Product Information
Pyralin® PI2525, PI2555, PI2556

The Pyralin® PI2525, PI2555 and PI2556 polyimides are suitable for applications where the high cure temperatures typically used for polyimides (ca. 350°C) cause problems. Typical applications for these materials are as stress buffer or interlayer dielectric layers over low temperature substrates. The Pyralin® PI2525, PI2555 and PI2556 polyimides 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 to provide adhesion to oxides to metals. Processing by wet or plasma etch is possible. Cured film thicknesses from 0.5 μm to 6 μm can be obtained.

Process Details

Coating

Pyralin® PI2525, PI2555 and PI2556 products can be coated onto a variety of metals, alloys, semiconductor and ceramic substrates. The silane adhesion promoter Pyralin® VM651 is recommended to provide good processability and adhesion. Bonding of the polyimide to the substrate is achieved during the softbake cycle as the priming chemistry is activated by temperature.

The polyimide solution is 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 necessary before the spin process starts. It may be necessary 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. 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 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.

Spin Speed Curves for cured Polyimide

**Spin Speed Curve Pyralin PI 2525**
(30 s spin; 120 s at 100°C + 30 min at 200°C + 30 min at 350°C)

![Spin Speed Curve Pyralin PI 2525 Graph]

**Spin Speed Curve Pyralin PI 2556**
(30 s spin; 120 s at 100°C + 30 min at 200°C + 30 min at 350°C)

![Spin Speed Curve Pyralin PI 2556 Graph]

**Spin Speed Curve Pyralin PI 2555**
(30 s spin; 120 s at 100°C + 30 min at 200°C + 30 min at 350°C)

![Spin Speed Curve Pyralin PI 2555 Graph]

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 prior to patterning. This bake stage leaves the polyimide coating dry, yet soluble in the etchant solution.

Choice of Photoresist

Polyimides 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 350 and 450 nm. 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:

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

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 vapour prime is permissible if installed on-line but is not really necessary for good resist adhesion to the polyimide.

Once coated, the resist should be given a softbake at 90°C 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.

Photoresist Development / Polyimide Etch

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

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

Thin polyimide layers up to around 5 μm can usually be developed quickly and cleanly with developer which is at ambient temperature. After development of the resist and then the polyimide using a spray 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°C and 25°C. This accelerates the dissolution of the polyimide 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 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 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 converts the polyamic acid to the insoluble imide form and drives out remaining solvent. This process requires elevated temperatures and controlled environments to achieve the best results. There is sufficient energy at 180°C 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°C 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 300°C when the coating is to be subjected to a high temperature process after curing. In such cases, temperatures up to 400°C have been used to ensure that there is no outgassing during subsequent processes.

![Imidization Rates - PI 2555 Type](image)

![Imidization Rates - PI 2540 Type](image)

Processing Pyralin® PI2525, PI2555, PI2556 products

(for ca. 7μm softbaked and 5μm cured film thickness)

Application of Adhesion Promoter

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

Bake Adhesion Promoter

- On hotplate at 120°C for 60 seconds (optional)
Polyimide Coating

- Dispense on static substrate
- Rotate at 500 rpm for 5 seconds
- Rotate at final speed for 30 seconds (see spin speed curves)
- EBR/Backside rinse, 10 seconds
- Spin dry, 15 seconds

Soft Bake

- In convection oven at 120°C for 30 minutes
- OR
- On hot plate at 120°C for 30 seconds followed by 150°C for 30 seconds

Coat Photoresist

- Dispense, 3 seconds
- Spread at 500 rpm for 5 seconds
- Spin at final speed for 30 seconds
- EBR / Backside rinse for 5 seconds
- Spin dry for 15 seconds

Softbake Photoresist

- On hotplate at 90°C for 60 seconds

Exposure

- 50 mJ to 150 mJ

Development

Developer: NaOH, KOH or TMAH developer or DE 1000 at 21°C
Rinse: DI water

Puddle Development process:

- Spray (100 rpm) 5 seconds
- Puddle 3 minutes
- Rinse (1000 rpm) 15 seconds
- Spin dry (5000 rpm) 15 seconds

Resist Strip

- Spray or spray/puddle acetone or standard resist stripper or N-Butlyacetate at ca. 2000 rpm
- Spin dry
Cure

Curing should be done in an oven using the following cure profile:

- Heating from room temperature to 200°C, ramp rate 4°C/minute in air
- Hold time at 200°C for 30 minutes in air
- Heating from 200°C to 300°C, ramp rate 2.5°C/minute in nitrogen
- Hold time at 300°C for 60 minutes in nitrogen
- Gradual cooling to room temperature

Solution Properties

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<tr>
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<th>PI2525</th>
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<tbody>
<tr>
<td>Solids content (%)</td>
<td>25.0 +/- 1.0</td>
<td>19.0 +/- 1.0</td>
<td>15.0 +/- 1.0</td>
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<tr>
<td>Viscosity (Poise)</td>
<td>60 +/- 10.0</td>
<td>14.0 +/- 2.0</td>
<td>3.5 +/- 1.0</td>
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<tr>
<td>Flash Point</td>
<td>93°C</td>
<td>60°C</td>
<td>54°C</td>
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<tr>
<td>Solvent (%)</td>
<td>N-Methyl-2-Pyrrolidone</td>
<td>N-Methyl-2-Pyrrolidone</td>
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<td></td>
<td>100</td>
<td>80/20 +/- 5</td>
<td>Propylene Glycol Methyl Ether</td>
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<tr>
<td>Ash content (%)</td>
<td>0.1 max.</td>
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<tr>
<td>Chloride content</td>
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<td>Sodium content</td>
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<td>Potassium content</td>
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<td>Copper content</td>
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<td>Iron content</td>
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<tr>
<td>Total metals</td>
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Cured Film Properties

<table>
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<tr>
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<th>Value</th>
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<td>Tensile strength (kg/mm²)</td>
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<td>Elongation (%)</td>
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<tr>
<td>Modulus (kg/mm²)</td>
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<td>Stress (dynes/cm²)</td>
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<tr>
<td>Moisture uptake (%)</td>
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<tr>
<td>Dielectric constant (at 1 kHz, 50% RH)</td>
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<tr>
<td>Dissipation factor</td>
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<tr>
<td>Dielectric strength (volts/mil)</td>
<td>4000</td>
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<tr>
<td>Volume resistivity (ohm-cm)</td>
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<tr>
<td>Surface resistivity (ohm)</td>
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<tr>
<td>Coefficient of thermal expansion (ppm)</td>
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<tr>
<td>Coefficient of thermal conductivity (cal/(cm)(sec)(°C))</td>
<td>35 x 10⁶</td>
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<tr>
<td>Glass transition temperature</td>
<td>&gt; 320°C</td>
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</table>
Decomposition temperature  560°C
Weight loss (% at 500°C, 120 min)  2.9
Specific heat (cal/g°C)  0.26
Refractive index  1.70

Packaging / Storage

Pyralin® PI2525, PI2555 and PI2556 are supplied in 1 kg bottles. All material should be refrigerated upon receipt unless it is used immediately.

Pyralin® PI2525, PI2555 and PI2556 are stable at clean room temperatures (21°C) for ca. four weeks with no significant change in properties. When stored at -18°C, shelf-life is 2 years from date of manufacturing. 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.

Safety and Handling

During all handling of Pyralin polyimide products, adequate ventilation must be provided. Skin and eye contact should be avoided. Exposed areas should be flushed with water immediately. For prolonged or repeated contact, milled butyl rubber or natural latex rubber gloves should be used. Disposable PVC gloves are not recommended.

<table>
<thead>
<tr>
<th>United States</th>
<th>Japan</th>
<th>Europe</th>
</tr>
</thead>
<tbody>
<tr>
<td>P0 Box 80010</td>
<td>10-13, Shibuya 3-chome, Shibuya-ku, Tokyo 150 Japan</td>
<td>DuPont Strasse 1 61352 Bad Homburg Germany</td>
</tr>
<tr>
<td>Wilmington, DE 19880</td>
<td>81-3-3407-9003 (Phone) 81-3-3407-9037 (Fax)</td>
<td>49-6172-87-1822 (Phone) 49-6172-87-1824 (Fax)</td>
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<tr>
<td>800-346-5656 (Phone)</td>
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<td>302-892-0597 (Fax)</td>
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</tr>
<tr>
<td>HD MicroSystems, LLC</td>
<td>1333 Lawrence Expressway Suite 212</td>
<td></td>
</tr>
<tr>
<td>1333 Lawrence</td>
<td>Santa Clara, CA 95051</td>
<td></td>
</tr>
<tr>
<td>Expressway Suite 212</td>
<td>408-260-0880 (Phone) 408-260-0885 (Fax)</td>
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Caution: Do not use in medical applications involving permanent implantation in the human body.