



DJ MICROLAMINATES, Inc
Dry Film Sheets

TIPS & TRICKS for SUEX PROCESSING

The following Tips and Tricks are a combination of questions and answers, best practices and suggestions based on the experience of SUEX users – including us.

QUESTIONS:

- Do you have SUEX parts falling off in the developer?
- Do you see discoloration under sections of the SUEX images?
- Has the SUEX pattern lifted along pattern edges or on fine features during development or after setting overnight after drying?

These questions mean you probably have an adhesion issue. If many, or all of the parts fall off in the developer then you have a major issue. Lesser problems may show up later. Color along the edges of the patterns or randomly over the SUEX surface or adhesion failure after plating or upon standing are indicative of resist delamination and will require better substrate prep/cleaning, higher doses, slow cooling after bakes and a hardbake quickly after rinse and dry. The delamination issue is partly due to the size and/or shape of your structures - large, thin square or round rings, large area features, very fine structures all have their problems. Delamination of the circles and squares rings are problematic because of the stress of the large feature size and small surface contact area to the substrate. However, the cause could be a wide number of things which can affect the adhesion to the substrate not just the feature characteristics. Cracks on the SUEX surface are a different issue. Both, however, are related to the residual stress formed in the resin coating during the curing process because the bond lengths shrink during crosslinking.

DATA SHEETS

The Data Sheets are written essentially for processing on typical Si substrates and new, clean silicon generally works very well. However, other substrates and other than pristine Si can have issues. So, let's start from the beginning of the process with some tips and suggestions to help.

The primary recommendation here is better preparation of the substrate and slow cooling (stress release) of the freshly cured parts.

QUESTIONS:

- What substrate are you working on?
- Did you do a chemical clean of the substrate?
- If you acid treated the copper, did you neutralize the acid before drying?

- Have you used a plasma treatment on the substrate surface?
- What power? Have you used a lower temperature (85°C) PEB?
- Did you cool the laminate down very slowly over 3-5hrs after the PEB?
- How did you dry your substrates after develop/rinse?
- Do you see rings or delamination under the SUEX features immediately after develop, after drying or only after standing a day or so?

If the features continue to delaminate or if you see delamination during develop you will need to work on the process more.

- Is this a copper or glass substrate?
- Are you using other difficult substrates which are known to have poor adhesion such as gold or nitride?
- What substrate cleaning and surface preparation did you do?

Here are our suggestions to remedy these issues. Over the years we have modified our process conditions from the original Data Sheet somewhat to give us what we think are the best conditions for adhesion. However, most do impact throughput as they usually require extra steps or longer processing times.

SUBSTRATE CLEANING

Cleaning of your tools: We use just acetone for removing fingerprints and resist from most of our processing items and either let it evaporate, blow dry with a soft stream of air or dry with a cleanroom wipe. The same goes for the mask as well. IPA is not a good solvent for residual resist and will tend to smear the surface, thus use acetone.

Cleaning/prep of the substrate: If there is a trace of acid on the substrate surface – or even a possibility of a trace of acid – we recommend a 5-10sec dip in 5% NH₄OH to neutralize the acid followed by a copious DI rinse or a quick wash with IPA and oven drying at approx. 100°C for a few minutes. Residual acid on any surface can cause premature crosslinking of a thin surface film at the substrate surface of the resist during handling or baking. Anything else that could act as an acid could also cause problems during the PEB. If ammonia is used to neutralize the residual acid from substrate pretreatment the basic ammonia could neutralize the photoacid, preventing cure. Fortunately, the ammonia evaporates quickly. We don't know of anything else that could poison the SUEX and inhibit crosslinking.

DIFFICULT SUBSTRATES

Copper: Cu is a very different animal and requires very different treatment to get the best adhesion. If you are using newly sputtered substrates cleaning with acetone/IPA followed by a 120-150°C dehydration bake may be sufficient. However, after the initial cleaning we have found that an O₂ plasma in a high power oxygen etching system at

450-600dB Rf power for 3-5 min or up to 10 min is most effective in activating the substrate surface and improving subsequent adhesion prior to lamination. Also, O₂ plasma treatment of a nitride surface for several minutes at 450-600dB prior to lamination may well help as well.

Gold: Au typically shows even poorer adhesion than Cu so you may have to work on getting better treatment for Au. Generally only O₂ plasma is used. We have heard that an SnO_x, Ti or TiO_x layer can help, if needed. Au is a bit more reflective than Cu so a lower dose compared to Cu should be needed, however.

Nitride: As for a nitride surface, which is known to exhibit poor adhesion to many materials, my suggestion would be to cover the nitride with a different coating such as oxide that would exhibit better adhesion to the SUEx. The other approach would be to treat the nitride prior to lamination of the SUEx to leave a surface coating on the nitride that would do the same thing. Delamination could also be indicative of under exposure.

The two biggest treatments used to improve adhesion are the O₂ plasma activation pretreatment and the slow-cool down after the PEB. Also heating in ovens offers significant advantages over a hot plate for all process steps as you more uniformly heat the films in an oven. On a hot plate the substrate side gets significantly more heat than the top of the SUEx film and the differential treatment will lead to differential curing of the film. The film also tends to skin over which inhibits proper development action.

We recommend laminating within 8hrs of the plasma treatment; however, in most cases up to 24hrs is acceptable.

LAMINATION CONDITIONS

Carrier/Cover sheet:

You will need a rigid or semi rigid sheet to carry the substrate through the laminator as well as a protective cover to prevent SUEx resin from being transferred to the laminator rollers during the lamination process. We have found the most durable carrier sheet to be a 1ft X 1ft (300mm X 300mm) square, (.019" (500µm) thick Plain Aluminum Sheet available from Amazon.com. For the cover sheet we use a 12" (300mm) square piece of 5mil (125µm) PET film or a standard size sheet of an overhead transparency. These can be taped together along one edge like a hinge so that the cover can be easily lifted to place the substrate/SUEx between the two.

Question: What temperature are the rollers?

Prior to first use, physically measure the temp between two rollers with a thermocouple wire? In some cases have found large difference between the set temperatures and the actual temp, particularly with the SKY laminators.

Typically, 60-65°C lamination temperatures are what we use. Some people like a bit hotter, around 70°C. Do not get the rollers too hot or you will squeeze out a bit of resin which will adhere to the rollers or the carrier sheet and will then both will need to be cleaned. Do not lay the SUEX sheet directly on the substrate prior to lamination. The heat from the laminator rollers will soften the sheet prior to laminating causing it to sag and trap air pockets between the SUEX and the substrates before it passes thru the rollers. These air pockets become locked in after the lamination rendering the laminated substrates unusable. Use a PET spacer sheet to separate the SUEX layer from the substrate until approx. 1cm from where the rollers meet.

For more lamination details please see our Lamination Video on our web site at www.djmicrolaminates.com.

POST LAMINATION BAKE

In general, we do not do a post lamination bake as we find that it does not make much difference in adhesion. However, some people claim that it gives a bit better adhesion.

EXPOSURE

To reduce mask sticking problems you should have an anti-stick coating applied to your mask by your mask house. Since SUEX films can be somewhat tacky, especially if warm or in warm rooms you should reduce the contact pressure as well. You will get much better images and much better side walls if you use an i-line filter or soda lime glass mask for exposing and increasing the exposure dose slightly as per our data sheet. Choose the proper dose for your thickness and substrate from Table 1.

Also note that the no-filter exposure will lead to undercut profiles because the shorter exposure wavelengths are significantly absorbed during exposure and will only partially pass through the resist film; thus the top of the SUEX gets a wider exposure profile than at the bottom of the sheet.

The laminated substrates should be cooled to 18-25°C for exposure, then the PET film removed just prior to exposure. Copper, glass and gold are quite non-reflective and will therefore need a much higher dose than our recommendations on Si, probably 2X, if not more. Aluminum will require less dose. Note that as a negative resist, the lines will always be larger than the mask for proper cure. Also, thicker resists and large sized features will have more adhesion problems as the residual stress increases with the volume of resist on the substrate and will require more overexposure. For SUEX K200 on a Cu panel, which is quite non-reflective, we typically work at 2500-2750mJ/cm². But different Cu substrates or different Cu treatments can require more or less as the reflectivity changes.

Direct write, LDW exposure systems, 365nm to 395nm, ($\sim 5 \text{ mW/cm}^2$ for 6 min = 1800 mJ/cm^2) work very nicely. The 375nm lasers will require more energy and 395nm lasers substantially more.

After exposure you can store the exposed substrates for up to a month or more before PEB and development.

PEB CONDITIONS

Question: Are you baking on a hot plate or in an oven?

We find much better uniformity, particularly for thicker films, with an oven and no surface skinning. We have found no benefit to the common slow ramp up or the two-temperature process for the PEB, just put it in an oven at temperature. Oven bakes are strongly recommended and we use a flat ceramic tile or glass placed in the hot oven to act as a heat sink then place the substrates flat on the surface. We do not use a hot-plate for the PEB. It creates stress patterns within the polymer film. Also, do not PEB the substrates placed vertically in a wafer carrier as the SUEX may flow giving bent structures.

We also do not use the two-step bake, 5min at 65°C , ramp up and then 5 min 95°C for PEB but do not find it to be detrimental. We prefer to use an $80\text{-}85^\circ\text{C}$ PEB for 30-60min or more instead and even lower temperatures for longer times if tenting. Also, when tenting we recommend baking face down on a non-stick surface to minimize sagging of the SUEX film into the cavity. A slow cool down rate is highly recommended ($<5^\circ\text{C}/\text{min}$ is best) and we don't take the substrates out of the oven for 3-5 hrs (temp $<45^\circ\text{C}$) to allow for maximum stress relaxation. Also, it is best to let the substrates rest overnight before developing to really minimize the residual stresses. With thinner films these precautions may not be as necessary. The key is not to cool the wafers rapidly on a cold surface.

After PEB, the wafers need to be developed within a day or two, otherwise you may see delamination due to the CTE difference between exposed and unexposed regions.

CRACKS

Cracks are typically only surface cracks but look much worse. They are very common with epoxy resists and are more prevalent with a hot plate bake process. They are also more prevalent if the dose is too low. You will find far fewer, more shallow cracks with the slower cool-down process. They are also frequently not seen if one waits overnight after PEB before development.

DEVELOP

We recommend face down development on a coarse (4 mesh) screen with little or no agitation. Face down makes for a faster development as the resist laden developer will fall out of the trenches rather than collecting at the bottom of the trench, even with agitation. It will also lead to cleaner trenches. We use a stainless steel screen with a 4mm mesh that was obtained on-line from Amazon.com. For agitation we use a very small or micro magnetic stirrer at a 200-300 rpm stir rate. We also recommend a 2-bath system, where the first bath will contain most of the dissolved unexposed resist and the 2nd bath remains relatively clean. Use two sequential developer baths doing the approximately 2/3rds of the development time in the bath one and the remainder in bath two. This will minimize dissolved polymer in the developer prior to rinse.

You will want to change the baths more frequently as there will be much more dissolved resist than you would commonly work with. When changing baths replace Bath 1 with Bath 2 and prepare a new Bath 2. It will also help to quickly wash the substrates with developer from a squirt bottle when transferring from Bath 1 to Bath 2 and letting the wash drain back into Bath 1. You should rinse with IPA after Bath 2 into a waste container before rinse to minimize drag-over to the IPA rinse. If you see any white haze or precipitation upon contact with the IPA you have not developed long enough or your developer bath is overloaded with dissolved polymer.

The develop time strongly depends on the resist thickness and develop process. Face down development with minimum agitation requires the least develop time. Face up needs the longest. Agitation gives uneven development. For K200 you will need 60 min total develop time (40min in a "used" Bath 1 and 20 min in a "clean" Bath 2). Wash the substrate quickly with IPA from a squirt bottle into a waste container to remove most of the developer before the rinse bath, which I do for 5 min. Rinse in IPA for 1 – 5min immediately after development is complete. Longer rinse times are recommended for higher aspect structures.

DO NOT blow dry the substrates after Rinse. This can cause SUEx structures to lift from the substrate and delaminate. We suggest immediately placing the IPA wet substrate vertically in a wafer carrier then immediately into an oven at ~100C before it starts to dry. I like to bake dry in a 100C oven for 15-30 min but time is not critical.

HARDBAKE

If the substrates look good after drying immediately give them a hardbake, the sooner you hardbake and the higher the hardbake temperature the better, up to 200°C for 1-2hr. This has been found to minimize edge delamination of the features. For quick results immediately bake a minimum of 125°C for 30min or more. This will almost eliminate any further delamination over time (days, weeks). For plating applications, a 125°C hardbake will normally suffice but for permanent applications where thermal

cycle testing is required a 200°C hardbake for 1-2hrs may be needed. Substrates can also be annealed to some degree by baking at 125-150°C for 15-30min, but with proper processing should not be needed.

COLOR & TRANSPARENCY

After development the SUEX film is very transparent but slightly yellow in color, but hardbaking will darken the crosslinked resist, particularly at higher temps. For SUEX there is only a slight color change up to approx. 150C for 1hr where it is still quite transparent but a light orange-red color. At 175C for 1hr it is still transparent but now considerably more colored and at 200C even more so.

RELEASE LAYERS

SUEX is essentially a permanent resist. After curing it is not possible to remove the SUEX structures from the substrate. Some users have successfully used release layers under the SUEX films for this purpose but only in limited applications. Using a release coating may work but will be a challenge. In preparing release layers on a substrate one probably won't be able to use the relatively poor adhesion of SUEX to glass to advantage as the resist will likely lift during the processing steps. However, Processing the SUEX sheets on a polyester substrate works quite nicely. We can supply SUEX films on special polyester substrates. Our recommended approach for removing the SUEX would be to remove the SUEX post processing. We are developing a laser based process for removing the SUEX after processing or plating even on large size substrates. The removal process will be capable of removing the cured SUEX features within an acceptable time frame once we have perfected the conditions.

CROSSLINK CONVERSION AND RATE

Data is available to show the crosslinking conversion vs time and temperature of laminated SUEX films. This data shows that the crosslink conversion at a 150C bake reaches a maximum after about 30min and that the conversion gets only slightly greater at higher temperatures but will reach a maximum much faster. However, at 95C bake the conversion is much slower and will never make even 50% in several hours. Yet, we know that only 5-10min at 95C is enough to give enough crosslinking to give good resolution after development. From this data we also estimate that a 125C bake would eventually reach 50-60% conversion. A hardbake at 200C will reach 75% conversion in 12-13 min and a maximum conversion of 80% in 20min. Longer heating will provide further removal of moisture and volatiles for up to 2hrs. Upon standing the hardbaked film will reabsorb moisture from the atmosphere and will reach an equilibrium level of about 1% in 24hrs.

Please email us if you would like more information: technical@djmicrolaminates.com