



# CYCLOTENE\* Advanced Electronic Resins

## *Processing Procedures for BCB Adhesion*

### **1. Introduction**

In fabricating a semiconductor device or package, adhesion of the materials used to one another is one of the most important considerations. Typically, multiple layers of polymers, metals, and inorganic glasses are used, and these layers must adhere well to each other to survive reliability testing. The adhesion of Cyclotene™ Advanced Electronic Resin\* (BCB) to various materials is thus of vital importance in fabricating a reliable device.

This application note discusses the various aspects of BCB adhesion. Section 2 describes the chemistry of the two adhesion promoters offered by Dow. Section 3 reviews processing procedures, and section 4 briefly summarizes the interfacial science involved. Section 5 covers pre-clean procedures, which are crucial in obtaining reliable adhesion. Section 6 describes the effects of processing chemicals, and section 7 reviews the adhesion test methods used to generate the data that is reported here. In sections 8-10 we review the adhesion of BCB deposited on top of other materials, while in sections 11-13 the adhesion of other materials on top of BCB is discussed.

### **2. Adhesion promoter chemistry**

Two adhesion promoter solutions are available from The Dow Chemical Company, AP8000 and AP3000. Both are based on organosilane primer chemistry.

These adhesion promoters are supplied as dilute solutions of the organosilane compounds in 1-

methoxy-2-propanol. They are ready to use, and dilution or mixing may result in reduced adhesion. Table 1 shows the composition and sales specs of the two products.

During the manufacture of AP3000 and AP8000, the silanes are hydrolyzed by a proprietary process. This converts some of the alkoxysilane or acetoxysilane groups to silanols (Si-OH), which partially condense to form siloxane oligomers. It is the silane oligomers containing silanol groups that are believed to be the active components in the adhesion promoter solutions.

AP3000 and AP8000 are the only adhesion promoters recommended for use with BCB. In particular, hexamethyldisilazane (HMDS) is not effective as a BCB adhesion promoter.

### **3. Adhesion promoter application**

To prepare the surface for BCB coating, the adhesion promoter solution is applied to the surface of the substrate, which is then spun dry. The substrate surface is immediately ready for BCB coating. AP3000 and AP8000 are designed for spin coating, and vapor priming of these is not recommended. The adhesion promoters improve the adhesion of BCB to most surfaces, and their use is virtually always recommended.

The basic function of an adhesion promoter is to form a compatible interface between two otherwise incompatible materials. The mechanism of adhesion derives from condensation of silanols with surface oxygen atoms or hydroxyl groups. The surface of the metal or inorganic material is then covered with an organosilane, with the organic portion facing outward. This renders the surface more compatible with an organic polymer.

\* trademark of The Dow Chemical Company



AP3000 often provides better adhesion than AP8000. The presence of the vinyl group in AP3000, which can react with the benzocyclobutene groups in the resin, is sometimes cited as a reason for this improvement. However, concrete data to support this is lacking. On some surfaces, AP8000 works quite well, despite its lack of apparent functional groups that can react with the BCB polymer. The actual mechanism of interaction between BCB and the adhesion promoter remains unclear. Table 2 shows adhesion to silicon as a function of time delay between AP3000 coat and bake, and BCB coat and bake. The adhesion does not degrade significantly even after a 7 day delay. Note that data is available only for cases where the AP3000-treated surface has been baked.

(T1100) there is still a thin layer remaining. Though the thickness of the AP3000 layer is dependent on the coating spin speed, the adhesion is independent of the thickness.

**4. The nature of the adhesion promoter layer**

The coating of adhesion promoter on the substrate is quite thin. Figure 1 shows thickness of an AP3000 layer (measured by ellipsometry) versus spin speed and bake temperature. Figure 2 shows that after a rinse with mesitylene

Table 1. Adhesion promoter composition and sales specs

	AP3000	AP8000
Silane	0.24–0.36%	0.096–0.144%
Particles	25 max*	25 max*
Sodium	1 ppm max	1 ppm max
Potassium	1 ppm max	1 ppm max
Copper	1 ppm max	1 ppm max
Iron	1 ppm max	1 ppm max
Nickel	1 ppm max	1 ppm max
Total Metals	10 ppm max	10 ppm max
Shelf Life	2 years	2 years

\*particles/ml, greater than or equal to 0.5µm

Table 2. Effect of time delay between AP3000 application (coat and bake) and BCB coating on adhesion to silicon.

CYCLOTENE 3022-57				Tape peel test	
AP3000	Bake	Time delay	m-ELT (Kc)	Dry	After 48 hr PCT
Yes	Yes	0	0.33 ± 0.02	5B	5B
Yes	Yes	18 hr	0.33 ± 0.02	5B	5B
Yes	Yes	24 hr	0.32 ± 0.02	5B	5B
Yes	Yes	40 hr	0.32 ± 0.03	5B	5B
yes	yes	166 hr	0.30 ± 0.03	5B	5B
CYCLOTENE 4024-40				Tape peel test	
AP3000?	Bake?	Time delay	m-ELT (Kc)	Dry	After 48 hr PCT
Yes	Yes	0	0.33 ± 0.01	5B	5B
Yes	Yes	18 hr	0.33 ± 0.03	5B	5B
Yes	Yes	24 hr	0.32 ± 0.03	5B	5B
Yes	Yes	40 hr	0.32 ± 0.03	5B	5B
yes	yes	166 hr	0.30 ± 0.03	5B	5B



The effectiveness of the adhesion promoter can often be enhanced by baking, and data on specific substrates will be shown below. The effect of a hot plate bake on the surface appearance of the coating, as measured by atomic force microscopy (AFM), is shown in Figure 4. Baking produces a more uniform coating.

### 5. Substrate Pre-clean

A clean surface, free of particles and organic contaminants, is important to achieve a robust interface between BCB and the underlying substrate. Several different pre-clean procedures have been used, depending on available equipment. Some examples are:

- O<sub>2</sub> plasma
- O<sub>2</sub> plasma; water rinse
- O<sub>2</sub>/CF<sub>4</sub> plasma; water rinse
- O<sub>2</sub>/CF<sub>4</sub> plasma; acetic acid rinse; water rinse
- Ar plasma; water rinse
- Resist stripper; water rinse
- O<sub>2</sub> plasma; resist stripper; water rinse

Each of these can be used successfully, but problems can arise if not done correctly. With some types of plasma tools, an O<sub>2</sub>/CF<sub>4</sub> plasma can create oxyfluorides on the metal surface which can degrade adhesion. Aluminum seems particularly prone to this. Resist strippers have been used but adhesion problems can arise from residues due to incomplete rinsing. The most generally applicable pre-clean procedure is an O<sub>2</sub> plasma, with or without water rinse. RIE (reactive ion etch) type plasma tools appear to give the best results, and are preferred over barrel etchers and microwave plasma tools.

An example of the effect of plasma cleaning treatments is shown in Table 3. In this case, O<sub>2</sub>/CF<sub>4</sub> plasma made adhesion worse relative to the control. Bump shear was used to measure these adhesion values (see section 6 below).

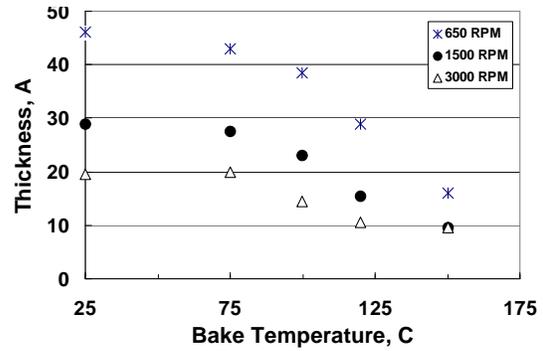


Figure 1. AP3000 thickness versus spin speed and bake temperature.

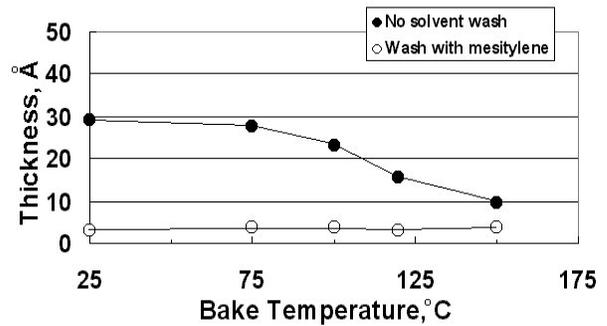


Figure 2. AP3000 thickness before and after rinse with mesitylene (T1100).

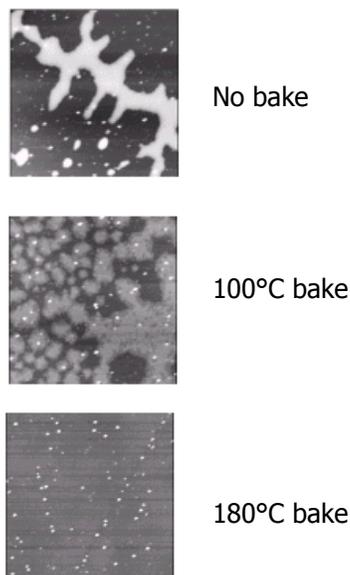


Figure 3. AP3000 coating uniformity vs bake



temperature (AFM).

## 6. Effect of processing chemicals

Some etch chemicals can attack the interface and cause undercut or delamination of BCB from the underlying surface. Sometimes this is not an attack of the adhesion promoter but rather etching of the substrate; delamination occurs because the cured BCB film is under tensile stress, causing the etchant to continue to attack the interface. The chemical sensitivity is substrate-dependent, as is shown in Table 4. In general, chemicals that can etch the material under the BCB can cause undercut or delamination.

Dilute solutions of HF, or buffered oxide etch (BOE) can be used with BCB films but higher concentrations should be avoided. For example we found that BCB on silicon nitride would tolerate 3 minutes in 2% HF, whereas in 11% HF the etch time could not be more than 10 seconds.

## 7. Adhesion test methods

A number of different methods have been used to measure the adhesion of thin films. This report uses data obtained from three different test methods: tape peel, modified edge lift-off (m-ELT), and bump shear. The tape peel test is based on ASTM D-3359-93. In this test, the film is scratched with a set of parallel blades to form a 10 x 10 grid of 1 mm squares. The rating is a semi-quantitative description of the damage after attachment and removal of the tape. Ratings range from 0B (large portions of the film delaminated) to 5B (no film loss or film damage). Films are tested dry (no temperature/humidity stressing) and after a pressure cooker test (PCT). The conditions in the PCT are: 121°C, 2 atmospheres pressure, 100% relative humidity.

m-ELT is a fracture mechanics-based approach to adhesion measurement. A thick layer of epoxy is applied on top of the device to be tested. The structure is cleaved to expose an edge, and the temperature of the device is lowered until the stress in the system causes the applied energy to exceed the fracture energy. The structure will fail either adhesively (peeling),

Table 3. BCB adhesion to aluminum with different plasma pre-clean procedures.

Pre-clean procedure	Bump shear force (g)
Control (water rinse)	49.1 ± 2.6
O <sub>2</sub> /CF <sub>4</sub> plasma	42.5 ± 3.5
O <sub>2</sub> plasma	60.7 ± 3.7

Table 4. Sensitivity of BCB interfaces to etch chemicals.

Interface	Etch chemical	Result
BCB on SiO <sub>2</sub>	HF	Delamination
BCB on SiO <sub>2</sub>	HCl or H <sub>2</sub> SO <sub>4</sub>	No delamination
BCB on Cu	HF	No delamination
BCB on Cu	HCl or H <sub>2</sub> SO <sub>4</sub>	Delamination

or cohesively (fracture inside one of the layers). Interfacial energy (Kc, in units of MPa\*m<sup>0.5</sup>) is calculated based on the temperature at which the structure fails. This test has been described in more detail elsewhere (1).

In the bump shear test, squares of BCB (typically 100µm x 100µm) are patterned on the substrate and are sheared off with a bump shear tool. The adhesion value reported is the force required to shear or fracture the bump. This test method has been described by Toepper et. al. (2).

## 8. BCB on top of metals

### 8.1 Aluminum

While excellent adhesion to aluminum is usually seen, this surface is especially sensitive to surface contamination, and the pre-clean procedure is very important (see section 5 and Table 3). If using an O<sub>2</sub>/CF<sub>4</sub> pre-clean procedure, care must be taken to ensure that the aluminum surface is not converted to a high proportion of aluminum oxyfluoride, which may degrade the adhesion. Note that if you are



integrating more than one layer of photo-BCB with aluminum, the first BCB layer will require a descum step with a fluorinated plasma, and this step must also leave an undamaged aluminum surface for the next layer of BCB.

Adhesion to aluminum can be improved by baking the adhesion promoter. Figure 4 shows adhesion of Cyclotene 4024-40 to aluminum vs AP3000 bake temperature. In general,  $K_{1c}$  values above 0.30 are associated with good adhesion and reliability.

AP8000 is not effective as an adhesion promoter for BCB on aluminum, and the use of AP8000 with aluminum is not recommended.

Table 5 shows adhesion of photo-BCB to various substrates, including aluminum, with and without AP3000 and with and without AP3000 bake. Adhesion is clearly enhanced by the adhesion promoter bake. These adhesion measurements were by tape peel test.

**8.2 Copper**

Figure 5 shows adhesion of Cyclotene 4024-40 to copper vs bake temperature. There is an improvement in adhesion when the bake is applied.

Issues have occasionally been seen with adhesion of BCB to the side wall of high aspect ratio copper lines. This is believed to be related to the difficulty of cleaning these narrow trenches. RIE may be relatively ineffective, especially if the side wall slope is retrograde.

Table 5 shows tape peel adhesion data for photo-BCB on copper, with and without AP3000, and with and without an AP3000 bake.

**8.3 Titanium**

Table 6 shows adhesion data for Cyclotene 4022-35 to titanium. Adhesion of BCB to Ti is poor without adhesion promoter but is quite good when AP3000 is used, particularly when the AP3000 is baked.

**8.4 Gold**

BCB does not adhere well to gold, or to other noble metals. BCB has been successfully used to completely cover gold lines and features, but

if an opening in the BCB lands on gold, loss of adhesion may occur. The most likely explana-

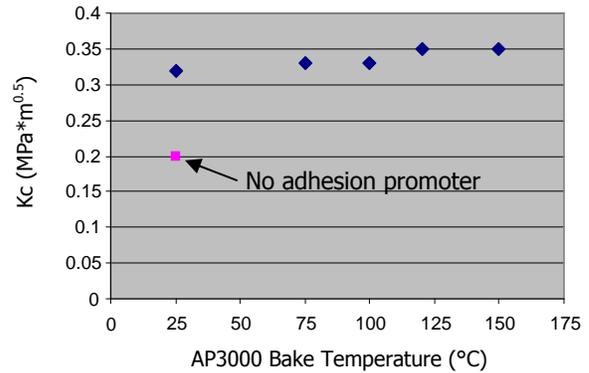


Figure 4. Adhesion of Cyclotene 4024-40 to aluminum, with and without a 60 second AP3000 bake (m-ELT data).

Table 5. Adhesion of photo-BCB to various substrates by tape peel test, after a 96 hour pressure cooker test.

Substrate	No AP3000	AP3000, No bake	AP3000, Bake ≥75°C
Aluminum	0B	3B	5B
Copper	0B	4B	5B
SiO <sub>2</sub>	0B	5B	5B
SiN <sub>x</sub>	5B	5B	5B

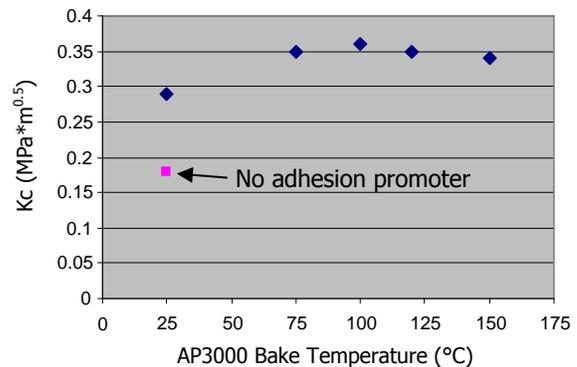


Figure 5. Adhesion of Cyclotene 4024-40 to copper, with and without a 60 second AP3000 bake (m-ELT data).



tion is that since gold does not form a surface oxide, there are no oxygen atoms on the surface for the silane to bond with. It is recommended that the BCB on gold interface be avoided if at all possible, and an intermediate layer (e.g., silicon nitride) be used to provide a surface for BCB adhesion.

**9. BCB on top of inorganic materials**

*9.1 Silicon nitride*

Figure 6 shows the adhesion of Cyclotene 4024-40 to silicon nitride as a function of AP3000 bake temperature. Table 5 shows data for BCB adhesion to nitride, with and without AP3000 and with and without a bake.

*9.2 Silicon oxide*

Figure 7 shows the adhesion of Cyclotene 4024-40 to silicon oxide versus AP3000 bake temperature. Table 5 shows data for BCB adhesion to oxide, with and without AP3000 and with and without a bake.

R. Dauskardt et. al. have studied the sub-critical debonding of Cyclotene 3022 to silicon oxide using double cantilever beam and 4-point bend test structures (3,4). While both adhesion promoters greatly improved the adhesion of BCB to oxide, they found slightly better adhesion with AP3000 than with AP8000.

*9.3 III-V semiconductors*

BCB adhesion to III-V semiconductors (GaAs, InP) tends to be marginal, even with the use of AP3000. As with gold, it is best to avoid this interface and employ an intermediate layer (e.g., silicon nitride or Ti) to which BCB has good adhesion once an adhesion promoter is applied to BCB.

**10. BCB on top of organic polymers**

*10.1 BCB to BCB*

Cure management is important when stacking multiple layers of BCB. For the top layer of BCB to properly stick to the layer below it, the bottom layer must be partially cured (75 – 85%). Figure 8 shows BCB-BCB adhesion as a

function of the cure temperature of BCB1 (2). At low cure temperatures (% cure = 77-85%)

Table 6. Adhesion of Cyclotene 4022-35 to Ti by tape peel test

Surface	m-ELT	Tape peel	
		Dry	48 hr PCT
No AP3000	0.14±0.04	0B	0B
AP3000, no bake	0.31±0.02	5B	4B
AP3000, baked	0.35±0.02	5B	5B

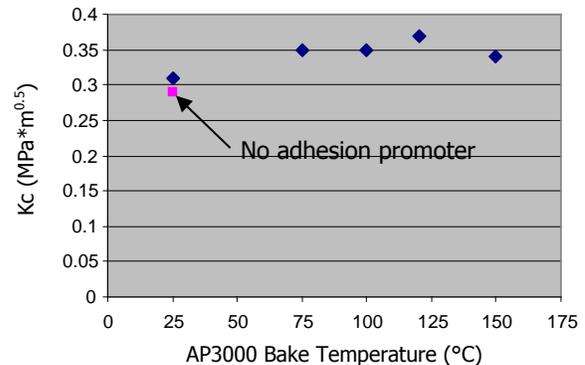


Figure 6. Adhesion of Cyclotene 4024-40 to silicon nitride, with and without a 60 second AP3000 bake (m-ELT data).

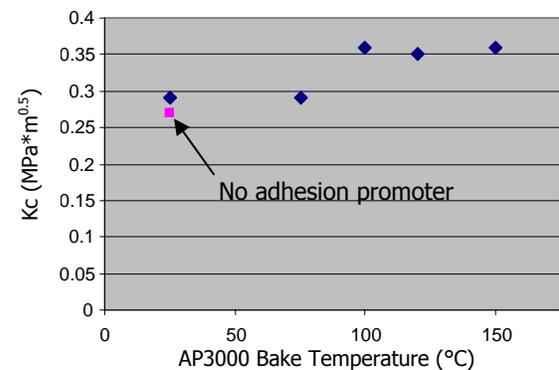


Figure 7. Adhesion of Cyclotene 4024-40 to silicon oxide, with and without a 60 second AP3000 bake (m-ELT data)



the adhesion is excellent; as the cure level increases the polymer-polymer adhesion degrades rapidly.

Because there is some silicon in the BCB polymer backbone, an oxidizing plasma process (e.g., O<sub>2</sub> plasma) can leave a thin layer of SiO<sub>2</sub> on the BCB surface. This layer has poor mechanical properties, and will adversely affect the adhesion of BCB (or anything else) deposited on top. Descum or plasma etch recipes must be optimized such that the silicon etch is balanced with the organic polymer etch, and silicon does not accumulate on the surface.

Adhesion promoter appears to have little effect on BCB-BCB adhesion. Normally, in a multilayer construction, the BCB will contact metals or other materials in addition to the underlying BCB layer. In the unusual circumstance where BCB is deposited over a blanket film of BCB, adhesion promoter is not required.

### 10.2 BCB to Polyimide

We have studied the adhesion of Cyclotene 4024-40 to polyimide. The polyimide was subjected to a dehydration bake at 150°C and an O<sub>2</sub> plasma treatment prior to BCB coating. The BCB layer was fully cured. Tape peel tests gave 5B ratings (excellent adhesion) immediately after cure as well as after a 48 hour pressure cooker test. Adhesion of BCB to polyimide appears to be strong. We believe the oven bake and O<sub>2</sub> plasma treatment are important in obtaining good adhesion between the polymers.

Note that there are several manufacturers of polyimides, and many different grades. We have not tested them all and cannot guarantee the same quality of adhesion to all polyimide types.

## 11. Metals on top of BCB

### 11.1 Sputtering

The most important consideration in ensuring good adhesion of a sputtered metal film to BCB is that a sputter etch is required. The sputter etch, typically argon, should be done *in situ* without breaking vacuum. A 100Å equivalent

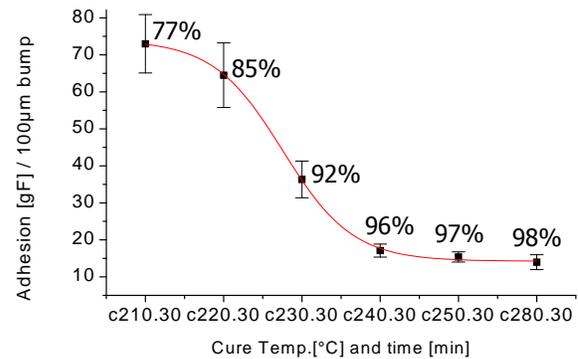


Figure 8. BCB-BCB adhesion vs BCB1 cure temperature, measured by bump shear test. The degree of cure of each point is indicated on the graph.

silicon oxide etch is very common and gives reliably good adhesion.

BCB surface damage by an oxidizing plasma can weaken the bond between metal and BCB significantly (see section 9.1). Minor surface oxidation might be repaired by the sputter etch, but significant surface oxidation will likely result in decreased adhesion.

Aluminum can be sputtered on top of BCB directly; no intermediate layers are needed. Copper generally does not adhere well when sputtered directly on BCB, so another layer is needed here. The adhesion layer is typically a thin layer of Ti or Ti:W. Shieu and Shiao (6) found poor adhesion of copper when either the argon ion pre-treatment or a Ti adhesion layer was omitted, whereas when both of these were employed the adhesion was quite strong. Evidence of direct bonding between BCB and Ti was seen in the form of Ti-C bonds at the BCB-metal interface.

Gutmann et. al. found that a Cu-C film (1-2% C) improved the adhesion of copper to BCB (7). They also found that the adhesion was better after damascene processing of the BCB, which they attributed to increased surface roughness and chemical modification of the BCB surface due to plasma etching.



Chromium can be sputtered onto BCB, and adheres well. Sputtered chromium, however can be highly stressed, and metal cracking can occur unless sputtering conditions are carefully controlled. It is generally preferred to use Ti or Ti:W instead of Cr.

### 11.2 Evaporation

In general, the metals that can be sputtered can also be evaporated. The need for an *in situ* sputter etch still holds, however. This means that the metal evaporation system should be equipped with an argon ion gun or ion mill. Table 7 lists the adhesion of Ti, Cu, and Cr under different deposition methods.

BCB surface damage by an oxidizing plasma can weaken the bond between metal and BCB significantly (see section 9.1). Minor surface oxidation might be repaired by the ion milling treatment, but significant surface oxidation will likely result in decreased adhesion.

## 12. Inorganics on top of BCB

### 12.1 Silicon nitride

Integration of PECVD silicon nitride on top of BCB has been implemented in production by several customers.

It is important to remain within the thermal budget of BCB, otherwise thermal degradation of the polymer may result. The deposition temperature should be no higher than 350°C, preferably lower.

The adhesion of nitride to BCB tends to be better when N<sub>2</sub> rather than NH<sub>3</sub> is the nitrogen source. NH<sub>3</sub> plasma can damage the BCB surface. If SiH<sub>4</sub>/NH<sub>3</sub> chemistry is used, the NH<sub>3</sub> pre-deposition time should be as short as possible; preferably, N<sub>2</sub> should be introduced to the chamber first, then NH<sub>3</sub> as the plasma stabilizes.

### 12.2 Silicon oxide

A study of the adhesion of PECVD silicon oxide (SiH<sub>4</sub> + N<sub>2</sub>O) to dry etch BCB found that a nitrogen plasma pre-treatment greatly improved the adhesion of the oxide (8). Pre-treatment

with NH<sub>3</sub> or N<sub>2</sub>O plasma, by contrast, yielded no improvement at all in adhesion. As with silicon Table 7. Adhesion of metals to BCB vs pre-deposition surface treatment.

Metal	Deposition	Pre-treatment	Tape peel result
Ti	Sputter	none	0B
Ti	Sputter	Ar sputter etch	4B
Ti	Evaporate	Ar ion gun	4-5B
Cr	Sputter	None	3B
Cr	Sputter	Ar sputter etch	4B
Cr	Evaporate	Ar ion gun	4B
Cu	Sputter	None	0B
Cu	Sputter	Ar sputter etch	0B
Cu	Evaporate	None	0B
Cu	evaporate	Ar ion gun	2B

nitride deposition, it is important to stay below the thermal decomposition temperature of BCB (about 350°C).

## 13. Organic Polymers on top of BCB

### 13.1 Underfill

Underfills, typically silica-filled epoxy resins, are used in flip chip applications. Researchers at Georgia Tech (9) studied the adhesion of several commercially available underfill materials to BCB by die shear testing. They found generally very good adhesion. Failure analysis of the sheared parts revealed mixed mode failures, with adhesive failure at the BCB/SiO<sub>2</sub> or BCB/nitride interface, adhesive failure at the BCB/underfill interface, and cohesive failure of the underfill all present.

Dimke and Edwards (10) studied adhesion of underfills to several difference passivation materials. They saw a large variation in the results depending on the choice of underfill, and the optimum choice was dependent on the type of passivation material. There are many commercially available underfill formulations, and it is important to work with the underfill



supplier to select the product most compatible with BCB.

Researchers at Stanford University also studied BCB adhesion to commercial underfill resins (11). Failure occurred predominantly at BCB-nitride, not at the BCB-underfill interface. Despite this, the adhesion values varied considerably depending on which underfill was used, again highlighting the importance of underfill selection. They found the adhesion values to be higher with partially cured BCB than with fully cured BCB.

### 13.2 Molding Compound

Molding compounds, like underfills, are filled epoxy resins. Problems with this interface are unusual. We have seen that the adhesion of molding compound to partially cured BCB is stronger than to fully cured BCB. A UV-ozone treatment of the BCB surface improves the adhesion. Table 8 provides data on these parameters. The test is similar to a m-ELT test; the thickness of the molding compound is varied and the device is inspected for failure at -196°C. A thicker molding compound layer without failure indicates better adhesion.

Table 8. Adhesion of Cyclotene 4026-46 to epoxy molding compound. Failure was only seen with full cure and no UV-ozone treatment.

Mold cmpd Thickness	BCB cure	UV-ozone	Failure at -196°C?
178µm	98%	No	No
184µm	98%	No	Yes
199µm	98%	No	Yes
199µm	98%	Yes	No
199µm	82%	No	No
231µm	82%	No	No
248µm	82%	Yes	No
318µm	98%	Yes	No

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