DRY ETCHING

With dry etching, the material is not eroded by the compounds dissolved in a liquid, but by the atoms or molecules of a, at least, partially ionised gas.

This chapter describes the basic physical and chemical processes of this etching process and sets out the specific requirements of the previous photoresist processing to define the desired resist mask.

Basic Etching Mechanisms and Parameters

Physical and Chemical Processes in Dry Etching

The erosion of the material to be etched can either be carried out physically by the kinetic energy of the particles (neutral or ionized atoms or molecules) from the gas, or through chemical reactions between the material and the gas.

The physical mechanism can be regarded as a partial-elastic collision of ions with the atoms of the medium to be etched. Chemical reactions play no or only a subordinate role, which is why etching is hardly materially selective. Since the ions from the gas are usually accelerated by electric fields perpendicular to the substrate, the material removal in this preferred direction is anisotropic.

If the 'chemical' mechanism dominates, etching occurs via the strong material selective formation of volatile compounds by radicals in the plasma which – towards high plasma pressure – hit the surface more and more isotropically. Compared to the physical erosion, the chemical erosion allows a significantly higher etching rate.

Dry Etching Technologies

Plasma etching is dominated by chemical erosion. In this way Si or SiO$_2$ is etched usually with chlorinated and fluorinated hydrocarbons isotropic and very material selectively.

With sputter etching (ion milling), the material is eroded physically by inert gas ions accelerated on the substrate.

RIE (Reactive Ion Etching) represents a combination of physical and chemical erosion: Here, chemically reactive radial is formed in plasma and accelerated towards the substrate.

Criteria with Dry Etching

The etching rate is defined by the eroded thickness of the material to be etched per time.

The selectivity is the ratio of the etching rate of two materials that are simultaneously exposed to the etching. This can be, for example, the photoresist structures of the etching mask, as well as the material to be etched, but also two vertically stacked materials that are to be etched in succession.

The anisotropy describes the ratio of etch rates perpendicular and parallel to the substrate surface. The more isotropic the etching process is, the stronger the etching mask is underetched during the etching process.

The uniformity describes the homogeneity of the etching rate over the entire substrate.

Plasma Etching of Si and SiO$_2$

Typical Etching Gases

Typical etching gases for the etching of SiO$_2$ are fluorinated hydrocarbons (C$_x$F$_y$H$_z$) such as tetrafluoromethane (CF$_4$). The basic reactions in plasma and on the SiO$_2$ to be etched are:

1. Formation of F-radicals through electron impacts: $e + CF_4 \rightarrow CF_3 + F + e^-$
2. Formation of volatile Si compounds: $SiO_2 + 4 F \rightarrow SiF_4 + O_2$

Typical etching gases for etching of Si are chlorinated and fluorinated carbon compounds (C$_x$F$_y$CL$_z$), again using the example of tetrafluoromethane:

1. Formation of F-radicals through electron impacts: $e + CF_4 \rightarrow CF_3 + F + e^-$
2. Formation of volatile Si compounds: $Si + 4 F \rightarrow SiF_4$
Adjustment of the Required Etching Rates Ratio Si : SiO$_2$

The addition of oxygen increases via the reaction CF$_3$ + O $\rightarrow$ COF$_2$ + F the concentration of fluorine radicals in the plasma with the following consequences:

- Via the reaction Si + 4 F $\rightarrow$ SiF$_4$ increases the etching rate of silicon to a maximum with a percentage of approx. 12% O$_2$ in CF$_4$.
- Via the reaction SiO$_2$ + 4 F $\rightarrow$ SiF$_4$ + O$_2$, the etching rate of SiO$_2$ increases with a maximum of approximately 20% O$_2$ in CF$_4$.
- Via O$_2$ combustion, an existing resist mask is eroded more strongly.

The addition of hydrogen to the process gas

- reduces the concentration of fluorine radicals in the plasma via the reaction H + F $\rightarrow$ HF and lowers the etching rate, however, for Si more than for SiO$_2$.
- leads to the chemically very inert fluorinated polymer deposition on Si surfaces via the reaction CF$_4$ + H + Si $\rightarrow$ CH$_x$F$_y$, thus stopping the etching of silicon.

**Deep Reactive Ion Etching: The "Bosch Process"**

The so-called Bosch Process lends itself to the dry chemical etching of structures with steep sidewalls and a very high aspect ratio.

Alternating anisotropic Si etching and the formation of a fluorinated polymer layer (which is inert against the plasma) on the etched sidewalls as well as the sidewalls of the resist structures allows aspect ratios > 50, Si etching rates > 10 µm/min, etching rate ratios > 450 (Si : SiO$_2$) and > 150 (Si : photoresist).

**Plasma Etching of Certain Metals**

**Aluminium**

Aluminium can be etched using gases such as hydrogen bromide (HBr) or chlorine-containing gases such as under the formation of sufficiently volatile compounds of aluminium bromide (AlBr$_3$) or aluminium chloride (AlCl$_3$).

**Tungsten**

Tungsten is etched with fluorine-containing gases with the formation of volatile tungsten hexafluoride (WF$_6$), the densest known gas under standard conditions.

**Titanium**

Due to the very low vapour pressures of titanium chloride (TiCl$_3$) and titanium fluoride (TiF$_3$), pure plasma processes with accordingly halogenated process gases are not suitable for the dry etching of titanium which is why argon is usually added to increase the erosion with sputter etching.

**Copper, Silver, and Gold**

These metals do not form sufficiently volatile halides for adequately high plasma etching rates at temperatures below 150°C. With the help of hydrogenous process gases however (unstable) hydrides of the metals can form, which via ion or photon-assisted processes can be desorbed from the surface at etching rates of a few nm/min.

**Photoresist Processing Requirements**

**Vertical Resist Sidewalls**

For the steepest possible resist profiles, a high-contrast, photoresist, as well as process parameters optimised for high contrast are required, i.e.

- depending on the desired resist film thickness and required thermal stability of the AZ® 701 MiR for resist film thicknesses below 1 µm, the AZ® ECI 3000 series for 1 - 3 µm resist film thickness, or AZ® 9260 for even thicker layers,
- the reduction of the dark erosion and maintenance of a possible high development rate of positive
resists via, among other things, optimised softbake parameters, and
  • the use of a highly selective, i.e. optimally diluted developer such as the MIF developers AZ® 326 and AZ® 726 or the AZ® 400K or AZ® 351B in a sufficiently high dilution.

**Resist Lenses**
If ellipsoid resist structures in the substrate are to be transferred, a normally processed resist profile with a rectangular cross-section is usually softened by heating via the softening temperature of the resist. For this process, all positive resists are suitable. The series AZ® 1500, AZ® 4500 and 9200 have a relatively low softening temperature of approx. 100 - 110 °C.

**Removal of the Photoresist Mask after Etching**
All standard strippers are generally suitable to remove the resist mask after dry etching. In case of increased temperatures during dry etching, possibly supported by the deep UV background radiation from the plasma, the resist structures can cross-link near their surface. If the removability of the resist mask suffers after the etching process, the measures listed in the following section can be applied against excessive heating. Ultrasonic treatment during stripping also supports removal of the resist structures. For highly cross-linked positive resists which can not be removed with standard removers, the high-performance stripper TechniStrip® P1316 is recommended for positive resists or the TechniStrip® NI555 for many Novolak based negative resists such as the AZ® nLOF 2000 series.

**Measures Against the Thermal Softening of Resist Structures**
Heat development during etching can soften the edges of the used photoresist mask which is transferred to the substrate during dry etching. Possible remedial measures are
  • an optimised heat coupling of the substrate to its holder (e.g. some drops of turbo pump oil for proper heat transfer from strained, curved substrates)
  • a sufficiently high heat buffer (massive substrate holder construction) or
  • heat removal (e.g. black anodised aluminium as rear infrared radiator) from the substrate holder
  • a reduced deposition rate and/or a multi-stage deposition with cooling interval(s) in between or
  • a thermally more stable photoresist like the AZ® 701 MiR or the AZ® ECI 3000 series
  • a sufficient softbake to minimise the residual solvent content.

**Measures Against Bubble Formation in the Resist Layer during Dry Etching**

**Appearance**
Sometimes, bubbles in the resist or even a foam-like resist appearance is observed after dry-etching. In most cases, nitrogen or evaporating solvent or water is the reason for this behaviour.

**Evaporation of Residual Solvents**
Another possible source of vapour bubbles is water which has penetrated during development in the resist film and can be evaporated after development with another baking. In this case, a baking step after development at approx. 80 - 100°C (always below the resist softening point!) helps reduce the water concentration and a thermal deformation of the resist structures.

**Evaporation of Water**
An insufficient softbake (too short/too cool) may cause the evaporation of the remaining solvent from the resist forming bubbles.

**Nitrogen Formation**
The developed resist structures of DNQ-based positive resists are still photo-active and can be exposed by the short-wave thermal or recombination radiation from plasma forming larger amounts of nitrogen. To ensure all photoinitiator has been converted and all nitrogen has been released from the resist film before the dry etch process, we recommend a flood exposure without mask, followed by a delay to out-
gas the nitrogen formed before the substrate is introduced to the dry etching.
Image reversal resists in image reversal mode are no longer photoactive after development and negative resists don't release nitrogen during exposure, so these resists are not affected by this problem.
### Inorganic Developers

#### AZ Developer
- Based on sodium phosphate and –metasilicate, optimized for minimal aluminum attack and typically used diluted 1:1 in DI water for high contrast or undiluted for high development rates. The dark erosion of this developer is slightly higher compared to other developers.
- **AZ® 351B**: Based on buffered NaOH and typically used diluted 1:4 with water, for thick resists up to 1:3 if a lower contrast can be tolerated.
- **AZ® 400K**: Based on buffered KOH and typically used diluted 1:4 with water, for thick resists up to 1:3 if a lower contrast can be tolerated.
- **AZ® 303**: Specifically for the AZ® 111 XFS photoresist based on KOH / NaOH is typically used diluted 1:3 - 1:7 with water, depending on whether a high development rate, or a high contrast is required.

#### Metal Ion Free Developers (TMAH-based)
- **AZ® 326 MIF**: 2.38 % TMAH (Tetramethylammonium Hydroxide) in water.

### Positive

<table>
<thead>
<tr>
<th>Recommended Applications</th>
<th>Resist Family</th>
<th>Photosists</th>
<th>Resist Film Thickness</th>
<th>Recommended Developers</th>
<th>Recommended Removers</th>
</tr>
</thead>
<tbody>
<tr>
<td>Improved adhesion for wet etching, no focus on steep resist sidewalls</td>
<td>AZ® 1500</td>
<td>AZ® 1505</td>
<td>0.5 µm</td>
<td>AZ® 351B, AZ® 326 MIF, AZ® 726 MIF, AZ® 826 MIF</td>
<td>AZ® 100 Remover, TechniStrip® P1316, TechniStrip® P1331</td>
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<tr>
<td></td>
<td>AZ® 1512 HS</td>
<td>AZ® 1514 H</td>
<td>1.0 - 1.5 µm</td>
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<tr>
<td></td>
<td>AZ® 1518</td>
<td></td>
<td>1.2 - 2.0 µm</td>
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<tr>
<td></td>
<td>AZ® 4500</td>
<td>AZ® 4533</td>
<td>3 - 5 µm</td>
<td>AZ® 400K, AZ® 326 MIF, AZ® 726 MIF, AZ® 826 MIF</td>
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<tr>
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<td>AZ® 4562</td>
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<td>5 - 10 µm</td>
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<td>AZ® P4000</td>
<td>AZ® P4110</td>
<td>1 - 2 µm</td>
<td>AZ® 400K, AZ® 326 MIF, AZ® 726 MIF, AZ® 826 MIF</td>
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<td></td>
<td>AZ® P4330</td>
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<td>3 - 5 µm</td>
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<td>AZ® P4620</td>
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<td>6 - 20 µm</td>
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<td></td>
<td>AZ® P4902</td>
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<td>10 - 30 µm</td>
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<tr>
<td>Spray coating</td>
<td>AZ® PL 177</td>
<td>AZ® PL 177</td>
<td>8 - 8 µm</td>
<td>AZ® 351B, AZ® 400K, AZ® 326 MIF, AZ® 726 MIF, AZ® 826 MIF</td>
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<tr>
<td>Dip coating</td>
<td>AZ® 4999</td>
<td></td>
<td>1 - 15 µm</td>
<td>AZ® 400K, AZ® 326 MIF, AZ® 726 MIF, AZ® 826 MIF</td>
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<tr>
<td>Steep resist sidewalls, high resolution and aspect ratio for e.g. dry etching or plating</td>
<td>AZ® ECI 3000</td>
<td>AZ® ECI 3007</td>
<td>0.7 µm</td>
<td>AZ® 351B, AZ® 326 MIF, AZ® 726 MIF, AZ® Developer</td>
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<tr>
<td></td>
<td>AZ® ECI 3012</td>
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<td>1.0 - 1.5 µm</td>
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<td></td>
<td>AZ® ECI 3027</td>
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<td>2 - 4 µm</td>
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<td>AZ® 9200</td>
<td>AZ® 9245</td>
<td>3 - 6 µm</td>
<td>AZ® 400K, AZ® 326 MIF, AZ® 726 MIF</td>
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<td></td>
<td>AZ® 9260</td>
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<td>5 - 20 µm</td>
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<td>AZ® 701 MR (14 cPs)</td>
<td>AZ® 701 MR (14 cPs)</td>
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<td></td>
<td>AZ® 701 MR (29 cPs)</td>
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<td>2 - 3 µm</td>
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### Image Reversal

<table>
<thead>
<tr>
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<th>Resist Film Thickness</th>
<th>Recommended Developers</th>
<th>Recommended Removers</th>
</tr>
</thead>
<tbody>
<tr>
<td>Elevated thermal softening point and undercut for lift-off applications</td>
<td>AZ® XT</td>
<td>AZ® 12 XT-20PL-05</td>
<td>3 - 5 µm</td>
<td>AZ® 400K, AZ® 326 MIF, AZ® 726 MIF</td>
<td>AZ® 100 Remover, TechniStrip® P1316, TechniStrip® P1331</td>
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<td>AZ® 12 XT-20PL-10</td>
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<td>AZ® 12 XT-20PL-20</td>
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<td>10 - 30 µm</td>
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<td>AZ® 40 XT</td>
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<td>15 - 50 µm</td>
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<td>AZ® IPS 6050</td>
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<td>20 - 100 µm</td>
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### Negative (Cross-Linking)

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<tr>
<th>Recommended Applications</th>
<th>Resist Family</th>
<th>Photosists</th>
<th>Resist Film Thickness</th>
<th>Recommended Developers</th>
<th>Recommended Removers</th>
</tr>
</thead>
<tbody>
<tr>
<td>Improved adhesion, steep resist sidewalls and high aspect ratios for e.g. dry etching or plating</td>
<td>AZ® nLOF 2000</td>
<td>AZ® nLOF 2020</td>
<td>1.5 - 3 µm</td>
<td>AZ® 326 MIF, AZ® 726 MIF, AZ® 826 MIF</td>
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<tr>
<td></td>
<td>AZ® nLOF 2035</td>
<td></td>
<td>3 - 5 µm</td>
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<td></td>
<td>AZ® nLOF 2070</td>
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<td>6 - 15 µm</td>
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<td></td>
<td>AZ® nLOF 5510</td>
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<td>0.7 - 1.5 µm</td>
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<td></td>
<td>AZ® nXT (115 cPs)</td>
<td>AZ® nXT (115 cPs)</td>
<td>2 - 3 µm</td>
<td>AZ® 326 MIF, AZ® 726 MIF, AZ® 826 MIF</td>
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<tr>
<td></td>
<td>AZ® nXT (450 cPs)</td>
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<td>5 - 20 µm</td>
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<td></td>
<td>AZ® nXT</td>
<td></td>
<td>20 - 100 µm</td>
<td>AZ® 326 MIF, AZ® 726 MIF, AZ® 826 MIF</td>
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### Negative (Curing)

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<th>Recommended Applications</th>
<th>Resist Family</th>
<th>Photosists</th>
<th>Resist Film Thickness</th>
<th>Recommended Developers</th>
<th>Recommended Removers</th>
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<tr>
<td>Positive (chem. amplified)</td>
<td>AZ® 5200</td>
<td>AZ® 5209</td>
<td>1 µm</td>
<td>AZ® 351B, AZ® 326 MIF, AZ® 726 MIF</td>
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<td>AZ® 5214</td>
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<td>1 - 2 µm</td>
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<td></td>
<td>Ti 35ESX</td>
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<td>3 - 4 µm</td>
<td>TechniStrip® Micro D2, TechniStrip® P1316, TechniStrip® P1331</td>
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<tr>
<td></td>
<td>Ti xLift-X</td>
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<td>4 - 8 µm</td>
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<tr>
<td>Image Reversal</td>
<td>AZ® nLOF 2000</td>
<td>AZ® nLOF 2020</td>
<td>1.5 - 3 µm</td>
<td>AZ® 326 MIF, AZ® 726 MIF, AZ® 826 MIF</td>
<td></td>
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<tr>
<td></td>
<td>AZ® nLOF 2035</td>
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<td>3 - 5 µm</td>
<td>TechniStrip® N555, TechniStrip® NF52, TechniStrip® MLO 07</td>
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<td></td>
<td>AZ® nLOF 2070</td>
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<td>6 - 15 µm</td>
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<tr>
<td></td>
<td>AZ® nLOF 5510</td>
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<td>0.7 - 1.5 µm</td>
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<td>AZ® nXT (151 cPs)</td>
<td>AZ® nXT (151 cPs)</td>
<td>2 - 3 µm</td>
<td>AZ® 326 MIF, AZ® 726 MIF, AZ® 826 MIF</td>
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<td>AZ® nXT (450 cPs)</td>
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<td>5 - 20 µm</td>
<td>TechniStrip® P1316, TechniStrip® P1331, TechniStrip® NF52, TechniStrip® MLO 07</td>
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</tbody>
</table>

### Metal Ion Free Developers (TMAH-based)
- **AZ® 326 MIF**: 2.38 % TMAH (Tetramethylammonium Hydroxide) in water.
AZ® 726 MIF is 2.38 % TMAH- (TetraMethylAmmoniumHydroxide) in water, with additional surfactants for rapid and uniform wetting of the substrate (e. g. for puddle development)

AZ® 826 MIF is 2.38 % TMAH- (TetraMethylAmmoniumHydroxide) in water, with additional surfactants for rapid and uniform wetting of the substrate (e. g. for puddle development) and other additives for the removal of poorly soluble resist components (resides with specific resist families), however at the expense of a slightly higher dark erosion.

Our Removers: Application Areas and Compatibilities

AZ® 100 Remover is an amine solvent mixture and standard remover for AZ® and Ti photoresists. To improve its performance, AZ® 100 remover can be heated to 60 - 80°C. Because the AZ® 100 Remover reacts highly alkaline with water, it is suitable for this with respect to sensitive substrate materials such as Cu, Al or ITO only if contamination with water can be ruled out.

TechniStrip® P1316 is a remover with very strong stripping power for Novolak-based resists (including all AZ® positive resists), epoxy-based coatings, polimyrides and dry films. At typical application temperatures around 75°C, TechniStrip® P1316 may dissolve cross-linked resists without residue also, e.g. through dry etching or ion implantation. TechniStrip® P1316 can also be used in spraying processes. For alkaline sensitive materials, TechniStrip® P1331 would be an alternative to the P1316. Not compatible with Au.

TechniStrip® P1331 can be an alternative for TechniStrip® P1316 in case of alkaline sensitive materials. TechniStrip® P1331 is not compatible with Au.

TechniStrip® NJ555 is a stripper with very strong dissolving power for Novolak-based negative resists such as the AZ® 15 nXT and AZ® nLOF 2000 series and very thick positive resists such as the AZ® 40 XT. TechniStrip® NJ555 was developed not only to peel cross-linked resists, but also to dissolve resists without residues. This prevents contamination of the basin and filter by resist particles and skins, as can occur with standard strippers. TechniStrip® NJ555 is not compatible with GaAs.

TechniClean® CA25 is a semi-aqueous proprietary blend formulated to address post etch residue (PER) removal for all interconnect and technology nodes. Extremely efficient at quickly and selectively removing organo-metal oxides from Al, Cu, Ti, TiN, W and Ni.

TechniStrip™ NFS2 is a highly effective remover for negative resists (liquid resists as well as dry films). The intrinsic nature of the additives and solvent make the blend totally compatible with metals used throughout the BEOL interconnects to WLP bumping applications.

TechniStrip™ Micro D2 is a versatile stripper dedicated to address resin lift-off and dissolution on negative and positive tone resist. The organic mixture blend has the particularity to offer high metal and material compatibility allowing to be used on all stacks and particularly on fragile III/V substrates for instance.

TechniStrip™ MLO 07 is a highly efficient positive and negative tone photorisest remover used for IR, III/V, MEMS, Photonic, TSV mask, solder bumping and hard disk stripping applications. Developed to address high dissolution performance and high material compatibility on Cu, Al, Sn/Ag, Alumina and common organic substrates.

Our Wafers and their Specifications

Silicon-, Quartz-, Fused Silica and Glass Wafers

Silicon wafers are either produced via the Czochralski- (CZ-) or Float zone- (FZ-) method. The more expensive FZ wafers are primarily reasonable if very high-ohmic wafers (> 100 Ohm cm) are required.

Quartz wafers are made of monocrystalline SiO₂, main criterion is the crystal orientation (e. g. X-, Y-, Z-, AT- or ST-cut).

Fused silica wafers consist of amorphous SiO₂. The so-called JGS2 wafers have a high transmission in the range of ≈ 280 - 2000 nm wavelength, the more expensive JGS1 wafers at ≈ 220 - 1100 nm.

Our glass wafers, if not otherwise specified, are made of borosilicate glass.

Specifications

Common parameters for all wafers are diameter, thickness and surface (1- or 2-side polished). Fused silica wafers are made either of JGS1 or JGS2 material, for quartz wafers the crystal orientation needs to be defined. For silicon wafers, beside the crystal orientation (<100> or <111>), the doping (n- or p-type) as well as the resistivity (Ohm cm) are selection criteria.

Prime-, Test-, and Dummy Wafers

Silicon wafers usually come as „Prime-grade“ or „Test-grade“, latter mainly have a slightly broader particle specification. „Dummy-Wafers“ neither fulfill Prime- nor Test-grade for different possible reasons (e. g. very broad or missing specification of one or several parameters, reclaim wafers, no particle specification) but might be a cheap alternative for e. g. resist coating tests or equipment start-up.

Our Silicon-, Quartz-, Fused Silica and Glass Wafers

Our frequently updated wafer stock list can be found here: www.microchemicals.com/products/wafers/waferlist.html

Further Products from our Portfolio

Plating

Plating solutions for e. g. gold, copper, nickel, tin or palladium: www.microchemicals.com/products/electroplating.html

Solvents (MOS, VLSI, ULSI)

Acetone, isopropyl alcohol, MEK, DMSO, cyclopentanone, butylacetate, … www.microchemicals.com/products/solvents.html

Acids and Bases (MOS, VLSI, ULSI)

Hydrochloric acid, sulphuric acid, nitric acid, KOH, TMAH, … www.microchemicals.com/products/etchants.html

Etching Mixtures

for e. g. chromium, gold, silicon, copper, titanium, … www.microchemicals.com/products/etching_mixtures.html
Further Information


Our Photolithography Book and -Posters

We see it as our main task to make you understand all aspects of microstructuring in an application-oriented way. At present, we have implemented this claim with our book Photolithography on over 200 pages, as well as attractively designed DIN A0 posters for your office or laboratory.

We will gladly send both of these to you free of charge as our customer (if applicable, we charge shipping costs for non-European deliveries):

www.microchemicals.com/downloads/brochures.html
www.microchemicals.com/downloads/posters.html

Thank you for your interest!

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