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DESIGN GUIDELINES FOR USE OF ADHESIVES IN HYBRID MICROCIRCUITS

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EDITOR'S NOTE

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INTRODUCTION

Recently, increased experience with adhesives and confidence in their performance have reached the point where they are being widely used in the assembly of hybrid microcircuits for both military and space electronics hardware. However, few programs have been conducted to develop adhesives specifically meeting the very stringent stability requirements for these applications. In many cases, selection of the adhesive being used has been essentially fortuitous, resulting from its initial inhouse availability (because it had been used for other, unrelated applications), or because it was recommended by the local adhesive representative. Thus in general, many of the classical polymer types of adhesives are being sold and used with little regard to essential reliability related factors, such as extent of outgassing, corrosivity, effect of outgassed constituents on uncased semiconductor devices, and retention of bond strength under long term operating conditions.

Adhesives presently used in hybrid microcircuits can be classified into the two very broad categories: electrically insulative and electrically conductive. In many circuit assemblies, both types are used. The various uses to which adhesives have been put include the following:

1. Electrically Insulative:

   a. Bonding substrates to packages.

   b. Lidding.

   c. Reinforcing edge connectors.

   d. Protecting fine wire leads.

   e. Molding or sealing packages.

   f. Bonding chip components (e.g., capacitors, resistors, semiconductors, etc.) to substrates.

2. Electrically Conductive:

   a. Bonding semiconductor die to substrates.

   b. Repairing conductor lines.

   c. Attaching capacitors to bond pads.

   d. Providing ohmic contact of connectors or lead frames.

Of these, the most important and common uses are bonding substrates (usually an alumina ceramic) to the base of metal or ceramic packages using an electrically insulative adhesive, and attaching semiconductor die and chip capacitors to conductor pads on substrates using an electrically conductive adhesive.

The use of adhesives in lieu of eutectic bonding or other metallurgical attachment is highly desirable from both a reliability and manufacturing process standpoint. Organic adhesives can be processed and cured at lower temperatures (usually below 150°C); allow easy rework, removal, and replacement of components without subjecting the circuits to high temperatures; and, because of their low moduli of elasticity, allow stress dissipation without damaging the component, bond line, or substrate. This latter property is particularly important during temperature cycling or thermal shocking of circuits, especially in the case of larger components such as capacitors. A previous investigation by S. V. Caruso et al. [1] has clearly delineated that solder joined capacitors are susceptible to catastrophic failures during temperature cycling and, consequently, it has been recommended that this assembly technique not be used for meeting the requirements of Classes "A" and "B" hybrid microcircuits as defined in MSFC 85M03926.

A. General Scope Of Study

Although it is generally agreed that the use of adhesives in the assembly of hybrid microcircuits offers advantages over other bonding methods, there currently does not exist a set of guidelines for the selection of adhesives which will insure the fabrication of hybrid microcircuits meeting the long use-life, high-reliability requirements of electronic equipment for space applications. Understandably, as a result, there is considerable reluctance on the part of the responsible agencies to accept the substitution of adhesive bonding for the long established metallurgical attachment methods.

In general, this attitude is justified considering the fact that while the use of adhesives offers several important advantages, it also can introduce severe reliability problems unless care is exercised to select only those specific adhesives which are chemically, physically, and electrically compatible with the components, processes, and metallization systems used in the fabrication of hybrid microcircuits.

This study was directed to the identification and investigation of such problems that could result from the use of electrically insulative adhesives, and to the development of suitable evaluation tests to quantify these effects for the various adhesives, thus forming the basis for guidelines and specifications for electrically insulative adhesives for hybrid microcircuits. To generate an adequate data base to verify the validity and establish the sensitivity of the selected tests, an indepth evaluation also was made of selected state-of-the-art adhesives representative of the major classes presently proposed for use in hybrid microcircuits.

B. Adhesives For Hybrid Microelectronics

1. Important Properties for Microcircuit Use. The properties of adhesives that are important in determining their suitability for use in the assembly of hybrid microcircuits include those listed and briefly discussed below.

Electrical	Stable electrical properties must be maintained over wide ranges of temperature and humidity (e.g., insulative adhesives should maintain a volume resistivity greater than 10^{14} ohm-cm in the dry condition).
Handling Convenience	From the user's standpoint for both economy and convenience, factors such as storage life and conditions, pot life, whether the adhesive is a single component or two component system, and whether or not it is available premixed, frozen and/or in a ready to use tube will influence selection.

Ease of Application	Adhesives must be capable of being applied in controlled amounts and thicknesses and give void free bonds. Insufficient thickness can result in electrical breakdown, while excessive amounts can produce excessive stresses during temperature cycling.
Flow During Cure	Excessive flow during cure must be prevented to avoid coating of adjacent areas which subsequently must be soldered, or bridging of conductor lines and possibly causing electrolytic corrosion.
Shrinkage During Cure	Excessive shrinkage during cure must be prevented to avoid mechanically stressing components and cracking them or inducing parameter changes.
Component Creep	The tendency of an adhesive component to separate due to capillary action or creep during cure is undesirable due to possible contribution to electrolytic corrosion or degradation of wire bonding.
Outgassing	Both the release of condensable volatiles during cure and continued outgassing after cure are undesirable. Outgassed constituents can adsorb onto electronic components and degrade properties.
Ionic Content	Adhesives must not contain water extractable ionic

constituents such as Cl^- or Na^+ which will promote corrosion or electrical leakage between conductors.

Tackiness	In the case of electrically insulative adhesives, exposed edges must be tack-free to avoid capturing of conductive particle contaminants which can cause electrical failure.
Solvent Resistance	Degradation of bond strength or leaching of adhesive components must not be caused by solvents used in cleaning electronic components, modules, or subsystems.
Corrosivity	Adhesives must not be innately chemically corrosive or electrolytically corrosive to the metallization system used.
Flexibility	Adhesives must be sufficiently pliable to relieve mechanical stresses among thermally mismatched materials to avoid warping or cracking of substrates and components.
Repairability	Because of the requirement for rework, it is desirable that the adhesive bond be fracturable at sufficiently low temperature and mechanical force to avoid damaging the metallization or breaking the substrate.
Hydrolytic Stability	Adhesives must not degrade chemically (e.g., revert back to a liquid) on exposure for long periods at high temperature and humidity.

Thermal Stability	Adhesives must not decompose at high temperatures or crack at low temperatures (-65 to +150°C).
Bond Strength	Adhesives must have a sufficiently high initial bond strength and must retain adequate bond strength at maximum use temperature, after exposure to commonly used solvents and high humidity, and after extended aging.
Reliability	Hybrid microcircuits assembled using the adhesive must be able to withstand the temperature cycling, thermal shock, and constant acceleration tests defined in MIL-STD-883.

2. Polymeric Types of Adhesives. A list of some of the better known general types of polymeric adhesives and their associated advantages and limitations is given in Table 1. Although there are numerous types of polymeric adhesives and many hundreds of variations within each type, to date only the epoxies have found extensive use in the assembly of hybrid microcircuits. Those types designed for structural bonding applications and, consequently, extremely high bond strength have been excluded because they usually require high temperature, long times, and, often, even high pressure for curing. These requirements are undesirable from a microcircuit assembly processing viewpoint, and the very high shear strengths obtained are not necessary in the bonding of the small, essentially flat, electronic components. Among the types thus excluded are the nitrile-phenolics, vinyl phenolics, polyimides, and polybenzimidazoles. Other generic types, such as polyurethanes, have been excluded because of their low use temperature (120 to 150°C) and their often high degree of outgassing and decomposition. On the basis of considerations such as these, the epoxies have been selected as the type of adhesive inherently most appropriate for use in microelectronics, and special development effort has been concentrated on them by manufacturers specializing in adhesives for this application.

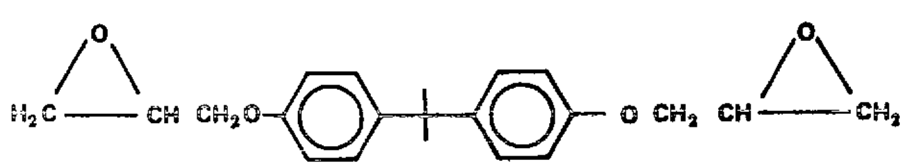
TABLE 1. ADHESIVE TYPES (ADVANTAGES AND LIMITATIONS)

Type	Advantages	Limitations
Phenolics	Very high bond strength	Used mostly for structural applications, Possibly corrosive, Difficult to process at low temperatures
Polyurethanes	Easy to rework	Not suitable for temperatures above 120-150°C, Relatively high outgassing, Some decomposition
Polyamides	Easy to rework	High moisture absorption, High outgassing, Variations in electrical insulation properties, especially in humidity
Polyimides	Very high temperature stability	High cure temperatures, Require solvents as vehicles
Silicones	High temperature stability, Easy to rework, High purity, Low outgassing	Moderate to poor bond strength, High expansion coefficient
Epoxies	Some are easy to rework (by thermo-mechanical means), Some are low outgassers, Easy to process, Can be filled to 60-70 percent with a variety of conductive or nonconductive fillers	Depending on type of curing agent used and degrees of cure: outgassing, catalyst leaching, corrosivity
Cyanoacrylates	Very rapid setting (≈10 sec), Give very high initial bond strengths	Bond strengths often degrade under moist or elevated temperature (≈150°C) conditions

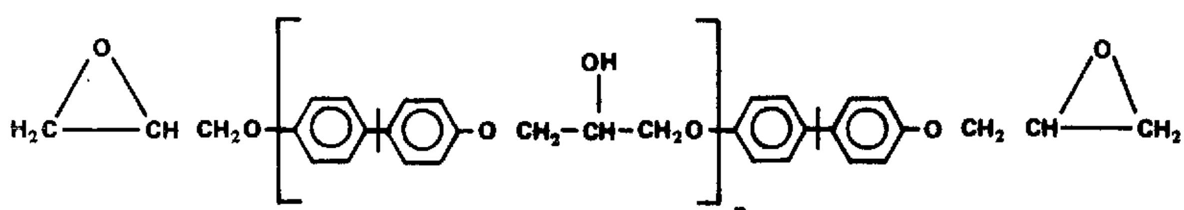
Considerable information of the type epoxy plastics required for commercial and structural aerospace applications is available [2][1]. However, little information related to their use in hermetically sealed microcircuit packages, and the specific properties required to meet the accompanying stringent demands, is available. Furthermore, many thousands of formulations are possible, and hundreds of proprietary epoxies are on the market, making it very difficult for the design or materials engineer to make a correct selection for his particular application. In the case of electrically conductive adhesives, new and reportedly improved versions are being announced almost weekly.

In general, epoxies can be categorized as to polymer types and curing agents used. All epoxy adhesives may be considered to be two component systems, consisting of an epoxy resin (a medium molecular weight difunctional compound) and a curing agent (any one of a number of acid or alkaline compounds which polymerize the resin via an ionic curing mechanism). Even the so-called one package or one component epoxies are in reality two components, wherein the curing agent has been rendered inert under normal storage conditions but becomes activated at elevated temperature. Although numerous epoxy resins exist [3], the type that invariably is used is the diglycidyl ether of bisphenol A (DGEBA) or its higher molecular weight analogs represented by the structural formulas given below:

Diglycidyl Ether of Bisphenol A (DGEBA)



Higher Molecular Weight Analogs of DGEBA



1. Some information was taken from reports sponsored by Wright Patterson Air Force Base, Materials Laboratory.

The commercial Epon series of resins (Epon 815, 828, 1001, etc.) belong to this class. Although there is the possibility of these resins carrying over some ionic impurities from their synthesis (sodium ions, chloride ions, and possibly others), generally they are quite pure and considered to be inert. Of more serious concern are the curing agents used in polymerizing or hardening these resins. Most of the curing agents are inherently corrosive, or deleterious to the electrical parameters of uncased semiconductor devices. Some curing agents must be used in stoichiometric amounts; that is, in precise precalculated ratios, in order to achieve the maximum degree of polymerization, and in order for them to be completely incorporated in the final molecular structure. If this ratio is slightly off or if the curing conditions are not carefully controlled, some of the ingredients (either resin or curing agent) may be left unreacted and can lead to an outgassing or corrosion problem. Other curing agents are employed in catalytic amounts but likewise can result in incomplete reactions and consequently cause similar circuit degradation effects.

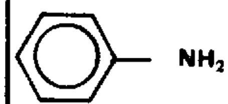
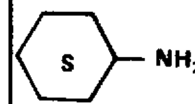
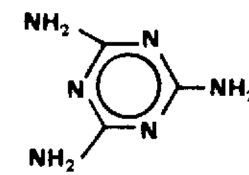
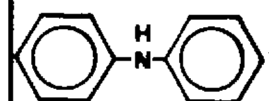
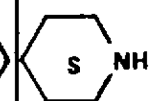
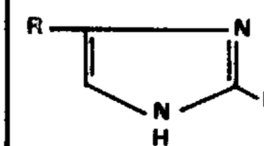
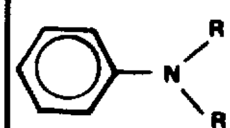
The most commonly used curing agents for epoxies are the amines, anhydrides, and boron trifluoride complexes. The amines in turn may be of several types: primary amines, secondary amines, tertiary amines, compounds containing both primary and secondary amines, and dicyandiamide (which decomposes at elevated temperature to primary and secondary amines). These amines may further be classified as aliphatic (straight chain), aromatic (ring type), or heterocyclic (ring type containing some atoms other than carbon). A matrix of the many permutations possible is given in Table 2. The reader is referred to any of several texts [4] for a better understanding of the chemistry and structure of these amines.

II. SELECTION OF ADHESIVES AND PROPERTIES FOR EVALUATION

A. State-Of-The-Art Survey

Two surveys were conducted during the course of this study in an attempt to insure that a complete list of candidate electrically insulative adhesives was obtained. The first was directed to some 50 adhesive manufacturers requesting them to identify the electrically insulative adhesives they manufacture and recommend for use in hybrid microcircuit assembly. Only 19 replies were received. Answers ranged from simple statements that they do not manufacture electrically insulative adhesives or that, while they do manufacture such adhesives, they are not familiar

TABLE 2. CLASSIFICATION OF AMINE HARDENERS AND CATALYSTS[a]

Type	Aliphatic	Aromatic	Alicyclic	Heterocyclic
Primary	$R-NH_2$			
Secondary	$R-\overset{H}{N}-R$			
Tertiary	$R-\underset{R}{\underset{\mid}{N}}-R$			

a. Specific examples are given; other permutations involving combinations of primary, secondary, and tertiary amines are possible.

with the special requirements related to their use in hybrid microelectronics, to the enthusiastic replies by two companies that they manufacture such adhesives specifically for use in the assembly of hybrid microcircuits. These two companies were Ablestik Laboratories and Epoxy Technology. Other manufacturers sent catalogs of all of their adhesives (resins and hardeners) with the statement that they felt certain that in it someplace was an appropriate electrically insulative adhesive with the desired characteristics, and that they would be pleased to supply it when it was identified.

The second survey was directed to over 30 possible manufacturers of hybrid microcircuits. Request was made that, to the extent that they could without divulging information considered company proprietary, they identify the specific electrically insulative adhesives they are using in the assembly of their hybrid microcircuits, the specific applications for which they are using them, and their satisfaction or dissatisfaction with the results obtained. Only eight responded. Answers varied from the simple statements that they do not use adhesives at all or that assembly processes and techniques are considered company proprietary, to specific identification of adhesives being used and frank opinions of their suitability. However, from this small sampling, indications are that in many cases the selection of adhesives has been fortuitous rather than the result of a systematic experimental study. Also, Ablestik and Epoxy Technology products were dominant among those specifically identified.

Lists of the adhesive manufacturers and hybrid microcircuit manufacturers contacted are given in Tables 3 and 4, respectively. Those marked with an asterisk are the ones that responded.

B. Selection Of Candidate Adhesives

Based on the results of this survey, the adhesives listed in Table 5 were selected for at least some testing (bond strength), and the following adhesives were selected for detailed testing and evaluation:

1. Hysol 0151.

2. Ablefilm 517A.

3. Eccobond 104.

4. Epo-Tek H74.

5. Epo-Tek H61.

TABLE 3. ADHESIVE MANUFACTURERS CONTACTED[a]

1. Ablestik Laboratories*	26. Fortin Laminating
2. Allaco Products	27. Furane Plastics
3. American Cyanamid*	28. G. C. Electronics*
4. Amicon Corp.*	29. General Electric Co.*
5. Aremco Products*	30. GFC Engineer & Sales
6. Armstrong Products*	31. Hardman, Inc.*
7. Bacon Industries	32. Hecht Rubber Co.
8. Baker Castor Oil*	33. Hysol
9. Biggs Co.	34. Isochem Resins, Co.*
10. R. H. Carlson	35. Job Ready Plastics
11. Castall Inc.	36. Kenics Corp.
12. Cermalloy	37. Lash, Labs
13. Chomerics	38. Loctite Corp.*
14. Conap, Inc.	39. Mystik Tape, Borden Inc.*
15. Dillon Stevens	40. Nitine Inc.
16. John C. Dolph Co.	41. Products Components*
17. Dow Corning Corp.	42. Ren Plastics
18. Dynaloy, Inc.	43. Sigma Plastronics
19. Eastman Chem. Products	44. Spectra Strip Corp.
20. Electro Materials Corp.	45. Starnetics*
21. Electro-Science Labs*	46. Reichhold Chem, Inc.
22. Emerson & Cuming	47. Techform Labs, Inc.*
23. Epoxylite Corp.	48. Tra-Con Inc.*
24. Epoxy Technology*	49. Transene Co.*
25. Fenwal Electronics	50. Vitta Corp.

a. An asterisk indicates that a response was received.

TABLE 4. HYBRID MICROCIRCUIT MANUFACTURERS CONTACTED[a]

1. American Electronics Laboratories
2. Amperex Electronics Corp.
3. Boeing Electronic Products
4. Centralab Electronics
5. Clemson University
6. Cogar Corp.
7. Collins Radio*
8. Conductron - Missouri*
9. CTS Microelectronics, Inc.
10. Dickson Electronics Corp.
11. Electronics Communications, Inc.*
12. General Electric ICPD
13. Halex, Inc.
14. Hewlett Packard
15. Johns Hopkins University*
16. Hughes Aircraft Corp.
17. Hybridyne, Inc.
18. Lear Siegler, Inc.*
19. LTV Electrosystems, Inc.
20. McDonnell Douglas Astronautics*
21. Micropac Industries
22. Raytheon-Components Division
23. Raytheon-Semiconductor Division
24. Sloan Technology Corp.
25. Sprague Electric
26. Teledyne Semiconductor
27. Texas Instruments
28. TRW Semiconductor Division*
29. Unisem Corp.
30. Varadyne, Inc.
31. Westinghouse, Defense Center*
32. MIT-Lincoln Lab
33. Lockheed Electronics Co.

a. An asterisk indicates that a response was received.

TABLE 5. ADHESIVES FOR WHICH SOME TESTING WAS PERFORMED

1. Hysol 0151	8. Epo-Tek H61
2. Hysol 0266	9. Epo-Tek H72
3. Ablefilm 517A	10. Epo-Tek H74
4. Ablefilm 532	11. Ablebond 161-3
5. Eccobond 104	12. Ablebond 450
6. Epo-Tek H54	13. Ablefilm 535
7. Epo-Tek H55	14. Epoxylite 6203

These particular adhesives were selected as representative of the types of electrically insulative adhesives presently used in microcircuit fabrication. All are epoxies, but they are cured with five of the different major types of curing agents. Hysol 0151 is cured with a primary/secondary amine; Ablefilm 517A is cured with a tertiary amine; Eccobond 104 is cured with an anhydride; Epo-Tek H74 is cured with a modified heterocyclic amine (imidazole); and Epo-Tek H61 is cured with a boron trifluoride complex.

The fact that all of the adhesives chosen for the present evaluation are epoxies was largely based on their extensive current use. However, it should also be pointed out that from fundamental considerations, silicones are highly promising and should be considered for future evaluation. None were selected for the present study due to the low bond strength of those commercially available [1.38 to 4.14 x 10^6 N/m^2 (200 to 600 psi)]. By preagreement, only those adhesives with room temperature bond strengths of at least 1.72 x 10^7 N/m^2 (2500 psi) were considered. To increase the bond strength of silicones to this value would require development effort beyond the scope of the present study.

C. Major Critical Properties Selected For Evaluation

Since it was impossible within the scope of this study to investigate all properties of electrically insulative adhesives previously listed, three major properties considered to be especially critical were selected for detailed attention:

1. Bond strength under various conditions.

2. Outgassing after cure.

3. Corrosivity to typical metallization systems.

III. TEST PROCEDURES

A. Bond Strength

Bond strength test specimens consisted of ten, 0.127 x 0.127 cm (0.050 x 0.050 in.), silicon dice bonded to both glazed and unglazed, 1.90 x 3.81 cm (0.75 x 1.5 in.), alumina substrates using the various selected adhesives. Adhesive application and die placement were done manually. A photograph of a typical specimen is given in Figure 1. Bond strength measurements were made using the tester shown in Figure 2. As can be seen from the photograph, the stage of this tester includes a hotplate to permit testing at elevated temperatures. The blade of the tester is flat and free to pivot so it can adjust to the orientation of the individual die insuring that the force is applied uniformly over the complete width of the die being tested and perpendicularly to its edge. Originally, the capability of the tester was limited to 2000 grams, or approximately 1.21×10^7 N/m^2 (1760 psi) for the 0.127 x 0.127 cm (0.050 x 0.050 in.) die being used. However, during the course of the study, it was first modified to increase its capability to approximately 3470 grams [approximately 2.11×10^7 N/m^2 (3060 psi) and ultimately redesigned to have a 4000 gram capability [approximately 2.43×10^7 N/m^2 (3530 psi)].

Measurements of bond strength were made at both room temperature and a maximum use temperature of 150°C. Room temperature bond strengths were determined for specimens immediately after cure; after 30 min immersion in Freon TF, isopropyl alcohol, and trichloroethylene; and after 10, 30, and 90 days aging at room temperature and at maximum use temperature. Bond strength at maximum use temperature was measured for freshly prepared specimens only.

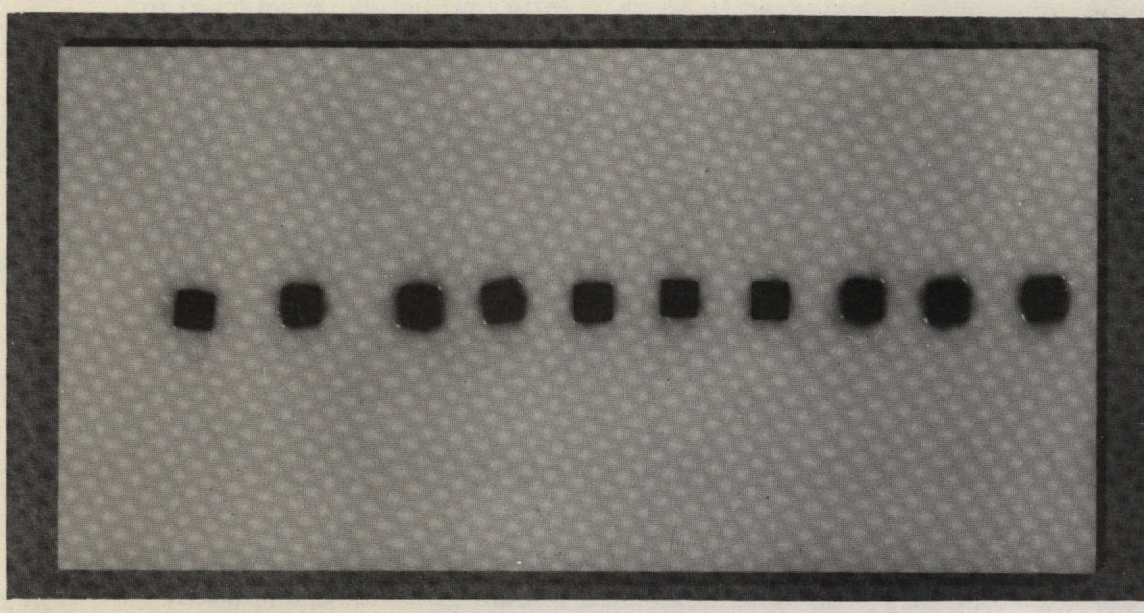


Figure 1. Typical bond strength test specimen.

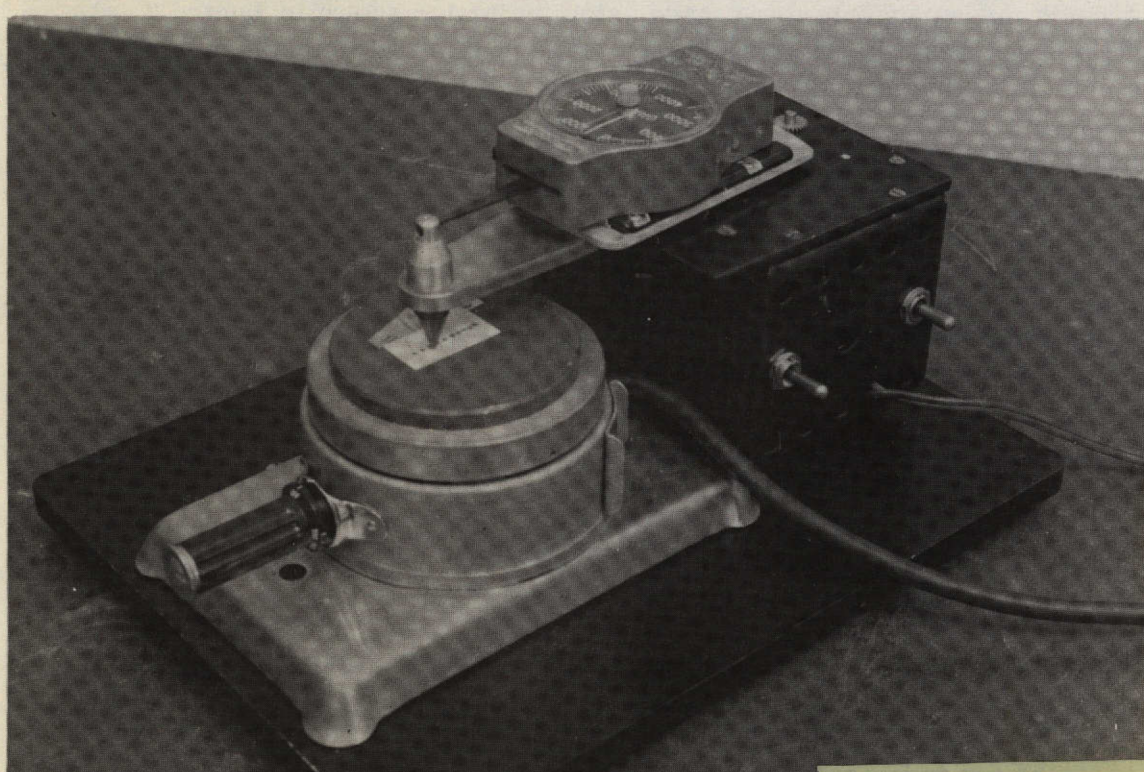


Figure 2. Bond strength tester.

B. Outgassing After Cure

After careful consideration of various methods of determining the extent to which adhesives outgas after cure, it was decided that the most practical method of obtaining meaningful data was to age samples of cured adhesives in a sealed enclosure and measure the pressure of the outgassed products. To implement this method, break-seal tubes were equipped with manometers; precisely weighed one gram samples of adhesives that had been spread thin, cured, and broken up were placed in them; and then they were evacuated and sealed off. These specimens then were mounted on a rack and placed in an oven held at the agreed-upon maximum use temperature of 150°C. A photograph of one specimen mounted on the rack is shown in Figure 3. Readings were taken of pressure versus time over some 55 to 60 days. Several readings were taken the first day, twice a day (morning and evening) for the next few days, and somewhat sporadically thereafter.

At the end of this time, gas chromatographic and mass spectrometric analyses were run to determine the major constituents outgassed from the various adhesives.

C. Corrosivity

A special test specimen shown in Figure 4 was designed and fabricated for use in evaluating the corrosive effect of electrically insulative adhesives on metallization systems commonly used in hybrid microcircuits. Specimens were fabricated of thin film aluminum and both thin and thick film gold. These specimens consist of seven pairs of parallel lines on a 1.90 x 3.81 cm (0.75 x 1.5 in.) unglazed alumina substrate:

1. Thin Film Pattern:

a. 2 pairs — 0.0127 cm (5 mils) wide, 0.00635 cm (2.5 mil) spacing.

b. 2 pairs — 0.0254 cm (10 mils) wide, 0.0127 cm (5 mil) spacing.

c. 2 pairs — 0.0254 cm (10 mils) wide, 0.0254 cm (10 mil) spacing.

d. 1 pair — 0.0254 cm (10 mils) wide, 0.0508 cm (20 mil) spacing.

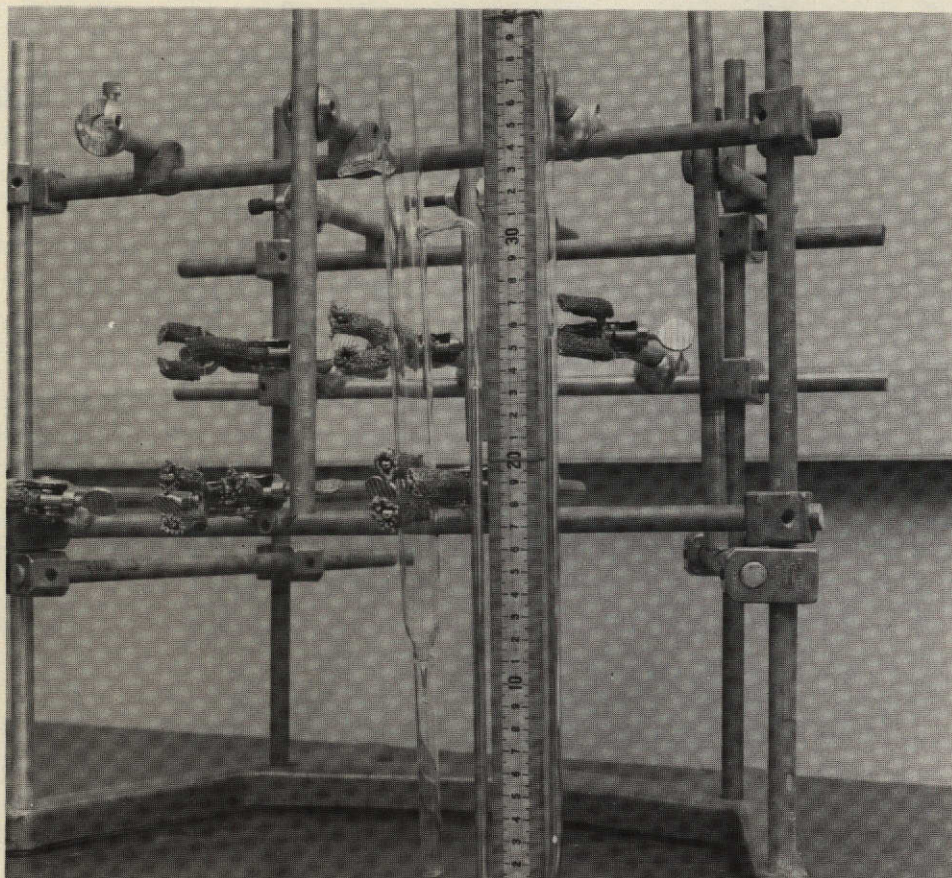


Figure 3. Test setup used for long term outgassing study.

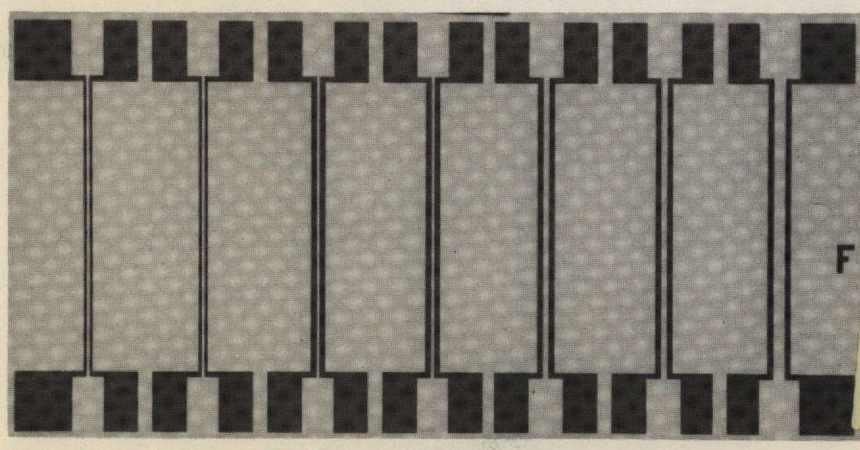


Figure 4. Corrosivity test specimen.
(Line pairs are counted 1 through 7 from left to right.)

2. Thick Film Pattern:

Same as thin film pattern, except spacing of first two pairs is 0.0127 cm (5 mils).

Test specimens were prepared for the various adhesives by applying a small dot of adhesive across each line pair or, in the case of the film adhesive, by placing a strip across all line pairs, and then curing at the proper cure schedule. Voltage was then applied across alternating pairs of lines (i.e., line pairs 1, 3, 5, and 7) only, and the specimens were subjected to a high temperature/relative humidity environment.

After several runs on specimens cured in normally available laboratory ovens, it was decided that in some cases there was uncertainty as to whether or not the specimens were being contaminated by extraneous residues from the ovens (e.g., residual products outgassed from the previously cured adhesive). Tests on specimens without adhesives applied but placed in the oven for a length of time representative of the average cure time of the adhesives used and on fresh specimens that had not been so treated indicated that there possibly was some validity to this suspicion. In any event, whether or not such was the case, the decision was made that the small additional effort involved to eliminate this uncertainty did not justify the risk, so all test specimens were cured on a carefully controlled and constantly monitored hotplate.

For the particular tests for which results will be discussed, the applied potential was 50 volts and the specimens were exposed to an 85°C/100 percent relative humidity environment for 24 hours. Specimens which did not have adhesive applied to them were similarly run as controls to provide normalization data. Both before and after subjection to these conditions, the test specimens were visually examined under a microscope (30X), the resistance of each of the individual lines and the interline resistance of line pairs were measured, and photographs were taken to provide a permanent record of any changes that occurred. Voltage was applied across alternating line pairs only so that information could be obtained on both the innate chemical corrosivity of the adhesives at the high temperature/relative humidity conditions and their electrolytic corrosivity. Visual observation also was made of the run-out, creep or capillarity of the adhesives after cure.

Metallization systems used were as follows:

1. Thin Film Aluminum:

   a. Titanium ≈100 Å (to improve adhesion).

   b. Aluminum ≈10 000 Å.

2. Thin Film Gold:

   a. Nichrome ≈170 Å (to improve adhesion).

   b. Nickel ≈1000 Å (chromium diffusion barrier).

   c. Vacuum Deposited Gold ≈10 000 Å.

   d. Electroplated Gold ≈25 000 Å.

3. Thick Film Gold:

   a. Conductor Ink — EMCA 212B.

   b. Thickness — 0.00178 cm (0.7 mil).

   c. Firing Temperature — 990^{+10}_{-5} °C.

Line resistances were measured with a Simpson Model 1699 Multi-Range Precision Milliohmmeter. The new Beckman Instruments Model L-8 Megohmmeter capable of measuring up to 10^{16} ohms was used to measure the interline or insulation resistance between line pairs.

IV. DISCUSSION OF RESULTS

A. Bond Strength

Bond strength measurements were made for a comparatively large number of adhesives at both room temperature and a maximum use temperature of 150°C (300°F). As previously stated, room temperature bond strengths were determined for specimens under a variety of conditions, and maximum use temperature bond strengths were determined only for freshly prepared specimens. Results are presented in Tables 6 through 10. Due to the fact that for the majority of the tests, the maximum capability of the tester was limited to approximately 3470 grams, or approximately 2.11×10^7 N/m^2 (3060 psi), for the 0.127 x 0.127 cm (0.050 x 0.050 in.) die used, results are stated by giving the number of specimens that exceeded this limit of 2.11×10^7 N/m^2 (3060 psi) and the number of specimens and the average value of their bond strength for those that did not. The following cure schedules recommended by the adhesive manufacturers were used:

Hysol 0151 — 2 hours at 60°C.

Hysol 0266 — 2 hours at 60°C.

Ablefilm 517A — 3 hours at 75°C.

Ablefilm 532 — 3 hours at 75°C.

Eccobond 104 — 6 hours at 120°C.

Epo-Tek H54 — 30 min at 100°C.

Epo-Tek H55 — 20 min at 100°C.

Epo-Tek H61 — 15 min at 150° C.

Epo-Tek H72 — 20 min at 100°C.

Epo-Tek H74 — 20 min at 100°C.

Ablebond 161-3 — 2 hours at 75°C.

Ablebond 450 — 1 hour at 120°C.

Ablefilm 535 — 2 hours at 120°C.

Epoxylite 6203 — 4 hours at 120°C.

TABLE 6. BOND STRENGTH OF FRESHLY PREPARED SPECIMENS

Adhesive	Substrate Type	Room Temperature (psi)[a]	150°C (psi)[a, b]
Hysol 0151	Glazed	All > 3060	All>3060
	Unglazed	All > 3060	8>3060 2 Avg 2450
Hysol 0266	Glazed	All > 3060	——
	Unglazed	All > 3060	——
Ablefilm 517A	Glazed	All > 3060	4>3060 6 Avg 2350
	Unglazed	All > 3060	Avg 2470
Ablefilm 532	Glazed	All > 3060	——
	Unglazed	All > 3060	——
Eccobond 104	Glazed	All > 3060	All>3060
	Unglazed	All > 3060	All>3060
Epo-Tek H54	Glazed	All > 3060	2>3060 9 Avg 2290
	Unglazed	All > 3060	7>3060 3 Avg 2250
Epo-Tek H55	Glazed	All > 3060	Avg 1090
	Unglazed	All > 3060	Avg 890
Epo-Tek H61	Glazed	All > 3060	9>3060 1 2830
	Unglazed	All > 3060	All>3060
Epo-Tek H72	Glazed	All > 3060	——
	Unglazed	9 > 3060 1 2600	——
Epo-Tek H74	Glazed	All > 3060	All>3060
	Unglazed	All > 3060	All>3060
Ablebond 161-3	Glazed	All > 3060	1>3060 9 Avg 1900
	Unglazed	All > 3060	3>3060 6 Avg 1940
Ablebond 450	Glazed	3 > 3060 7 Avg 2100	2>3060 8 Avg 2295
	Unglazed	9 > 3060 1 2820	3>3060 7 Avg 2115

TABLE 6. (Concluded)

Adhesive	Substrate Type	Room Temperature (psi)[a]	150°C (psi)[a, b]
Ablefilm 535	Glazed	All > 3060	Avg 855
	Unglazed	All > 3060	Avg 1140
Epoxylite 6203	Glazed	All > 3060	9>3060 1 2645
	Unglazed	All > 3060	All>3060

a. 1 psi = 6895 N/m^2.
b. — means measurement was not made due to loss of specimens.

TABLE 7. ROOM TEMPERATURE BOND STRENGTH AFTER 30 MIN IMMERSION IN COMMONLY USED SOLVENTS

Adhesive	Substrate Type	Freon TF (psi)[a]	Isopropyl Alcohol (psi)[a]	Trichloroethylene (psi)[a]
Hysol 0151	Glazed	All>3060	All>3060	9>3060 1 2450
	Unglazed	All>3060	All>3060	All>3060
Ablefilm 517A	Glazed	All>3060	All>3060	All>3060
	Unglazed	All>3060	All>3060	All>3060
Eccobond 104	Glazed	All>3060	All>3060	All>3060
	Unglazed	All>3060	All>3060	All>3060
Epo-Tek H54	Glazed	All>3060	All>3060	All>3060
	Unglazed	All>3060	All>3060	All>3060
Epo-Tek H55	Glazed	9>3060 1 2910	All>3060	All>3060
	Unglazed	All>3060	All>3060	All>3060
Epo-Tek H61	Glazed	All>3060	All>3060	All>3060
	Unglazed	All>3060	All>3060	All>3060
Epo-Tek H72	Glazed	All>3060	All>3060	All>3060
	Unglazed	All>3060	All>3060	All>3060
Epo-Tek H74	Glazed	All>3060	All>3060	All>3060
	Unglazed	All>3060	All>3060	All>3060

a. 1 psi = 6895 N/m^2.

TABLE 8. ROOM TEMPERATURE BOND STRENGTH AFTER 10 DAYS AGING

Adhesive	Substrate Type	Room Temperature (psi)[a]	150°C (psi)[a]
Hysol 0151	Glazed	7>3060 2 Avg 2450	All>3060
	Unglazed	7>3060 3 Avg 1810	All>3060
Ablefilm 517A	Glazed	All>3060	All>3060
	Unglazed	All>3060	All>3060
Eccobond 104	Glazed	All>3060	All>3060
	Unglazed	All>3060	All>3060
Epo-Tek H54	Glazed	All>3060	All>3060
	Unglazed	All>3060	All>3060
Epo-Tek H55	Glazed	All>3060	All>3060
	Unglazed	All>3060	All>3060
Epo-Tek H61	Glazed	All>3060	All>3060
	Unglazed	All>3060	All>3060
Epo-Tek H72	Glazed	9>3060 1 2300	All>3060
	Unglazed	All>3060	All>3060
Epo-Tek H74	Glazed	All>3060	All>3060
	Unglazed	All>3060	All>3060
Ablebond 161-3	Glazed	All>3060	All>3060
	Unglazed	All>3060	All>3060
Ablebond 450	Glazed	Avg 1115	All>3060
	Unglazed	7>3060 3 Avg 2110	All>3060
Epoxylite 6203	Glazed	All>3060	All>3060
	Unglazed	All>3060	All>3060

a. 1 psi = 6895 N/m^2.

TABLE 9. ROOM TEMPERATURE BOND STRENGTH AFTER 30 DAYS AGING

Adhesive	Substrate Type	Room Temperature (psi)[a]	150°C (psi)[a]
Hysol 0151	Glazed	9>3060	All>3060
		1 1380	
	Unglazed	All>3060	All>3060
Ablefilm 517A	Glazed	All>3060	All>3060
	Unglazed	All>3060	All>3060
Eccobond 104	Glazed	All>3060	All>3060
	Unglazed	All>3060	All>3060
Epo-Tek H54	Glazed	All>3060	All>3060
	Unglazed	All>3060	All>3060
Epo-Tek H55	Glazed	All>3060	9>3060
			1 2300
	Unglazed	All>3060	All>3060
Epo-Tek H61	Glazed	All>3060	All>3060
	Unglazed	All>3060	All>3060
Epo-Tek H72	Glazed	All>3060	All>3060
	Unglazed	All>3060	All>3060
Epo-Tek H74	Glazed	All>3060	All>3060
	Unglazed	All>3060	All>3060
Ablebond 161-3	Glazed	8>3060	All>3060
		2 Avg 2205	
	Unglazed	9>3060	All>3060
		1 2205	
Ablebond 450	Glazed	Avg 2560	9>3060
			1 2515
	Unglazed	7>3060	All>3060
		3 Avg 2375	
Epoxylite 6203	Glazed	All>3060	All>3060
	Unglazed	All>3060	All>3060

a. 1 psi = 6895 N/m^2.

TABLE 10. ROOM TEMPERATURE BOND STRENGTH AFTER AT LEAST 90 DAYS AGING[a]

Adhesive	Aging Interval (days)	Substrate Type	Room Temperature (psi)[b]	150°C (psi)[b, c]
Hysol 0151	123	Glazed	All>3060	9>3060 1 2515
		Unglazed	4>3060 6 Avg 1535	All>3060
Ablefilm 517A	149	Glazed	All>3060	All>3060
		Unglazed	All>3060	All>3060
Eccobond 104	122	Glazed	All>3060	All>3060
		Unglazed	All>3060	All>3060
Epo-Tek H54	99	Glazed	1>3060 9 Avg 1985	All>3060
		Unglazed	5>3060 5 Avg 2025	All>3060
Epo-Tek H55	98	Glazed	8>3060 1 2190	Avg 1865
		Unglazed	5>3060 5 Avg 2515	8 Avg 1280 1 Zero
Epo-Tek H61	99	Glazed	All>3060	8>3060 2 Avg 2450
		Unglazed	All>3060	All>3060
Epo-Tek H72	111	Glazed	6>3060 4 Avg 2545	All>3060
		Unglazed	3>3060 7 Avg 2175	—
Epo-Tek H74	99	Glazed	3>3060 6 Avg 2450	All>3060
		Unglazed	All>3060	All>3060
Ablebond 161-3	90	Glazed	All>3060	All>3060
		Unglazed	All>3060	All>3060

TABLE 10. (Concluded)

Adhesive	Aging Interval (days)	Substrate Type	Room Temperature (psi)b	150°C (psi)b, c
Ablebond 450	156	Glazed	9 Avg 1005 1 Zero	All>3060
		Unglazed	1>3060 9 Avg 1805	All>3060
Epoxylite 6203	156	Glazed	All>3060	All>3060
		Unglazed	All>3060	All>3060

a. Actual aging interval is given since it varied quite widely from 90 days.
b. 1 psi = 6895 N/m ^{2}.
c. — means measurement was not made due to loss of specimen.

Summary conclusions obtained from a review of Tables 6 through 10 are as follows:

1. All adhesives tested form bonds on unglazed substrates with initial room temperature strengths exceeding the measurable limit of the tester, i.e., 2.11×10^7 N/m^2 (3060 psi), and all except Ablebond 450 give bond strengths exceeding this limit on glazed substrates.

2. The room temperature bond strengths of the adhesives tested are not affected by prolonged immersion in the commonly used solvents — Freon TF, isopropyl alcohol, or trichloroethylene. Hysol 0266, Ablefilm 532, Ablebond 161-3, Ablebond 450, Ablefilm 535, and Epoxylite 6203 included in the previous list were not tested.

3. All adhesives tested except Epo-Tek H55 and possibly Epo-Tek H61 retained a room temperature bond strength greater than the measurable limit of the tester [2.11×10^7 N/m^2 (3060 psi)] on both glazed and unglazed substrates after prolonged exposure (at least 90 days) to the maximum use temperature of 150°C. Severe degradation of the room temperature bond strength of Epo-Tek H55 occurred sometime after 30 days, dropping to an average value of 1.29×10^7 N/m^2 (1865 psi) for glazed substrates and 8.83×10^6 N/m^2 (1280 psi) for unglazed substrates at the end of 98 days. Degradation of the bond strength of Epo-Tek H61 is somewhat questionable

since 8 of the 10 specimens still exceeded 2.11×10^7 N/m^2 (3060 psi). Hysol 0266, Ablefilm 532, and Ablefilm 535 included in the original list were not tested.

4. Only 5 of the 11 adhesives tested retained room temperature bond strengths exceeding the measurable limit of the tester [2.11×10^7 N/m^2 (3060 psi)] on both glazed and unglazed substrates after long term aging of at least 90 days at room temperature. These are Ablefilm 517A, Eccobond 104, Epo-Tek H61, Ablebond 161-3, and Epoxylite 6203. Degradation occurred for Hysol 0151 and Epo-Tek H55 on unglazed substrates only, and for Epo-Tek H74 on glazed substrates only. Degradation occurred on both glazed and unglazed substrates for Epo-Tek H54, Epo-Tek H72, and Ablebond 450, and was greater on glazed substrates for Epo-Tek H54 and Ablebond 450. Again, Hysol 0266, Ablefilm 532, and Ablefilm 535 included in the original list were not tested.

5. Of the 11 adhesives tested at the maximum use temperature (150°C), only Eccobond 104, Epo-Tek H61, Epo-Tek H74, Epoxylite 6203, and possibly Hysol 0151 gave bond strengths exceeding 2.11×10^7 N/m^2 (3060 psi). Bond strengths of the other 6 adhesives varied over a range down to an average value of 6.14×10^6 N/m^2 (890 psi) for unglazed substrates and 7.52×10^6 N/m^2 (1090 psi) for glazed substrates for Epo-Tek H55, and 7.86×10^6 N/m^2 (1140 psi) for unglazed substrates and 5.90×10^6 N/m^2 (855 psi) for glazed substrates for Ablefilm 535. Hysol 0266, Ablefilm 532, and Epo-Tek H72 were not tested under this condition.

B. Outgassing After Cure

Curves showing total outgassed pressure as a function of time for the five selected adhesives are given in Figures 5 through 9. Specimens were aged at 150°C in a controlled oven. Cure schedules used for the adhesives were as follows:

Hysol 0151 — 2 hours at 60°C.

Ablefilm 517A — 1 hour at 120°C.

Eccobond 104 — 6 hours at 120°C.

Epo-Tek H74 — 20 min at 100°C.

Epo-Tek H61 — 15 min at 150°C.

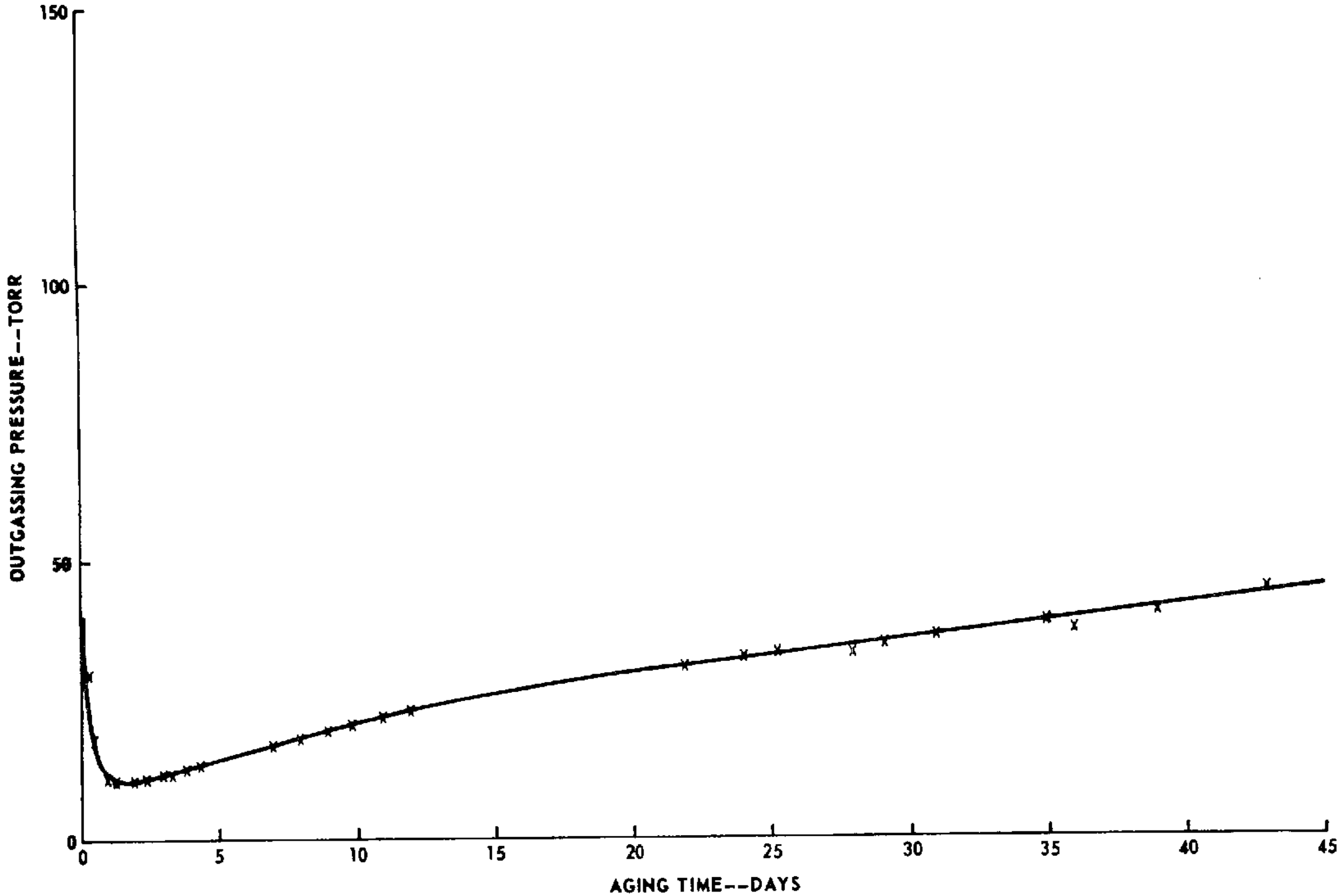


Figure 5. Outgassing of Hysol 0151 after curing for 2 hr at 60°C (140° F).

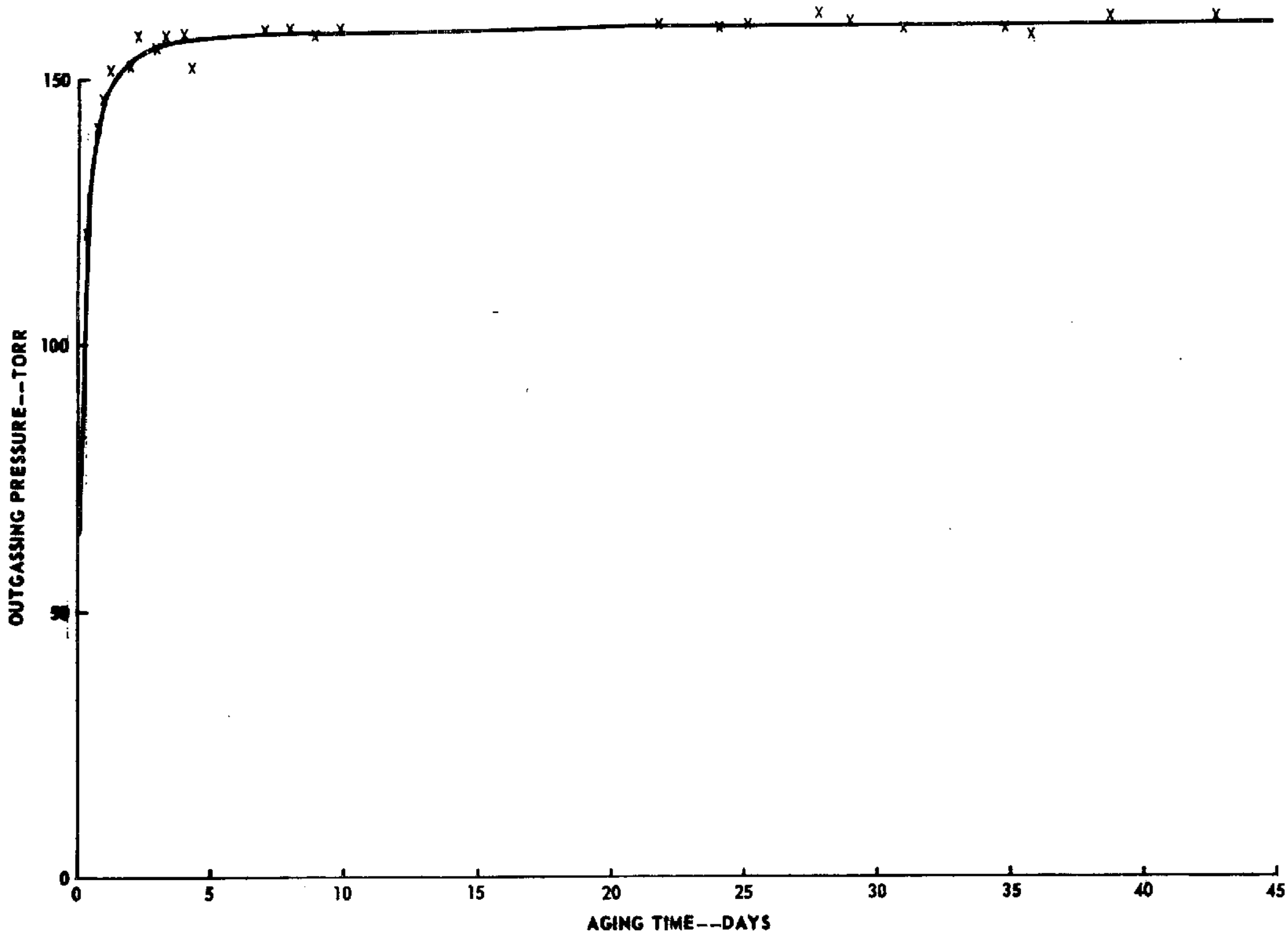


Figure 6. Outgassing of Ablefilm 517A after curing for 1 hr at 120°C (250° F).

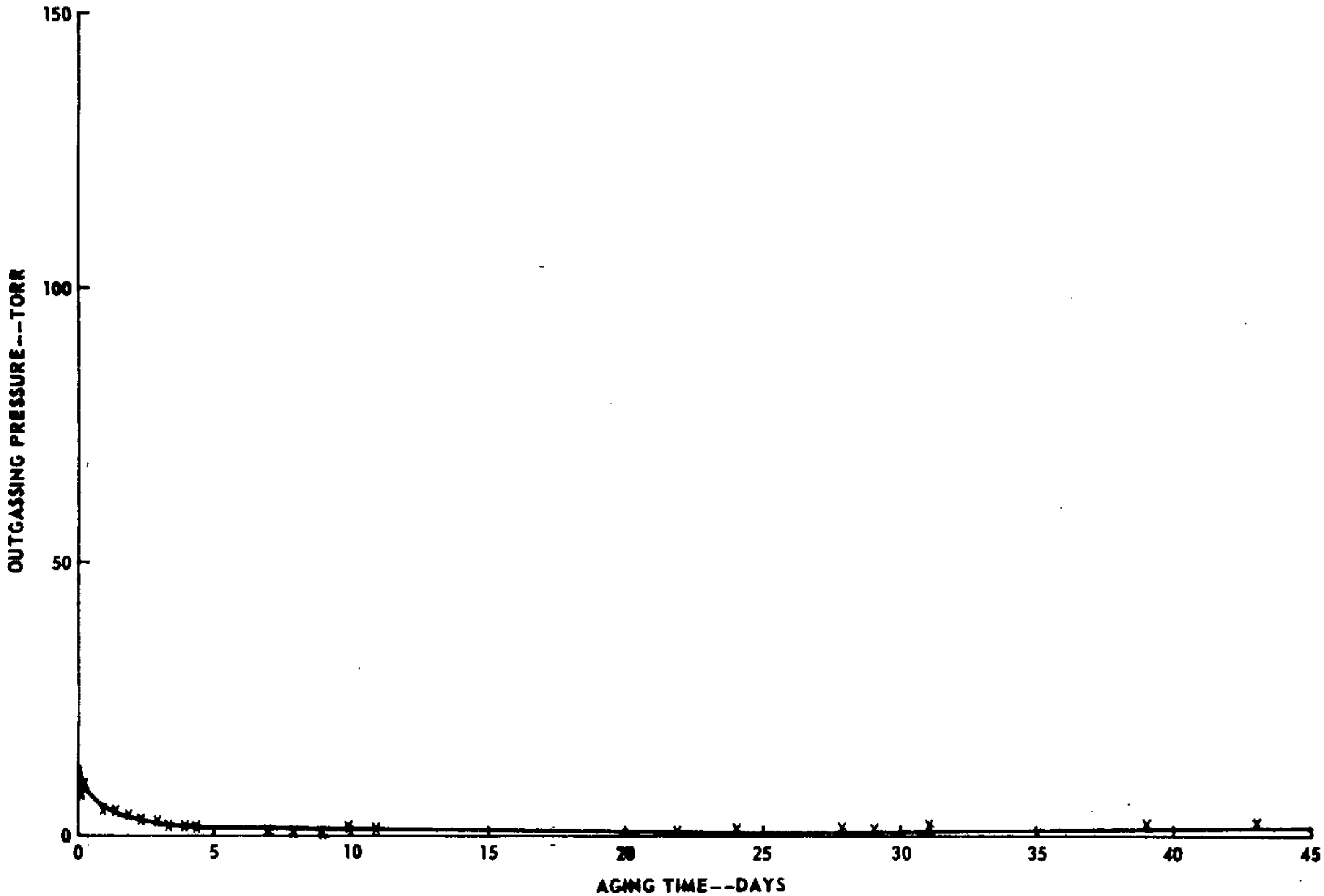


Figure 7. Outgassing of Eccobond 104 after curing for 6 hr at 120°C (250° F).

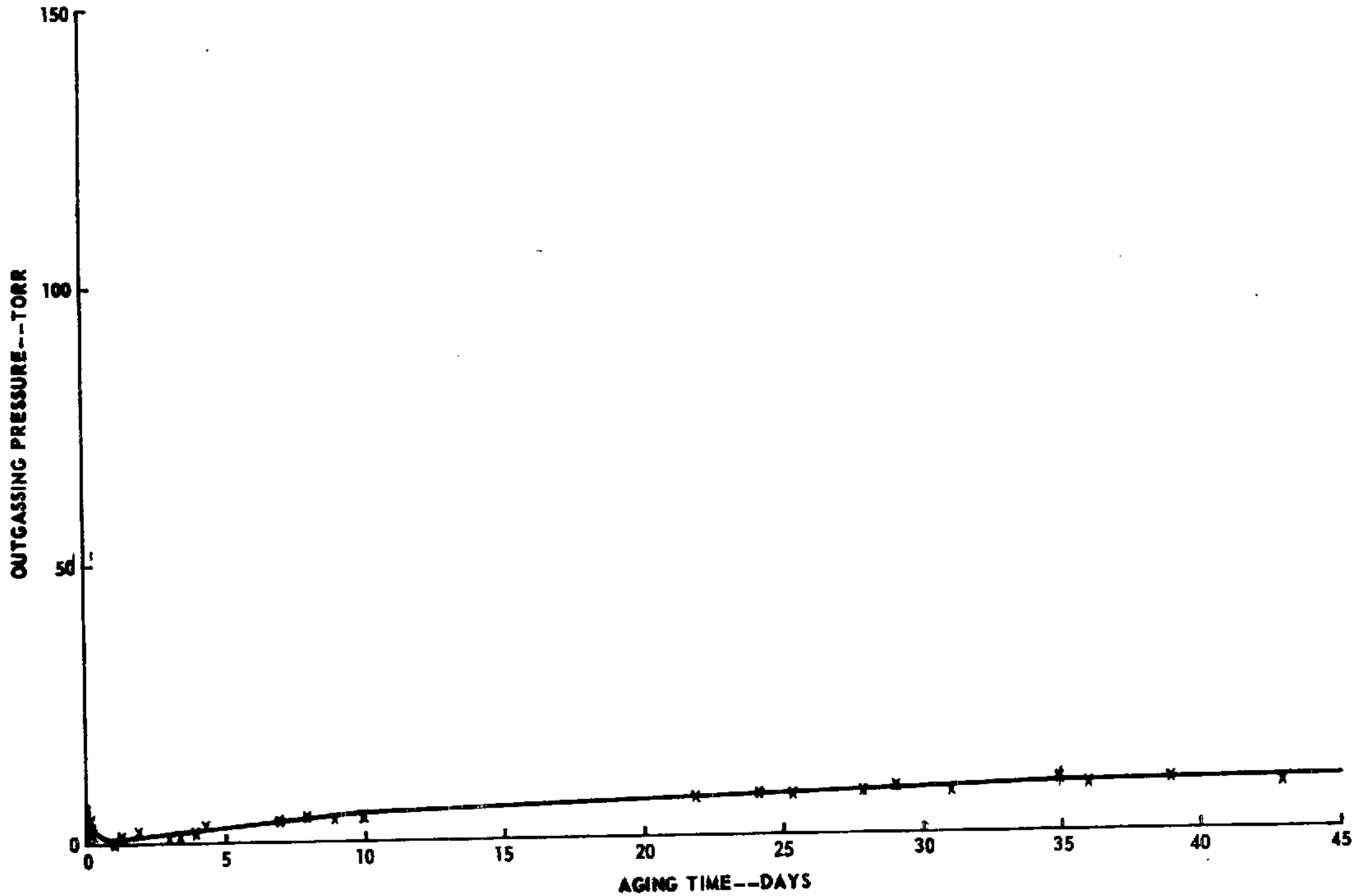


Figure 8. Outgassing of Epo-Tek H74 after curing for 20 min at 100°C (212° F).

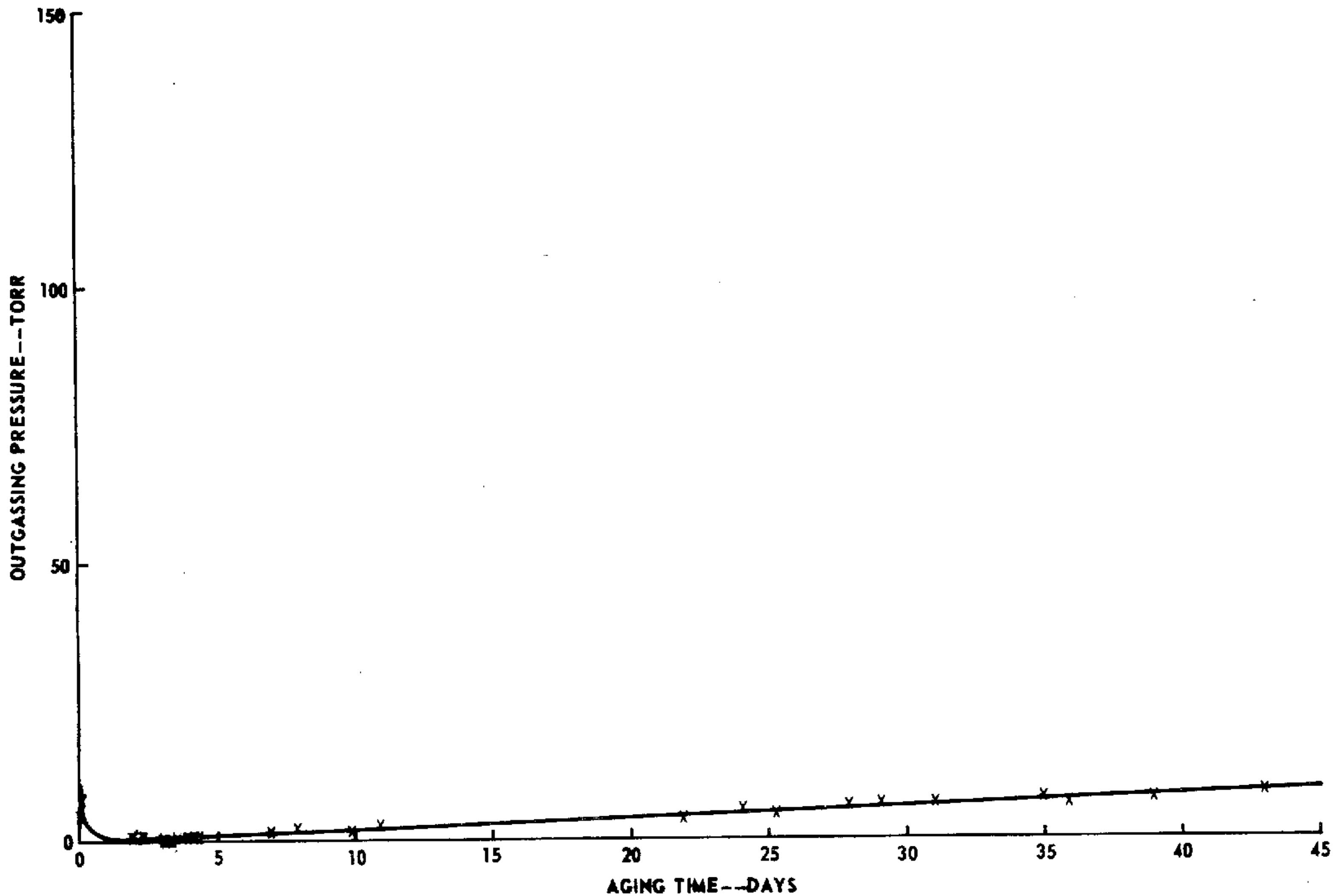


Figure 9. Outgassing of Epo-Tek H61 after curing for 15 min at 150°C (300° F).

It is obvious from a comparison of these curves that Ablefilm 517A (for the given cure schedules) is by far the largest outgasser, and that Eccobond 104 is the smallest, outgassing a comparatively negligible amount. Also, both Epo-Tek adhesives outgas relatively little. However, it also should be noted that while Ablefilm 517A outgasses a substantial amount, essentially all outgassing occurs in the first few days and the outgassed pressure remains almost constant thereafter. A similar situation also occurs for Eccobond 104; i.e., after the initial outgassing, the outgassed pressure does not continue to increase. However, for the other three adhesives, and particularly for Hysol 0151, while initial outgassing is relatively small compared to that of Ablefilm 517A, outgassing continues and the pressure in the tube continues to increase with extended aging. This suggests that Ablefilm 517A and Eccobond 104 are stable at the aging temperature (150°C) while the other three adhesives are slowly outgassing or, perhaps, decomposing. Use of higher curing temperature (particularly for Hysol 0151) or extension of curing time may improve the performance of these adhesives. Further investigation should be made to determine whether or not this is the case.

Analysis of the curves also shows that in all cases except Ablefilm 517A there is an initial positive offset in the outgassed pressure after which the pressure drops to its minimum value in a few hours or in a day or two. It is felt that this effect should be ignored since it probably is not indicative of the real situation occurring but is due to some vagary of the experimental setup, such as unequal outgassing of the mercury columns of the manometers. Also, it is felt that this situation occurred for Ablefilm 517A but was masked by the initial large outgassing of this adhesive. In all cases, the true initial portion of the curve is probably more correctly represented by extending the curve from its minimum pressure point back through the origin. The validity of this supposition is supported by the fact that similar tests (on other adhesives) now in progress using manometers filled with fresh triple distilled mercury and carefully outgassed before seal-off do not show this effect.

Results of gas chromatographic and mass spectrometric analyses of the outgassed products of the various adhesives showed that in general the major components consist of normal atmospheric gases (N_2, H_2, CO_2, CO) and various organics. Exceptions are Ablefilm 517A, which was by far the largest outgasser, and Hysol 0151, which while a medium initial outgasser continues to outgas substantially with extended aging. On the basis of supportive tests, it was found that the major portion of the large quantity of material outgassed from Ablefilm 517A is tetrahydrofuran, a solvent used in its formulation. The analyses of Hysol 0151 showed large quantities of organics and a considerable quantity of water, supporting the previously suggested speculation that long term aging at 150°C causes decomposition of this adhesive.

C. Corrosivity

Corrosivity tests were performed for the same five adhesives selected for the outgassing study just discussed. The adhesives also were cured using the same cure schedules identified in that discussion. Testing consisted of exposing the specimens to an 85°C/100 percent relative humidity environment for 24 hours with 50 volts applied to alternating line pairs (line pairs 1, 3, 5, and 7) and the other line pairs left open (line pairs 2, 4, and 6). For those line pairs to which potential was applied, the right line (or the line toward the side of the substrate where the F is located) was made positive. Photographs showing the results obtained are given as Figures 10 through 24. Photographs of new (unexposed) specimens, and of control specimens similarly tested but with no adhesive applied are given as Figures 25 through 27 and 28 through 30, respectively.

The following is a discussion of the results obtained from visual observation under a microscope at 30X; they can be substantiated at least to a reasonable extent by reference to the appropriate photographs. Detailed data on the resistance of each of the lines and the interline or insulation resistance of line pairs before and after exposure corroborating the following discussion are tabulated in the Appendix. In several cases, line pairs arced over during application of the high voltage (up to 700 volts) required to measure the interline or insulation resistance. Evidence of such arcing is obvious and easily distinguished from corrosion.

1. Control Set

a. Thin Film Aluminum — Some general overall corrosion that appears to be independent of whether or not voltage was applied to the line pair.

b. Thin Film Gold — No evidence of corrosion; however, several small arcing spots on first line pair.

c. Thick Film Gold — No evidence of corrosion; however, evidence of slight arcing on third line pair.

2. Hysol 0151

a. Thin Film Aluminum — Very slight (negligible) creeping or haloing of adhesive. Evidence of extensive corrosion. Positive lines of line pairs 1, 3, and 5 are corroded through, a large portion of line pairs 1 and 3 at the adhesive edges and a small portion of line pair 5 under the adhesive. Also, the negative line of line pair 7 is considerably darkened under the adhesive. Even line pairs 2 and 4 appear discolored and the negative line of line pair 2 is corroded through but at quite some distance from the adhesive.

b. Thin Film Gold — Essentially no creeping or capillarity of adhesive. No corrosion of line pairs 2, 4 and 6. Pinkish-red to black discoloration under adhesive on the positive lines of line pairs 1, 3, 5, and 7 with the extent of discoloration also decreasing in that order. However, measurements of line resistances do not indicate any change.

c. Thick Film Gold — Again, no haloing of adhesive or corrosion of line pairs 2, 4, and 6. However, dark red or black discoloration under adhesive on the positive lines of line pairs 1, 3, 5, and 7 of about the same intensity in all cases. However, again line resistances have not changed.

3. Ablefilm 517A

a. Thin Film Aluminum — No creeping or capillarity of adhesive. Evidence of general overall corrosion that appears to be independent of whether or not voltage was applied to the line pair similar to that obtained for the control set. However, resistance measurement indicates that the positive line of line pair 1 is excessively corroded (line resistance >50 ohms).

b. Thin Film Gold — No creeping or capillarity of adhesive or evidence of corrosion. Line pairs 1, 3, and 4 arced over during insulation resistance measurement.

c. Thick Film Gold — No creeping or capillarity of adhesive or evidence of corrosion. Some line pairs show slight arcing.

4. Eccobond 104

a. Thin Film Aluminum — Considerable creeping or haloing of adhesive extending for a distance from the edge of the adhesive equal to approximately two-thirds of the radius of the adhesive dot. Also, there is additional capillarity along the lines. No corrosion on even numbered line pairs except for line pair 4 on which there are three small spots where metallization has blistered on left line (probably not due to adhesive). Some slight corrosion of the positive lines of all odd pairs with line pair 7 particularly bad, both lines appear open.

b. Thin Film Gold — Slight creeping or haloing of adhesive. No apparent corrosion. Line pairs 1, 2, and 3 have arced over.

c. Thick Film Gold — Considerable haloing of adhesive. No evidence of corrosion.

5. Epo-Tek H74

a. Thin Film Aluminum — Considerable creeping or haloing of adhesive extending out from the adhesive dot for a distance equal to approximately one-half to three-fourths of the radius of the dot. Also, considerable additional capillarity of about the same amount along the lines. No evidence of corrosion on line pairs 2, 4, and 6. Positive lines appear corroded through at edge of adhesive in the halo area for line pairs 1, 3, and 5 and at the bottom edge of halo on line pair 7. The resistance measurements substantiate these results.

b. Thin Film Gold — Some creeping or haloing out to a distance of about one-fourth of the radius of the adhesive dot plus considerable capillarity along the lines greater than the haloing by a factor of about 2 plus. No corrosion on line pairs 2, 4, and 6. Dark reddish brown spots on each side of the adhesive on the positive lines at the adhesive-line interface for line pairs 1 and 3 and similarly located reddish-pink dots for line pairs 5 and 7. Cannot tell if this discoloration runs through the adhesive since it is opaque. Resistance measurements do not show degradation due to this effect.

c. Thick Film Gold — Haloing of the adhesive out to a distance of approximately one-half of the radius of the dot, and capillarity along lines for an additional distance of about one-half of the radius of the dot. Purple discoloration on positive lines and continuing around outside of adhesive in semicircle on right side on line pairs 1, 3, 5, and 7. Discoloration appears to be about the same intensity in all cases. Also, slight light pink discoloration at adhesive-line interface on left-hand line of line pairs 4 and 6. Line resistance measurements do not indicate any effect due to this discoloration; however, the insulation resistance has dropped by 3 or 4 orders of magnitude in all cases.

6. Epo-Tek H61

a. Thin Film Aluminum — Substantial capillarity of the adhesive along the lines for a distance about equivalent to the radius of the dots. No evidence of corrosion on line pairs 2, 4, and 6. Positive lines of line pairs 1, 3, 5, and 7 are corroded through, the latter three at the epoxy line interface. Negative lines of line pairs 1, 5, and 7 are also corroded through. Resistance measurements show that both lines of all odd numbered line pairs are open.

b. Thin Film Gold — No evidence of creeping or capillarity of adhesive. No evidence of corrosion on even numbered line pairs. Evidence of arcing on line pairs 1, 3, and 7. Line resistance measurements do not show any change indicative of corrosion.

c. Thick Film Gold — No evidence of creeping or capillarity of adhesive. No evidence of corrosion on even numbered line pairs. Evidence of arcing or corrosion (grayish-black between lines) at adhesive-line interface on all odd numbered line pairs. Also, some slightly purplish discoloration at bottom adhesive-line interface on positive line of line pair 7. Again, line resistance measurements do not show a substantial change indicative of corrosion.

Review of the above detailed discussion supports the following general conclusions:

1. The 85°C/100 percent relative humidity environment selected for the above testing is exceedingly harsh on the aluminum metallization system, possibly to the extent that it invalidates conclusions based on interpretation of the above results for aluminum specimens. In many cases a substantial amount of aluminum oxide is formed.

2. In all cases, the major evidence of discoloration or corrosion occurs on the positive lines of the line pairs.

3. Of the five adhesives tested, only Ablefilm 517A (tertiary amine cured) and Eccobond 104 (anhydride cured) appear to be suitable for use with aluminum metallization systems.

4. These adhesives also appear to be the best of those tested for use on thin and thick film gold metallization systems, particularly if discoloration is considered to be indicative of corrosion. However, Hysol 0151 (primary, secondary amine cured) and Epo-Tek H61 (boron trifluoride complex cured) may also be usable. Also, while Epo-Tek H74 (probably imidazole cured) may also be usable on thin film gold metallization systems, its use on thick film metallization is questionable because of the large decrease in interline or insulation resistance measured for this case.

5. In order of increasing corrosivity, these results indicate that the tested adhesives should be listed as follows:

a. Eccobond 104 or Ablefilm 517A.

b. Hysol 0151.

c. Epo-Tek H61.

d. Epo-Tek H74.

However, in conclusion of this discussion, it should be emphasized that the test environment was purposely chosen to be exceedingly harsh (hopefully to accelerate the corrosion effect and simulate long term usage) and that the conclusions given are based upon observations of only a single set of test specimens subjected to this environment. Thus, in the less severe environments associated with packaged hybrid microcircuits, all of the adhesives may well be acceptable for use.

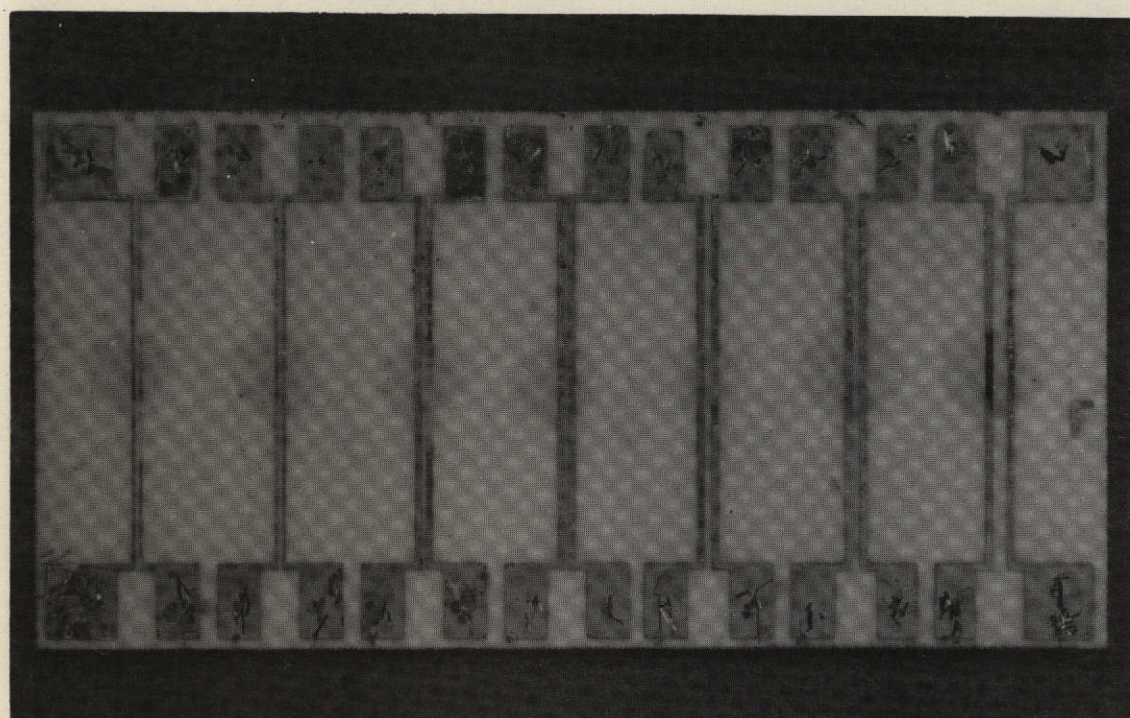


Figure 10. Hysol 0151-thin film aluminum exposed for 24 hr.

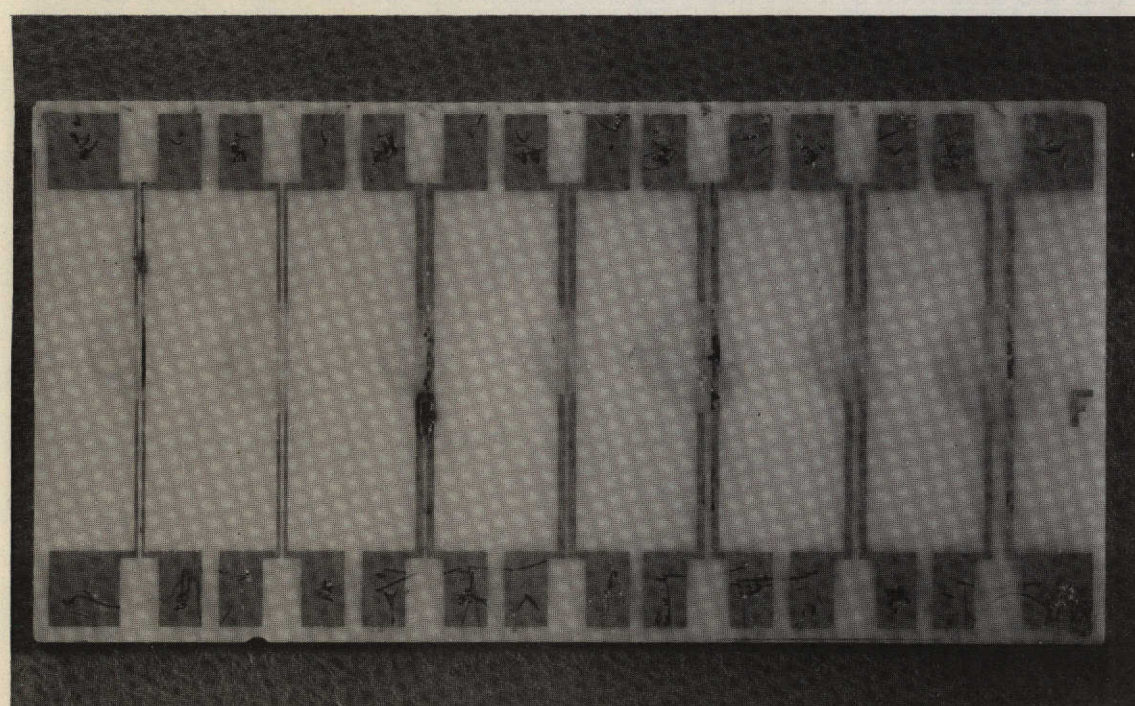


Figure 11. Hysol 0151-thin film gold exposed for 24 hr.

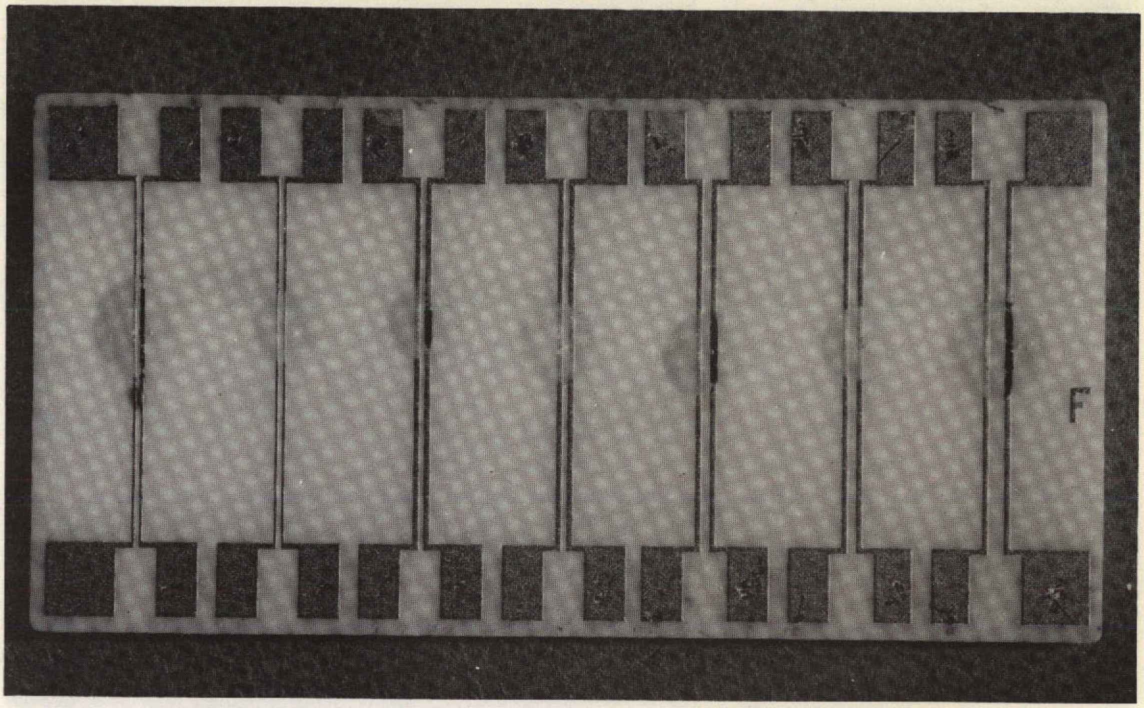


Figure 12. Hysol 0151-thick film gold exposed for 24 hr.

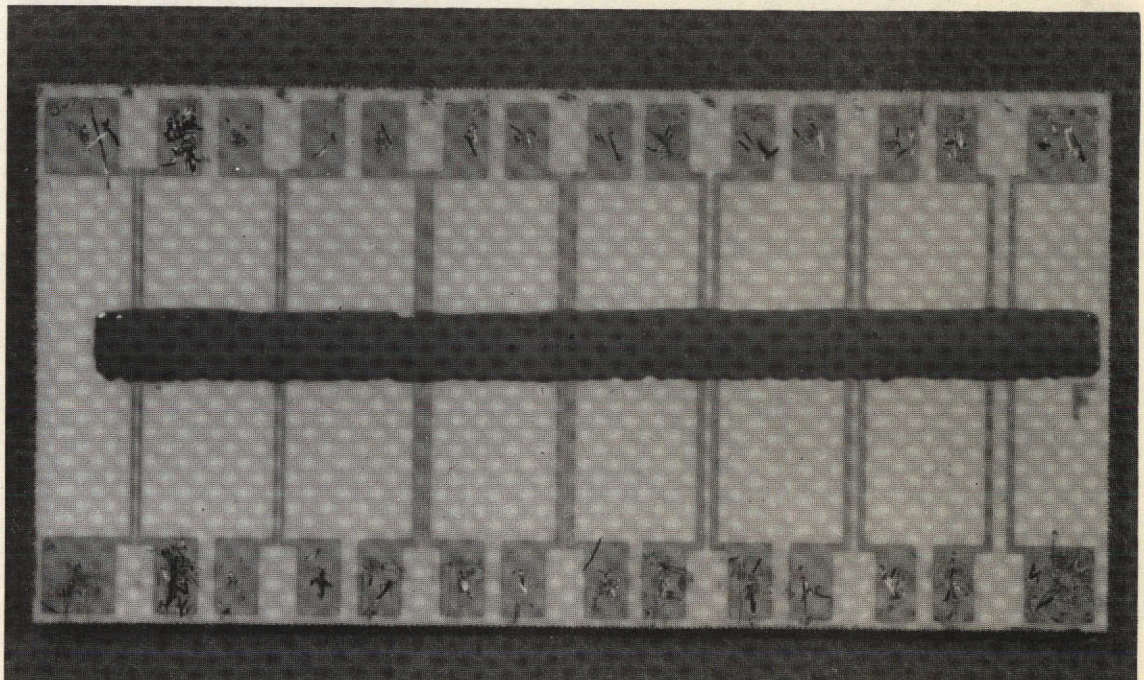


Figure 13. Ablefilm 517A-thin film aluminum exposed for 24 hr.

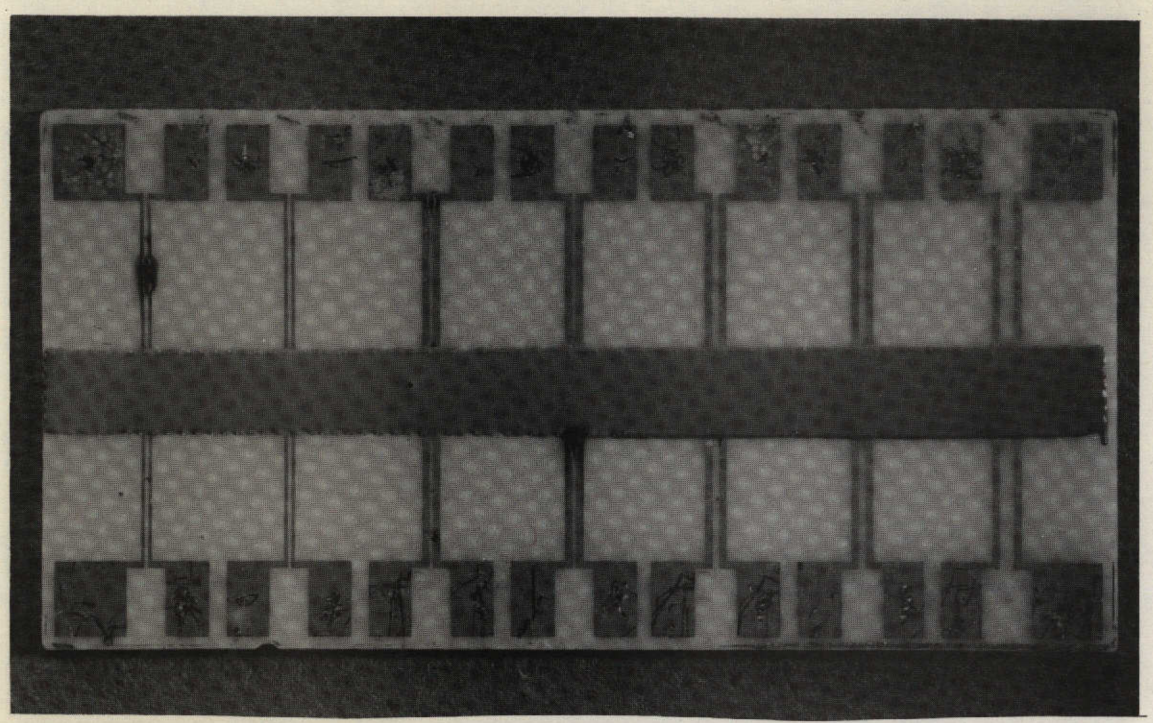


Figure 14. Ablefilm 517A-thin film gold exposed for 24 hr.

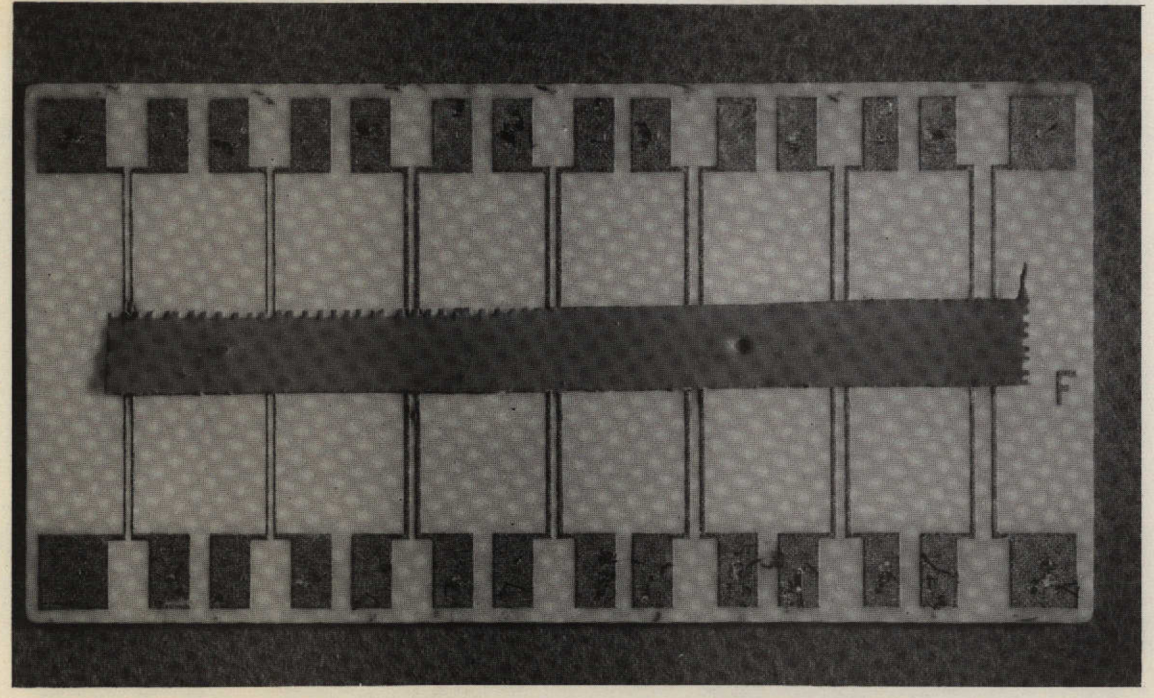


Figure 15. Ablefilm 517A-thick film gold exposed for 24 hr.

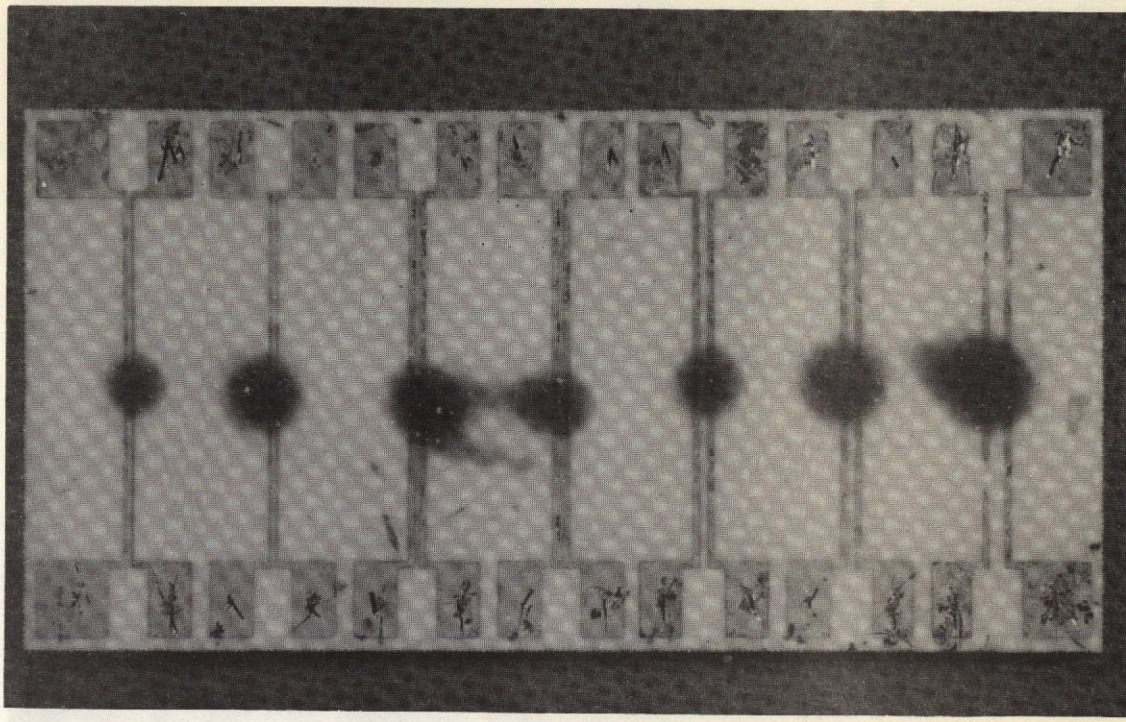


Figure 16. Eccobond 104-thin film aluminum exposed for 24 hr.

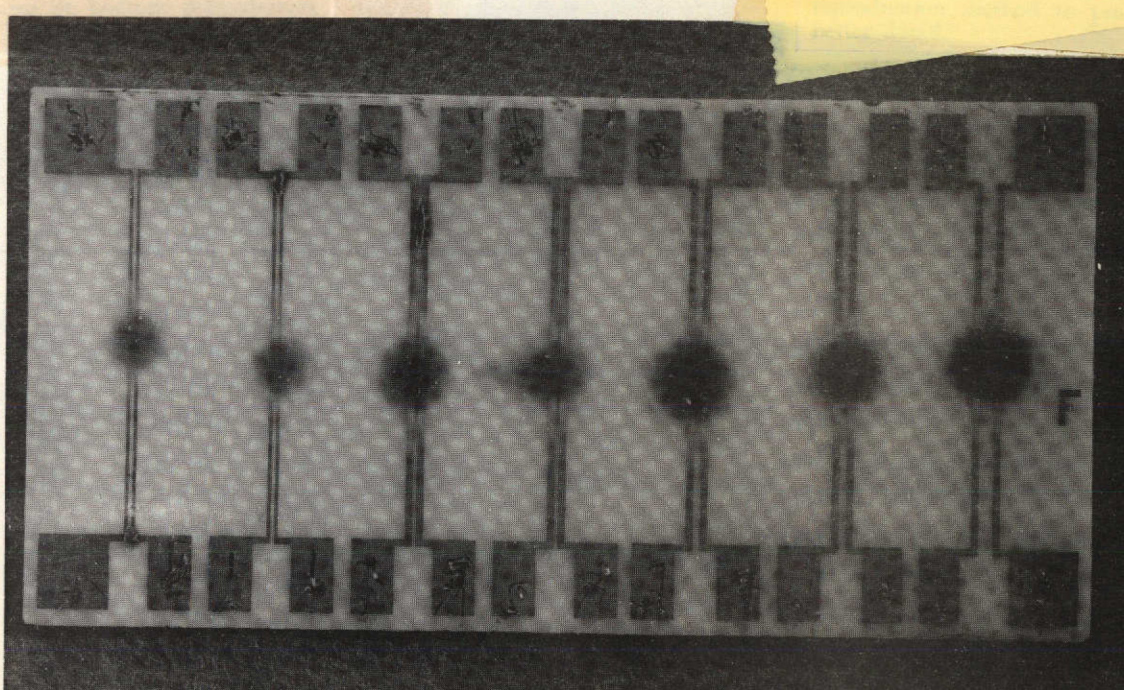


Figure 17. Eccobond 104-thin film gold exposed for 24 hr.

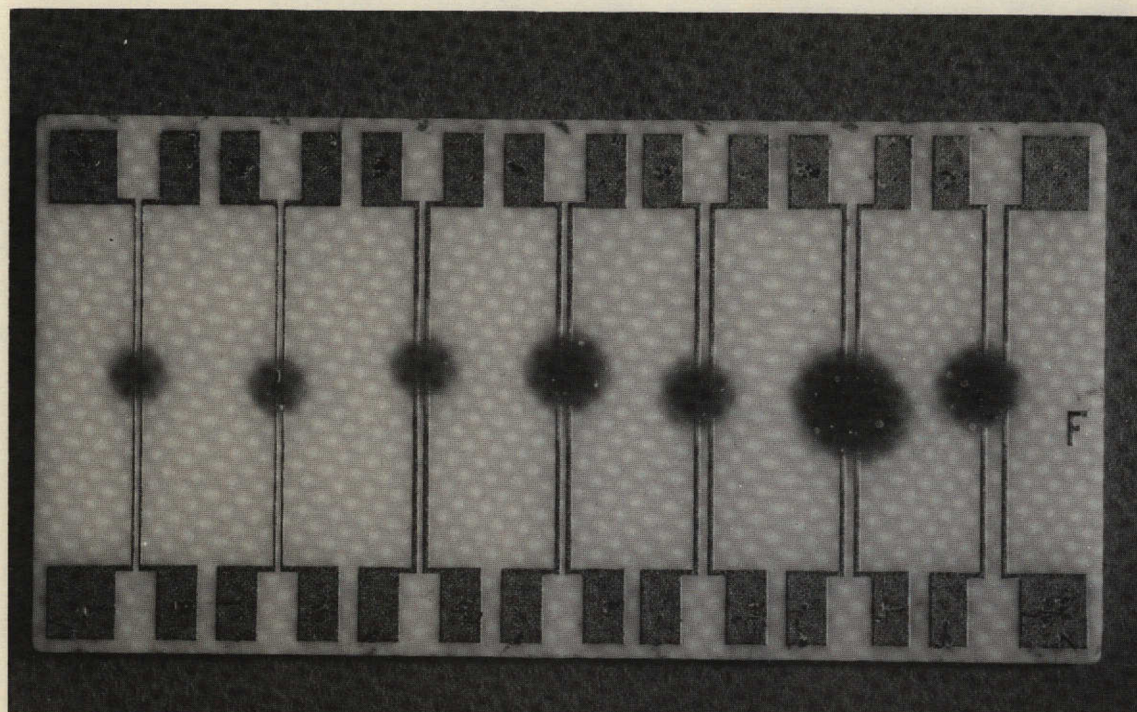


Figure 18. Eccobond 104-thick film gold exposed for 24 hr.

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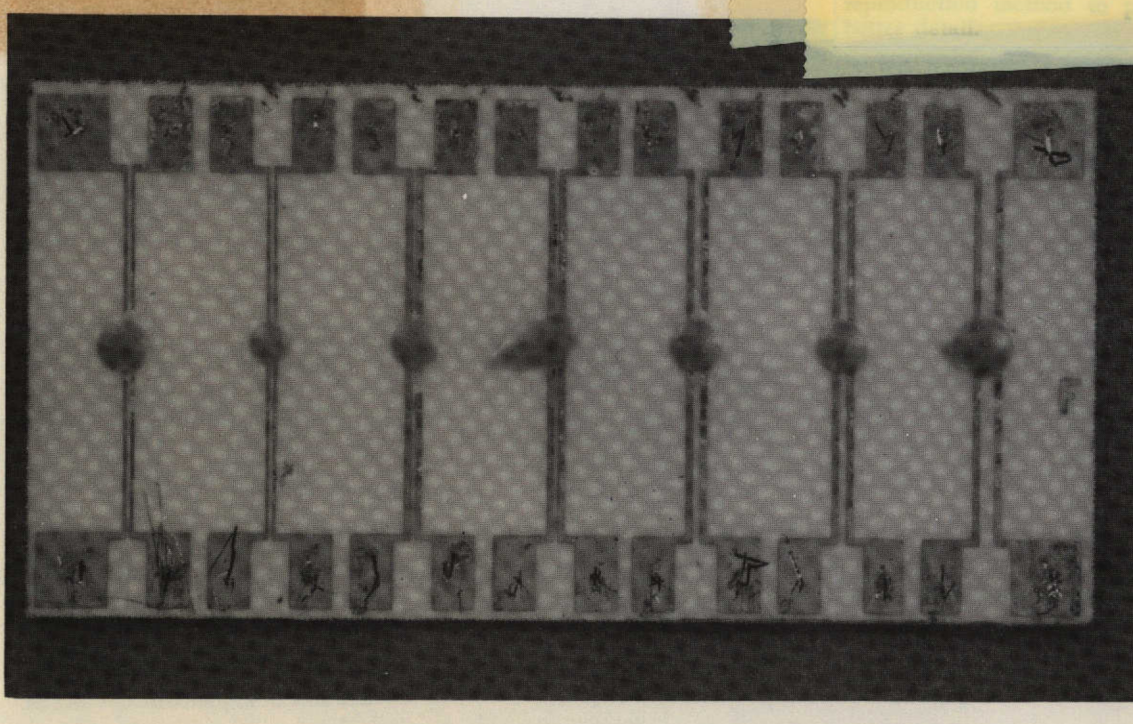

Figure 19. Epo-Tek H74-thin film aluminum exposed for 24 hr.

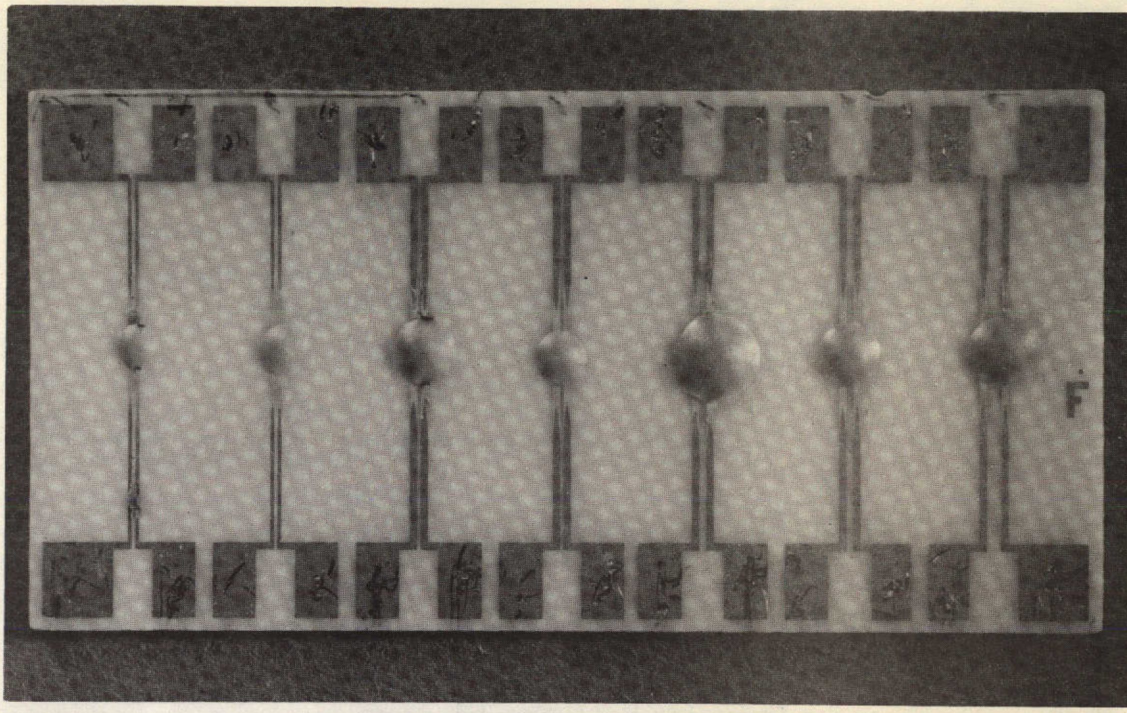

Figure 20. Epo-Tek H74-thin film gold exposed for 24 hr.

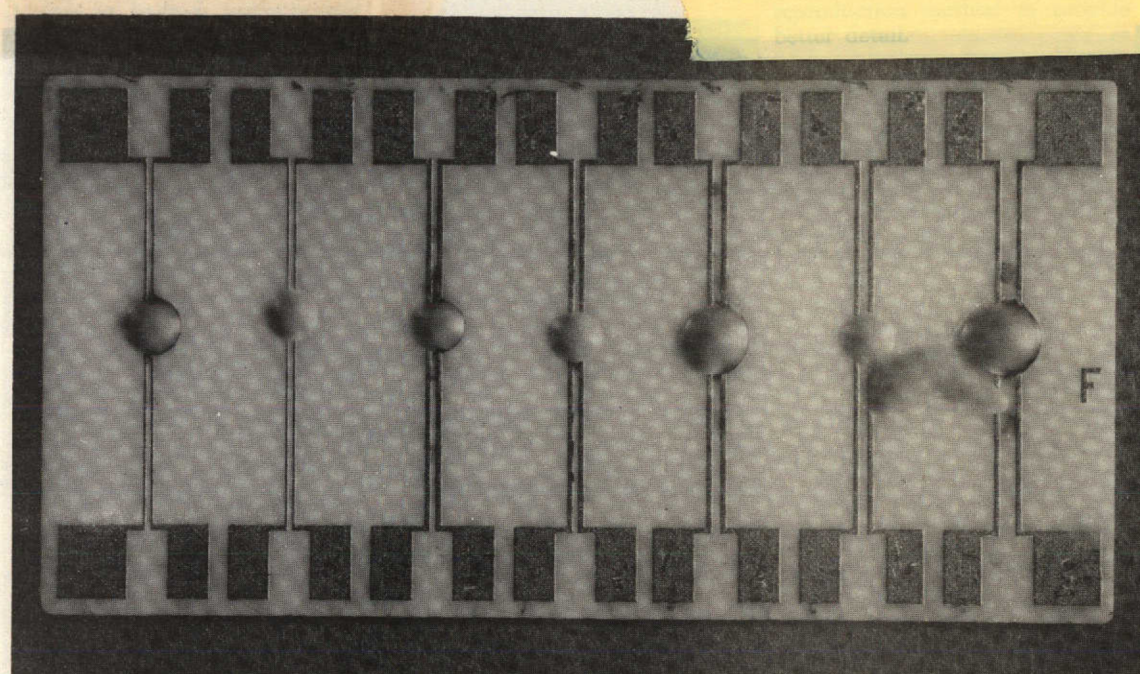

Figure 21. Epo-Tek H74-thick film gold exposed for 24 hr.

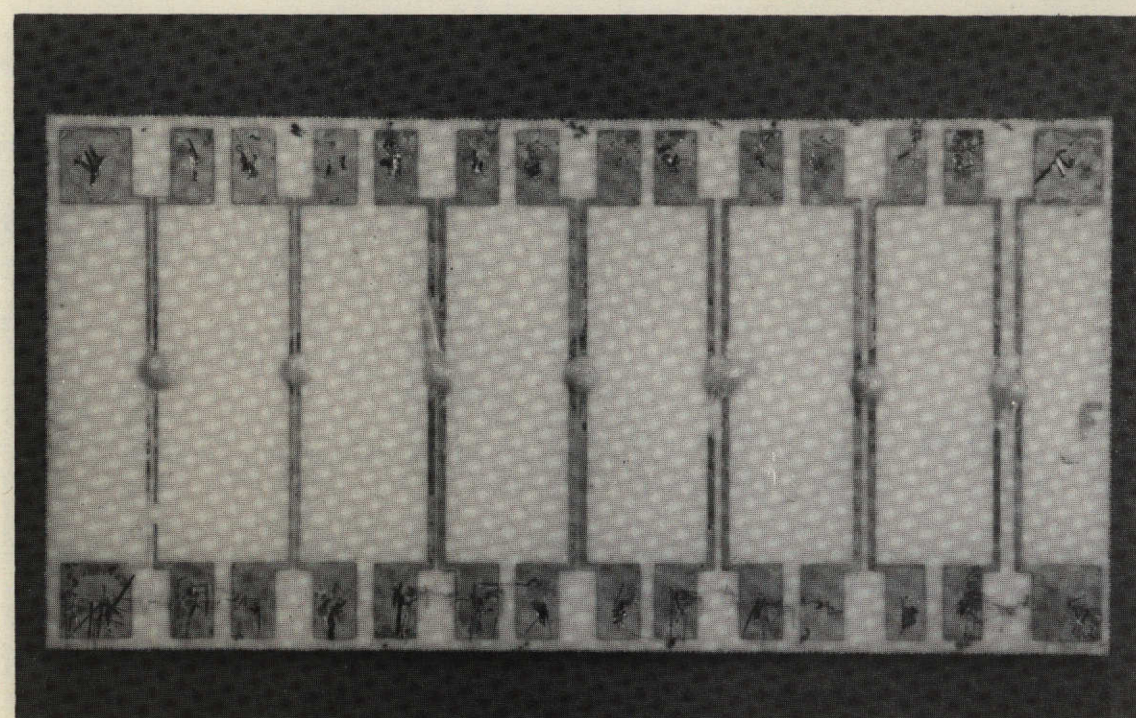

Figure 22. Epo-Tek H61-thin film aluminum exposed for 24 hr.

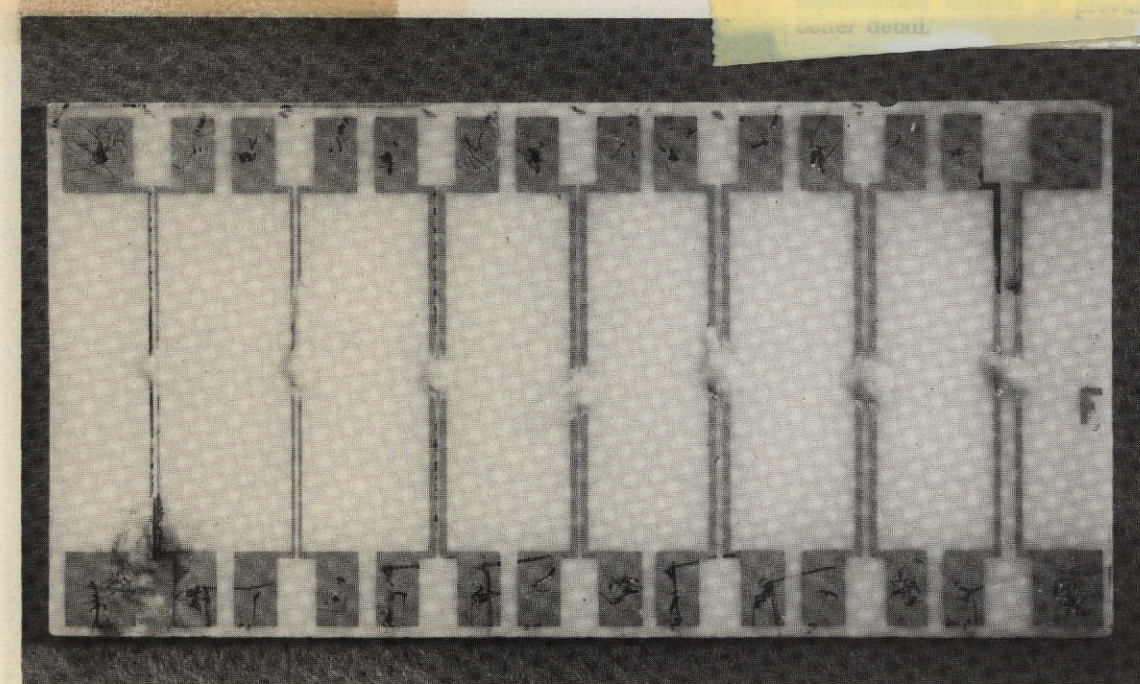

Figure 23. Epo-Tek H61-thin film gold exposed for 24 hr.

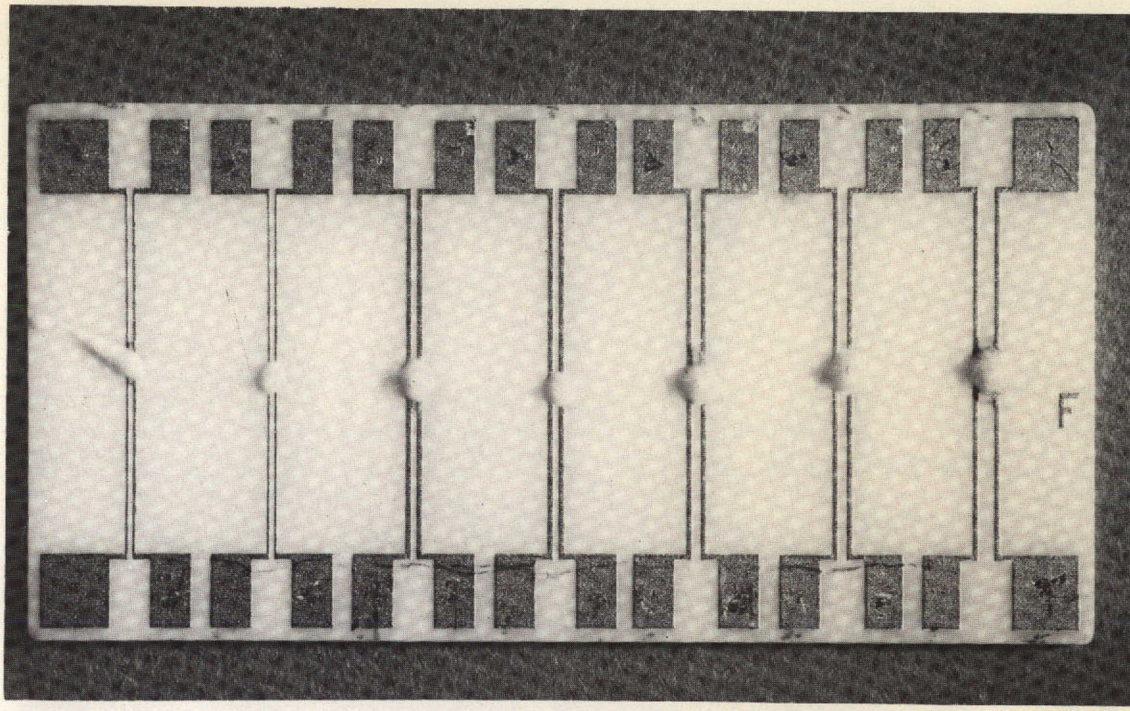

Figure 24. Epo-Tek H61-thick film gold exposed for 24 hr.

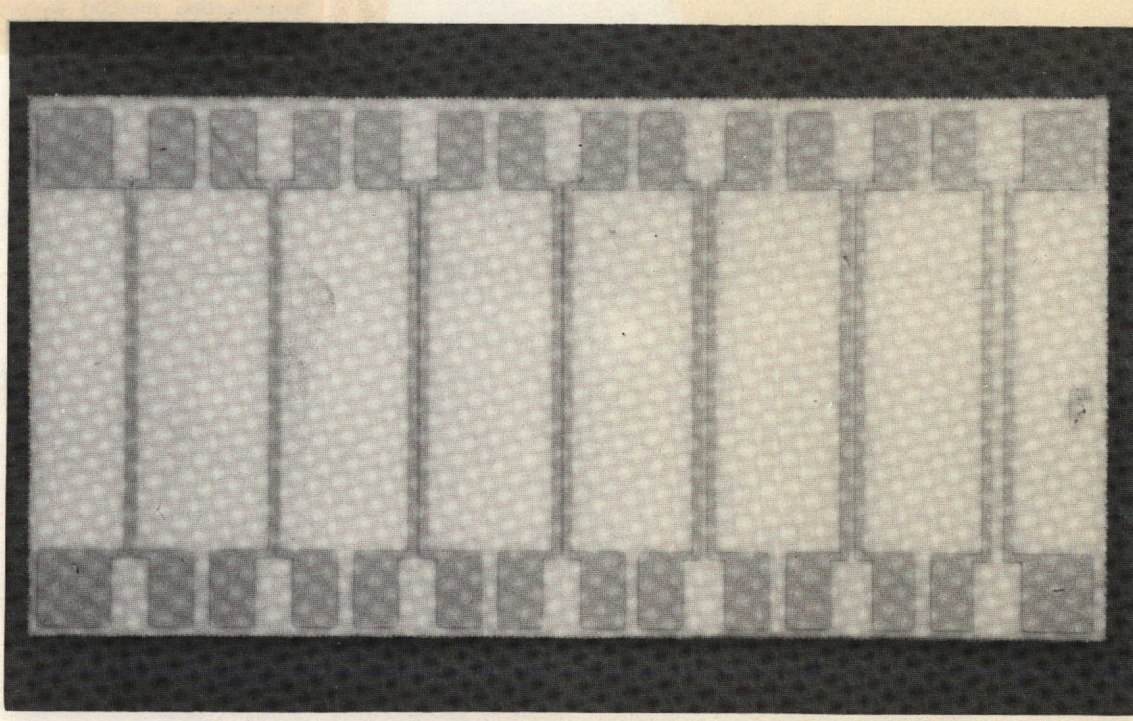

Figure 25. Unexposed thin film aluminum.

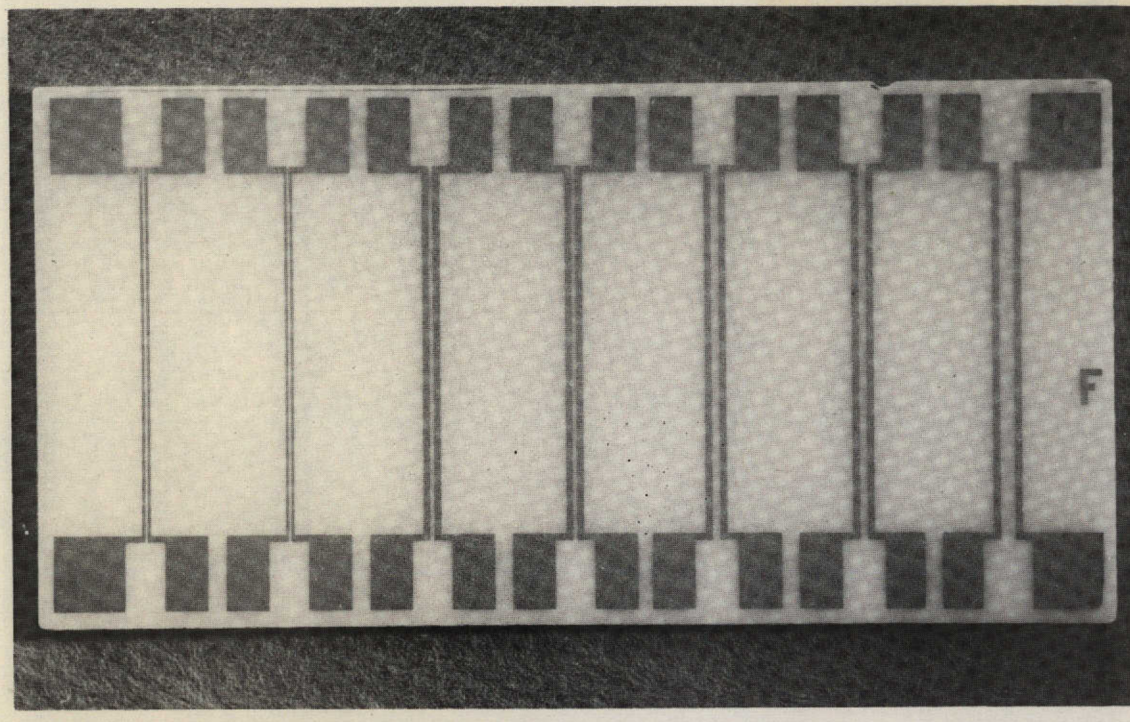

Figure 26. Unexposed thin film gold.

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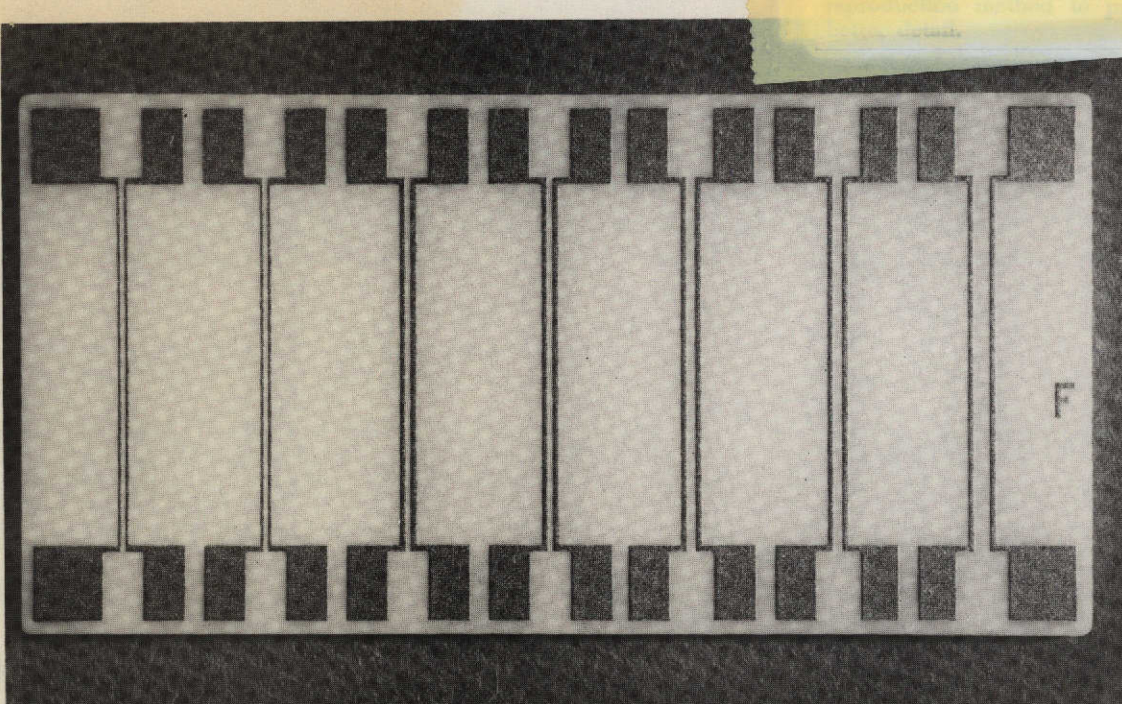

Figure 27. Unexposed thick film gold.

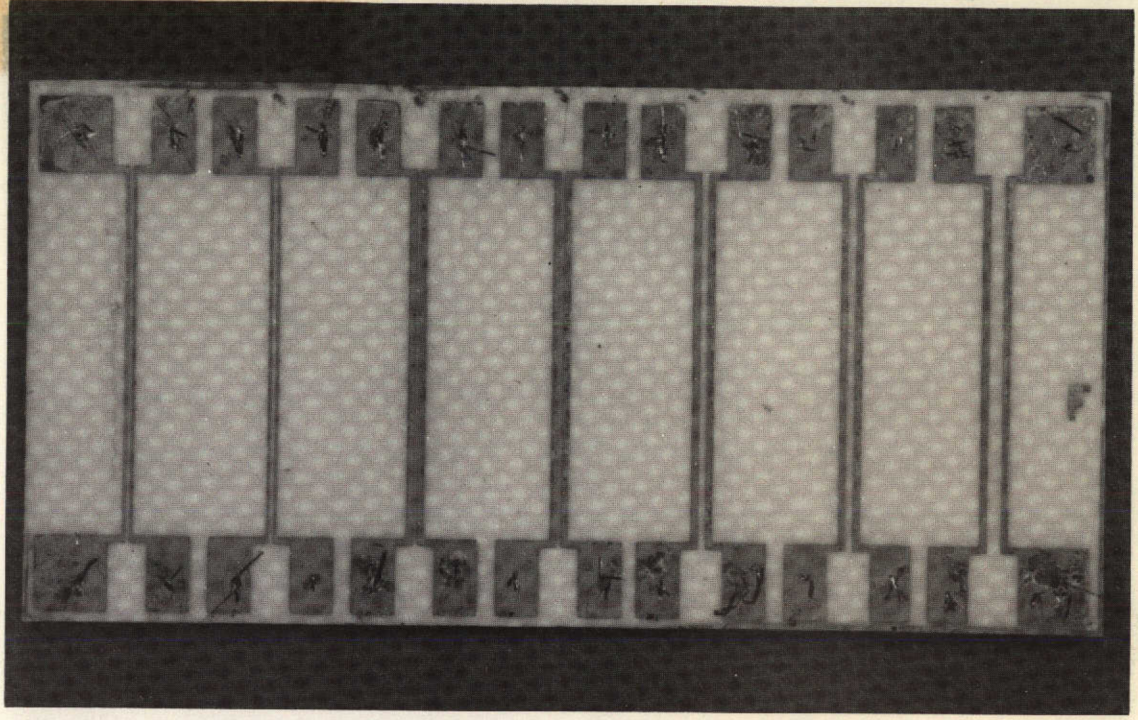

Figure 28. Control (no adhesive) — thin film aluminum exposed for 24 hr.

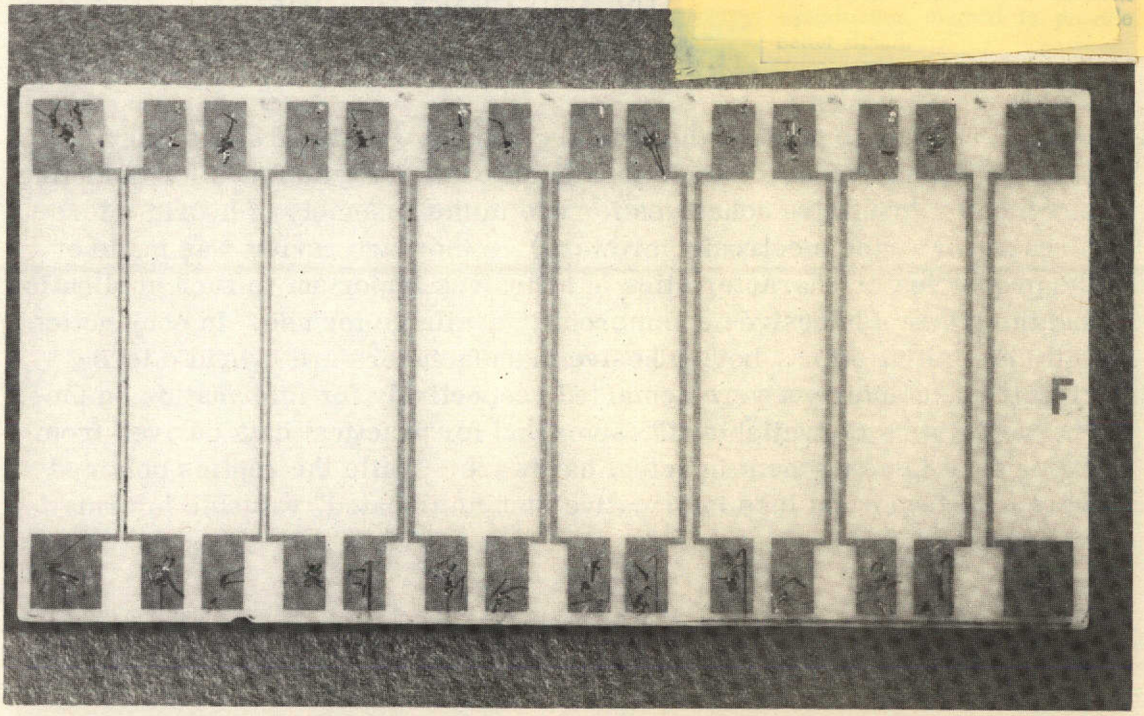

Figure 29. Control (no adhesive) — thin film gold exposed for 24 hr.

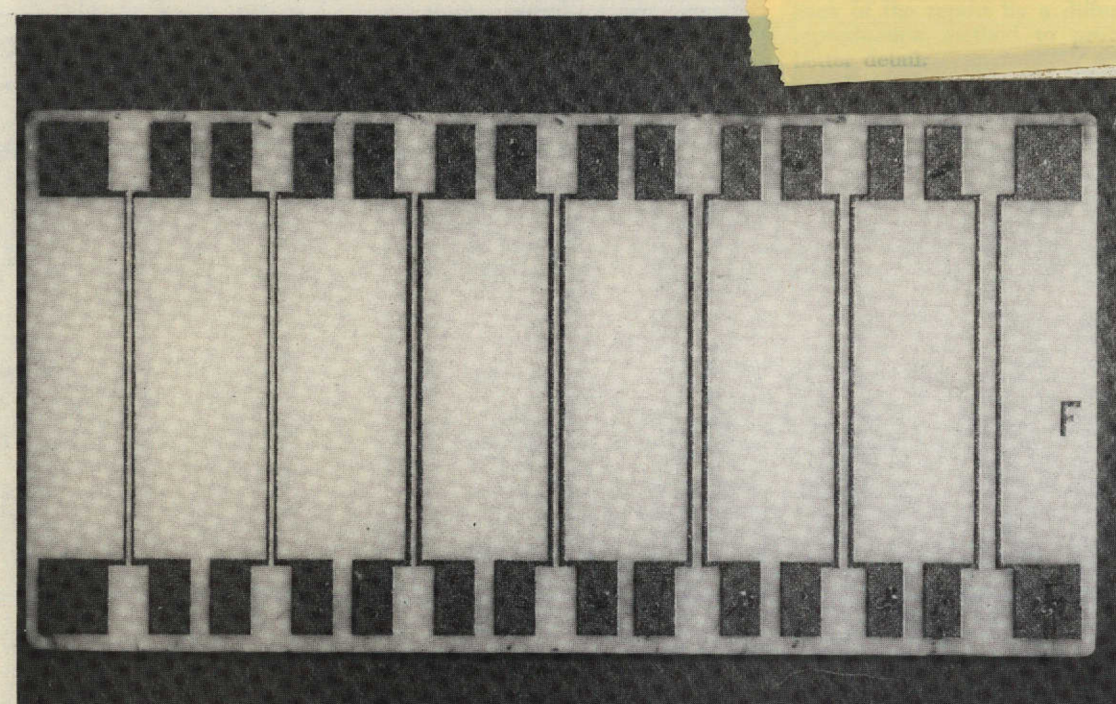

Figure 30. Control (no adhesive) — thick film gold exposed for 24 hr.

V. SUMMARY AND CONCLUSIONS

In order to achieve the primary objective of the present study (i.e. to define evaluation tests forming a basis for guidelines for selecting electrically insulative adhesives for use in the assembly of hybrid microcircuits for space electronic hardware), a thorough review was made of the properties or characteristics of adhesives important to such application and the types of adhesives commercially available for use. In conjunction with this initial effort, both adhesive manufacturers and hybrid microcircuit manufacturers were contacted respectively for information on the characteristics of available adhesives and for practical data derived from experience in using them in actual hardware. While the replies obtained were both fewer and less informative than anticipated, valuable information was obtained.

Since it was impossible within the scope of this study to investigate all of the important properties of adhesives that were identified, the following three major properties considered to be especially critical were selected for detailed attention:

1. Bond strength.

2. Outgassing after cure.

3. Corrosivity.

Then, tests were defined to quantify these characteristics, and selected adhesives were tested to generate an adequate data base to verify the validity and to establish the sensitivity of the selected tests. While a larger number of adhesives were evaluated for bond strength, again because of practical considerations, only the following five representative epoxy adhesives were evaluated for outgassing after cure, and corrosivity:

1. Hysol 0151 (cured with a primary, secondary amine).

2. Ablefilm 517A (cured with a tertiary amine).

3. Eccobond 104 (cured with an anhydride).

4. Epo-Tek H74 (cured with an imidazole).

5. Epo-Tek H61 (cured with a boron trifluoride complex).

Briefly, the tests defined were as follows:

1. <u>Bond Strength</u> — Test specimens consisted of 10 silicon dice bonded to both glazed and unglazed alumina substrates. Bond strength was determined by exerting a shear force on the die and measuring it with a dynamometer. Measurements were made of the room temperature bond strength for specimens freshly cured, after immersion in commonly used solvents, and after aging at room temperature and 150°C for intervals up to at least 90 days. Measurements also were made of the bond strength at 150°C for freshly cured specimens.

2. <u>Outgassing After Cure</u> — Cured samples of the adhesives were placed in breakseal tubes equipped with manometers and the pressure was monitored over an extended period of time while the tubes were aged at

150°C. At the end of this time, gas chromatographic and mass spectrometric analyses were run.

3. Corrosivity — Test specimens consisted of alumina substrates containing pairs of lines of different width and spacing. Thin film aluminum and both thin and thick film gold metallization systems were used. Small dots or strips of adhesive were applied across all line pairs, the adhesives were cured, 50 volts of current were applied across the lines of alternating line pairs, and the specimens were exposed to an 85°C/100 percent relative humidity environment for 24 hours. The test specimens then were visually examined at 30X, and the resistances of each line and between line pairs were measured both before and after exposure to this environment.

A cursory summary of the results is as follows:

1. Bond Strength — The bond strengths of all adhesives tested are unaffected by 30 min exposure to Freon TF, isopropyl alcohol, and trichloroethylene. The room temperature bond strengths of all adhesives tested except perhaps Ablebond 450 and Epo-Tek H55 are adequate for the present application even after prolonged aging (at least 90 days) at room temperature or maximum use temperature. Bond strength measurements at 150°C indicate that all adhesives tested except Epo-Tek H55, Ablefilm 535, and perhaps Ablebond 161-3 retain adequate bond strength for the present application.

2. Outgassing After Cure — Ablefilm 517A is by far the largest outgasser and Eccobond 104 is the smallest, outgassing a comparatively negligible amount. Also, both Epo-Tek adhesives (H61 and H74) outgas relatively little. After the first few days, the outgassed pressure remains essentially constant for Ablefilm 517A and Eccobond 104; but for Hysol 0151, the pressure in the tube continues to increase with extended aging. This suggests that Ablefilm 517A and Eccobond 104 are stable at the aging temperature (150°C) while Hysol 0151 may be slowly decomposing. Gas chromatographic and mass spectrometric analyses show that in general the major components outgassed by the adhesives are normal atmospheric gases and water; exceptions are Ablefilm 517A and Hysol 0151. The major portion of the large quantity of material outgassed from Ablefilm 517A is the solvent tetrahydrofuran. Analyses for Hysol 0151 showed large quantities of organics and a considerable quantity of water, supporting the suspicion that long term aging at 150°C causes decomposition.

3. Corrosivity — Of the five adhesives tested, only Eccobond 104 and Ablefilm 517A appear to be suitable for use on aluminum metallization

systems. These adhesives also appear to be best for use on thin and thick film gold metallization systems. In order of increasing corrosivity the adhesives probably should be listed as Eccobond 104 or Ablefilm 517A, Hysol 0151, Epo-Tek H61, and Epo-Tek H74. In all cases, the major evidence of discoloration or corrosion occurs on the positive lines of the line pairs. However, it should be noted that the 85° C/100 percent relative humidity environment used for the test may be too severe to give valid results for the aluminum specimens. In many cases, large amounts of aluminum oxide were formed.

APPENDIX
RESISTANCES BEFORE AND AFTER CORROSION TESTS

Tables A-1 through A-6 give the measured values of the individual line resistances and the interline or insulation resistances of the line pairs for the control set, Hysol 0151, Ablefilm 517A, Eccobond 104, Epo-Tek H74 and Epo-Tek H61, respectively. Entries from top to bottom correspond to lines from left to right on the test specimens, with the right side of the specimen being the side where the F is located. The second line of the odd pairs were at positive potential.

TABLE A-1. LINE AND INSULATION RESISTANCES OF CONTROL SET[a]

Thin Film Aluminum				Thin Film Gold				Thick Film Gold			
Before Test		After Test		Before Test		After Test		Before Test		After Test	
Line Resistance	Insulation Resistance	Line Resistance	Insulation Resistance	Line Resistance	Insulation Resistance	Line Resistance	Insulation Resistance	Line Resistance	Insulation Resistance	Line Resistance	Insulation Resistance
1.75	4×10^{13}	1.90	1.5×10^{11}	1.25	2×10^{14}	1.20	1×10^{10}	0.30	6×10^{14}	0.30	1×10^{15}
1.75		1.80		1.25		1.20		0.30		0.30	
1.70	5×10^{13}	1.95	6×10^{12}	1.25	2.5×10^{14}	1.20	6×10^{14}	0.29	6×10^{14}	0.29	6×10^{14}
1.70		1.80		1.25		1.20		0.29		0.29	
0.90	7×10^{13}	1.00	2×10^{11}	0.65	4×10^{14}	0.65	5×10^{14}	0.15	7×10^{14}	0.15	7×10^{14}
0.90		1.00		0.65		0.65		0.15		0.14	
0.90	6.5×10^{13}	1.00	1.2×10^{12}	0.65	4×10^{14}	0.65	8×10^{14}	0.15	1×10^{15}	0.15	5×10^{14}
0.90		1.00		0.65		0.65		0.15		0.14	
0.90	1.4×10^{13}	1.05	3×10^{12}	0.65	7×10^{14}	0.65	1.5×10^{15}	0.15	9×10^{14}	0.15	7×10^{14}
0.90		1.15		0.65		0.65		0.15		0.15	
0.90	1.1×10^{13}	1.40	2×10^{12}	0.65	7×10^{14}	0.65	1×10^{15}	0.15	1×10^{15}	0.15	1×10^{15}
0.90		1.15		0.65		0.65		0.15		0.15	
0.90	4×10^{13}	1.30	1.5×10^{12}	0.60	2×10^{15}	0.60	1.5×10^{15}	0.15	2×10^{15}	0.15	1×10^{15}
0.90		1.20		0.60		0.60		0.15		0.15	

a. All resistances measured in ohms.

TABLE A-2. LINE AND INSULATION RESISTANCES WITH HYSOL 0151[a]

Thin Film Aluminum				Thin Film Gold				Thick Film Gold			
Before Test		After Test		Before Test		After Test		Before Test		After Test	
Line Resistance	Insulation Resistance	Line Resistance	Insulation Resistance	Line Resistance	Insulation Resistance	Line Resistance	Insulation Resistance	Line Resistance	Insulation Resistance	Line Resistance	Insulation Resistance
1.90	1.2×10^{14}	>50	1.5×10^{10}	1.20	2×10^{14}	1.20	Arced	0.33	8×10^{14}	0.33	Arced
1.90		>50		1.20		1.20		0.33		0.33	
1.80	1.0×10^{14}	>50	8×10^{9}	1.15	1.4×10^{14}	1.15	2×10^{13}	0.32	8×10^{14}	0.31	1.5×10^{12}
1.80		2.30		1.15		1.15		0.31		0.30	
0.95	1.2×10^{14}	>50	1.5×10^{11}	0.65	7.5×10^{11}	0.65	Arced	0.15	9×10^{14}	0.15	6×10^{14}
0.95		>50		0.65		0.65		0.15		0.15	
0.95	1.0×10^{14}	1.05	4×10^{10}	0.65	4.5×10^{14}	0.65	7×10^{13}	0.15	7×10^{14}	0.15	3×10^{14}
0.95		1.05		0.65		0.65		0.15		0.15	
0.95	2×10^{14}	1.10	4×10^{9}	0.65	4.5×10^{13}	0.65	4×10^{14}	0.15	4×10^{14}	0.15	2×10^{9}
0.95		>50		0.65		0.65		0.15		0.15	
0.95	2×10^{14}	1.00	4×10^{13}	0.65	3×10^{13}	0.65	3×10^{14}	0.15	2×10^{14}	0.15	6×10^{14}
0.95		1.00		0.65		0.65		0.15		0.15	
0.95	3×10^{14}	1.20	2×10^{9}	0.65	1.5×10^{13}	0.65	1.5×10^{14}	0.15	3×10^{14}	0.15	6×10^{14}
0.95		1.05		0.65		0.65		0.15		0.15	

a. All resistances measured in ohms.

TABLE A-3. LINE AND INSULATION RESISTANCES WITH ABLEFILM 517A[a]

Thin Film Aluminum				Thin Film Gold				Thick Film Gold			
Before Test		After Test		Before Test		After Test		Before Test		After Test	
Line Resistance	Insulation Resistance	Line Resistance	Insulation Resistance	Line Resistance	Insulation Resistance	Line Resistance	Insulation Resistance	Line Resistance	Insulation Resistance	Line Resistance	Insulation Resistance
1.80	2.5×10^{14}	2.05	3×10^{10}	1.15	6×10^{14}	1.30	Arced	0.30	1.5×10^{15}	0.30	Arced
1.80		>50		1.15		1.25		0.30		0.29	
1.75	2×10^{14}	1.90	7×10^{10}	1.15	1×10^{15}	1.20	5×10^{13}	0.29	1×10^{15}	0.29	6×10^{13}
1.75		1.90		1.15		1.20		0.28		0.28	
0.95	2.5×10^{14}	1.00	2×10^{10}	0.60	1×10^{15}	0.65	Arced	0.14	6×10^{14}	0.15	Arced
0.95		1.00		0.60		0.65		0.14		0.14	
0.95	3×10^{14}	1.00	1×10^{11}	0.60	1.5×10^{15}	0.65	Arced	0.14	6×10^{14}	0.15	4×10^{10}
0.95		1.00		0.60		0.65		0.14		0.14	
0.95	4×10^{14}	1.10	1×10^{12}	0.60	2×10^{15}	0.65	4.5×10^{14}	0.14	7×10^{14}	0.15	5×10^{13}
0.95		1.10		0.60		0.65		0.14		0.14	
0.95	7×10^{14}	1.05	1×10^{12}	0.60	1×10^{15}	0.65	2×10^{14}	0.14	8×10^{14}	0.15	5×10^{13}
0.95		1.05		0.60		0.65		0.14		0.14	
0.95	3×10^{14}	1.20	2×10^{14}	0.60	1.5×10^{15}	0.65	6×10^{14}	0.14	2×10^{15}	0.15	1.5×10^{14}
1.00		1.35		0.60		0.65		0.14		0.15	

a. All resistances measured in ohms.

TABLE A-4. LINE AND INSULATION RESISTANCES WITH ECCOBOND 104[a]

Thin Film Aluminum				Thin Film Gold				Thick Film Gold			
Before Test		After Test		Before Test		After Test		Before Test		After Test	
Line Resistance	Insulation Resistance	Line Resistance	Insulation Resistance	Line Resistance	Insulation Resistance	Line Resistance	Insulation Resistance	Line Resistance	Insulation Resistance	Line Resistance	Insulation Resistance
2.10 2.05	9×10^{13}	2.15 2.55	3.5×10^{13}	1.10 1.10	1×10^{15}	1.10 1.10	Arced	0.31 0.31	8×10^{14}	0.31 0.31	8×10^{14}
1.95 1.95	8×10^{14}	2.05 2.00	Arced	1.05 1.05	8×10^{14}	1.10 1.10	Arced	0.33 0.30	1.5×10^{15}	0.33 0.30	2×10^{15}
0.95 0.95	1.5×10^{15}	1.10 1.15	Arced	0.60 0.60	1×10^{15}	0.60 0.60	Arced	0.14 0.14	6×10^{14}	0.14 0.14	8×10^{14}
0.95 0.95	1.5×10^{15}	1.00 1.00	Arced	0.60 0.60	1×10^{15}	0.60 0.60	2×10^{14}	0.14 0.14	1×10^{15}	0.14 0.14	7×10^{14}
0.95 0.95	1×10^{15}	1.05 1.35	6.5×10^{12}	0.60 0.60	2×10^{15}	0.60 0.60	1.4×10^{14}	0.14 0.14	1×10^{15}	0.14 0.14	7×10^{14}
0.95 0.95	1×10^{15}	1.00 1.00	9×10^{9}	0.60 0.60	1×10^{15}	0.60 0.60	9×10^{13}	0.14 0.14	1.5×10^{15}	0.14 0.14	7×10^{14}
0.95 0.95	1×10^{15}	>50 >50	3×10^{11}	0.60 0.60	1.5×10^{15}	0.65 0.65	1.2×10^{14}	0.14 0.14	1×10^{15}	0.14 0.14	1×10^{15}

a. All resistances measured in ohms.

TABLE A-5. LINE AND INSULATION RESISTANCES WITH EPO-TEK H74[a]

Thin Film Aluminum				Thin Film Gold				Thick Film Gold			
Before Test		After Test		Before Test		After Test		Before Test		After Test	
Line Resistance	Insulation Resistance	Line Resistance	Insulation Resistance	Line Resistance	Insulation Resistance	Line Resistance	Insulation Resistance	Line Resistance	Insulation Resistance	Line Resistance	Insulation Resistance
1.75	5×10^{14}	>50	2×10^{10}	1.15	9×10^{14}	1.20	Arced	0.34	1×10^{15}	0.33	Arced
1.75		>50		1.15		1.15		0.34		0.33	
1.70	4×10^{14}	2.05	2×10^{11}	1.15	5×10^{14}	1.15	3×10^{12}	0.32	1×10^{15}	0.31	6×10^{11}
1.70		2.05		1.15		1.15		0.31		0.30	
0.90	6×10^{14}	1.30	5×10^{12}	0.65	5×10^{14}	0.65	4×10^{14}	0.15	1×10^{15}	0.15	Arced
0.90		>50		0.65		0.65		0.15		0.15	
0.90	6×10^{14}	1.20	Arced	0.65	4.5×10^{14}	0.60	2×10^{12}	0.15	1×10^{15}	0.15	1×10^{9}
0.90		1.25		0.65		0.60		0.15		0.15	
0.90	8×10^{14}	1.70	7×10^{9}	0.65	1×10^{15}	0.60	Arced	0.15	1.2×10^{15}	0.15	7×10^{9}
0.90		>50		0.65		0.60		0.15		0.15	
0.90	7×10^{14}	1.25	2×10^{10}	0.65	6×10^{14}	0.60	4×10^{14}	0.15	1.5×10^{15}	0.15	3×10^{12}
0.90		1.10		0.65		0.60		0.15		0.15	
0.90	1×10^{15}	1.55	1.5×10^{12}	0.65	1×10^{15}	0.60	9×10^{12}	0.15	2×10^{15}	0.15	5×10^{11}
0.90		>50		0.65		0.60		0.15		0.15	

a. All resistances measured in ohms.

TABLE A-6. LINE AND INSULATION RESISTANCES WITH EPO-TEK H61[a]

Thin Film Aluminum				Thin Film Gold				Thick Film Gold			
Before Test		After Test		Before Test		After Test		Before Test		After Test	
Line Resistance	Insulation Resistance	Line Resistance	Insulation Resistance	Line Resistance	Insulation Resistance	Line Resistance	Insulation Resistance	Line Resistance	Insulation Resistance	Line Resistance	Insulation Resistance
2.1	2.2×10^{11}	>50	4×10^{11}	1.35	2×10^{14}	1.40	1×10^{11}	0.34	1×10^{15}	0.32	1.5×10^{12}
2.1		>50		1.35		1.30		0.33		0.32	
2.1	2.2×10^{13}	2.15	2×10^{13}	1.05	4×10^{14}	1.05	8×10^{14}	0.32	9×10^{14}	0.31	1×10^{15}
2.1		2.25		1.05		1.05		0.31		0.30	
1.05	1.5×10^{13}	>50	3×10^{11}	0.55	6×10^{14}	0.55	8×10^{11}	0.15	1.2×10^{15}	0.14	2×10^{14}
1.05		>50		0.55		0.55		0.15		0.14	
1.05	8×10^{12}	1.15	1.5×10^{14}	0.55	1.0×10^{15}	0.55	6×10^{13}	0.15	2×10^{15}	0.14	2×10^{14}
1.05		1.15		0.55		0.55		0.15		0.14	
1.1	2×10^{14}	>50	4×10^{11}	0.55	1.2×10^{15}	0.55	2×10^{9}	0.16	1.5×10^{15}	0.15	4.5×10^{14}
1.1		>50		0.55		0.55		0.15		0.15	
1.1	2.4×10^{14}	1.20	8×10^{14}	0.55	1.6×10^{15}	0.55	1.2×10^{14}	0.15	8×10^{14}	0.15	8×10^{14}
1.1		1.20		0.55		0.55		0.15		0.15	
1.15	5.0×10^{14}	>50	3×10^{10}	0.60	1.6×10^{15}	0.60	8×10^{12}	0.15	2×10^{15}	0.15	2×10^{15}
1.15		>50		0.60		0.60		0.16		0.15	

a. All resistances measured in ohms.

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APPROVAL

DESIGN GUIDELINES FOR USE OF ADHESIVES IN HYBRID MICROCIRCUITS

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The information in this report has been reviewed for security classification. Review of any information concerning Department of Defense or Atomic Energy Commission programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

This document has also been reviewed and approved for technical accuracy.

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