WET-CHEMICAL ETCHING OF METALS

In microelectronic and mechanical components, various metals are used due to their respective electrical, optical, chemical or mechanical properties. The elements aluminium, chromium, gold and copper, which can also be wet-chemically structured, are particularly common.

This chapter describes the specifics of the etching of these metals with different etching mixtures also with regard to the processing of a resist mask used for it. All substances marked with an asterisk (*) in this chapter refer to the commonly available concentration of the respective substance as listed in the last section.

Etching of Aluminium

Properties and Application Areas of Aluminium

Aluminium has a density of 2.7 g/cm^3 and therefore belongs to the light metals. Its crystal structure is cubic face centred. Due to its high electric conductivity, aluminium is used for conductors in microelectronics where it is often alloyed with copper in order to prevent electro-migration, or with silicon in order to prevent the formation of (silicon-consuming) aluminium-silicon alloys.

With a standard potential of -1.66 V, aluminium does not belong to the noble metals. However, the formation of a very thin (few nm) Al_2O_3 film makes it very inert in many substances.

Aluminium Etchants

Typical aluminium etchants contain 1 - 5 % HNO_3^* (for Al oxidation), 65 - 80 % $H_3PO_4^*$ for etching the native aluminium oxide as well as oxide steadily newly formed by the HNO_3, acetic acid to improve the wetting of the substrate with the etching solution, as well as for the buffering of the nitric acid and water to adjust the etching rate at a given temperature.

Aluminium can also be etched with alkaline liquids, e.g. with diluted sodium or potassium hydroxide. However, photoresist masks are not suitable for this since the correspondingly high pH value dissolves the resist film layer in a short time or can peel it off in the case of cross-linked negative resists.

Homogeneity of Al Etching

The actual aluminium etching starts when, for example, using phosphoric acid as a component of typical Al-etching mixtures, dissolves a few nm thick native aluminium oxide film present on aluminium surfaces. For this reason, photoresist processing also impacts a subsequent Al etching step:

The alkaline developers preferentially dissolve the native aluminium oxide layer where the resist is primarily developed. These include thinner resist areas, laterally larger exposed areas or areas which, e.g. due to diffraction or from an inhomogeneous exposure, received a higher exposure dose. Dependant on the extent of (desired or undesired) over-developing as well as any delay between development and Al-etching, the process parameters may lead to a laterally inhomogeneous Al etching start and thus to different etching depths or times (Fig. 118).

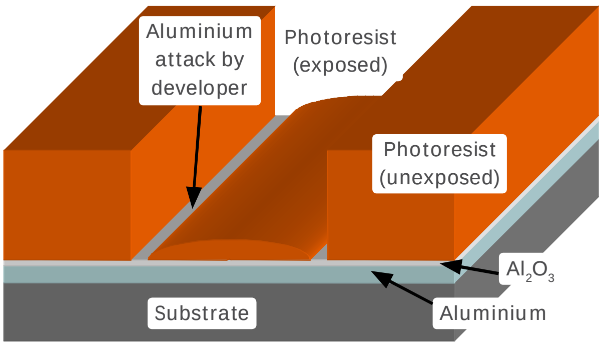


Fig. 118: Alkaline developers attack the native oxide on aluminium layers where the resist is first developed. There, the later, actual Al-etching can begin more quickly than at the areas which are protected longer by the resist film during development.

The formation of hydrogen in the etching reaction is also problematic for a homogeneous etching result. The constantly produced H_2 bubbles stick to the surface and block the etching process through a suppressed supply of fresh etching medium. In this case, it can help to interrupt the etching process several times by a brief dipping in water, which at least temporarily removes the H_2 bubbles.

Compatibility and Selectivity

All of our photoresists are sufficiently stable in H_3PO_4/HNO_3-based etching mixtures to be used as a resist mask, at least after a sufficient hardbake.

The etching rate of aluminium etching based on H_3PO_4/HNO_3 mixtures is strongly temperature-dependent and doubles every few degrees of temperature rise. Aluminium alloyed with only a few percent silicon has a similar etching rate as pure aluminium.

Copper is etched by this Al-etchant much more greatly than aluminium, nickel is comparatively weakly etched. Titanium, chromium and silver are not noticeably etched, precious metals such as gold, platinum are not etched at all.

Our Aluminium Etchant

Our aluminium etchant *TechniEtch Al80* has the composition $H_3PO_4^*$: HNO_3^* : CH_3COOH^* : H_2O = 80 %: 5 % : 5 % : 10% comes in 2.5 L containers in MOS quality.

Etching of Chromium

Properties and Application Areas of Chromium

Chromium is used in the field of microstructuring for the production of photo masks as well as adhesion promoters for the subsequently applied metal films because of its high hardness and good adhesion to many materials.

Chromium Etchants

Chromium etchants are usually based on ceric ammonium nitrate $(NH_4)_2[Ce(NO_3)_6]$ with perchloric acid ($HClO_4$) as an optional additive. Perchloric acid is almost completely dissociated as an extremely strong acid in aqueous solution (P_{KS} value < -8) and serves as a very strong oxidising agent for the stabilisation of the ceric ammonium nitrate. Ceric ammonium nitrate itself is a very powerful oxidising agent.

The sum formula for the etching of chromium with ceric ammonium nitrate and perchloric acid is

$$3\,(NH_4)_2Ce(NO_3)_6 + Cr \rightarrow Cr(NO_3)_3 + 3(NH_4)_2Ce(NO_3)_5$$

according to which the cerium is reduced from the oxidation stage IV to III and the chromium is oxidised to oxidation stage III. The chromium nitrate, which during the etching process forms a dark, constantly new formation on the chromium layer, is very soluble in water and thus in the chromate etchants.

Compatibility and Selectivity

All of our photoresists are sufficiently stable in ceric ammonium nitrate and perchloric acid-based etching mixtures to be used as resist masks.

Copper, silver and vanadium are strongly etched by this etching mixture. Aluminium, titanium, tungsten and nickel experience only a weak etching. The noble metals gold, platinum and palladium are not etched. Experience has shown that with copper in (electrical) contact with chromium, a greatly reduced etching rate of chromium can occur.

Our Chromium Etchants

Our chromium etchant *TechniEtch Cr01* consists of ceric ammonium nitrate : Perchloric acid : water = 10.9 % : 4.25 % : 84.85 % with an etching rate of about 60 nm/minute at room temperature comes in 2.5 L containers in VLSI quality.

Etching of Gold

Properties and Application Areas of Gold

Gold is a metal of very high density of 19.3 g/cm^3, its crystal structure is cubic face centred. With a standard potential +1.5, gold belongs to the noble metals. The electron configuration [Xe] $4f^{14}5\,d^{10}6\,s^1$ strongly prevents the oxidation of gold: The completely occupied 5d orbital extends beyond the single valence electron which hereby is well shielded against any reaction partners.

Wet chemical etching of gold therefore requires a strong oxidiser for the separation of the unpaired valence electron, as well as a complexing agent which suppresses the reassembly of oxidised gold atoms back into the crystal.

By virtue of this high chemical stability against most acids and bases, gold is used in microelectronics as a material for electrical contacts or their protection.

Gold Etching with HCl/HNO_3

Mixtures of nitric acid and hydrochloric acid (in a mixing ration of 1 : 3 also called *aqua regia* are able to etch gold at room temperature. The very strong oxidative effect of this mixture stems from the formation of nitrosyl chloride (NOCl) via the reaction $HNO_3 + 3\ HCl \rightarrow NOCl + 2\ Cl + 2\ H_2O$, while free Cl radicals formed in the solution keep the noble metal dissolved as Cl-complex (chloroauric acid = $HAuCl_4$). Aqua regia consumes itself and decomposes under formation of nitrous gases and chlorine gas.

The etch rate of aqua Regia for gold is approx. 10 µm/min (at room temperature) and can be increased to several 10 µm/min at elevated temperatures.

Palladium, aluminium, copper and molybdenum are also etched at room temperature in aqua regia. For etching platinum or rhodium, the etching solution has to be heated to attain a reasonable etch rate. Etching of iridium requires strongly heated (boiling) aqua regia.

Silver is not attacked by aqua regia due to the formation of a silver chloride passivation film. Chromium, titanium, tantalum, zirconium, hafnium and niobium also form a very stable passivationfilm (in many cases, the metal oxide) protecting the metal against the attack of aqua regia at least at room temperature. For same reason, tungsten reveals a very slow etch rate in aqua regia.

Gold Etching with KI/I_2

Gold and iodine form gold iodide via $2\ Au + I_2 \rightarrow 2\ AI$. The solubility of AI is improved by adding KI to the solution. Iodine/iodide can be substituted by other halogenides excepting fluorine which does not form soluble gold compounds.

In a mixing ratio $KI : I_2 : H_2O$ = 4 g : 1 g : 40 ml, a room temperature etch rate of approx. 1 µm/min gold is attained. Copper reveals a comparable etch rate, while nickel is only etched when in contact with gold.

Gold Etching with Cyanides

Aqueous solutions of the very toxic sodium cyanide (NaCN) or, respectively, the also very toxic potassium cyanide (KCN) dissolve gold via the formation of the soluble cyano-complex $[Au(CN)_2]$. This reaction requires oxygen from the air or supplied by decomposing hydrogen peroxide H_2O_2 added to the etching solution.

In addition to gold, cyanide solutions etch, for example, silver and copper, which also form water soluble, cyano-complexes.

Our Gold Etch ACI2

Our ready-to-use gold etchant solution *TechniEtch* ACI_2 is based on a potassium iodide/iodine solution which we offer in 2.5 L containers in VLSI quality.

Copper Etching

Properties and Application Areas of Copper

Due to its high electrical conductivity and its lower cost compared to silver, copper is widely used as a material for conductors in microelectronics as well as in printed circuit boards. Due to the lack of possibility of dry-chemical structuring by means of plasma etching, wet-chemical etching methods have to be used for this purpose, if necessary, in combination with a subsequent fortification by electroplating.

Copper Etching

Copper is etched by (also diluted) nitric acid as well as saturated 30% $Fe(Cl)_3$ solution.

Mixtures of NH_4OH and H_2O_2 etch copper as well.

Nickel Etching

Properties and Application Areas of Nickel

The transition metal nickel with a density of 8.9 g/cm³ is one of the heavy metals. Its crystal structure is cubic face centred. Due to its hardness and high chemical resistance, nickel coatings are used as corrosion protection of surfaces against chemical and mechanical attack.

Nickel Etching

Oxidising acids coat nickel with a passivating oxide layer, which prevents further etching. For this reason, nickel etching mixtures require a medium which is able to dissolve the initially present as well as the constantly forming oxide, as well as an oxidiser.

Like titanium, nickel can be etched using H_2O_2 (for the oxidation of Ni) and HF (dissolution of oxide). As an oxidiser, nitric acid and instead of the HF, hydrochloric acid can be used. An aqueous 30% $Fe(Cl)_3$ solution also etch nickel.

Silver Etching

Properties and Application Areas of Silver

The noble metal silver's crystal structure is cubic face centred. Silver has the highest electrical conductivity of all metals and is used in microelectronics as a material for conductors, where the requirement for maximum electrical conductivity warrants the higher material costs as compared to copper.

Silver Etching

The corresponding etching solutions require a component which oxidises silver, and a further one that dissolves silver oxide.

In addition to the KI / I_2 / H_2O etching solution described in the section Gold etching, silver is also etched by NH_4OH^* : $H_2O_2^*$: methanol = 1 : 1 : 4. The toxic methanol is not a mandatory component and can be omitted with a loss in etching uniformity or replaced by water. A further etching mixture for silver is an aqueous solution of HNO_3^* : HCl^* : H_2O = 1 : 1 : 1.

Titanium Etching

Properties and Application Areas of Titanium

Titanium is often used as a very hard and corrosion-resistant metal in microstructuring as an adhesive layer between the substrate and metal films deposited on top of it. As separating layer between silicon and aluminium, it serves as a barrier against the diffusion of Si in Al in order to prevent the so-called "aluminium spiking" where Al diffuses into spaces left by diffused Si and thereby may cause short circuits.

Titanium Etching

Titanium forms a very stable oxide layer on air to be etched by HF, which is thus often a component of titanium etchants. H_2O_2 is suitable for the oxidation of the underlying layers as a second component. In a mixing ratio of HF^* : $H_2O_2^*$: H_2O = 1 : 1 : 20, titanium can be etched at room temperature with approx. 1 µm/min.

Standard Concentrations

All concentration data of all substances mentioned in this chapter and marked with a (*) refer to following basic concentrations:

HCl^* = 37 % HCl in H_2O	HNO_3^* = 70 % HNO_3 in H_2O
$H_2SO_4^*$ = 98 % H_2SO_4 in H_2O	HF^* = 50 % HF in H_2O
$H_2O_2^*$ = 30 % H_2O_2 in H_2O	$H_3PO_4^*$ = 85 % H_3PO_4 in H_2O
NH_4OH^* = 29 % NH_3 in H_2O	CH_3COOH^* = 99 % CH_3COOH in H_2O

Our Photoresists: Application Areas and Compatibilities

	Recommended Applications[1]	Resist Family	Photoresists	Resist Film Thickness[2]	Recommended Developers[3]	Recommended Removers[4]
Positive	Improved adhesion for wet etching, no focus on steep resist sidewalls	AZ® 1500	AZ® 1505 AZ® 1512 HS AZ® 1514 H AZ® 1518	≈ 0.5 µm ≈ 1.0 - 1.5 µm ≈ 1.2 - 2.0 µm ≈ 1.5 - 2.5 µm	AZ® 351B, AZ® 326 MIF, AZ® 726 MIF, AZ® Developer	AZ® 100 Remover, TechniStrip® P1316 TechniStrip® P1331
		AZ® 4500	AZ® 4533 AZ® 4562	≈ 3 - 5 µm ≈ 5 - 10 µm	AZ® 400K, AZ® 326 MIF, AZ® 726 MIF, AZ® 826 MIF	
		AZ® P4000	AZ® P4110 AZ® P4330 AZ® P4620 AZ® P4903	≈ 1 - 2 µm ≈ 3 - 5 µm ≈ 6 - 20 µm ≈ 10 - 30 µm	AZ® 400K, AZ® 326 MIF, AZ® 726 MIF, AZ® 826 MIF	
		AZ® PL 177	AZ® PL 177	≈ 3 - 8 µm	AZ® 351B, AZ® 400K, AZ® 326 MIF, AZ® 726 MIF, AZ® 826 MIF	
	Spray coating	AZ® 4999		≈ 1 - 15 µm	AZ® 400K, AZ® 326 MIF, AZ® 726 MIF, AZ® 826 MIF	
	Dip coating	MC Dip Coating Resist		≈ 2 - 15 µm	AZ® 351B, AZ® 400K, AZ® 326 MIF, AZ® 726 MIF, AZ® 826 MIF	
	Steep resist sidewalls, high resolution and aspect ratio for e. g. dry etching or plating	AZ® ECI 3000	AZ® ECI 3007 AZ® ECI 3012 AZ® ECI 3027	≈ 0.7 µm ≈ 1.0 - 1.5 µm ≈ 2 - 4 µm	AZ® 351B, AZ® 326 MIF, AZ® 726 MIF, AZ® Developer	
		AZ® 9200	AZ® 9245 AZ® 9260	≈ 3 - 6 µm ≈ 5 - 20 µm	AZ® 400K, AZ® 326 MIF, AZ® 726 MIF	
	Elevated thermal softening point and high resolution for e. g. dry etching	AZ® 701 MiR	AZ® 701 MiR (14 cPs) AZ® 701 MiR (29 cPs)	≈ 0.8 µm ≈ 2 - 3 µm	AZ® 351B, AZ® 326 MIF, AZ® 726 MIF, AZ® Developer	
Positive (chem. amplified)	Steep resist sidewalls, high resolution and aspect ratio for e. g. dry etching or plating	AZ® XT	AZ® 12 XT-20PL-05 AZ® 12 XT-20PL-10 AZ® 12 XT-20PL-20 AZ® 40 XT	≈ 3 - 5 µm ≈ 6 - 10 µm ≈ 10 - 30 µm ≈ 15 - 50 µm	AZ® 400K, AZ® 326 MIF, AZ® 726 MIF	AZ® 100 Remover, TechniStrip® P1316 TechniStrip® P1331
		AZ® IPS 6050		≈ 20 - 100 µm		
Image Re-versal	Elevated thermal softening point and undercut for lift-off applications	AZ® 5200	AZ® 5209 AZ® 5214	≈ 1 µm ≈ 1 - 2 µm	AZ® 351B, AZ® 326 MIF, AZ® 726 MIF	TechniStrip® Micro D2 TechniStrip® P1316 TechniStrip® P1331
		TI	TI 35ESX TI xLift-X	≈ 3 - 4 µm ≈ 4 - 8 µm		
Negative (Cross-linking)	Negative resist sidewalls in combination with no thermal softening for lift-off application	AZ® nLOF 2000	AZ® nLOF 2020 AZ® nLOF 2035 AZ® nLOF 2070	≈ 1.5 - 3 µm ≈ 3 - 5 µm ≈ 6 - 15 µm	AZ® 326 MIF, AZ® 726 MIF, AZ® 826 MIF	TechniStrip® NI555 TechniStrip® NF52 TechniStrip® MLO 07
		AZ® nLOF 5500	AZ® nLOF 5510	≈ 0.7 - 1.5 µm		
	Improved adhesion, steep resist side-walls and high aspect ratios for e. g. dry etching or plating	AZ® nXT	AZ® 15 nXT (115 cPs) AZ® 15 nXT (450 cPs)	≈ 2 - 3 µm ≈ 5 - 20 µm	AZ® 326 MIF, AZ® 726 MIF, AZ® 826 MIF	
			AZ® 125 nXT	≈ 20 - 100 µm	AZ® 326 MIF, AZ® 726 MIF, AZ® 826 MIF	TechniStrip® P1316 TechniStrip® P1331 TechniStrip® NF52 TechniStrip® MLO 07

[1] 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.
[2] Resist film thickness achievable and processable with standard equipment under standard conditions. Some resists can be diluted for lower film thicknesses; with additional effort also thicker resist films can be achieved and processed.
[3] Metal ion free (MIF) developers are significantly more expensive, and reasonable if metal ion free development is required.

Our Developers: Application Areas and Compatibilities

Inorganic Developers

(typical demand under standard conditions approx. 20 L developer per L photoresist)

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

(typical demand under standard conditions approx. 5 - 10 L developer concentrate per L photoresist)

AZ® 326 MIF is 2.38 % TMAH- (TetraMethylAmmoniumHydroxide) 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 (residues 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, 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 alkaline sensitive materials, TechniStrip® P1331 would be an alternative to the P1316. Nicht kompatibel mit Au oder GaAs.

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

TechniStrip® NI555 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® NI555 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® NI555 is not compatible with Au or 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 photoresist 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 SiO_2, main criterion is the crystal orientation (e. g. X-, Y-, Z-, AT- or ST-cut)

Fused silica wafers consist of amorphous SiO_2. 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: è www.microchemicals.com/products/wafers/waferlist.html

Further Products from our Portfolio

Plating

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

Solvents (MOS, VLSI, ULSI)

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

Acids and Bases (MOS, VLSI, ULSI)

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

Etching Mixtures

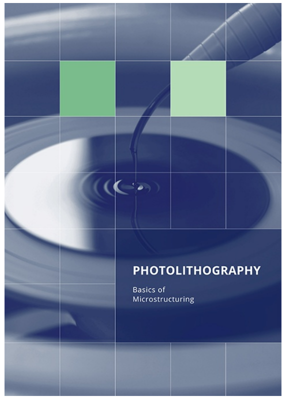
for e. g. chromium, gold, silicon, copper, titanium, ... è www.microchemicals.com/products/etching_mixtures.html

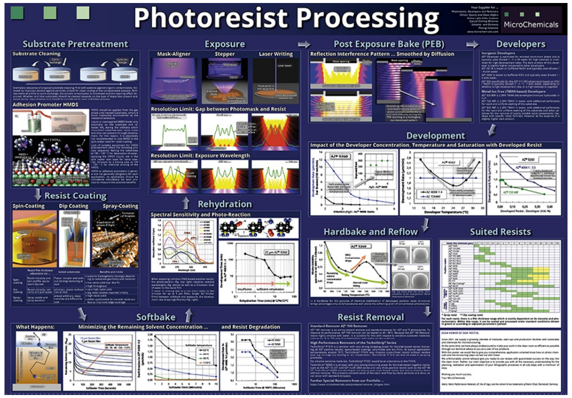
Further Information

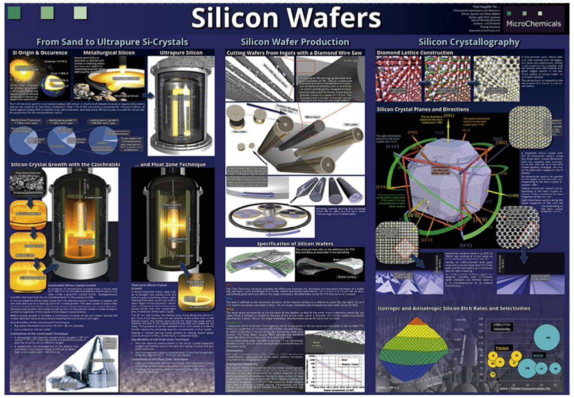
Technical Data Sheets: www.microchemicals.com/downloads/product_data_sheets/photoresists.html

Material Safety Data Sheets (MSDS): www.microchemicals.com/downloads/safety_data_sheets/msds_links.html

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):

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