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Etchback Smear-Removal-Process Characterization

By J. H. Richardson

Published March 1981

MASTER

Final Report

Prepared for the United States Department of Energy
Under Contract Number DE-AC04-76-DP00613.



**Kansas City
Division**

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By J. H. Richardson

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Final Report

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Department of Energy under Contract Number
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SUMMARY

A study evaluated the tolerance limits of three variables for each of five chemical solutions used in etchback smear removal on multilayer printed wiring boards (MLPWBs). The purposes of the study were to verify the variables' applicability, isolate potential sources of erratic etchback behavior, and establish etchback data for later use.

Etchback, as a form of smear removal, is a basic process used at Bendix Kansas City in the manufacture of about 40 multilayer parts. However, the erratic behavior of etchback contributes to reduced yield on multilayer parts.

A series of 43 tests on 172 multilayer boards under controlled conditions examined part immersion time, and solution concentration and temperature in each of the etchback chemical solutions: chromic acid, hydrofluoric acid, chrome neutralizer, hydrochloric acid, and ammonium persulfate. The generation of many data points from etchback measurements taken from samples required a specially prepared computer program to manipulate the data.

Although the results showed that recessed conductors occurred in 38 of the 43 tests on MLPWBs, the results did not indicate a direct relationship between recessed conductor existence and process control limits.

The study indicated that chemical interaction may not cause erratic etchback behavior and that slight changes in variables do not influence the presence of recessed conductors. The study verified the adequacy of existing tolerances on main process variables to produce uniformly etched holes.

DISCUSSION

SCOPE AND PURPOSE

A basic processing step in the manufacture of multilayer printed wiring boards (MLPWBs) is smear removal. Etchback, the form of smear removal used at Bendix Kansas City, is a complex chemical process that removes epoxy resin smear and a portion of the epoxy-glass matrix from the side walls of drilled holes. Since its inception, etchback has been a contributor to the reduced yields in multilayer boards. The purpose of this development project was to evaluate the limits of the principal process variables for each solution comprising the etchback process: part immersion time, solution concentration and solution temperature.

Approximately 40 multilayer parts requiring etchback now are being manufactured.

ACTIVITY

Plated through-holes in multilayer printed wiring boards (MLPWBs) are a unique processing problem. Intimate, physical contact between each conductive layer is essential to the function of MLPWBs. This contact, however, can be impeded by epoxy smear. Heat generated by drilling softens the epoxy resin in the laminate. Movement of the drill bit smears the softened resin across the exposed conductive layers. Unless it is removed before plating, the smeared epoxy prevents complete contact between the inner layers and through-hole plating. In severe cases, even electrical continuity between layers can be prohibited.

Another area of concern is stress-induced separation of the through-hole plating from the side walls of MLPWBs. Considerable disagreement exists within the printed wiring board industry about the extent of this type of failure. However, proponents of the etchback form of smear removal suggest that copper surface area exposed by drilling and conventional smear removal is insufficient to prevent separation between the inner conductive layers and through-hole plating during thermal or mechanical stress.

The etchback process used at Bendix is designed to accommodate both stress-induced separation and epoxy resin smear. Removal of both the epoxy resin smear and a portion of the epoxy-glass matrix from the side walls of drilled holes provides additional copper contact surface area to strengthen through-hole plating. Although etchback successfully has eliminated stress-induced

separations and has provided complete smear removal, it also has been a source of recurring production problems because of erratic and inconsistent material removal.

The etchback process shown in Figure 1 uses five chemical solutions: ammonium persulfate for copper oxide removal; hydrochloric acid for removal of sulfate smut and other residues; chromic acid to remove epoxy; chrome neutralizer to neutralize the chromic acid, and hydrofluoric acid to remove glass. Each of the solutions that make up the etchback process has three main process variables: immersion time, solution concentration, and solution temperature. The allowable range for each of these variables is given in Table 2. The project goals were to evaluate the tolerances placed on these three process variables; confirm the current validity of those tolerances; isolate and identify unforeseen problem areas created by the interaction among the five chemical solutions; and establish a data foundation for future smear removal and etchback development projects.

Test Procedure

To carry out this experiment, 258 innerlayers, 152.4 x 228.6 mm consisting of 0.036 mm-thick copper plated on two sides of a 0.127 mm-thick glass epoxy laminate were black oxide-coated and laminated in accordance with the standard process that uses prepreg to yield 86 six-layer panels. Each of these panels was then drilled in a predetermined pattern to yield two separate boards to a panel. The drill pattern consisted of 92 holes from each of three different drill sizes 0.610, 0.840, and 1.07 mm in diameter. All panels were cut in half prior to etchback for a total 172 parts.

Etchback was performed using 3.8 L glass beakers. None of the solutions was agitated, and all were replaced or replenished as necessary to maintain proper solution concentrations. Forty-three separate trials, four parts to a trial, were conducted according to the chart in Table 3. An additional cleaning in ammonium persulfate was added after etchback to duplicate that portion of the preplate cleaning that removes copper. This cleaning was done as a result of a recently observed condition called recessed conductors, wherein the copper innerlayers themselves appear to be overetched (Figure 2). The first board in each of the 43 test runs received a 15-second preplate cleaning in ammonium persulfate, but the remaining three boards in each test run received the standard 60-second preplate cleaning. After etching, all parts were copper-plated. After plating, one hole from each drill size was chosen at random from each board to be cross-sectioned and measured.

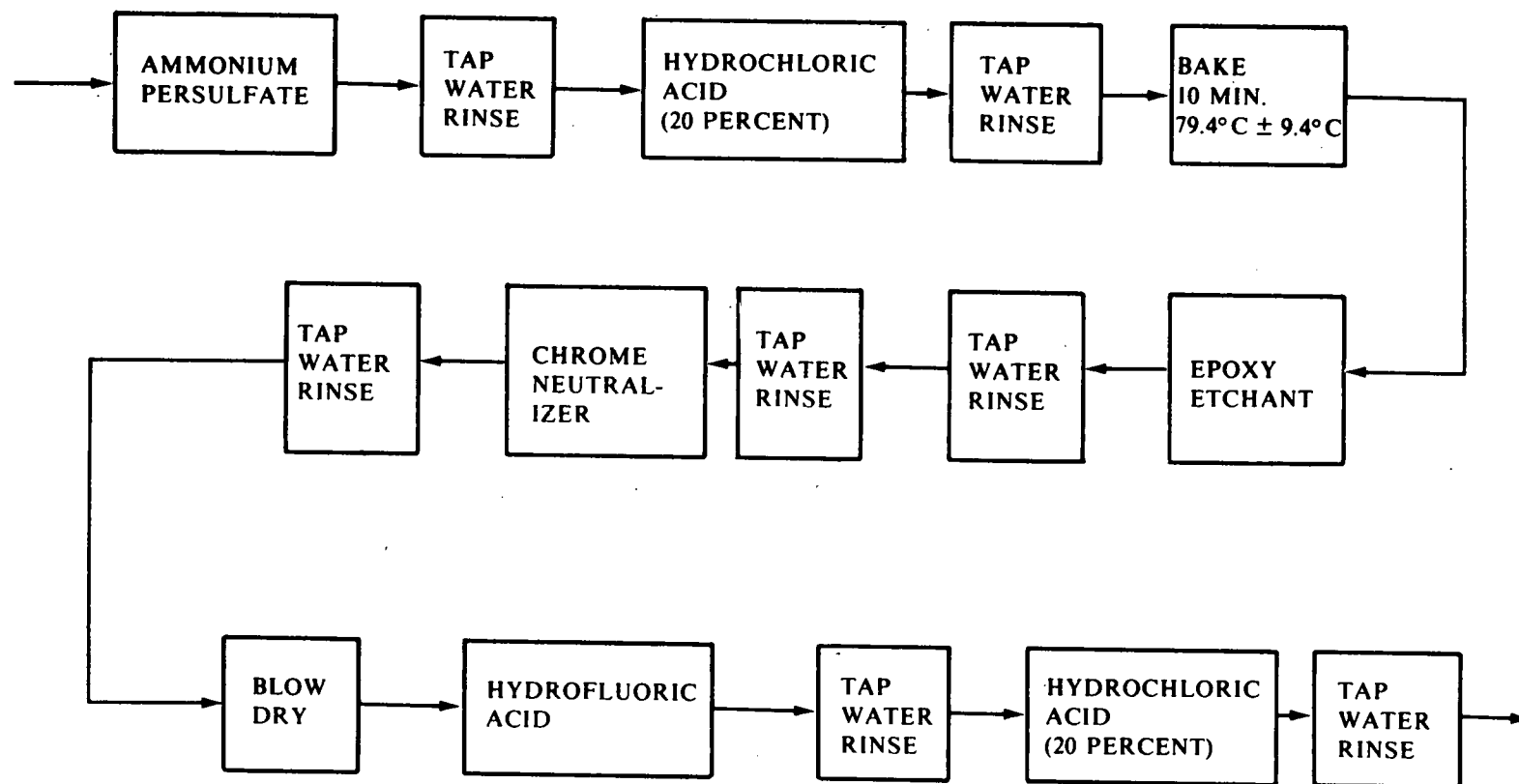
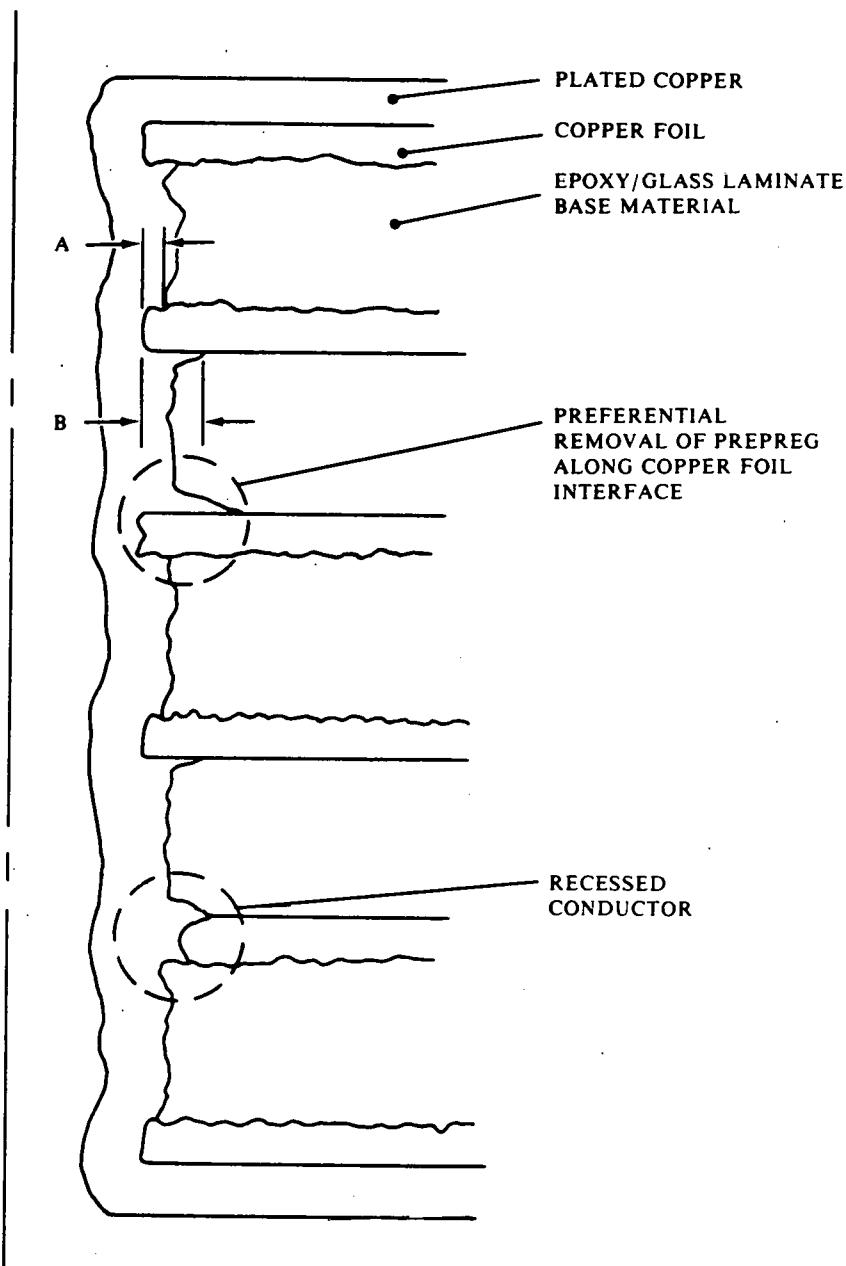


Figure 1. Etchback Process



CENTER LINE OF
DRILLED HOLE

$A + B$ EQUAL TO OR GREATER THAN 0.010 mm
 A OR B EQUAL TO OR LESS THAN 0.05 mm

Figure 2. Etchback Showing Recessed Conductors

Table 1. Solution Control

Etchback Process

Solution Ammonium Persulfate

Hydrochloric Acid (20% reagent)

Epoxy Etchant

Chrome Neutralizer

Hydrofluoric Acid

Twenty measurements of etchback were taken for each of the 12 samples in a test run for a total 10,320 possible data points. The many data points required a specially prepared computer program to manipulate the data and allow the maximum amount of useful information to be extracted from the data. A summary of that information is given in the Appendix.

Analysis

The acceptable etchback range is from 0.010 mm, representing the sum of two sides, to 0.051 mm, which corresponds to the maximum of any one side. All of the samples measured conform to these requirements. This suggests that the control limits on time, temperature, and concentration for each solution are valid and capable of yielding acceptably etched holes for through-hole plating. The actual amount of etchback ranged from 0.008 to 0.028 mm with a standard deviation of 0.005 to 0.008 mm. This is a much tighter grouping of test results than is commonly found from analysis of production parts.

Recessed conductors were a problem throughout the evaluation. Thirty-eight of the 43 test runs indicated at least one sample hole displaying one or more recessed conductors. This condition was particularly unusual because copper is only susceptible to attack from the ammonium persulfate cleaning solution. The project was expected to indicate evidence of recessed conductors when the process variables of the ammonium persulfate baths took maximum values, but not at any other time. Although holes from these conditions showed a high incidence of recessed conductors, the 12 holes from the test run that represented maximum exposure time and solution temperature showed no evidence of recessed conductors.

Table 2. Process Variables

Solution	Concentration		Time (s)	Temperature (°C)
	g/L	Volume Percent		
Ammonium Persulfate				
Minimum Value	210		45	24
Maximum Value	270		60	27
Chromic Acid (Epoxy Etchant)				
Minimum Value	900		88	77
Maximum Value	950		92	82
Hydrofluoric Acid				
Minimum Value		34	30	24
Maximum Value		37	35	29
Chrome Neutralizer				
Minimum Value	45		60	24
Maximum Value	75		90	29
Hydrochloric Acid				
Minimum Value		17	60	24
Maximum Value		23	70	29
Hydrochloric Acid				
Minimum Value		17	45	24
Maximum Value		23	60	29
Ammonium Persulfate (Preplate Cleaning)				
Minimum Value	240		15	24
Maximum Value	240		60	24

Project results did not indicate any direct relationship between variances in the process control limits and the existence of recessed conductors in plated through-holes. Consequently, the project test conditions were compared to the production etchback system to detect variations. The only significant difference was in the degree of agitation. None of the test baths was agitated; however, in the production area, the operator manually agitates each panel throughout the etchback cycle. The ammonium persulfate and chromic acid solutions are air agitated. Stagnant or poorly agitated solutions possibly may influence etchback more than was previously considered. As a result, additional studies are

Table 3. Test Runs

Test Variable		Solution*	Test Variable		Solution*
1	All	All	23	Conc/Temp.	3
2	Concentration	1	24		4
3		2	25		All
4		3	26	Conc/Time	1
5		4	27		2
6		5	28		3
7		All	29		4
			30		5
			31		All
8	Temperature	1	32	Temp/Time	1
9		2	33		2
10		3	34		3
11		4	35		4
12		5	36		5
13		All	37		All
14	Time	1	38	All	1
15		2	39		2
16		3	40		3
17		4	41		4
18		5	42		5
19		All	43		All
20	Conc/Temp.	1			
21		2			
22		3			

*Solutions are 1, ammonium persulfate; 2, chromic acid; 3, hydrofluoric acid; 4, chrome neutralizer; and 5, hydrochloric acid. All but Test 1 were conducted at the maximum limit; Test 1 was conducted at the minimum.

investigating the exact relationship between degrees of agitation and the occurrence of recessed conductors.

ACCOMPLISHMENTS

The accomplishments of this project include the following:

- The existing tolerances on the main process variables of time, temperature, and concentration are adequate and capable of producing uniformly etched holes.

- The presence of recessed conductors in etched holes is independent of slight variations in solution temperature and concentration or exposure time.
- Erratic etchback results, including recessed conductors, are not caused by chemical interactions but may be a function of physical limitations of the production etchback facility.

FUTURE WORK

Future work on etchback will include a complete evaluation of agitation effects on recessed conductors and the development of a fully automated etchback line to minimize the variations in etchback now evident in production.

Appendix

ETCHBACK DATA

Table A-1. Etchback Data

	Holes With Recessed Conductors**				
Variable*	15-s Preplate	60-s Preplate	Recess (μm)	Etchback (μm)	Standard Deviation (μm)
Ammonium Persulfate					
Concentration	3/3	3/9	13	18	8
Temperature	1/3	0/9	3	25	8
Time	0/3	3/9	13	18	8
Concentration/ Temperature	3/3	2/9	20	13	5
Concentration/ Time	3/3	8/9	20	15	5
All	3/3	0/12	8	23	8
Chromic Acid (Epoxy Etchant)					
Concentration	3/3	3/9	18	20	8
Temperature	0/3	7/9	23	8	5
Time	1/3	3/9	8	20	8
Concentration/ Temperature	3/3	1/9	18	18	5
Concentration/ Time	0/3	3/9	18	20	5
Temperature/ Time	2/3	0/9	10	23	8
All	0/3	0/9	0.0	23	5
Hydrofluoric Acid					
Concentration	0/3	0/9	0.0	23	8
Temperature	0/3	0/9	0.0	23	8
Time	3/3	6/9	10	15	8
Concentration/ Temperature	0/3	3/9	13	18	5
Concentration/ Time	1/3	3/9	13	15	5
Temperature/ Time	1/3	3/9	20	15	5
All	3/3	1/9	13	18	5
Chrome Neutralizer					
Concentration	1/3	3/9	18	20	8
Temperature	3/3	3/9	18	15	8

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ELECTRICAL: Etchback Smear Removal

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