WET-CHEMICAL ETCHING OF SILICON AND SiO₂

Silicon is the most common substrate material used in microelectronics and micro-mechanics. It is used not only as a passive substrate, but also as an active material in electronic or mechanical components. The necessary patterning can also be achieved by means of wet-chemical etching methods, as described in this chapter.

Anisotropic Etching of Silicon

Etching Mechanism

Strongly aqueous alkaline media such as KOH-, NaOH- or TMAH-solutions etch crystalline silicon via

\[ \text{Si} + 2 \text{OH}^- + 2 \text{H}_2\text{O} \rightarrow \text{Si(OH)}_4 + \text{H}_2 \rightarrow \text{SiO}_2(\text{OH})_2^- + 2 \text{H}_2 \]

Because the Si atoms of the different crystal planes have different activation energies for the etching reaction and the KOH etching of Si is not diffusion-limited but etching-rate-limited, the etching process takes place anisotropically: The \{100\} and \{110\} planes are much more rapidly etched than the stable \{111\} plane that act as etch stops.

(111)-oriented Wafers

(111)-oriented Si wafers are hardly attacked by alkaline solutions, since here the entire wafer surface forms an etch stop. Because the real orientation of wafers is usually tilted to a few 0.1° against the ideal crystal plane, with nominally (111)-oriented wafers, an etching attack in the form of very shallow steps also occurs.

(100)-oriented Wafers

(100)-orientated wafers in alkaline etchants form square-based pyramids with \{111\} surfaces. These pyramids can be realised on mono-crystalline silicon solar cells for the purpose of reflection minimisation.

(110)-oriented Wafers

(110)-orientated wafers in alkaline etchants form perpendicular trenches with \{111\} side-walls, used as e.g. micro-channels in micro-mechanics and micro-fluidics.

Etch Rates

The anisotropy, the absolute etch rates and the homogeneity of the etching depend on both defects in

![Si Etch Rate in KOH](image1)

![Si Etch Rate in TMAH](image2)

Fig. 119: The concentration and temperature-dependent etching rate of (100) and (110) planes of crystalline silicon in KOH (left graph) and TMAH (right graph). The alkaline etching of Si requires in addition to OH ions, free water molecules. Therefore, the etching rate, but also the surface roughness of the etched silicon surface, decreases to stronger alkaline solutions.
the silicon as well as contamination of the etching by metal ions and already etched Si ions in addition to etching temperature. Also the doping of Si plays an important role:

During etching, boron-doped Si forms borosilicate glass on the surface which acts as etch stop if the boron doping concentration exceeds ($>10^{19}$ cm$^{-3}$).

Fig. 120 and Fig. 121 show the temperature and concentration-dependent etch rates of (100)- and (111) planes in KOH- and TMAH-solutions (Fig. 119), as well as the selectivity of the SiO$_2$ etching (Fig. 120 and Fig. 121), which is often used as masking.

Typical Etching Mixtures

We supply 25% TMAH and 44% KOH in VLSI quality. Because these media only attack SiO$_2$ to a very small extent, the (native) SiO$_2$ film must be removed before the anisotropic Si etching in diluted or buffered hydrofluoric acid.

Suitable Etching Masks

The high pH values and temperatures required for the anisotropic etching of silicon attack even heavily cross-linked negative resists in a short time, so that photoresist masks do not come into question for this purpose. Instead, hard masks usually made of silicon nitride, SiO$_2$ or alkaline-stable metal films such as chromium are used, which in turn can be structured using photoresist masks.
Isotropic Etching of Silicon with HF/HNO₃

Etching Mechanism

The basic etching mechanism in the isotropic etching of Si is divided into the oxidation of silicon using nitric acid and the etching of the oxide constantly formed on the surface from this with hydrofluoric acid:

1. Formation of NO₂ from nitric acid: 4 HNO₃ → 4 NO₂ + 2 H₂O + O₂
2. Oxidation of silicon by NO₂: 2 NO₂ + Si → SiO₂ + 2 NO
3. Etching of SiO₂: SiO₂ + 6 HF → H₂SiF₆ + 2 H₂O

with the formula of the overall reaction:

4 HNO₃ + 2 Si + 12 HF → 4 NO + 6 H₂O + O₂ + 2 H₂SiF₆

The resulting hexafluorosilicic acid (H₂SiF₆) is stable in aqueous solution.

Etching Rates of Silicon

Fig. 122 shows the rate of etching of crystalline silicon in different HF : HNO₃ mixtures at room temperature.

The etch rate drops towards zero when either the HF or HNO₃ concentration becomes very low, since in pure HF no SiO₂ forms which can be etched in HF, and HNO₃ only oxidises the Si without etching it.

An accurate control of the etching rate requires temperature accuracy within ± 0.5°C. A dilution with acetic acid improves the wetting of the hydrophobic Si-surface and thus increases the spatial homogeneity of the etch rate.

Doped (n- and p-type) silicon exhibits a higher etching rate than undoped silicon.

Etch Selectivity of Si : SiO₂

As the etching triangle in Fig. 123 shows, high HF : HNO₃ ratios promote rate-limited etching (strong temperature dependency of the etch rate) of Si via the oxidation step.

Low HF : HNO₃ ratios promote diffusion-limited etching (lower temperature dependency of the etch rate). Pure HF does not attack silicon, pure HNO₃ only results in an oxidation of its surface.

The SiO₂ etch rate is determined by the HF-concentration, since the oxidation does not play a role.

Etching of SiO₂ with HF or BHF

Hydrofluoric Acid

Hydrofluoric acid (HF) is the only wet-chemical medium with which SiO₂ can be isotropically etched at a reasonable rate. Due to the high toxicity of concentrated HF, one has to consider the concentration that is really required for each individual application. 1 % HF is sufficient for re-
moving native SiO$_2$ in a so-called HF-Dip, and even 200 - 300 nm oxide can be etched in 10 % HF or buffered HF in a reasonable amount of time. We supply 1 %, 10 % and 50 % HF in VLSI-quality.

**Buffered Hydrofluoric Acid**

The etching of Si and SiO$_2$ consumes F-ions via the reaction SiO$_2$ + 4 HF → SiF$_4$ + 2 H$_2$O. HF buffered with ammonia fluoride (BHF = NH$_4$F + H$_2$O + HF):

- maintains the free F$^-$ ion concentration via NH$_4$F + H$_2$O $\rightarrow$ H$_3$O$^+$ + F$^-$ + NH$_3$ allowing a constant and controllable etch rate as well as spatial homogeneous etching,
- an increase in the etch rate (factor 1.5 - 5.0) by highly reactive HF$_2^-$ ions and
- an increase in the pH-value ($\rightarrow$ minor resist underetching and resist lifting).

Despite the increased reactivity, strongly buffered hydrofluoric acid has a pH-value of close to 7 and therefore may not be detected by chemical indicators. We offer buffered HF (BOE 7: 1 = AF 87.5 -12.5) in 2.5 L containers in VLSI quality optionally with or without surfactant for improved wetting and etching homogeneity.

**Etch Rates of SiO$_2$ in HF or BHF**

Compared to thermal oxide, deposited (e.g. CVD) SiO$_2$ has a higher etch rate due to its porosity; wet oxide a slightly higher etch rate than dry (thermal) oxide for the same reason, i.e. thermally via O$_2$ produced SiO$_2$. Phosphorus-doped SiO$_2$ etches faster than undoped SiO$_2$.

**Etching of Glasses**

Unlike SiO$_2$, glasses with various compositions show a strong dependency between their etch rate and additives in the etch. Such additives (e.g. HCl, HNO$_3$) dissolve surface films formed on the glass during etching, which are often chemically inert in HF and would stop or decelerate glass etching with pure HF. Therefore, such additives allow a continued etching at a constant and high rate. This allows one to increase the etch rate at a reduced HF-concentration (= increased stability against resist peeling).
## Inorganic Developers

**AZ® Developer** is based on sodium phosphate and –metasilicate, is optimized for minimal aluminum attack and is 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 is 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 is 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 diluted 1 : 3 - 1 : 7 with water, depending on whether a high development rate, or a high contrast is required.

**Metal Ion Free (TMAH-based) Developers**

AZ® 326 MIF is 2.38 % TMAH- (TetraMethylAmmoniumHydroxide) in water.

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## Our Photoresists: Application Areas and Compatibilities

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<td>Improved adhesion for wet etching, no focus on steep resist sidewalls</td>
<td>AZ® 1500</td>
<td>AZ® 1505</td>
<td>$0.5 - 1.0 \mu m$</td>
<td>AZ® 351B, AZ® 326 MIF, AZ® 726 MIF, AZ® Developer</td>
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<td>AZ® 1512 HS</td>
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<td>AZ® 1514 H</td>
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<td>AZ® 4500</td>
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<td>AZ® 4562</td>
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<td>Spray coating</td>
<td>AZ® P4000</td>
<td>AZ® P4110</td>
<td>$1.0 - 2.0 \mu m$</td>
<td>AZ® 351B, AZ® 326 MIF, AZ® 726 MIF, AZ® 826 MIF</td>
<td>AZ® 100 Remover, TechniStrip® P1316, P1331</td>
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<td>AZ® P4330</td>
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<td>AZ® P4620</td>
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<td>AZ® PL 177</td>
<td>AZ® PL 177</td>
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<td>AZ® 4999</td>
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<td>Dip coating</td>
<td>MC Dip Coating Resist</td>
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<td>AZ® 351B, AZ® 400K, AZ® 726 MIF, AZ® 826 MIF</td>
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<td>$2.0 - 5.0 \mu m$</td>
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<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>$0.7 - 1.5 \mu m$</td>
<td>AZ® 400K, AZ® 326 MIF, AZ® 726 MIF, AZ® 826 MIF</td>
<td>AZ® 100 Remover, TechniStrip® P1316, P1331</td>
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<td>AZ® ECI 3012</td>
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<td>AZ® 9200</td>
<td>AZ® 9245</td>
<td>$3.0 - 5.0 \mu m$</td>
<td>AZ® 351B, AZ® 326 MIF, AZ® 726 MIF, AZ® Developer</td>
<td>AZ® 100 Remover, TechniStrip® P1316, P1331</td>
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<td>AZ® 9260</td>
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<td>Elevate thermal softening point and high resolution for e. g. dry etching</td>
<td>AZ® 9701 MR</td>
<td>AZ® 701 MR (14 cPs)</td>
<td>$3.0 - 5.0 \mu m$</td>
<td>AZ® 400K, AZ® 326 MIF, AZ® 726 MIF, AZ® 826 MIF</td>
<td>AZ® 100 Remover, TechniStrip® P1316, P1331</td>
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<td>AZ® 701 MR (29 cPs)</td>
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<td>$1.0 - 2.0 \mu m$</td>
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<td><strong>Positive (chem. amplified)</strong></td>
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<td>Steep resist sidewalls, high resolution and aspect ratio for e. g. dry etching or plating</td>
<td>AZ® XT</td>
<td>AZ® 12 XT-20PL-05</td>
<td>$3.0 - 4.0 \mu m$</td>
<td>AZ® 351B, AZ® 326 MIF, AZ® 726 MIF, AZ® Developer</td>
<td>AZ® 100 Remover, TechniStrip® P1316, P1331</td>
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<td>AZ® 12 XT-20PL-10</td>
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<td>AZ® 40 XT</td>
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<td>AZ® IPS 6050</td>
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<td>Elevated thermal softening point and undercut for lift-off applications</td>
<td>AZ® 5200</td>
<td>AZ® 5209</td>
<td>$1.0 - 2.0 \mu m$</td>
<td>AZ® 351B, AZ® 326 MIF, AZ® 726 MIF</td>
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<td>AZ® 5214</td>
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<td>Ti</td>
<td>Ti 35ESX</td>
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<td>Ti xLift-X</td>
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<td>Negative resist sidewalls in combination with no thermal softening for lift-off application</td>
<td>AZ® nLOF 2000</td>
<td>AZ® nLOF 2020</td>
<td>$1.5 - 3.0 \mu m$</td>
<td>AZ® 326 MIF, AZ® 726 MIF, AZ® 826 MIF</td>
<td>TechniStrip® NiS55, P1316, P1331, MLO 07</td>
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<td>AZ® nLOF 2035</td>
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<td>AZ® nLOF 2070</td>
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<td>AZ® nLOF 5550</td>
<td>$0.7 - 1.5 \mu m$</td>
<td>AZ® 326 MIF, AZ® 726 MIF, AZ® 826 MIF</td>
<td>TechiniStrip® P1316, P1331, P1331, MLO 07, P1331</td>
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<tr>
<td>Improved adhesion, steep resist sidewalls and high aspect ratios for e. g. dry etching or plating</td>
<td>AZ® nXT</td>
<td>AZ® 15 nXT (151 cPs)</td>
<td>$2.0 - 3.0 \mu m$</td>
<td>AZ® 326 MIF, AZ® 726 MIF, AZ® 826 MIF</td>
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<td>AZ® 15 nXT (450 cPs)</td>
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<td>AZ® 125 nXT</td>
<td>$2.0 - 100 \mu m$</td>
<td>AZ® 326 MIF, AZ® 726 MIF, AZ® 826 MIF</td>
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</tbody>
</table>

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**Inorganic Developers**

In general, almost all resists can be used for almost any application. However, the special properties of each resist family makes them specially suited for certain fields of application.

In general, metal ion free (MIF) developers are significantly more expensive, and reasonable if metal ion free development is required.
Our Silicon-, Quartz-, Fused Silica and Glass Wafers 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.

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 Prime-, Test- and Dummy Wafers besides the crystal orientation (<100> or <111>) the doping (n- or p-type) as well as the resistivity (Ohm cm) are selection criteria.

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

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, polyimides 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 alkali 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® N1555 is a remover with very strong stripping 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® N1555 was developed not only to peel cross-linked resists, but also to dissolve them without residues. This prevents contamination of the basin and filter by resist particles and skins, as can occur with standard strippers. TechniStrip® N1555 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™ NF52 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 photore sist 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 Czechralski- (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 monocristalline 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, besides 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!

Disclaimer of Warranty & Trademarks

All information, process descriptions, recipes, etc. contained in this document are compiled to the best of our knowledge. Nevertheless, we can not guarantee the correctness of the information. Particularly with regard to the formulations for chemical (etching) processes we assume no guarantee for the correct specification of the components, the mixing conditions, the preparation of the batches and their application.

The safe sequence of mixing components of a recipe usually does not correspond to the order of their listing. We do not warrant the full disclosure of any indications (among other things, health, work safety) of the risks associated with the preparation and use of the recipes and processes. The information in this book is based on our current knowledge and experience. Due to the abundance of possible influences in the processing and application of our products, they do not exempt the user from their own tests and trials. A guarantee of certain properties or suitability for a specific application can not be derived from our data. As a matter of principle, each employee is required to provide sufficient information in advance in the appropriate cases in order to prevent damage to persons and equipment. All descriptions, illustrations, data, conditions, weights, etc. can be changed without prior notice and do not constitute a contractually agreed product characteristics. The user of our products is responsible for any proprietary rights and existing laws.

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MicroChemicals GmbH
Nicolaus-Otto-Str. 39
89079, Ulm
Germany
Fon: +49 (0)731 977 343 0
Fax: +49 (0)731 977 343 29
e-Mail: info@microchemicals.net
Internet: www.microchemicals.net