Photoresist Lifting Solved Using Design of Experiments

By Dr. Richard Mayes & Dr. John Ganjei

Statistical experimental design enabled personnel at MacDermid Imaging Technology, Inc., to pinpoint surface preparation as the cause of a customer's electroplating problem. The group was able to isolate the problem at photoresist lifting during the acid copper and tin/lead plating process. It caused severe underplating and sporadic shorts after etching. To accomplish this, the MacDermid team used an experiment designed to isolate surface preparation from the variables and interactions involved in printed circuit board (PCB) manufacturing.

By using a statistical software package, the solution to the photoresist lifting problem was relatively easy. Increasing the rinse time after electroless copper plating, plus maintaining the pH of the rinse at an acidic level, eliminated the resist lifting problem. Isolating the variables involved in the process was the toughest challenge for the team.

Information is Essential To Developing a Design

The most important part of developing a design is interviewing supervisors and line operators. The choice of variables is determined through interaction and discussion with the customer who knows the process being used, and the supplier who knows the product and the process. This interaction helps to eliminate unnecessary experimental design points.

MacDermid is a producer of dry film photoresist, a product used in PCB manufacturing to transfer a circuit's image to the copper-clad laminate. The company's proprietary* dry film is a photosensitive photopolymer sandwiched between polyester and polyethylene. The photopolymer is laminated to the substrate by removing the polyethylene layer, followed by hot-roll lamination. The remaining polyester sheet acts as a protective cover and oxygen barrier through the exposure process.

Atmospheric oxygen inhibits the exposure process. MacDermid engineers point out that many processing conditions affect photoresist performance, such as: Lamination speed, light intensity, surface preparation, spray pressure and others. These variables, and their interactions, make the PCB manufacturing process a prime candidate for experimental design techniques.

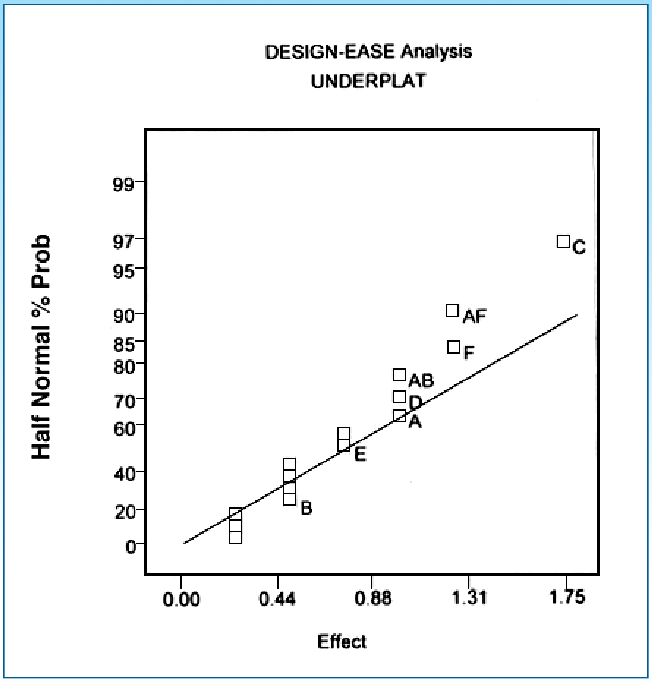


Fig. 1—A half-normal probability plot of the effects of the six variables (factors).

Processing conditions vary greatly among the thousands of shops that produce PCBs. As a result, the continuing challenge to MacDermid scientists is to develop products that perform well within the spectrum of specified conditions. The company must quickly resolve customer problems when they occur, either by providing a different product or by suggesting a change in processing conditions.

In the early 1980s, the company started to use statistical experimental design techniques for product development and in-the-field troubleshooting. This change resulted in faster development times for new products and increased quality of technical service field calls. At first, they used a program written by a statistician at their research center. They found it somewhat awkward to use and switched to a commercial software package.**

Quicker Turnaround

The software uses dialog boxes, simple forms, and on-line prompts to guide them through the steps needed to design and analyze experiments. The program generates recipe-like worksheets for running the experiments in random order. This ensures lurking variables will not confound the results. The software then performs statistical analyses and generates graphical outputs that clearly identify factors. It also validates statistical assumptions and shows key interactions.

MacDermid engineers used these methods to help a customer improve the quality of a double-sided electroless printed circuit board. Examine

*Aqua Mer®, MacDermid Imaging Technologies, Inc., Wilmington, DE

**Design-Ease, Stat-Ease, Inc., Minneapolis, MN

Arrangement of Variables Into a Fractional Factorial Design

Std	Dsn ID	Run	Block	Prel Hold Day Factor	Lam Spd ft/min Factor	Anti-tarn Factor	Expose SS (21) Factor	Develop Dwell BP Factor	Plate A/ft^2 Factor	Underplt Degree Response
14	14	1	1	8.00	10.00	0.00	9.00	60.00	30.00	2.00
15	15	2	1	0.00	4.00	0.00	9.00	60.00	60.00	4.00
16	16	3	1	8.00	4.00	0.00	9.00	40.00	60.00	4.00
1	1	4	1	0.00	10.00	1.00	7.00	60.00	30.00	0.00
3	3	5	1	0.00	4.00	1.00	7.00	40.00	60.00	2.00
9	9	6	1	0.00	10.00	1.00	9.00	60.00	60.00	3.00
5	5	7	1	0.00	10.00	0.00	7.00	40.00	60.00	6.00
12	12	8	1	8.00	4.00	1.00	9.00	60.00	30.00	2.00
11	11	9	1	0.00	4.00	1.00	9.00	40.00	30.00	0.00
2	2	10	1	8.00	10.00	1.00	7.00	40.00	30.00	1.00
8	8	11	1	8.00	4.00	0.00	7.00	40.00	30.00	1.00
4	4	12	1	8.00	4.00	1.00	7.00	60.00	60.00	0.00
10	10	13	1	8.00	10.00	1.00	9.00	40.00	60.00	1.00
13	13	14	1	0.00	10.00	0.00	9.00	40.00	30.00	4.00
7	7	15	1	0.00	4.00	0.00	7.00	60.00	30.00	1.00
6	6	16	1	8.00	10.00	0.00	7.00	60.00	60.00	1.00

the customer's procedures to make the board:

1. Drill the panel.
2. Deposit electroless copper inside the drilled holes. (Provides an electrical connection between the two sides of the finished panel.)
3. Add an anti-tarnish agent to the copper after it goes through the electroless line. (Prevents oxidation.)
4. Apply photoresist in dry film lamination process.
5. Expose photoresist.
6. Develop off photoresist that has not been exposed to light. (Developing exposes the surface in preparation for electroplating.)
7. Electroplate areas with copper followed by tin and lead where photoresist was removed. (Defines the correct conductive pattern on the board.)
8. Strip the remaining photoresist with a strong caustic. (Exposes the unwanted copper substrate.)
9. Etch away the exposed copper using an alkaline etching solution. (The tin/lead plating resists the etchant, leaving the circuit line and pads intact.)

Correcting the Faults

At the customer's facility, the photoresist infrequently lifted during the plating process. Tin/lead would be deposited in unwelcome areas, causing sporadic shorts when tested. Costly rework would follow, or more importantly, generation of scrap product.

In correcting the quality faults, MacDermid engineers studied six process variables (factors):

- Prelamination hold time. (The waiting time before the board is laminated.)
- Lamination speed. (Rate the board travels through the lamination machine.)
- Use or non-use of anti-tarnish. (Prevents oxidation of the copper surface.)
- Exposure energy. (Amount of ultraviolet energy used to cure the photoresist.)
- Developer dwell. (Amount of time that the board remains in developer.)
- Plating current density. (Speed at which the copper is plated.)

Results of the Experiment

MacDermid engineers arranged the variables into a fractional factorial design. Using the proprietary software, they constructed a 1/4-replicate,

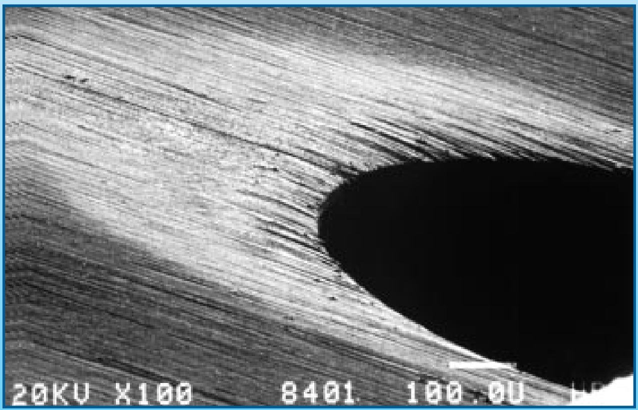


Fig. 2—SEM of stained area.

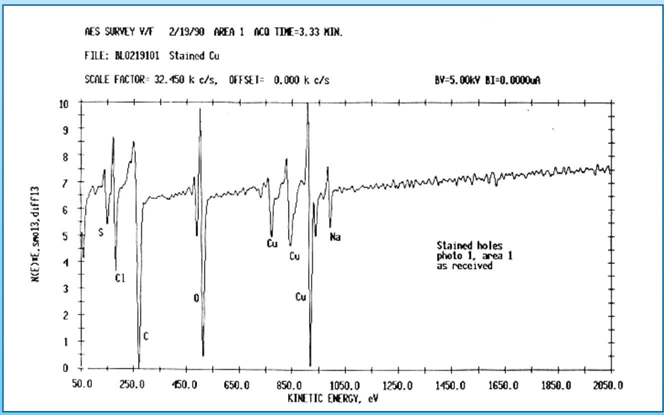


Fig. 3—AUGER surface analysis of stain.

six-factor experimental design requiring only 16 experiments (See accompanying table).

A half-normal probability plot (Fig. 1) of the effects clearly showed the variables that stood out:

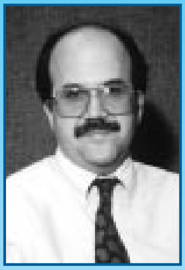
1. Anti-tarnish (Factor C), significant at 97 percent confidence level.
2. Plating current (Factor F), at 85 percent confidence level.
3. Interaction between plating current and prelamination hold (Factor AF), at 90 percent confidence level.

The strong correlation to anti-tarnish level and prelamination hold-time suggested that the preclean process may be suspect. The correlation to plating current density suggested that photoresist adhesion was compromised at the electroless copper surface. In general, the higher the current density, the more aggressive the plating. If adhesion of the resist is not optimal, the aggressive nature of the plating will lift the resist. With these factors in hand, the engineers focused their attention on the surface preparation process.

They examined panels—after electroless copper deposition, but prior to lamination—at 100X magnification (Fig. 2). They discovered telltale stains. These stains centered on the annular pads where the photoresist lifting was concentrated. Using spectroscopic analysis (Fig. 3), the chemists studied the chemical composition of the stained area. Results showed the stain was primarily copper, chlorine, sulfur and sodium. This analysis matched the composition of copper chloride, copper sulfate, sodium chloride and sodium hydroxide salts generated during the electroless process. To ensure proper photoresist adhesion, these salts must not be present. Of particular concern was the presence of sodium, suggesting that sodium hydroxide was trapped in the thru-hole. Sodium hydroxide is extremely detrimental to photoresist adhesion.

Armed with this discovery, the MacDermid engineers gave their customer this advice: If you improve the quality of your rinse operation after electroless deposition, and closely monitor the pH on the rinse to neutralize the hydroxide, your problem will be eliminated. **P&SF**

About the Authors



Dr. Richard Mayes

Dr. Richard T. Mayes is the manager of R&D, electronics at MacDermid Imaging Technologies, Inc. He was previously with Hercules, Inc., which divested the Hercules Electronics and Printing Group to MacDermid, Inc. His primary interests include fine pitch imaging for the PCB industry, lead frame applications and the chemical milling industry.

Dr. John Ganjei

Dr. John D. Ganjei is the market development manager, electronics at MacDermid Imaging Technologies, Inc. He has been involved in the electronics industry for 10 years, performing R&D and technical service duties.He is focusing on marketing a systems approach to manufacturing innerlayer circuit boards at low cost and high-yield using liquid protoresist technology.