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**Title: Effects of Copper Foil Type and Surface Preparation on Fine Line Image Transfer in Primary Imaging of Printed Wiring Boards**

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**Abstract**

A laboratory study compares fine line print & etch (P/E) and pattern plate performance on base materials with a variety of copper foils and surface preparations. Foil types include fine grain copper, double treat copper, reverse treated foil, vendor copper, and electroless copper surfaces. Surface preparation methods include alumina jet cleaning, pumice scrubbing, brush cleaning, and chemical cleaning. Surfaces are characterized by contact profilometry, off-contact optical profilometry, Optically Stimulated Electron Emission (OSEE), and water break test. Evaluation criteria are instant adhesion (tape test), P/E minimum line and space resolution, minimum isolated line held, and (AOI) yields on a 75 micron test pattern. Furthermore, pattern plate performance is judged by degree of underplating, minimum developed space, and resolution. Limitations of such a laboratory study are pointed out and results are compared to production experience.



**Biographical Information**  
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## 1. Introduction

Laminate construction, chemical composition of the copper foil surface and its topography affect dry film photoresist adhesion, conformation, and, ultimately PWB (Printed Wiring Board) yields. This area has been studied extensively over the years (Ref. 1-5). Substrate weave patterns, foil manufacturing process, and surface preparation all have been found to contribute to the macro and micro-topography of the surface, which will determine the contact area, or anchoring points for the dry film resist, depending on lamination conditions and flow characteristics of the resist. The final chemical composition of the copper surface before lamination will influence wetting behavior and the magnitude of the adhesive forces between the resist and the foil for a given contact area. If mechanical or chemical forces at the film/copper interface exceed adhesion and mechanical interlocking forces, the dry film will lift ("break down"). The study of Ref. 1 examined the net effect of these forces on PWB yields. While it gained valuable insight, the study had inherent limitations and has lost some relevance over the years due to the advent of new foil constructions, new surface preparation methods, improved analytical techniques to characterize surfaces, and a shift to aqueous processable film compositions. Inherent limitations of the study included the use of semi-aqueous processable dry film, "chemical cleaning" being represented only by hydrogen peroxide/ sulfuric acid, and a lack of applying the rigor of statistical significance tests to the data. In addition, compared to the relatively simple print & etch process study, pattern plate yield data reflect not only the contribution of copper surface characteristics to yield, but to a large extent the process noise of a large number of individual steps contributing to yield variations, even though great care is being exercised in controlling these process parameters.

The objective of this study then was to update the earlier work in light of pertinent changes in materials, processes, and analytical techniques in recent years.

## 2. Background

Let us now look at some of the more important developments of recent years that have impacted dry film performance as it relates to resist/ copper surface interactions.

Driven by environmental concerns, the conversion of solvent and semi-aqueous processable resists to fully aqueous processable photoresists has accelerated in recent years in all regions of the world and is practically complete. These aqueous processable resists tend to be less robust to a variety of chemical attacks, especially aggressive plating chemicals, which deteriorate adhesion. This includes the effect of localized high pH due to hydrogen evolution in low efficiency plating baths, and adhesion interferences from trace organics accumulating in recycled water. Thus, concern about resist adhesion to a variety of surfaces has become a greater issue.

We are also witnessing changes in surface preparation and substrate construction which are driven by cost pressures and the need to establish the capability to handle and clean thin cores, e.g. 50-75 microns (2-3 mil) dielectric with 17 microns (1/2 ounce) copper, without damage or distortion. New analytical tools such as OSEE (Optically Stimulated Electron Emission), ISS (Ion Scattering Spectroscopy), SIMS (Secondary Ion Mass Spectrometry), SERA (Sequential Electrochemical Reduction Analysis), and Interferometry (Ref. 3, 6-9) as well as traditional measurement techniques (Ref. 10) are available to characterize new cleaning processes and surfaces.

To avoid dimensional distortion of thin cores, mechanical surface preparation methods are being avoided in favor of chemical cleaning (microetch, or acid clean/ microetch combinations) or no-clean options. Single step chemical cleaners ( Ref. 11) are being offered. Reverse current cleaning methods are being introduced to remove chromate with a minimum of copper while maintaining dimensional stability of thin cores (Ref. 12). No-clean options include double treat copper (Ref. 13), "reverse treated foils" (Ref. 14-16), and foils with aluminum carrier/ protector sheets (Ref. 17), or combinations thereof.

On the other hand, we are noticing the growing popularity of aluminum oxide jet and brush scrubbing (Ref. 18-20) on thicker boards, apparently driven by cost and productivity considerations.

Low profile, fine grain foils (Ref. 21) and non-woven dielectric constructions such as layers of parallel glass fiber alternating in direction (Ref. 22) or aramid non-wovens offer the promise of better conformation of thin resists to planar foil surfaces.

Let us now look at some of these developments in more detail:

## 2.1 Double-Treat (DT) Copper

We have noticed increased popularity of double-treat foil in the US and Japan. A Japanese supplier is reporting increased use of his 18 micron DT foil on innerlayers. Several reasons have been reported:

- The price differential between double-treat and standard foil appears to be offset by true value-in-use, at least in certain applications.
- DT does not require the application of a multilayer bonder and avoids the potential of pink ring defects. On very thin foils the conversion of a substantial amount of copper to copper oxide for use as a bonder may not be acceptable.
- No prelamination surface preparation is required, resulting in cost savings. More importantly, as innerlayer cores become very thin, brush or pumice scrubbing can cause unacceptable distortion.

On the other hand, double-treat surfaces do present some problems. Double-treat foils are not necessarily compatible with wet lamination. Wetting agents can overcome these problems. Other concerns: difficult AOI recognition in the reflectance mode, tendency for etch retardation with alkaline etchants, "scuffing" of the surface, staining or lock-on with extended hold times, and stripping residues with some photoresists.

## 2.2 Reverse Treated Foils (RTF)

Reverse Treated Foils have entered the market under names such as "Drum Side Treated Foil" (DSTF) (Ref. 14 & 15) and MLS foil (Ref. 16). As ED (electro-deposited) foil is formed on a revolving cathodic drum, it typically has a smooth side facing the drum and a rough side facing the copper solution. In the RTF process, the foil receives a zinc treatment only on the smooth side which is laminated to the dielectric. The rough, non-treated side, faces the dry film. Because of its surface roughness, no mechanical or chemical roughening step is needed for dry film adhesion. However, since the roughness ( $R_a > 0.3$  micron) exceeds optimal topography for dry hot roll lamination of photoresist films, best results are achieved with wet lamination and/or higher lamination temperatures. The omission of an acid cleaning step for tarnish and chromate removal is somewhat controversial: careful yield studies are needed to define the trade-offs, and multilayer oxide bonder formation may suffer as a result.

## 2.3 Fine Grain Low Profile Foil and Non-Woven Dielectrics

The need for impedance control and fine line etching has accelerated the development of so called "low profile copper foils" which often have a fine grain structure. "Low profile" in this context refers to the smoother, more uniform topography of the copper foil side facing the dielectric. Suppliers of fine grain foils have introduced such foils under acronyms such as SQ-VLP, JTCAM, and VLP (very low profile). SQ-VLP is reported to yield a better etch factor. DT-VLP is a fine grain innerlayer foil. Fine grain foils seem to yield a more uniform, high surface area, planar topography, especially when combined with non-woven dielectric constructions. This development will accommodate surface mount needs as well as provide good conformation of thin resists for high resolution and etch uniformity. Reported yield improvements do not appear to be directly related to the grain structure, but more so to the overall quality of the laminate and shorter etcher residence times.

## 2.4 Very Thin Foils

For "ultra thin copper" (UTC) for MCMs and BGA's, minimal copper removal during surface preparation is an issue. A typical ultra thin foil thickness is 12 microns. Electrochemical (reverse current) cleaning systems may be viable solutions to removing chromate conversion coatings with only minimal copper removal. The use of 12 microns double-treat foil without surface preparation would be a viable alternative. A major manufacturer of foils can supply such material in developmental quantities.

To facilitate the handling of very thin foil, and to eliminate surface cleaning and to not contaminate the copper surface with epoxy spots, thin foils are offered as more rigid sandwiches of copper/aluminum/copper or copper/aluminum (Ref. 17).

## 2.5 Aluminum Oxide Jet or Brush Cleaning

The growing popularity of the aluminum oxide surface preparation process, driven by cost and productivity considerations, was mentioned above. Al<sub>2</sub>O<sub>3</sub> jet scrubbing was first introduced in Japan (Ref. 18), then a domestic US supplier of comparable equipment entered the field (Ref. 19), and then Al<sub>2</sub>O<sub>3</sub> brush scrubbers were offered (Ref. 20). This process is not to be confused with pumice cleaning. Compared to pumice, there are some important differences:

Aluminum oxide does not disintegrate to fines as rapidly as pumice does. As judged by the particle size distribution, aluminum oxide lasts much longer. There is less maintenance, downtime, and sludge disposal. On the other hand, aluminum oxide particles wear "round", i.e. with time the particles become smoother, "peening" the copper, and creating a smoother surface which is detrimental for dry film adhesion. Thus, aluminum oxide needs to be replaced before fine particle build-up suggests replacement is due. Suppliers of aluminum oxide equipment don't seem to have hard and fast rules, based on data, when it comes to aluminum oxide replenishment frequencies. Particle size and grade of aluminum oxide are also variables with evolving specifications. According to the study of Ref. 20, brushing of aluminum oxide gives a rougher, more preferred surface than jetting. There are also data indicating that jetting distorts (elongates) thin copper foil more than brushing. If jet pressure is lowered to counteract this phenomenon, there may be a problem with insufficient surface roughening due to reduced particle impact.

### 3. Test Design

In view of the above mentioned developments, the following test parameters were selected:

- A fully aqueous processable dry film resist, approx. 40 microns (1.5 mil) thick, suitable for print & etch as well as pattern plating ( henceforth referred to as "film").

Copper Surfaces:

- Vendor copper, prepared in four different ways: chemical clean (Fig. 1), brush scrub (Fig. 2), hand pumiced (grade: 3F; Fig. 3), and "as is" (no preclean, Fig. 4).
- Double-treat copper, no preclean (Fig. 5).
- Fine grain foil, chemically cleaned (Fig. 6); for reference, see fine grain foil, no clean (Fig. 7)
- Reverse treated foil, acid dip (10% sulfuric acid, one minute, room temperature, rinse, dry; Fig. 8); for reference, see reverse treated foil "as is" (Fig. 9)
- Electroless copper, aluminum oxide jet scrubbed, acid dip, rinse, dry (Fig. 10).
- Electroless copper, post-electroless rinse sequence, dry, brush scrub (Fig. 11); for reference, see electroless copper, no clean (Fig. 12).

Chemical cleaning consisted of the following conveyORIZED spray cleaning sequence: alkaline cleaner ( 20% v/v; 30 seconds, 50°C, 1.5 bar spray pressure), rinse, microetch ( mono-persulfate 150 g/l; 50 g/l sulfuric acid; 0-22 g/l copper; 30 seconds, 30°C, 1.5 bar spray pressure), rinse, dry.

Brush scrubbing was done with a 320 grit bristle brush, 7 mm "foot print".

- Lamination: dry lamination, manual laminator, 105°C roll temperature, lamination speed 1.0 m/ min.
- Exposure: manual exposure unit, 5 kW UV lamp, low degree of collimation, good vacuum contact (frame: Mylar® top, glass bottom), 14 RST (Riston® Steps) held.
- Development: 0.85% sodium carbonate, 30°C, 1.5 bar spray pressure, 60-65% breakpoint (wash-off point), rinse, dry.
- Etch: conveyORIZED spray cupric chloride etch, 50°C, oscillating cone nozzles, 1.5 bar spray pressure, 510-530 ORP control, 2.4 N normality, 1.3 Sp. Gr., 170 g/l copper.
- Plating: hot soak acid preplate clean, mono-persulfate microetch, standard high-throw acid copper plating cycle, followed by tin/ lead plating ( methanesulfonic acid pre-dip).
- Stripping: conveyORIZED spray stripping, 50°C, 1.5-2 bar spray pressure, 3.0% NaOH plus 1% monoethanol amine (MEA), resist loading up to 0.6 mil-m<sup>2</sup>/liter (25 mil-sqft/ gal), rinse, dry.

Panels were assigned computer generated random numbers for the processing sequence through lamination, exposure, and development.

#### **4. Surface Characterization**

Prior to dry film lamination, the prepared copper surfaces were characterized by the following techniques:

- Water Break Test
- Surface Profilometry
- Interferometry (non-contact)
- Optically Stimulated Electron Emission (OSEE)

##### **4.1 Water Break Test**

The panel (copper surface) is completely wetted with water to form a contiguous sheet of water on the surface. The panel is then held vertically and the time elapsed for the sheet to break up into beads of water is recorded. The longer it takes for the water to break, the "cleaner" the surface is deemed. A water break test gives a number ("seconds held"). The water break time can be used for SPC to track the removal of hydrophobic (water repelling) organics (typical specification: 30 seconds minimum). The test will not be useful to detect hydrophilic organics such as wetting agents or inorganic impurities.

##### **4.2 Surface Profilometry (Ref. 23)**

The surface roughness was characterized by sub-micron stylus profiling with a Dektak<sup>3</sup>ST Surface Profile Measuring System, Veeco Sloan Technology. Conditions: Stylus tip radius: 0.7-0.8 micron; scan length 1000 microns; cut-off filter: 50 microns; scan speed: low (50 seconds total); smoothing: none; stylus force 10 mg. An earlier study (Ref. 24) had shown some promising empirical correlations between  $R_a$ ,  $R_z$  values, brushing variables such as cutting speed, conveyor speed, oscillation frequency, dry film adhesion, and PWB yields. "Good" values for  $R_a$  (0.2-0.3 micron) and for  $R_z$  (2-3 micron) were used for guidance. However, the study had not been extended to non-brushed surfaces, and the stylus had been coarser. The inclusion of the 50 microns cut-off filter also reduces the reported numbers because it effectively removes the weave component from the reported  $R_a$  and  $R_z$  values.

##### **4.3 Optical Surface Profilometry (Interferometry)**

This non-contact technique is based on a combination of a new technique for vertical-scanning, white light interferometry with traditional phase-shifting interferometric techniques (Ref. 25). The instrument used was a RST Plus, Surface Measurement System, WYKO Corporation (Ref. 6, Fig. 13). In addition to the traditional surface roughness parameters obtained by contact profilometry (Fig. 14), this method can produce 3-dimensional images (Fig. 15) contour plots (Fig. 16), and a surface area parameter ("Surface Area Index") which is a good indicator of the total true surface area. Since surface area translates into film contact area, within a reasonable roughness range, we were expecting that this surface area index would correlate better with dry film adhesion than  $R_z$  and  $R_a$  values which we have used in the past.

##### **4.4 Optically Stimulated Electron Emission (OSEE) (Ref. 26)**

When applying this technique, the surface is bombarded with high energy ultraviolet radiation, causing photo emission of electrons from the surface which register as a photoelectric current in a detector. Clean copper gives a strong signal, while most unwanted surface impurities attenuate (lower) the signal (Fig. 17). This way, organics and heavy oxides are detected. We have used this technique to follow the reoxidization of cleaned copper surfaces with time. A study (Ref. 27) showed a good correlation between OSEE results and the more costly ESCA (XPS) surface analysis. The same study concluded that OSEE also serves as a good indicator for the removal of vendor stain-proof coatings as evidenced by XPS correlation. Our own earlier studies demonstrated that clean vendor copper, after removal of chromate conversion coatings, produced OSEE values above approximately 700 count. The instrument in use was an OP-1020 (Ref. 28, Fig. 18).

#### **5. Responses**

The effect of copper surface differences on dry film performance, i. e. the response, was evaluated by the following criteria:

- "Instant adhesion"
- Resolution after development, as judged by
  - a) Minimum isolated line remaining (surviving development)
  - b) Minimum line/ space resolution
- Degree of Resist Lifting in Pattern Plating
- Yield (Print & Etch; AOI Data)

### **5.1 "Instant Adhesion"**

This "instant adhesion" test is a pull (peel off) test performed at different time intervals after resist lamination (0, 15, 30, 60 minutes, and 24 hours). An adhesive tape is pressed against the laminated resist, the tape is pulled off, and the area % of the resist remaining on the copper surface is reported. There is no pre-scoring or cross-hatching of the resist before applying the adhesive test, as it is practiced in other standard adhesion tests; e. g., in the paint industry.

### **5.2 Resolution after Development**

Resolution after development was judged by the smallest lines and spaces resolved, and the smallest, isolated line "held". A multiple pitch pattern with 20 to 200 micron lines and spaces, isolated and nested (paired) lines, in a print & etch version and the corresponding pattern plate version, served as a test vehicle.

### **5.3 Degree of Resist Lifting in Pattern Plating**

Degree of resist lifting was measured by inspecting pattern plated boards after copper plating, but before resist stripping, with a high power microscope. The width of the observed color change under the lifted resist was recorded in microns. It had been observed that the degree of lifting on a given board varied with the dimension of the resist feature, not the size of the adjacent copper area. Therefore we measured lifting on the edge of a well defined large rectangular resist area. Three measurements from each of six test boards were averaged.

### **5.4 Yield**

Print & etch yields were determined by Automatic Optical Inspection (AOI) (Ref. 29). Defects called out by the AOI are visually inspected, verified, recorded as opens, shorts, space and line width violations, and downloaded to a PC. Yield analysis software then allows one to eliminate certain types of defects from the calculation which are not of direct interest to the study. For example, "repeat defects" which have to be associated with the imaging step (e.g. phototool flaw, trapped dirt, etc) are irrelevant to a surface study and dilute defects pertinent to surface characteristics. Likewise, "short" defects in a print & etch study, don't speak to film adhesion and conformation issues, however "opens" do. So we selected random "opens" as the yield indicator for the purpose of this study.

The test vehicle was a single pitch, 75 micron (3 mil) line and space pattern with "one-up" repeat patterns of nested pair conductors, alternating in direction. The "one-ups" consist of a 2 X 2 inch (5 X 5 cm) pattern, repeated in an 8 X 10 array which covers a 16 X 20-inch (approx. 40 X 50 cm) total area with 36,000 inches (0.6 miles or 1km) of circuitry. Two test patterns were used, which were mirror images of each other: one for the panel front side and one for the back side. Five boards were processed for each test condition.

When a test pattern is used, there arises some difficulty in defining yield. With real boards (at least a 1-UP real board), it is simple. A real board either has no defects, and is "good", or contains one or more defects, and is therefore "bad". The yield fraction is the number of good boards divided by the total number of boards made.

Because a test pattern is not a "real" board, defining yield becomes more complex. First, the test pattern may contain conductor length equivalent to that of 5-10 real boards. Our test pattern does not contain all of the features present on a real board, and it contains conductors and spacing finer than most real boards today. The best one can do

is to measure the total defect count, divide by the total conductor lineage to find defects per unit length, then calculate the average number of defects per panel for a hypothetical "real" panel by multiplying defects per unit length (from the test vehicle) times the conductor length of the real panel. Finally, one can then estimate a yield from average defects per hypothetical panel. In our case, raw "yield" data are reported in "DEMI's" ( defects per million inches of circuitry; 1 million inches equal about 16 miles or 25 km). The test pattern has 36,000 inches ( 0.6 miles or 1 km) of circuitry on each side. The space length is half the length of the lines. For reference, a typical high density 75 micron line & space production panel might have 5,000 inches (125 m) of circuitry.

## **6. Statistical Significance Tests (Ref. 30)**

Are print & etch yield results truly different for the copper foils and surface preparations tested? To answer this question, several variations of "significance tests" based on either the binomial distribution or the Poisson distribution were applied. AOI yield data are attribute data. This means that the common "Student t" significance test, applicable to measurement data with a "normal" distribution, is not applicable here. Significance tests are used to determine the probability that the observed differences in the data are due to chance alone. If the probability is high that the observed difference is due to chance alone, it is pretty safe to conclude that there is no real (significant) difference. If, on the other hand, the probability of getting the observed difference is very remote or unlikely, then it is fairly safe to conclude that there really is a difference between the two groups. An important caveat needs to be kept in mind: once a statistically significant difference between several populations has been calculated, statistics really don't guarantee that the cause for the difference is the variable (foil/ surface) which we deliberately varied. There is always the outside chance that another significant factor varied without our knowledge and that this unknown factor is the true cause of the observed difference.

The significance tests applied in this study were the Chi Square Test, the 50% Probability Chi Square Test, and the "zI" Test ("z" test for "Instances"). The Chi Square Test served as a screening test to tell us that there was indeed a significant difference somewhere among the populations (test conditions). Where parent populations (number of boards) per condition were the same, the 50% Chi Square test was used to first probe for differences between pairs of sets (e.g. fine grain foil vs. double-treat), and then across more than two sets (e.g. fine grain vs. double-treat vs. reverse treated foil). In the case of dissimilar parent populations (e.g. different number of boards per condition), the "zI" test was applied.

### Chi Square Test for More than Two Samples

The Chi Square test is a method for testing more than two samples. It was applied to AOI yield data from nine different (foil/ surface) conditions with unequal parent populations (number of panels). This test tells whether there is a statistically significant difference among the nine conditions but it will not tell which ones are different.

### 50% Probability and 50% Chi Square Tests

Generally, we run 5 panels per condition, resulting in parent populations that are equal. Since we run all the panels at the same time, our assumption is that aside from normal error any differences we observe should be due chance alone. We assume that, all things being equal, each group should have roughly the same number of defects. This is the null hypothesis. Significance tests are based on this assumption that any difference detected is due to chance alone. The probability of this being the case is then determined using the Binomial Formula or Poisson distribution. If our analysis tells us that the probability of the observed difference being due to chance alone is very unlikely, we reject the null hypothesis and conclude that there is indeed a real difference between the two groups. When two groups are made up of equal numbers of things (boards), the 50% probability test can be used. This is the condition we have when we test 5 panels for each copper surface. Although we try to run an equal number of panels for each condition, there are times where 1 or more of the panels is damaged and not included in the AOI analysis. Now we have a case where the parent groups are of different sizes and we cannot use the 50% Probability Test. In this case we use the "zI Test".

### The zI Test

Without going into a lot of detail, the assumption is made that there is no difference between the samples. A value, “z”, is calculated and compared to a table of critical values. The “z” value obtained from the calculation is the difference between the two samples expressed in terms of standard deviations. As “z” becomes larger there is less and less chance that our assumption of the two samples being the same is correct and we can then confidently conclude that the samples are really different.

## 7. Results, Discussion of Findings, and Search for Correlations

### 7.1 Surface Characterization Results

#### 7.1.1 Ranking of Surfaces by Water Break Test

Fine Grain Foil, no cleaning:	< 1 second
Vendor Copper, no cleaning:	3 seconds
All others:	• 30 seconds

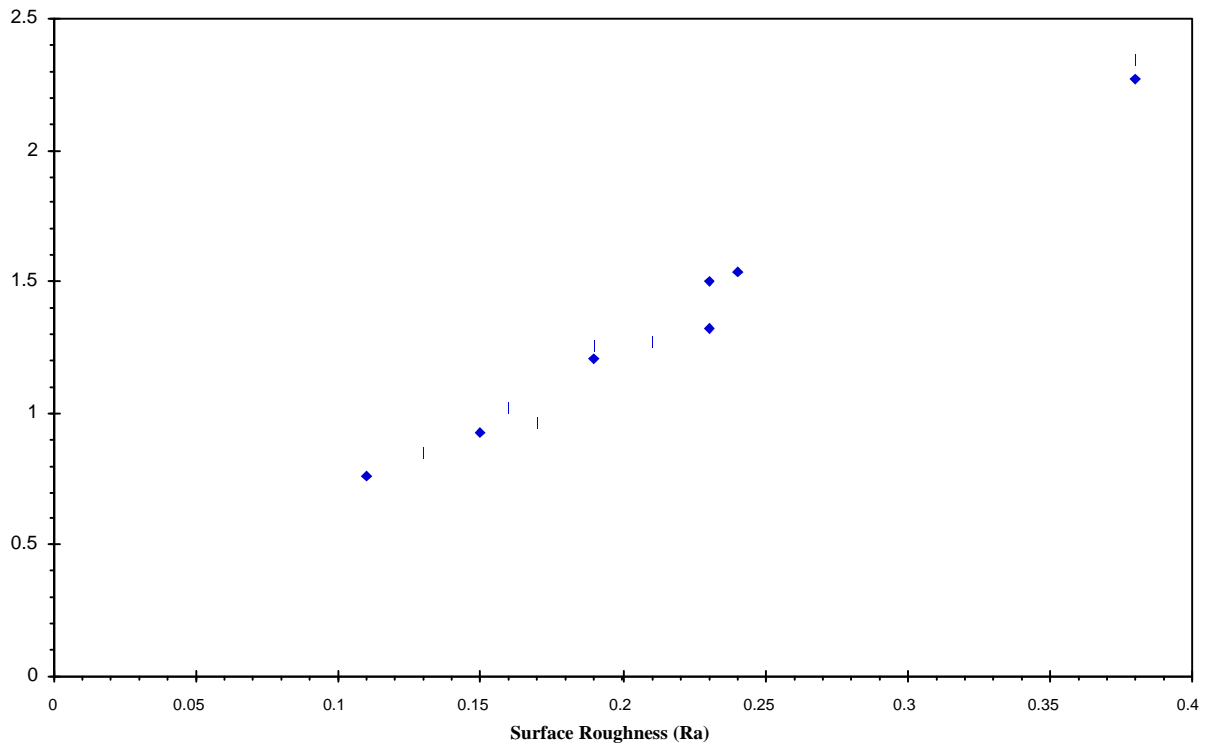
The expectation was that cleaned surfaces and virgin double-treat surfaces would pass our empirical specification of • 30 seconds. "Uncleaned" surfaces may or may not pass the specification depending on the handling history. Therefore it is no surprise to find unacceptably low numbers with some, but not all uncleaned boards.

#### 7.1.2 Ranking by $R_a$ from Surface (Contact) Profilometry

<u>Surface</u>	<u><math>R_a</math> (microns)</u>
Electroless Copper, Al <sub>2</sub> O <sub>3</sub> scrubbed, acid dip	0.11
Fine Grain Foil, no cleaning	0.13
Vendor Copper, brush scrubbed	0.15
Electroless Copper, brush scrubbed	0.16
Fine Grain Foil, chemical clean	0.17
Electroless Copper, Al <sub>2</sub> O <sub>3</sub> jet scrubbed	0.19
Electroless Copper, no cleaning	0.19
Vendor copper, chemical clean	0.21
Double-Treat Copper, no clean	0.23
Vendor Copper, no clean	0.23
Vendor Copper, hand pumice	0.24
Reverse Treated Foil, no clean	0.38
Reverse Treated Foil, acid dip	0.38

We had expanded the empirical "good"  $R_a$  range from 0.2-0.3 micron to 0.15-0.3 micron to reflect experience with non-brush scrub methods and minor changes in the test method. As expected, most of the measured  $R_a$  values fell in that range, with fine grain foil and aluminum oxide scrubbed surfaces at the lower end and pumice scrubbed surfaces at the high end of the range. Also as expected, the reverse treated foil showed the highest  $R_a$  values which confirms that special lamination conditions, e.g. high lamination temperature or wet lamination, are likely to give the best dry film conformation results.

Historically we had collected some evidence suggesting a better correlation between resist adhesion performance and  $R_z$  values than with  $R_a$  values. However, no firm data existed. Plotting profilometry  $R_a$  vs.  $R_z$  (Fig. 19) readily showed a strong correlation between these two surface roughness parameters within the range measured, so that we felt comfortable proceeding with  $R_a$  data only for further analysis.



**Fig. 19:  $R_z$  vs.  $R_a$  by Contact Profilometry**

### 7.1.3 Ranking by $R_a$ from Non-Contact Surface Profilometry (Interferometry)

<u>Surface</u>	<u>Ra (microns)</u>
Electroless Copper, no cleaning	0.16
Vendor Copper, 320 grit brush scrub	0.19
Vendor Copper, chemical clean	0.25
Electroless Copper, Al <sub>2</sub> O <sub>3</sub> scrubbed, acid dip	0.26
Fine Grain Foil, no cleaning	0.27
Electroless Copper, Al <sub>2</sub> O <sub>3</sub> scrubbed, no cleaning	0.30
Fine Grain Foil, chemical clean	0.32
Electroless Copper, 320 grit brush scrub	0.39
Vendor Copper, no Cleaning	0.40
Vendor Copper, hand pumice	0.40
Double-Treat Copper, no cleaning	0.45
Reverse Treated Foil, no cleaning	0.95
Reverse Treated Foil, acid dip	0.95

As one notices, the  $R_a$  values measured by interferometry give about the same ranking sequence for surface roughness as the contact profilometry, however, the absolute values are approximately twice as high. The reason for the difference lies in the fact that the non-contact profilometry did not use a low frequency cut-off filter to remove the contribution of the surface waviness to the roughness factor. The scan mode employed a "low pass" filter.

#### 7.1.4 Ranking by Surface Area Index from Interferometry

<u>Surface</u>	<u>Surface Area Index</u>
Vendor Copper, brush scrubbed	1.01
Fine Grain Foil, no clean	1.01
Electroless Copper, Al <sub>2</sub> O <sub>3</sub> jet scrubbed, acid dip	1.01
Electroless Copper, brush scrubbed	1.01
Vendor Copper, no clean	1.02
Vendor Copper, chemical clean	1.02
Fine Grain Foil, chemical clean	1.02
Electroless Copper, Al <sub>2</sub> O <sub>3</sub> jet scrubbed	1.02
Electroless Copper, no clean	1.02
Vendor Copper, hand pumiced	1.03
Double-Treat Foil, no clean	1.06
Reverse Treated Foil, acid dip	1.17
Reverse Treated Foil, no clean	1.18

The "surface area index" computed by the interferometry software is a good indicator of the true, microscopic surface area of the sample. As such, it should be a meaningful indicator of the magnitude of available anchoring points for the resist, within certain limits, of course, imposed by the conformation characteristics of the dry film. The index is a dimensionless number. I=1 for a mirrorlike surface. Again, reverse treated foil shows the highest number. Most other surfaces have an index of 1.01 to 1.02 with the exception of pumiced copper (1.03) and double-treated copper (1.06), both surfaces that offer good dry film adhesion.

#### 7.1.5 Ranking by OSEE Count

<u>Surface</u>	<u>OP-1020 Reading (Counts)</u>
Fine Grain Foil, no cleaning	125
Reverse Treated Foil, acid dip (stained after dip)	173
Double-treat, no cleaning	195
Electroless Copper, no cleaning	253
Reverse Treated Foil, no cleaning	266
Vendor Copper, no cleaning	321
Vendor Copper, hand pumice	736
Electroless Copper, Al <sub>2</sub> O <sub>3</sub> jet scrubbed, no cleaning	787
Vendor copper, brush scrubbed	975
Electroless Copper, brush scrubbed	1111
Electroless Copper, Al <sub>2</sub> O <sub>3</sub> jet scrubbed, acid dip	1138
Vendor Copper, chemical clean	1153
Fine Grain Foil, chemical clean	1176

As was mentioned earlier, the work of reference Nr. 2 and our own foil characterizations correlating chromate removal (Auger analyses), OSEE counts, and print/etch yield performance (Ref. 5), had tentatively established an OSEE count of •700 as an indicator of a freshly cleaned surface, free of chromate. As we review the above data, we notice that all no-clean surfaces fail this specification. It should be noted that our correlations were established only for copper and not for the brass surface of double-treat foils. Therefore, the fact that the double-treat foil yields a low OSEE count is not necessarily a matter of concern. Also failing the specification was the acid dipped reverse treated foil, however, based on the operator's observations and notes, we have to assume that the acid had not been removed properly from the test specimen, leading to rapid retarnish. The standard deviation for this suspect OSEE count of 173 was also unusually high (86). All other cleaned samples recorded OSEE counts of •700 and passed our empirical specification.

## 7.2 Performance Results

The following foils/surfaces were not laminated with resist: Fine Grain Foil (no clean), Electroless Copper Al<sub>2</sub>O<sub>3</sub> jet scrubbed but not acid dipped, and uncleaned Electroless Copper. They served only as topography reference samples, but since they were never cleaned, or badly retarnished, no meaningful yield performance results were expected.

### 7.2.1 "Instant Adhesion"

The following table shows "instant adhesion" results for different surfaces as a function of time after lamination. The reported numbers are area % of resist not pulled off the board by the tape test. The data generated have limited information value: historically, such adhesion tests were used to confirm that dry film resists did not require unusual hold times after lamination and exposure to achieve reasonable adhesion of the resist to the board to survive manual or automatic handling of the laminated boards. Also, the manual or automatic removal of the Mylar® coversheet after exposure, before development, requires the right balance of resist/ copper adhesion and resist/ Mylar® tack. However, neither excessive hold time requirements nor balance of adhesion before wet processing are big issues today. The actual "survival" of resist during wet processing (etching, plating) will not necessarily correlate with the "instant adhesion" test as described here. There is also considerable noise in the data, especially at "zero hold time", partly attributable to variations in the operator's test technique and interpretation of the result. No statistical significance tests were applied to the data. Some of the "zero minute" results appear to confirm conventional wisdom and intuition: the good (instant) adhesion on double treat, pumiced copper, and brushed electroless would be expect while uncleaned vendor copper types would yield lower numbers. A new data point is the good instant adhesion of the tested dry film on fine grain foil.

**"Instant Adhesion" Results**

Surface	0 min.	15 min.	30 min.	60 min.	24 hrs.
Vendor Copper- no cleaning	14	94	98	95	100
Vendor Copper- chemical clean	6	98	99	76	98
Vendor Copper- brush scrub	42	97	98	86	100
Vendor Copper- hand pumiced	86	95	94	100	100
Double Treat Copper- no clean	100	100	100	100	100
Fine Grain Foil- chemical clean	98	97	98	100	100
Reverse Treated Foil- no clean	40	93	91	94	99
Reverse Treated Foil- acid dipped	0	88	75	93	100
Electroless Copper- Al <sub>2</sub> O <sub>3</sub> scrubbed, acid dipped	53	72	97	98	97
Electroless Copper- brush scrubbed	92	80	100	93	98

### 7.2.2 Developed Resist Resolution

On both test patterns, print and etch as well as pattern plate, the average line and space resolution for all surfaces measured was 50 microns, with no significant differences attributable to surfaces or other factors. The isolated line retention on the print & etch pattern averaged 30 microns. The double- treat sample had a 40 micron resolution, however, statistical significance criteria would not permit us to conclude that DT was different from other surface with regard to the performance criteria of developed resist resolution. On the pattern plate pattern the average isolated space resolution was 60 microns with no distiguishable differences attributable to surfaces. From these data one could draw the conclusion that differences in surfaces and adhesion do not affect resolution through the aqueous development step. However, it remains to be seen if differences will show up in the more aggressive process environments of etching and plating.

### 7.2.3 Resist Lifting (Adhesion) Performance in Pattern Plating

Lifting ranged from 5 microns to 59 microns. Depending on the operator inspection technique, "minimal" lifting, "no lifting", and up to "5 micron" lifting are essentially the same results.

### Results of Resist Lifting Tests

Surface	Resist Lifting (microns)	Standard Deviation (microns)
Electroless copper, Al <sub>2</sub> O <sub>3</sub> (fresh) scrubbed, acid dip	5	1
Double-treat copper, no clean	15	2
Vendor copper, hand pumiced	16	4
Vendor copper, brush scrubbed	17	2
Vendor copper, chemical clean	18	3
Electroless copper, brush scrubbed	22	4
Fine grain foil, chemical clean	22	6
Reverse treated foil, acid dip	47	15
Vendor copper, no clean	59	21
Reverse treated foil, no clean	heavy lifting	N/A

Not all of these surfaces are typically found in plating processes. However, to get a broader picture of suitable surfaces for resist adhesion through plating processes, we included surfaces such as double-treat. Certainly the traditional electroless copper surfaces had to be included in the study, but also different vendor copper surfaces to reflect the growing use of direct metallization processes. The no-clean vendor copper and no-clean reverse treated foils were included as reference points and showed the expected poor performance (large lifting). As mentioned earlier, the OSEE data flagged the reverse treated, acid dipped foil as suspect, and the heavy resist lifting confirmed that something went wrong during surface preparation. Next, we notice the range of conditions which yielded 15 to 22 micron lifting, which does not meet the criteria of showing statistically significant differences. The interpretation of the similarity of lifting results of this group of surfaces, despite a relatively large spread of  $R_a$  values in the group, would point in the following direction: adhesion performance of resists in plating environments is less dependent on the topography of the substrate and more dependent on the nature of the chemical bond between the resist and the copper and how this bond is attacked by the plating chemistries. Internal resist stresses due to resist volume changes through the various wet chemistry steps will aggravate adhesion failure.

On the other hand, for a given surface preparation method, such as aluminum oxide jet scrubbing, acid dip, rinse, and dry, and for a given resist, fixed lamination and plating conditions, we were able to show the effect of varying  $R_a$  values on resist lifting. Experimental conditions: electroless copper plated boards were given a mild acid clean (10% sulfuric acid, followed by a 1.7 bar rinse), then aluminum oxide jet scrubbed (15-20% solids, 220 grit, 1.7 bar jet pressure, 1.8 meter/minute line speed, followed by three rinses of 1.4, 1.7, and 1.7 bar pressure; dry). Since the boards were transported to another site for further processing, they were given a mild acid dip, rinse, and dry before resist lamination.

The boards represented changes in the surface preparation process over the course of two months' production: the first board had been cleaned with fresh aluminum oxide shortly after the jet nozzles had been replaced. The next three boards represent one week's of use of the aluminum oxide particles, one month, and two months respectively. The monthly throughput is roughly equivalent to 60,000 panels, average size of 18X21 inches, corresponding to about 2,700 m<sup>2</sup> of surface area, counting both sides. Scanning Electron Microscope (SEM) pictures were taken of the aluminum oxide particles in use at the time when these four boards were processed (see Fig. 20, 21, 22, 23). The pictures show the gradual wear on the surface: with time the particles become smoother and less effective. Contact profilometry of the first board cleaned with fresh aluminum oxide revealed directionality of surface roughness parameters  $R_a$  and  $R_z$ . In addition, the initial measurements of the next three boards showed a trend toward a smoother surface as the aluminum oxide aged. To determine whether the trend toward smoother surface was real, multiple measurements were made. Profilometry scans were made in two directions on each sample. The first direction was the one showing a high  $R_a$  value and the data are shown in the first table below. In the second direction the  $R_a$  values were low and are shown in the second table. The corresponding resist lifting results showed little difference between the one week old and the one month old samples, but did show significantly more lifting on the surface prepared with the two month old aluminum oxide (see table below). These findings are in good agreement with the production results. In production a different dry film resist with slightly better adhesion was in use, and incidents of underplating were reported when aluminum oxide, and nozzles, are not exchanged within three months.

### CONTACT PROFILOMETRY DATA FROM BOARDS

**CLEANED BY ALUMINUM OXIDE JET SCRUBBING**

(Data from **high** Ra value scan direction)

Scan	1 Week	1 Week		1 Month	1 Month		2 Months	2 Months
Number	Ra ( $\mu$ )	Rz ( $\mu$ )		Ra ( $\mu$ )	Rz ( $\mu$ )		Ra ( $\mu$ )	Rz ( $\mu$ )
1	0.236	1.66		0.223	1.18		0.170	1.05
2	0.204	1.33		0.201	1.19		0.179	1.06
3	0.185	1.13		0.213	1.27		0.171	1.09
4	0.259	1.57		0.205	1.19		0.181	1.08
5	0.216	1.40		0.200	1.36		0.175	1.32
6	0.198	1.27		0.226	1.51		0.190	1.24
7	0.188	1.19		0.185	1.32		0.192	1.13
8	0.216	1.33		0.167	0.96		0.177	1.03
9	0.185	1.31		0.166	1.09		0.193	1.17
10	0.222	1.34		0.164	1.07		0.190	1.17
11	0.202	1.34		0.215	1.22		0.188	1.21
12	0.190	1.13		0.215	1.35		0.207	1.30
13	0.242	1.74		0.208	1.31		0.184	1.05
14	0.229	1.59		0.206	1.42		0.207	1.32
15	<u>0.241</u>	<u>1.55</u>		<u>0.199</u>	<u>1.40</u>		<u>0.204</u>	<u>1.27</u>
Average	0.214	1.39		0.200	1.26		0.187	1.17
Std. Dev.	0.024	0.19		0.020	0.15		0.012	0.10

**CONTACT PROFILOMETRY DATA FROM BOARDS  
CLEANED BY ALUMINUM OXIDE JET SCRUBBING**

(Data from **low** Ra value scan direction)

Scan	1 Week	1 Week		1 Month	1 Month		2 Months	2 Months
Number	Ra ( $\mu$ )	Rz ( $\mu$ )		Ra ( $\mu$ )	Rz ( $\mu$ )		Ra ( $\mu$ )	Rz ( $\mu$ )
1	0.143	0.97		0.156	0.96		0.096	0.68
2	0.125	0.79		0.159	1.01		0.099	0.64
3	0.125	0.87		0.148	0.88		0.088	0.58
4	0.134	0.83		0.125	0.79		0.096	0.60
5	0.119	0.78		0.146	1.00		0.089	0.69
6	0.139	0.86		0.140	0.88		0.084	0.56
7	0.124	0.75		0.152	1.40		0.097	0.62
8	0.130	0.78		0.117	0.71		0.119	0.79
9	0.098	0.63		0.167	1.32		0.093	0.78
10	0.144	0.90		0.149	1.07		0.096	0.62
11	0.132	0.88		0.137	0.87		0.099	0.86
12	0.142	0.89		0.162	0.99		0.085	0.65
13	0.134	0.95		0.141	0.98		0.078	0.50
14	0.162	1.26		0.131	0.76		0.093	0.66
15	<u>0.128</u>	<u>0.89</u>		<u>0.124</u>	<u>0.78</u>		<u>0.089</u>	<u>0.64</u>
Average	0.132	0.87		0.144	0.96		0.093	0.66
Std. Dev.	0.014	0.14		0.015	0.19		0.009	0.09

The data from each panel were compared by calculating the t statistic for two means to determine if the observed differences were statistically significant. For this comparison, the t value for a 95% confidence level and 28 degrees of freedom was 2.05. The roughness values corresponding to the one week old and one month old samples were right on the border line of showing a statistically significant difference. The comparison of the 1 week panel and the 2 month panel showed that both Ra and Rz were statistically different. The calculated t value for the Ra data was 8.78 and the t value for the Rz data was 4.93. Thus, after 1 month there may still be equivalent roughness but by two months there is a real drop in the roughness of the surface as measured by contact profilometry.

### RESIST LIFTING RESULTS FOR AGED ALUMINUM OXIDE MEDIA

“AGE” OF Al <sub>2</sub> O <sub>3</sub> MEDIA	Resist Lifting (microns)	Standard Deviation (microns)
Fresh	5	1
1 Week	No Lifting	-
1 Month	No Lifting	-
2 Months	10	1

#### 7.2.4 Yield Results

##### Rank by AOI Yield (DEMI's)

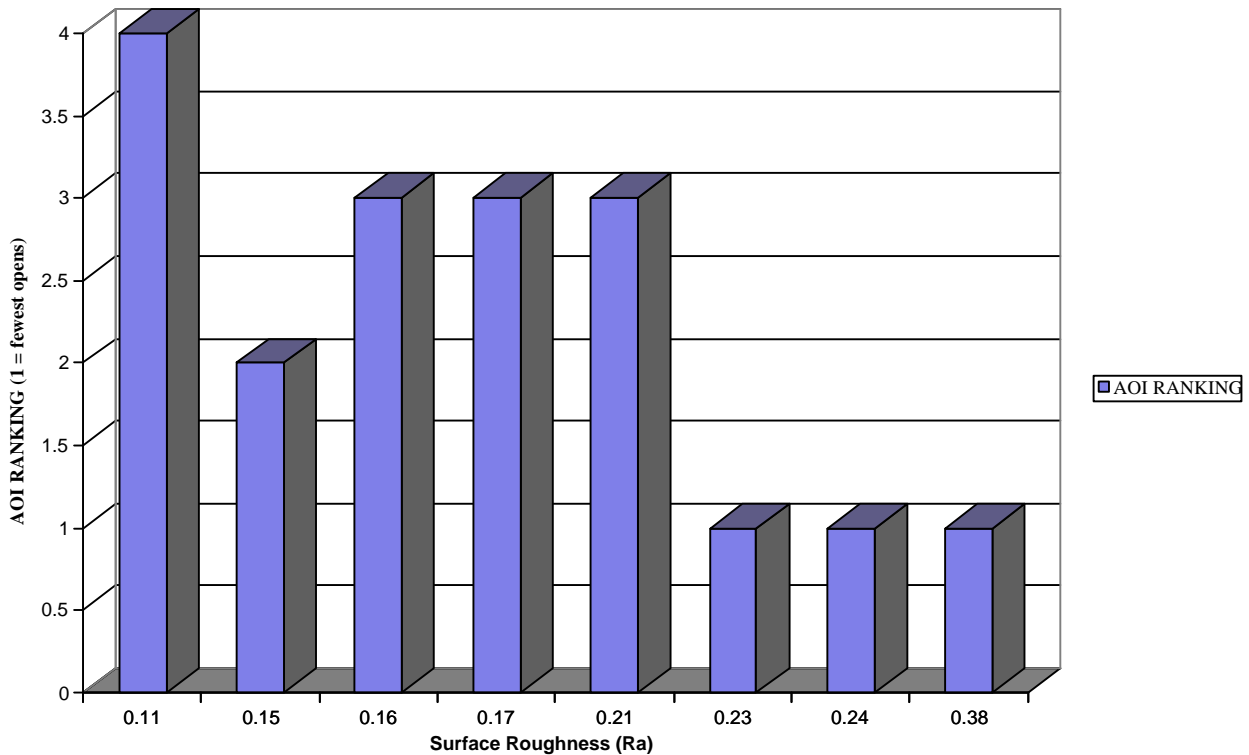
<u>Surface</u>	<u>Average DEMIs</u>
Double Treat, no cleaning	124
Reverse Treated Foil, acid dip	152
Vendor Copper, hand pumiced	154
Vendor Copper, brush scrubbed	220
Fine Grain Foil, chemical clean	308
Electroless Copper, brush scrubbed	382
Vendor Copper, chemical clean	412
Vendor copper, no cleaning	443
Electroless Copper, Al <sub>2</sub> O <sub>3</sub> jet scrubbed, acid dip	890
Reverse Treated Foil, no cleaning	7104

Looking at the above data without statistical tools left us puzzled: there were some data that appeared to confirm conventional wisdom, others didn't seem to make sense. For example, the fact that double-treat foil gives the lowest number of random open defects per million inches (DEMI's) and that pumiced and brushed copper are in the low defect group, with pumiced copper slightly better than bristle brushed copper, all makes sense in view of production experience and earlier studies. Also, that the no-clean reverse treated foil might give a high defect rate is plausible, especially if the surface had a chance to pick up contaminant in handling prior to dry film lamination. The very favorable results obtained with the acid dipped reverse treated foil; i. e., dipped to remove excessive oxide tarnish, are in line with the good yield results reported in references Nr.15 and 16, but seem to contradict the low OSEE count and large lifting in pattern plating. However, if the demands on dry film adhesion in print & etch are sufficiently different from plating, with a greater beneficial contribution from mechanical interlocking, and less chemical attack on the resist/copper interface than in plating, then we can rationalize even these seemingly odd results.

When we apply statistical significance criteria to the above AOI data and probe for a correlation with R<sub>a</sub> results, we find a correlation between high R<sub>a</sub> numbers and low random open defects within the limits tested. The correlation is not overwhelmingly convincing, but of significance (Fig. 24).

The AOI yields obtained on the chemically cleaned fine grain foil appear slightly better than on chemically cleaned standard vendor copper but the difference is not deemed significant by statistical criteria. This finding is

probably not in conflict with the much more extensive studies of references Nr. 21 and 31. The referenced studies used a much larger number of panels, narrowing down the standard deviation, and allowing to detect 4-7% first pass yield increases in print & etch with fine grain, low profile foil. Second pass yield increases were less impressive but still averaged 1-3%. According to the author, the improvements may not so much correlate with a larger surface area of the fine grain foil, but have to do with the overall quality of the foil (e.g. fewer nicks), and even more importantly, with the "low profile" characteristics of the foil. This feature allowed the process engineer to shorten the process time in the etcher which in turn reduced the potential for etched line width reduction and opens.



**Fig. 24: AOI Yield Data Ranking by Contact Profilometry  $R_a$**

## Summary and Conclusions

This study expanded the scope and updated an earlier study on the subject of copper foils, surface preparation, surface characterizations, and dry film resist performance parameters. New surface analysis methods were applied and correlated with the traditional tools of surface characterization. Response factors were expanded from print & etch yields to "instant adhesion", resolution after development, and resist lifting in pattern plating. Statistical significance tests were applied to the results to assess their validity. A correlation between  $R_a$  values and innerlayer AOI yields was reconfirmed. The aluminum oxide jet scrubbing surface preparation was investigated, and a correlation between aluminum oxide particle shape, time in the machine of the abrasive particles, and resist lifting was established, confirming observations in a production environment. Based on a comparison of the print & etch yields with the data on resist lifting in plating baths, we are concluding that the dominant failure mode of the chemical bonding mechanism between resist and copper in the plating environment is due to chemical attack and stresses within the tested dry film resist.

## Acknowledgements

We gratefully acknowledge Tom Poole's help in procuring test samples and Doreen Thornton's administrative and documentation assistance.

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Figure 1

Fig.1: Vendor Copper, Chemical Clean

Figure 2

Fig. 2: Vendor Copper, Brush Scrubbed

Figure 3

Fig. 3: Vendor Copper, Pumiced

Figure 4

Fig. 4: Vendor Copper, No Clean

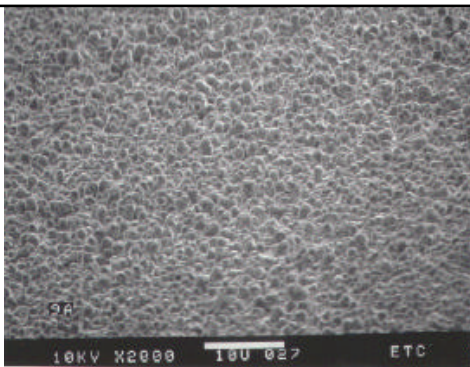


Fig. 5: Double-Treated Foil

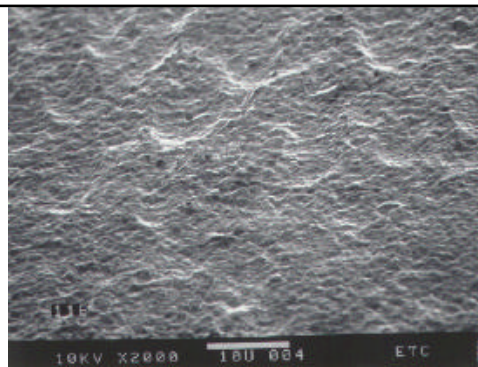


Fig. 6: Fine Grain Foil, Chemical Clean

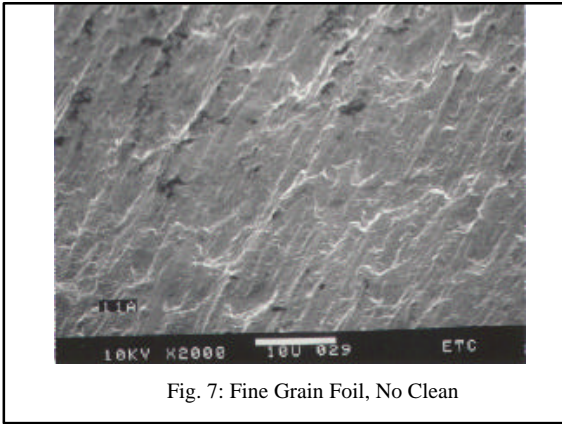


Fig. 7: Fine Grain Foil, No Clean

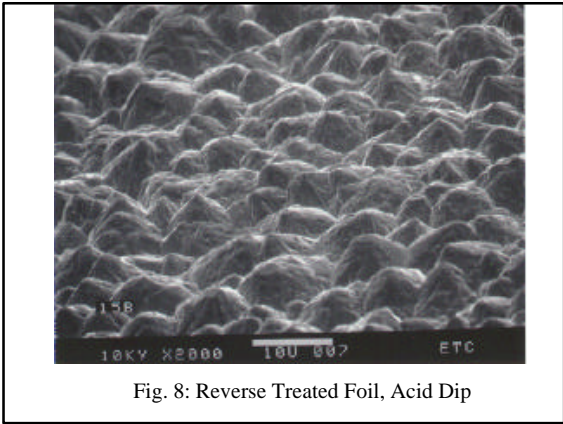


Fig. 8: Reverse Treated Foil, Acid Dip

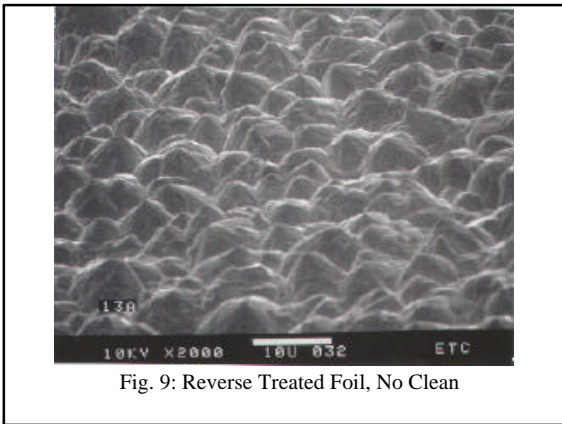


Fig. 9: Reverse Treated Foil, No Clean

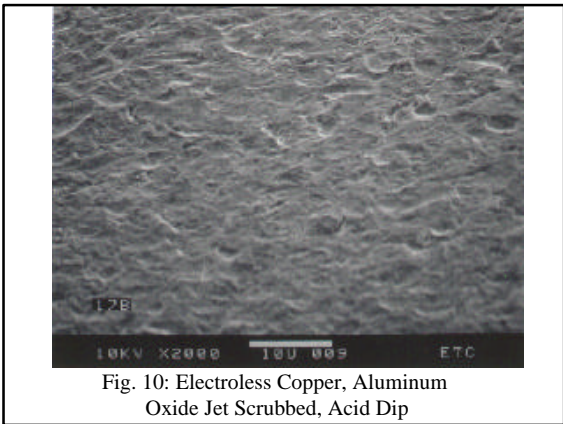


Fig. 10: Electroless Copper, Aluminum Oxide Jet Scrubbed, Acid Dip

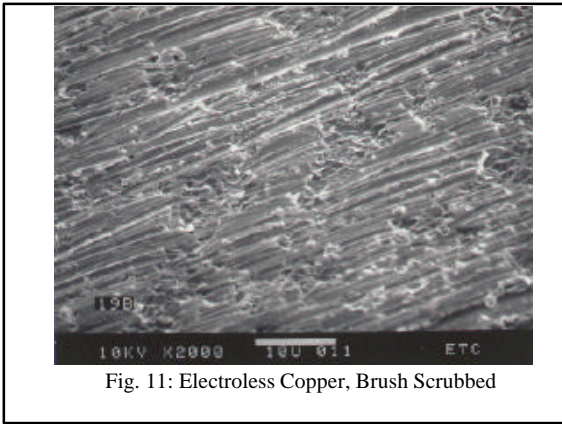


Fig. 11: Electroless Copper, Brush Scrubbed

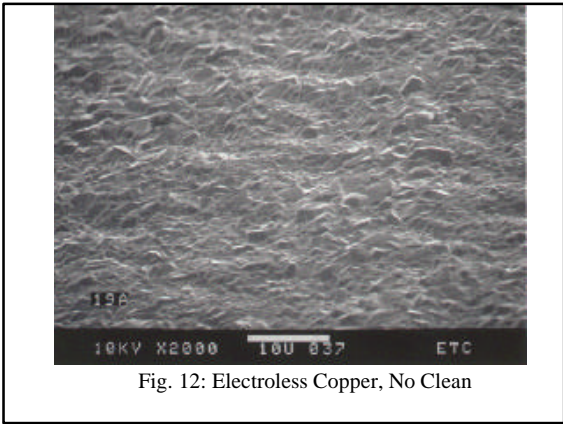


Fig. 12: Electroless Copper, No Clean

