PL 177

General-Purpose
Positive Resist

Clariant

General

PL 177 is a positive tone liquid resist for the application in various coating techniques. *PL 177* can be used in all those places, where layouts are directly to be copied onto and subsequently transferred into a substrate by etching, plating, sputtering and related processes. The essential features of **PL 177** include:

- high resolution potential
- good drying behaviour
- aqueous-alkaline processability
- halogen free coating solvent
- possibility of multiple exposure (selective plating process) light or bright day light should be avoided.
- storability of coated substrates
- blue coloured for easy inspection

PL 177 can be applied by dip coating. By dilution with suitable solvents *PL 177* can be adjusted to also meet the viscosity and drying requirements of other coating techniques (spray coating, roller coating, spin coating). *PL 177* is resistant towards acidic and ammoniacal etchants as well as acidic and neutral plating baths. The final removal of the temporary resist layer, the stripping, is done with low concentrated bases. *PL 177* is used in the manufacturing of printed circuit boards, multilayer inner flexible boards and in chemical milling.

Physical properties

Solid content	36 %
Viscosity at 25°C	l35 mm²/s
Absorptivity at 398 nm	0.92 l/g*cm
Water content	max. 0.5%

Storage Conditions

Liquid Resist *PL 177* is to be stored in sealed original containers at temperatures below 25°C. The shelf life under these conditions is a minimum of 1 year from date of manufacturing. *PL177* contains inflammable solvents. Respective safety measures are to be regarded when handling *PL177*.

Illumination of working areas

Because of its light sensitivity *PL177* and *PL 177* coated substrates should be handled under yellow safety lights. Direct exposure to sun light or bright day light should be avoided. For the illumination of working areas yellow fluorescent lamps are recommended, e.g. Philips 1.2 m TL-D 36 W-1 6, the emission of which does not affect dry film performance. Windows are to be coated by a non-bleachable yellow film which has to be in-transparent for light of wave length below 450 nm. Instead yellow plexi glass plates like Röhm's type Yellow 303 can be used.

Clean Room Facilities

In general the application of resists of lower thickness results in a more pronounced influence of contaminating particles on the production yield as compared with standard 38 µm dry film resist technology. Especially critical with respect to particle or dust inclusion in the resist are the process steps of coating and drying. A reduction of particle concentration at reasonable cost can be achieved by application of clean room technology in an object scale, just limited to the respective equipment. Depending on technological requirements a clean room class 100 to 10.000 area is suggested.

Pre-treatment of Surfaces

To assure optimum adhesion on metallic surfaces these have to be free of grease and oxides. This can be assured by mechanical or chemical means.

Coating

For typical applications *PL 177* is applied in film thicknesses between 3 and 10 µm. For these thicknesses commercial equipment is available for the following coating technologies:

- spray coating
- dip coating
- roller coating
- spin coating

PL 177 can be adjusted in concentration and viscosity to meet the requirements of all of the above techniques.

General Remarks to Viscosity Adjustment

It is advisable to dilute the liquid resists immediately before application. In the simple case this is done by vigorous shaking of resist concentrate and thinner in a closed bottle, which should not be filled by more than 75 % of its volume. In any case dilution must result in a homogenous solution. Air bubbles thus included in the liquid normally are easily removed and do not lead to any coating failure.

The replenishing of diluted resist into the coating equipment must be done with a thoroughly homogenated material of well-adjusted viscosity. Dilution must not be done within the coating equipment (dipping vessel, spraying tank) but rather in a separate vessel. The degree of dilution and the choice of thinner depend on the coating technique and the desired resist thickness. Solvent losses due to evaporation are to be compensated with the thinner in use.

When using higher boiling thinners the drying process has to be adjusted depending an the extent of dilution and the resist thickness. The parameters are to be checked by pre tests for the respective case.

Storage of coated substrates

Coated substrates are to be stored in the dark and - especially for long term storage – at reduced temperature. Under these conditions storage over several weeks has proved to be uncritical with respect to resist performance.

Viscosity Control, Filtration, Selection of Equipment and Material

Since *PL 177* contains low boiling solvents, it is recommended - especially for dip coating - to keep the coating vessels closed when not in use. A regular control of the viscosity of the resist is recommended. Characteristic data for the dilution of *PL 177* with *AZ EBR Solvent* and the respective viscosity values are given below.

Viscosity reduction of *PL 177* by dilution with *AZ EBR Solvent*

Dilution by volume (resist : solvent)	1 : 0.0	1 : 0.1	1 : 0.2	1 : 0.3	1 : 0.4	1 : 0.5
Viscosity [mm²/sec]	135	70	45	30	20	16

When controlling viscosity it has to be considered that it is strongly dependent with temperature. The table below gives the viscosity of undiluted *PL 177* in the temperature range from 20 to 35°C.

Temperature dependence of the viscosity of *PL 177*

Temperature [°C]	20	22	25	27	30	33	35
Viscosity [mm²/sec]	177	155	135	118	100	84	75

In practice viscosity control by measuring the drain time of standardised cups has proven useful. For the viscosity range of interest *Zahn Cup 2* or *Zahn Cup 4* is recommended.
Especially in continuously working production facilities increasing contamination of the liquid resist mainly by particles from the substrate material is observed. It therefore is recommended to slowly pump the liquid resist through an appropriate filtration module. The filter size depends on the product specification, good results have been obtained using a 10 µm curled filter.
All equipment and material used in production must withstand the used solvents. Appropriate materials include glass, stainless steel and polytetrafluoro ethylene. The coating rollers should not consist of Viton or EPDM rubber, while the use of butyl rubber offers advantages. In case of doubt the resistance of the material is to be checked in a pre test.

Resist thickness [µm]	4	6	8	10
Yield [m²/litre]	90	60	45	36

These calculated values refer to coated area and are to be halved for double sided coating of the substrate.

Coating

In the following suggestions are given on how to dilute for the various coating techniques. Depending on the specific application the indicated parameters can differ reasonably from the given values. It is therefore recommended to countercheck the optimum dilution conditions prior to running the process.

Spraying

For cylinder and flat panel coating a fast drying formulation has proved useful, which is obtained from

1.0 volume part *PL 177*
1.0 volume part Methylethylketone (MEK) and
1.0 volume part *AZ EBR Solvent*

Alternatively dilution with pure medium boiling solvent is possible, like:

1.0 volume parts *PL 177*
2.5 volume parts butyl acetate.

The use of solvents with low MAK value (high toxicity) is to be avoided, especially considering dip coating. The spraying process (spray nozzle diameter, spraying air pressure and distance spraying head to substrate) is to be optimised for any specific situation.

Dip Coating

can be used for the coating of single or double sided copperclad material without metallized through holes. Good results are obtained with the following formulations:

for higher lifting speeds (resist thickness 5.0 µm at 40 cm/min.):
1.0 volume part *PL 177* and 0.5 volume parts of Methylethylketone (MEK)
for lower lifting speeds (resist thickness 3.0 µm at 20 cm/min):
1.0 volume part *PL 177* and 0.7 volume parts of Methylethylketone (MEK)

For printed circuit boards with through holes, dip coating is not suitable. To avoid any coating failure the dip coating equipment must be installed shock free. The lifting of the substrate should be performed pneumatically.

Roller Coating

PL 177 can be used on all commercial roller coaters with ungravured rollers, using the original concentration.

Spin Coating

For spin coating of *PL 177* on commercial spin coating equipment the following minimum dilution is suggested:

1.0 volume parts *PL 177*
0.5 volume parts *AZ EBR Solvent*

The desired resist thickness is obtained by adjustment of the rotational speed. Spinning is to be continued until the resist is sufficiently dried to avoid drawing back of the resist to the centre of the substrate.

Drying

The coated substrates can be dried in IR-, hot air or combination ovens, both of stand-alone or conveyorised type. For resist thicknesses of 3 to 6 um, drying times of 10 to 20 min at 70 to 90°C are recommended. For other thicknesses and in dependence on the dilution by higher or lower boiling solvents other drying parameters have to be used, which are to be determined by test runs.

Not sufficiently dried resist layers can result in sticking of the phototool in the subsequent exposure step or in bubble formation during the evacuation of the exposure frame. During development this can result in partial or overall loss of image. Over dried layers result in prolonged development times.

Exposure

Before exposure the substrates have to cool down to room temperature. *PL 177* has a maximum photo sensitivity in the spectral range between 340 and 420 nm. Good exposure results are obtained with iron doped or undoped mercury lamps as installed in almost all commercial exposure equipment. The values given in the table below can be considered as a guide line for the exposure of *PL 177* of thickness 7 µm with 5 kW lamps.

Guidelines for exposure of *PL 177*

Step Wedge BK01: Cu-free	1 - 2
Stouffer (21 Step): Cu-free	2 – 3
Exposure Energy (ORC-probe UV 350, through tool)	Approx. 100 mJ/cm²
Exposure time (iron doped, 5 kW, MO61 Sylvania)	Approx. 10 sec.
HI at 2.5 kW	8 sec.
POK at 2 kW	20 sec.

The sensitivity of *PL 177* (for the given optimum step wedge reproduction) depends on resist thickness and drying. The exposure time is to be determined in test runs to include the specific parameters of resist thickness, phototool and exposure equipment. We recommend the use of step wedge BK01 for this purpose. To avoid deviations due to the UV transparency of the applied phototool this is to be placed above the step wedge during the test exposure. Too short exposure times result in resist residues after development, which in subsequent process steps (etching, plating) result in failure.

Development

Exposed *PL 177* is developed with *AZ 351B Developer*, diluted 1:4 with water in an appropriate vessel (vertically or horizontally). The development time can be reduced by slightly wiping with a wad or a non-fraying cloth or by spraying with the developer solution. Any movement of the developer or the substrate will speed up the development. After development the image must be clean and free of resist residues, since otherwise subsequent etching or plating will result in failure.

Instead of *AZ 351B Developer* (1:4), pure diluted sodium hydroxide solution of 1 wt.% can be used. In this case the optimisation of the parameters for drying, exposure and kind and duration of development have to be done more thoroughly. When using commercial conveyorized equipment the use of NaOH is recommended.

The temperature of the developer should be between 20 and 25°C. Lower temperatures retard development, too high temperatures increase the loss of fine patterns. Underexposed layers result in slower development rate or even in residues which remain in the exposed areas.

The development has to be followed by a thorough water rinse (temp. > 18°C, nozzle pressure 1.0 - 1.5 bar) and subsequent drying. The pH value of the rinsing water should not be below 6.8 to avoid redepositing of dissolved resist components. The calcium and magnesium content ('hardness') of the water should be in a medium range (5 to 10°dH). Good drying after the development offers advantages in subsequent process steps.

It is of particular importance that no developer solution is allowed to dry on the substrate between development and rinsing. Testing for shadow free development can be carried out with sodium persulfate solution, with pre etch solution or with a chemically reductive tin bath.

For correctly exposed resist of a thickness of 4 to 6 µm, the development time in unloaded developer is between 30 and 60 sec. Thinner layers develop at much faster rate, while thicker resist requires longer development.
The aqueous alkaline developer is consumed by the up-take of carbon dioxide from the air. It is therefore recommended to close the development vessels after use or to place the developer in closed bottles.

The table below gives recommendations for a typical development process for *PL 177*.

Developer type and make-up (7 µm thick *PL 177*)	1.0% NaOH or alternatively *AZ 351B Developer* 1:4 diluted with D.I. water
Developer temperature	20 – 25 °C
Nozzle pressure	1 – 2 bar
Development time (1% NaOH, 25 °C)	Approximately 60 sec.

Should foaming occur during development with sodium hydroxide, a suitable anti foam agent can be added at a ratio of 0.2 to 1.0 ml per litre. To assure even distribution of the anti foam agent it should be added continuously to the tank by means of a feeding pump. Best results are obtained when the surface active agent is added to the backflow of the developer. Since the developer is free of organic solvents processors made of both stainless steel and plastic (PVC, PP) are suitable.

Thermal Cure

For the uncritical requirements of the resist in etching of easily etched materials in typical acidic or ammoniacal etchants no additional curing step is required. However, for etching of difficult to etch materials (like steel or steel alloys) requiring in general rather long etching times, an additional thermal cure of the resist is recommended. This also applies for subsequent plating processes in critical plating baths.

For difficult to etch metals a thermal cure of 10 to 30 min. at 160 to 190° C is suggested. Fine tuning of both curing time and temperature mainly depends on the etchability and the thickness of the metal. For critical plating processes curing of 10 to 30 min. at 120°C normally is sufficient.

Retouch

As the resist is non-soluble in pure aliphatic and aromatic hydrocarbons they can be retouched before etching, plating or sputtering. Retouch can be done with benzene based varnishes, like asphalt varnish. Deletions can be done with *PL 177* resist.

Etching

The residual resist after development is resistant towards all common etchants, thus yielding well defined images, conductor patterns or milled parts. Sufficient resistance is also obtained for ammoniacal etchants as long as the residual resist is protected from light. Concentration and type of etchant are to be chosen as to assure minimum undercutting in the etching step, while keeping etch times as short as possible. Etching can be performed at increased temperatures. Towards weakly alkaline or buffered alkaline solutions up to pH 9, *PL 177* shows limited or short-term resistance.

Plating

The residual resist after development is sufficiently resistant towards all common acidic and neutral plating baths as long as proper pre-treatment of the metal surface is assured. The recommendations of the bath supplier, especially in terms of current density, are to be followed. A further requirement for accurate plating is a uniform coating. Film thickness not below 8 µm are therefore suggested. For non-critical baths (like nickel sulfamate or brilliant tin) a thermal cure of the sufficiently thick resist layer is not necessary. Due to the variety of commercial plating baths general recommendations are difficult to be given. In case of doubt a test run is suggested.

Stripping

Positive liquid resists can be removed:

- by flood exposure and subsequent development
- by rinsing with polar solvents like acetone
- by rinsing with 4 to 30 % sodium hydroxide or
- potassium hydroxide at elevated temperature (ref.: 50°C)

Resist, which has been cured at temperatures above 120°C is to be removed by 10 to 30% alkali at elevated temperature. An increase in stripping speed is obtained by mechanical aid of the stripping process by spraying or wiping or by addition of solvent. In case of doubt, especially with roughened surfaces, a test run is suggested.

Waste Disposal and Environmental Aspects

The working up of resist loaded developer solutions and rinsing waters can be done in neutralisation equipment by addition of acid. The pH value of the used developer solution is about 13. By acidification to pH 3 most of the dissolved organic material is precipitated and can be removed by filtration. The waste water loading is thus reasonably reduced. Before draining the solution is to be adjusted to pH 6.5 to 9. The regulations of the local water authorities are to be observed.

The pH value of loaded aqueous alkaline stripper solutions is about 13.5. Working up follows the process outlined above. The so obtained solid waste and solvent containing stripper solutions are to be deposited or alternatively incinerated in locally authorised sites.

Safety informations

Skin contact with uncovered resist as well as with the processed developer is to be avoided to prevent allergic reactions particularly of people with sensitive skin. It is suggested to coat in a well-ventilated room. Preferably the coater should be installed under a hood. The exhaust air from the drying unit is to be removed in suitable form.

All directions contained in our material safety data sheets must be adhered to.

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