	1.383	-1.19	1.730	-1.39
7000.00	1.205	-1.13	1.374	-1.29
7500.00	1.166	-1.26	1.641	-1.30
8000.00	1.164	-1.49	1.376	-1.48
8500.00	1.263	-1.37	1.308	-1.34
9000.00	1.170	-1.53	1.209	-1.57
9500.00	1.365	-1.69	1.573	-1.69
10000.00	1.049	-1.74	1.141	-1.74
10500.00	1.208	-1.83	1.441	-1.83
11000.00	1.145	-1.76	1.146	-1.77
11500.00	1.039	-1.81	1.362	-1.79
12000.00	1.186	-1.79	1.261	-1.77

REPRODUCIBILITY OF THE ORIGINAL PAGE IS POOR

C-2

TABLE 5. DATA TAKEN AFTER EXPOSURE FOR 1000 HOURS AT +150°C IN AIR AMBIENT

*** HUGHES AIRCRAFT COMPANY *** 5/ 3/74

SUBSTRATE S/N 8

	FORWARD		REVERSE	
FREQ	VSWR	GAIN	VSWR	GAIN
2000.00	1.041	-.75	1.049	-.72
2500.00	1.302	-.93	1.250	-.93
3000.00	1.084	-.89	1.091	-.92
3500.00	1.042	-.87	1.063	-.93
4000.00	1.328	-1.13	1.353	-1.14
4500.00	1.243	-1.16	1.240	-1.16
5000.00	1.246	-1.27	1.274	-1.28
5500.00	1.274	-1.25	1.260	-1.25
6000.00	1.133	-1.27	1.137	-1.29
6500.00	1.308	-1.55	1.322	-1.55
7000.00	1.311	-1.46	1.298	-1.47
7500.00	1.113	-1.44	1.155	-1.43
8000.00	1.232	-1.70	1.224	-1.68
8500.00	1.196	-1.65	1.234	-1.67
9000.00	1.108	-1.76	1.159	-1.81
9500.00	1.430	-1.74	1.417	-1.80
10000.00	1.040	-1.76	1.198	-1.88
10500.00	1.295	-1.62	1.277	-1.91
11000.00	1.044	-1.85	1.103	-1.90
11500.00	1.092	-2.24	1.064	-1.96
12000.00	1.210	-2.17	1.299	-1.96

APPENDIX B

TABLE 1. DATA REDUCTION FOR MICROWAVE INTEGRATED CIRCUIT TEST PATTERNS

Nos. 1, 2, 3, and 4 were not parylene coated. Nos. 1, 2, 5, and 6 were sealed in nitrogen. Nos. 3, 4, 7, and 8 were sealed in air.

Test Pattern Number	All Values are Line Loss in dB per Inch														
	Before Coating			After Coating (Patterns 5, 6, 7 & 8 only)			After 168 Hr. at +150°C			After 500 Hr. at +150°C			After 1000 Hr. at +150°C		
	2 GHz	8 GHz	12 GHz	2 GHz	8 GHz	12 GHz	2 GHz	8 GHz	12 GHz	2 GHz	8 GHz	12 GHz	2 GHz	8 GHz	12 GHz
1	0.2119	0.4825	0.5260	0.1987	0.4428	0.4844	0.1949	0.5582	0.6168	0.1041	0.3974	0.4768	0.1249	0.4106	0.5582
2	0.2554	0.6414	0.7304	0.2384	0.6055	0.7133	0.2233	0.6604	0.7890	0.2573	0.6036	0.7190	0.1589	0.5752	0.6887
3	0.1173	0.2933	0.3217	0.1154	0.2763	0.3273	0.1192	0.2668	0.2630	0.0889	0.2838	0.3614	0.1381	0.3027	0.3349
4	0.1211	0.2838	0.4011	0.1192	0.2819	0.3009	1.078	1.310	1.504	0.0870	0.2952	0.3822	0.1173	0.2857	0.3425
5	0.2157	0.5166	0.5904	0.1968	0.4541	0.5241	0.2308	0.5298	0.6149	0.1835	0.5676	0.6623	0.2081	0.5525	0.6793
6	0.1533	0.4371	0.5033	0.1608	0.4201	0.4957	0.1514	0.4541	0.5430	0.1268	0.5128	0.6074	0.1400	0.5203	0.6225
7	0.1665	0.3160	0.3614	0.1306	0.2876	0.3236	0.1495	0.2365	0.3217	0.1135	0.3122	0.3482	0.1419	0.3311	0.3898
8	0.1230	0.3179	0.4863	0.1268	0.2876	0.3330	0.1287	0.2668	0.3065	0.1041	0.2800	0.3349	0.1362	0.3179	0.3709

TABLE 2. DATA REDUCTION FOR MICROWAVE INTEGRATED CIRCUIT TEST PATTERNS SHOWING PERCENTAGE CHANGE IN LINE LOSS AS A RESULT OF ENVIRONMENTAL EXPOSURE TO +150°C FOR 1000 HOURS

Test Pattern Number	Percentage Change in Line Loss from 0 Hours to 1000 Hours at +150°C		
	2 GHz	8 GHz	12 GHz
1 – Not Coated	-37.14	- 7.27	+15.24
2 – Not Coated	-33.35	- 5.00	- 3.45
3 – Not Coated	+19.67	+ 9.55	+ 2.32
4 – Not Coated	- 1.59	+ 1.35	+13.83
5 – Coated	+ 5.74	+21.67	+29.61
6 – Coated	-12.94	+23.85	+25.58
7 – Coated	+ 8.65	+15.13	+20.46
8 – Coated	+ 7.41	+10.54	+11.38

NOTE: A minus (-) sign indicates a decrease in loss and thus an improvement. A plus (+) sign indicates an increase in loss and thus some degradation.

TABLE 3. PERCENTAGE CHANGES IN LINE LOSS BEFORE AND AFTER PARYLENE COATING

Test Pattern Number	Percentage Change in Line Loss		
	2 GHz	8 GHz	12 GHz
1 – Not Coated	- 6.23	- 8.23	- 7.91
2 – Not Coated	- 6.66	- 5.60	- 2.34
3 – Not Coated	- 1.62	- 5.80	+ 1.74
4 – Not Coated	- 1.57	- 0.67	-24.98
5 – Coated	- 8.76	-12.10	-11.23
6 – Coated	+ 4.89	- 3.89	- 1.51
7 – Coated	-21.56	- 8.99	-10.46
8 – Coated	+ 3.09	- 9.53	-31.52

NOTE: The change in line loss for the "NOT COATED" samples is due only to exposure to room ambient for 65 days between measurements. A "-" value means an improvement in loss. A "+" value indicates the loss got worse.

APPENDIX C

TABLES REPRESENTING DATA REDUCTION OF THE THICK/THIN FILM RESISTOR TEST RESULTS

Table 1. Average percentage resistance changes for thin film resistors (225 Ω/□ Nichrome)

Table 2. Average percentage resistance changes for glazed thick film resistors (10 Ω/□)

Table 3. Average percentage resistance changes for glazed thick film resistors (1 KΩ/□)

Table 4. Average percentage resistance changes for glazed thick film resistors (100 KΩ/□)

Table 5. Average percentage resistance changes for unglazed thick film resistors (10 Ω/□)

Table 6. Average percentage resistance changes for unglazed thick film resistors (1 KΩ/□)

Table 7. Average percentage resistance changes for unglazed thick film resistors (100 KΩ/□)

TABLE 1. RESISTANCE CHANGES FOR THIN FILM RESISTOR (225 Ω/□ NICHROME)

Specimen Description	Nominal Resistor Value KΩ	Effect of Cleaning and/or Coating (percent)	Subsequent Change after Exposure at 150°C for 1000 Hours (percent)	Portion of Change Occurring between 500-1000 (percent)
Uncoated N_2 Control	10 to 50	+0.03	0.42/0.35	75/80
Uncoated N_2O_2 Control	10 to 50	+0.05	0.34/0.37	75/75
Uncoated Air Control	10 to 50	+0.05	0.34/0.37	75/75
P+P/N_2	10 to 50	+0.03	0.31/0.42	75/80
P+P/N_2O_2	10 to 50	+0.02	0.38/0.47	85/80
P+P/Air	10 to 50	0.0	0.36/0.38	75/75
P/N_2	10 to 50	0.0	0.37/0.30	75/80
P/N_2O_2	10 to 50	-0.02	0.37/0.42	75/80
P/Air	10 to 50	-0.02	0.34/0.37	75/75

TABLE 2. RESISTANCE CHANGES FOR GLAZED THICK FILM RESISTORS (10Ω/□)

Specimen Description	Nominal Resistor Value KΩ	Effect of Cleaning and/or Coating (percent)	Subsequent Change after Exposure at 150°C for 1000 Hours (percent)	Portion of Change Occurring between 500-1000
10Ω/□ Ink				
Uncoated N_2 Control	0.035	-0.2	+6.59	50
Uncoated N_2O_2 Control	0.035	-0.2	+2.67	20
Uncoated Air Control	0.035	+1.0	+1.89	75
P+P/N_2	0.035	+0.0	+3.39	25
P+P/N_2O_2	0.035	0.0	+3.28	25
P+P/Air	0.035	+2.0	+1.94	50
P/N_2	0.035	0.0	+3.65	50
P/N_2O_2	0.035	0.0	+3.93	50
P/Air	0.035	+3.0	+1.42	75

TABLE 3. RESISTANCE CHANGES FOR GLAZED THICK FILM RESISTORS (1 KΩ/□)

Specimen Description	Nominal Resistor Value KΩ	Effect of Cleaning and/or Coating (percent)	Subsequent Change after Exposure at 150°C for 1000 Hours (percent)	Portion of Change Occurring between 500-1000
1 KΩ/□ Ink				
Uncoated N_2 Control	4.0	+0.02	+0.58	90
Uncoated N_2O_2 Control	4.0	+0.01	+0.55	80
$P+P/N_2$	4.0	+0.01	+0.55	90
$P+P/N_2O_2$	4.0	+0.01	+0.48	80
P/N_2	4.0	+0.02	+0.49	90
P/N_2O_2	4.0	+0.01	+0.48	90

TABLE 4. RESISTANCE CHANGES FOR GLAZED THICK FILM RESISTORS (100 KΩ/□)

Specimen Description	Nominal Resistor Value KΩ	Effect of Cleaning and/or Coating (percent)	Subsequent Change after Exposure at 150°C for 1000 Hours (percent)	Portion of Change Occurring between 500-1000
<u>100 KΩ/□ Ink</u>				
Uncoated N_2 Control	350	+0.03	+0.33	90
Uncoated N_2O_2 Control	350	-0.02	+0.35	80
$P+P/N_2$	350	0.00	+0.34	90
$P+P/N_2O_2$	350	-0.01	+0.30	80
P/N_2	350	+0.02	+0.06	90
P/N_2O_2	350	+0.01	+0.13	90

TABLE 5. RESISTANCE CHANGES FOR UNGLAZED THICK FILM RESISTORS (10Ω/□)

Specimen Description	Nominal Resistor Value KΩ	Effect of Cleaning and/or Coating (percent)	Subsequent Change after Exposure at 150°C for 1000 Hours (percent)	Portion of Change Occurring between 500-1000
10 Ω / □ Ink				
Uncoated N_2 Control	0.035	+0.5	+ 8.38	50
Uncoated N_2O_2 Control	0.035	+0.2	+ 7.55	50
Uncoated Air Control	0.035	+2.5	- 0.01	0
P+P/N_2	0.035	+0.3	+ 6.16	50
P+P/N_2O_2	0.035	+0.2	+ 7.44	25
P+P/Air	0.035	+1.0	+14.81	90
P/N_2	0.035	+0.2	+23.13	75
P/N_2O_2	0.035	+0.3	+18.50	75
P/Air	0.035	+1.5	+ 6.47	75

TABLE 6. RESISTANCE CHANGES FOR UNGLAZED THICK FILM RESISTORS (1 KΩ/□)

Specimen Description	Nominal Resistor Value KΩ	Effect of Cleaning and/or Coating (percent)	Subsequent Change after Exposure at 150°C for 1000 Hours (percent)	Portion of Change Occurring between 500-1000
1 KΩ/□ Ink				
Uncoated N_2 Control	4.0	+0.05	+1.93	25
Uncoated N_2O_2 Control	4.0	+0.02	+1.81	75
P+P/N_2	4.0	+0.05	+3.75	75
P+P/N_2O_2	4.0	+0.03	+3.15	75
P/N_2	4.0	+0.03	+2.00	75
P/N_2O_2	4.0	+0.02	+1.87	75

TABLE 7. RESISTANCE CHANGES FOR UNGLAZED THICK FILM RESISTORS (100 KΩ/□)

Specimen Description	Nominal Resistor Value KΩ	Effect of Cleaning and/or Coating (percent)	Subsequent Change after Exposure at 150°C for 1000 Hours (percent)	Portion of Change Occurring between 500-1000
100 KΩ/□ Ink				
Uncoated N_2 Control	350	+0.02	+1.59	75
Uncoated N_2O_2 Control	350	+0.05	+1.99	75
P+P/N_2	350	+0.02	+1.71	75
P+P/N_2O_2	350	+0.02	+2.02	75
P/N_2	350	+0.02	+1.84	75
P/N_2O_2	350	+0.05	+1.71	75