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MASTER Effects of Humidity During
Photoprocessing on Thin Film
Metallization Adhesion

By D. P. Norwood

Published March 1980

Topical Report

Prepared for the United States Department of Energy
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EFFECTS OF HUMIDITY DURING PHOTOPROCESSING
ON THIN FILM METALLIZATION ADHESION

By D. P. Norwood

Published March 1980

Topical Report
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Prepared by D. P. Norwood

Humidity effects during photoprocessing on tantalum/chromium/gold thin film networks (TFNs) were investigated. Humidity conditions at various process steps were controlled by placing either desiccant or water in handling containers for the TFNs. The TFNs photoprocessed in humid conditions had a much higher occurrence of metallization failures compared to TFNs processed in dry conditions. Ceramic surface defects were shown to cause pores in the thin films, and these pores enhanced corrosion susceptibility for the films. This study resulted in a desiccated storage process for production of TFNs.

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SUMMARY

This work was conducted to determine the effects of humidity on metallization adhesion of thin film networks (TFNs) during photoprocessing. Thin film metallized substrates were scribed and broken into quarters to provide uniform samples for comparison of humid and dry processing conditions. Between processing steps, the thin film metallized substrates were placed in plastic boxes containing either desiccant or water to simulate extremely dry (<6 percent RH) or humid (>85 percent RH) conditions. In addition, some of the substrates were placed in a humidity chamber at 50 percent RH. Each in-process storage time was approximately 16 hours except for a 1-hour storage period after etching and before stripping to simulate the maximum time allowed by photoprocessing specifications. After dry and wet photoprocessing, the metallization adhesion of the TFNs was tested by lead frame bonding and pull testing.

Initial tests on substrates metallized in the Microelectronics Laboratory and photoprocessed in high humidity containers resulted in gold lifting or peeling during pre-bond ceric ammonium nitrate (CAN) etching and cleaning. On substrates processed in the desiccated containers, metallization failures occurred on about 80 percent of the TFNs, but pull strengths averaged 1.5 kg. This indicated high humidity during photoprocessing is a contributor to corrosive effects on metallization. Pre-bond CAN etching was shown to contribute to metallization adhesion failures. Dry photoprocessed parts, which were pre-bond CAN etched, had good lead frame bond pull strengths; however, metallization de-adhesion was found in the lifted bond zones. Non-CAN etched parts, which had been dry photoprocessed, had good pull strengths and 80 percent heel failures in lead frame pull tests.

High humidity photoprocessing was shown to promote corrosion in thin film metallization processed with either liquid resist (used for non-via patterning) or sheet resist (used for via patterning). Liquid resist processing, however, yielded TFNs with good bond pull strengths and metallization failures, whereas sheet resist processing resulted in loss of adhesion prior to bonding. No explanation was found for the differences in corrosion protection provided by liquid and sheet resist. Gold etchant was shown to penetrate developed sheet resist in a time exceeding the 1-hour limit imposed on resist stripping after etching. Although a critical humidity level for the triggering of corrosive effects was not determined, photoprocessing in a 50-percent humidity environment (provided by a humidity chamber for in-process storage) was shown to cause corrosive effects. Wet processing enhanced corrosion effects on all thin films that were susceptible to corrosion. The corrosion susceptibility of thin films was found

to depend on pre-photolithography processing as well as the porosity in the ceramic substrate. Substrates metallized in the production vacuum systems at the time of these tests were not susceptible to corrosion induced by humid photoprocessing.

Auger analysis of the bond zone and peeled gold from the wet-processed TFNs which exhibited metallization failure indicated that corrosion was occurring at the chromium-gold interface. Chromium remained on the TFN surface, but none was found on the back side of the peeled gold. In addition, iodine was found on the peeled gold indicating that gold etch ($KI + I_2$) was responsible in part for the corrosion. Once it was established that different thin films had varying degrees of corrosion susceptibility, scanning electron microscope (SEM) analysis was performed on both types to determine what differences could be detected in film morphology. This analysis showed that voids were present in the gold surface of films which were sensitive to corrosion during photoprocessing; resistant films did not have voids. When the metallization was removed to examine the ceramic surface below, voids between the ceramic grains were found; however, it was not possible to relate each gold void to a ceramic void.

This work resulted in the definition of in-process desiccated storage to reduce the occurrence of the metallization failures which tend to occur in TFN manufacturing during humid months. The dry versus wet storage of TFNs during photoprocessing also has provided a good test for corrosion susceptibility.

DISCUSSION

SCOPE AND PURPOSE

The purpose of this project was to determine the effects of humidity variation on thin film network (TFN) metallization during photoprocessing and to develop an in-process method of humidity control during TFN manufacturing.

PRIOR WORK

Previous work on chromium-gold thin film corrosion has shown that corrosion can occur when these films are subjected to chemical vapors used in photoprocessing.¹ Chemical vapors such as gold etchant ($KI + I_2$) and ceric ammonium nitrate (CAN) etchant were found to attack chromium-gold films to the point that peeling of the films occurred indicating loss of adhesion. Other production work showed that a decrease in humidity during photoprocessing resulted in less corrosion of thin films and resulted in a higher yield as determined by lead frame bonding and pull testing. Humidity reduction to approximately 25 percent RH was maintained during etching and stripping operations, and this resulted in yield improvement over performing these operations in a production environment of 50 to 60 percent RH.

ACTIVITY

Thin Film Network Processing

This work was directed primarily toward determining effects of photoprocessing at different humidity conditions on thin film adhesion. A brief summary of TFN processing leading up to photolithography is given here for convenience.

Thin film networks are manufactured on large (95 by 114 mm) alumina substrates of 99.5 percent alumina oxide. A layer of tantalum nitride is sputtered on the substrate followed by vacuum-evaporated layers of chromium and gold. The tantalum nitride is used for resistor fabrication, and the chromium-gold is used for conductors. Chromium acts as an adhesive layer between the tantalum nitride and the gold.

Multi-image photoprocessing techniques are used to process substrates to the thin film network level. Two levels of photoprocessing are required to define the TFN pattern--the first for

the conductor pattern and the second for the resistor pattern. TFNs which contain vias are photoprocessed with sheet resist which furnishes protection of the metallization in the plated-through holes. Second level resist patterning is done with liquid resist applied by roller coating. TFNs not containing vias use liquid resist for both levels of photoprocessing. Exposure is made on a machine capable of a high degree of light collimation.

After photoprocessing, resistors are thermally stabilized in air at 300°C for 2 hours. Laser scribing and hand breaking are used to separate the large substrate into smaller TFNs. Resistors are then laser trimmed to nominal value using an automated yttrium aluminum garnet (YAG) system. After a visual inspection, TFNs are assembled with the devices required to convert them to functional circuits. The block diagram in Figure 1 shows simplified processing flow of product from TFN to HMC levels. This block diagram shows processing required for manufacturing TFNs containing vias.

General Description of Humidity Tests

The initial evaluation was performed using storage containers in place of an area of low or high humidity. The containers chosen were plastic boxes having a watertight seal. To simulate conditions of low and high humidity, desiccant was used in one container to provide a dry condition, and the bottom was covered with water in the other to provide high humidity. The level of water was carefully maintained such that it did not touch the substrates. The desiccant, a dust-free silica gel which was contained in individual packages, was packed so as to not touch the substrate surface. Humidity levels inside the dry and wet containers were <6 and >85 percent RH, respectively, as determined by hygrometers.

The procedures used for conducting humidity controlled photoprocessing are shown in Figure 2. Generally, they consisted of substrate storage in dry and humid containers after the resist patterning and etching steps. Overnight storage was done at each major level (conductor patterning and resist patterning) of photoprocessing. This ensured each substrate had sufficient time to dry out or absorb moisture from the container environment. An additional storage was done after etching and prior to stripping to simulate the 1-hour maximum limit (between etch to strip) imposed by photoprocessing specifications; it is required that resists be stripped within 1 hour after etching. Other than the additional time in desiccated or humid storage, photoprocessing requirements according to specifications were observed. The humidity controlled photoprocessing spanned a 4-day period and

Figure 1. Diagram of Processes for TFN Manufacture

Figure 2. Diagram of Humidity Controlled Photolithography

required approximately 75 hours of clock time. This time period was from initial application of sheet resist to stabilization. (Refer to Figure 2.)

For the adhesion evaluations, a pattern which produced a 26- by 26-mm TFN having 19 lead frame bond sites on each side was used. Initially, gold-plated, 0.18-mm lead frames were used for bonding and pull testing. These type lead frames were replaced with bare copper lead frames for subsequent tests. There were several reasons for this decision:

- Copper lead frames could be bonded at different process levels, whereas bonding of gold-plated lead frames required extensive surface cleaning and pre-bond etching of the TFN;

- Copper lead frames were less expensive; and
- Copper lead frames were easily cleaned for bonding by immersion in a dilute acid and rinsing with deionized (DI) water.

Copper lead frames could be bonded to gold surfaces which had not been prepared by CAN etching (chromium etchant) to remove residual amounts of chromium. This allowed analysis of bondability, corrosion, or both effects prior to removing the chromium and proved to be advantageous since CAN-etch, prebond cleaning was found to be part of the corrosive effects of humidity photoprocessing.

Lead frame pull testing acceptance criteria include both strength and failure modes. After leads are bonded, they are bent 90° away from the substrate and individually pull tested to break the lead-to-substrate bond. The force required to pull the lead from the substrate is recorded and each bond zone on the TFN is then optically examined for the mode of failure. Acceptance requirements are: the average pull strength must exceed 0.7 kg, the minimum pull strength for any one lead must be 0.34 kg, and less than 10 percent metallization failures may be present in the exposed bond area (regardless of strengths). Since failure modes are an important aspect of the bond criteria, a general description of the six types of failure mode is given in Table 1.

In this evaluation, the primary concern was for failure modes of M and B categories. The M failure mode indicates the possible occurrence of corrosive effects resulting in delamination in the base metallization; B failure modes indicate good adhesion and that the lead has been destroyed at the weakest link (at the heel of the bond zone). Other failure modes would be associated with lead frames, cleaning, and substrate properties. It is noteworthy that an M failure mode could occur without peeling the lead from the bonded zone; if greater than 10 percent of the exposed bond imprint showed signs of M failure, the failure mode of that lead would be classified as M.

Initial Humidity Tests on Laboratory Deposited Films

The purpose of the initial evaluation was to determine if photoprocessing in high and low humidity would create corrosive effects leading to metallization failures on TFN circuits. Testing was conducted on substrates which had been metallized in the Microelectronics Laboratory. The samples were metallized at a time when metallization failures were occurring with parts produced in the laboratory metallization system. Type M failures occurred on metallization lots produced before and after the humidity process evaluation was conducted. The standard Ta₂N/Cr/Au metallization was deposited on the substrates. Gold thickness was nominal

Table 1. Failure Modes for 90° Pull Test of Lead Frames

Failure Mode	Definition
Type A (Lead Tensile)	Lead breaks at a point away from the bond zone.
Type B (Heel Failure)	Lead breaks at heel of bond with at least 25 percent of bonded lead remaining.
Type C (Bond Failure)	Failure in substrate gold-to-lead frame gold interface.
Type M (Metallization Failure)	Lead peels away exposing tantalum or chromium base metallization in greater than 10 percent of the exposed bond zone.
Type E (Substrate-Ta ₂ N)	Tantalum peels leaving bare ceramic in the bond zone.
Type F (Lead Plating Failure)	Plating removed from the lead during pull test.

6 μ m. The mask pattern used in photoprocessing provided a 26- by 26-mm circuit with 19 bond sites on each edge for lead frame bonding.

One large size (95 by 114 mm) substrate was scribed and broken into four smaller substrates for processing after metallizing. This was to ensure that no differences in substrate or metallization characteristics were introduced into the evaluation; if different substrates were used, other factors could be introduced such as cleaning, ceramic, or metallization differences even though they were produced in the same processing lot. By using one substrate and dividing it for different tests, it was more likely that no other substrate or metallization characteristics entered into the results.

The substrates were photoprocessed using the humidity controlled photolithography process outlined in Figure 2. Sheet resist was used for conductor patterning and liquid resist was used for resistor patterning. Processing time covered a 4-day period with a cumulative container time of 74 hours representing dry or humid storage between processing steps. Immediately after photoprocessing, substrates were stabilized for 2 hours at 300°C.

Evaluation of the processing steps then consisted of laser scribe and separation of TFNs followed by a pre-bond cleaning of CAN etching and thermocompression (TC) cleaning according to normal manufacturing procedures. After cleaning, lead frames were attached by thermocompression bonding on a lead frame bonder. Lead frames used for bonding were standard 0.18-mm, gold-plated copper lead frames.

Lead frame pull test results between the dry and humid photoprocessing were quite different. Substrates processed through photolithography in humid containers did not survive the CAN etching and TC cleaning process: the gold metallization was found to peel from the base metallization of chromium or tantalum. Substrates processed in the dry environment exhibited pull strengths averaging 1.5 kg. These results are shown in Figure 3. Metallization failures occurred in the exposed bond zone for approximately 80 percent of the dry processed parts, but pull strengths met or exceeded normal lead frame test results for production testing. The minimum pull strength was 1.0 kg and the maximum pull strength was 1.7 kg.

Photographs of the failure mode differences are shown in Figure 4. It can be seen (Figure 4A) that in dry photoprocessed TFNs some base metallization is exposed where the lead frame was lifted; however, sufficient metallization adhesion exists such that the heel fractured rather than the bond zone. Figure 4B shows the lifting or adhesion failure prior to bonding in humid photoprocessed TFNs. This indicated high humidity during photoprocessing is a major contributor to corrosive effects on metallization.

Since gold metallization had peeled during CAN etching and TC cleaning, another experiment was conducted to determine what part these pre-bond cleaning processes contributed to corrosive effects. Lead frames were bonded to wet and dry processed substrates from the evaluation without performing the pre-bond cleaning. Since gold-to-gold thermocompression bonding results in bond delamination on uncleaned substrates, copper lead frames were used for this test.

The humid (wet) photoprocessed substrates survived the bonding operation; however, when bent 90° for pull testing, the gold pads lifted exposing base metallization. This indicated the corrosive effects were established during high humidity photoprocessing. Dry processed substrates showed high pull strengths, but this time they did not show evidence of metallization (type M) failures. The majority of lead failures were heel failures (type B) with approximately 30 percent type C failures; type C failures are expected where contamination or chromium remains on the bond surface. The results (Figure 5) indicated that pre-bond CAN etching was entering into the corrosive mechanism since no metallization failures occurred on these dry samples during pull testing. (They had occurred on samples that were CAN etched and cleaned.)

Figure 3. Bonding Test Results on TFNs Photoprocessed at High and Low Humidity Conditions, Laboratory Metallized Substrates

A. DRY PROCESSING

B. WET PROCESSING

Figure 4. Failure Mode Differences Resulting From Wet and Dry Photo-processing Conditions on Laboratory Metallized Substrates

Figure 5. Bonding Test Results on TFNs Photoprocessed in Humidity Extremes With No Pre-Bond CAN Etching, Laboratory Metallized Substrates

To test the hypothesis that CAN etching contributes to corrosion, another experiment was conducted on the dry processed substrates only. Two dry photoprocessed TFNs were bonded with copper lead frames, except one was CAN etched prior to bonding; the other was not. These results are shown in Figure 6. Both TFNs exhibited good lead frame pull strength; however, parts that were CAN etched showed 100 percent metallization failures (in the bond area exposed after pull testing), and the lead broke at the heel leaving the bond in place in all instances. Substrates which were not CAN etched showed approximately 80 percent heel failures with no indication of metallization failures (approximately 25 percent were type C failures). Therefore, it was shown possible to change from type B to type M failures on the same substrate by introducing pre-bond CAN etching.

These initial results established that high humidity contributes to corrosive effects during photoprocessing. Also, CAN etching after photoprocessing contributed to the corrosive mechanism. The differences in these results were independent of substrate or metallization properties since evaluation was conducted on the same physical substrate.

Humidity Effects on Different Photoresists

Production history had indicated that the majority of metallization failures (as determined by lead frame bonding tests) were occurring on substrates containing vias and back side metallization. The photoprocessing differences between these circuits and those with single-sided metallization was in the type of resists used in conductor patterning. TFNs with single-sided metallization used liquid resist, whereas TFNs having vias and two-side metallization used sheet resist.

Another evaluation was planned using humidity photoprocessing to determine if either resist contributed to or prevented corrosive effects during photoprocessing. Also, it was desired to repeat the initial experiment to verify humidity effects on substrates metallized in different systems. As part of the evaluation, lead frame bonding tests were planned on the back side metallization to determine if corrosive effects were present there. Photoprocessing of back side metallization was different than front side photoprocessing in that there is no resist patterning on the back side. The entire surface is protected by resist during etching operations.

Metallized substrates used in this evaluation were deposited in different systems from those in the initial evaluation. Sputtering was done in a continuous process (CP) sputtering machine which was being characterized for production. Chromium/gold was deposited in a planetary E-beam system which also was being characterized

Figure 6. Pre-Bond CAN Etching Effect on Laboratory Metallized Substrates Which Were Dry Photoprocessed

for production use. Only 99.5 percent alumina substrates were used in this evaluation. Nominal production thicknesses of Ta₂N and Cr/Au were deposited; even though no vias were present in the substrate, both front side and back side Cr/Au were deposited (nominal 6- μ m gold thickness) to simulate double-sided TFN processing.

Prior to photoprocessing, a large substrate was divided into quarters for various photoprocessing analyses. As in the first evaluation, this minimized the effects of substrate or metallization variables. Photoprocessing was identical to those operations described in Figure 2, except two additional categories of substrates were processed with liquid resists used in first-level (conductor) patterning; the other two samples were photoprocessed with sheet resists as before. Normal processing of single-sided metallized TFNs does not require liquid resist application to the back side; however, in this instance both sides of the substrates were coated for comparison to sheet resist processing.

After humidity photoprocessing, all substrates were stabilized for 2 hours at 300°C. Substrates were then laser scribed and separated followed by pre-bond CAN etching and cleaning. Copper lead frames were bonded to the TFNs and then pull tested. Substrates, which had been processed with the sheet resist, repeated the metallization failure modes seen in the initial evaluation. Those substrates processed in a dry environment exhibited good pull strength, whereas those processed in a wet environment had low pull strengths with metallization failures. Several bond pads did not survive the 90° bend (9 of 23), and those that did survive were low strength. Dry processed parts showed average pull strengths of 1.6 kg, and those processed in a humid environment averaged 0.8 kg if "zero" pull strengths were disregarded. Wet processed parts had 100 percent type M failures. Dry processed parts had mixed failure modes: 26 percent type E, 21 percent type C, 48 percent type B, and 3 percent type M. These results are shown in Figure 7.

First-level photoprocessing with liquid resist produced fewer corrosive effects. Substrates processed in the dry environment with liquid resist had good pull strengths averaging 1.7 kg and 97 percent type B failures. Substrates processed in a humid environment with liquid resist had good pull strengths averaging 1.4 kg with 100 percent type M failure modes. These results (Figure 7) showed that processing with the liquid resist resulted in lesser corrosive effects than pre-cleaning with the sheet resists; however, the differences in failure mode between dry and humid processing were still evident.

Reference data was available from an unrelated evaluation on substrates metallized at the same time. This data is shown in Figure 7 also. These substrates were processed routinely through

Figure 7. Humidity Effects on Liquid and Sheet Resist Photo-processing

photoprocessing, stabilization, and pre-bond cleaning without any special humidity conditions. The reference sample pull strengths were slightly less than those processed dry with an average pull strength of 1.4 kg. No metallization failures occurred; however, 37 percent type C failures were present. These results correlated closely to those processed in the dry environment using sheet resists.

Since corrosive effects caused by photoprocessing in humid environments repeated in this evaluation, back side metallization was evaluated for similar effects. For back side metallization no Ta₂N is deposited under the Cr/Au metallization. Therefore, metallization failures (type M) which were characteristic of front side corrosion should show up as type E failure modes (bare ceramic) if adhesion failures were resultant. Lead frames were bonded to back side metallization on the same samples which had been used for front side metallization testing. The results were similar for dry versus humid processing in that high pull strengths occurred on substrates processed in the dry environments and lower pull strengths occurred on those processed in humid conditions. Failure modes and pull strengths are shown in Figure 8. Dry processed TFNs had average pull strengths greater than 1.6 kg; average pull strengths for leads on humid processed parts were less than 1.1 kg. Very little difference was found in the protective characteristics of sheet resists versus liquid resists when the back side was protected with each.

These results established that high humidity contributes to corrosion of thin films regardless of the type of resist used. Liquid resists appear to offer more protection than sheet resists. The results indicated that back side metallization also was affected during high humidity photoprocessing even though the entire back side is coated during first-level patterning. It was significant that the results established in the initial evaluation on laboratory deposited films repeated for substrates processed in different sputtering and evaporation systems.

Effects of Different Humidity Levels

To this point extreme humidity differences had been maintained during photoprocessing of the test samples. Typical levels of humidity during storage were <6 percent RH and >85 percent RH for dry and wet conditions. In practice, humidity levels in the factory (clean room) during spring and summer months were in the 40 to 60 percent range. Because of differences in air exhaust systems due to venting, etching room humidities generally were 5 to 10 percent higher than the photopatterning room. In order to relate the corrosive effects of high humidity processing to these levels of humidity, another evaluation was planned to include a third humidity level between the high and low extremes evaluated earlier. This evaluation was conducted using the desiccated and wet container approach with an additional storage condition of 50 percent RH. A humidity chamber capable of maintaining 50 percent RH in a temperature range of 25 to 30°C was used to obtain this environment.

Substrates were processed for this evaluation identically to the previous evaluation. The same kind of substrate was used and Ta₂N was sputtered in the Continuous Process system. Chromium-gold was

MODE	FAILURES	(PERCENT)	
B	42	16	
E	32	37	100
B/E	26	47	100

Figure 8. Humidity Effects on Back Side Metallization for Different Resists and Humidities

evaporated in the E-beam planetary system; nominal gold thickness was 6 μm . Photoprocessing was conducted as before (Figure 2) except that a third storage condition was maintained for TFNs stored at 50 percent RH. The standard sheet resist photoprocessing was used characteristic of the double-sided, metallized substrates containing vias. Figure 9 shows the flow diagram for these experiments. As before, the large substrate was divided into smaller ones to eliminate potential substrate or metallization differences. After the humidity photoprocessing, routine TFN procedures of stabilizing, scribe/separate, and pre-bond cleaning were conducted. One TFN was removed from the sequence (reference

Figure 9. Process Sequence for Humidity Evaluation Using Three Humidity Levels

Figure 9) prior to pre-bond cleaning for lead frame pull tests so that the effects of CAN etching could be established. All bonding was done with 3.2-mm-wide copper lead frames.

The corrosive effects of high humidity photoprocessing repeated again. TFNs processed in a high humidity environment did not survive the lead frame bonding test; when leads were bent 90°, the gold bonding pads lifted exposing base metallization. TFNs photoprocessed in the dry containers showed exceptional pull strength averaging 1.5 kg with 90 percent type B failure modes; there were two minor incidences of metallization (type M) failures and one type C failure. These results are shown in Figure 10. TFNs photoprocessed in the 50 percent humidity environment resulted in good pull strengths averaging 1.4 kg, but there was a 100 percent incidence of metallization (type M) failures. In 45 percent of the cases, the failure occurred at the heel; however, base metallization was exposed in the uncovered bond zone. These results were typical of metallization failures in production where high pull strengths are realized yet metallization failures occur. This indicated that photoprocessing in a 50 percent humidity environment can cause corrosive effects; however, they are not as severe as those occurring in an extremely humid environment of 85 percent or greater.

Bond pull tests on TFNs which had not received pre-bond CAN etching showed similar strength to those which had been CAN etched. Metallization (type M) failures were not found after pull testing on these non-CAN etched parts; pull strengths averaged 1.4 kg with predominately heel failures (63 percent). The remaining failure modes (32 percent) were type C failures. These results substantiated the conclusion that CAN etching contributes to the corrosion of TFNs. Reference data on substrates metallized in the same lot is shown in Figure 10 for comparison; this pull strength data was obtained through routine processing of substrates in a normal factory environment.

It was concluded from this evaluation that photoprocessing in humidity environments greater than 50 percent RH causes corrosive effects. Also, CAN etchant was confirmed as being a contributor to the corrosive mechanism. TFNs which had been processed in a dry environment showed high pull strengths with one particular lead having a strength approaching 2.3 kg. Two metallization failures did occur on these dry processed parts indicating that some corrosion may have occurred even though very low humidity was maintained during storage.

Humidity Tests on Production Substrates

Similar humidity tests were conducted on substrates which had been metallized in production vacuum systems. Processing differences between laboratory and production substrates are minimal.

Figure 10. Results of Multi-Humidity Testing on Metallized Substrates

The major difference is that processes and equipment used in manufacturing have been characterized to find optimum operating conditions, and the equipment is operated within limits of a specified set of parameters. In addition, manufacturing equipment is operated daily and a production history has been established for it and the product which is manufactured.

The substrates used for humidity photoprocessing experiments were cleaned in the manufacturing cleaning room and then sputtered with Ta₂N. Evaporation of Cr-Au was done in the production E-beam planetary system to nominal 6- μ m gold thickness. The substrates contained vias and were metallized on both sides. Routine production testing showed that sheet resistivity for Ta₂N and gold metallization were within required limits. The substrates, cleaning, and metallizing processes were typical of parts being processed for product usage during this time interval. It is noteworthy that the humidity level in the production facility at the time these substrates were processed was in the 10 to 20 percent RH range.

More definitive information on corrosive effects was sought in this evaluation. In addition to the three storage conditions used previously (wet, dry, and 50 percent humidity), a storage condition was imposed at room conditions where humidity levels during testing were in the 10 to 15 percent RH range. Enough parts were started through the photoprocessing to allow TFNs to be pulled out of the sequence at various intervals: this would more accurately define at what process point the corrosive effects were occurring. A diagram of the planned evaluation is shown in Figure 11. Storage conditions during photoprocessing were essentially the same as before except several TFNs would be available for bonding tests at different photoprocess operations in the sequence. To eliminate any storage abnormalities which might occur prior to bonding, these TFNs were immediately stabilized after removal from the sequence. As in previous evaluations, the large size substrates were separated into quarter (1/4) substrates to minimize variables of substrates and metallization properties. Since six substrates were required for each storage condition in the evaluation, two additional quarter substrates were included and were pulled from the sequence during the tantalum etching evaluation point (the least likely candidate for corrosive effects). After all processing was concluded, the various TFNs were scribed, separated, pre-bond etched, and cleaned at the same time (same lot). Parts were then bonded with 177.8- μ m-thick copper lead frames for pull testing.

Results of the evaluation on production substrates were negative in that no differences in bond pull strengths or failure modes were detected for the different humidity process levels. All TFNs processed produced high lead frame pull strengths with heel (type B) failure modes. These results are shown in Figure 12 for

Figure 11. Process Sequence for Humidity Evaluation Using Four Levels of Humidity on Substrates Metallized in Production Vacuum Systems

Figure 12. Results of Production Substrate Evaluation

TFNs photoprocessed through the entire sequence. Average pull strength for all TFNs tested were above 1.4 kg regardless of wet or dry humidity photoprocessing. There was no evidence of metallization failures for any TFNs tested.

Substrates which had been pulled at various intervals in the process sequence had no significant bond test differences. Substrates which were processed in high humidity conditions yielded bond results that were as good as those for TFNs processed in dry conditions and in some instances exhibited higher pull strengths. Since the gold etching operation was considered a likely candidate for producing corrosive effects, the bonding data for TFNs removed at this portion of the process sequence is shown in Figure 13. Only the wet and dry humidity conditions are shown. No significant difference was noted in bond strength or failure modes in delaying the stripping operation 1 hour or in immediately stripping after etching. Pull strengths for all parts averaged in excess of 1.5 kg and failure modes were predominantly heel (type B) failures.

Results of this evaluation appeared to contradict the results of the previous evaluations where corrosive effects were attributed to high humidity conditions during photoprocessing. No apparent differences in corrosion were attributable to humidity levels in this experiment. An overtest was planned to determine if any small differences existed in the metallization films due to humidity photoprocessing. TFNs which had completed all operations in the photoprocessing sequence (reference Figure 11) were used for this test. Overtesting consisted of immersing TFNs in CAN etchant for a period of 16 hours and then repeating bond tests using copper lead frames. Normal CAN etch time for prebond cleaning is 60 seconds; since previous results had shown CAN etching to be a contributor to corrosive effects, this test was expected to result in noticeable corrosive effects if any differences were present. Results of the test are shown in Figure 14. There was no evidence of corrosive effects for parts subjected to the 16-hour CAN etchant. All samples processed at different humidities yielded high lead frame pull strengths with a majority of heel failures.

It was concluded that the production films used in this evaluation were not susceptible to corrosive effects during photoprocessing. No degradation or corrosion was found regardless of humidity storage conditions during photoprocessing. Overtesting conducted with extended times in CAN etchant did not produce a significant difference between parts. Specific tests to yield information on where corrosive effects were initiated were similarly unsuccessful. Therefore, the thin film metallization on these production substrates was determined extremely resistant to corrosive effects. The results indicated that corrosion susceptibility was initiated prior to photoprocessing.

Figure 13. Effects of Humidity Processing on Gold Etching Operation for Substrates Metallized in Production Vacuum Systems

Figure 14. Overtest Results for Humidity Processed Production Parts

Subsequent Humidity Testing and Conclusions

Three of the four tests to this point had shown corrosive effects caused by photoprocessing in high humidity environments. Evaluations subsequent to these four were planned to get more details on the process point at which corrosion occurred during photoprocessing and to determine the percent humidity that was the threshold level to cause corrosion. These continued evaluations were unsuccessful in that the metallized substrates used were not susceptible to corrosive effects during photoprocessing regardless of humidities. These results substantiated previous conclusions that susceptibility of thin film metallization to corrosion was established prior to the photolithography process.

Additional tests were conducted using different bonders to ensure that bonding was not entering into the results; production bonding equipment was used to substantiate results found with the laboratory lead frame bonder. Regardless of overtest, bonding equipment, and evaluation sequence, once the thin film metallization was found to be insensitive to corrosive effects, it generally was stable and impervious to any overtests.

A total of six evaluations were conducted on different lots of metallized substrates; three lots of which were found to be susceptible to corrosion during humidity photoprocessing. Analysis of these results provided several conclusions which were valuable for further work in corrosion testing.

- Corrosive effects of photoprocessing are compounded by high humidity environment. Levels of humidity in excess of 50 percent RH were determined to be detrimental during photoprocessing; no minimum limit of humidity was established.
- Film corrosion susceptibility is created prior to the photoprocessing operations.
- CAN etching during pre-bond cleaning after photoprocessing is a contributor to the corrosive process.
- Sheet resist photoprocessing used for two-sided metallized substrates containing vias is more likely to produce corrosive conditions.
- Some films are not susceptible to corrosive effects during photoprocessing or pre-bond etching. The films in this category demonstrated extraordinary resistance to corrosion during extended times in CAN etch.
- The general result of these experiments is that a test has been established for corrosion susceptibility by a photoprocessing evaluation using extremes of humidity for storage.

Further analysis of the corrosive characteristics of thin film metallization was planned for substrate and film properties. Since several groups of metallized substrates were now identified as being susceptible, analysis of the metallization morphology and ceramic characteristics could be studied to determine the cause of corrosion susceptible films.

Analysis of Peeling Gold

In the initial evaluation using humidity photoprocessing, substrates processed in wet conditions were found to exhibit poor adhesive characteristics with subsequent peeling of gold during cleaning. (Refer to Figure 4B.) To determine where in the metallization layers the loss of adhesion had occurred, analysis of the substrate surface (at the bond zone) and the back side of the peeled gold was performed using Auger analysis. Results of this analysis are shown in Table 2.

Initial Auger sputtering showed that the bond zone on the substrate surface (where the gold had peeled) contained traces of chromium, oxygen, carbon, and copper present in that order of magnitude. After 2 minutes of sputtering, only chromium and oxygen were present at the bond location. Analysis of the back side of the peeled gold showed the elements gold, iodine, copper, and carbon were present; after 2 minutes of sputtering, only gold was present. This information established the corrosive mechanism was occurring at the chromium-gold interface since chromium remained on the substrate surface when loss of adhesion occurred. Also, iodine was discovered reaching the chromium-gold interface to set-up the corrosive conditions. The discovery of iodine was significant because iodine normally sublimates at room temperature and was not expected to be found in a sample subjected to vacuum pressures (during Auger analysis). This indicated a substantial amount of iodine had been present and was probably trapped within the gold structure. Further work was then performed to determine how the iodine penetrated the metallization.

Iodine and Etchant Penetration of Resist

The work thus far had established that iodine in gold etchant contributes to the corrosion of chromium-gold thin films. It was not fully understood how the gold etchant entered into the corrosive mechanisms, however, its presence was always found to be detrimental to a corrosion susceptible thin film. Since the gold surface was protected during conductor etching, the hypothesis was that gold etchant must be reaching the surface through the resist. Tests were conducted to determine the permeability of dry film photoresist by gold etchant ($KI + I_2$).

Table 2. Elements Found by Auger Analysis of Peeling Metallization in Evaluation of Laboratory Metallized Substrates

Process	Bond Zone Surface	Back Side of Peeled Gold
Presputter	Chromium Oxygen Carbon Copper	Gold Iodine Copper Carbon
Postsputter (2 Minutes)	Chromium Oxygen	Gold

Initial experiments were conducted using a starch solution and gold etchant separated by a membrane of dry film photoresist. Starch is a good indicator of iodine and was expected to indicate when the etchant penetrated the resist. This method of testing was not successful because it was used several times and required hours or days for the resist to penetrate. Because these times were possibly in error, another approach was attempted.

In a subsequent test, resist was laminated on a substrate, exposed, developed, and then subjected to gold etchant. After predetermined times, visible evidence of etchant penetration on the substrate surfaces were expected to occur. This approach was first used on gold evaporated substrates and was not successful since any indication was removed when the resist was stripped.

The permeability test was repeated using copper clad printed circuit board material. This material was chosen because copper is a good indicator of corrosive effects by iodine. The 50- by 50-cm copper plates were first cleaned to remove any stains, and then dry film photoresist was laminated onto them at nominal photoprocessing parameters. The photoresist was exposed and developed (even though no pattern was present) using routine processing to simulate TFN conditions. Testing was then conducted by applying several drops of gold etchant in the center of the resist-covered copper plate for a predetermined time; the etchant was not allowed to dry out and fresh etchant was added when this occurred. Etchant penetration through the photoresist was found to be detectable on the copper after stripping the resist. Using the stain criteria, etchant penetrated through the exposed dry

film photoresist in 60 to 70 minutes. This experiment was repeated using unexposed and undeveloped resist; etchant penetration was found to occur in 30 to 45 minutes. Therefore, an initial conclusion was that the exposure and development of dry film resist acts to toughen the resist against etchants as suggested in the manufacturer's literature.

These test results indicated that iodine corrosive effects were not occurring because of resist penetration of gold etchant. Specification limits require the resist to be removed within 1 hour of etching. Generally, stripping is accomplished in production before this time limit expires. This could be occurring during resist stripping as some of the etchant is absorbed by the dry film resist during etching. It could be released into the stripping solution and then reach the exposed surface conductors. Further tests on iodine penetration of photoresist are being conducted and results are not yet available.

Effects of Gold and Ceramic Surface Structure on Corrosion Susceptibility

Results of humidity testing had indicated that some films were highly sensitive to humidity during photoprocessing and others were not. To establish the differences in these films, scanning electron microscope (SEM) analysis was performed. The differences are shown in Figure 15 for each type of metallization film (corrosion sensitive and corrosion resistant). These photographs were taken at 1000 and 3000X; arrows indicate the voids found in the corrosion sensitive films. The voids are indicated by a dark area which is surrounded by a white halo and occur between the grains. The halo is caused by an excess amount of electrons at this area created by a cavity or a void. Although the depth of these voids cannot be determined by SEM methods, it does offer a reasonable hypothesis that voids penetrate the thin film interface allowing any iodine present to permeate the film. A further assumption is that keeping these pores or voids dry would tend to minimize corrosion by gold etchant. This could explain the reason some films were sensitive to high humidity photoprocessing while void-free films were not affected.

Although there was no reason why voids occur on some metallization films and not others, there was a theory that the ceramic structure could be part of the explanation. To further explore this theory, the metallization was removed from the circuits (Figure 15) and SEM analysis was conducted on the ceramic surface. These results are shown in Figure 16. The ceramic was found to contain the same void conditions as the substrate surface, although it was not possible to relate the gold voids to the ceramic voids on a one-to-one basis. The references show the same voids in the 300

1000X

CORROSION SENSITIVE

CORROSION RESISTANT 1000X

3000X

CORROSION SENSITIVE

CORROSION RESISTANT 3000X

Figure 15. SEM Photographs of Gold Metallization (Substrate on left was sensitive to high humidity during photoprocessing; substrate on right was not. Arrows indicate gaps in grain boundary.)

300X

1000X

SENSITIVE GOLD METALLIZATION LOT

300X

1000X

STABLE GOLD METALLIZATION LOT

Figure 16. SEM photographs of Ceramic Surface (Metallization deposited on substrate containing voids, arrows, was found susceptible to high humidity photo-processing.)

and 1000X magnification photos. The white halo effects around the voids are caused by electron charging effects as before; a thin layer of metallization (30-nm Pd-Au) is deposited on the ceramic to permit SEM pictures, but the penetration of evaporant into the voids is insufficient to coat the walls. This results in electron charging and the resultant halo around the void.

Conclusions from this SEM work were that some ceramic surfaces contain voids which may proliferate up through the gold metallization during deposition and allow penetration of corrosive chemicals to the chromium-gold interface.

An abnormally high reject rate occurred when all production bond monitors failed to pass the metallization adhesion tests. This condition was investigated by examining the gold and ceramic characteristics of several TFNs produced during that month. It was found by SEM analysis that the gold metallization exhibited a very porous surface structure (Figure 17A). The gold grain boundaries appeared to be separated by a lattice of voids throughout the surface unlike the occasional void found in previous SEM studies. The occurrence of 100 percent metallization failures on substrates with these metallization properties was not surprising; the problem was compounded because humidity in that month was extreme. For comparison, metallization characteristics of the same month the next year are shown in Figure 17B. The gold structure is relatively void free, resulting in less corrosion susceptibility.

One void found in the TFN surface gold was of such magnitude that an attempt was made to examine the identical ceramic surface below it. Prior to this, it had not been possible to relate voids in metallization to voids in ceramic structures on a one-to-one basis. Because of its size and position relative to a conductor edge, it was possible in this instance to relate the gold void to a void in the ceramic (after gold removal) at identical locations. This confirmed the earlier conclusion that voids in ceramic could proliferate to the surface during metallization and provide a path for etchants to reach the metallic interface (Figures 18).

Desiccated Photoprocessing for Production of TFNs

The previous results indicated that improvements in TFN yield could be made if an effort was made to maintain dry conditions during photoprocessing. This could be accomplished by lowering the humidity in the photoprocessing area or maintaining films in a dry container during critical times in the process sequence. The most expedient manner to accomplish the dry conditions was to use desiccated containers. Requirements for this process were established from the results of the corrosion evaluation study.

METALLIZATION A

**VOID IN CERAMIC SURFACE
AT SAME LOCATION**

METALLIZATION B

**METALLIZATION B WAS
INSENSITIVE TO HUMIDITY
DURING
PHOTOLITHOGRAPHY:
METALLIZATION A
WAS SUSCEPTIBLE**

Figure 17. SEM Photographs of Metallization

300X

1000X

LARGE GOLD METALLIZATION VOID

300X

1000X

VOID IN CERAMIC SUBSTRATE AT IDENTICAL LOCATION

Figure 18. SEM Photographs of Gold and Ceramic Surface (Large gap in grain boundary correlated to the same location as a ceramic void. Smaller gaps in gold metallization also are visible.)

The procedure established for photoprocessing production TFNs were similar to the methods used in the humidity photoprocessing evaluation. An abbreviated procedure is given below.

- Immediately after evaporation, substrates are placed in desiccated, airtight containers.
- Substrates are patterned with dry film resist.
- Substrates are stored overnight in desiccated containers.
- Chromium/gold is etched and photoresist is stripped.
- Substrates are stored for a minimum of 1 hour in desiccated containers.
- Wet resist is applied and patterned for resistor photolithography.
- Substrates are stored overnight in desiccated containers.
- Tantalum is etched and wet photoresist is stripped.
- Substrates are returned to desiccated containers until stabilization.

The containers, desiccant, and substrate arrangement, which were used for storage of substrates during photoprocessing, are shown in Figure 19. The container has a carrier which holds the substrates with desiccant stored around the periphery so the substrates do not contact the desiccant.

An analysis (or history) of Bendix Kansas City TFN metallization showed improvement in bond qualification yield during the humid spring months after desiccated storage was initiated.

ACCOMPLISHMENTS

Humidity effects on chromium-gold TFNs during photoprocessing were investigated. An increase in humidity during photoprocessing was found to generally cause an increase in corrosive effects which can result in metallization failures and low lead frame pull strengths. Some substrates were found to be more susceptible to humidity effects than others, and the corrosion susceptibility was initiated before photolithography. Corrosion susceptibility was traced to voids or pores in the gold surface; these were on the ceramic surface before metallization. Many of these pores were still detectable by SEM examination after metallization.

Figure 19. The Container, Desiccant, and Substrate Arrangement

This work resulted in a desiccated storage process currently in use for TFN production fabrication.

FUTURE WORK

The following areas are recommended for further improvements in control of corrosion effects in TFN manufacturing:

- More effective ways to etch the gold and chromium, such as changes in etchant concentrations,
- Additional methods of rinsing the gold and chromium etchants to more effectively remove them or neutralize their effects, such as various rinsing time periods and various pre-rinse solutions,
- Gold spray etching, which allows faster removal of the thicker gold required for RF circuits and subsequently less exposure time to the iodine solution, and
- More work is needed on substrate voids to determine their relationship to film properties. A test for gross substrate conditions could be beneficial in screening out exceptionally poor (or porous) substrates.

REFERENCE

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BDX-613-2312

EFFECTS OF HUMIDITY DURING PHOTOPROCESSING ON THIN FILM METALLIZATION ADHESION, D. P. Norwood, Topical, March 1980.

Humidity effects during photoprocessing on tantalum/chromium/gold thin film networks (TFNs) were investigated. Humidity conditions at various process steps were controlled by placing either desiccant or water in handling containers for the TFNs. The TFNs photoprocessed in humid conditions had a much higher occurrence of metallization failures compared to TFNs processed in dry conditions. Ceramic surface defects were shown to cause

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