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IMPROVED PRECISION ALUMINUM ETCHANT

PDO 6984887, Topical Report

W. H. Knopf, Project Leader

Project Team:
J. W. Chesney

Published August 1976

MASTER

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IMPROVED PRECISION ALUMINUM ETCHANT

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August 1976

Prepared by W. H. Knopf, D/812, under PDO 6984887

An improved solution was developed to etch 0.00045-inch-thick (11.43 μm) 1145 rolled aluminum for precision pattern hardware. The new etchant produces smooth etched edges with little or no undercut.

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CONTENTS

Section	Page
SUMMARY.	5
DISCUSSION	6
SCOPE AND PURPOSE.	6
PRIOR WORK	6
ACTIVITY	6
ACCOMPLISHMENTS.	18
FUTURE WORK.	20
REFERENCES	21
DISTRIBUTION	22

ILLUSTRATIONS

Figure		Page
1	Cross Section of Element Illustrating Undercut and Slope of Etched Edges.	8
2	Element Edge Etched With RCE, Phosphoric Acid, and Water Solution, 425X (Polaroid).	8
3	Large Grain (Left) and Small Grain (Right) in Different Lots of 0.00045-Inch-Thick (11.43 μ m) Aluminum, 100X (Polaroid)	9
4	Element Edge Spray Etched in 62 Volume Percent Hydrochloric Acid, 33 Volume Percent Phosphoric Acid, and 5 Volume Percent Nitric Acid, 425X (Polaroid).	11
5	Modified Dynamil 3500 Etch Machine.	12
6	Aluminum Particles Remaining Unetched on Mylar Surface, 150X (Polaroid)	14
7	Typical Etched Edge Before (Left) and After (Right) Rework Etching, 425X (Polaroid).	16
8	Aluminum Conductor Edge Etched in 55 Volume Percent Hydrochloric Acid, 27 Volume Percent Phosphoric Acid, 18 Volume Percent Acetic Acid, 0.07 g/l Duponol C, and 0.8 ml/l RCE, 425X (Polaroid).	17
9	Rough, Undercut Etched Edge Caused by Etchant Beyond Its Useful Life, 425X (Polaroid)	18
10	Element Edges Etched With New Etchant (Top Left at 1000X, Top Right at 3000X) and Old Etchant (Bottom Left at 1000X, Bottom Right at 3000X) (Polaroids)	19

SUMMARY

Etching precision patterns in 450- μ inch-thick (11.4 μ m) 1145 aluminum is a primary fabricating operation for development hardware. The etchant¹ used from 1965 to 1974 (5 parts by volume phosphoric acid, 5 parts by volume water, and 1 part volume RCE*) did an acceptable job, but the etched edges of 0.007-inch-wide (0.18 mm) elements were rough. The edge roughness, combined with the measurement systems used, caused element width measurement repeatability to be little better than ± 300 μ inch (± 7 μ m).

One of the goals of precision pattern fabrication is etching elements with a width spread of 50 μ inch (1.27 μ m) across each part. Therefore, a new etchant that can produce smoother element edges was required.

In anticipation of the tighter width tolerance, 80 etchants were investigated during a preliminary etchant development program.³ The most promising ones obtained during this preliminary study were investigated in depth during the current study. The etchant selected contained 65 volume percent phosphoric acid, 30 volume percent hydrochloric acid, and 5 volume percent nitric acid. Although the useful life of this solution is short, the solution was used from April to July 1974 to meet hardware delivery schedules. The edge roughness produced by this etchant was an order of magnitude below that obtained with the previous etchant. Further development to extend the useful life of the etchant changed the etchant composition to 55 volume percent hydrochloric acid, 27 volume percent phosphoric acid, 18 volume percent acetic acid, 50 to 100 mg/l Duponol C** and 0.35 to 1.0 ml/l RCE. This composition, although not ideal, has been used since August 1974 and will continue to be used to etch precision pattern hardware until further development efforts can be completed.

*RCE, Rapid Circuit Etch, 42 Baume FeCl_3 , plus proprietary additives made by Philip A. Hunt, Palisades Park, New Jersey.

**DuPont trademark.

SUMMARY

Etching precision patterns in 450- μ inch-thick (11.4 μ m) 1145 aluminum is a primary fabricating operation for development hardware. The etchant¹ used from 1965 to 1974 (5 parts by volume phosphoric acid, 5 parts by volume water, and 1 part volume RCE*) did an acceptable job, but the etched edges of 0.007-inch-wide (0.18 mm) elements were rough. The edge roughness, combined with the measurement systems used, caused element width measurement repeatability to be little better than ± 300 μ inch (± 7 μ m).

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DISCUSSION

SCOPE AND PURPOSE

The purpose of this effort was to develop an etchant capable of producing smooth edges in 450- μ inch-thick ($11.4\ \mu\text{m}$) 1145 aluminum while minimizing the amount of undercut. The smooth edge requirement is part of an overall program to improve etched element width and measurement repeatability from element to element and from part to part. The goal of the etching and measurement effort is to produce and measure etched elements to a width tolerance of $\pm 25\ \mu\text{inch}$ ($0.63\ \mu\text{m}$), to reduce the variation in conductor width across a part ($W_{\text{max}} - W_{\text{min}}$) to not more than $50\ \mu\text{inch}$ ($1.27\ \mu\text{m}$), and to produce a difference in average element width from part to part ($Av_{\text{max}} - Av_{\text{min}}$) no greater than $100\ \mu\text{inch}$ ($2.54\ \mu\text{m}$).

PRIOR WORK

Detailed characteristics of the solution used to etch development hardware for the last 8 years has been reported.¹ More than 80 different solutions capable of etching aluminum were studied to develop an improved etchant.² The most promising solutions of the 80 candidates were used as the starting point for the present study.

ACTIVITY

The precision pattern development hardware fabricated at Bendix Kansas City includes parts etched from aluminum-Mylar laminates. The base laminate, 450- μ inch-thick ($11.4\ \mu\text{m}$) 1145 aluminum foil, is bonded to a 0.005-inch-thick (0.12 mm) Mylar substrate. In general, a second Mylar layer and a 0.004-inch-thick (0.10 mm) layer of aluminum is bonded to the back of the laminate. Precision elements ranging in nominal width from 0.005 to 0.050 inch (0.12 mm to 1.27 mm) are etched in the 450- μ inch-thick aluminum. A relatively imprecise pattern is etched in the 0.004-inch-thick aluminum.

The criteria used to evaluate etchants are smoothness of etched edges and control of undercut and slope. Undercut is the distance

between the photoresist pattern edge and the etched edge; slope is the angle of the etched edges (Figure 1). An additional criterion requires that no residual aluminum remain in the etched areas of the part. From 1965 to early 1974, an etchant containing 1 part by volume RCE, 5 parts by volume water, and 5 parts by volume phosphoric acid was used to etch aluminum.¹ This etchant produced no significant fumes and could be used for several weeks without replenishment. Although acceptable development hardware was fabricated with this etchant for 8 years, the element edges were relatively ragged (Figure 2). The edge roughness, in conjunction with the measurement system used, resulted in element width measurement repeatability of 300 μ inch (7 μ m). Beginning in 1973, the element width inspection tolerance was changed from 300 μ inch to 25 μ inch (0.63 μ m). As a result of this change, a new etchant capable of producing smoother element edges was required.

In addition to the etchant, the aluminum grain size also significantly influences the etch time, edge roughness, and consequently element width. Figure 3 shows the grain structure of two different lots of aluminum etched in a solution containing 9 volume percent hydrofluoric acid, 13 volume percent hydrochloric acid, and 78 volume percent water (Flick's Etchant) for 2 minutes. The large grain material required up to 10 times as long to etch as the small grain material when the RCE-phosphoric acid-water etchant was used. The resulting elements were narrower and had rougher edges than when small grain material was used. Because development hardware has been restricted to small grain material, the etchant development program was directed toward etching small grain material only.

In anticipation of the tighter width tolerance, a one-year preliminary etchant development program was conducted.² More than 80 solutions capable of etching aluminum were evaluated by etching test panels with solutions made up in beakers. Thirteen of the eighty solutions etched element edges as smooth as or smoother than the RCE-water-phosphoric acid etchant. Eight of the thirteen potential etchants were not pursued because they attacked the preferred positive photoresists. To use these etchants, negative photoresist and negative photomasters would be required. If new positive resists are developed or if negative photomasters are produced, further investigation of these eight etchants will be warranted.

Of the five remaining etchant candidates, one was eliminated because of its slow etch rate. The remaining four were variations of hydrochloric acid, phosphoric acid, nitric acid, and a wetting agent. This combination of materials was used as a starting point to develop an improved etchant.

Figure 1. Cross Section of Element Illustrating Undercut and Slope of Etched Edges

Figure 2. Element Edge Etched with RCE, Phosphoric Acid, and Water Solution, 425X

Figure 3. Large Grain (Left) and Small Grain (Right) in Different Lots of 0.00045-Inch-Thick ($11.43\ \mu\text{m}$) Aluminum, 100X

As with the preliminary investigation, test panels were etched with solutions made up in beakers. Solutions were formulated by incrementing the level of concentrated hydrochloric acid in 5 percent by volume steps from 5 to 100 percent, with the balance of the solution consisting of concentrated phosphoric acid. The best results were obtained from the beaker experiments when the volume of hydrochloric acid was approximately twice the volume of phosphoric acid.

The experiments were scaled up to a Dynamil 50 (Western Technology Associates, Santa Ana, California) spray etch machine operating in a laboratory fume hood. Typical etch solution volume for this machine is 3 to 6 liters. The etchant composition (by volume) giving the smoothest etched edges with a minimum of undercut was 5 percent concentrated nitric acid, 62 percent concentrated hydrochloric acid, and 33 percent concentrated phosphoric acid. The nitric acid was used to passivate the titanium components in the etch machine. Figure 4 is a photomicrograph of an element edge spray etched in this solution; the test panel was small grain material, but this etchant produced equivalent edges with large grain aluminum.

Because the rotating part holding fixture was 12 inches (305 mm) in diameter in the Dynamil 50 etch machine, parts larger than this had to be etched in a larger Dynamil 3500 etch machine (Figure 5) with a 30-inch-diameter (762 mm) part holding fixture operating with a minimum of 40 liters of solution. This machine was modified by adding a sliding exhaust hood to the top of the machine; the hood was necessary to remove the hydrochloric and nitric acid fumes. A 6-inch-diameter (150 mm) exhaust duct was added to the etch chamber but was later changed to a 9-inch-diameter (230 mm) duct connected to the hood because of excessive solution pickup by the exhaust in the chamber.

The modified Dynamil 3500 was used to etch large development hardware, and the Dynamil 50 was used to etch small development hardware from May through August 1974. The etchant used during this time was 5 volume percent nitric acid, 62 volume percent hydrochloric acid, and 33 volume percent phosphoric acid.

When this etchant was used, it became apparent that its ability to etch smooth edged elements lasted only 2 to 3 hours; an average of five parts could be etched before the etchant produced ragged and undercut edges caused by the formation of aqua regia (3 to 1 ratio of hydrochloric acid and nitric acid). The presence of aqua regia was demonstrated by the ability of the etchant to dissolve gold.

To increase the useful life of the solution, the nitric acid was eliminated from the etchant to prevent formation of aqua regia. However, removing the nitric acid allowed the hydrochloric acid to attack the titanium components in the etch

Figure 4. Element Edge Spray
Etched in 62 Volume
Percent Hydrochloric
Acid, 33 Volume
Percent Phosphoric
Acid, and 5 Volume
Percent Nitric Acid,
425X

machine. The amount of titanium, therefore, was reduced by removing the heating and cooling coils. The titanium pump shafts, however, were retained because their thickness permitted them to endure the etchant longer. The heaters were not replaced because it is not necessary to heat the etchant. The solution tends to heat up while being used, so a Teflon cooling coil was added to the Dynamil 3500 machine and a glass coil was added to the Dynamil 50. Chilled water is circulated through the coils to maintain the etch solution between 19 and 25°C.

Figure 5. Modified Dynamil 3500 Etch Machine

The spray nozzle on the Dynamil 3500 was also moved closer to the part holding fixture, which increased the spray density at the part from approximately $0.8 \text{ ml/cm}^2 \cdot \text{s}$ to approximately $2 \text{ ml/cm}^2 \cdot \text{s}$. The etchant density for the Dynamil 50 was $14 \text{ ml/cm}^2 \cdot \text{s}$, accounting at least in part for an etch rate 2 to 3 times slower for the Dynamil 3500 than for the Dynamil 50.

Removing the nitric acid from the etchant gave poorer etching results, although they were better than those of the old etchant. The etched edges were slightly more undercut and wavier. The nitric acid was retained, therefore, in new batches of etchants made up. Meanwhile, study continued with an effort to find a replacement for nitric acid.

One of the first replacement additives tried was DuPonol C (E. I. du Pont de Nemours and Company Inc., Dyes and Chemicals Division, 7 South Dearborn Street, Chicago, Illinois 60603), a wetting agent (sodium lauryl sulfate). Small amounts (0.25 g/l) lengthened etch times and reduced, or virtually eliminated, undercut. Two other wetting agents, Petrowet R (E. I. du Pont de Nemours and Company Inc., Dyes and Chemicals Division, 7 South Dearborn Street, Chicago, Illinois 60603) and Aerosol OT (American Cyanamid Company), were evaluated but did not work as well as DuPonol C.

A solution of 65 volume percent hydrochloric acid, 35 volume percent phosphoric acid, and 0.25 g/l Duponol C was used in the spray etch machines with good results: the parts were high quality, but quantities of aluminum particles about 0.001-inch -diameter ($25 \mu\text{m}$) and smaller remained unetched on the Mylar substrates (Figure 6). These particles appeared as star clusters under normal microscopic examination and were therefore termed "stardust."

The effect of specific amounts of these aluminum particles on the development hardware function was unknown. Although it might have been possible to set a maximum size and density limit for residual aluminum particles, the inspection procedure would have been tedious and subjective. Because of this, coupled with the uncertainty associated with the particles, the development effort was directed toward eliminating them.

Previous experience had shown that additions of ferric chloride increased the etch rate significantly. A small amount of liquid RCE, ferric chloride, added to the etchant removed the aluminum particles during etching. In concentrations up to approximately 0.1 ml/l of RCE, corresponding to about 0.02 g/l of iron, the etched edges were smooth with little undercut. With this concentration, the first two or three parts etched in a newly made solution had few or no aluminum particles. As additional parts

Figure 6. Aluminum Particles
Remaining Unetched on
Mylar Surface, 150X

were etched, the density and size of the particles increased. As the concentration of iron was increased, the etched edges became more undercut and ragged. Etch time also decreased as the concentration of iron was increased from 200 seconds at 80°F (26.7°C) with no RCE in the etchant to 140 seconds at 70°F (21.1°C) with 0.05 ml/l of RCE, and 90 seconds at 70°F (21.1°C) with 0.25 ml/l of RCE. Such faster etch times were undesirable because as the etch rate increased the etch time became more critical. Because of the minute quality of RCE necessary to the etchant, small amounts of iron from sources other than RCE might have affected the etchant. The small quantity of iron would also have made it difficult to control the iron concentration. Formulations containing hydrochloric acid, phosphoric acid, RCE, and Duponol C were used to etch development hardware during August 1974. The etched edges were essentially the same as those shown in Figure 4.

Parts showing the unetched aluminum particles were reworked by pouring approximately 150 ml of the old etch solution (RCE, water, and phosphoric acid) into the concave side of the part and swirling the heated (40°C) etchant over the surface part for 30 to 60 seconds, depending on the size and concentration of the particles. This technique was effective in removing the residual aluminum without significantly increasing the edge roughness. Figure 7 shows a typical edge before and after rework etching.

The addition of glacial acetic acid to the etchant, suggested by LLL personnel, slowed the etch rate and increased the tolerance of the etchant for ferric chloride. It also appeared that the exact makeup of the etchant was not as critical when it contained acetic acid. The following composition has been used since August 1974 for etching development hardware: 55 volume percent hydrochloric acid, 27 volume percent phosphoric acid, 18 volume percent glacial acetic acid, 0.05 to 0.1 g/l Duponol C, and 0.35 to 1.0 ml/l RCE. The concentration of acetic acid can be increased by 50 percent or more, with a corresponding increase in ferric chloride, without significantly affecting the etch results. With this etch solution, the 450- μ inch-thick (11.4 μ m) aluminum etched in 150 to 200 seconds at 75°F (23.9°C) in the Dynamil 50 etch machine (Figure 8).

The useful life of a new etch solution usually followed this pattern: three to four parts were etched with no residual aluminum particles; the next three to four parts had good element edges and widths but the quantity of particles increased, however these parts were usually capable of being reworked; the following parts had more aluminum particles with increasingly narrow elements and excessively rough edges which prevented rework of these parts (Figure 9).

However, this etch pattern was not consistent in a given etch machine or between the two etch machines. Some solutions were bad when made up--that is, the first part etched was unacceptable--while other solutions etched 16 to 18 acceptable parts.

Apparently, this variability was related in part to the fume removal rate: the faster the fume removal rate, the lower the useful life of the etchant. Fumes were continuously removed from the interior of the Dynamil 3500 through the 9-inch-diameter (230 mm) exhaust duct in the added hood. The Dynamil 50, however, was small enough to be placed in a laboratory fume hood, so that only exterior fumes are removed. Dynamil 50 etch solutions retained their etch quality for 2 days or more whereas the same composition in the Dynamil 3500 produced acceptable parts for only about 2 hours. When more air flow was added to the interior of the Dynamil 50, the useful life of the solution was reduced to about 2 hours. Repeating the same test but substituting nitrogen

Figure 7. Typical Etched Edge Before (Left) and After (Right) Rework Etching, 425X

Figure 8. Aluminum Conductor
Edge Etched in 55
Volume Percent Hydro-
chloric Acid, 27 Volume
Percent Phosphoric
Acid, 18 Volume Percent
Acetic Acid, 0.07 g/l
Duponol C, and 0.8 ml/l
RCE, 425X

and then argon gave essentially the same results as those obtained with air.

The comparative etchant material cost of etching the 0.00045-inch-thick ($11.43\ \mu\text{m}$) aluminum with the old etchant (RCE, water, and phosphoric acid) was \$1.00 per part. Using the Dynamil 3500 and the new etchant, the cost is \$12.00 per part. Using the Dynamil 50 and the new etchant, the cost is \$1.50 per part.

Figure 9. Rough Undercut Etched Edge Caused by Etchant Beyond Its Useful Life, 425X

ACCOMPLISHMENTS

An improved etchant has been formulated for etching the 450- μ inch-thick (11.4 μ m) aluminum used for precision pattern hardware. Etched edges were smooth with little undercut, and the edge slope was almost vertical. Figure 10 shows scanning electron photomicrographs comparing the new etchant (used since August 1974) and the old etchant (used from 1965 to 1974). The new etchant permitted closer width control of etched elements. Because edges of elements were smoother, there was less uncertainty as to the exact edge location, making element measurements more meaningful than they previously were. Other assets of the new etchant were that it etched at a reasonably uniform rate over the surface of the part and that lengthening the etch time did not appreciably affect the undercut.

0 0.001 INCH

0 0.0005 INCH

Figure 10. Element Edges Etched With New Etchant (Top Left at 1000X, Top Right at 3000X) and Old Etchant (Bottom Left at 1000X, Bottom Right at 3000X)

FUTURE WORK

Further etchant development will be conducted to eliminate residual aluminum particles. The 1145 aluminum will be metallurgically evaluated to establish the cause of the residuals.

The etchant will be chemically analyzed in detail to establish the cause of etchant degradation, and the development effort will be directed toward increasing the effective life of the etch solution. When an etchant with a long useful life is developed, analysis and control procedures will be developed to maintain the etchant at an effective operating level.

A multiple-nozzle spray head will be installed in the Dynamil 3500 to increase the spray density at the part. Solution flow rate and pressure, as well as nozzle configuration, will be examined in detail.

Etch solution development will be conducted on physical vapor deposited aluminum.

Caustic etchants in conjunction with negative photoresists will be examined.

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