HD MicroSystems™
An Enterprise of Hitachi Chemical and DuPont Electronics

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PRODUCT BULLETIN

PI 2525, PI 2555 & PI 2574

PI 2525, PI 2555 and PI 2574 polyimide precursors are suitable for applications where the high temperatures typically used for polyimide curing (350°C) cause problems. Typical applications for these materials are as stress buffer or interlayer dielectric layers over low temperature substrates. These materials imidize faster and at lower temperatures than for standard polyimide precursors. The materials are supplied as solutions suitable for spin or roller coating application. A separate adhesion promoter is recommended with PI 2525 and PI 2555 to improve adhesion to oxides and to metals. PI 2574 is self priming and does not require an adhesion promoter. Processing by wet or plasma etch is possible. Cured film thicknesses from 2 µm to 9 µm can be obtained.

Process Details

Coating

These products can be coated onto a variety of metals, alloys, semiconductor and ceramic substrates. HD MicroSystems adhesion promoters VM651 or VM-652 are recommended to provide good processability and adhesion with PI 2525 and PI 2555. Bonding of the polyimide precursor to the substrate is achieved during the softbake cycle as the priming chemistry is activated by temperature.

These solutions are highly viscous. There are some guiding principles for dispensing materials of this type. Always coat substrates which are at room temperature. Never trap air into the solution. This can occur for example when the solution is moved during dispense. All bubbles take time to dissipate out of solution. If left in, coating "comets" will result. Dispensing should be in the centre and as close to the substrate as possible. A clean cut-off at dispense is neccessary before the spin process starts. It may be neccessary in the case of highly viscous solutions to have a short delay prior to spin to allow the polyimide to flow as far as possible and relax.

Both static and dynamic dispense may be used. Static dispense is the easiest, but requires more material to be dispensed for each substrate. Dynamic dispense uses less material, but requires greater control during dispense to ensure that the polyimide strikes the exact centre of the substrate. Any deviation will result in poor coating quality. The acceleration to final speed should be as low as possible to allow gradual flow of the polyimide across the substrate. Often one or more intermediate spin speeds are used to allow the polyimide to gradually cover more than 80% of the substrate before continuing on to the final speed. To reduce the backside contamination potential it is often beneficial to prolong the spread cycle until the bulk of the excess polyimide has been removed from the substrate.

The final spin speed and time is determined by the film thickness required (see spin speed curves on following page. Longer spin times will improve coating uniformity, but will also reduce the film thickness. In semiconductor applications, an Edge Bead Removal (EBR) and Backside Rinse process may be added to the coating cycle to remove polyimide precursor from the edge and back of the wafer prior to baking. NMP (N-methyl-2-pyrrolidone) or NMP/IPA (isopropanol) can be used for this purpose.

Soft Bake

After application of the polyimide, a bake process is required. Both convection oven and hotplate bake methods may be used. The purpose of this stage is to partially cure the polyimide precursor prior to patterning. This bake stage leaves the coating dry, yet soluble in the etchant solution.

Choice of Photoresist

Files of this type can be patterned using common photolithography techniques centered around positive photoresist. The underlying polyimide is an effective anti-reflective layer as there is significant absorption between 350nm and 450nm. This absorption can significantly reduce substrate reflection effects on the photoresist, usually seen as “notching“ after development.

The photoresist should be selected with the correct wavelength to suit the exposure tool in use. As a general guide, formulations with good adhesion in “wet etch“ semiconductor applications perform well. Other attributes include:

- Compatability to standard alkaline positive photoresist developers
- Low contrast performance so that a soft sidewall profile is always produced
- Capability to produce cleanly developed via holes in thick resist coatings
- Good development latitude, especially when over-developed
- The ease of producing a minimum dried 2.5 µm film thickness

The coating after softbake has minimum solubility in typical photoresist solvents. Photoresist can therefore be coated directly onto the polyimide coating without layer inter-mixing occuring.

Photoresist Application

Substrates should be coated directly with the resist selected. No dehydration bake should be given as this would make the polyimide totally insoluble in the developer. Instead an HMDS vapor prime is permissable if installed on-line but is not really neccessary for good resist adhesion to the polyimide.

Once coated, the resist should be given a softbake at 90℃ either in a convection oven for 30 minutes or on a vacuum hotplate for 60 seconds. Once coated and baked, coatings may be held up to 24 hours before exposure.

Photoresist Exposure

Typical exposure: 50 mJ to 150 mJ

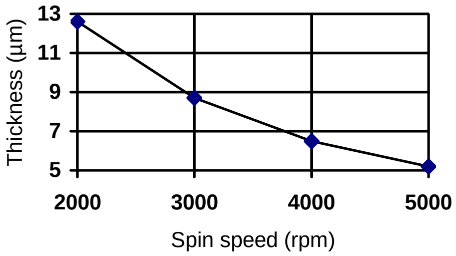
Once exposed, development should take place within 8 hours.

Spin Speed Curves for Cured Polyimide

Pyralin PI 2525/PI2574

(30 s spin; 120 s at 100℃

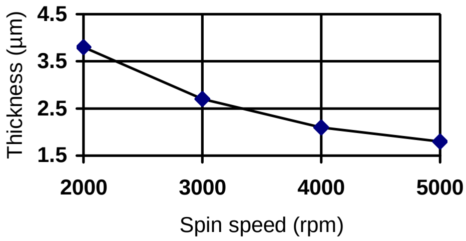
Cure: 30 min at 200℃ + 30 min at 350℃)



Spin Speed Curve Pyralin PI 2555

(30 s spin; 120 s at 100℃

Cure: 30 min at 200℃ + 30 min at 350℃)



Photoresist Development Polyimide Precursor Etch

A single step is used to develop the photoresist and etch the polyimide precursor.

Most alkaline positive resist developers will dissolve both exposed photoresist and polyimide precursor at varying rates. The choice of developer affects the quality of the image after development. Best results have been obtained using a NaOH based developer.

Thin layers up to around 5 µm can usually be developed quickly and cleanly with developer which is at ambient temperature. After development using a spray or puddle technique, a water rinse should be used to remove the developer. The substrate should subsequently be spun until dry.

When thicker polyimide layers need to be processed, it is often beneficial to heat the developer to between 23℃ and 25℃. This accelerates the dissolution of the polyimide precursor while having minimal effect on the solubility of the photoresist. In more extreme situations, this may be coupled to a double puddle process. The second puddle is used to develop only the polyimide precursor layer. Once developed, wafers may be held up to 8 hours before stripping the resist.

Resist Strip

After developing, the photoresist needs to be stripped off the polyimide precursor surface before curing. This step is usually carried out on automated track equipment to reduce surface contamination with resist residue which may result if clean solvents are not used. Resist solvent strippers are normally used.

Cure

The cure heating cycle imidizes the polyimide precursor converting it to a polyimide and driving out remaining solvent. This process requires elevated temperatures and controlled environments to achieve the best results. There is sufficient energy at 180℃ to complete the imidization of the polyimide, but higher temperatures are required to completely drive off solvents, thus achieving the ultimate electrical and mechanical properties. A programmable high temperature oven with typical nitrogen flow rate of 10 litres per minute is recommended for best results.

To activate the adhesion promoter, it is recommended that the cure be carried out up until 200℃ in air (min 50% RH). Above this temperature, a nitrogen atmosphere should be used. The ramp rates (up and down) should be low to avoid high stress in the polyimide. The maximum cure temperature may be higher than 200℃ when the coating is to be subjected to a high temperature process after curing. In such cases, temperatures up to 400℃ have been used to ensure that there is no outgassing during subsequent processes.

Storage/Shelf Life

Pyralin® PI 2525 and PI 2555 are stable at cleanroom temperatures (21℃) for about three weeks with no significant change in properties. When stored at -18℃, shelf-life is two years from date of manufacture. PI 2574 should be used within one week at room temperature and has a frozen shelf life of one year. Moisture contamination is detrimental to stability and must be avoided. Containers should be brought to room temperature before opening to avoid moisture condensation inside the bottle.

Example of Typical Process Conditions

Application of Adhesion Promoter
(not required for PI 2574)
(VM 652 or diluted VM 651)

- Dispense on static substrate, 3 seconds
- Hold for 20 seconds
- Spin Dry for 30 Seconds

Apply Polyimide Precursor Coating

- Dispense on static substrate
- Spread at 500rpm for 5 seconds
- Spin at final speed for 30 seconds
- EBR / Backside rinse, 10 seconds
- Spin Dry, 15 seconds
- Hot plate bake at 120^{o}C for 30 seconds, followed by 150 ^{o}C for 30 seconds.

Coat Photoresist

- Dispense, 3 seconds
- Spread at 500rpm for 5 seconds
- Spin at final speed for 30 seconds
- EBR / Backside rinse, 10 seconds
- Spin Dry for 15 seconds.
- Contact hot plate bake at 90^{o}C for 60 seconds

Expose Photoresist – 50mj to 150mj

Develop Photoresist / Polyimide Etch

Developer: TMAH, DE-1000, KOH or NaOH
Rinse: DI water
Double Puddle Development Process:

- Spray (100rpm) 5 seconds
- Puddle 20 seconds
- Spray (100rpm) 5 seconds
- Puddle 20 seconds
- Rinse (1000rpm) 15 seconds
- Spin Dry (5000rpm) 15 seconds

Resist Strip

Stripper: PGMEA, acetone, N-Butlyacetate

- Spray (100rpm) 5 seconds
- Puddle 20 seconds
- Spray (100rpm) 5 seconds
- Puddle 20 seconds
- Spin Dry (5000rpm) 15 seconds

Polyimide Cure (in Nitrogen)

- Heat from RT to 200^{o}C, ramp rate 4^{o}C/min
- Hold 200^{o}C, 30 minutes
- Heat to 300^{o}C, ramp rate 2.5^{o}C/min
- Hold at 300^{o}C for 60 minutes
- Gradual cooling to RT

Solution Properties

	PI 2525/PI 2574	PI 2555
Solids content (%) **Viscosity (Poise)** **Flash Point**	25.0 +/-1.0 60 +/- 10.0 93℃	19.0 +/-1.0 14.0 +/-2.0 60℃
Solvents (%)	N-Methyl-2-Pyrrolidone 100%	N-Methyl-2-Pyrrolidone Aromatic Hydrocarbon 80%/20%±5%
Ash Content (%) **Chloride Content** **Sodium Content** **Potassium Content** **Copper Content** **Iron Content** **Total Metals**	0.1 ppm max. 2.0 ppm max. 1.0 ppm max. 0.5 ppm max. 0.5 ppm max. 1.0 ppm max. 10.0 ppm max.	0.1 ppm max. 2.0 ppm max. 1.0 ppm max. 0.5 ppm max. 0.5 ppm max. 1.0 ppm max. 10.0 ppm max.

Cured Film Properties

Tensile strength (kg/mm^2)	13.1
Elongation (%)	10
Modulus (kg/mm^2)	245
Stress ($dynes/cm^2$)	3.6×10^8
Moisture uptake (%)	2 - 3
Dielectric constant (at 1 kHz, 50% RH)	3.3
Dissipation factor	0.002
Dielectric strength (volts/mil)	4000
Volume resistivity (ohm-cm)	10^{16}
Surface resistivity (ohm)	10^{15}
Coefficient of thermal expansion (ppm)	40
Coefficient of thermal conductivity (cal/(cm)(sec)(℃))	35×10^{-5}
Glass transition temperature	> 320℃
Decomposition temperature	550℃
Weight loss (% at 500℃, 120 min)	2.9
Specific heat (cal/g/℃)	0.26
Refractive index	1.70

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Caution: Do not use in medical applications involving permanent implantation in the human body.