

# ADHESION TO VERY SMOOTH SUBSTRATES UTILIZING AN OPTIMIZED SEMI-ADDITIVE PROCESS

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## ABSTRACT

Mobile phones, tablets and other hand held devices are pushing technology to new heights. These devices continue to add greater functionality while decreasing in size. This miniaturization demands tighter pitch, finer lines and better signal integrity. Nowhere is this clearer than in the IC substrate market. Semi-Additive Processing or SAP has been widely used in the manufacture of tighter pitch and finer lines for many years, however with the growing need to maintain and improve signal integrity, different dielectric materials are being evaluated.

One of these materials is polyimide (PI), and although not new to the industry, PI shows good chemical resistance, low moisture adsorption and good UV absorption. In addition, PI has excellent thermal stability, low dielectric constant and good processability. These unique chemical, mechanical and electronic properties make PI one of the best candidates for interlayer dielectric.

MacDermid developed an optimized semi-additive process (SAP-PI), which was tailored for PI film in build up application. This SAP-PI combines low-roughness desmear and efficient adhesion promotion processes. After desmear, the PI surface showed Ra <60 nm, while achieving initial peel strengths between 900-1700 gf/cm on different commercial PI substrates. After 7-day baking test at 150 °C, the peel strengths can be maintained between 500-1100 gf/cm.

This paper will discuss an optimized process flow and associated steps in detail to alleviate concerns on processing complexity. It will include characterization and analysis on PI and metal plating topography using scanning electron microscopy, their compositions using energy dispersive X-ray spectroscopy, and adhesion between them from 90° peel tests.

## INTRODUCTION

Higher functionality, higher performance and miniaturization are the mantras of any electronic device, and the future guarantees more of the same. To achieve the requirements of these devices, designs must incorporate laser drilled blind microvias, tighter pitch, finer line while maintaining good circuitry adhesion and improving signal integrity. Semi-Additive Process (SAP) is a production proven method used on build up materials that enables the manufacture of ultra fine circuitry with good signal quality.

The traditional SAP process utilizes some roughening or texturing of the dielectric substrate in order to achieve sufficient adhesion. However rough surface negatively impacts signal integrity. The

amount and size of the dielectric filler have an impact on adhesion as well. Filler-less dielectric material that possesses good chemical resistance, excellent thermal stability and low dielectric constants are being considered as replacements for conventional build up material. One of these materials is polyimide (PI).

PI film is one of the most popular dielectric materials for flexible printed circuitry (FPC), which proliferate rapidly to replace traditional wire harnesses. With special properties of PI film, PI based FPC shows many benefits compared to traditional cabling and rigid boards. Physically, it presents smaller size and weight as well as better thermal stability and wider operating temperature. Regarding electronic properties, it provides higher wiring accuracy and circuit density, stronger signal quality, better reliability and impedance control. In addition, it shows good design flexibility and enables the elimination of mechanical connectors.

MacDermid developed an optimized semi-additive process (SAP-PI), which was tailored for PI film. This paper will discuss its process flow and associated steps in detail to alleviate concerns on processing complexity. It will include characterization and analysis on PI and metal plating topography using scanning electron microscopy (SEM), their compositions using energy dispersive X-ray spectroscopy (EDX), and adhesion between them from 90° peel tests. The role of each processing step as well as its influence on metal-PI adhesion will be discussed.

## OPTIMIZED SEMI-ADDITIVE PROCESS FLOW

The process flow for SAP-PI is listed in Figure 1. It includes similar steps as in traditional SAP, which makes it adaptable to existing equipment sets. Meanwhile, the SAP-PI combined etching (desmear), conditioning, electroless and electrolytic plating techniques optimized for PI films from MacDermid. In the etching and conditioning step, innovative MacDermid product, XD 7805 conditioner was utilized to impart the PI film with a very smooth but highly functionalized surface. After activating and reducing, a Ni plating step using MacDermid MACUPLEX J-64 electroless Ni introduced a thin Ni layer bonding directly to PI film, functioning as metal tiecoat to enhance PI-plating adhesion. Then, a thin Cu seed layer was applied on the film surface using MacDermid M-Copper 20 electroless Cu plating process. This Cu layer provided the film surface with increased electrical conductivity which results in a more uniform electrolytic copper thickness across the panel width. In the last electrolytic Cu plating step, MacDermid HiSpec3 acid copper plating process was used to build up idea Cu thickness.

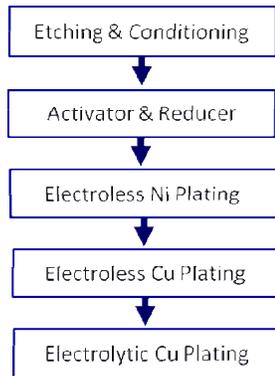


FIGURE 1. OPTIMIZED SEMI-ADDITIVE PROCESS FLOW

Following each plating step, there was a 15 min baking treatment at 150 °C. To re-activate the baked metal surface for the next plating process, a 3-min pre-dip treatment at 24 °C was also inserted using MacDermid MULTIPREP 506 acid pickle and activator. In the following part of this paper, PI film indicated DuPont 150 EN-C PI film unless otherwise specified. This PI film was chosen to compare traditional SAP and optimized SAP-PI regarding PI-plating adhesion. The influence of each processing step was demonstrated and discussed as the following.

## RESULTS AND DISCUSSION

### Etching and Conditioning

With optimized SAP-PI, etching and conditioning of the PI film is conducted at lower caustic solution (NaOH wt% as low as 5~10) and a lower and wider temperature range (from room temperature to 70 °C) for a short time of 2~5 min. It is much milder condition compared to traditional SAP for PI material, which normally uses a more concentrated NaOH solution up to 25 wt% at temperature like 70 °C or even higher. As a result, in our studies, the PI film after SAP-PI etching and conditioning maintained a very smooth surface. It was well demonstrated by the SEM images in Figures 2-(2, 3), which demonstrate its topographies at 1000× and 5000× magnifications, respectively.

For comparison, Figure 2-(1) also shows the topographies of raw PI film at 1000× and 5000× (inserted image). Compared to the raw film, the PI film after etching and conditioning showed some tiny pores on the top, indicating mild surface roughness. However, their surface roughness couldn't be directly quantified using an instrument like Zygo because the raw PI and the etched PI films were both transparent and could not return valid signals during Zygo's laser scanning. Meanwhile, Figure 2-(4) presents the topography of a typical resin at 5000×, which was etched by a traditional SAP desmear process separately and presented a low Ra of 60 nm. By comparing Figures 2-(3, 4), it was obvious that the PI film treated by SAP-PI etching and conditioning exhibited a much lower surface roughness and its Ra was much less than 60 nm.

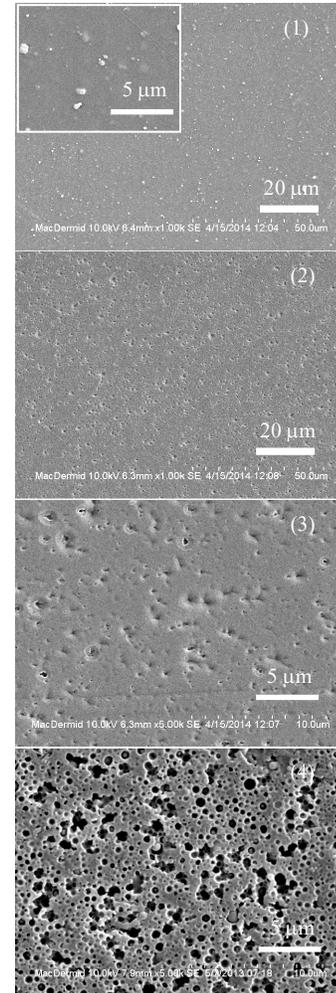


FIGURE 2. SEM IMAGES FOR PI FILM BEFORE (1) AND AFTER (2, 3) SAP-PI ETCHING AND CONDITIONING. (4) SHOWS A TYPICAL RESIN WITH LOW RA= 60 NM.

### Electroless Ni Plating

Following etching and conditioning, direct metallization processes were utilized to build up PI-metal laminate. To apply tiecoat and seed Cu coating on PI film, there are two popular techniques, sputter deposition and electroless plating. Sputter deposition method is simple but costly, which limits its use in production. In the optimized SAP-PI, MacDermid MACUPLEX J-64 electroless Ni was used to introduce a thin layer of Ni serving as the tiecoat. This Ni plating process was performed at 39 °C for 8-15 min. As shown by the SEM image in Figure 3-(1), after Ni plating, the PI film surface was uniformly covered by Ni grains and the thickness of this Ni tiecoat was approximately 0.2-0.3 μm (i.e., 8-12 μin).

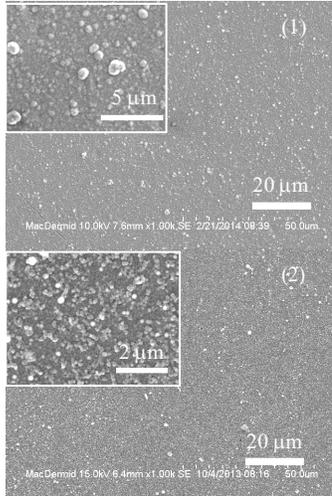


FIGURE 3. SEM IMAGES FOR PI FILM (1) AFTER ELECTROLESS NI PLATING AND (2) AFTER ELECTROLESS CU PLATING

Using EDX built in SEM, composition of the Ni plating was characterized and is shown in Table 1. It proved that the phosphorus content in the Ni layer was only around 2.5 wt%. Compared to high-phosphorus Ni plating, this Ni tiecoat with low phosphorus content can provide much better electrical conductivity, which is preferred for upcoming electroplating process.

#### Electroless Cu Plating

In order to further promote the electrical conductivity of the film surface, MacDermid M-Copper 20 electroless Cu plating was applied on top of the Ni layer. As shown in Figure 3-(2), the Cu seeds were much smaller than Ni grains and homogeneously distributed on the Ni surface. This Cu layer was as thin as 0.05 μm (~2 μin), so the EDX data from its surface (Table 1) still showed Ni signal in majority and only 5.3 wt% Cu. Nevertheless, this thin Cu seed layer together with low phosphorus Ni tiecoat provided good conductivity on the PI surface for the final electrolytic Cu plating.

TABLE 1. EDX RESULTS FOR SAMPLE SURFACE COMPOSITION FOR PI FILM AFTER ELECTROLESS NI AND ELECTROLESS CU PLATING.

Electroless			
Plating	Element	Wt.%	At.%
Ni	C	0.6	2.9
	O	1.4	4.7
	P	2.5	4.3
	Ni	95.5	88.1
Cu	C	0.5	2.5
	O	1.0	3.5
	P	2.4	4.2
	Ni	90.8	85.2
	Cu	5.3	4.5

#### Electrolytic Cu Plating

In optimized SAP-PI, MacDermid HiSpec3 acid copper was used to build up Cu thickness. Figure 4-(1) exhibits a cross-section of the PI film after electrolytic Cu plating. A Cu thickness of

approximately 20-22 μm was obtained after a one-hour plating with a current density of 20 ASF at 23 °C. By adjusting current density and plating time, the Cu thickness could be tailored to the ideal value with preferred plating rate, which proved to show little influence on PI-plating adhesion using the optimized SAP-PI.

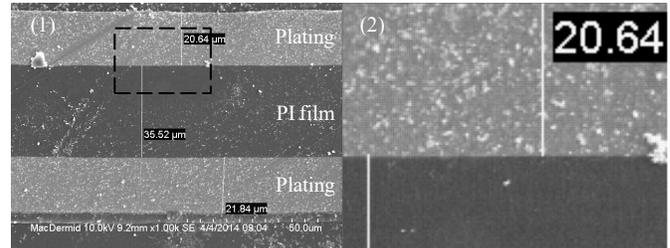


FIGURE 4. (1) SEM IMAGE SHOWING CROSS-SECTION OF PI FILM AFTER ELECTROLYTIC CU PLATING. THE AREA IN DASH FRAME WAS ENLARGED AND SHOWN IN (2).

In addition, an area of PI-plating interface was framed using black dash line as shown in Figure 4-(1), and enlarged and demonstrated in Figure 4-(2). It was obvious that the PI-plating interface was very smooth, which again proved that the PI film maintained very good smoothness after etching and conditioning in SAP-PI.

#### PI-Plating Adhesion

The adhesion between the very smooth PI surface and plating was evaluated by the peel strength at PI-plating de-adhesion in 90° peel test. Freshly processed with SAP-PI, plated PI samples were cut into 1 in W × 2.25 in L specimens and tested, whose results were recorded as initial PI-plating adhesion. Meanwhile, specimens of same size were cut from same samples and subjected to a 7-day baking test at 150 °C. After baking test, these specimens were also peeled for PI-plating adhesion evaluation, whose results were recorded as 7D adhesion.



FIGURE 5. DIGITAL CAMERA IMAGE SHOWS THE PEELED OFF NI PLATING WAS MIRROR-SHINING.

Figure 5 presents a picture of a sample after peel test. Apparently, both the PI film top and the back of plating were mirror shining, indicating the PI-plating interface was very smooth. In other words, it proved again that the PI film maintained very smooth surface through the SAP-PI flow.

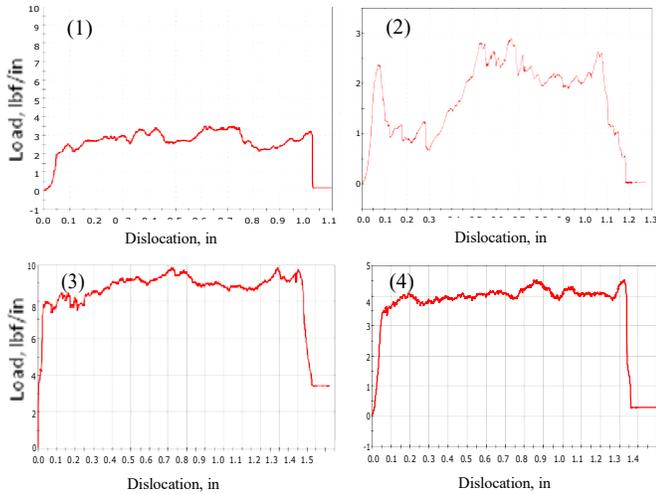


FIGURE 6. REPRESENTATIVE LOAD VS. DISLOCATION PROFILES FROM 90° PEEL TESTS SHOWING (1) INITIAL PI-PLATING ADHESION PROCESSED WITH TRADITIONAL SAP; (2) 7D PI-PLATING ADHESION PROCESSED WITH TRADITIONAL SAP; (3) INITIAL PI-PLATING ADHESION PROCESSED WITH SAP-PI; (4) 7D PI-PLATING ADHESION PROCESSED WITH SAP-PI.

Figure 6 presented representative load vs. dislocation profiles from peel tests. As shown in Figure 6-(1), samples processed with traditional SAP flow presented good initial adhesion. The peel strength from PI-plating de-adhesion averaged 2.8 lb/in, i.e. 500 gf/cm. However, after baking test, the PI-plating adhesion became very non-uniform and dropped dramatically to only 1.9 lb/in, 339 gf/cm (Figure 6-(2)). Meanwhile, the sample processed with the optimized SAP-PI flow showed excellent initial peel strength averaged at 5.5 lb/in, i.e. 983 gf/cm (Figure 6-(3)). Moreover, baked at 150 °C for 7 days, the sample still demonstrated very uniform PI-plating adhesion. Nevertheless the PI-plating adhesion was maintained at very good high level, averaged at 3.8 lb/in, i.e. 679 gf/cm (Figure 6-(4)).

adhesion data were shown as the outlined black points, averaging at  $536 \pm 21$  gf/cm (indicated by dash line). These results well proved that the optimized SAP-PI process can render PI film with excellent initial PI-plating adhesion, which can be maintained at high level after 7-day baking at 150 °C with very good consistency.

In addition, the optimized SAP-PI technique was also utilized on silicon-containing PI film from a customer in Taiwan, which showed initial peel strength averaged at 1731 gf/cm and a 7D peel strength averaged at 1155 gf/cm (Profiles not shown here). It well demonstrates the good potential of applying this optimized SAP-PI process to PI films other than DuPont 150 EN-C.

## CONCLUSIONS

The optimized SAP-PI process combines new etching, conditioning and direct metallization techniques from MacDermid. It contained similar steps like traditional SAP process, which makes it adaptable to existing equipment sets. Moreover, the processed PI surface showed very low Ra, which was much less than 60 nm. In addition, the initial PI-plating adhesion was achieved to 900-1700 gf/cm on different commercial PI substrates. After 7-day baking test at 150 °C, the PI-plating adhesion can be maintained between 500-1100 gf/cm. In other words, the optimized SAP-PI can render excellent initial and 7D adhesion on smooth PI. Therefore, it demonstrates great potential in manufacturing of PI based FPC and other build-up packaging.

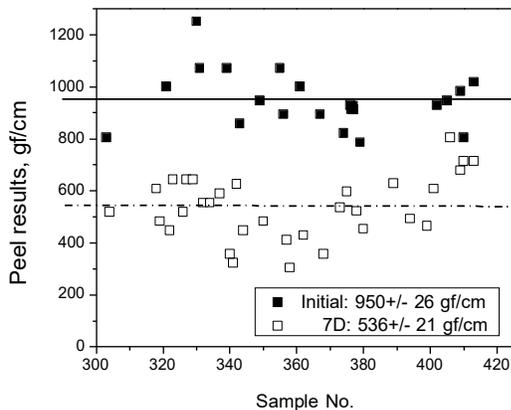


FIGURE 7. PI-PLATING ADHESION RESULTS FROM REPEATING SAMPLE No 303-416.

To evaluate its repeatability and consistency, 114 replicate experiments were performed by processing PI films using the SAP-PI flow. And the results are shown in Figure 7. In Figure 7, the solid black points represent initial PI-plating adhesion, which showed an average of  $950 \pm 26$  gf/cm indicated by the black line. And the 7D